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THESE

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Microwear and residue analyses of quartzite stone tools. Experimental development of a method and its application to the assemblages from the Pleistocene sites of Gran Dolina-TD10 (Sierra de Atapuerca, Burgos, Spain) and Payre (Ardèche, France)

Sous la direction de **: Andreu Ollé Cañellas** et **Marie-Hélène Moncel**

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UNIVERSITAT ROVIRA I VIRGILI

I STATE that the present study, entitled "**Microwear and residue analyses of quartzite stone tools. Experimental development of a method and its application to the assemblages from the Pleistocene sites of Gran Dolina-TD10 (Sierra de Atapuerca, Burgos, Spain) and Payre (Ardèche, France)**", presented by **ANTONELLA PEDERGNANA** for the award of the degree of Doctor, has been carried out under my supervision at the Department D'HISTÒRIA I HISTÒRIA DE L'ART of this university.

[Tarragona [data] /

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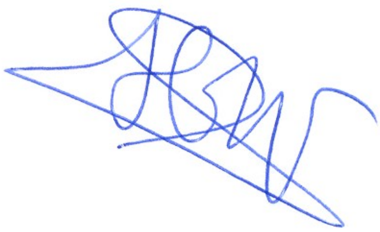

Doctoral Thesis

ANTONELLA PEDERGNANA

Microwear and residue analyses of quartzite tools:

Experimental development of a method and its application to the assemblages from the Pleistocene sites of Gran Dolina-TD10 (Sierra de Atapuerca, Burgos, Spain) and Payre (Ardèche, France)

directed by Dr. Andreu Ollé Cañellas and Dr. Marie Hélène Moncel

in a joint PhD programme with the Muséum National d'Histoire Naturelle of Paris

VOLUME I

UNIVERSITAT ROVIRA I VIRGILI
Departament d'Historia i Historia del'Art

MNHN

Looking first at the material apparatus of culture, we can say that every artefact is either an implement or else an object of more direct use, that is, belonging to the class of consumers' goods. In either case, the circumstances as well as the form of the object are determined by its use. Function and form are related.

Malinowski, 1944

Tem duas formas, ou modos, o que chamamos cultura. Não é a cultura senão o aperfeiçoamento subjectivo da vida. Esse aperfeiçoamento é direto ou indireto: ao primeiro se chama arte, ciência ao segundo. Pela arte nos aperfeiçoamos a nós; pela ciência aperfeiçoamos em nós o nosso conceito, ou ilusão, do mundo.

Fernando Pessoa, 1924

I love fools' experiments. I am always making them.

Charles Darwin

Acknowledgements

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- the projects CGL2015-65387-C3-1-P (Comportamiento ecosocial de los homínidos de la Sierra de Atapuerca durante el Cuaternario III) (Spanish Ministerio de Economía y Competitividad) and SGR 2014-899 (AGAUR, Generalitat de Catalunya) contributed with regular help during all the stages of this research;
- Fieldwork in Atapuerca is founded by the Junta de Castilla y León and the Fundación Atapuerca.

This thesis was developed under a joint supervision arrangement (*cotutelle*) between the Rovira i Virgili University (Tarragona, Spain)-IPHES (Institut Català de Paleoecologia Humana i Evolució Social) and the Muséum National d'Histoire Naturelle (MNHN)- Institut de Paléontologie Humaine (IPH) (Paris, France). I would like to acknowledge all the people who made possible the enrolment of this thesis in this programme. In particular, the responsible for the Mérimée projects, Dr. Robert Sala Ramos, Dr. Carlos Lorenzo Merino and Dr. Dominique Grimaud-Hervé. I am also grateful for the administrative help I received from Marina Vives i Cabré and Chafika Falgueres.

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To Dr. Marie Hélène Moncel, *du côté français*, for having helped me so much with incredible efficiency in what concerns all the administrative obligations to render this *cotutelle* a reality. Also, for providing me access to the archaeological collection of Payre and for being always present during my stays in Paris. Her feedbacks on my work were of inestimable value, as well as her constant support during the preparation of some of my papers.

Remembering all steps of this long trip, there are many people who allowed this research to be developed. I apologise if I forget to mention some of them.

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Finally, I thank my family for the constant support throughout all the years of my research and for understanding my absence.

And thanks to “my mountains” which are always there to welcome and cherish me, even if the summit of the highest one, embodied by this thesis, has finally been reached by always remembering:

“If you have a high-way on Everest, you don’t meet the mountain. If everything is prepared and you have a guide who is responsible for your security, you cannot meet the mountain. Meeting mountains is only possible if you are out there in self-sufficiency!”

Reinhold Messner, mountaineer

Abstract

This thesis derives from the need for a robust experimental set of data regarding use-wear on quartzite. Are the methods developed for the analysis of flint appropriate to analyse coarse-grained materials like quartzite?

To assess this question, it was necessary to develop a strong experimental programme, including different actions (movements, elapsed time, etc.) as well as worked materials. Several varieties of quartzite (4) compose this collection, the main reason for this being the assessment of the internal use-wear variability. Hence, this thesis is the result of a big effort coinciding with the construction of an experimental use-wear collection for this rock type.

Also, with the same need of producing reliable experimental collections to later infer functions of archaeological tools, morphological and elemental data was obtained from the analysis of experimental residues of the worked materials.

The methodology developed is strongly based on the systematic monitoring of sequential experiments and on the use of different microscopic techniques (mainly Optical Microscopy and SEM/EDX but also occasionally Confocal Microscopy), with the main aim being to understand the way wear originates and propagates on quartzite surfaces. The systematic comparison of OLM and SEM micro-graphs is also the base of our characterisation of residues.

The methodology developed was then tested on two archaeological samples from the Middle Pleistocene site of Gran Dolina (Burgos, Spain) and the Late Pleistocene site of Payre (Southern France). Use-wear results were then obtained from the study of the two collections, while no significant evidence regarding micro-residues was obtained.

Since the function of stone tools is the main concern of microwear analysists, a deeper understanding of the object itself is needed. If use-wear results are not accompanied by technological data, only a limited part of the information hidden within prehistoric tools is attained. This is why we applied techno-functional analysis on the quartzite flakes and retouched flakes of the Gran Dolina-TD10.1 assemblage. Results coming from the two types of analysis have then been crossed and interesting insights have come out.

Eight publications form the backbone of the thesis, the first one being an introduction of the research as a whole; the second, third and fifth gather the experimental use-wear evidence on quartz and quartzite; the fourth discusses the issue of contamination in lithic residue analysis; the sixth and eighth present the experimental data of the characterisation of micro-residues; finally, the seventh contains preliminary data of the use-wear study of the quartzite assemblage from Payre.

Key-words: quartzite, use-wear analysis; residue analysis; Optical Light Microscopy; Scanning Electron Microscopy; Energy Dispersive X-rays spectroscopy.

Résumé

Cette thèse a comme objectif de montrer la nécessité d'avoir un ensemble rigoureux de données expérimentales pour identifier des traces d'utilisation sur le quartzite. Notre question est : les méthodes développées pour l'analyse tracéologique du silex sont-elles appropriées pour analyser des matériaux à gros grains tel que le quartzite ?

Pour répondre à cette question, il nous a fallu mettre en œuvre un solide programme expérimental, incluant des activités (mouvements, durée, etc…) et des matériaux travaillés variés. Plusieurs types de quartzite ont été utilisés dans ce référentiel, principalement pour comprendre la variabilité des traces d'utilisation. Ce travail de thèse est le résultat d'un grand effort afin de constituer une collection de référence de traces d'usures sur cette roche.

De plus, dans le but d'obtenir des collections de référence fiables pour pouvoir interpréter le matériel archéologique, des données sur la morphologie et sur la composition élémentaire des micro-résidus ont été obtenues.

La méthodologie développée repose sur une observation systématique des expérimentations et sur le recours à différentes techniques de microscopie (essentiellement microscopie optique et MEB/EDX, et occasionnellement microscopie confocale), avec pour objectif principal de comprendre la formation des traces d'usure et leur développement sur la surface du quartzite. Une comparaison systématique entre les micro-graphiques du microscope optique et du MEB fonde notre caractérisation des résidus.

La méthodologie développée a été testée sur deux échantillons archéologiques provenant du site du Pléistocène Moyen de Gran Dolina (Burgos, Espagne) et de Payre (Sud-est de la France). Des résultats tracéologiques ont été obtenus pour l'étude de ces deux collections. En revanche aucun résultat significatif n'a été fourni par l'analyse des résidus.

Puisque la fonction des artefacts lithiques est l'intérêt principal des tracéologues, nous avons considéré qu'il fallait arriver à une compréhension profonde de l'objet lithique. Si l'analyse tracéologique n'est pas accompagnée de données technologiques, seule une partie limitée de l'information sur la fonction des instruments préhistoriques est atteinte. Pour cette raison, nous avons appliqué également une analyse techno-fonctionnelle sur l'ensemble des éclats bruts et retouchés de Gran Dolina-TD10.1. Les résultats provenant des deux types d'analyse ont, ensuite, été croisés et des considérations très intéressantes ont pu être obtenues.

Huit publications forment la trame de cette thèse : la première est une introduction à la recherche présentée ; les deuxième, troisième et cinquième exposent les données expérimentales sur le quartz et le quartzite ; le quatrième discute le problème de la contamination dans l'analyse des résidus ; les sixième et huitième présentent les données expérimentales sur la caractérisation des micro-résidus ; enfin, le septième contient des données préliminaires sur l'étude tracéologique de pièces archéologiques de Payre.

Mots-clés : quartzite ; analyse tracéologique ; analyse des résidus ; Microscopie optique ; Microscopie électronique à balayage ; Spectroscopie à rayons X à dispersion d'énergie.

Resumen

Este trabajo de tesis nace de la necesidad de disponer de un sólido conjunto de datos experimentales de referencia sobre huellas de uso en cuarcita. ¿Son los métodos desarrollados para analizar el sílex apropiados para el análisis de rocas de grano grueso como la cuarcita?

Para responder a esta cuestión resultaba necesario el desarrollo de un programa experimental completo que incluyese distintas acciones (movimientos, tiempos, etc.) y también diferentes materiales trabajados. Con el objetivo de evaluar la variabilidad interna de huellas de uso, la colección se compone de distintas variedades de cuarcita (4). Por lo tanto, esta tesis es el resultado de la inversión de un gran esfuerzo en la elaboración de una colección experimental de huellas de uso para este tipo de roca.

Paralelamente, y con el mismo objetivo de producir colecciones experimentales fidedignas para después inferir las funciones de los instrumentos líticos, se obtuvieron también datos experimentales sobre la morfología y composición elemental de los residuos de los materiales trabajados.

La metodología desarrollada en este trabajo se basa principalmente en el control sistemático de experimentos secuenciales y en el uso de distintas técnicas microscópicas (principalmente microscopía óptica y MEB/EDX, pero ocasionalmente también microscopía confocal), siendo el objetivo principal la monitorización y comprensión de los procesos de formación de las huellas de uso en la cuarcita. Asimismo, la base de nuestra caracterización de residuos se presentó como la comparación sistemática de las imágenes obtenidas con microscopía óptica y MEB.

Esta metodología fue posteriormente testada en dos muestras arqueológicas del yacimiento del Pleistoceno Medio de Gran Dolina (Burgos, España) y del yacimiento del Pleistoceno Superior de Payre (sur de Francia). Si bien se obtuvieron resultados funcionales en el estudio de estas dos colecciones, no se logró obtener datos significativos en cuanto al análisis de micro-residuos.

Pese a que en la mayoría de los casos la función de los instrumentos líticos es la mayor preocupación de los traceólogos, se hace evidente la necesidad de un entendimiento más profundo del objeto en sí mismo. Por ello, si los resultados funcionales no se acompañan de datos tecnológicos, solamente alcanzaremos una pequeña parte de la información retenida por los instrumentos prehistóricos. Por esta razón, aplicamos el análisis tecno-funcional a las lascas simples y lascas retocadas en cuarcita del conjunto recuperado en el nivel TD10.1 de Gran Dolina. Los resultados obtenidos con los dos tipos de análisis fueron posteriormente cruzados y surgieron interesantes ideas.

Ocho publicaciones forman la columna vertebral de la tesis: la primera es una introducción general a la investigación realizada; la segunda, tercera y quinta agrupan los datos experimentales obtenidos referentes a las huellas de uso en cuarzo y cuarcita; en la cuarta se discute la problemática de la contaminación en el análisis de residuos sobre elementos líticos; la sexta y la octava exponen los datos experimentales sobre la caracterización de micro-residuos; y, finalmente, la séptima publicación incluye datos preliminares provenientes del análisis funcional de la colección en cuarcita del yacimiento de Payre.

Palabras-clave: cuarcita; análisis de huellas de uso; análisis de residuos; Microscopía Óptica; Microscopía Electrónica de Barrido; Espectrometría de dispersión de energía de rayos X.

Resum

Aquest treball de tesi neix de la necessitat de disposar d'un conjunt sòlid de dades experimentals de referència sobre traces d'ús en quarsita. Els mètodes desenvolupats per analitzar el sílex són apropiats per l'anàlisi de les roques de gra gruixut com la quarsita?

Per respondre a la qüestió resulta necessari el desenvolupament d'un programa experimental complet que inclogui diferents accions (moviments, temps, etc.) i també diferents materials treballats. Amb l'objectiu d'avaluar la variabilitat de traces d'ús de la pròpia roca, la col·lecció es composa de distintes varietats de quarsita (4). Així doncs, aquesta tesi és el resultat d'un gran esforç invertit en l'elaboració d'una col·lecció experimental de deformacions d'us per aquest tipus de roca.

Paral·lelament, i amb el mateix objectiu de produir col·leccions experimentals fidedignes per després inferir les funcions dels instruments lítics, també es van obtenir dades experimentals sobre la morfologia i la composició elemental dels residus dels materials treballats.

La metodologia desenvolupada en aquest treball, doncs, es basa principalment en el control sistemàtic d'experiments seqüencials i en l'ús de diferents tècniques microscòpiques (principalment microscòpia òptica i MER/EDX, tot i que també ocasionalment microscòpia confocal), essent l'objectiu principal la documentació i comprensió dels processos de formació de les traces d'ús en la quarsita. Tanmateix, la base de la nostra caracterització de residus es va presentar com la comparació sistemàtica de les imatges obtingudes amb el microscopi òptic i amb el MER.

Aquesta metodologia ha estat posteriorment testada en dues mostres arqueològiques del jaciment del Plistocè Mitjà de Gran Dolina (Burgos, Espanya) i del jaciment del Plistocè Superior de Payre (sud de França). Si bé s'han obtingut resultats funcionals en l'estudi d'aquestes dues col·leccions, no ha estat possible, però, adquirir dades significatives referents a l'anàlisi de micro-residus.

Malgrat que en la majoria dels casos la funció dels instruments lítics és la principal preocupació dels traceòlegs, resulta evident la necessitat d'una comprensió més en profunditat de l'objecte en sí mateix. Per això, si els resultats funcionals no s'acompanyen de dades tecnològiques, únicament obtindrem una petita part de la informació continguda als instruments prehistòrics. Per aquesta raó, apliquem l'anàlisi tecno-funcional a les ascles simples i ascles retocades en quarsita del conjunt recuperat al nivell TD10.1 de Gran Dolina. Els resultats obtinguts amb els dos tipus d'anàlisi han estat posteriorment creuats, i s'han obtingut idees molt interessants.

Vuit publicacions formen la columna vertebral de la tesi: la primera és una introducció general a la investigació realitzada; la segona, la tercera i la cinquena agrupen les dades experimentals obtingudes referents a les deformacions d'ús en quars i quarsita; a la quarta es discuteix la problemàtica de la contaminació en l'anàlisi de residus sobre els elements lítics; la sisena i la vuitena exposen les dades experimentals sobre la caracterització de

micro-residus; i, finalment, la sèptima publicació inclou les dades preliminars provinents de l'anàlisi funcional de la col·lecció en quarsita del jaciment de Payre.

Paraules-clau: quarsita; anàlisi de traces d'ús; anàlisi de residus; microscòpia òptica; microscòpia electrònica de rastreig; espectrometria de dispersió d'energia de rajos X.

List of publications resulting from the thesis

1. **Publication 1:**

   Pedergnana, A., Ollé, A., 2014. **Use-wear and residues analyses on quartzite stone tools: setting up a methodology.** In: Lemorini, C., Nunziante, S. (Eds.), Proceeding of the international conference "An integration of use wear and residues analysis for the identification of the function of archaeological stone tools". *BAR International Series* 2649, Oxford, pp. 43-62.

2. **Publication 2:**

   Ollé, A., Pedergnana, A., Fernández-Marchena, J.L., Martin, S., Borel, A., Aranda, V., 2016. **Microwear features on vein quartz, rock crystal and quartzite: a study combining Optical Light and Scanning Electron Microscopy.** *Quaternary International* 424, 154-170.

3. **Publication 3:**

   Pedergnana, A., García-Antón, M.D., Ollé, A., 2017. **Structural study of two quartzite varieties from the Utrillas facies formation (Olmos de Atapuerca, Burgos, Spain): From a petrographic characterisation to a functional analysis design.** *Quaternary Interna*tional 433, 163-178.

4. **Publication 4:**

   Pedergnana, A., Asryan, L., Fernández-Marchena, J.L., Ollé, A., 2016. **Modern contaminants affecting microscopic residue analysis on stone tools: A word of caution.** *Micron* 86, 1-21.

5. **Publication 5:**

   Pedergnana, A., Ollé, A., 2017a. **Monitoring and interpreting the use-wear formation processes on quartzite flakes through sequential experiments**. *Quaternary International* 427(Part B), 35-65.

6. **Publication 6:**

   Pedergnana, A., Ollé, A., 2017b. **Building an experimental comparative reference collection for lithic micro-residue analysis based on a multi-analytical approach.** *Journal of Archaeological Method and Theory* (in press), DOI 10.1007/s10816-017-9337-z

7. **Publication 7:**

   Pedergnana, A., Ollé, A., Borel, A., Moncel, M.H., 2016. **Microwear study of quartzite artefacts: Preliminary results from the Middle Pleistocene site of Payre (South-eastern France).** *Journal of Anthropological and Archaeological Sciences* (in press)., doi: 10.1007/s12520-016-0368-2

8. **Publication 8:**

   Pedergnana, A., Blasco, R., 2016. **Characterising the exploitation of avian resources: An experimental combination of lithic use-wear, residue and taphonomic analyses.** *Quaternary International* 421, 255-269.

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Chapter 1: Introduction

1.1 Presentation of the thesis

Questions about how stone tools were used, what types of activities took place at sites, how settlement functioned in past cultural systems have raised a variety of studies, whose main aim was to develop an innovative analytical method to respond to those questions. Traceology, use-wear analysis, microwear analysis, functional studies are some of the terms used to name the discipline which investigates the function of past tools.

This work represents a contribution to this broad discipline and focuses on a sole lithic raw material: quartzite. Quartzite, as other coarse raw materials, has been poorly studied from a functional perspective and not extensive experimental frameworks have been published. In fact, research focused on microwear on quartzite has been rather unsystematic and often lacked strong experimental references.

Because of these reasons, it was urgent to constitute a robust set of experimental data in order to be able to deal with the archaeological assemblages made of this type of rock. For example, there are entire regions in the World, like the Iberian Peninsula, and long chronological periods, such as the Lower Palaeolithic, where the use of quartzite is extremely widespread.

In such cases, quartzite embodies unique information, which might be lost if the knowledge of this raw material is only superficial. Often, the chances of reaching a thorough understanding of the subsistence strategies of prehistoric hunter-gatherers are linked exclusively to this type of rock. These are the main reasons why we undertook this research.

Broadly speaking, we pursuit an innovative way to look at quartzite from a functional perspective, which could incorporate a combination of different microscopic techniques. The main reason of not resorting to only one type of microscopes is that we wanted to construct a sound method, applicable worldwide with no restrictions.

Our desire was that this method was flexible, meaning that its reliability needed to be guaranteed even if one of the microscopes used in this work were not available.

Above all, this thesis is the conjunct of different sub-topics, all inserted in a broader purpose, which always turns around the functionality of stone tools. It is organised in a way that, although its core is the function of quartzite tools, many concepts discussed are applicable to other lithic raw materials. For instance, a great effort has been put to characterise the experimental organic residues available from our experimentation with quartzite tools. In the same framework, observations on possible sample contamination have been provided and may be useful when any archaeological tool is subjected to microscopic analysis (*e.g.* bones, teeth).

Moreover, one of the central interests was that microwear studies do not depart from the main core of lithic studies which is technology. The clear inter-relation between the two disciplines is discussed and the effort to manifest this unity is constantly present in this work.

The last part of this work, nowise being less important than the previous ones, is a recollection of the two case studies where the methodology proposed here has been applied. As the main goal of any experimental research is to ultimately apply a methodology designed in laboratory conditions to archaeological assemblages, we selected two Middle-Pleistocene sites yielding abundant quartzite material. Therefore, we analysed quartzite assemblages collected in the Gran Dolina site in Northern Spain and in the Payre site in Southern France.

The main reason why we selected these two assemblages, beside their similar chronological span (GD-TD10.1= MIS9-11, Payre= MIS5-8), is that it was important to understand the functional significance of quartzite in both assemblages. In the case of GD-TD10.1, the poor conditions of chert normally impede the preservation of wear. The chemical damage of chert mostly affects one of the varieties present at Atapuerca (Neogene chert) (Sala, 2007; Font Rosselló, 2009; Font et al., 2010). Although some chert artefacts were successfully found bearing use-wear evidence, it was not thinkable to apply use-wear analysis on the chert assemblage to a large scale. Therefore, quartzite is the second most abundant raw material at GD-TD10.1 and it is the sole material whose microscopic study may provide large functional information of the human occupations of the site.

Regarding Payre, the main archaeological question was to understand the functional role of quartzite throughout the entire sequence of the site (MIS5-6, MIS7-8) and its relationship with the other raw materials (quartz, flint, basalt and limestone).

Moreover, we put much energy into the participation in the scientific debate through different means. An extensive bibliographic research and consultation have been the basis of each section of this work. Besides, the most informative parts of this work have been already published on scientific journals. These parts are included in the text as they were originally published (with no further modifications).

1.2 Description of aims

The main aims of this thesis have been already mentioned in the previous paragraph. However, considering the importance of a detailed explanation, our objectives will be listed below. When we refer to the publications included in this manuscript, please take as a reference the list of publications found at page XVII, before the table of contents.

1. The main goal of this research is to design a method which allows for the identification of use-wear on quartzite (Publication 1). Hence, one of the main objectives of this work is the thorough description of the appearance of use-wear on this type of rock. Within this major aim, the general use-wear bibliography has been carefully revised and, due to a largely unclear and confused terminology, we attempted to systematise a general terminology, valid for all quartzose rocks (and by extension, to all raw materials). Generic and broad categories of use-wear are used, which allows our results to be compared with the functional studies performed on other rocks. Then, some specific terms and descriptions are only valid for quartzite. This differentiation has been extensively explained in this work (Publication 2). Experiments are the main corpus of this investigation and serve to respond to several questions (Chapter 5). They formed an extensive use-wear reference collection, including different contact materials and actions. Five different varieties of quartzite have been included in order to evaluate the microwear intra-variability (Publications 3 and 4). The use of sequential experiments was crucial to understand how use-wear forms and, by extension, the mechanical behaviour of this rock under stress. Concepts of tribology are of paramount importance to reach this type of understanding and are then taken as the main basis to explain the formation of use-wear, which are nothing more than physical modifications of the micro-topography of the rock. Furthermore, the quantitative data that are available for a restricted experimental sample help in the definition of different polish features on quartzite (Annex 1).
2. The secondary pillar of this thesis is related to the study of the micro-residues of the contact materials (Publications 6 and 8). Assuming that a combined methodology comprising both use-wear and residue analyses is more reliable than when only one analysis is applied, analysts need to have at least a basic knowledge of both. In fact, if functional interpretations are based on multiple sources, results are more satisfactory, since they are contrastable.
3. The third objective was the intention to find the best combination of microscopes to image microwear on quartzite. Optical Light Microscopy (OLM), Scanning Electron Microscopy (SEM) and, to a lesser extent, Laser Scanning Confocal Microscopy (LSCM) have been used and the best advantages of each microscope have been underlined (Chapter 4; Annex 1). The same approach has been applied for the observation of micro-residues, where only OLM and SEM have systematically been

used.

4. **4.** The fourth objective was to discuss the inter-relationship between functional and technological studies. We wanted to achieve an understanding on how these two analyses are inter-dependent. A theoretical framework is first provided (Chapter 2), then a combined use of technological and functional data is tested for one archaeological case study (Chapter 6) and considerations are then extrapolated from these results (Chapter 8). More specifically, the techno-functional approach has been adapted to our case in order to understand if this can be a helpful tool to select the artefacts prior to microscopy analysis. This adaptation is meant to understand whether the grouping of unmodified and retouched flakes based on their recurrent technical characters is a reliable source of functional information or not. In other words, does the definition of the technical characters have always a correspondent value in the tools which have been actually used? Does the presence of particular combinations of technical features have a major impact in the selection of the lithic blanks to be used (and/or retouched?). And finally, is it the case to re-think the criteria on which the selection of the implements that undergo use-wear analysis is generally done? Would it be better to systematically base this selection on technological criteria, rather than on random or subjective ones? We are making a first trial on an archaeological sample to understand whether technological considerations are helpful in giving some clues about the functional potential of the lithic tools and if so, how this would influence further works on use-wear.

5. The fifth and last objective was to test the whole methodological corpus by studying two archaeological assemblages. On the one hand, we analysed quartzites from TD10.1 level of Gran Dolina by using techno-functional data to select the samples for microscopic analysis (Chapter 6). On the other hand, the Payre assemblage has been microscopically analysed and no additional technological data have been collected (Chapter 7; Publication 7). The main interest in applying use-wear analysis was in both cases to use the functional information to infer the main activities which took place at the sites. In this way, functional information would contribute in the definition of the type of settlement and help to decipher the behaviour of the human groups inhabiting Gran Dolina and Payre during the end of the Middle Pleistocene.

6. The last point of discussion, which is basic for both use-wear and residue analyses, is the issue of contamination. We thought it was important to discuss the main contaminants which might be present on the surfaces of stone tools, how they might compromise the functional interpretation and how our capacity of recognising them can be improved (Publication 5). Certainly, the more the awareness about this is raised and stabilised the less contamination is expected to be present on newly excavated material. The key to this issue is obviously found in how stone tools are handled after excavation and prior to microscopic analysis.

1.3 Publication 1:

Pedergnana, A., Ollé, A., 2014. **Use-wear and residues analyses on quartzite stone tools: setting up a methodology.** In: Lemorini, C., Nunziante, S. (Eds.), Proceeding of the international conference “An integration of use wear and residues analysis for the identification of the function of archaeological stone tools”. *BAR International Series* 2649, Oxford, pp. 43-62.

This publication was prepared during the very initial stage of this research. The main goals of the research are exposed, the first experimental trials are described and first impressions about the formation of use-wear are given. Use-wear and residue analyses are both considered in this text.

USE-WEAR AND RESIDUES ANALYSES ON QUARTZITE STONE TOOLS: SETTING UP A METHODOLOGY

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Abstract

With this contribution we aim to stress a possible specific methodology to study quartzites from a microscopic point of view. The main purpose of our research project is the observation and the comprehension of microwear formation on quartzite implements. The urgency of setting up a specific methodology for quartzite comes from the idea that its structural behaviour, when a force is applied, is different from that of flint. So, quartzite has to be treated individually recognizing its specific attributes.

We started to define a detailed methodology for this project, which combines the use of Optical Light Microscopy (OLM), Scanning Electron Microscopes (SEMs) and experimental archaeology.

One of the main aims of this project is the creation of a quartzite stone tool reference collection including different activities, whose use-wear features are going to be compared with those found on the archaeological material. Directly linked to the experimental program is the purpose of systematically documenting residues of the worked materials with the employment of low vacuum SEM and EDS elemental microanalysis.

Microscopic observations of experimental flakes allowed us to make a preliminary assessment of quartzite behaviour under stress. Brittle respond and the constant fracturing as well as the different steps in the formation of microwear have been underlined.

1. Introduction

Microwear analysis has been largely employed to determine functions of stone tools, but not so many efforts have been done to extend this methodology to non-flint raw materials. In fact, the available functional studies refer almost entirely to flint (among others, Tringham *et al*., 1974; Keeley, 1980; Vaughan, 1985; Grace, 1989; González and Ibáñez, 1994; Levi-Sala 1996; Van Gijn, 2010). When other raw materials have been analysed, very frequently the same analytic method which had been set up specifically for flint, has been applied with the result of a great scale of bias errors.

Although some specific studies including both experimental and archaeological samples have been done, just very few analysts created specific methodologies for non-flint rocks (Knutsson, 1988; Richards, 1988; Sussman, 1988; Hurcombe, 1992; Pereira, 1996; Sternke *et al*., eds., 2009).

This research focuses on quartzite tools because of several reasons. First of all, as mentioned, it is not an adequately studied rock in the field of use-wear. Then, the obtaining of a reference collection will help to analyse lithic assemblages which are poor of flint elements or absolutely flint lacking. Secondly, as quartzite is one of the most used rocks in some regions such as the Iberian Peninsula, it is necessary to provide a functional record concerning this rock.

As archaeological application, we propose the study of a sample of quartzite stone tools from the Middle Pleistocene Gran Dolina site (TD10 level) (Ollé *et al*., 2013). Generally speaking, concerning Atapuerca sites, quartzite embodies a special importance, as it is present in huge percentages and apparently shows a good state of preservation, while chert and sandstone stone tools might pose serious problems when applying functional analysis (Márquez *et al.*, 2001).

In this paper we will present a thorough characterization of the employed methodology within our on-going project. The fundamental methodological issues will be exhaustively exposed and also preliminary results regarding the development of use-wear on quartzite stone tools will be included. A limited sample of archaeological objects has been analysed in order to assess their state of preservation and to evaluate our methodological approach using preliminary results to set up new experiments.

2. Materials and Methods

As we have already said, the main purpose of this research is the comprehension of the mechanical behaviour during use of quartzite. In addition, we want also to methodically document organic residues (morphology and elemental composition) found on experimental tools in order to create a reference collection. Both to document the plurality of use-wear traces developed on different lithological varieties of quartzite and to be able to recognize possible worked material residues present in the Gran Dolina TD10 record, a strictly controlled experimental activity is necessary.

2.1 Experimental activity: a necessary background

Experimental flakes have been obtained employing raw material coming from the studied site vicinity to guarantee the possibility to compare them with edges and surfaces modifications found on archaeological tools. The more similar to the original archaeological sources the experimental raw material is, the more suitable the functional approach is.

To be able to detect use-wear internal variability, knowing that different activities on different materials produce characteristic modifications of the lithic micro-topography, it is very important that experimental activity comprises various materials (meat, bone, antler, hide, wood, vegetal matters…). So, the experimental program will include several activities divided in groups of different quartzite varieties, worked materials, movements and actions. The experimental activity has been carried out basically by one of the authors (A. P.), who presents a medium grade of experience in performing this kind of tasks, though occasionally some of the experiments have been carried by students with lower experience. Part of the experimental work has been already finalized, providing us with a considerable portion of the required reference collection. This allowed us to perform functional analysis on a number of archaeological implements.

The experimental protocol comprises sets of sequential experiments. That means that the same lithic tool is used to perform the same activity during different time intervals (15’-30’-60’). To make possible the recognition of the surface modifications of the active edge after use, we used casts of the original edges (before use). The imprints of the fresh edges are prepared by making moulds using silicon based dental impression material (*Provil® novo Light*) from which casts are obtained using a bicomponent rigid polyurethane resin (*Feropur PR-55)* (Ollé, 2003; Vergès, 2003; Ollé and Vergès, 2008, *I.P*.).
By dividing experiments in sequential sessions we mean to monitor the development of the control points which we have previously chosen. The collected data will allow us to assess the

general behaviour of the different quartzite varieties under stress and which are the effects of the different experimental variables (edge angle, worked material, action, motion…) on the development of the different wear types.

Regarding the efficiency of quartzite edges during the performance of the experiments, we must underline that, with respect to the performed action, quartzite edges can respond differently. Generally, edges of the finer varieties work better in the sense that they take more time to worn out and become useless, while coarser varieties loose efficacy relatively in a short time. Specifically, as for other raw materials, when used to butcher (skinning, disarticulation, meat cutting, removal of the periosteum) or to scrape fresh hide, quartzite tools need to be frequently cleaned to eliminate grease residues from the edge which would limit the edge functionality. Concerning wood, bone and plants processing and dry hide working, no particular problems emerged. In fact, quartzite edges showed a notably high level of durability in terms of efficacy, with few exceptions which could depend on the edge morphology (edge angle and sagittal outline).

2.1.1 Experimental reference collection and experimental independent variables

The experimental activity is still on-going, that means that the presently available experimental collection is not complete. However, all the experimental activities have been divided into various categories, including different varieties of raw material, worked materials, different actions, meaning both movement directions (unidirectional-bidirectional) and position of the used edge regarding the worked material (transversal-longitudinal) (Table 1).

Although several quartzouse varieties present in the Atapuerca archaeological record (which includes also orthoquartzites and quartz-arenites) will be added to the experimental protocol, so far we have started employing two of the most common ones. Both varieties come from the *Arenas de Utrillas* formation (*Sierra de la Demanda*, near Burgos) (García Antón and Mosquera, 2007). The first one (QTFU1) is a fine grained variety which presents colours ranging from reddish-brown to light grayish tones. Pebbles within this formation frequently show rubefaction signs. The second variety (QTFU2) differs from the first one in its coarser grain size and its yellow to brown colouring. Both of them are perfectly apt to knapping activity, presenting conchoidal fracture, high quartz content and high degree of metamorphization.

Hardness of worked material has not been thoroughly evaluated yet. For this reason, a general division into soft, medium and hard materials has been maintained. Soft materials are meat, fresh hide, tendons and plants. Medium materials are woody plants, soft wood, dry hide and humidified antler. Finally, dry antler, bone, shell and stone are considered to be hard materials. The state of the material while being worked is also recorded (fresh, dry, humidified).

We chose to establish clear and unequivocal variables for each experiment in order to monitor use-wear formation. Anyway, the environmental conditions have been maintained as much as possible close to those which might have been experienced during prehistoric times. That is to say, performing all the actions in external locations, on the ground, and trying to employ the more functional prehension mode for the experimenter. What emerged from these considerations is that the experiments have been designed based on two different, but complementary, definitions, “ethic” and “emic” types (Knuttson, 1988: 11-12). It has been chosen not to apply a strict ethic type of experimentation, because of the unreal conditions supposed by a completely mechanical development of it. At the same way, performing a pure emic type of experiment, would mislead the interpretation of the results.

REFERENCE CODE	EDGE ANGLE	WORKED MATERIAL	WORKED MATERIAL TYPE	ACTION	MOVEMENT	WORKING ANGLE	TIME *
QTFU1-01	40	Fresh wood	*Quercus ilex*	Sawing	Longitudinal-Bidirectional	60°<α<90°	15+15+30+ 30
QTFU1-02	60	Shed antler	*Cervus elaphus*	Sawing	Longitudinal-Bidirectional	90°	15+15+30
QTFU1-03	40	Skin	*Cervus elaphus*	Cutting (skinning)	Longitudinal-Unidirectional	80°<α<40°	35
QTFU1-04	45	Fresh hide	*Cervus elaphus*	Scraping (hide processing)	Transversal-Unidirectional	60°<α<30°	15
QTFU1-05	50	Fresh bone	*Cervus elaphus/ Bos taurus*	Sawing	Longitudinal-Bidirectional	80°<α< 90°	15+15+30
QTFU1-06	25	Fresh bone	*Cervus elaphus/ Bos taurus*	Scraping	Transversal-Unidirectional	90°	15+15+30
QTFU1-08	30	Fresh wood	*Quercus ilex*	Scraping	Transversal-Unidirectional	40	15
QTFU1-10	35	Woody plant	*Arundo donax*	Sawing	Longitudinal-Bidirectional	90°	15
QTFU2-01	55	Fresh wood	*Quercus ilex*	Sawing	Longitudinal-Bidirectional	90°	15+15+30
QTFU2-02	50	Shed antler	*Cervus elaphus*	Sawing	Longitudinal-Bidirectional	80°<α< 90°	15+15+30
QTFU2-03	35	Flesh, tendons bone	*Cervus elaphus*	Cutting (defleshing)	Longitudinal-Uni(bi)directional	80°<α< 90°	35
QTFU2-04	45	Fresh hide	*Cervus elaphus*	Scraping (hide processing)	Transversal-Unidirectional	80°	15
QTFU2-05	45	Fresh bone	*Cervus elaphus/ Bos taurus*	Sawing	Longitudinal-Bidirectional	80°<α<90°	15+15+30
QTFU2-06	40	Fresh bone	*Cervus elaphus/ Bos taurus*	Scraping	Transversal-Unidirectional	45°	15+15+30
QTFU2-08	50	Fresh wood	*Quercus ilex*	Scraping	Transversal-Unidirectional	45°	15+15
QTFU2-10	45	Woody plant	*Arundo donax*	Sawing	Longitudinal-Bidirectional	90°	15+15

Table 1: The main independent variables
of the performed experiments
(for actions definitions we refer to Keeley, 1980).
* Although all the experiments
are planned to be sequential,
here we show the current state of experimentation;
some flakes have just been used for 15 or 30 minutes.

So, regarding the performed action the recorded parameters are the used edge position regarding the movement (parallel or tangential, that is to say longitudinal or transversal actions), the working angle and the relative exerted pressure (subjective parameter), lithic handling during action (prehension or hafting, after Odell, 2004). In addition to the general time intervals (15'-30'-60') the number of executed strokes is also calculated.

Concerning the lithic object, several parameters are recorded: measurements, the used edge angle and morphology (horizontal and sagittal delineations) and presence of retouch. Systematic photographic and video-clip supports are provided for each activity.

2.2 Microscopic analyses

2.2.1 Use-wear analysis

Regarding experimental stone tools, microscopic analyses will be performed by applying two different but highly complementary approaches: the description of use-wear and, for each one of the worked materials, the morphological and chemical characterization of the residues adhered on lithic surfaces. Experimental flakes surfaces are microscopically observed between one experimental session and the other aiming to reach a better comprehension of the mechanical behaviour of quartzites under stress and, as a result, to control use-wear features, its internal variability and its development.

A specific work sheet has been designed in order to be able to document and subsequently plot diagnostic features. Photographic record of edge modification is kept and each image is put on its exact position on the sketch of each specimen (either experimental or archaeological). We employed high-power microscopy, using an optical light microscope (OLM) equipped with Differential Interference Contrast and Nomarski prisms, with magnifications from 50X to 500X (Zeiss Axio Scope A1), an extended focus system provided by the software *Delta Pix Insight 3.2.5*, as well as two scanning electron microscopes at magnifications from 15X to 5000X[1], a JEOL JSM-6400 and an ESEM FEI Quanta 600 (the latter used for low vacuum mode analysis of residues).

During SEM observations a series of control points is chosen (comparing used edges with fresh ones), allowing us to document the gradual stages of edges modifications during use as well as use-wear formation processes.

So, use-wear is recorded by means of two different methodologies developed in the past decades, which basically divided scholars into two distinct school of thought: analyses with optical microscopes (among others, Keeley, 1980; Odell, 1981; Vaughan, 1985) or with electronic devices (Knutsson, 1988; Ollé, 2003; Vergès, 2003), being actually scarce the combined approaches (among others, Sussman, 1985, 1988; Yamada, 1993). The scarcity of publications regarding use-wear on quartzite forced us to refer to the main works concerning use-wear terminology, habitually employed for analysing chert artefacts. We are aware that a universal terminology specific to quartzite is urgently needed, especially for defining the analytical criteria for our sample, but as this is not the main aim of this paper, we will just refer to the most general literature on use-wear, favouring works focused on quartz (e.g. Tringham *et al.*, 1974; Keeley, 1980; Knutsson, 1988; Sussman, 1988). So, specific use-wear terminology will be not here exhaustive and will be probably redefined by our future contributions.

Furthermore, use-wear features detected with different methods have to be differentiated: some of them are visible with both optical and electronic microscopes, whilst some are just detectable at highest magnifications (SEM).

[1]Although SEMs are capable to much higher magnifications, for this study we did not reach magnifications superior to 5000X.

Both edge modifications and quartz grains alterations are investigated. In general terms, macroscopic wear, such as edge damage (micro-fracturing or micro-scarring) are detectable with optical devices, whilst aspects related to the grains edges and surfaces are much better documented with SEM. Edge rounding is visible through both techniques employment, although micro-rounding (when the edge is barely affected) imaging is only possible at highest magnifications.

2.2.2 Residues analysis

By analysing the organic residues on the experimental artefacts, we aim to create a reference collection in order to be able to recognize remains of the worked material on the archaeological stone tools surfaces. A multi-analytical approach will be adopted for an in-depth characterization of experimental residues. Optical microscopy observations and photographic documentation will be carried out (Fullagar, 2006; Lombardand and Wadley, 2007; Lombard, 2008; Monnier *et al.*, 2012).

SEM observations (low vacuum) and photographic documentation will be conducted for each type of worked material included in our experimental protocol in order to acquire high quality images comparable to those obtained with optical devices. Moreover, X-ray spectra will be collected with X-ray Energy-Dispersive Spectroscopy (EDS) system to perform elemental analysis of the organic specimens. To improve the EDS results, we observed residues using low vacuum mode SEM (which avoids the need of specimens being coated with gold or carbon).

Both for optical and electron microscopes observations, a polar coordinate system has been used (Lombard, 2008) to record use-wear and residues distribution aiming at a more precise distribution allowing a further application of statistical analysis.

2.2.3 Sample preparation

For experimental residues characterisation using either OLM or low vacuum SEM techniques, as we are dealing with organic residues, no cleaning procedure is usually taken into account.
As SEM analyses allow reaching evidently greater magnifications than optical ones, the sample surface needs to be potentially perfectly cleaned because some micro-particles, such as dust, pollen, human skin cells or other elements can be detected and also cover use-wear. Usually, OLM does not detect this kind of particles. So, for use-wear analysis experimental flakes have previously undergone an ultrasonic bath in H_2O_2 for 15 minutes (with the aim of removing organic residues). Then, both experimental and archaeological specimens have been soaked in a 10% HCl solution for several minutes in order to respectively remove residues or sediment. Acid remains are eliminated by cleaning with running water. After that, we included two more ultrasonic baths: 15 minutes in a detergent solution (Derquim®), with the objective of removing all the acid residues and finally, two minutes in pure acetone to eliminate manipulation residues. For the ultrasonic baths zippered plastic bags were used in order to avoid frictions against the tank.

For SEM analysis, flakes are then mounted on a metal stub using hot melted glue. When working with conventional (high vacuum) SEM, the procedure is eventually more complex and certainly more time consuming, as specimens need to be conductive (Ollé and Vergès, 2008). To make this possible, before observation samples are covered with a 30 A thick gold layer and then a colloidal silver path has to be created in order to enhance conductivity. After the SEM analysis, the conductive gold coating can be removed with nitro-hydrochloric acid (*aqua regia*, an acid mixture containing ¼ concentrated nitric acid (HNO_3) and ¾ hydrochloric acid (HCl). This

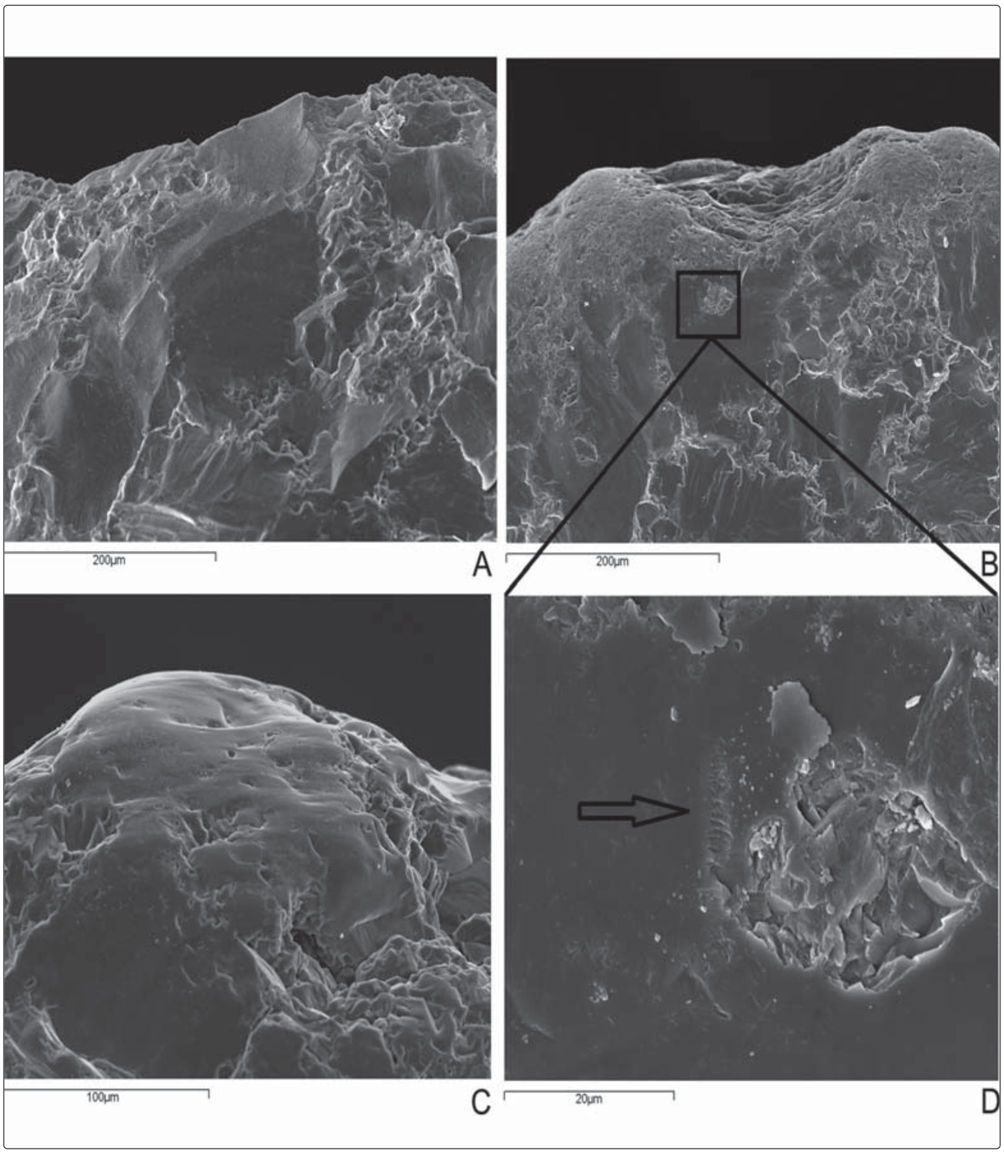

Figure 1: A-B) A control point on an active edge (QTFU1-04),
used to scrape fresh hide (15 minutes).
Original magnification: 250X; Scale bar: 200 µm.
Comparing the fresh edge (A) with the same point after 15’ of use (B) the very rapid change
in morphology is evident, particularly the edge rounding caused by micro-abrasion;
C) Extremely polished area due to 60’ of bone sawing (QTFU2-05).
Original magnification: 500X; Scale bar: 100 µm;
D) Enlarged picture of the rectangle in the B image showing a hole on the grain interior surface
caused by a subsurface crack.
It can also be seen a striation (“linear groove” or “furrow”) produced by a dragged particle across the surface,
perpendicular to the edge (indicative of a transverse motion).
Original magnification: 2000X; Scale bar: 20 µm.

procedure has proven not to damage the surfaces of siliceous rocks such as flint, quartz and quartzite (Ollé and Vergès, 2008).

So, when archaeological implements are analysed, depending on which type of analysis is applied, cleaning procedures are contemplated or no. When searching for residues, just a very soft cleaning procedure is used in order to avoid the risk of destroying possible present remains, for which a mixture of alcohol and acetone is used (to remove dust particles and manipulation signs). For use-wear analysis, the complete processes described above are employed.

3. Results

3.1. Wear patterns on quartzite

Structurally quartzite is different from chert, in the sense that it has an extremely irregular topography. In fact, when observed microscopically surfaces of quartzite flakes appear to be more uneven than those made of chert. This characteristic causes micro-wear to develop from the highest points of the relief to the lowest ones, thing that happens also in chert artefacts, but at a very slighter degree. Therefore, the very irregular surface relief of quartzite might strongly affect the process of use-wear formation in the sense that plausibly the wear patterns connected with the various worked materials differ from those found when analysing chert artefacts. This means that it is no longer possible to try to infer the worked material on non-flint raw materials making comparisons with the wear features found on chert.

From our preliminary results concerning observations of experimental lithic objects we are now able to assess to some extent the behaviour of quartzite tools edges under stress. Quartzite is essentially a brittle material, so at the first stages of use it shows a progressive loss of material due to micro-fracturing. Entire portions of quartz grains are detached as soon as friction with the worked material starts (Figure 1A-B). Afterwards, as friction continues, a very erosive wear comes into play making the used part of the edge rounded. Indeed, the rounding degree and

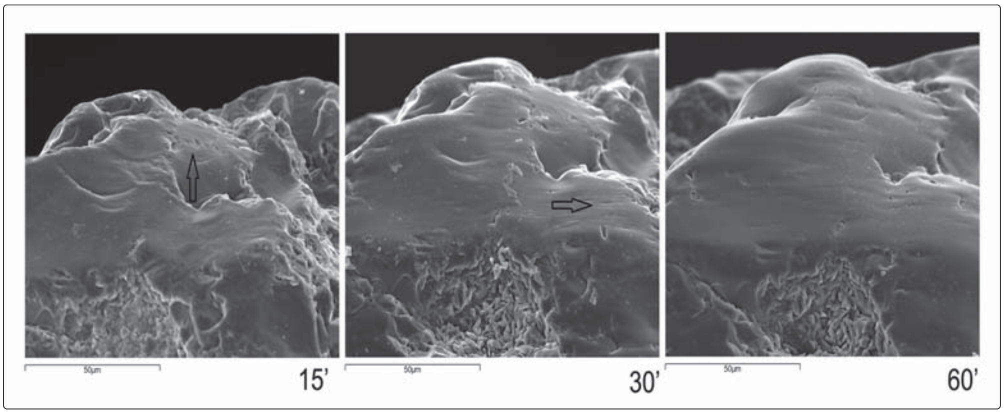

Figure 2: The development of polish (smoothing) after sawing a limb bone (QTFU2-05).
The original magnification of the three SEM micrographs is 1000X and the scale bar is 50 µm.
The attritional character of the polish is evident.
The arrows are pointing to linear features (parallel to edge)
which dissapear after 60' of use
(in fact, it seems to be a combination of mechanical grooves and plastic sleeks).
Polish areas appear as smooth, even regions,
with a wavy relief clearly distinguishable from the original rock surface texture.

wear texture depend upon the worked material. It can be in fact very developed and have a rough appearance (Figure 1B), as in hide working (scraping fresh hide in this case), or it can be quite smooth, like in the case of bone polish (Figure 1C). The deeper this feature is found in relation to the edge rim, the more acute is the working angle related to the performed action.

Many concepts coming from material sciences (as tribology and rheology) have been employed to better interpret the wear formation processes (i.e. Knutsson, 1988; Sala *et al.*, 1998), some of which have been deeply used in this study. Nevertheless, here we focus more on the monitoring of the experimental processes than on the interpretation of the origin of each type of wear feature.

The remarkable rapidity with which the edge is becoming worn has generally been documented in relation to dry hide processing or to the employment of abrasives (Mansur, 1983; Araújo Igreja *et al.*, 2007). From this first set of experiment we can regard fresh hide as a very abrasive material leading to a general loss of material, an extreme rounding of the entire portion of the used edge and the sporadic presence of tiny linear features, perpendicular to the used edge (Figure 1D). Therefore, we noticed that particularly when referring to transversal actions the presence of linear features is sporadic and they are generally much shorter than those forming on flakes used for sawing actions (particularly on hard and medium materials).

In fact, especially after the first phase of use (in sawing actions) quartz grains surfaces show several linear features, very often grouped together on a same, nearly plane, grain surface (Figure 4F). Striations occur in the form of linear signs with the very characteristic half-moon shapes caused by microparticles of the rock which are then dragged along the surface during the friction process (linear grooves after Sussman, 1988). No striations normally called as “furrows”, have been documented.

Polish usually refers to a shiny, mainly smooth, altered zone of the stone tools topography and it is recognized by the particular reflection of the incident light using to illuminate the specimen, allowing differentiating it from the original rock surface (mostly defined for chert). Through SEM imaging polished areas do not show this sleek appearance (SEM does not use incident light to form the magnified image) and appear as extremely worn-out areas (Figure 2). This has been interpreted as the product of actual plastic deformation which takes part of the general attritional processes involved in use-wear formation (Knutsson, 1988; Ollé and Vergès, 2008). The very irregular topography of quartzite, together with its essentially brittle behaviour, does not allow polish do develop on very large areas. Anyway, at least on the highest topography points, polish is likely to be formed punctually depending on the action performed, the elapsed time and the worked material (Clemente Conte, 2009).

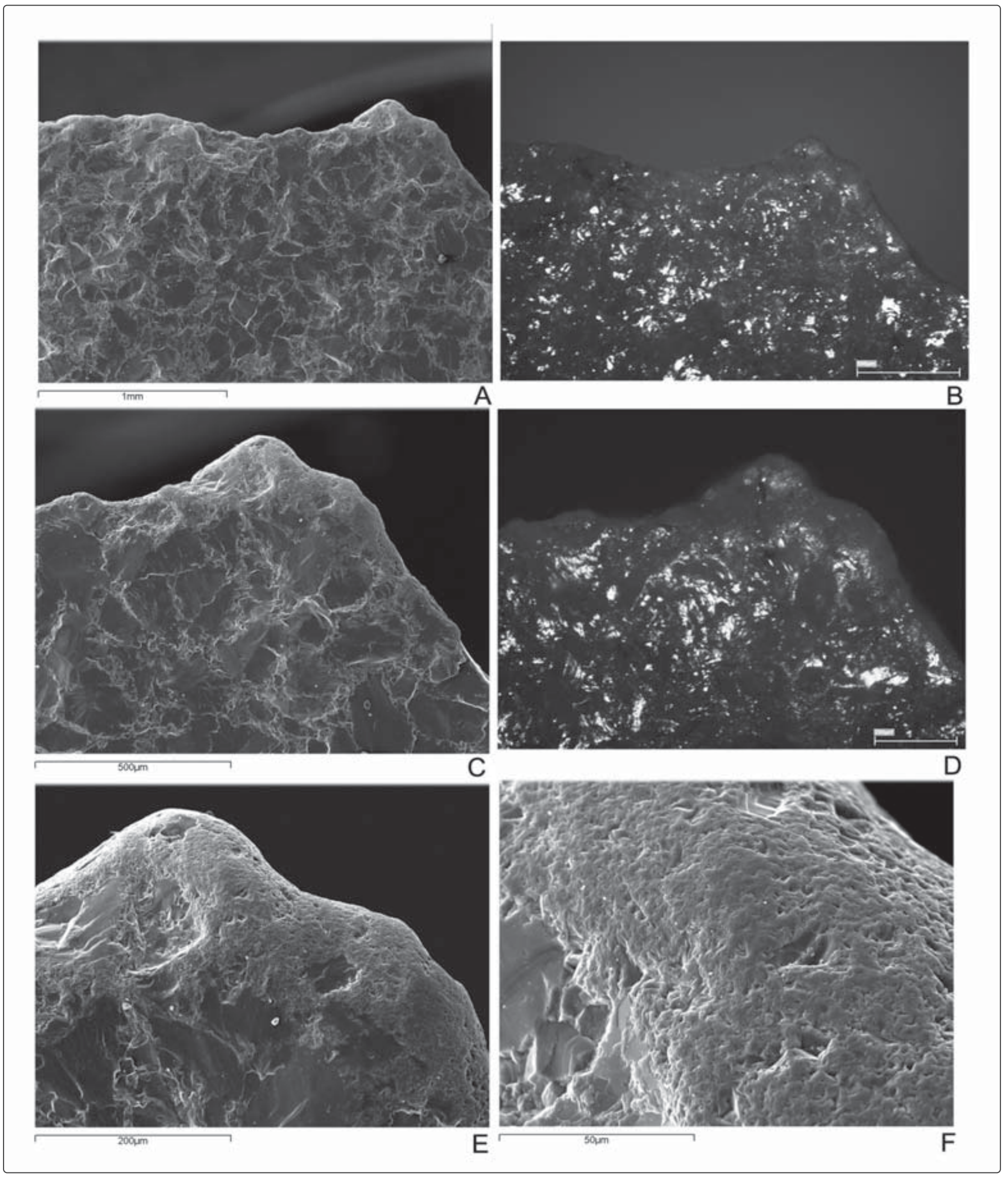

Figure 3: Metaquartzite flake (QTFU1-04) used to scrape fresh hide for 15 min.
It shows a well-developed edge rounding with a rough texture,
with a punctual presence of pits and smooth areas due to plastic deformation (F).
Pictures B and D are taken with a metallographic microscope (stacking of 15 images, extended focus mode),
the rest with a SEM.
A) Original magnification: 50X, scale bar: 1 mm;
B) Original magnification: 50X, scale bar: 500 µm;
C) Original magnification: 100X, scale bar: 500 µm;
D) Original magnification: 100X, scale bar: 200 µm;
E) Original magnification: 250X, scale bar: 200 µm;
F) Original magnification: 1000X, scale bar: 50 µm.

The visual differences of the same wear pattern (for instance fresh hide scraping) depending on the recording employed technique are very notable (Figure 3). Considering that this flake has been used only for 15 minutes, the very high degree of surface abrasion and edge rounding can be appreciated either with OLM and SEM. With optical devices greater magnifications which offer a fine vision of the texture of the modified surface are impossible to obtain. At highest magnifications (1000X, Figure 3F), "pits" can be observed, due to the survival of the lowest parts of the original topography, not yet affected by abrasion (Diamond, 1979).

Use-wear related to antler and wood sawing (Figure. 4 and 5 respectively) seem to develop to the same degree. In both cases linear features (parallel to the edge) form during the first 15 minutes of work and then gradually disappear due to quartz grains breakage, grains levelling or grains edges rounding. The origin of the linear feature which crosses the entire crystal surface (Figure 4 D-F) has clearly an abrasive origin. The abrasive particle might have had a considerable size, noting the rough appearance of the inflicted cracks on the grain surface. This statement derives also from the comparison of the overall size of this wear with the extremely thinner linear features developed after sawing a wood branch (Figure 5D-F). In general terms, having noted that some use-wear features on quartzite tend to disappear or become slighter, and that this vanishing is directly proportional with the elapsed work time, we should be cautious in the interpretation of a quartzite assemblage when applying a functional approach.

From our preliminary results it seems clear that use-wear on quartzite develops very rapidly in the earliest phase of use (15-20 minutes), due to the probable edge breakage caused by the contact with the worked material and then stabilize showing different patterns (less linear features, possible presence of fresh parts of the edge, so more or less developed edge rounding…). Moreover, use-wear formed at the various stages of use might eventually be continuously replaced by fresh edge portions, due to the mechanics of quartzite (Clemente Conte, 2009).
That means that a general feeble presence of use-wear could not be indicative of a short time of utilization. Nevertheless, it seems that after a certain amount of time, something like 30 minutes of use, the edge stabilizes and no more micro-fracturing happens, while abrasion processes generated by the unceasing friction with the worked material erase or weaken previously formed use-wear, particularly linear features.

Clearly, in an ideal scheme of lithic object life, the contact with the worked material and subsequently the mechanism of edge modification are suspended whenever the edge is no longer usable. So, the extreme edge rounding and the increasing of the edge angle might be responsible of the decreasing of the potential efficiency of the edge.

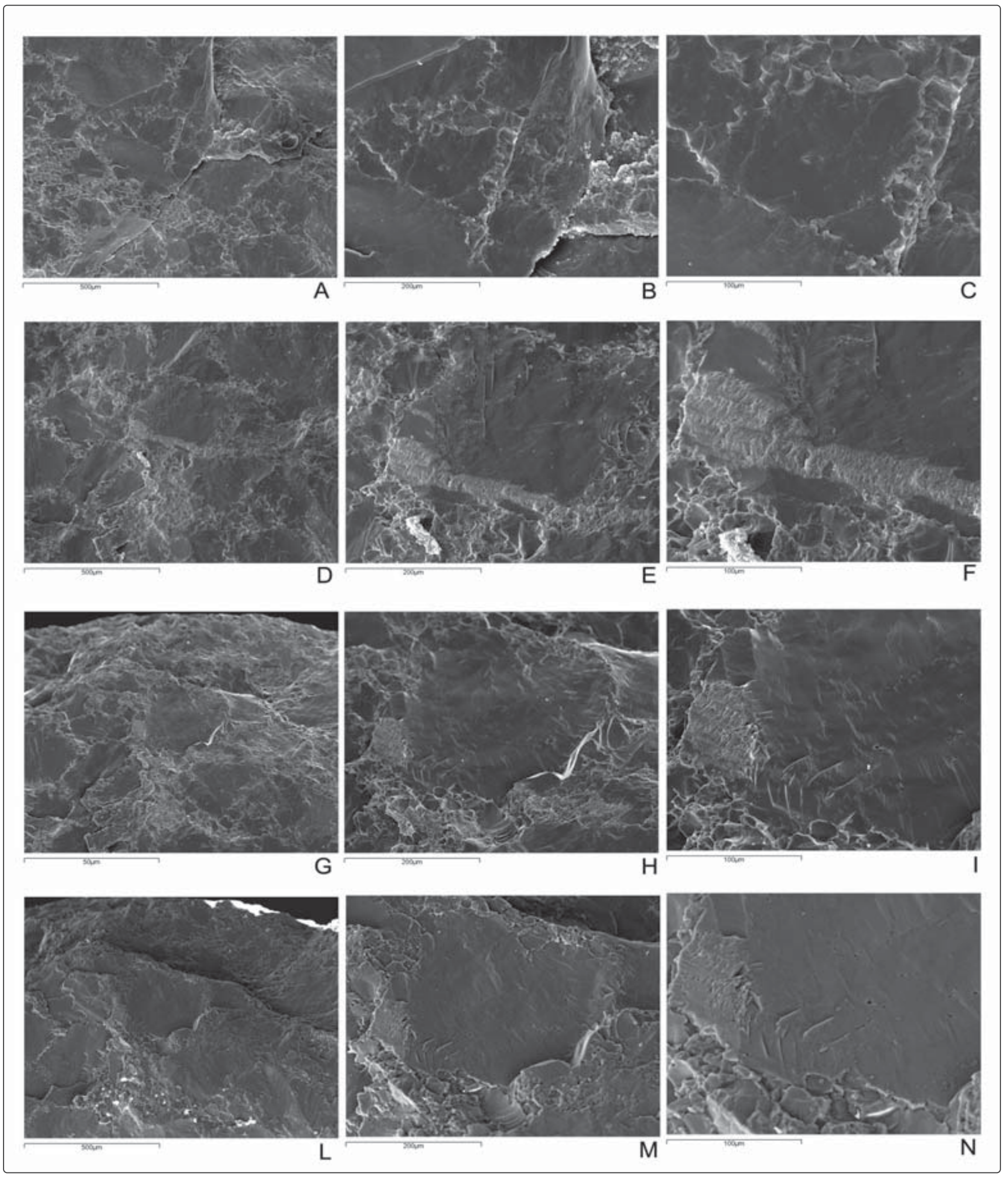

Figure 4: Development of a control point on an experimental quartzite flake(QTFU2-02)
employed to saw an antler during a complete set of experiments: first line, before use;
second line, 15' of use; third line, 30' of use;
fourth line, 60'of use. For pictures A, D, G, L)
Original magnification: 100X, scale bar: 500 µm;
B, E, H, M) Original magnification: 250X, scale bar: 200 µm;
C, F, I, N) Original magnification: 500X, scale bar: 100 µm.
The very abrasive linear feature develop after 15' of use (D-F),
and then disappear after 30' (G-I) because of the detachment of some parts of the grain surface.
Only a small portion of the wear survives after that.
The flake is oriented with the used edge on the top.

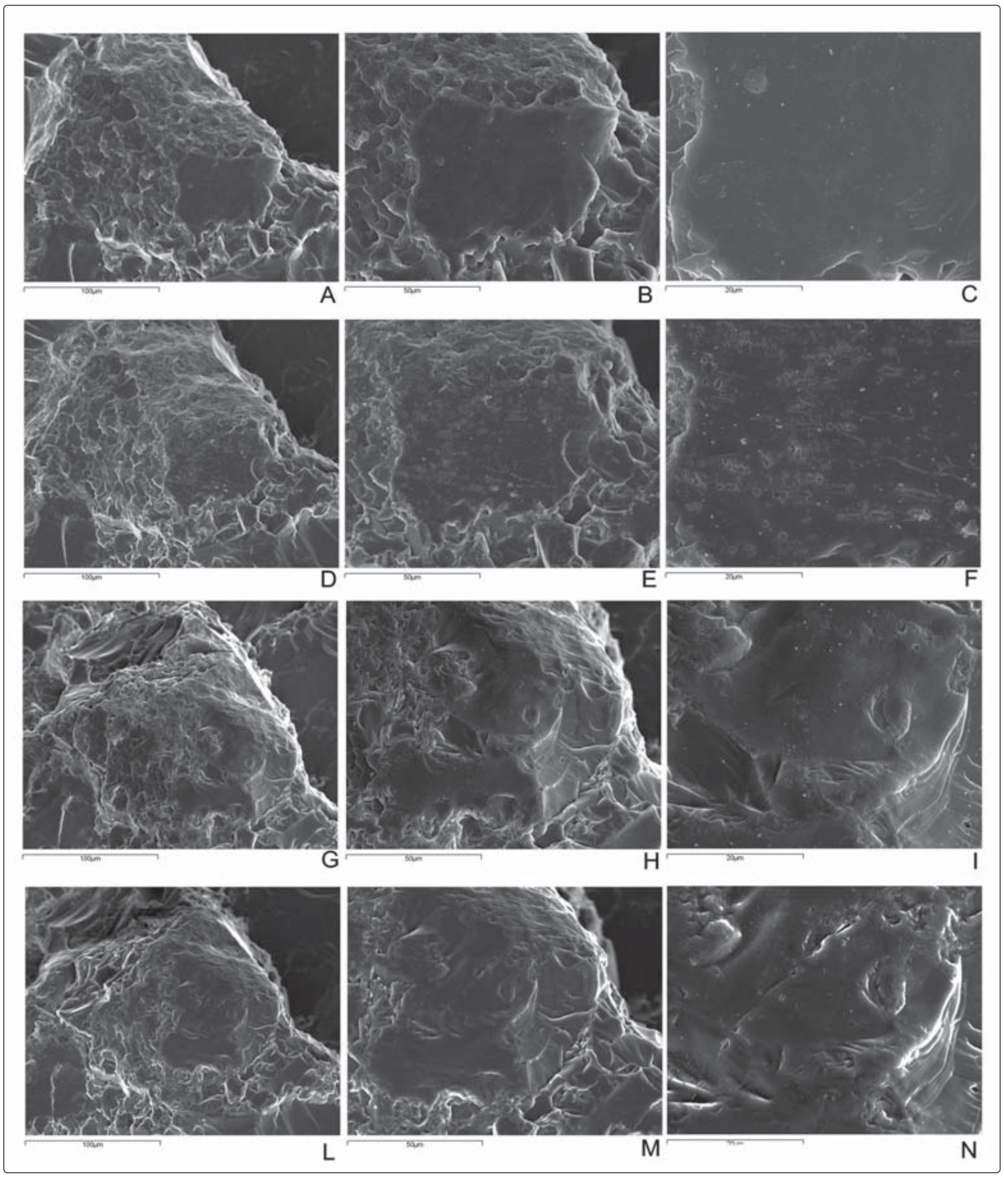

Figure 5: Development of a control point on an experimental quartzite flake (QTFU2-01) employed to saw hard wood during a complete set of experiments: first line, before use; second line, 15' of use; third line, 30' of use; fourth line, 60' of use.
For pictures A, D, G, L) Original magnification: 500X, scale bar: 100 µm;
B, E, H, M) Original magnification: 1000X, scale bar: 50 µm;
C, F, I, N) Original magnification: 2500X, scale bar: 20 µm.
A set of linear features develops on a smooth surface of a quartz grain (F) and then disappears because of some grain ruptures (I).
The survived striations are erased by the grain surface levelling due to the prolonged use and friction with the worked material.
The flake is oriented with the used edge on the top.

3.2 Experimental organic residues

Worked materials adhered to stone tools surfaces constitute a very important clue regarding tool function. In archaeological contexts, their detections appear to be very challenging due to poor preservation conditions, weathering and post-excavation treatment (Langejans, 2010). Afterward, not all the residues which might be found on archaeological samples are necessary connected with the material which had been worked at the site. In fact, they can be the result of various phenomena, such as bioturbation, contamination from the soil enrichments, modern contamination. They can also be connected with the tool life, giving some clues about more complex *chaînes opératoires*. For instance, a number of organic residues related to the hafting practice might be detected (glues, ochre or binding materials) (Rots, 2010).

Anyway, to be able to correctly identify them on archaeological material, we thought to provide a photographic and chemical characterization of the different worked materials which are likely to be found within archaeological contexts. For this reason SEM and conventional microscope images are systematically obtained in order to make a further analytical comparison between the two techniques. Then, chemical spectra (EDS) of each worked material are acquired and stored in our digital catalogue.

At the following image (Figure 6) we have an example of the employed recording procedure; in that case a limb bone has been scraped with a quartzite flake and after having analysed it a protruding residue has been noted and then photographed with both SEM and OLM. It appeared to be particularly effective in showing the original residue morphology, avoiding the contrast with the background rock surface. In optical images animal tissue (probably periosteum) is distinguished from the inorganic matter basing upon the different colour (red-brown, opaque white respectively), while the SEM micrograph (from a secondary electrons detector, low vacuum mode) provides the vision of fine bone residue topographical features. Inorganic parts seem to be melted together in a compact paste showing indeed linear traits, and their texture differs completely from that of organic matter.

Fresh wood residues pertaining to *Quercus ilex* (Figure 7) present characteristic brown-reddish colour when observed with optical devices. Elongated structures (fibres or vessels) can be distinguished (in the enlarged figures). Here both secondary electrons and backscattered detectors have been used (Figure 7C-F). While the former provides high morphological detail, the latter, which gives contrast based on atomic number, has proved to be extremely helpful in detecting organic residues when scanning rapidly the rock surface.

Antler residues appear at SEM micrographs in the form of mud-cracking layers (backscattered electrons detector, Figure 8B), feature also documented through optical microscopes (Monnier *et al.*, 2012). For this residue type, an elemental mapping is also provided (Fig. 8.1-5), which is very useful for checking the spatial distribution of the various elements. Apart from the rock chemical elements (Si and O), the major antler components have been detected (C, Ca, P, Mg, K) (Chen, *et al.*, 2009).

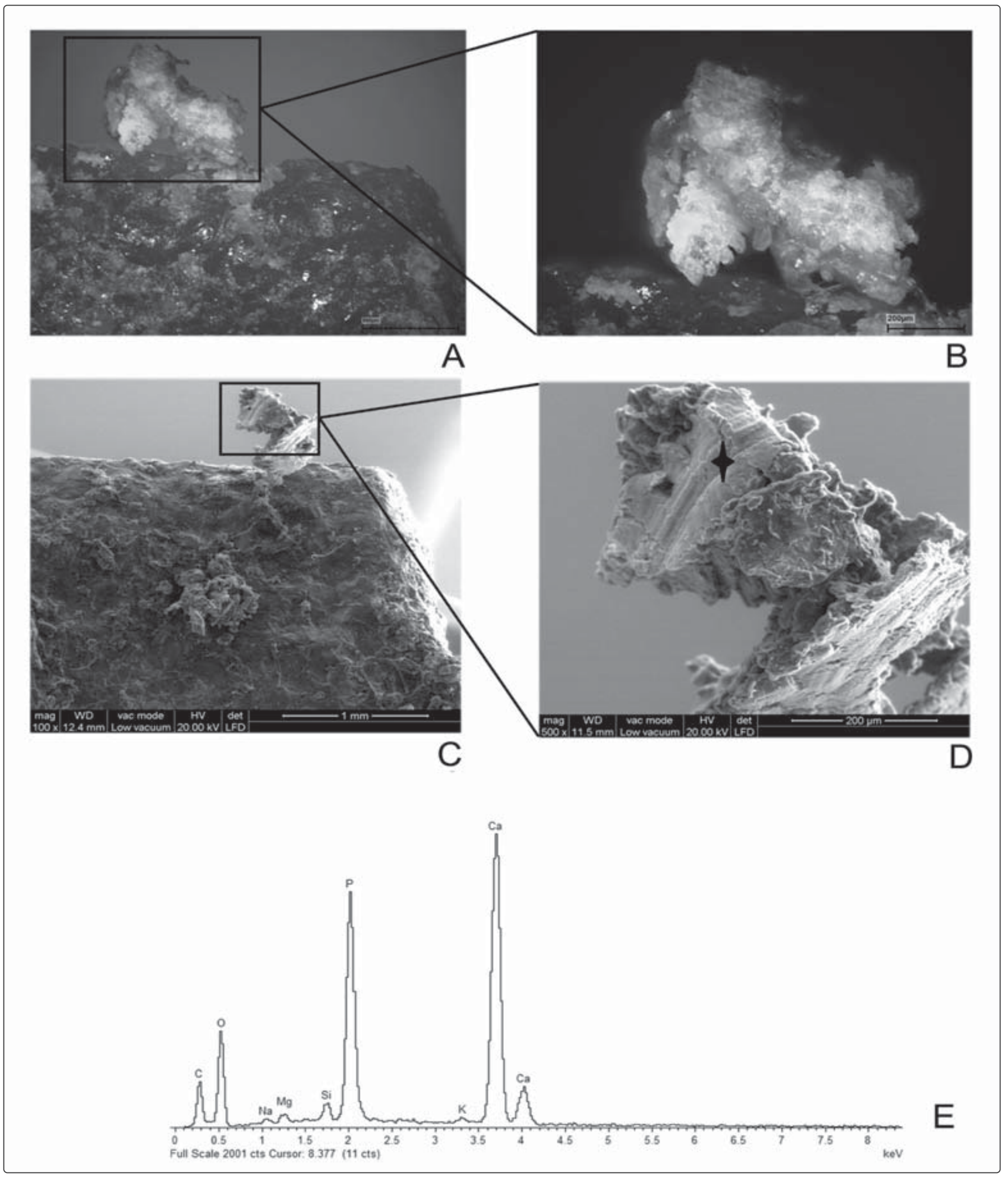

Figure 6: Experimental bone residue detected on a flake edge (QTFU1-06) used for scraping a bone.
A) Metallographic image (stacking of 20 images, extended focus mode):
original magnification, 50X, scale bar: 500 µm;
B) Metallographic image (stacking of 20 images, extended focus mode):
original magnification: 100X; scale bar: 200 µm;
C) SEM secondary electrons micrograph: original magnification: 100X; scale bar: 1 mm;
D) SEM secondary electrons micrograph: original magnification: 500X; scale bar: 200 µm.
The four points star indicates the exact point where the microanalysis (E) has been done;
E) The compositional analysis of this residue showed picks of calcium (Ca), phosphorus (P), oxygen (O)
and the presence of magnesium (Mg) and potassium (K),
all the main components of osseous tissue.

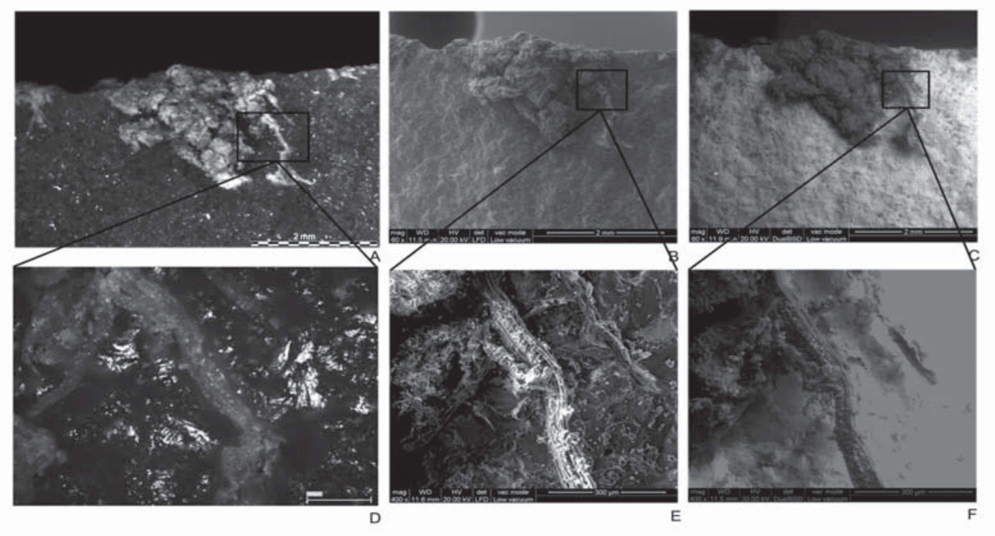

Figure 7: Wood residue from flake QTFU1-08:A) Stereomicroscope image: original magnification: 20X; scale bar: 2 mm; B) SEM secondary electrons micrograph: original magnification: 60X; scale bar: 2 mm; C) SEM backscattered electrons micrograph: original magnification: 60X; scale bar: 2 mm; D) Metallographic image (stacking of 20 images, extended focus mode): original magnification: 100X; scale bar: 200 µm; E) SEM secondary electrons micrograph: original magnification: 400X; scale bar: 300 µm; F) SEM backscattered electrons micrograph: original magnification: 400X; scale bar: 300 µm.

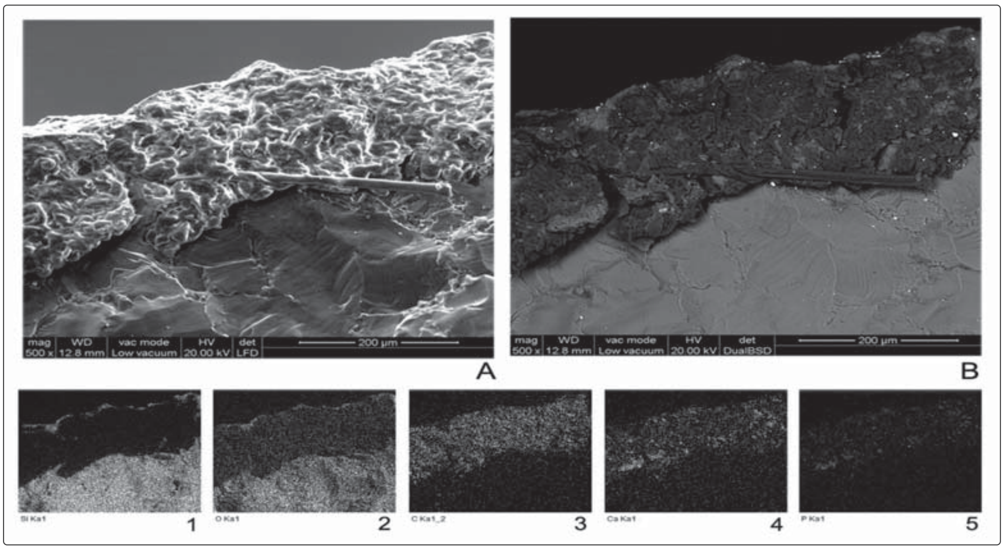

Figure 8: Antler residue including a fibre from flake QUFU1-02: A) SEM secondary electrons micrograph: original magnification: 500X; scale bar: 200 µm; B) SEM backscattered electrons micrograph: original magnification: 500X; scale bar: 200 µm; 1-5) Residues mapping of the detected chemical elements, 1=Si; 2=O; 3=C; 4=Ca; 5=P (the lighter parts indicate when the corresponding element is present). Mapping is very important to show the spatial distribution of the different chemical elements.

3.3. Archaeological preliminary results

Analysing archaeological material is something complementary to the creation of an experimental reference collection. That means that use-wear found on archaeological implements can influence the development of the experimental activity as well as induce to change some parameters of the experimental protocol.

Up to now only four archaeological samples have been analysed following this protocol. No residues analyses have been performed yet. First, a primary evaluation of surfaces modification due to post-depositional events is done. Then, we proceed to record all the modifications due to use on apposite sheets with sketches of the analysed flakes. Some of the analysed tools exhibited wear features slightly different from those found on experimental artefacts (Figure 9: 1; 5), so additional experiments and more archaeological data are necessary before making any kind of interpretation.

Anyway, preliminary results are promising, as use-wear traces have been documented. For instance, the hafting practise, already recorded on flint tools (Márquez *et al.*, 2001), seems to be possibly present.

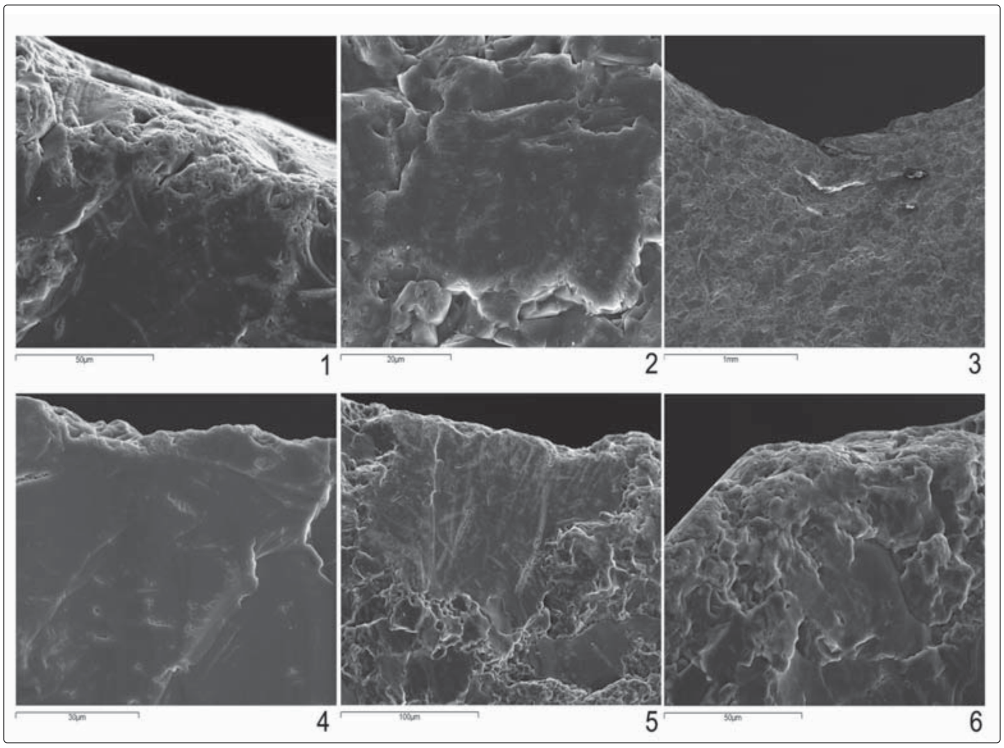

Figure 9: Some wear features on archaeological quartzite samples from Gran Dolina TD10 level:
1) Well developed edge rounding accompanied by striations, mainly perpendicular to the edge;
2) Striations parallel to the edge on the surfaces of grains and general grain edges rounding;
3) Incipient fractures, showing portions of the edge which have not been detached after retouch;
4) Feeble edge rounding plus clear striations parallel to the edge;
5) A massive concentration of very marked striations randomly distributed,
pointing to the occurrence of a certain degree of post-depositional surface modification;
6) Very well developed edge rounding with the presence of some polished areas.

4. Discussion and final remarks

This paper is a first step of a wider research aiming to reach a thorough understanding of the mechanical behaviour of quartzite flakes under stress. Part of a quartzite reference collection comprising various activities and worked material is already available for comparison with the archaeological material coming from TD10 level of Gran Dolina site.

From preliminary results it emerges that quartzite has a brittle behaviour in its earliest phase of use, producing a relatively vast amount of micro-flakes which might be incorporated in a sort of paste, formed by the worked material fine particles. This leads to the intense formation of wear due to use, specifically linear features (striations) on quartz grains which are extremely useful for the kinematics identification, edge rounding and polish areas (depending on the worked material).

This fragile behaviour can be also the reason of the loss of some surface deformations due to use. In fact, the protracted use of the edge makes use-wear to disappear both because of grains ruptures (the highest parts of the micro-topography can constantly suffer some phenomena of breakage) and abrasion. So, friction can cause extreme surface levelling and abrasion, which might erase use-wear previously created. On the other side, during the process of use, the gradual loss of material together with edge rounding make the edge more stable to breakage, having probably slightly increased the edge angle and eliminated the more prominent parts.

Afterwards, preliminary results from the analysed archaeological sample allowed us to set up the following experiments. Some variables which had been not taken into account have been subsequently added, based on some unidentified wear found on some archaeological samples. For instance, experiments with hafted objects are going to be performed as well as others dealing with post-depositional modification. Also technological features connected with retouch are going to be investigated. To find out the standardization degree of use-wear development on quartzite more research is surely needed

Finally, we need to underline the full complementary of both of the employed microscopic techniques to achieve a more accurate use-wear analysis: OLM observations have to be combined with the SEM ones. It is worthy to try to take advantage of both techniques, dealing with the immediacy of OLM and the SEM high resolution potential. Clearly, for understanding the processes of use-wear formation high magnifications and resolution are necessary, so SEM employment plays a predominant role.

Acknowledgements
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1.4 Structure of the thesis

This manuscript is the result of the conjunction of two models of thesis writing: the traditional one and the North-European model, which proposes that the doctoral thesis is composed of a set of published works on peer-reviewed journals.
The unitary concept of the traditional doctoral thesis has been therefore adapted to our research purposes and, as far as possible, we have been seeking at producing informational blocks which could be transformed into publishable works. All of these packages needed to follow the same background line of thoughts. A contextual coherence should be the solid base on which the thesis is built up. In this way, the denser informational blocks should be developed into papers, which will replace chapters or part of chapters of doctoral theses. In the introductory and conclusive sections, one should explain and justify the unity of the published works and their coherence within the main objectives of the research.
Therefore, eight peer-reviewed papers compose the main body of our thesis. Based on the main topic of the papers, their location along the body of the manuscript has been chosen.
One of the publications, although it partially uses the methodology developed here, goes beyond the scopes of this thesis. Therefore, it has been inserted in this work as an annex (Annex 2).

The thesis has been divided into five main blocks: theoretical part, materials and methods, results, synthesis of results and discussion, and annexes.
It is organised across eight chapters:

Chapter 1 deals with the introduction of the main ideas of this research and discusses very briefly the aims of this study. It comprises a paper (Publication 1), published at the very beginning of the research, which helps in clarifying the main objectives of the research itself;

Chapter 2 is dedicated to the theoretical aspects of lithic studies. It begins to explore concepts as technique and gestures, fundamental in technological studies as well as in traceology. It then describes the main steps of a stone tool life's cycle and focuses on the types of wear that may be added at each step. Moreover, other important concepts in the domain of lithic studies, such as object, tool and instrument, are underlined.
It is concluded with the presentation of the techno-functional approach and the proposal of its combined application with use-wear analysis.

Chapter 3 begins with a brief review of functional analyses in lithic studies. Different methodological approaches are discussed and a second publication (Publication 2) is inserted to introduce the topic dealing with microwear research on quartzose rocks. A small note about microwear research on quartzite only is also given and the main use-wear studies of Lower Palaeolithic collections are listed. A brief review of residue analysis on lithic material is also provided. The chapter concludes with some remarks on experimentation and methodological problems in functional studies;

Chapter 4 forms the framework of the research and discusses the methodology used in this thesis. The experimental programme is described in detail. This chapter discusses the utilisation of sequential experiments, the cleaning procedures applied before microscopic observations and the microscopic techniques employed in this study. It also discusses the characteristics of the different varieties of quartzite included in this study, with major details on two of them. The petrographic study of these is provided in the form of a published article (Publication 3). Another article (Publication 4) discusses the problematic related to modern contamination in microscopic studies involving stone tools;

Chapter 5 constitutes the main body of this thesis. It outlines the results of the experiments designed to test a short range of contact materials and use-actions. It discusses the appearance of use-wear traces and their formation processes. The variation of microwear appearance as well as a large photographic documentation are presented (Publication 5). Moreover, this chapter includes a photographic catalogue of our experimental residues (Publication 6). Finally, the limitations and possible future developments of residue analysis are discussed;

Chapter 6 opens a new section of the thesis. It presents the archaeological sites studied in this work. This chapter provides wide contextual information of the Sierra de Atapuerca and the main research steps undertook in the last decades. It then focuses on the studied site: the Gran Dolina site. In addition, recent studies about the TD10.1 level are presented. Then, the results of techno-functional and use-wear analyses of the quartzite assemblage are described and contextualised;

Chapter 7 is dedicated to the presentation of the Payre site and the microwear results of the quartzite assemblage. The results are contained in a paper (Publication 7);

Chapter 8 is divided into a discussion explaining the research goals were addressed and a summary of the main results. The impact of our research on the scientific community and its major beneficial aspects regarding functional studies are discussed. The chapter ends with the conclusions and future perspectives which act as a direction for future research.

The Annexes constitute an important part of the corpus of the thesis and their location at the end of the manuscript only responds to organisational constraints. Annexes 3 to 6 are only provided in digital format.

Annex 1 contains a first systematic attempt to quantify micro-polish on experimental quartzites by means of Laser Confocal Microscopy;

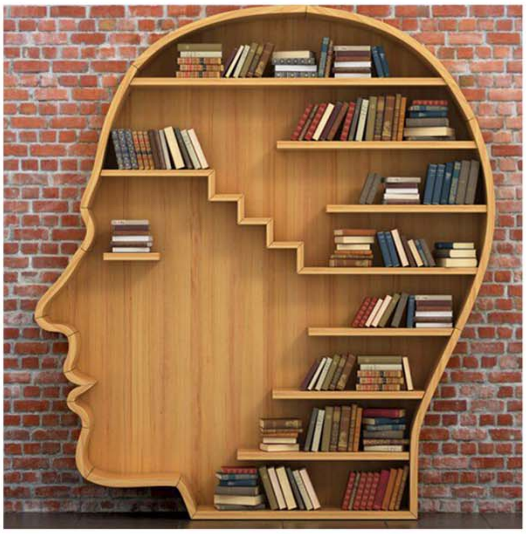
Annex 2 is the result of a published work and deals with butchering experiments involving avian species (Publication 8). The comparison of data coming from functional analysis of stone tools and analysis of cut-marks on bones is conducted. It provides important insights about the correct identification of micro-residues types;

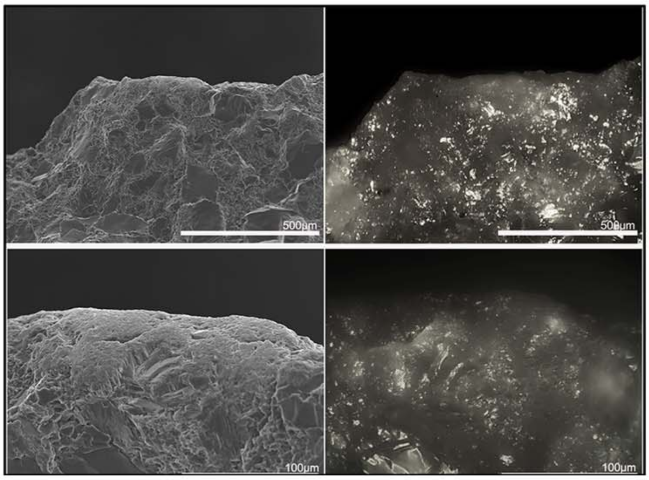
Annex 3 includes all the field and laboratory documentations of the performed experiments and the use-wear forms for all the experimental tools. It is available only in digital format;

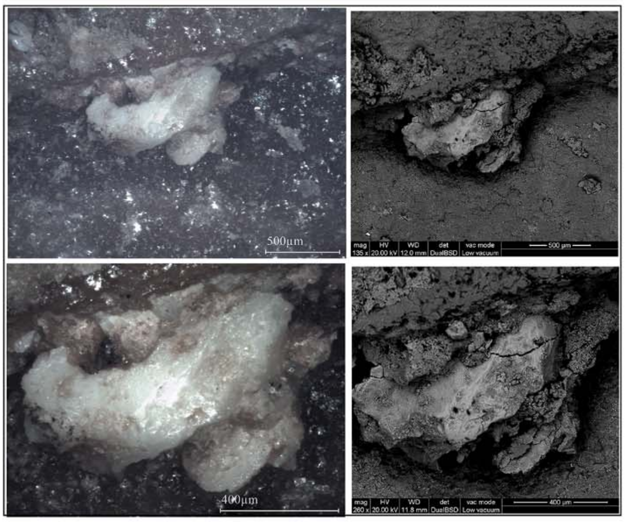
Annex 4 includes all the use-wear recording forms for all the observed archaeological implements. It is divided into two parts, separating the material coming from the two archaeological sites. It is available only in digital format;

Annex 5 refers to the compiled database of the analysed archaeological tools and the related use-wear results. It specifies the absence/presence of use-wear, post-depositional traces, their respective intensity, and the interpretation resulting from the analysis. It is available only in digital format.

Annex 6 is a recollection of the technological data of all unretouched and retouched flakes analysed. Moreover, a table including all artefacts ascribed to techno-functional categories is provided.







PART I: THEORY

Chapter 2: Theoretical concepts in the study of stone tools function

Techniques are to be defined as traditional actions combined in order to produce a mechanical, physical, or chemical effect, these actions being recognised to have that effect (Mauss, 1967:24).

2.1 Technic and gesture

The function of a tool comes into being through the application of a force (or energy), in the form of an action, to reach a definite objective. To perform this action, it is necessary to possess a set of techniques.

Technique is a traditional and efficient act, which cannot exist without tradition (Mauss, 2003: 407, 1st ed. 1947). Although most techniques are materialised through the presence of an external object or instrument, Mauss incorporates the notion of bodily action (*technique du corps,* in French) into the broader and more general idea of technique. Hence, corporal attitudes and movement must be considered as manifestations of techniques and therefore, traditionally determined. The main aspects of bodily techniques are that they are in constant development and they are culturally determined (Mauss, 2007:25-26, 1st ed. 1967). In fact, there can be no technique or transmission without tradition (Mauss, 2003: 407, 1st ed. 1947).

The genesis of technique is to be found in the human intervention on the external world. It involves the application of sets of rules, through a conjunction of actions carried out in order to reach an objective (Leroi-Gourhan,1945). Technique can be seen as an indispensable interface between Man and Nature. The study of technique allows us to better understand the know-how and socio-economic behaviours of past human groups (Fogaça and Boëda 2006:674). Undoubtedly, technique is an important part of the cultural heritage of the people who are using it (Lemonnier, 1986).

Techniques can also be combined, thus forming complex technical systems. According to Lemonnier (1992), a technical system should be considered on three different levels: 1) the techniques themselves; 2) different techniques and technical conjuncts developed by a society, which can mutually influence each other and which are the technological system itself; 3) the interaction of the technical system with other cultural phenomena. If technique is the interaction of different elements, such as matter, gesture, energy, objects and knowledge (Lemonnier, 1992: 4-6), the description of a technical system starts with the analysis of the *chaînes opératoires* (operating or operational sequences) from which the objects originate (Leroi-Gourhan, 1964). These are also composed of a series of acts (or gestures) which have the same aim. The elements are, on the one hand, the agents and the energy they use, and on the other hand, the instruments used and the raw material which will be transformed. The agents can be either human or animal and the energy can have different sources (human, animal, natural). The tools comprise both the manual ones (active and passive) and the machines. The raw material varies and may be directly transformed into a final product, or it may be the result of an anterior operational sequence (Leroi-Gourhan, 1987, 1st ed. 1964).

If technique involves gestures and tools at the same time, organised by a true syntax which gives the operational series both their stability and flexibility, therefore, it is crucial to study the only manifestation of these complex systems, found at the archaeological excavations. In archaeology, the sole techniques which can be analysed and deduced are those embodied by the material culture which survives post-depositional alteration.

Another concept which is necessary to understand technique is 'gesture'. In fact, gesture allows the manifestation of technique. Technical gesture is a term used to refer to those specific movements that are applied when an action comes into being through technique. Gesture can be at once individual and collective, concrete and abstract. When gesture is defined as 'a manner of carrying the body', it may often express an attitude or an emotion or simply an everyday action, carried out practically without thinking (Darwin, 1965, 1st edition 1872; Leroi-Gourhan, 1983, 1sr ed. 1965; Mauss, 2003, 1st ed. 1950).

Broadly speaking, *chaînes opératoires* may be realised through a single gesture, a repetition of the same gesture or a series of different gestures (Balfet, 1975). During lithic production, gestures are seen as a product of trajectory, strength, prehension and body posture (Whittaker 1994; Fogaça, 2006: 19). Hence, technique is a combination of actions and constitutes the means necessary to obtain a blank from a core (Boëda, 1997). If technique always implies a transfer of energy, it generally happens through the use of hard or soft hammers when stones are knapped to produce flakes or blades (*e.g.* Inizan et al., 1992; Whittaker, 1994).

Lithic technology is the rational study of techniques, defined by Inizan et al. (1995) as a conceptual approach to study material culture through the analysis of techniques and gestures (Inizan et al., 1995: 13). Therefore, technological studies consider both the concepts of material culture and gestures. If we see material culture as the product of the relationship between people and objects, including the meaning they give to them (Miller, 2007: 6), it means that material culture encodes a great amount of information about the technology that is related to the objects' manufacture.

The limitation of the technological approach is to over-emphasise the technique itself, sometimes forgetting who stands beyond the tools. All mental predetermination, abstract thought, projected function related to a knapped object and, ultimately, the performance of function through the use of instruments, are generated by human groups existing within determined social environments and governed by specific rules, which all form a part of culture.

Conversely, by trying to understand how tools work, more attention is given to the manipulator-agent, who applies a set of gestures to take ownership of the tools (Mello et al., 2007:35). In fact, "*without the action animating it, the tool is nothing*" (Lemonnier, 1986:154). Through this perspective, the manufacture of a tool and the performance of an action are intimately connected. In fact, a tool is normally produced to be used, that is to say to perform an action aimed to satisfying a specific need. Tools are therefore manufactured while bearing in mind possible tasks for them and that is why form and function are inter-dependent. The necessary criteria (morphology, angle and delineations of cutting edges) that the tools will have needs to be pre-determined and so tools are shaped in accordance to the objective at hand which, in turn, is determined by a problem-solving attitude.

In Prehistory, the cultural items which have most favourably survived through time are stone tools. In fact, very frequently these tools (or parts of tools) constitute the only testimony of technique in the archaeological record. They therefore embody, on the one hand, the diachronic evidence of their production (Dauvois, 1976) and, (or parts of tools) (or parts of tools) and in some cases, the traces of their utilisation. They thus represent alternative sets of techniques (*chaînes opératoires*), which were applied by humans to manipulate the external world (*e.g.* cutting meat, chopping wood).

2.2 Stone-tool's life cycle

The life cycle of a stone tool is defined as the transposition of moments from its manufacture until its abandonment. Based on this assumption, it comprises all phases of technical production, use, curation and even abandonment. A subsequent phase may be present, which refers to its post-depositional exposition to external agents (trampling, soil movements, etc.). After burial, the life cycle of a stone-tool is momentarily interrupted until it is unearthed either by ancestral human groups (who might re-use it), or by archaeologists. If a tool is re-used, it may display newly-formed traces. Archaeologists may also modify stone tool surfaces by adopting careless protocols. In early excavations, archaeological material was often assembled into large bags for storage, and this obviously provoked extensive damage to the lithics (friction traces, gloss, linear streaks of polish, rounding of ridges, etc.).

The sequence of a tool's life is initiated when it is made or selected, usually to fulfil a particular need to modify or access matter. Surface modifications as well as residues may be accumulated on a tool during practically all the stages of the its life. Use-wear studies aim to differentiate the modifications caused by the use of tools from those having other causes.

Production:

During the early phases of a stone-tool's life cycle, several kinds of technical traces may be produced. Striations, plastic deformations, macro-fractures may be produced during this phase (Fig. 2.1).

- *Blank production*: this may require the creation of a specified shape, either by knapping it from a core or by shaping cobbles (*façonnage*);
- *Retouching*: when the blanks do not present the criteria required for performing specific tasks, edges are retouched either on the active part (the cutting edge) or

on the prehensile part (generally opposed to the active part). This is done in order to guarantee a better grip (for instance reducing the sharpness of an edge, to remove unnecessary, prominent parts, etc.) or for hafting purposes;

- *Hafting:* tools may be hafted into a handle before being used. Therefore, traces of this manufacturing stage as well as of the de-hafting practise can be present along with those related to use (Rots, 2010).

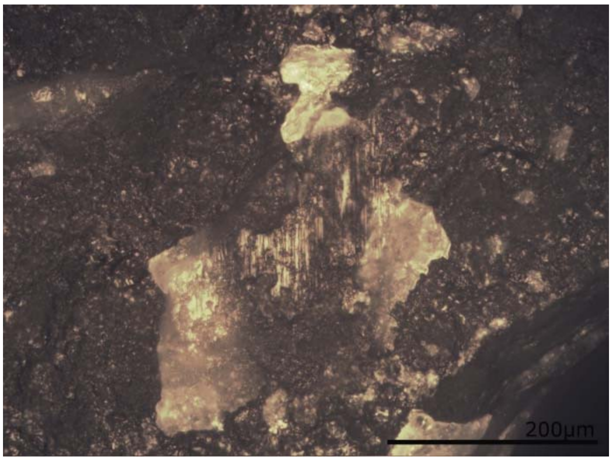


Fig. 2.1: *Technological wear due to the retouching of a flint implement. Plastic deformation found near the retouched edge and produced by the use of a hard hammer (quartzite), 200x (courtesy of J.L. Fernández-Marchena).*

Use:

- *Use to perform specific activities:* longitudinal, transversal, rotational, repetitive gestures on various kinds of organic or inorganic matter (*e.g.* cutting meat, scraping bone, perforating skins). During this phase, surface modifications and fractures which most intrigue use-wear analysists are formed. Residues directly connected to the action performed always adhere onto the surface of tools. If these survive the burial processes, this is another issue;
- *Re-sharpening:* edges are sometimes retouched again during use to re-formulate some qualities necessary for them to be functional, therefore usable (such as, the angle or the edge outline). Additional technical traces may be produced at this stage;
- *Transportation:* Tools can be transported from one site to another before use or in between multiple stages of use. Transportation may cause what is called 'bag wear';

Abandonment:

after all the possible uses of a tool, it is finally abandoned. The character and intensity of any further surface modifications it is subjected to depend on several factors (environment, weather conditions, time and context of the pre-burial exposure, type of sediments, etc.).

- *Burial phenomena:* These generally include post-depositional movements of the soil, trampling, bioturbation and all the activities which could modify the burial conditions and therefore, the stratigraphy (holes, tunnels, etc.).

Extraction of the tool from the sediment:

- Performed by prehistoric groups: this may start a new life cycle of the tool including sub-sequential phases of use (re-sharpening, use and abandonment);
- Performed by archaeologists involving specific methodological procedures normally aimed at the careful extraction of the objects. Even so, some additional traces may occur (such as large polished lines due to the contact with metal trowels or other tools used to excavate) (Fig. 2.2);
- *Storage and laboratory analysis of the tool:* a tool begins a new life at this moment, during which modern wear and residues may be accumulated. Wear can be related to improper storage of the artefacts or to accidental breakage. Residues, however, are far more likely to be produced as the minimum interaction with the archaeological tools is capable of depositing some kinds of substances (better described in 4.6 section of this thesis: Publication 4).

Fig. 2.2: *Metal residues left by trowels during excavation (courtesy of L. Tumung).*

In sum, many actions may mechanically alter the surface of stone tools in different ways. Experimentation is used to infer the cause of the formation of wear (production-related, functional, post-depositional). To do so, the archaeological evidence is systematically compared with experimental referential data. It is important to bear in mind that use-wear connected to an archaeological occupation of a site may be overlaid with traces of subsequent use by the same or different human groups. Moreover, transportation or even deposition, excavation or further analyses may all leave traces on tools that must be distinguished from use-related wear. At any stage of the life-cycle of a stone tool, from the time of manufacture to final curation and analysis, changes of the surfaces which affect the presence of wear and residues may constantly occur.

Hence, this issue is quite complex and functional interpretations should always take into account all the possible sources for the production of wear. The highest aim is, of course, to be able to distinguish wear caused by factors other than use and to eliminate this background noise from the final interpretations.

2.3 Object, tool, instrument

Tout objet est porteur d'un schème de fonctionnement. Sa fonction essentielle est de transformer des matériaux. Ce schème est l'essence même de l'objet, il est la raison de son existence (Boëda, 1997: 41).

Broadly speaking, the term 'object' can be used to define every kind of external physical manifestation perceived by a subject. Objects can be both manufactured and used or only used by humans (natural, un-modified objects). Tools (or artefacts), on the contrary, are always objects which are created by humans, by modifying the surrounding reality.

In Anthropology, tools, or technical objects, are seen as the materialisation of the interaction of matter with the means to transform it (Simondon 1968, as cited in Rabardel, 1995). The question is, are all technical objects artificial? It seems that an object is 'technical' if it can function, as a means or as a result, within a technical action (Mello et al. 2007). A more specific distinction is made for the 'material object which has been manufactured' or 'artefact', which defines all objects which have been minimally altered by man (Rabardel, 1995: 49). The interesting point here is that an artefact does not need to be used to be considered a tool. The mere fact that it has been *produced* is sufficient, as a human agent is responsible for its creation. Traditionally, only lithic artefacts which have been secondarily modelled by retouch are called tools. The un-modified flakes are not called tools, even if they bear traces of use.

If the term tool comprises every object that has been used (Rabardel, 1995: 49), which is a 'testimony of the exteriorisation of an efficient gesture' (Leroi-Gourhan, 1984, 1st edition 1943), therefore un-modified but used flakes should also be considered as 'tools'.

From this perspective, Rabardel (1995) defined the concept of 'instrument'. Basically, every instrument is a tool accompanied by a movement. Hence, all lithic objects should be inserted into a system wherein there is an interaction between the object itself, the person who is using the object and the environment (Lepot, 1993). The techno-functional approach in lithic studies is based on this concept and sees a tool as a moving entity, always as a part of a system comprising the users of the objects and the surrounding environment.

Therefore, the function of stone tools acquires a fundamental meaning, as it is the ultimate aim of all lithic production. The tools we analyse embody all the efforts which led to their production as well as their functioning mode. In other words, tools are produced by people in accordance with their future functions and, particularly when edges are affected by secondary modification (retouch), a particular morphology has clearly been sought after. Therefore, *"Every artifact is either an implement or else an object of more direct use, that is, belonging to the class of "consumers" goods. In either case, the circumstances as well as the form of the object are determined by its use. Function and form are related"* (Malinowski, 2002:151, 1st edition 1944). This does not mean that specific functions correspond to determined morphologies, as in the traditional typological vision. If we assume this, then the human agent is excluded from the whole vision of the object. In fact, from this perspective, the object itself encompasses all explanatory and existential possibilities and specific functions would always correspond to determined morphologies. Since it is known that standardised types might have served to perform different activities, the main focus goes back to the producers and users of tools: humans.

However, form also matters since tools that are used are always chosen by humans based on combinations of features which render them 'usable' and 'functional'. The volumetric structure and technical characters of the artefacts are then fundamental aspects for the comprehension of their essence.

The decomposition of the object into different components acting in synergy to obtain a desired effect, led Boëda to see an object as a mixed entity, comprising the object *sensu strictu* and the schemes of its utilisation (Boëda, 1997; 1998 2001: 52).

The object itself is the conjunction of its volumetric construction and its ways of functioning (Fr. '*instrumentalisation',* Boëda, 2001: 52). According to this author, the production of a specific object depends on a number of constraints: from the set of techniques available in the period of production, to the specific know-how of the human group to which the producer of the object belongs (Boëda. 2005).

The study of tool use schemes (Fr. '*instrumentation*') is based upon the view of the moving object that is constantly related with the user and the matter being transformed. This interaction takes place within a specific spatial context. This vision allows to decompose tools into different *techno-functional units* and has led to the formulation of a new approach to lithic studies: the techno-functional approach.

2.4 The techno-functional approach: a definition

The techno-functional approach, firstly named 'techno-morpho-functional approach', was formulated by E. Boëda (Boëda, 1997). Based on the Rabardel's concept, which sees in objects intrinsic schemes of utilisation and therefore, functioning modes, Lepot first described the system into which objects are inserted as acting in synergy with humans and worked materials (Lepot, 1993). He then defined three types of contact related to each of the participants in the system: 1) a receptive contact between Man and energy; 2) a prehensive contact between Man and the objects; 3) a transformative contact between the object and the worked material (Lepot, 1993). In the Lepot's work, based mainly on the approach of Rabardel, a new conception of the object has been proposed, which involves its insertion into a system composed of humans and their needs, as well as the object itself and the material which is being transformed.

Based on this assumption, the object itself can be decomposed into three different parts, acting again in synergy with respect to each other (Boëda, 1997, 2001). Such decomposition is achieved based on the concept of the techno-functional unit. The latter is formed by a number of technical characteristics which coexist within a synergy of effects. Some technical features involved in the definition of techno-functional units are: edge angles, delineations of surfaces and edges, planes of intersections of surfaces, etc. (Boëda, 1997:46). The presence of these characters on specific portions of the edges of stone tools allows the individualisation of coherent units, which, when observed together, provide a whole perspective of a tool. The analysis of the combination of the various techno-functional units allows to understand an object in movement (Boëda, 2001). More specifically, the criteria which permit to define techno-functional units are: angle, frontal and sagittal delineations of edges and the morphology of the intersection of the ventral and dorsal surfaces forming a dihedral (Fr. *plan de coupe*). Then, depending on the assemblage analysed, different scales of analysis can be attained. For instance, much attention may be given to the description of retouch (analysing the consequences of retouch on the final morphology of the edges) or on the volumetric conception of tools (*e.g.* bifaces).

Therefore, three different techno-functional units are described on every object (Fig. 2.3):

- the <u>transformative techno-functional unit</u> (t-TFU), defined as the part of the object which is in contact with the worked material;
- the <u>prehensile techno-functional unit</u> (p-TFU), defined as the portion of the object which is in contact with the user with or without an intermediary body (hand-held instruments *vs*. hafted ones);
- the <u>transmitting techno-functional unit</u> (tr-TFU), the part of the object which receives the motion energy and transmits it to the worked material (Boëda, 1997). As it can be confused with the p-TFU, it is not always considered in the analysis (Boëda, 2001:53; Bonilauri, 2010:55; Lourdeau, 2010:67).

Numerous works appearing in the last decades have contributed to the formulation of the techno-functional approach and to its application to the study of lithic industries (Bourguignon, 1997; Soriano, 2000; Koehler, 2009; Bonilauri, 2010; Lourdeau, 2010; Chevrier, 2012; Rocca, 2013).

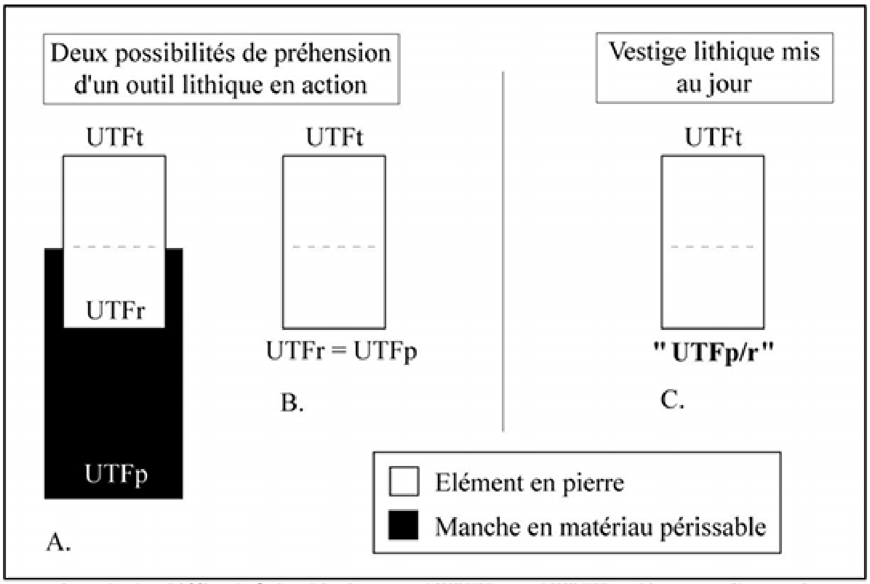


Fig. 2.3: *Scheme of the disposition of the three techno-functional units on tools. A) Hafted element, the distinction between the three different TFUs is clear; B) hand-held element, the prehensile and receptive TFUs are confused; C) Archaeological tools, whether they were hafted or not, the prehensile and receptive TFUs are always difficult to be differentiated (Lourdeau, 2010:67).*

During the overview of the available literature, parallels were noticed between the techno-functional approach and the morpho-potential analysis proposed by the Spanish Logical Analytical System (Carbonell et al., 1983, 1992; Ollé, 2003; Vergès, 2003). The basic concepts of the analysis of the morpho-potential of lithic tools were largely formulated based on an Airvaux' paper (1987). The 'morpho-potential element' is defined as the theoretical capacity of intervention of determined morpho-technical structures, which are identified by the morphology and angles of edges (Airvaux, 1987, 1994). The idea of the decomposition of a tool into different dimensions (*e.g.* morpho-technical, morpho-potential and morpho-functional) is present in the Logic Analytic System (Carbonell et al., 1983, 1992). Regarding tool function, the concept of 'morpho-potential unit' has been used to refer to the portions of the tools presenting a given set of features that confers them a potentiality for use (Ollé 2003; Vergès 2003). These parts were conceived as the basic units according to which use-wear data had to be organised and assessed. Indeed, some parallels between the SLA 'morpho-potential units' and the 'transformative-techno-functional units' found in the techno-functional approach may be seen.

Generally speaking, techno-functional analysis is applied after the study of the lithic production (technological analysis). When techno-functional analysis is coupled with microwear analysis, two analytical levels are described. First, the prehensile and transformative units are identified through the documentation of use-wear on tools. Second, the correlation between the techno-functional characters observed on tools and the functions documented is evaluated (Bonilauri, 2010: 35, 36).

2.5 A multi-differential approach to lithic studies

… on peut dire que la totalité de l'outillage de pierre est constituée par des tranchants destines à couper, à gratter, à percer (André Leroi-Gourhan, « Milieu et technique »).

If a technical object includes every functioning object and form and function are related, then the morphology of an object must be seen as a structural element of the action to be performed. In order to be functional, an object must present a combination of characteristics which allow to perform an action by applying a series of gestures.

The pre-determination of blanks, followed (or not) by retouching (transformation into tools), considers the disposition and inter-relation of active/transformative parts which are in contact with the materials that are being transformed, prehensile parts (which favour prehension, directly or through a third body, the haft) (Fig. 2.3, B, A) and parts which receive the energy (transmitted by muscular action, therefore the repetitive gestures (Lepot, 1993:20).

Consequently, the selection of a tool to perform an action is done by visualising a function related to a particular object. Therefore, the object to be used needs to respond to several rules and requires specific, physical features. In other words, obtaining 'cutting edges' or their transformation into tools through retouch, are often dictated by functional intentions, intrinsic to the objects, which eventually give meaning to them. If we think about it, traceologists unconsciously perform a similar kind of evaluation of lithic artefacts during the selection process for use-wear analysis, making general assumptions about how 'functional' an edge can be or on how a tool could have been held, etc.

From the considerations exposed above, we can see that the essence of a tool lies in its function, whether it has been performed (manifested) or only thought. The performance of an action is the execution of the purpose for which the tool has been created. The non-utilisation of the tool does not modify its essence, being the result of pre-determined ideas and gestures, inscribed in a sequence of actions, composing the *chaîne opératoire* of their production. For this reason, the modified objects (retouched blanks) are always considered tools, whether they have been used or not.

It is evident that the technical vision of objects englobes their functionality and the functional vision of the same objects intrinsically implies their mode of production. Therefore, it is necessary to find an analytical way to link both the domain of stone tool technological analysis (with techno-functional analysis) and use-wear analysis.

The determination of function is of paramount importance, not only to establish functional patterns of a studied settlement, but also to define which artefacts are to be considered tools, that is to say to assess which artefacts were actually used to modify matter during human occupations at archaeological sites. Always taking into account the limitations of microwear analysis (wear on tools might be not present or might not be developed enough), its application with technological studies can contribute to determining the significance of lithic objects.

Since form, or retouch type cannot be linked *a priori* to specific functions, how can function be determined? We think the answer to this question is to seek out a deeper understanding of the object itself. Given that the same morphology of a retouched edge can be used to perform different actions and that the same action can be performed using different morphologies, we must not limit ourselves only to studying the active parts of objects.

In fact, a stone tool is not only a cutting edge or the function related to that edge. A stone tool, as any other, is composed of different volumetric structures and different parts (techno-functional units); it has an organised structure which allows it to be used. To reach the physical structure necessary to perform specific activities, a particular technological know-how is applied using a set of technical gestures. In other words, the morphology of the instruments can lead to its way of functioning. At the same time, the mode of functioning determines its production. The analysis of the recurrence of the technical intentions (technical choices) on tools allows to discover the functioning intentions. The production schemes are then linked to the function schemes. Finally, the recurrent morphological combinations on the objects embody their functional intentions. Through the analysis of the functional intentions, hypotheses on the utilisation schemes of tools can be proposed.

By analysing the recurrence of combinations of technical traits on objects, techno-functional groups can be defined within studied assemblages. The application of use-wear analysis on the groups defined by the techno-functional analysis can help researchers to evaluate the significance of the production of different technical objects within the same assemblage. In this way, a deeper comprehension of the assemblage itself and of the human intentions beyond it, could be reached.

Microwear analysis can make a step forward, in the sense that it can discriminate which un-retouched blanks might be tools (used artefact). Since normally techno-functional analysis focuses on the presence of retouch (although assemblages with no or few retouched implements have been considered, Koehler, 2010; Rocca, 2013), use-wear results could also complete the vision of the techno-functional groups which do not present any retouch.

This combined approach has been firstly presented by Bonilauri (2010) in her doctoral thesis, where she applied use-wear analysis on a set of techno-functional groups defined within the Levallois points of the Umm el Tlel (central Syria) assemblage. She named the methodology 'techno/traceo-functional' (Fr. '*méthode techno/tracéo-fonctionnelle*') (Bonilauri, 2010:33). The application of this type of analysis has succeeded in determining the functional significance of each techno-functional group defined and also to describe technical relationships among different groups. It also helped in evaluating the functional strategies connected to the intention of the lithic production and specified the relationships with the surrounding environmental resources (Bonilauri, 2010).

Depending on specific research needs, the techno-functional approach can be moulded and used to assess specific questions. For instance, in this thesis, it will be applied only on a selection of artefacts and it will not follow a deep description of the operational sequences of the assemblage. This is because this goes beyond the main aims defined in this work.

Moreover, since the integrated application of techno-functional and use-wear analyses is relatively new and, so far, not many published works on this topic are available, we consider our contribution as a test to better evaluate its potential.

As a matter of fact, the combination of technological and microwear studies have the potential to provide a global vision of lithic assemblages and more efforts should be made to join these two disciplines which, up to now, have remained relatively distinct. Such potential, in our opinion, has not been fully explored and more work is surely needed in the near future.

Chapter 3: Functional Analysis applied on lithic artefacts

3.1 Functional analyses in lithic studies

The function of stone tools has always intrigued prehistorians since the first discovery of stone tools. Due to this widespread interest, speculations on tool functionality have been performed in conjunction with the first studies of lithic assemblages. Typology, which was one of the main branches of lithic studies, is based upon the classification of stone tool morphologies. Stone tools were often named after their speculative function, and types like scrapers, points and knives started to enter into the common terminology utilised in the field of lithic studies.

In the mid-nineteenth century, some scholars started to remark the presence of macro-traces visible to the naked eye on some tools. Edge rounding, linear features and bright polish were documented for the first time, sometimes with the aid of magnifying lenses (*e.g.* Evans, 1872; Spurrell, 1892, as cited in Stemp et al., 2016). Polish on sickle was one of the most commonly studied wear features, due to its visibility and clear attribution to use (Curwen,1930, as cited in Stemp et al., 2016).

Systematic microwear studies began with the publication of a pioneering work, which not only led to the foundation of a new discipline, often referred to as *traceology*, but also deeply influenced its future development and modern refinement. This work, entitled “Prehistoric Technology”, was written by a Russian scientist, Sergei Semenov, and was first published in 1957. It was subsequently translated into English, in 1964, which allowed the discipline to reach Western Europe and America. In his work, Semenov (1964) stressed that even tools of the hardest raw material retained traces of their use. He was primarily able to observe and classify a variety of polishes and striations (linear features) using a binocular microscope. He also defined the major aim of microwear analysis, which is essentially to explain how a lithic tool was used, by detecting what kind of material was worked, how long the action lasted, and what gestures were employed by the craftsman (kinematics).

The methodology designed by the Russian archaeologist was then adopted by a number of scholars. Particularly, foreign researchers were able to learn the method directly from Semenov in his laboratory and then to diffuse it in their own countries (Phillips, 1988). This was the case of Ruth Tringham, who made an important contribution to the development of microwear studies in America. In the 1970’s and 1980’s, she formed a group of students at Harvard University in the recognition of use-wear traces by using a stereomicroscope, just as the creator of the discipline did. Meanwhile, on the other side of the Atlantic Ocean, metallographic microscopes were adopted to perform microscopic analysis of lithic surfaces, and therefore incorporated into the discipline. The foremost scholar in developing functional studies on the European continent was without a doubt Lawrence Keeley (1980). He performed the identification of microwear on lithic tools basically by using metallographic microscopes and, therefore, higher magnifications compared to previous traceologists (Tringham, Odell). His work clearly influenced the further development of the method (*e.g.* Vaughan, 1985; Van Gijn, 1990; Juel Jensen, 1994).

Hence, different methods or approaches within the same discipline were progressively defined and the consequences of this methodological differentiation continue even today. However, more than different and opponent schools, as they have been sometimes considered, it is preferable to see in them a divergent and parallel development of the same basic know-how, which eventually came to be split into two specialised tendencies. In fact, the methodology is the same; the observed (wear) is also the same. What changes is the way the observer observes (hence, the technical equipment he/she uses) as well as the types of wear which can be recorded (micro-scars, polish, striations) using different kinds of equipment. Different sets of data are extrapolated and then used to propose interpretations about the original function of the tools. Therefore, different methods or approaches can be identified, rather than different schools.

Traceology (Semenov, 1970:5), from the French word '*traces'* (Eng. traces), is the study of artefacts' productive functions and modes of use through the analysis of the modifications due to use preserved on their surfaces. It was officially introduced into the Western World following a conference held at the Simon Fraser University (British Colombia, Canada), in 1977 (Hayden, 1979), which brought together eminent scholars in the field (*e.g.* Del Bene, Hayden, Kamminga, Keeley, Newcomer, Odell). During this conference, some of the, at that time, most debatable topics about microwear, were discussed. Such issues included terminology, experimentation, the mechanics of scar formation, the role played by raw material variation in use-wear studies, the adequacy of different microscopic techniques, etc.

Although during the formulation of the discipline, materials other than flint have been sporadically incorporated into functional studies (*e.g.* Knutsson, 1988a; Mansur-Franchomme, 1988, 1991; Sussmann, 1988), it must be underlined that not many efforts have been undertaken to extend this methodology to non-flint raw materials. In fact, the available functional studies refer almost entirely to flint (among others, Tringham et al. 1974; Keeley and Newcomer 1977; Keeley 1980; Vaughan 1985; Grace 1989; Van Gijn 1990; González-Urquijo and Ibáñez-Estévez, 1994; Rots 2010). When other raw materials have been analysed, very frequently the same analytic method, which had been set up specifically for flint, has been applied with the result of a great scale of biases.

Coarse-grained rocks, such as basalt (Price-Beggerly, 1976; Stafford, 1977; Odell and Odell-Vereecken, 1980; Richards, 1988; Rodríguez-Rodríguez, 1997-1998), rhyolite (Foix and Bradley, 1985; McDevit, 1994; Clemente-Conte and Gibaja-Bao, 2009) and quartzite (Toll, 1978; Greiser and Sheets, 1979; Kamminga, 1982; Plisson, 1986; Alonso and Mansur, 1990; Pereira, 1993, 1996; Leipus and Mansur, 2007; Hroníková et al., 2008; Aubry and Igreja, 2009; Cristiani et al., 2009; Gibaja et al., 2009) have not been thoroughly studied from a functional point of view. However, very few analysts have focused their research on non-flint rocks and specific methods for analysing them were proposed (Knutsson 1988a; Richards

1988; Sussman, 1988; Hurcombe, 1992). Among non-flint raw materials, quartz has received the most attention from use-wear analysts and therefore, their expertise in recognising wear on this material has been enlarged (Beyries and Roche 1982; Kamminga, 1982; Sussman, 1985, 1988; Fullagar, 1986; Pant, 1989; Knutsson, 1988a, 1988b; Alonso and Mansur, 1990; Bracco and Morel, 1998; Pignat and Plisson, 2000; Derndarsky and Ocklind, 2001; Lombard, 2011; Igreja et al., 2007).

Recently, an increasing interest to deepen knowledge about microwear on non-flint materials is noted, but these contributions are still relatively isolated studies in the discipline of traceology. For example, basalt (Asryan et al., 2014), obsidian (Kononenko, 2011), quartz (Derndarsky, 2009; Eigeland, 2009; Taipale, 2012; Taipale et al., 2014; Venditti, 2014; Knutsson et al., 2015), rock crystal (Fernández-Marchena and Ollé, 2016), quartzite and rhyolite (Clemente-Conte and Gibaja-Bao, 2009; Lemorini et al., 2014) and limestone (Hortelano-Piqueras, 2016) have appeared in recent works on use-wear. Moreover, use-wear on non-flint raw materials has been the central object of sessions in recent international conferences (Clemente-Conte and Igreja, 2009; Sternke et al., 2009).

Since the appearance of the first pioneering work (Semenov, 1964), raw material has been considered as a key variable in the development of wear and, as a consequence, in its visual features. This topic was extensively debated in the historical conference at the Simon Fraser University (Greiser and Sheet, 1979; and discussion in Hayden et al., 1979:297-299). This proves that the high impact which the raw material type has on the appearance of use-wear has been a generally acknowledged fact, since the origins of traceology. This knowledge has been somewhat ignored by many who simply adapted the methodology developed for flint to other raw material types (especially on coarse-grained materials).

The new tendency is to perform specific experiments to control the development of wear on the different raw material types. In that way, reliable experimental and comparable use-wear data are obtained for each raw material. Creating use-wear collections for each raw material highly improves the reliability of the functional interpretations proposed.

3.2 Different approaches

The current field of microwear analysis recognises two main levels of magnification, depending on the technical equipment employed in the study. These two approaches were named after the range of magnifications used; the low-power approach for low magnifications and the high-power approach when use-wear was observed under higher magnifications. While the use of optical microscopes can be linked with both low and high-power approaches, the introduction of Scanning Electron Microscopy can only refer to the high approach, if we consider only sample magnification.

The basic functioning of optical microscopy is briefly introduced to provide an elementary understanding of the method (Murphy, 2001). An optical light microscope is an instrument that uses visible light to produce a magnified image of a specimen. This image is projected onto the retina of the eye or onto an imaging device (*e.g.* from a camera to a computer's screen). It works through the aid of two lenses; the objective lens and the eyepiece (or ocular), to produce a final magnified image of the object (Fig. 3.1). During observations, a real image of the specimen is produced by the objective lens. When the eye is looking through the objective, the cornea and lens of the eye, working together with the objective lens, produce a second real image of the object, which is then perceived and interpreted by the brain (or projected onto a camera to take a picture of it).

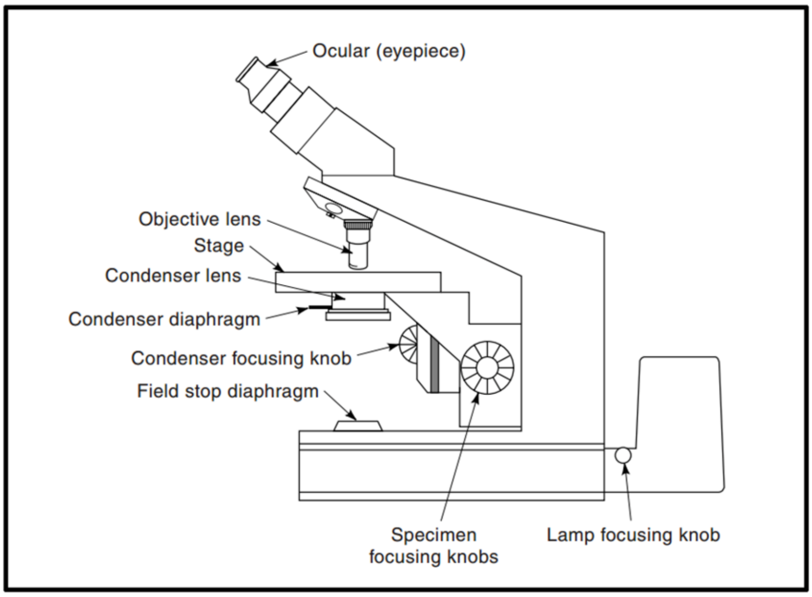


Fig. 3.1: *Scheme of a regular optical light microscope (modified after Murphy, 2001:2 Figure 1-1).*

3.2.1 The Low-power approach

The classification of microwear by means of stereoscopic binocular microscopes with magnification generally ranging from *ca.* 5x to 70x and either incident or external lighting is called the low-power approach (Odell, 1975, 1981; Odell and Odell-Vereecken, 1980). In 1974, a group of researchers led by Ruth Tringham published a contribution where they described a large series of experiments with different kind of worked materials (Tringham et. al*.* 1974). They succeeded in classifying the negative scars found on the used edges, which are produced by conchoidal micro-fractures when a certain amount of pressure is exerted from contact with the worked material (different loading conditions). The morphological characteristics of scars, their orientation and distribution allowed different materials and gestures to be recognised. Striations and well-developed polishes, like those on sickle-blades, are sometimes detectable by stereomicroscopes. The main exponent of this method was George Odell, a member of the original Harvard group (Odell and Odell-Vereecken, 1980; Odell, 1981, 1988).

During the Conference on Lithic Use-Wear in Burnaby, in 1977, scholars discussed fracture mechanics among various topics (Cotterell and Kamminga, 1979; Lawn and Marshall, 1979; Lawrence, 1979; Tsirk, 1979). Scar initiation (Hertzian or bending) and termination (feather, step, hinge or snap) types were also discussed and definitions were included in the Ho Ho Classification, provided during the conference (Hayden, 1979:133-135).

The Low-power approach is regarded as useful for determining the used edges and kinematics. The hardness of a worked material is also inferred, but specific types of worked materials are not identified. The acknowledgment of these limitations was asserted as soon as this method was presented to the scientific community (Tringham et al., 1977). For instance, it is not applicable to edges modified by retouch, as the negative scars from retouching exhibit equivalent morphologies to scars resulting from use. Soon after, further similarities with scars produced by trampling and other post-depositional surface modifications were pointed out (Levi Sala, 1996; Burroni et al., 2002)

3.2.2 The High-power approach

The High-power approach is based on the employment of metallographic microscopes to analyse wear on stone tools. Metallographic microscopes have an effective range of magnification from 50x to 500x.

This method was first developed by Lawrence Keeley (1980) and generally uses magnifications from 100x to 200x (although the equipment has a more varied range of magnifications, as seen above). The main use-wear features included in the observations are polish and striations. Polish texture, reflectivity and distribution along the tool edge are classified in order to define worked materials. Keeley established six broad categories of possible tool use on: wood, bone, hide, meat, antler, and non-woody plants. The kinematic (*e.g.* piercing, cutting, scraping) is mainly inferred by striations, as in Semenov (1964). Other scholars (*e.g*. Moss, 1983; Vaughan, 1985, Juel Jensen, 1994; Levi-Sala, 1986) have refined and further developed this method. The method itself was defined by testing it on siliceous materials and it is therefore more feasible when applied to fine-grained or vitreous materials (chert, flint, obsidian), rather than to coarse-grained ones.

Problems encountered when analysing coarse-grained materials with a high quartz content are related to slower processes of polish formation, non-reflectivity of polish and extreme reflection of quartz grains (Grace, 1989, 1990; Igreja, 2009). Therefore, the polished areas appear more reduced relative to those found on flint (Clemente-Conte and Gibaja-Bao, 2009). A general low depth of field is also perceived, as coarse-grained materials have very irregular micro-surfaces. They may be described as a succession of ‘valleys’ and ‘hills’ and this is why wear is generally found principally on the highest parts of the topography (hills). In fact, it is logical to think that those parts have the most prolonged contact with the worked material during use and therefore, are the most “modified”.

The effectiveness of the High-power method was verified by a historic blind test, in which Keeley was able to correctly identify the working portions of the tools, the way in which they were used and even the type of worked material in almost every case (Keeley and Newcomer 1977).

Although Keeley is mainly known for his efforts in classifying polish types, he never advocated the sole employment of this particular feature to formulate functional hypotheses.

On the contrary, he clearly described different kinds of striations, based on size and morphology (Keeley, 1980:23), and classified scars according to shape, depth and size, defining the following types: large, small, and microscopic deep scalar; large and small shallow scalar; large, small, and microscopic stepped; half-moon breakages (Keeley, 1980:24-25).

3.2.3 The use of SEM in functional analysis

Scanning Electron Microscopy (SEM) functions through the interaction between primary electrons and the observed sample. A beam of electrons (primary electrons) bombards the surface of a sample, which responds with different phenomena, producing secondary electrons, backscattered electrons and X-rays (Dunlap and Adaskaveg, 1997) (Fig. 3.2). Topographic images are built up when a detector (in high vacuum conditions, Everhart-Thornley detector) reads the signals of the secondary electrons emitted by the surface as a consequence of its excitation by the primary electron beam. Secondary electrons are generated when a primary electron displaces a specimen electron from the specimen surface. The topographical information obtained through the secondary-electron imagining mode always has high resolution. The contrast and soft shadows of the image closely resemble that of a specimen illuminated with light, therefore the images obtained are easily readable and interpretable. Topographic contrast is obtained when backscattered electrons are detected (BSE, Back-scattered electron detector). Backscattered electrons are those primary electrons that have been scattered back to the surface. They give both relative atomic density and topographical information. Elements with an average high atomic number appear lighter in the resulting image, while elements having a low atomic number are always darker.

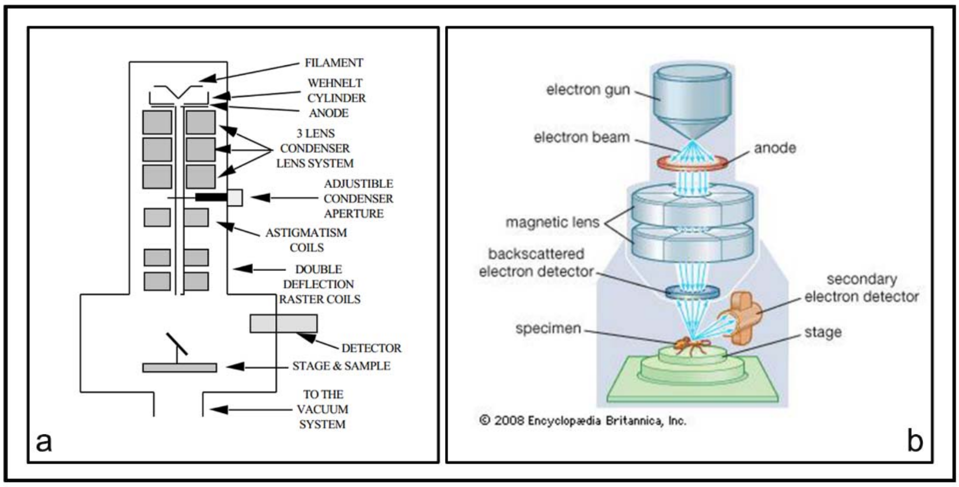


Fig. 3.2: *a) Technical scheme of a Scanning Electron Microscope) (Dunlap and Adaskaveg, 1997:63); b) Visual scheme of a specimen in a SEM chamber (Encyclopaedia Britannica online, https://global.britannica.com/technology/scanning-electron-microscope).*

Originally, SEM was not extensively employed to study microwear on stone tools. It has occasionally been used to investigate problematic issues, such as the detailed aspect of striations (Fedje, 1979; Mansur-Franchomme, 1983; d'Errico and Varetto, 1985; Knutsson, 1988a; Sussman, 1988) or the formation of polish (Anderson, 1980b; Christensen, 1998).

Yamada (1993) tested the potential of systematically observing individually selected locations on the stone tools' edges with SEM for the first time to understand the processes of use-wear formations. Afterwards, Longo (1994) and Sala (1997) were the first who systematically used SEM to analyse both experimental and archaeological stone tools. Further works intensively discussed the value of sequential experiments (and SEM observations of the tools after each stage of use), to categorise use-wear on different lithic raw materials (Ollé, 2003; Vergès, 2003). These studies have highly improved the use-wear analysis method itself, as they took great advantage of the high resolution and magnifications capabilities of SEMs for analysing entire archaeological assemblages.

The high resolution reached with SEM is of great importance when the main aim is to observe how modifications on the surfaces occur. Besides, tiny striations or cracks are only detected at very high magnifications, although they are invisible under regular optical microscopes.

By only referring to the magnifications reached with this equipment, it should be considered as part of the High-Power approach, even if it can achieve magnifications far beyond the 500x reached by conventional optical devises. The higher resolution, compared to OLM, is due by the fact that electrons have shorter wavelengths than photons. As the maximum resolution of OLM is determined by the wavelength of the photons used to illuminate the sample, SEM resolution is dependent on the electrons used to bombard the sample. Apart from higher resolution, SEM also offers other advantages, including the possibility of investigating the elemental composition of a sample. Energy Dispersive X-ray Spectroscopy (EDS or EDAX) uses the characteristic X-rays generated from a sample bombarded with electrons to identify its elemental constituents. The amount of energy released in the form of X-rays is due to the interaction between the electron beam and the sample. It derives from the replacement of the secondary electrons, emitted by the sample, with other electrons, which 'jump' from more peripheral orbitals of the atoms. The detection of these X-rays generates a spectrum in which the peaks correspond to specific X-ray lines, making the elements easily identifiable. Quantitative data can also be obtained by comparing peak heights or areas of a sample with those of a known material.

3.2.4 Combination of techniques

The complementarity of low and high-power observations has been proposed as early as the first establishment of lithic microwear analysis as a distinct discipline (Hayden and Kamminga, 1979). In fact, the two approaches provide analysts with different sources of information, which could eventually be combined. Crossing data obtained using different techniques provides more reliable information, as the inferences made by only using a type of wear can be supported by other observations.

Roger Grace (1989) was the first to conceptualise the idea of hierarchical analysis in microwear studies. He suggested that analysis should be performed on three different levels:

analysis of 1) the edge morphological attributes (macro wear), 2) microwear and rounding using a power microscope, and 3) microwear analysis integrating the above two analysis types with polish analysis employing a high-power microscope.

Regarding quartzite, Grace (1990:10) pointed out that the observation of microwear on quartzite with a conventional optical microscope is very challenging, while macro-wear seems to be more discernible. Polish information seems to be available only by resorting to SEM.

Obviously, there are advantages and disadvantages in employing each kind of microscope (Knutsson, 1988a; Borel et al., 2014). The most feasible solution is clearly to combine various techniques and adapt this combination case by case. Thus, the best approach is to select the most useful techniques and adapt them to the analyst's specific needs, after having considered the raw material type and the kinds of wear which are to be imaged.

3.2.5 Quantification of wear

During the foundation and development of traceological studies, microwear analysis has mainly relied upon qualitative attributes. In early publications, the need to find a way to quantify wear seemed to be the main imperative (Shiffer, 1979:18). From that time onward, relatively little effort was put into developing techniques to quantify lithic use-wear.

Nevertheless, during the last decades, the urgent need to provide more objective data gave way to sporadic works. Digital image acquisition (Grace et al., 1985; Grace, 1989; González-Urquijo and Ibáñez-Estévez, 2003; Lerner et al., 2007; Mansur, 2009; Lerner, 2014a, 2014b) was used to try to recognise recurring patterns of wear by analysing images taken with optical light microscopes. More sophisticated equipment was then incorporated to measure the degree of surface roughness. The atomic force microscope (Kimball et al., 1995), focus variation microscopy (Macdonald, 2014), laser profilometry (Stemp, 2014), and laser confocal microscopy (Evans and Donahue, 2008; Stemp and Chung, 2011; Evans et al., 2014) were developed. Mathematical models, such as fractal analysis, were also occasionally used (Stemp and Chung, 2011; Stemp, 2014). Due to the paucity of standards in this kind of study, none of the above mentioned methods has been systematically incorporated into the domain of traceology.

3.3 Publication 2:

Ollé, A., Pedergnana, A., Fernández-Marchena, J.L., Martin, S., Borel, A., Aranda, V., 2016. **Microwear features on vein quartz, rock crystal and quartzite: a study combining Optical Light and Scanning Electron Microscopy.** *Quaternary International* 424, 154-170.

In the following publication, the main problems related to the analysis of quartzose rocks are discussed. Terminological issues are introduced as well as the implications of using different microscopic techniques to analyse these rock types.

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Microwear features on vein quartz, rock crystal and quartzite: A study combining Optical Light and Scanning Electron Microscopy

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ABSTRACT

In general, quartz and most of non-flint rocks have not been extensively studied from a functional point of view. Very frequently the definitions of micro-features connected with flint surfaces have been used to describe those encountered on non-flint tools. This circumstance has repeatedly posed serious methodological problems for evaluating the accuracy of functional results when analysing use-wear on quartz and quartzite implements. This is due to the intrinsic divergences in morphology and distribution of use-wear with regard to the different lithic raw materials.

Even though important efforts to systematise use-wear features on quartz have been done almost since the beginning of the discipline, there continues to be confusion and lack of standardisation regarding terminology in this aspect.

In this paper, we try to contribute to new insights in this research by means of selecting examples from an extensive experimental programme involving different raw materials: from rock crystal (the purest form of quartz found in nature) to vein quartz and quartzite, with the latter two materials extensively used for knapping throughout Prehistory and still poorly understood in terms of microwear. For data recording, we preferentially used sequential experiments and resorted to both Optical Light and Scanning Electron Microscopy.

We focused our interest on describing the main groups of wear features. The results obtained allowed us to assess the different mechanical behaviours under the stressors induced by tool-use from a group of raw materials with the same chemical composition but very different in structure. Furthermore, we propose the revision of some terms commonly employed when documenting micro-wear on quartz and similar rocks, as well as recurring concepts coming from materials and geological sciences (e.g. tribology, quartz exoscopy...).

1. Introduction

Use-wear studies of non-flint/chert raw materials have not been sufficiently developed in the past and for this reason functional interpretation of such materials is still problematic. This relies on the fact that analysts concentrated their efforts in analysing assemblages mainly composed by flint or chert (generically referred thereafter as chert), because of the feasibility of these material to the easy observation of wear with light microscopy. Therefore, based on wide reference collections, analysts came to broadly know the specific use-wear patterns connected with different actions and worked materials contributing to the creation of a solid methodology (e.g.Semenov, 1964; Tringham et al., 1974; Keeley, 1980; Vaughan, 1985; Van Gijn, 1990).

Most attention is presently paid to the improvement of the technological studies of assemblages composed by quartzose materials (quartz and quartzite), as demonstrated by the contributions to this volume. To join this increasing interest in those materials, it

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is worth reviewing their role within the history of lithic use-wear analysis, and evaluating the methodological problems connected with detecting use-wear on them. For instance, previous studies (e.g. Grace, 1990; Igreja, 2009; Borel et al., 2014) have discussed the difficulty of microscopically analysing coarse materials, such as quartz, quartzite and basalt (as well as for other rocks including coarse particles of other minerals). This can be explained sometimes by the high reflectivity and the resulting bright diffraction halo of the rocks analysed (quartz, quartzite, rock crystal or hyaline quartz) and sometimes by the great irregularity of the flaked surfaces (sandstone, quartzite, basalt, rhyolite). However, a paradox on the suitability of use-wear analysis of quartz using the standard high-power method, especially when post-depositional processes affect the lithic assemblages, has been highlighted (Knutsson, 1988b:122).

At the same time, when definite circumstances promoted the functional study of non-chert raw materials, very extensive and complete methodologies have been constructed (Knutsson, 1988a; Knutsson et al., 1988; Richards, 1988; Sussman, 1988; Hurcombe, 1992; Clemente-Conte, 1995/2008). Usually, this occurred when the great abundance of these types of rocks in some regions was largely reflected in the archaeological lithic assemblages coming from those regions (e.g. Kamminga, 1982; Knutsson, 1988b; Derndarsky, 2009; Eigeland, 2009; Kononenko, 2011). Although based on very in-depth investigations, those contributions alone were not enough to establish a universally recognised method to perform use-wear analysis of those materials. In some cases (Knutsson, 1988a) a very thorough description was presented, combining specific traces with relative actions and worked material, which resulted in very useful comparative tables. Of course, the fact that the method did not reach a general acknowledgement has nothing to do with the quality of the method as such, but with the fact that, in this case, quartz hardly gained the interest of use-wear analysts.

Often the analyses of those materials required procedures to overcome the methodological limitations posed by the classical microscopic analysis, which is based on the reflected light observation. Other microscopic techniques have been employed to improve the potential of use-wear analysis on non-flint materials. Among these techniques, the Scanning Electron Microscope (SEM) revealed to be very useful for imaging purposes from almost the beginning of the discipline (Borel et al., 2014; Ollé and Vergès, 2014, and references therein), and, more recently, the Laser Scanning Confocal Microscope (LSCM) ushered a really promising progress in terms of wear quantification (Derndarsky and Ocklind, 2001; Evans and Donahue, 2008; Stemp et al., 2013; Ibáñez et al., 2014).

Moreover, terminological confusion introduced new problems in an already complicated discipline predominantly dependant on the personal experience of the analyst (Grace, 1996). In fact, very frequently different terms were employed to define the same use-wear trait or sometimes the same term was used to describe different traces. Also, direct analogies between traces found on chert and non-chert implements were made, underestimating the fact that use-wear develops differently on distinct raw materials (Greiser and Sheets, 1979; Clemente-Conte, 1995/2008, 2015; Lerner et al., 2007; Clemente and Igreja, 2009).

Quartzose materials were extensively used in the knapping activity in Prehistory and so it would be desirable to improve use-wear analyses on them. Beside, these materials tend to present better preservation conditions than chert, for example, which is more resistant to post-depositional processes (Knutsson, 1988b). Actually, sometimes use-wear analysts are not able to analyse chert artefacts because of the presence of strong patinas or desilicification processes. This is one of the reasons why we initiated an extensive experimental programme aimed to monitor use-wear formation on lithologies with a very similar basic chemical composition (vein quartz, rock crystal and quartzite). All of those materials are formed by macrocrystalline quartz crystals, but their structures are very different (grain size, flatness, etc.). This programme is currently being built to assist the study of the archaeological materials from the following Palaeolithic sites: Gran Dolina-TD10, Burgos, Spain (Ollé et al., 2013), Santa Ana, Cáceres, Spain (Carbonell et al., 2005), Payre, Ardèche, France (Moncel et al., 2008) and Cova Eirós, Lugo, Spain (Rodríguez et al., 2011).

The main aims of the current project are to assess the degree to which inter-rocks variability among quartzose materials affects use-wear formation and development and also to assess the point at which they present a similar use-wear pattern. For this reason, we highlight the need to precisely and independently describe the main groups of use-wear features on each lithology, to then later compare them. In parallel to the general description of the use-wear patterns associated to each raw material, we consider some propositions on terminological aspects to describe use-wear on quartzose raw materials.

Additionally, we explore the advantages and disadvantages of different microscopic techniques in relation to each of the materials taken into consideration. In fact, the type of microscopic equipment employed to perform functional studies, and the specific expertise of the analysts in doing it, might influence the description of use-wear to some degree. For example, use-wear traits are imaged differently depending on the employed microscope and analysis conditions, and also some traces may or may not be detectable depending on the resolution reached by each observation technique and settings chosen.

2. Materials and methods

2.1. Experimental programme

Experiments and results shown here do not take part of an *ad hoc* programme, but derive from different recent or still ongoing programmes aimed to furnish the needed reference collections to interpret the results obtained in the aforementioned archaeological sites (Ollé, 2003; Martin, 2012; Fernández-Marchena, 2013; Aranda et al., 2014; Pedergnana and Ollé, 2014; Fernández-Marchena and Ollé, 2016; Pedergnana and Ollé, 2016; Pedergnana et al., 2016). All these experimental programmes share the use of different quartzose materials, from different varieties of quartzite to vein quartz and rock crystal. Although these programmes include traditional, controlled and sequential experiments (Ollé and Vergès, 2014), we especially selected examples of the latter type, as they allow the subsequent phases of surface modification to be monitored and the evolution of the micro-relief to be precisely tracked throughout the course of the activity performed.

The monitoring of the wear process was especially interesting in this context because we did not aim to offer a catalogue of wear traces, but to learn how the main wear features originate and evolve on the selected materials after having performed similar actions. In other words, we study the mechanism of wear formation from the progressive development of a worn surface, tracing its progressive modification at single points throughout the use process.

The detailed procedures and general advantages of such sequential experiments have been recently discussed (Ollé and Vergès, 2014). In short, we systematically record the development of use-wear traces at several points in order to document the variability of the effects of a given action on the active edge of a tool as closely as possible. Thus, the experimental tools were analysed before use and then at specific intervals during their use.

2.2. Microscopic analysis

The results presented in this study were obtained by the combined use of optical light and scanning electron microscopes, as these demonstrated to be very complementary techniques (Borel et al., 2014) (Fig. 1). The low magnification approach based on stereomicroscope analyses was only followed for sample screening and location of points of interest.

The Zeiss Axioscope A1 reflected light microscope was used with the differential interference contrast (DIC) system, in which Nomarski prisms confers a 3D-like look to the image, as it has been proved to be suitable for the analysis of transparent and birefringent materials (e.g. Pignat and Plisson, 2000; Igreja, 2009; Fernández-Marchena and Ollé, 2016; Márquez et al., 2016). Here the images were taken with a motorised extended focus system. SEM microscopes were used as shown in previous articles (Ollé and Vergès, 2008, 2014; Borel et al., 2014). Table 1 shows the specific details of the equipment used.

Although initially we planned to carry out the analyses equally combining both optical and electron microscopes in all the materials, we have only extensively the former for the rock crystal while milky quartz and quartzite were more extensively documented under the SEM. This directly stems from the grain size, texture and irregularity of these raw materials, as will be further discussed below.

Although in just an exploratory way, and without any quantification approach so far, we occasionally added the confocal laser scanning to the imaging techniques used, with the aim to get insight on some specific details of the wear process of these materials (see Fig. 9).

3. Results

In this section we grouped the main wear features into the following big groups: edge fracturing, linear features and polish. For each of these essential categories of features we comment on a

Table 1
Specifications of the OLM and the two SEM used during this study. EC = Enhanced Contrast objectives; DIC = Differential Interference Contrast; LD = Long Distance.

Optical Light Microscope (OLM)				
Brand and model	Zeiss Axio Scope A1			
Lineup description	Stand column Axio Scope Vario, 560 mm Upper stand part M27 – HD/FL reflected-light illumination for HAL 100 4 positions reflector turret Binocular phototube 30°/23 (5:50) 100 W halogen illuminator with collector Stop slider A 14 × 40 mm with aperture stop Stop slider A 14 × 40 mm with luminous-field diaphragm Stop slider C-DIC 6x60 EC LD EPN 20×- 50× Polarizer slider A 6 × 30 mm, 90° rotable Analyzer slider D/A, fixed Reflector module brightfield ACR P&C for reflected light Reflector module darkfield ACR P&C for reflected light Reflector C-DIC for reflected light Z motor controler Prior ES10ZE Z focus motor H112			
Objectives				
Model	EC epiplan 5×	EC epiplan 10x	LD epiplan Noefluar 20x HD DIC	LD epiplan Neofluar 50x HD DIC
Model number	422030-9901	422040-9901	422852-9960	442855-0000
Numerical aperture (na)	0.13	0.2	0.4	0.5
Working distance (wd) in mm	16.1	16.1	7.1	6.5
Field of view (fov) in mm	23	23	20	20
Magnification range Eyepieces	50× to 500×			
Oculars	PL 10×/23			
Camera				
Brand and model	Invenio 5S vII			
Resolution	5 Megapixels			
Adapter	CCD adapter 1x			
Software	DeltaPix Insight			
Scanning Electron Microscopes (SEM)				
Brand and model	JEOL JSM-6400			
Detectors	Secondary electron Everhart-Thornley detector (ETD) Back-scattered electron detector (DualBSD) EDX-EXL II system Link Analytical Oxford			
Beam energy set up	15/20 kv			
Working distance used	Between 15 and 20 mm			
Captured image resolution	1024 × 832 pixels			
Software	Oxford Instruments, INCA suite v.4.01			
Brand and model	FEI Quanta 600			
Detectors	Secondary electron Everhart-Thornley detector (ETD) when working at high vacuum Large Field detector (LFD) when working at low vacuum Back-scattered electron detector (DualBSD) for both high and low vacuum EDX-EXL II system Link Analytical Oxford			
Beam energy set up	20 kv			
Working distance used	Between 8 and 18 mm			
Captured image resolution	Up to 4096 × 3536 pixels (used resolution: 1024 × 943 pixels)			
Software	Oxford Instruments, INCA suite v.4.01			

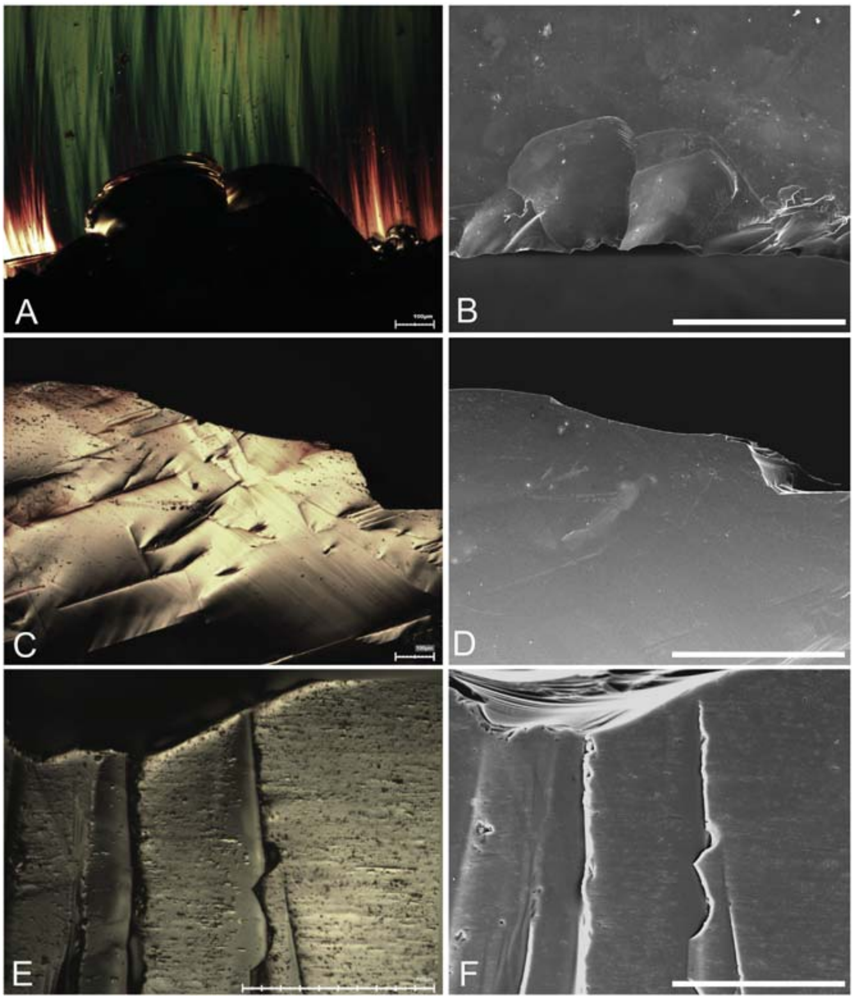


Fig. 1. Use-wear on rock crystal documented trough OLM (A, C, E) and SEM secondary electron detector (B, D, F). Scars outlines are more visible with SEM (B) than with OLM (A), especially because a higher depth of field, while striations are more visible with OLM (C, E) than with SEM (D, F). A) orig. mag.: 100×, scale bar: 100 μm; B) orig. mag.: 100×, scale bar: 500 μm; C) orig. mag.: 100×, scale bar: 100 μm; D) orig. mag.: 100×, scale bar: 500 μm; E) orig. mag.: 100×, scale bar: 500 μm; F) orig. mag.: 100×, scale bar: 500 μm. A and B correspond to bone cutting, and C to F to wood sawing actions.

selection of experimental cases, we broadly assess how they appear on the different raw materials, we determine which terminological issues must be taken into account, and how effective the aforementioned microscopes are to document them.

3.1. *Edge fracturing (scarring, microchipping)*

This is a very well described feature in the literature, also referred as scarring or microchipping. It refers to the micro scars produced on the tools' edges as a consequence of the applied force during use. Studies have traditionally considered the distribution along the edges, the morphology and the termination of the scars as dependent variables of the actions performed and worked materials. This, indeed, has been the base of the so-called low approach analysis, which is still being used in a way that maintains the guidelines stablished since the first proposals (Tringham et al., 1974; Odell et al., 1976; Hayden, 1979; Kamminga, 1982; Prost, 1990).

In all the studied materials different types of conchoidal fractures appear; these include scalar scars, step fractures, half-moon fractures and small crushing. These fractures appear on the edges of the tools, predominantly on their rims, but also on all the exposed quartz crystal ridges.

In general, the bigger the crystals, the better these scars can be documented and consequently used as diagnostic features. Regarding the materials studied here, these features are very clear for rock crystal (see Fig. 1a–b), just clear for vein quartz (Fig. 2), and really difficult to record on quartzite. As already noted (Kamminga,

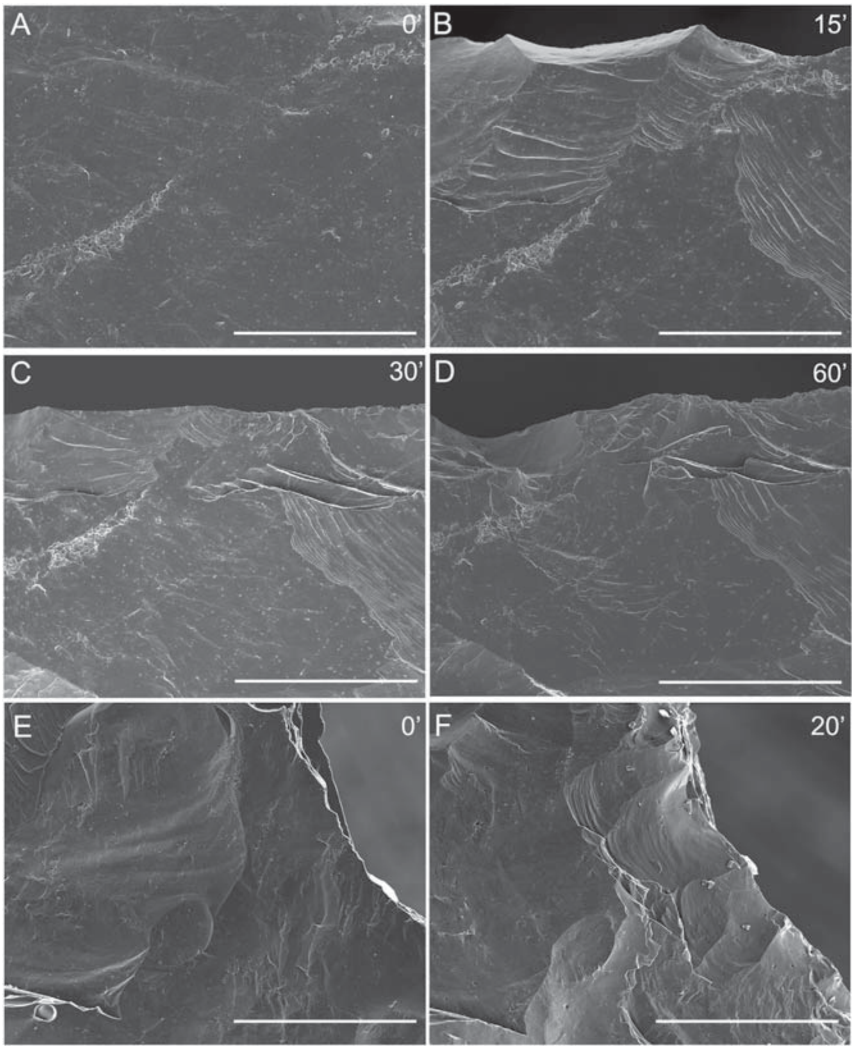


Fig. 2. Edge fracturing on quartz implements. Sequence of micrographs of the same portion of the edge before (A) and after 15, 30 and 60 min of wood scraping (B, C and D respectively); orig. mag.: 100×, scale bar: 500 μm; images of the same portion of the edge before (E) and after 20 min of bone scraping (F); orig. mag.: 100×, scale bar: 500 μm.

1982), problems appear when trying to distinguish individual scar patterns due to surface reflectivity, edge irregularities and unevenness in the quartz grains. These problems can be considerably overcome with the use of SEM; however, case materials such as quartzite continue to pose problems, as the scar limits are very hard to follow (Fig. 3).

There is no doubt that the identification of such scarring in experimental materials can effectively be achieved, especially when sequential series are available. The problem is the identification and the interpretation of these traces on archaeological materials. Probably, the main issue restricting the diagnostic value of this generic feature has to be with its equifinality. Indeed, many potential processes can lead to quite similar scarring patterns. These processes include anthropic actions as the edge modification by retouch, but also different postdepositional phenomenon quite common in archaeological contexts (as object transport, trampling, excavation and post-excavation damage…).

3.2. *Linear features*

We generically refer to as linear feature any naturally or anthropically induced mark on a stone surface susceptible to be microscopically identified. In spite of a certain trend to refer to these marks generically as striations, it must be said that many authors already focused their interest in establishing differences. For example, categories as “linear polishes” (Fischer et al., 1984) or

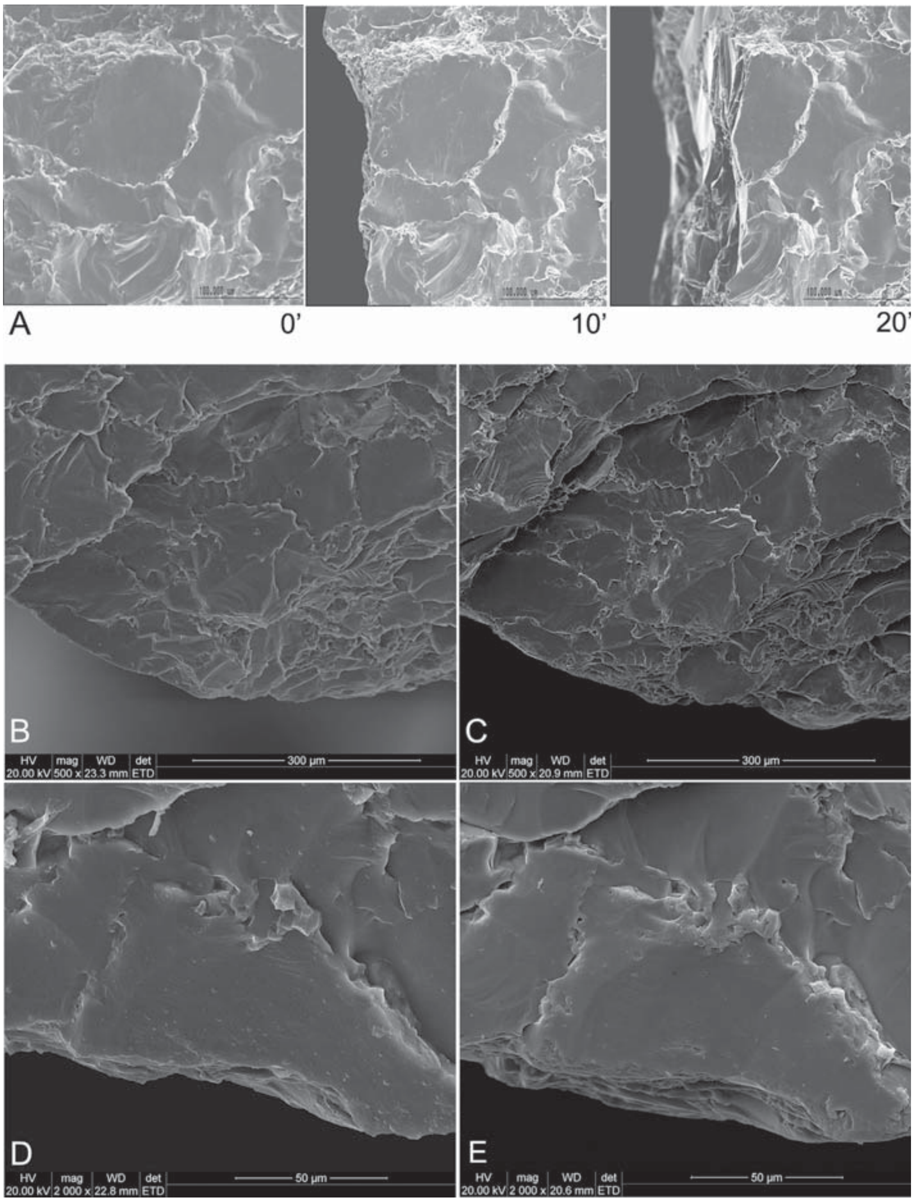


Fig. 3. Sequential experiments on quartzite. A) Edge scarring after 10 and 20 min of wood scraping; orig. mag. 250×, scale bar 100 μm; B and C microscarring on the edge before (B, cast of the fresh edge) and after 20 min of a butchery action; orig. mag. 500×, scale bar 300 μm; D and E details of B and C respectively; orig. mag.: 2000×, scale bar: 50 μm. Note on E some rounding and polish formation on the edge, as well as some linear friction features on the crystal.

"linear impact traces" (Moss, 1983), which usually appear associated to projectiles, or "bands of polish" and "lineal components of polish" (González Urquijo and Ibáñez Estévez, 1994), or even "linear trends" (Kamminga, 1982), which do not show clear limits and thus cannot be strictly considered striations, have been proposed.

Linear features are obviously important for microwear studies, as they are indicative of how a tool was orientated during use, of which type of motion was performed, and can even provide some clues on the type of worked material. The central role of these features was highlighted since the beginning of the discipline (e.g. Semenov, 1964; Keeley, 1980; Kamminga, 1982; Plisson, 1985; Mansur-Franchomme, 1986; Juel Jensen, 1994). Also, a kind of threefold dimension on them (functional, technological and post-depositional) has been noticed by most of authors.

The formation processes of the different linear friction features have been intensively debated. Globally, they are understood as marks produced by abrasive particles on the stone surfaces, these particles being variated in nature and origin. These particles can come from the worked material, from the damage of the tool during use, or they can be intentionally or unintentionally added to the interfacial medium. Obviously, linear features are not restricted to the active edges, as they often appear on other parts of the tools, due to the result of friction actions occurred during production, use and postdepositional processes.

Some basic variables such as length and width have been proposed to be measured to classify the linear features (e.g. Keeley, 1980; Mansur-Franchomme, 1983, 1986), but this quantitative approach has only been occasionally used to assist the usewear interpretation of archaeological tools. Actually, questions regarding their type, intensity and association degree with other wear features, are the preferred interpretative criteria.

Although different classifications and a variated terminology (which sometimes turns out to be quite confusing) have been proposed, linear features have mainly been divided into two big groups: sleeks (narrow and fine striations), and furrows (large and rough ones) (Table 2, and references therein). The former tend to have smooth and regular margins, and seem to respond to the plastic behaviour of the stone surface. The latter, on the contrary, tend to show irregular margins, "which are torn, or broken, or shattered as material was removed by excavation or microfracturing in a way that is somewhat analogous to ploughing" (Kamminga, 1982: 12), and must be explained by the brittle behaviour of the stone surfaces (Knutsson, 1988a).

As it has been noted before (e.g. Kamminga, 1982; Sussman, 1988; Knutsson, 1988a) on the materials studied here there is a clear predominance of the furrow-type. Thus, on quartz, rock crystal and quartzite it is easy to observe scratches in the form linear arrays of microscopic cracks, or holes formed from cracks, caused by brittle fracture after material fatigue in the subsurface zone (Fig. 4). These features have been also referred to as "chatter marks", "crescent", or "incipient cone cracks with shoulder breakage" in other contexts (e.g. Knutsson, 1988a; Madhavaraju et al., 2009). Differences between the studied materials do not seem to be linked to the chemical composition or to the toughness of these raw materials, so we likely have to take into account their differences in terms of crystal grain size. The larger the crystals are, the longer the linear marks tend to appear. Apart from simply the dimensions, it is very important that the homogeneity of the crystal topography (derived from the cleavage planes characteristic of each variety), which situates the rock crystal on one end and the quartzite at the other, placing the vein quartz in an intermediate position.

Sleek striations are really rare. Among the materials studied here, they have only been documented with a certain abundance on rock crystal, and usually associated to already modified surfaces (Fig. 5). Fig. 6 illustrates a continuous obliteration process sequentially recorded in a wood-cutting action, where in advanced stages of use narrow striations with very clear margins appear superimposed to furrow-like striations. These sleeks mostly consist of V-shaped grooves apparently created by an abrasive particle on an already plastically deformed surface, likely due to dislocation within the crystal structure, although in other

Table 2
Summary of the main terminological contributions on the description of the linear features regarding the two main groups considered here: sleeks and furrows.

References	Sleeks	Furrows
Semenov, 1964: 115	Striations: Tiny streaky scratches	Striations (generic use of terms such as scratches, furrows, lines, grooves, and wear striations)
Del Bene, 1979	Striae: formed by addition and translocation of materials	
Lawn and Marshall, 1979:72		Partial Hertzian Cracks
Keeley, 1980:23	Striations: subdivided according to width and depth	Abrasion tracks: often with parallel running, deep tracks
Kamminga, 1979:148 1982:12	Sleeks: linear features caused by plastic deformation	Scratches or furrows: tears in the surface due to microfracturing
Mansur, 1982	Smooth-bottomed through	Rough-bottomed through
Mansur-Franchomme, 1986:95	*Striae à fond lise, en forme de ruban*	*À fond rugueux*
Levi Sala, 1996: 12–13; 68	Linear features: generic term Sleeks: plastic deformation	Striations: furrows or grooves in the polished surface Grooves: opposed to linear features, form on the polished surface principally by microchips removed from the used edge during work
Knutsson et al., 1988	Striations- Linear features- Sleeks: narrow plastic deformations	Striations-Linear features: irregular striations
Sussman, 1988: 13–14	Striae: linear features with smooth-bottom	Linear grooves: gouges or rough bottom striations; Partial hertzian cones
Hurcombe, 1992:58	Sleeks	Crescent cracks: partial surface rings around the contact zone
Fullagar, 2006:222	Sleeks: smooth cross-section, likely plastic deformation of the surface	Furrows: ripping the surface and with jagged margins; continuous or discontinuous
Taipale, 2012: 36, 39	Sleeks: narrow plastic deformations	Discontinuous striations; Irregular striations; Hertzian cone cracks
Quartz exoscopy (Torcal and Tello, 1992; Madhavaraju et al. 2009, and refs. therein)		Striations, grooves, chatter marks

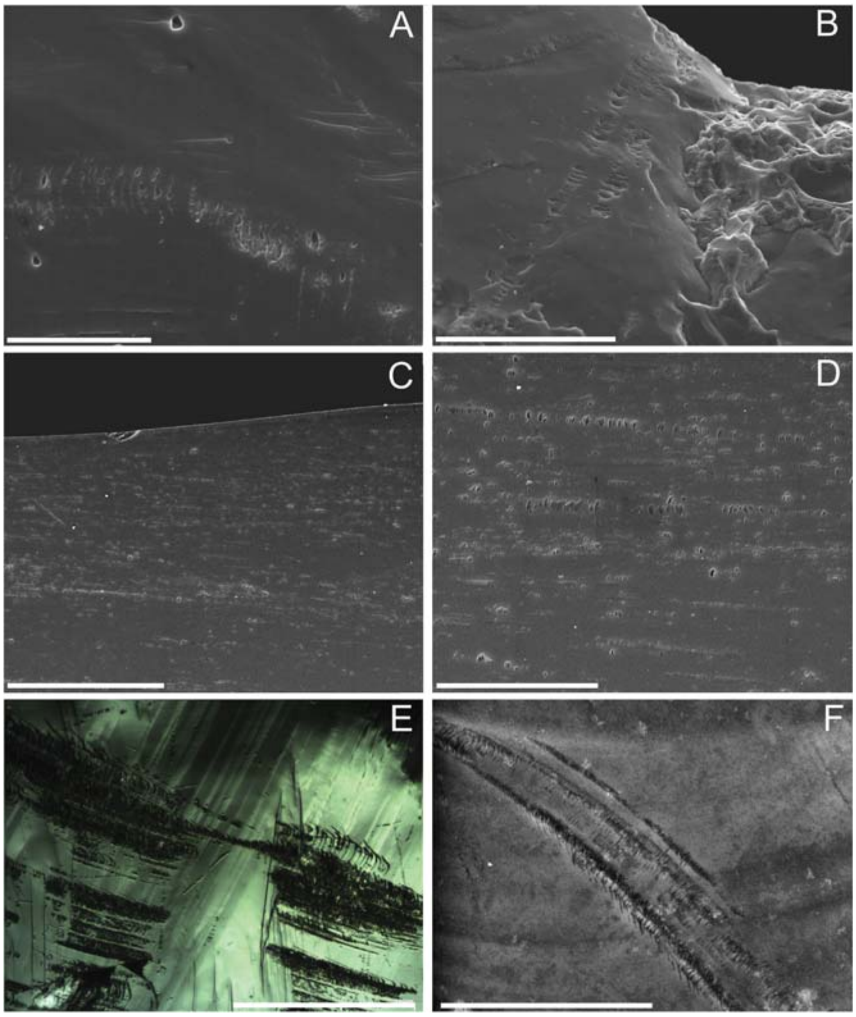


Fig. 4. Linear features exhibiting Hertzian cones on quartz (A), quartzite (B), and rock crystal (C, D, E and F). A) orig. mag.: 2000×, scale bar: 20 μm, cutting fresh bone; B) orig. mag.: 1000×, scale bar: 50 μm; butchering C) orig. mag.: 500×, scale bar: 100 μm, wood sawing; D) orig. mag.: 1500×, scale bar: 30 μm, wood sawing; E) orig. mag.: 500×, scale bar 100 μm, archaeological artefact; F) orig. mag.: 3000×, 50 μm, archaeological artefact.

cases what is preserved seems to be just the deeper part of a furrow-like striation.

Morphological differences among the linear friction features can be noted, but relationships between them and worked materials are hard to establish. There is a general trend to record clearer "chatter-mark" morphologies and more detachment of particles when working hard materials. This leads, for example, to propose the terms "wood striation" and "straight-sided striation" (Knutsson, 1988a; Knutsson et al., 2015) (Fig. 7). Although accepting the appropriateness of these observed associations, it is worth noting that in the tribo-systems several variables take part in the generation of these linear friction features: the physical characteristics of the elements (whether these are abrasive particles or an incident body), the characteristics of the interfacial medium (which affect the contact conditions), and the energy of the dynamic contact between surfaces (Knutsson, 1988a; Ollé and Vergès, 2008; Key et al., 2015). The different combinations of these variables (and other more specific like the holding properties of the worked material) would then promote the formation of more or less linear friction features and lead to the variability recorded in this group of traces.

3.3. Polish

Probably, this is the more described and debated wear feature in microwear analysis literature. It generally refers to the stone tool surface levelling due to the contact with another material. During use (but also during production or once the stone tool is

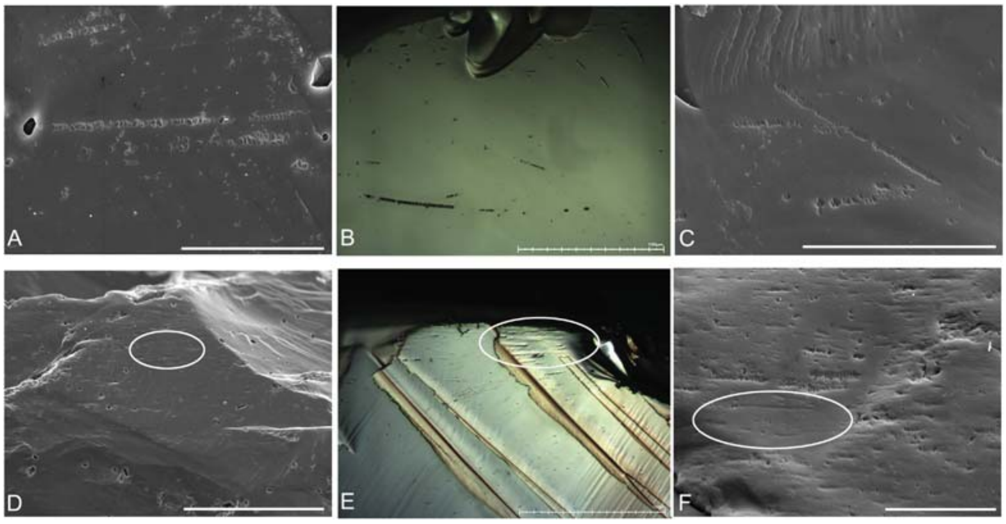


Fig. 5. Furrow striations on quartz (A), rock crystal (B) and quartzite (C). Combination of furrows and sleeks on quartz (D), rock crystal (E) and quartzite (F). White ellipses mark some of the sleeks. In all cases the action was wood cutting/sawing, except in E, which corresponds to a bone cutting action. A) orig. mag.: 2500×, scale bar: 20 μm; B) orig. mag.: 500 ×, scale bar: 100 μm; C) orig. mag.: 3500×, scale bar: 20 μm; D) orig. mag.: 500×, scale bar: 100 μm; E) orig. mag.: 500×, scale bar: 100 μm; F) orig. mag.: 2000× scale bar: 20 μm.

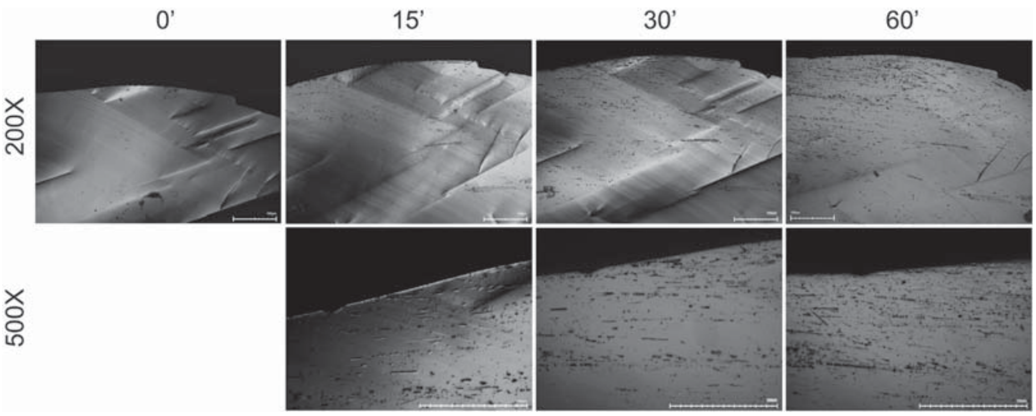


Fig. 6. Sequential experiment on rock crystal in a wood sawing activity, with intensive striation formation and a final smooth edge; orig. mag. 200×, scale bar: 100 μm.

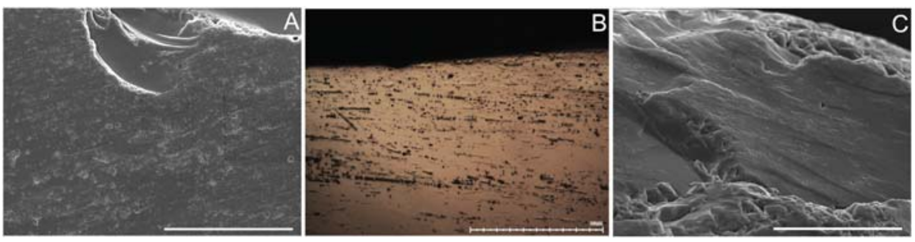


Fig. 7. Furrow striations after sawing wood. A), quartz, orig. mag.: 1000×, scale bar: 50 μm; B) rock crystal, orig. mag.: 500×, scale bar: 100 μm; C) quartzite, orig. mag.: 1000×, scale bar: 50 μm.

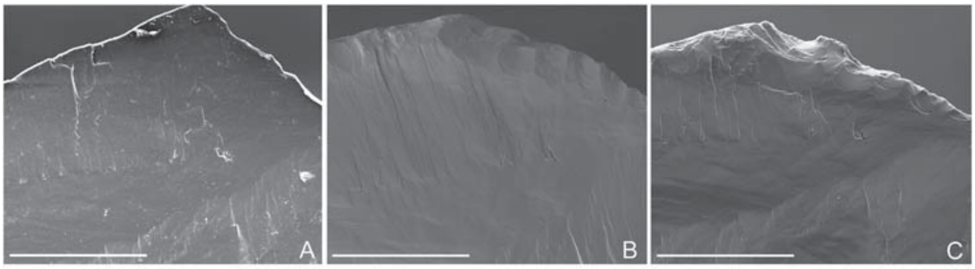


Fig. 8. A control point on a quartz edge after hide scraping: before use (A), after 30 min of use (B) and 60 min (C). Micrographs taken with a high vacuum SEM secondary electron detector, orig. mag.: 250×, scale bar 200 μm.

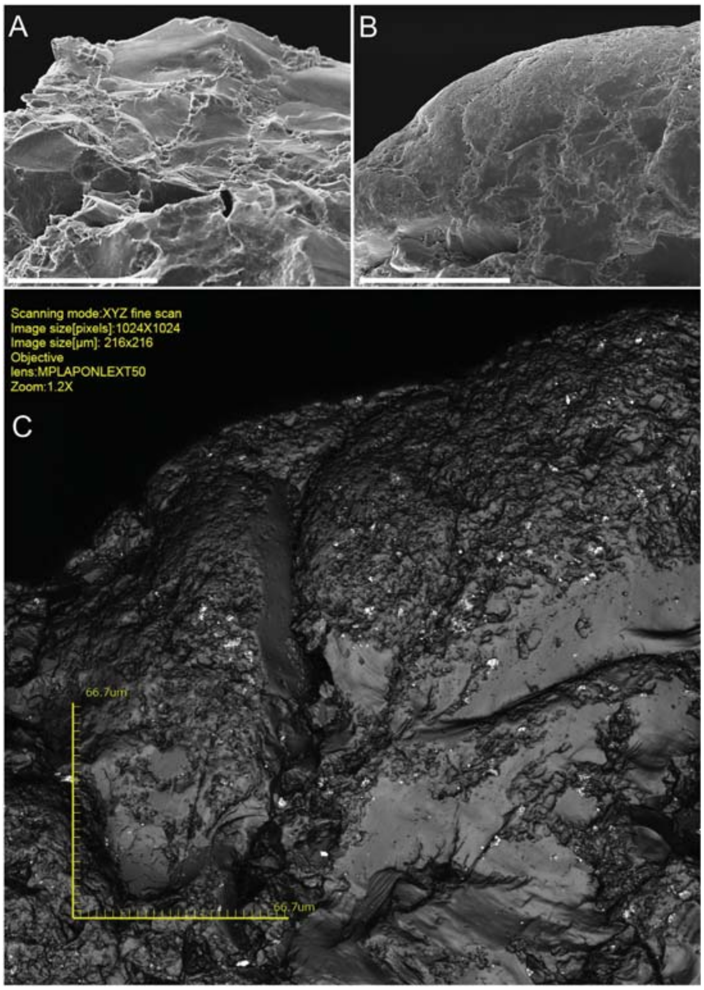


Fig. 9. Formation of abrasive wear on a quartzite artefact after hide scraping for 45 min; A) large crystals on the fresh edge; B) worn surface on the same spot showed in A; C) detail of the central area in B under the LSCM, with the abraded crystal and a clear smoothing of the surface. A, B) orig. mag.: 250×, scale bar: 200 μm; C) scale bar: 66.7 μm.

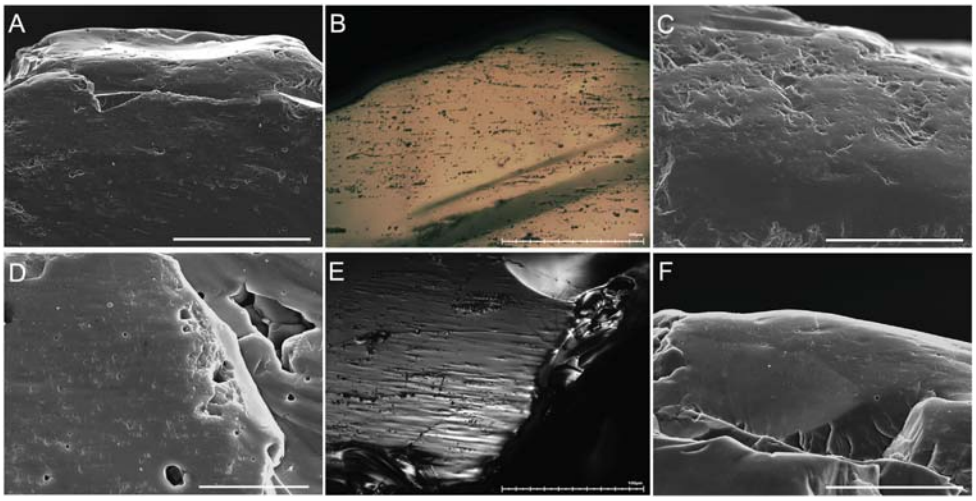


Fig. 10. Well-developed polish on quartz (A, D), rock crystal (B, E) and quartzite (C, D). A) orig. mag.: 250×, scale bar: 200 μm, cutting fresh bone; B) orig. mag.: 500×, scale bar: 100 μm, wood sawing; C) orig. mag.: 500×, scale bar: 100 μm, cane scraping; D) orig. mag.: 2000×, scale bar: 20 μm, cutting fresh bone; E) orig. mag.: 500×, scale bar: 100 μm, bone sawing; F) orig. mag.: 1000×, scale bar: 50 μm, bone sawing.

abandoned), the friction caused by that contact causes a removal of surface material (abrasion) on several scales, from broad roughening to smoothing into a glossy surface.

The polish formation processes have been widely discussed (e.g. Witthoft, 1967; Anderson, 1980a, 1980b; Masson et al., 1981; Meeks et al., 1982; Mansur-Franchomme, 1983; Unger-Hamilton, 1984; Vaughan, 1985; Plisson and Mauger, 1988; Fullagar, 1991; Hurcombe, 1992; Yamada, 1993; Levi Sala, 1996; Christensen, 1998; Lerner et al., 2007). In previous works we contributed to this debate mainly basing on SEM analysis and sequential experiments (Ollé and Vergès, 2008; Aranda et al., 2014; Ollé and Vergès, 2014; Pedergnana and Ollé, 2014), and borrowing theoretical concepts from materials sciences and tribology (e.g. OECD, 1969; Bhushan and Gupta, 1991; Hutchings, 1992; Williams, 2005; Kato, 2006; Momber, 2015), as this approach had been previously demonstrated especially useful (Knutsson, 1988a; Fullagar, 1991; Levi Sala, 1996; Donahue and Burroni, 2004; Anderson et al., 2006; Adams et al., 2009; Delgado-Raack et al., 2009).

Our results led us to consider the polish formation as a clear attritional process, in which a combination of brittle and plastic deformations occur in a very dynamic way, and that leads to worn surfaces by smoothing of the asperities on the stone microrelief. No layer formation has been observed. As other authors already noted (in a very special way after the works by Knutsson and colleagues continuously referred in this article), quartz and similar materials have a more brittle behaviour compared to cryptocrystalline materials such as chert (where plastic deformation is more evident).

Bearing this in mind, we propose to interpret features as edge rounding, smoothing by attrition and smoothing by plastic deformation as different steps of a single general process. Our sequential experiments including quartz, rock crystal and quartzite showed that a quite similar process can be observed, which starts with a strong edge microfracture and is followed by a progressive rounding of the crystal edges by micro-abrasion, and a more or less developed plastic deformation (compression) restricted to the more exposed points of the topography (and only if no subsequent fracturing occurs) (Fig. 8). Obviously, the particular representation, development, and distribution of the different wear features will vary depending on the performed actions, worked materials and use conditions.

Recently, the particularities of polish appearance and distribution for this group of rocks with respect to chert have been properly stressed (Clemente and Gibaja, 2009; Lemorini et al., 2014; Clemente-Conte et al., 2015; Márquez et al., 2016). However, the description of wear phenomena seems to require more accuracy. For instance, what seems to be a mechanical process has been sometimes referred to as corrosion, without having proved the existence of a "wear process in which chemical or electrochemical reaction" occurred (OECD, 1969). Another issue would be the reference to the different appearance of polish on the surfaces of crystals and on the "matrix". Combining different microscopes, we have documented that what can be interpreted as the "cement matrix" is, in fact, the abraded crystal. This is particularly clear in the case of quartzite; as discussed elsewhere (Pedergnana et al., 2016), the term matrix has no sense in the case of metaquartzites. At any rate, and, sequential experiments as the one in Fig. 9 show how a single crystal is abraded by use (hideworking in this case), which may resemble the smaller fraction of sedimentary rocks known as matrix when scanned with OLM. By taking advantage of the resolution of LSCM, a clear polish is observable on the more exposed points of the former microfractured crystal, which appear clearly smooth. This phenomenon can be explained, in our opinion, by a combination of brittle and plastic response of the rock surface to the stress caused by use.

Regarding the diagnostic value of the appearance of the polished surfaces with respect to the worked materials, so far, just a few general observations can be made for the raw materials studied here. In all cases, for instance, hard and/or silica-rich materials tend to produce flatter (more levelled) topographies, as while softer ones tend to show a rough surface, usually ploughed by a variable amount of furrows, and with really scarce plastic deformation. On the former, other than the levelling, other topographical traits (roughness, waviness) observed on well-developed polishes seem to be quite diagnostic. These are the cases of the really smooth and

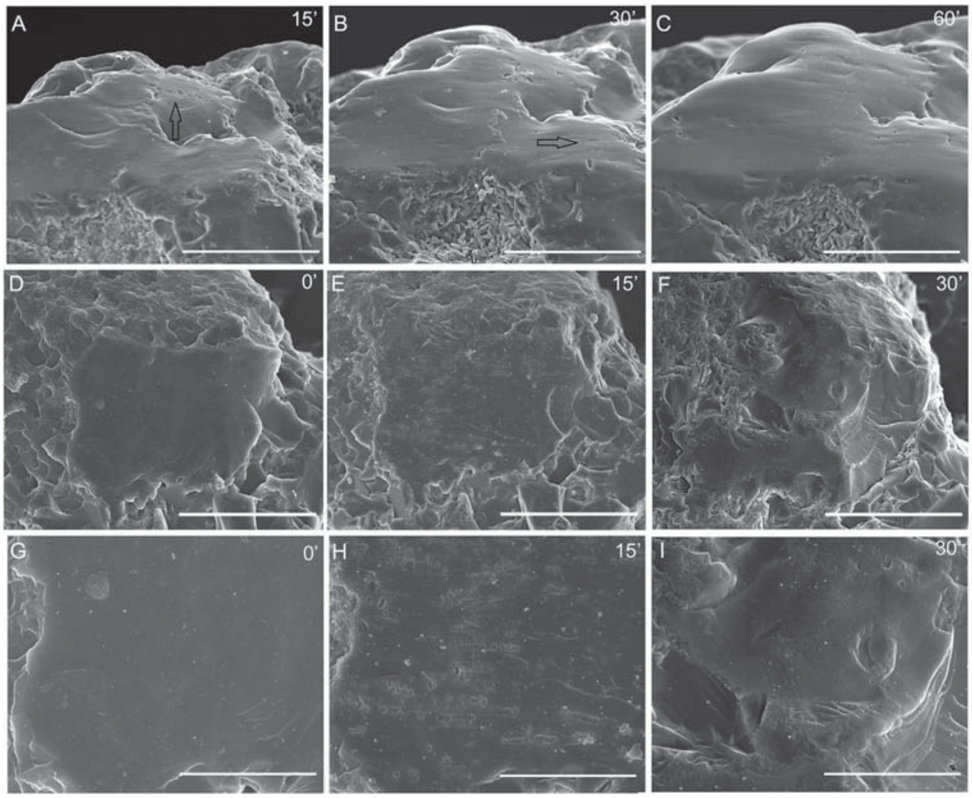


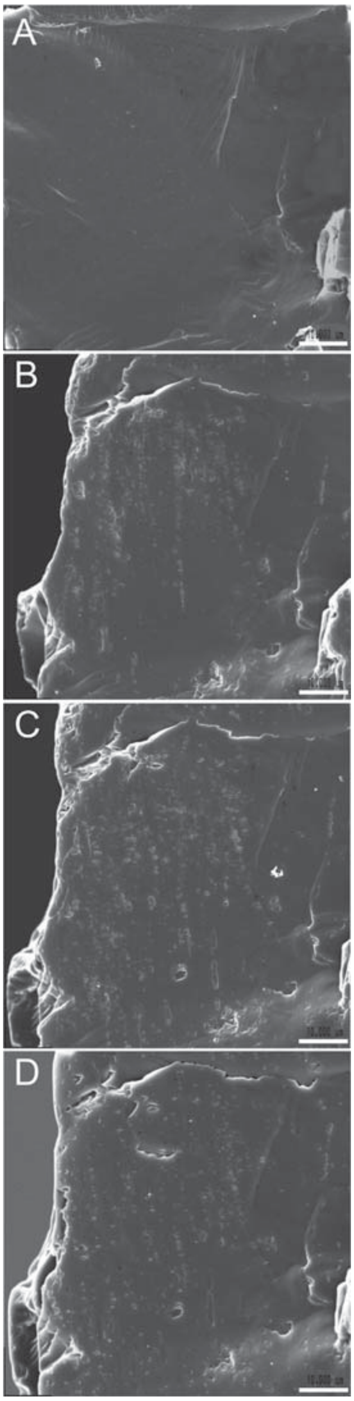
Fig. 11. Sequential experiments on quartzite showing wear development. A to C show the polish development after 15, 30, and 60 min of bone sawing respectively. Sleek striations may form on the polish and then disappear after the flattening of the surface during the process of polishing. Note the clear wavy aspect in the last stage. D to F show the wear formation on a flat surface of a quartz crystal after wood sawing before use (D, cast) and after 15 and 30 min. G to I are details of the same points. Note how after 15 min of use a number of furrows plough the crystals' surface, and how after 30 min these features absolutely disappear because of the detachment of part of the crystal. This, in turn, shows clear wear features in the form of edge microscarring and some smoothing. Micrographs taken with a high vacuum SEM secondary electron detector, A–F) orig. mag.: 1000×, scale bar: 50 μm; G–I) 2500×, scale bar 20 μm.

doomed surfaces for plant materials, of the wavy ones associated to fresh bone (Figs. 10 and 11).

4. Discussion

In summary, if we put aside the differences between raw materials, our research mainly supports the essentially attritional character of the wear process. As such, all the wear features referred to can be framed into this conception as, in all recorded observations, we have identified a high or small loss of matter as a result of the smoothing of the asperities of the microrelief and the loss of an edge portion by fracture and polish. Furthermore, the experiments clearly show the very dynamic behaviour of the tool's contact surfaces: use-wear traces are continuously generated and destroyed, so what we record through microscopic observation is the state at a specific moment during the process, not the final phase of an accumulative phenomenon (Fig. 11). We have also noticed differences between certain episodes in which brittle fracture or plastic deformation alternatively dominates during the use, as well as how edge microflaking often removes pre-existing plastically deformed areas (Ollé and Vergès, 2008). Therefore, "although many factors influence the final appearance of wear features, we do not distinguish between different processes, but rather between different phases of a single process, and therein lies the interest of the sequential monitoring method we propose" (Ollé and Vergès, 2014:69). The experiment shown in Fig. 6 illustrates how the edge of a rock crystal progressively wears during a three-step sawing action on green wood. This process begins with some long furrows produced after the edge microscarring, continues with the progressive scarring, more striations and slight edge rounding, and after 60 min of use shows obliterated furrows, a polished surface with clear signs of plastic deformation, partially covered by sleeks. A similar process can be observed in Fig. 12 on a quartzite tool used a similar action. Here a stronger edge scarring is initially appreciated, being the detached portions of it the responsible of the same type of furrows as in the previous example, and finishing after 30 min of work with a really smooth and doomed surface case, where the furrows practically disappeared.

So far, our results and observations are insufficient to properly assess the wear features on the materials studied here, especially taking into account the huge petrological variability in raw materials as vein quartz or quartzite. At any rate, what is clear is that



these rocks share an essentially brittle behaviour, quite different from other cryptocrystalline quartz varieties as chert of flint. It is for this reason that it made sense consider them together for this study.

Nevertheless, some differences between the wear features have been documented, and they likely derive from the specific material structure. This, and not the rock's chemical composition or hardness, determines its toughness and the way in which the edges wear. Variables like grain size, surface homogeneity, continuity, microtopography and smoothness seem to be determinant on the appearance and development of the wear features.

In this sense, for example brightness, which is one of the main criteria when describing microwear, seems to be strongly dependant on crystal size and arrangement. In rocks like chert, the more worn the surface, the brighter it is because the attritional process smooths it. But in rock crystal the original surface is extremely smooth (and so it naturally shines), and what wear always does is turning the surface rougher and duller. Neither on vein quartz nor on quartzite brightness can be used to properly identify polished areas (Figs. 10 and 13).

The ongoing experimental programmes consider a “checklist approach” to make easier in the future the comparisons on the wear features occurring on quartzose raw materials. Such an approach is commonly used in Earth sciences when dealing with microtextures of quartz grains (Torcal and Tello, 1992; Madhavaraju et al., 2009), and was effectively applied to usewear analysis by Knutsson (1988b: 65). Future steps on this field would require more specific results on each of the raw materials studied here, incorporating available systems of classification of the wear features (e.g. Adams et al., 2009) and already observed specific phenomena (e.g. Clemente-Conte and Gibaja, 2009; Márquez et al. in press).

Although postdepositional processes have not been considered in this article, it is worth noting that most of them imply mechanical phenomenon highly coincident with the stone tool use, and so, their effects on the stone surfaces tend to present morphological coincidences. So, a proper distinction of use-wear and postdepositional wear on archaeological artefacts would require a specific consideration of the distribution of the traces and, sometimes, a high magnification to properly identify features as the v-shaped impact pits (Fig. 14).

In terms of microscopes, we strongly support the complementary character of different techniques (e.g. Borel et al., 2014; Knutsson et al., 2015; Marreiros et al., 2015) (Fig. 13). The reflected light microscope is appropriate for analysing the varieties with large crystals, as rock crystal and vein quartz, but they revealed more limited when dealing with quartzite (Grace, 1990). Of course, results improve clearly when using differential interference contrast (DIC) and extended focus applications. SEM, on his part, adds a higher depth of field, the possibility of a higher magnification, and a general better quality view of the textural features thanks to the removal of the glare derived from the rock's optical properties. These advantages are very useful to describe features as the striations. Finally, we occasionally used the LSCM, and it proved to be highly efficient to image the details on crystal modification at high magnification. Its potential in terms of wear quantification has been proved for flint (Evans and Donahue, 2008),

Fig. 12. Sequential experiment on quartzite showing wear development during a sawing action on green wood. Images of the fresh edge (A, cast), and after 10, 20 and 30 min of use. Note the initial loss of a portion of the edge, the progressive formation of parallel striations, and their obliteration at the third use-stage, in which a domed polish can be seen. Micrographs taken with a high vacuum SEM secondary electron detector, orig. mag.: 1000×, scale bar: 10 μm.

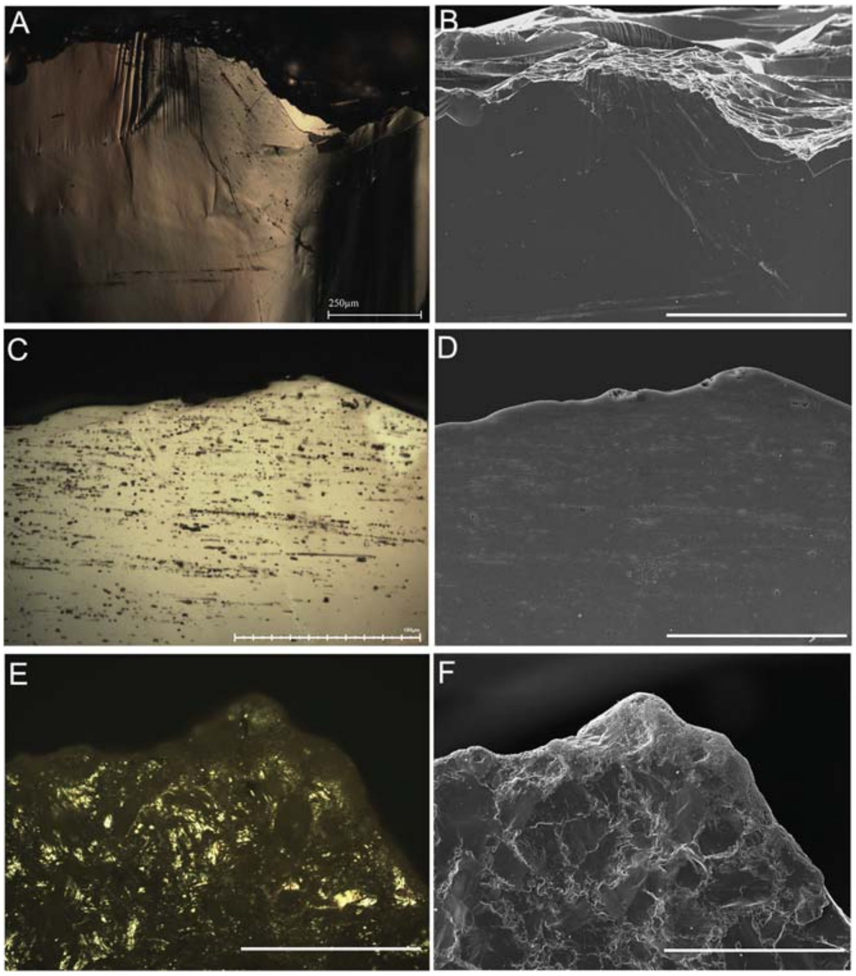


Fig. 13. Comparison of use-wear features on quartzose materials. The same points the lithic edges are imaged by means of two complementary microscopic techniques, OLM (A, C, E) and SEM secondary electron detector (B, D, F). A–B) Quartz, orig. mag. 100×; scale bars: 250 and 500 µm respectively, archaeological traces; C–D) rock crystal, orig. mag.: 500×, scale bar: 100 µm, wood sawing; E–F) quartzite, orig. mag.: 100×, scale bar: 500 µm hide scraping.

and only few data is available for the rocks we are dealing with (Stemp et al., 2013).

5. Final remarks

The results presented here aimed to contribute new data and observations to the up to date still scarce of reliable published works to be used as referential wear patterns on quartz-like materials.

After appropriate methodological procedures, use-wear analysis can effectively tackle functional as well as technical issues from quartz materials. In fact, we think that methodology should be adapted to each type of rock, possibly varying the combinations of microscopic techniques, magnifications, cleaning processes, etc. In fact, we noticed that for some lithologies optical light microscopy was enough to observe use-wear, but for others it was far to provide satisfactory results. And sequential experiments allowed making progress in the comprehension of the wear formation processes on a group of materials mineralogically similar (macrocrystaline quartz), but quite different in structure.

The use of a very wide range of magnifications is extremely efficient, as use-wear is made up of a variety of features (microflaking, edge rounding, striations, polishes, etc.), each of which has specific requirements in terms of magnification and image resolution in order to be properly documented. So, different magnifications can give rise to very accurate descriptions of the diagnostic traits of wear features.

The trials with the LSCM proved to be really encouraging, not only by the clear potential of the technique in terms of quantification, but also in terms of imaging. The above mentioned "flatting" effect of the SEM is eliminated, keeping a very good image resolution up to c. 2000×, which allows, for example, properly interpret

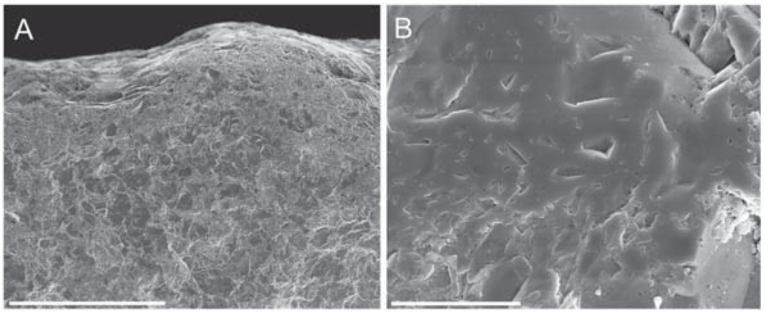


Fig. 14. Example of a naturally abraded edge on a quartzite fragment. At low magnification (A) a wear pattern (edge rounding and rough surface) very similar to some use experiments (e.g. hide working) can be observed. But at a higher magnification some diagnostic features as v-shaped impact pits can be documented, which allows us to identify this piece as naturally abraded instead of a used one. A) orig. mag.: 50×, scale bar: 1 mm; B) orig. mag.: 2000×, scale bar: 20 μm.

as a progressive abrasion of a large crystal what under the optical microscope would seem to be just an area of smaller crystals.

The essentially brittle behaviour of quartz restricts both the appearance and the preservation of wear features (striations, plastic deformations …). For that reason, isolated wear features, which in other materials perhaps would not be considered when analysing archaeological materials, must in this case be taken into account.

The diagnostic character of the described features depends on what we are searching for: the identification of the used edges (high), the tool's kinematics (medium), and the worked material (medium to low).

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3.4 Use-wear studies on quartzite

It is generally agreed that out of all the crystalline lithic materials, quartz is the most problematical because it does not seem to be susceptible to polishing, smoothing, or striating under most conditions (Hayden and Kamminga, 1979:8). In fact, no striations were normally observed in previous studies. The main features documented were only worn edges and general surface abrasion (Hayden, 1979:299). Quartz was not understood as a coherent material in use-wear analysis until the first solid investigations entirely focusing on this topic filled in this gap (*e.g.* Knutsson, 1988a, 1988b; Sussman, 1988).

Following mainly the methodology created for flint and with a few adaptations, researchers analysed quartzite assemblages (Plisson 1986; Pereira 1993, 1996; Alonso and Mansur, 1990; Carbonell et al., 1999b, 1999c; Igreja et al. 2007; Hroníková et al. 2008; Igreja, 2008; Aubry and Igreja 2009; Cristiani et al., 2009; Lemorini et al. 2014). In few cases, detailed studies have been also carried out, considering the specificities of this rock (Beyries 1982; Gibaja et al. 2002; Leipus and Mansur 2007; Clemente-Conte and Gibaja-Bao 2009).

The high-power approach was thought to be insufficient to provide reliable use-wear results on highly reflective materials like quartzite (Grace 1989, 1990). Even if the employment of the DIC (Differential Interference Contrast) can be useful to avoid light reflection of reflective materials (Igreja 2008, 2009; Knutsson et al. 2015), it does not always provide satisfactory results (Pedergnana and Ollé, 2017a).

3.5 Functional studies on Lower and early Middle Palaeolithic assemblages

Although one of the founders of use-wear analysis focused his research on English Lower Palaeolithic assemblages (Keeley, 1980), it is not usual to apply this method to ancient assemblages. This is due basically to two reasons. Firstly, the general poor preservation of the surfaces of lithics in older assemblages that are often recovered from lacustrine or fluvial environments contexts where the incidence of post-depositional processes may be very high. Secondly, excluding some exceptions (France, England), these assemblages are almost entirely composed of coarse-grained rocks (*e.g.* quartz, quartzite, basalt). As use-wear analysists have mostly focused their efforts on the development of a solid method through the analysis of fine-grained lithologies (*e.g.* chert), the minor interest for ancient assemblages is easily explained.

However, parallel to the most known and comparable studies of the 1970's and 1980's, some sporadic works considered even extremely ancient chronologies, such as the Oldowan sample from the Koobi Fora region (Kenya) analysed by Keeley and Toth (1981). Further studies followed these first trials on ancient materials (Longo, 1994; Peretto et al., 1998; Vergès, 1996, 2003; Sahnouni et al., 2013). Although minor research focused on the initial phases of the Lower Palaeolithic, most of the studies have focused on significantly more recent lithic industries, ascribed to the Middle Pleistocene. Flint handaxes from the Lower Palaeolithic site of Boxgrove (*ca.* 500 ka) in England were microscopically analysed by Mitchell (1998), who concluded that they were usually used for short periods of time and then abandoned without re-sharpening.

Donahue and Evans (2012) attempted to analyse the Lower Palaeolithic assemblage of Linford Quarry in England. The analysis did not provide significant functional results due to the poor preservation of the surfaces. Out of 109 samples, only two display evidence of use-wear. Post-depositional modifications were very abundant and widespread. Several kinds of post-depositional alterations were described (*e.g.* mild polishing, metal traces, natural severe wear, bright spots) and categorised into different degrees of development.

Other Middle Pleistocene sites whose collections were submitted to use-wear analysis, like Coudoulous in France, dating to *ca.* 300 ka (Jaubert et al., 2005; Venditti 2014). Quartz is the most abundant material in this assemblage and its high reflective index has rendered use wear analyses quite difficult.

Use-wear analysis applied to small lithic samples recovered in association with large animals have provided interesting insights into the ways that small flakes were used for butchery during the early Middle Pleistocene (Lemorini, 2001; Aureli et al., 2015; Mosquera et al., 2016). The sample analysed from the Ficoncella site (Italy) is particularly interesting for its limestone implements, given that this type of material has not been well-studied from a functional point of view until very recently (Aureli et al., 2015:16). In fact, the first systematic study of limestone, comprising an extensive experimental reference collection, focused on the Lower Palaeolithic site (MIS9) of Bolomor in Spain (Hortelano-Piqueras, 2016).

Among the studies available on Lower Palaeolithic material, work on the Sierra de Atapuerca's sites stand out. For example, studies carried out on the Galería site have underlined the presence of wood-working and butchering activities despite the occasional bad preservation of some lithologies (mainly chert), (Ollé, 1996, 2003; Sala, 1997; Márquez, 1998; Carbonell et al., 1999b, 1999c). Also, the same activities are described for samples analysed from the Gran Dolina site (TD6, TD10 units) (Márquez et al., 2001).

A recent work combining use-wear and residue analyses concerned two different locations of the Middle Pleistocene site of Shöningen in Germany, underlining the presence of mainly woodworking and butchering activities, with some uncertainties about evidence for hafting on two samples (Rots et al., 2015).

More frequently, use-wear analysis has been successfully applied to assemblages dating from MIS9 onwards (*e.g.* Martínez et al., 2003; Lazuén et al., 2011; Lazuén, 2012; Clemente et al., 2014). The most abundantly documented activities are generally woodworking and butchery. Middle Palaeolithic sites are thought to be characterised by less weathered assemblages, compared to those with more ancient chronologies, such as the Acheulean and Oldowan ones.

The site of Biache-St-Vaast (MIS7) in France is an example of a successful functional study providing very interesting insights. Activities linked to butchery and woodworking activities were recorded, and, more recently, the practises of hafting and throwing spears have also been documented (*e.g.* Beyries, 1988; Claud et al., 2013; Rots, 2013, 2015).

A preliminary study of the collection of Maastricht-Belvédère (MIS7) has also recently been performed, allowing to identify at least one spear tip (Rots, 2015).

Functional data is also available for the Payre site (MIS7-8; MIS5-6), for which use-wear as well as residue data has been collected (Moncel et al., 2009; Hardy and Moncel, 2011). Use-wear traces on a sample of convergent flint tools revealed that they were used by applying longitudinal and transversal actions. However, it is unclear what materials were worked due to the poor development of the wear. Residues play also an important role in defining site function. Specifically, the identification of animal and vegetal fragments have contributed to the reconstruction of the Neanderthal diet (Hardy and Moncel, 2001).

Within the same chronological framework, studies of the artefacts from San Quirce in Spain (Palencia) (MIS5) revealed that wood and vegetal fibres were commonly worked materials at this site (Clemente et al., 2014).

Bifaces have also been an important object of investigation in recent years. Non-exhaustive experimental programmes focusing on the use of bifaces are available (*e.g.* Jones, 1980; Mitchell, 1995; Ollé, 2003; Claud et al., 2009; Viallet, 2016). A low power approach is generally preferred for biface analysis, since the main parameter considered is that of macro-scars. Thus, their morphology and distribution of scars are documented (Claud, 2009; Viallet, 2016). Functional data concerning bifaces is still relatively rare and results usually relate them to woodworking and butchery activities (Keeley, 1980; Binneman and Beaumont, 1992; Ollé, 2003; Soressi and Hays, 2003; García-Medrano et al., 2014). Percussive activities have also been documented (Moncel, 1995; Mitchell, 1998; Domínguez-Rodrigo et al., 2001; Rots and Van Peer, 2006; Claud et al., 2013).

Outside of Europe, recent studies have explored the possibility of analysing Oldowan quartz and quartzite assemblages (Lemorini et al., 2014). In the Levant, sites with early chronologies have also yielded interesting functional data (Lemorini et al., 2015). Evidence of residues has also been rarely documented on ancient material. One of the most ancient residues recorded so far comes from the Acheulean assemblage of Peninj, analysed by Domínguez-Rodriguez et al. (2001), where remnants of phytoliths were attributed to woodworking activities.

However, functional studies of very old assemblages are still quite rare, mainly due to problems relating to the preservation of the artefacts. In fact, we should not underestimate the fact that post-depositional alterations, especially when presenting high degrees of development, are capable of obliterating wear related to use by compromising its original appearance as well as its spatial patterning. Residues have hardly been preserved on the surfaces of stone tools with ancient chronologies, although they may be found in extraordinary circumstances.

3.6 Residue analysis

Worked materials adhered onto stone tool surfaces constitute a very important clue regarding tool function. In archaeological contexts, their detection appears to be very challenging due to poor preservation conditions, weathering and/or post-excavation treatment of the artefacts (Langejans, 2010).

Early on, important work on residue analysis was undertaken by Briuer (1976), Broderick (1979), and Shafer and Holloway (1979), but it was Loy's paper on blood residue identification (1983) that was to attract more attention to the potential of residue analysis.

Animal remains, such as hairs (Shafer and Holloway 1979; Loy and Hardy 1992; Hardy et al., 2013), blood cells, collagen fibres (Loy and Dixon 1998; Lombard, 2008) and feathers (Hardy et al., 2001, 2013; Hardy and Moncel, 2011), have all been recognised on stone tools. Some researchers have also identified plant fibres and cells, starch grains and other remains such as, phytoliths trapped onto stone tool surfaces (*e.g.* Shafer and Holloway 1979; Hardy and Garufi, 1998; Domínguez-Rodrigo et al., 2001; Fullagar, 2006; Lombard 2008).

Apart from the obvious issues connected with the poor preservation conditions of many assemblages and the regular decay of organic materials (Eerkens, 2007; Langejans, 2010), difficulties of simple recognition of the residue types have been highlighted by all the blind tests set up to test this methodology (Wadley et al., 2004; Lombard and Wadley, 2007; Rots et al., 2016). The most bewildering aspect is that a high level of uncertainty exists even at an experimental level (hence, even when the variables are controlled and there are no taphonomic alterations).

Furthermore, it is known that residues can be the result of either direct or indirect activities. In fact, they can be related to the direct action performed (residues of the material worked with the stone tools), or they can be connected with the tool's life and give some clues about more complex *chaînes opératoires*. For instance, a number of organic and inorganic residues related to the hafting practice (Rots, 2010) might be detected (glues, ochre or binding materials) (*e.g.* Boëda et al., 2007; Cârciumaru et al., 2012; Charrié-Duhaut et., 2013; Hauck et al., 2013; Helwig et al., 2014).

Different methods have been formulated to analyse ancient residues on archaeological tools (ceramics, lithic material being both grinding tools and knapped objects, and others). To simplify, two distinct, though complementary, approaches can be identified within the domain of residue analysis:

- Direct observation of residues by means of reflective optical devices (both stereomicroscopes and metallographic microscopes) (*e.g.* Hardy et al., 2001, 2013; Lombard, 2005, 2008; Lombard and Wadley, 2007; Langejans, 2010, 2011; Cristiani et al., 2014, 2016);

- Indirect observation of residues through transmitted light microscopes (Fullagar, 2006; Haslam et al. eds., 2009; Fullagar et al., 2015). This kind of observation is only possible after the residues have been extracted from the surfaces of the artefacts and subsequently prepared in the laboratory. Chemical reaction agents are likely to be used when samples are prepared (staining procedures) (Stephenson, 2015). These chemicals serve to identify specific proteins (*e.g.* haemoglobin, lignin, collagen) (Loy and Dixon, 1998; Barnard et al., 2007; Högberg, et al., 2009; Yohe II et al., 2013; Fullagar et al., 2015; Rots et al., 2016).

SEM analysis has proved to be very useful in residue characterisation, providing best image resolution and elemental analysis by means of the EDX system (Anderson 1980a; Jahren et al., 1997; Pawlik, 2004; Hortolà, 2005; Pawlik and Thissen, 2011; Monnier 2012; Borel et al., 2014; Xhauflair , 2014; Xhauflair et al., 2016).

In any case, despite numerous studies on residue identification, no detailed catalogue (photographs or chemical characterisation) is available yet and the potential of SEM for recognising different types of residues has not been entirely exploited up to now.

3.7 Experimentation in functional studies

Experimentation is an unavoidable tool in functional studies. As a branch of 'Experimental Archaeology' (Coles, 1979), it became very important to the domain of lithic microwear research from the onset (Semenov, 1964; 1970). In this kind of studies, the main objective of the manufacture of stone tools is not their mere replication of a morphotype, but rather to use replication to investigate the past function of the tools (Coles, 1979). Therefore, experiments involving the use of lithic tools are set up and often have a significant influence on the final results. Ultimately, experimental data is compared with the archaeological evidence. The selected variables are of paramount importance when the resulting use-wear is to be analysed and interpreted.

General rules should be taken into account when starting to construct a microwear reference collection. First of all, all microwear analysts should construct their own reference collection to be aware of the precise actions carried out with the stone tools. The application of the scientific method is expected to be suitable for later comparison with the archaeological material.

Some basic criteria to be considered are:

- Variables must be controlled throughout the experiments;
- All the events need to be entirely observable;
- Experiments need to be measurable and replicable by others;
- Experimental conditions need to be described in detail;
- Subjectivity should be limited as much as possible.

Following these fundamental steps, different types of experiments can be designed. 'Emic' experiments aim at gaining total control of subjectivity, taking the role of experimenter from people and giving it to a machine (Knutsson, 1988a:11-12). Robotic equipment is used in this kind of experiments to maximise the objectivity and the measurability of the results (Iovita et al., 2014). On the other hand, 'ethic' experiments are performed at exterior locations, re-creating the original conditions where prehistoric activities took place (on the ground, etc...) (Knutsson, 1988a:11-12). The same concept is expressed by the term 'replicative experiments', wherein that the main aim is to reproduce activities which are hypothetically analogous to those that occurred in the past (*e.g.* Semenov, 1964; Keeley, 1980).

The 'analytic experiments' (*e.g.* Tringham et al., 1974; Vaughan, 1985; González and Ibáñez, 1994; Gutiérrez, 1996) are closer to what is discussed above. They consider dependent and independent variables and analyse their inter-relationships.

The concept of 'sequential experiments' is a natural development of analytic experiments (Ollé and Vergès, 2014) that are also based on the control of the dependent and independent variables and respond to the need of monitoring a repetitive sequence of events. This kind of experimental approach has been specifically designed to monitor the formation of use-wear on stone tools, by observing the same portions of the surface after several periods of use-time.

Besides the experimental data that one can produce in laboratory conditions, ethnographic accounts might also add interesting information about the tool use of in general, helping the analyst to better design his/her experimental activity (Rots and Williamson, 2004; Xauflair, 2014). In the field, personal observations buttress the large body of available published works, contributing to enriching research in the countless ways that stone tools are produced and used by indigenous people (*e.g.* Stout, 2002; Shott and Sillitoe, 2005; Diamond, 2012). These 'traditions' are obviously different from those employed by the researchers in retrospect. First of all, researchers do not produce and use the lithic artefacts for their own subsistence requirements. Secondly, they do not follow any particular cultural trends. In other words, they have not been culturally formed to perform the tasks at hand. They do not have a preference for particular sets of gestures nor do they follow any specific sequences of actions. All of these factors could have tremendous impact on the experimental results. The experimenters' gestures are not inserted in any social context and therefore, they lack significance from a strict anthropological point of view (Mauss, 2007). All of these reflections serve as a reminder about the complexity of the applications of experimental data in functional studies. In fact, we need to bear in mind that the reproduction of prehistoric activities is undertaken under laboratory conditions and therefore, it is extremely difficult to make any direct analogies with the archaeological collections. That is to say, comparisons with the archaeological evidence are expected to provide functional data, and they certainly accomplish this objective. The fact is that the researcher's interpretation of the data plays a major role in defining stone tool functionality. Hence, the subjectivity of the interpretations needs always to be taken into account, as a full objectivity cannot be guaranteed. This is maybe the biggest limit of traceology, which can hardly be overcome. It is a paradox, intrinsic to the methodology selected. Therefore, we should be reminded that the results discussed in the domain of traceology always derive from our own experience as experimenters and probably the capacity of providing precise interpretations of the archaeological evidence is directly proportional to our own experimental baggage.

3.8 Methodological problems in functional studies

The analysis of microwear on stone tools can be very challenging on many levels. As seen in the previous paragraph, experiments lacking solid foundations are likely useless for comparison for a number of reasons. First of all, if the variables are not kept constant, results are not trustworthy. As a consequence, all the archaeological studies carried out by comparing traces with improperly obtained experimental replicas are not conclusive. The same remark is made when the only consulted materials are the pictures found in the literature or when analogies with rock types other than that under analysis are made. Interpretations based only on limited photographic material cannot come close to the reality of the original function of the tools. When the rock type of the reference collection does not match that one of the analysed material and functional interpretations are proposed anyhow, this should be considered a methodological error and results cannot be reliable.

A second and less controllable issue regards traces which might compromise a correct functional reading of a tool. Post-depositional alteration of lithics is a factor that might reduce the effectiveness of functional identification of tools (Levi-Sala, 1986, 1996; Burroni et al., 2002; Donahue and Evans, 2012). This is due to the sometimes-equivalent aspect of some post-depositional traits with those resulting from use. For instance, regular scars on the edges can be observed after the utilisation of the tools, the retouching of them or when tools are subjected to trampling phenomena.

A third aspect which might raise confusion regards the presence of non-use related micro-residues. It is known that not all the residues found on archaeological samples are necessary connected with the material which had been worked at the site. In fact, they can be the result of various phenomena, such as bioturbation, contamination from the soil enrichments, modern contamination. This topic will be addressed in more detail in section 4.6 of this thesis.

Moreover, the microscope type used in the analysis can strongly affect the quality of the results. We can expect that some micro-traces are detectable by some microscopes and invisible when using different equipment. For instance, scars are better imaged by a stereo-microscope, barely visible with a metallographic microscope and invisible with a SEM (at least on quartzite)[1]. Micro-scars on quartzite, located on the very rim as well as scars with step-terminations are visible also with a SEM. Conversely, thin and short striations (few microns length) are only visible with SEM and do not appear under optical microscopes. Hence, depending on the selected way of analysing the material, some traces may or may be not observed and therefore, recognisable.

Considering all of the above exposed points, it is important to try to overcome methodological problems in traceology, as well as to be constantly aware of the intrinsic limitations of the methods available to us.

3.8.1 Post-depositional processes

Special attention is devoted to the difficulties originating from post-depositional surface modifications (PDSM) on tool surfaces. Soil sheen, patination, weathering and trampling are a major challenge for any microwear analyst. Their effects can either cover up use-related wear (as in the case of patination) (Levi-Sala, 1986), or they can be mistaken for use-wear (*e.g.* trampling). Soil movements and trampling can produce regular macro-scars on stone tools' edges, very similar to human-induced ones (retouch). The presence of water during soil displacements (*e.g.* lacustrine and fluvial environments) can accelerate the processes of wear formation (abrasive erosion).

The differentiation between human-induced wear and post-depositional wear is very challenging; morphological similarities are huge, as their formation processes are very similar (Levi-Sala, 1986, 1996; Burroni et al., 2002; Donahue and Evans, 2012; Pedergnana and Rosina, 2015). In fact, wear basically originates from the mechanical friction between two solid bodies, namely, the stone tool and the lithic fragments embedded in the worked materials' residues. In the case of post-depositional soil movements, the second solid body is composed of the sand particles of the soil, plus fragments of any rock found in the sediment which may contribute to the friction.

The only way to limit the uncertainty originating from the presence of PDSM is to carefully consider the distribution of wear on both the edges and the internal surfaces of the tools. Generally, use-wear presents logical distributional patterns, whereas PDSM are chaotically distributed. When PDSM are superimposed on the wear originating from use, it is indeed impossible to recognise any use-wear. Nevertheless, when the degree of PDSM is relatively low, it is possible to identify at least the used portions of the edges. This is why we created a section to record the presence/absence of post-depositional surface modifications and the relative degree of development in the data sheet used to localise the use-wear evidence on the archaeological material (Table 4.5, Chapter 4). In this way, one can also assess the degree of reliability for each interpretation proposed.

[1] Micro-scars on smooth raw materials (flint, rock crystal) are visible at SEM-low magnifications (30-50x).

3.8.2 Similarities between intentionally retouched edges and macro-scars due to use

Morphological analogies between negative scars caused by the retouching of edges and the scars originating from use have been described since the first investigations on use-wear began (Tringham et al., 1974). The regular pattern of scar distribution observed on edges used to perform different tasks strongly resembled the appearance of slightly retouched edges. This is why, perhaps, the expression “retouched due to use”, frequently used by lithic technologists, was coined.

Analogies are not restricted to retouch. In fact, the process of detachment of any scar from the edge of a flake responds to clear physical laws (Cotterell and Kamminga, 1979). Therefore, the pressure exerted to the rim of an edge by different means may produce very analogous results. In fact, the enduring contact between a stone tool and a haft or the hand-held prehension with the interposition of other materials (sand, leather wraps, etc.) between the hand and the stone tool may create non-intentional tiny regular scars all along the hafted edge or the prehensile part of a hand-held tool. Prehensile wear and evidence of hafting have been thoroughly described and categorised (Rots, 2010). Despite the similarities between scar morphology and size, the regular distribution of such features allows the identification of the areas coincident with the presence of the haft (Rots, 2010; Rots et al., 2001).

Conversely, scars due to trampling or PDSM in general, usually show non-patterned features, and present distinct size patterns. They are also discontinuous and may be located on multiple edges and surfaces (dorsal, ventral). When PDSM are abundant, even the internal surfaces are affected. Moreover, damage caused by mechanical friction between artefacts carried together in the same bag also produces scars similar to those originating from trampling (Rots, 2010: 44). This kind of evidence is denominated ‘bag wear’ (*e.g.* Odell and Odell-Vereecken, 1980; Kamminga, 1982).

Due to these extreme analogies of scars derived from very different phenomena, functional interpretations considering macro-traces should be always accompanied by other sources of data. General caution is necessary, as the identification of scars on tool edges is not a certain prove of their past utilisation.

PART II: MATERIALS AND METHODS

Chapter 4: Methodology

4.1 Experiments

A wide-ranging experimental programme was set up in order to provide a thorough comparative use-wear reference repository for quartzite. Data was collected over a period of approximately three years. A total of 42 un-retouched blanks and 4 retouched flakes were included in the experiments. Each tool was catalogued by the attribution of both a basic and an experimental reference code (Table 4.1). Therefore, 46 experiments were elaborated, but a combined average of 81 experimental sessions were in fact performed. Since the experiments were sequential, each tool was used for different stages (1, 2, 3 or 4) and then observed microscopically. This strategy was carried out so as to systematically monitor any microscopic edge transformations throughout the different phases of use. The performance of '*sequential observations*' of lithic surfaces on individually selected points was introduced to the domain of traceology by Yamada (1993), and proved to be very useful for monitoring the formation of use-wear, giving important clues about how it can develop during different sequences of utilisation. This procedure was then extended to other studies and incorporated in the standard experimental methodology aimed at the creation of use-wear reference collections (Ollé, 2003; Vergès, 2003; Ollé and Vergès, 2008, 2014). When tools are systematically observed prior to their use and then throughout several stages of use, the concept of '*sequential experiment'* is employed.

All of our experimental activities were carefully recorded by filling-in experimental forms to describe each sequence. Videotapes and photographic material was also generated for most of the activities. Data collected during the experiments was subsequently transferred to digital catalogues, comprising all of the various stages. Each experiment is represented by a separate form, thus making it easily available for consultation and providing additional information for this thesis in digital format (Annex 3). The template of the experimental form (Table 4.2) shows how the reference codes used for both the experiment and the experimental tool are recorded. Basic information, such as the date of each experiment and the name of the experimenter are indicated at the top of the form. Generic technological information of the tool is then provided, followed by the measurements of each tool. Length is measured as the greatest distance parallel to a line perpendicular to the long axis of the tool, while width is measured as the greatest linear distance perpendicular to the long axis (the same criteria are used to measure archaeological tools). A photo of each tool and a general view of the experiment are also uploaded onto this document.

Moreover, detailed information regarding each experiment is provided: the prehensive mode used to wield the tool, the elapsed time of use, the number of stages or experimental sessions, the contact angle between the used edge and the worked material, etc. The most used part of the edge is generally indicated by a circle directly on the photograph of the tool.

Details about the type of experimental activity and those pertaining to aspects such as the gesture (longitudinal *vs* transverse, unidirectional *vs* bidirectional) and variables referring to the worked material (type, scientific species denomination, state, and hardness), are all provided in the third line of the experimental form. Next, the period of time associated with each stage is specified and the approximate number of strokes per use-unit is calculated. The latter is obtained based on stroke counts from full experimental sessions observed in slow-motion on videotapes. For experiments which were not entirely videotaped, approximations on this variable were obtained by multiplying the number of strokes counted for the recorded sequence by the elapsed time in each case. For those which were not videotaped at all, the same procedure was applied, but calculations were done based on analogous videotapes. In such cases, the number of strokes per minute was extrapolated from videotapes of similar experiments (same activity and experimenter) and then multiplied by the elapsed time of the experiments.
Continuing on the form descriptive, we provide references for any casts of the lithics that may have been realised. Finally, we specify the type of microscopic analysis applied (OLM, high or low-vacuum-SEM,). Generally, the experiments involving entire animal carcasses were performed in outdoor situations. In the case of the butchering of adult red deer, the outdoor location was the National Hunting Reserve of Boumort, in Northern Catalonia (la Pobla de Segur, Lleida), on the border with France (Fig. 4.1). Most of the hide scraping activities were also performed in this reserve, mainly because we were able to reproduce environmental conditions similar to those of the Prehistoric groups performing the same task.

	EXPERIMENT REFERENCE	EXPERIMENTAL TOOL	MOVEMENT	N. STAGES
1	ANTLER-01	qtfu1-02	longitudinal	3
2	ANTLER-02	qtfu2-02	longitudinal	3
3	ANTLER-03	qtfu1-14	longitudinal	1
4	ANTLER-04	qtfp1-07	transverse	1
5	BONE-01	qtfu1-05	longitudinal	3
6	BONE-02	qtfu2-05	longitudinal	3
7	BONE-03	qtfu1-06	transverse	3
8	BONE-04	qtfu2-06	transverse	3
9	BONE-05	qtfp1-02	longitudinal	1
10	BONE-06	qtfp1-03	transverse	1
11	BONE-07	pay-06	transverse	1
12	BONE-08	qtfu1-17	transverse	1
13	BUTCHERING-01	qtfu1-03	longitudinal	3
14	BUTCHERING-02	qtfu2-03	longitudinal	2
15	BUTCHERING-03	qtfp1-02	longitudinal	2
16	BUTCHERING-04	qtfu1-12	longitudinal	1
17	BUTCHERING-05	qtfu1-15	longitudinal	1
18	CANE-01	qtfu1-10	longitudinal	2
19	CANE-02	qtfu2-10	longitudinal	2
20	HIDE-01	qtfu1-04	transverse	3
21	HIDE-02	qtfu2-04	transverse	2
22	HIDE-03	qtfu1-07	transverse	3
23	HIDE-04	qtfu2-07	transverse	2
24	HIDE-05	qtfu1-09	longitudinal	2
25	HIDE-06	qtfu2-09	longitudinal	3
26	HIDE-07	qtfu1-11	longitudinal	1
27	HIDE-08	qtfu2-11	longitudinal	1
28	HIDE-09	qtfp1-04	transverse	2
29	HIDE-10	pay1-05	transverse	1
30	HIDE-11	qtfu1-16	transverse	1
31	WOOD-01	qtfu1-01	longitudinal	3
32	WOOD-02	qtfu2-01	longitudinal	4
33	WOOD-03	qtfu1-08	transverse	2
34	WOOD-04	qtfu2-08	transverse	2
35	WOOD-05	qtfp1-05	transverse	1
36	WOOD-06	qtfp1-06	longitudinal	1
37	WOOD-07	qtfu1-13	long./trans.	1
38	WOOD-08	pay-01	longitudinal	1
39	WOOD-09	pay-02	longitudinal	1
40	WOOD-10	pay-03	transverse	1
41	WOOD-11	pay-04	transverse	1
42	WOOD-12	qtfu1-18	transverse	1
43	TUMBL-01	TUMBL1-qtfu1	-	1
44	TUMBL-02	TUMBL2-qtfu1	-	1
45	TUMBL-03	TUMBL3-qtfu2	-	1
46	TUMBL-04	TUMBL4-qtftp1	-	1
TOT=46	-	-	-	tot. stages: **81**

Table 4.1: *Total number of experiments performed, experimental reference and associated tool, type of movement and number of stages for each sequential experiment.*

<table>
<tr><td>Experimentation code:</td><td>Experimentation type:

N. intervals:</td><td>Dates:
Stage 1:
Stage 2:
Stage 2:</td><td>Experimenter:</td></tr>
<tr><td colspan="2">Lithic artefact reference nº:</td><td colspan="2">Technological category:
Knapping technique:
Raw material:
Granulometry:
Cortex: Position of cortex:
Dimensions mm:
Butt:
Used edge: Retouched:
Angle of the used edge: Length mm:
Horizontal delineation:
Sagittal delineation:
Prehension:
Hand used:
Working angle: Surface referenced:
Most functional part: Length mm:</td></tr>
<tr><td colspan="2">Activity:
Action:
Movement 1:
Movement 2:
Macro use-wear:</td><td colspan="2">Worked material type:
Worked material state:
Scientific species:
Hardness:
Observations:</td></tr>
<tr><td colspan="2">Experiment intervals:
Total time:
Approximate number of strokes:</td><td colspan="2">Photos:
Videos:</td></tr>
<tr><td colspan="2">Reference codes of moulds/casts:</td><td colspan="2">Applied analyses:</td></tr>
<tr><td colspan="4">Observations:</td></tr>
</table>

Table 4.2: *Blank experimental form. A total number of 46 forms, one for each experiment, were filled in and are available in Volume II in digital form only (Annex 3).*

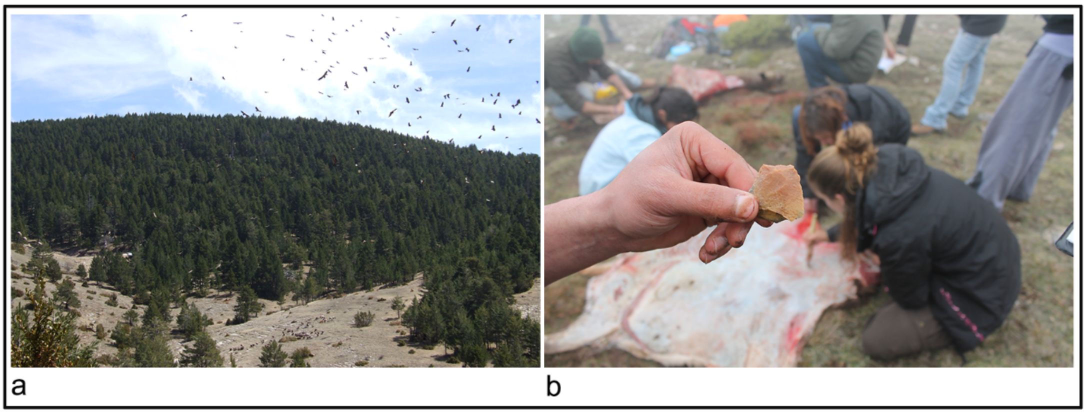

Fig. 4.1: *Boumort Natural Hunting Reserve in Northern Catalonia. a) General view of the location where the outdoor butchering experiments were carried out. Different species of vultures are present in this reserve, and they are usually attracted to the location of the experiments; b) One of the quartzite tools composing our reference collection (qtfu1-04, second stage, fresh hide scraping). In the background, students performing hide scraping experiments on the ground.*

The worked materials comprised in the experimental programme were: animal carcasses, (Fig. 4.2: a, b), antler (Fig. 4.2: c), bone (Fig. 4.2: d), fresh and dry skins (Fig. 4.2: e, f), cane (Fig. 4.2: g) and wood (Fig. 4.2: h). Although there were some exceptions, the state of the worked materials was generally fresh; a variable which might deeply influence the development of wear on the lithics. The water content contained in the worked materials is also thought to be an important aspect affecting the extent to which wear develops on lithic surfaces (among others, Levi-Sala, 1986; Xhaufflair et al., 2016).

Bones were always worked soon after the butchering process, which means that the periosteum, as well as some remnants of meat, were still present. Antler was always worked in a dry state (no previous soaking)[2], while both fresh and dry skins were scraped. While the term 'hide scraping' is universally employed to refer to the fleshing of any kind of animal's outside dermal layer, a differentiation is generally made between hide and skin depending on the size of the animal. In fact, the term 'skin' is applied to refer to small-sized animals (*e.g.* goats, deer, rabbits), whereas 'hide' is only employed for larger-sized animals (*e.g.* cattle, horses, bison) (Wiederhold, 2004). Only one hard-wood and one reed species were used in the experiments (*Quercus ilex* and *Arundo donax*)[3].

[2] The only experiments where antler was soaked are those included in Annex 1 (Laser Scanner Confocal Microscope).

[3] For the two experiments with wood of the same study (Laser Scanner Confocal Microscope-Annex 1), a soft-wood species was used (*Pinus halaepensis*).

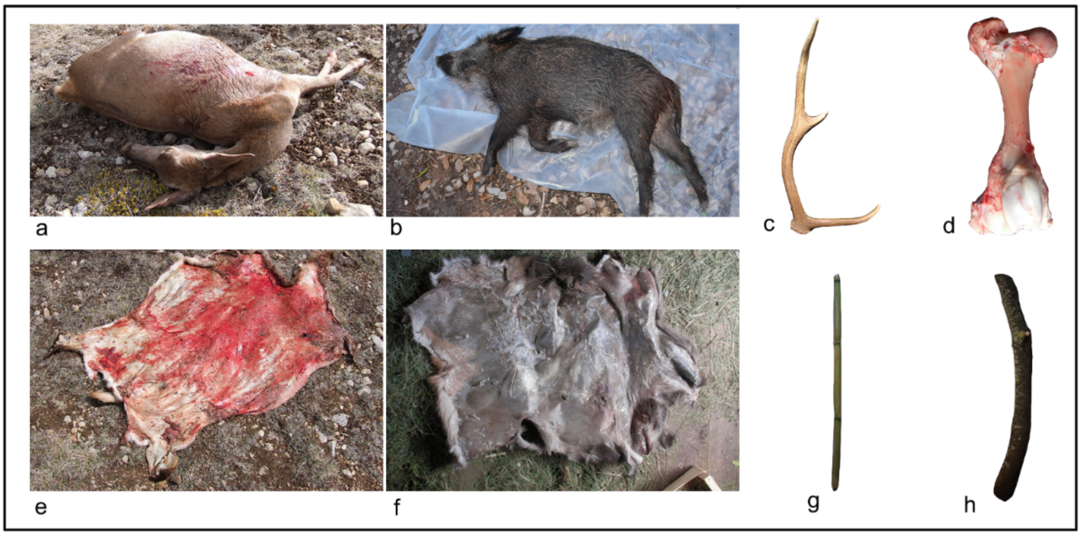

Fig. 4.2: *The materials included in the experimental programme. a-b) Deer (Cervus elaphus) and wild boar (Sus scrofa) carcasses; c) Cervid shed antler; d) Bovid long bone; e) Fresh deer skin; f) Dry deer skin; g) Giant cane stem (Arundo donax); h) Wood branch (Quercus ilex).*

Both deer (*Cervus elaphus*) and wild boar (*Sus scrofa*) were butchered with quartzite tools. The entire process was carried out with the aid of two or more tools. Entire animal carcasses were processed, starting with the removal of the skin (Fig. 4.3: a, b), the disarticulation of the limbs (Fig. 4.3: c, d), the removal of the flesh from both the axial and appendicular parts of the animal (Fig. 4.3: e, f).

All of the activities involved in the butchering process were generally performed using unidirectional and longitudinal movements (Fig. 4.4: a), with the rare occurrence of punctual bidirectional strokes, aimed at the breakage of resilient materials such as tendons or ligaments. Contact with bone, though not intentional, was always recorded. For the other worked materials, both longitudinal and transverse actions were performed (Table 4.3). Examples of these are: scraping fresh (Fig. 4.4: b) and dry (Fig. 4.4: c) skins, cutting fresh skin (Fig. 4.4: d), scraping (Fig. 4.4: e) and sawing (Fig. 4.4: f) wood, and sawing bone (Fig. 4.4: g). All of the tools were hand-held and none of the blanks were hafted into composite tools.

The differentiation between cutting and sawing was maintained (Keeley, 1980). Cutting thus refers to unidirectional, longitudinal movements on any kind of material and uni or bidirectional movements performed on soft materials (*e.g.* animal tissue, non-woody plants). Sawing is used only for bidirectional and longitudinal strokes on hard materials (*e.g.* wood, bone). Scraping and whittling are both unidirectional and transverse backward movements. What changes is the amplitude of the contact angle, from quite open (close to 90°) in the former case, to more acute, for the latter. In general, one bidirectional movement consisted of two strokes.

Fig. 4.3: *Butchering of an adult specimen of red deer (Cervus elaphus): a-b) Skinning; c-d) Dismembering; e-f) Removal of flesh from long bones.*

Fig. 4.4: *Detailed pictures of tool prehension and movement during various experimental activities: a) Unidirectional longitudinal movement during the skinning of an animal; b-c) Unidirectional scraping of fresh and dry skins respectively: d) Unidirectional cutting of fresh skin; e) Unidirectional scraping of fresh wood; f) Bidirectional movement (sawing) of a wooden branch; g) Sawing a cow long bone.*

	Ex. Reference	Edge angle	Contact material	Type of c. material	Action	Contact angle	n. stages	Total time min	Approx. n. strokes
1	**qtfu1-01**	40°	Fresh wood	*Quercus ilex*	Sawing	60°<α<90°	3	60	18 000
2	**qtfu1-02**	45°<α<60°	Shed antler	*Cervus elaphus*	Sawing	90°	3	60	24 000
3	**qtfu1-03**	40°	Skin, flesh, tendons, bone	*Cervus elaphus/ Sus scrofa*	Cutting (uni)- Skinning/ Dismembering	80°<α<40°	3	80	4 000
4	**qtfu1-04**	45°	Fresh hide	*Cervus elaphus/ Sus scrofa*	Scraping	60°<α<30°	3	45	6 000
5	**qtfu1-05**	50°	Fresh bone	*Cervus elaphus/ Bos taurus*	Sawing	80°<α<90°	3	60	18 000
6	**qtfu1-06**	25°<α<30°	Fresh bone	*Cervus elaphus/ Bos taurus*	Scraping	90°	3	60	7 200
7	**qtfu1-07**	60°	Dry skin	*Cervus elaphus*	Scraping	40°	3	45	6 000
8	**qtfu1-08**	30°	Fresh wood	*Quercus ilex*	Scraping	40°	2	30	3 000
9	**qtfu1-09**	45°	Dry skin	*Cervus elaphus*	Cutting (bi)	90°	2	30	6 000
10	**qtfu1-10**	35°	Giant cane	*Arundo donax*	Sawing	90°	2	45	13 500
11	**qtfu1-11**	35°	Fresh skin	*Cervus elaphus*	Cutting (uni)	90°<α<70°	1	10	1 200
12	**qtfu1-12**	60°	Flesh, tendons, bone	*Cervus elaphus*	Cutting (uni)- Defleshing	90°<α<80°	1	20	1 600
13	**qtfu1-13**	40°<α<30°	Wood	*Quercus ilex*	Multiple actions (sawing, scraping, whittling)	40°<α<90°	1	25	2 000
14	**qtfu1-14**	35°	Shed antler	*Cervus elaphus*	Cutting (uni)	90°	1	15	1000
15	**qtfu1-15**	45°	Skin, flesh	*Cervus elaphus*	Skinning (uni)	90°	-	32	-
16	**qtfu1-16**	50°	Fresh skin	*Cervus elaphus*	Scraping (uni)	60°	1	-	-
17	**qtfu1-17**	65°	Fresh bone	*Cervus elaphus*	Whittling (uni)	45°	1	15	-
18	**qtfu1-18**	65°	Fresh wood	-	Scraping (uni)	-	1	-	-
19	**qtfu2-01**	55°	Fresh wood	*Quercus ilex*	Sawing	90°	4	90	27 000
19	**qtfu2-01**	55°	Fresh wood	*Quercus ilex*	Sawing	90°	4	90	27 000
20	**qtfu2-02**	50°	Shed antler	*Cervus elaphus*	Sawing	80°<α<90°	3	60	24 000
21	**qtfu2-03**	35°	Flesh, tendons, bone	*Cervus elaphus*	Cutting (uni)- Defleshing	80°<α<90°	2	85	6 800
22	**qtfu2-04**	45°	Fresh hide	*Cervus elaphus*	Scraping	80°	2	30	4 000
23	**qtfu2-05**	45°	Fresh bone	*Cervus elaphus/ Bos taurus*	Sawing	80°<α<90°	3	60	18 000
24	**qtfu2-06**	40°	Fresh bone	*Cervus elaphus/ Bos taurus*	Scraping	45°	3	60	7 200
25	**qtfu2-07**	40°	Dry skin	*Cervus elaphus*	Scraping	40°	2	30	4 000
26	**qtfu2-08**	50°	Fresh wood	*Quercus ilex*	Scraping	45°	1	15	1 500
27	qtfu2-09	35°	Dry skin	*Cervus elaphus*	Cutting (bi)	90°	3	45	9 000
28	qtfu2-10	45°	Giant cane	*Arundo donax*	Sawing	90°	2	30	9 000
29	qtfu2-11	45°	Fresh skin	*Cervus elaphus*	Cutting (uni)	90°<α<70°	1	10	1 200
30	qtfp1-01	40°	Skin, flesh, tendons, bone	*Cervus elaphus/ Sus scrofa*	Cutting (uni)- Skinning/ Dismembering	50°<α<40°	2	60	3 000
31	qtfp1-02	55°	Fresh bone	*Cervus elaphus*	Sawing	90°	2	30	9 000
32	qtfp1- 03	75°	Fresh bone	*Cervus elaphus*	Scraping	60°	2	30	3 600
33	qtfp1-04	70°	Fresh skin	*Cervus elaphus/ Sus scrofa*	Scraping	80°<α<70°	2	30	4 000

34	qtfp1-05	45°	Wood	*Quercus ilex*	Scraping	60°	1	11	1000
35	qtfp1-06	45°	Wood	*Quercus ilex*	Cutting (uni)	90°	1	15	1000
36	qtfp1-07	30°	Shed antler	*Cervus elaphus*	Cutting (uni)	90°	1	13	1000
37	pay1-01	45°	Wood	*Quercus ilex*	Cutting	90°	1	13	1000
38	pay1-02	45°	Wood	*Quercus ilex*	Sawing	90°	1	6	500
39	pay1-03	45°	Wood	*Quercus ilex*	Scraping	70°<α<60°	1	6	500
40	pay1-04	50°	Wood	*Quercus ilex*	Planning	50°<α<30°	1	5	500
41	pay1-05	45°	Fresh skin	*Cervus elaphus*	Scraping	70°<α<60°	1	30	4000
42	pay1-06	45°	Fresh bone	*Cervus elaphus*	Scraping	70°<α<60°	1	7	500
43	TUMBL1-qtfu1		Sediment, water	-	Rolling	-		1 800 (30h)	-
44	TUMBL2-qtfu1		Sediment, water	-	Rolling	-		1 200 (20h)	-
45	TUMBL3-qtfu2		Sediment, water	-	Rolling	-		1 800 (30h)	-
46	TUMBL4-qtfp1		Sediment, water	-	Rolling	-		1 800 (30h)	-

Table 4.3: *Total number of experimental tools and the main variables of the experiments performed. The number of strokes was calculated a posteriori, with the aid of videotapes.*

4.1.1 Quartzite varieties

The experimental reference collection is composed of four different varieties of quartzite (Fig. 4.5). Three of them were collected in the vicinity of the Sierra de Atapuerca (Burgos, Spain), while the fourth one comes from the Rhône Valley (France), near the site of Payre. All the flakes pertaining to one variety were knapped from the same cobble to limit the raw material intra-variability. In all the cases, the technique used was direct percussion with quartzite hammers. The referents were kept constant (Tables 4.1, 4.3), summing up the whole experimental corpus. Two varieties are ascribed to the Utrillas facies (Fig. 4.5: a, b), being denominated by QTFU1 and QTFU2 respectively, and were collected at the Olmos sand quarry, near the village of Olmos de Atapuerca (Northern Spain) (Fig. 4.6: a, b). These two varieties are described in detail in section 4.1.2: the third publication included in this thesis.

The third variety (QTFP1, Fig. 4.5: c) pertains to the facies Pedraja (García-Antón, 2010) and the cobble from which flakes were obtained was collected on the most recent fluvial terrace associated to the Arlanzón River, specifically at the 3rd kilometre of the BU-820 national road (Fig. 4.6: c, d). The last type (PAY1, Fig. 4.5: d) is the only variety used as a reference for the study of the Payre site. In this case, six flakes were knapped from a single cobble which was collected in the vicinity of the site.

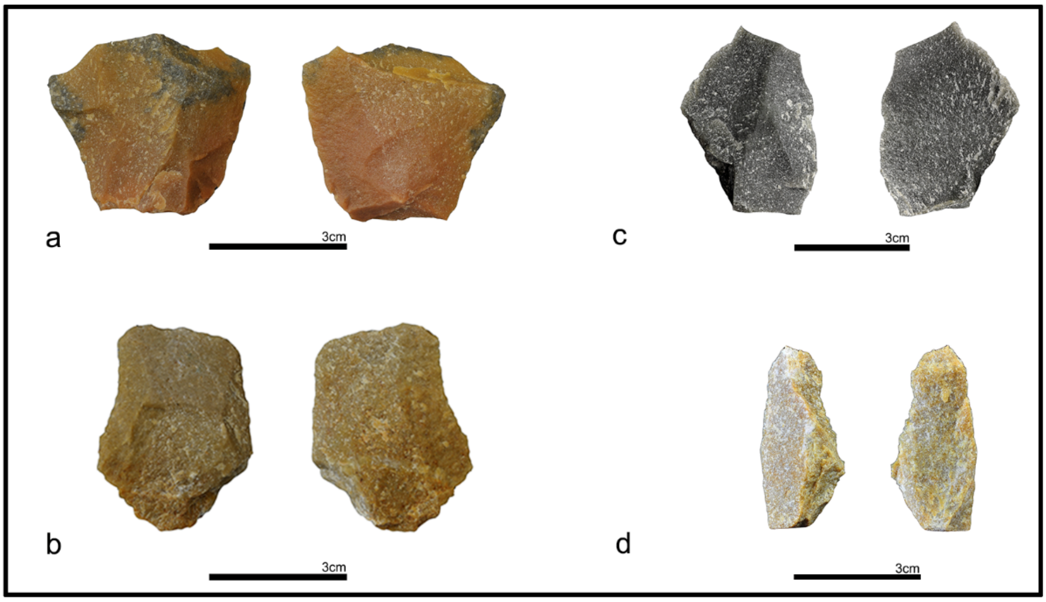

Fig. 4.5: *The four quartzite varieties included in the experiments: a) Qtfu-1, facies Utrillas; b) Qtfu-2, facies Utrillas; c) Qtfp-1, facies Pedraja; d) Pay-1, French variety. All are metaquartzites exhibiting different degrees of metamorphism.*

Fig. 4.6: *Modern localities where the cobbles used for experimentation were collected: enlarged pictures showing the morphology of the pebbles and cobbles. a) Modern sand quarry at Olmos de Atapuerca (Northern Spain); b) BU-820 highway (Northern Spain).*

Although micro-morphological data is available only for the Utrillas facies samples (Fig. 4.5: a, b), SEM observations might help in describing the surface peculiarities of the other varieties. Assuming that wear forms and develops differently on different lithic raw materials (and also on different varieties of the same raw material) (*e.g.* Beyries, 1982; Leipus and Levi-Sala, 1996; Mansur, 2007), a good characterisation of the raw material before analysing wear on it is vital in order to provide reliable functional interpretations. Differences in the micro-topography affect the formation of use-wear and therefore, it is extremely important to be familiar with the unused surfaces of the analysed lithologies before observing worn areas on them.

Quartzite or metaquartzite is a metamorphic rock very rich in quartz content, originating from quartz-arenites, which are sandstones containing less than 15% of matrix (the finer fraction) and at least 95% quartz grains (Tucker, 2001). Macroscopically, it is not always possible to distinguish between quartzites, orthoquartzites and sandstones. Although only the analysis of thin sections can provide secure results, the employment of other microscopic techniques may be helpful. SEM gives enough resolution to image single grains and EDX can rapidly identify inclusions.

Hence, by simply magnifying the unused surfaces with high resolution microscopes, it is possible to distinguish different varieties of the same raw material, based on their topographical features. Concerning the varieties selected for this study, SEM images prove that they are all metaquartzites with different degrees of metamorphism. The two Utrillas varieties are quite similar, though QTFU2 presents rare larger quartz grains compared with QTFU1 (Fig. 4.7, a, b). QTFP1 is extremely heterometric and borders between crystals are in the process of disappearing (which means that this variety has a higher degree of metamorphism than the other three) (Fig. 4.7: c). PAY1 variety is quite distinct from the others, showing a large presence of neoblasts (Fig. 4.7: d). This variety is described in further detail in section 7.3.

SEM inspection can be very useful also to discover the presence of accessory minerals. Knowing that the type of minerals and their relative proportions can provide parameters to differentiate among different rock varieties and to determine their provenance, it is important to learn to identify the aspect of those minerals under the microscope. Reflected metallographic microscopes do not seem to be appropriate, as they do not always allow to locate the inclusions, which may present similar colours of the rock substrate. Conversely, SEM can very easily detect differences in the general topography of the rocks and, after performing the EDX analysis, aid in identifying the elemental composition of the minerals. For more precise mineral identifications, specialised atlases need to be consulted (*e.g.* Welton, 1984)

Therefore, a general mineral characterisation of the studied varieties is provided, although no quantification calculations have been performed. Results about the Utrillas varieties (QTFU1 and QTFU2) are included in the 4.1.2 section, showing a very varied composition of the accessory minerals (Ca, Fe, K, Ti and Zr). The Payre variety (PAY1) seems to be quite homogeneous in its composition; no elements other than Si and O were found, indicating that the presence of accessory minerals is very low or absent. It is difficult to be more precise, as we do not dispose of petrographic data for this variety. The Pedraja variety (QTFP1) showed a very high presence of accessory minerals, mostly Fe and Ti (Fig. 4.8). The topographic aspect of minerals other than quartz is quite distinct and easy to detect under a SEM. Iron accumulations, at least in this variety, are organised in a layer pattern (Fig. 4.8: d), while titanium accumulations are more compact (Fig. 4.8: f). If those crystals had been observed through a backscattered electron detector, they would appear very bright (due to their high atomic number).

Both the micro-topography (granulometry, grain orientation and compaction, etc.) and composition (accessory minerals) influence the way use-wear forms and distributes over the surface area. An example of this is provided in Figure 4.9, where the use-wear resulting from the same activity performed during the same time interval on three varieties of quartzite is compared. Specifically, the worn edges are compared to the same portions before use (on replicas of the original surfaces) (Fig. 4.9: a, c, e). This is a particularly explicative example, as we selected one of the activities producing highly developed wear over short periods of time, being fresh hide scraping. We can see that, after only 15min of use, wear is very developed and quite continuous on QTFU1 (Fig. 4.9: b), while it is less continuous and more evident on protruding ridges of the micro-topography on QTFU-2 (Fig. 4.9: d). Very curiously, on the Pedraja variety wear is not visible at 100x (Fig. 4.9: c). The typical edge rounding is not present and only a slight polishing of the very edge is visible, when imaged at higher magnifications (at least 2000x).

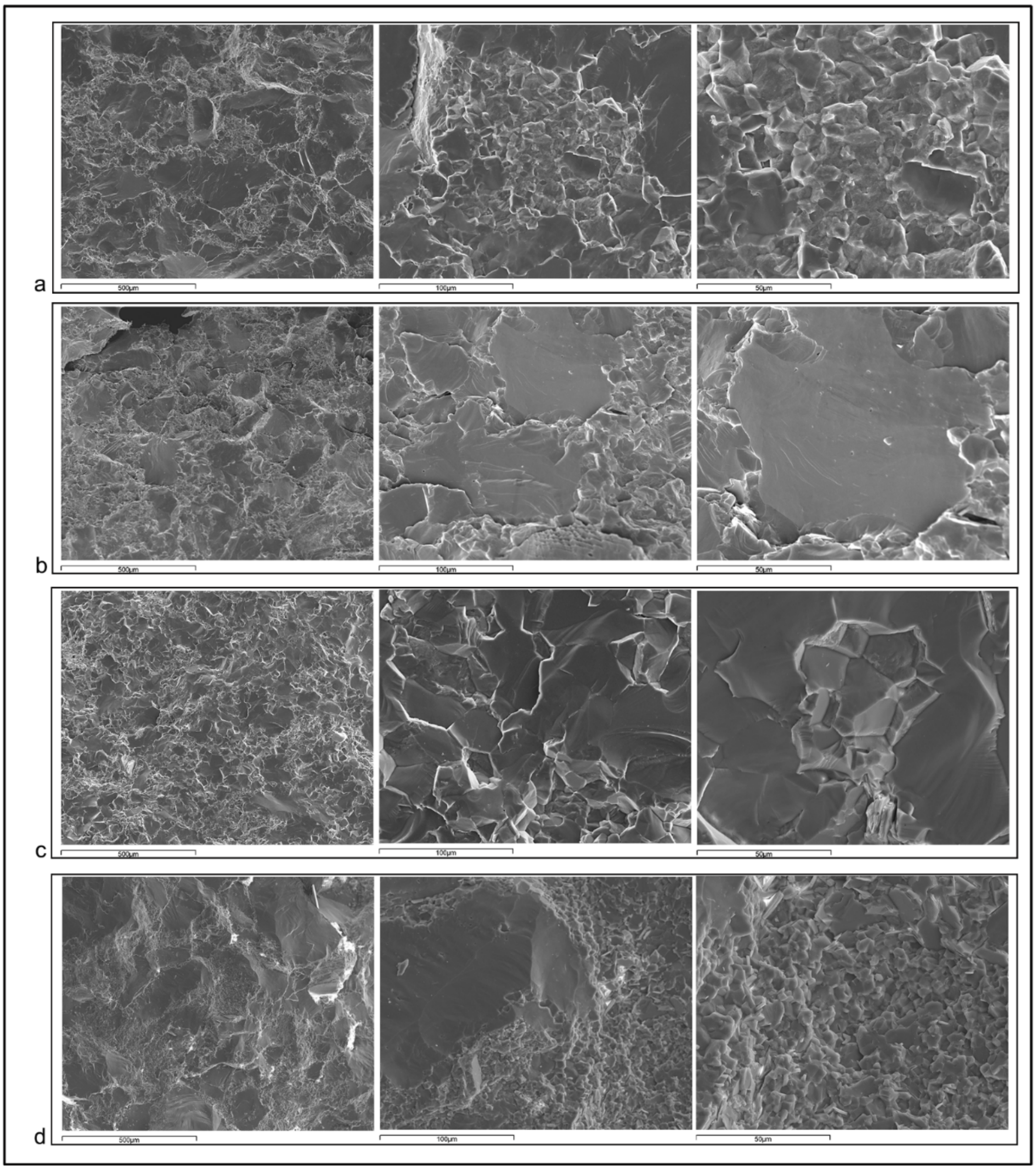

Fig. 4.7: *Different aspects of the micro-surface of the four metaquartzite varieties: a) Utrillas variety (qtfu1), b) Utrillas variety (qtfu2), c) Pedraja variety (qtfp1), d) Payre variety (pay-1). Magnifications and scale bars: first column, 250x (500µm), second column, 500x (100 µm), third column, 1000x (50µm).*

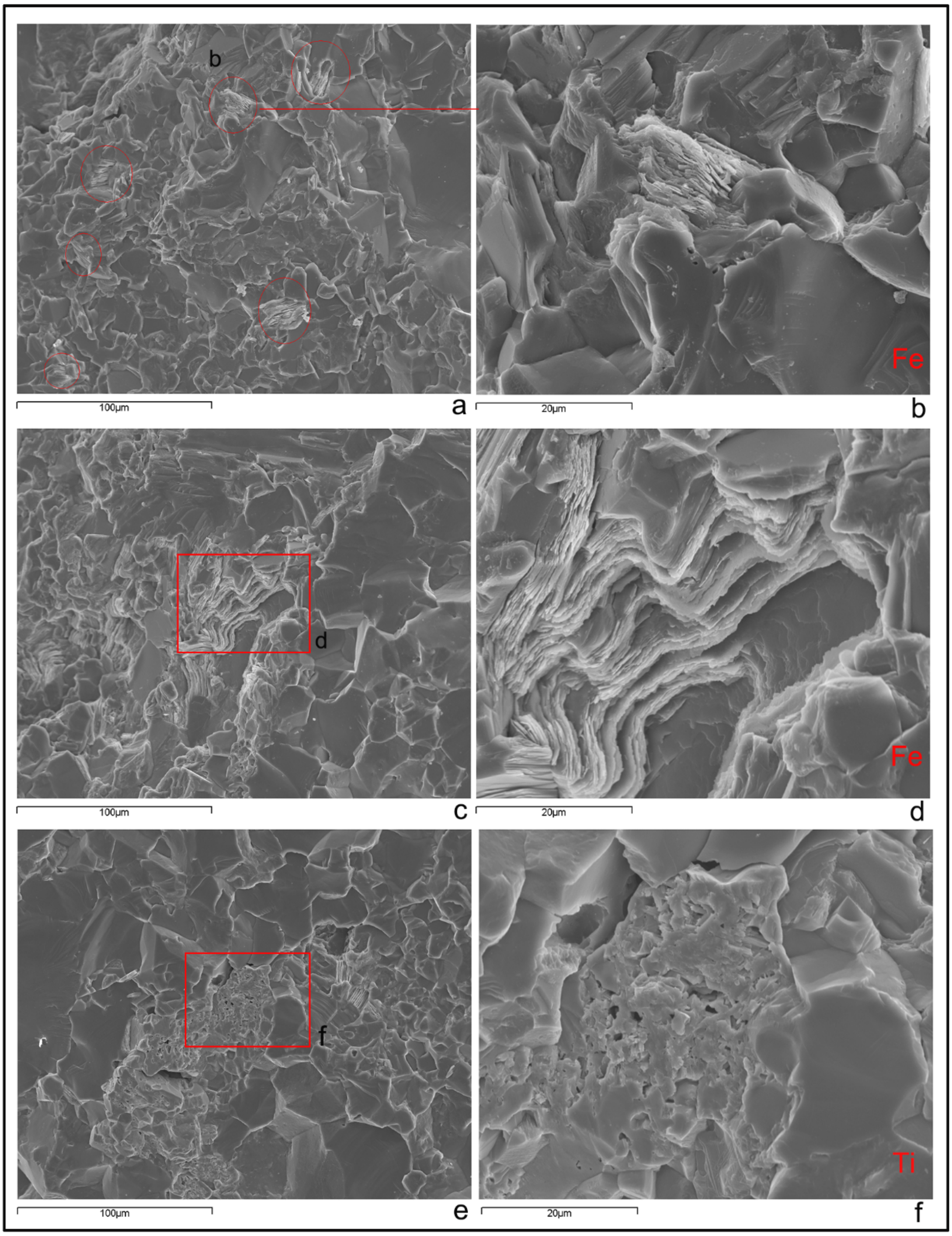

Fig. 4.8: *Mineral inclusions in the Pedraja variety (qtfp1): various iron inclusions and b) on enlarged image of one of them; c) iron inclusion and d) a close-up of it; e) a titanium inclusion and f) a close-up of it. Original magnifications: a, c, e) 500x; b, d, f) 2000x.*

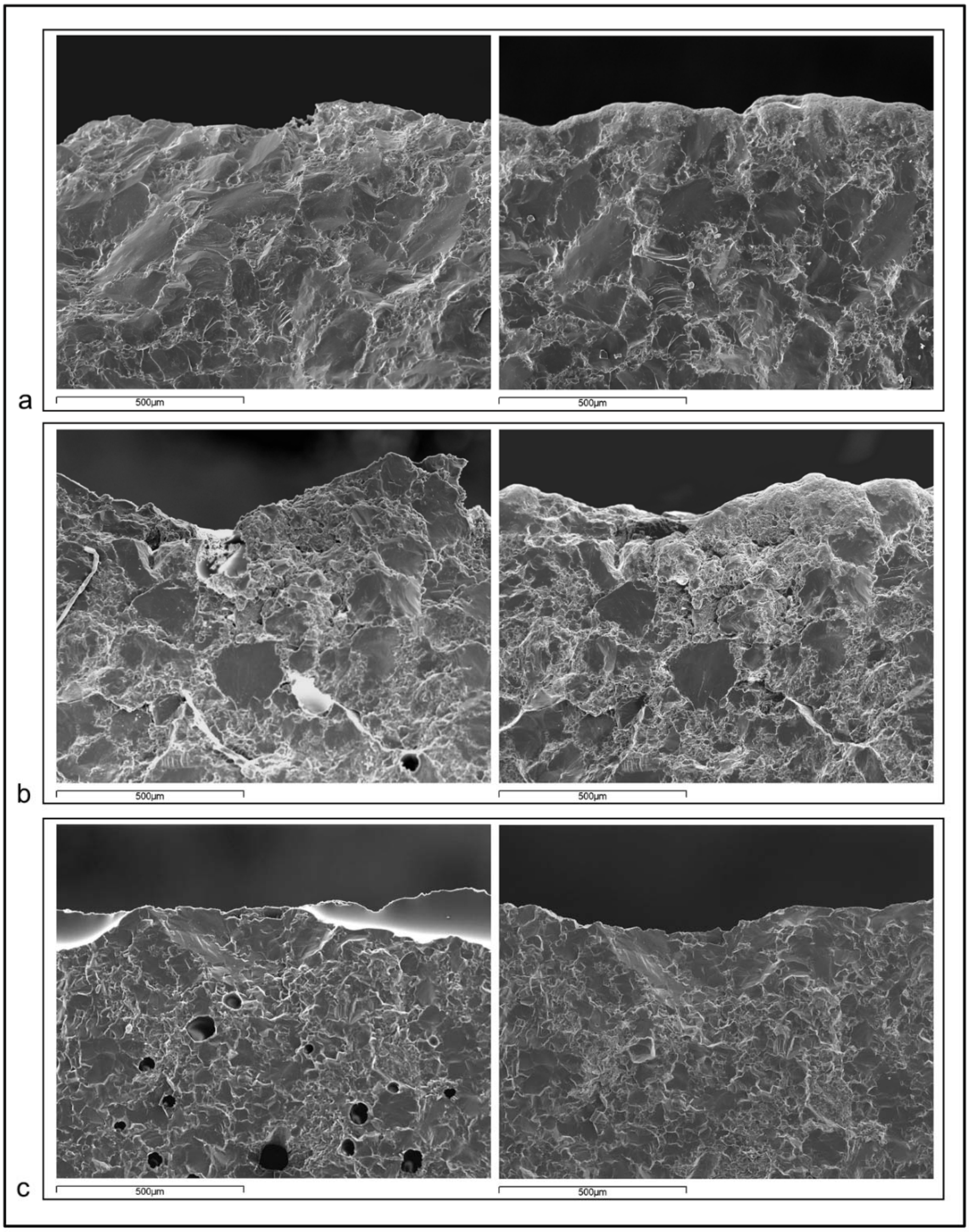

Fig. 4.9: *Comparison of use-wear developed on three quartzite varieties at the same magnification (100x). In the right column wear resulting from fresh hide scraping (15min). The controlled variables of the experiments are the same, but the resulting appearance of wear is quite distinct. The left column shows the same points as the right column, before utilisation. Faults of the replica are sometimes visible and can disturb the process of comparison (c). a) Utrillas variety, qtfu1; b) Utrillas variety, qtfu2; c) Pedraja variety, qtfp1.*

4.1.2 Publication 3:

Pedergnana, A., García-Antón, M. D., Ollé, A., 2016. **Structural study of two quartzite varieties from the Utrillas facies formation (Olmos de Atapuerca, Burgos, Spain): From a petrographic characterisation to a functional analysis design.** *Quaternary International* 433, 163-178.

The main objective of this publication is to provide petrographic characterisations of two quartzite varieties employed in the experiments. Starting from the analysis of composition and textural characters of the two varieties, inferences on the way wear forms and develops on them are discussed. The application of petrographic analyses to lithic materials before the development of methods for describing use-wear on them is regarded to be a valid contribution to traceology. The promising results encourage us to suggest that the previous characterisation of lithic raw materials' specific varieties is fundamental to be able to recognise use-wear on them.

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Structural study of two quartzite varieties from the Utrillas facies formation (Olmos de Atapuerca, Burgos, Spain): From a petrographic characterisation to a functional analysis design

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A B S T R A C T

This overall research initiative was undertaken to evaluate the role of lithic raw material variability in use-wear formation processes, focusing specifically on two lithological varieties of quartzite present in the Lower and Middle Pleistocene lithic assemblages of the Gran Dolina, Galería and Sima del Elefante sites (Sierra de Atapuerca, Burgos). Two Arenas de Utrillas facies varieties (from a sand quarry located in Olmos de Atapuerca) were studied using petrographic and SEM analyses. They were both identified as metaquartzites, revealing differences in their granular structure and metamorphic grade.

Sequential experiments employing the same quartzite varieties were then conducted involving different activities and several worked materials, which made it possible to determine use-wear formation on these two quartzite varieties. The results helped to clarify the role of intra-raw material variability in the process of use-wear formation on quartzite and contributed to ongoing studies of the lithic materials from these archaeological sites.

1. Introduction

In the past few decades, use-wear analyses have become an increasingly valuable interpretative tool in lithic studies. A functional analysis is defined as the interpretation of traces resulting from the various production processes that a lithic object has been involved in. Since the discipline was first founded (Semenov, 1964), the Russian legacy has been sustained by several subsequent developments and additions (Keeley, 1980; Vaughan, 1985; Moss, 1987; Van Gijn, 1990), which have made it a productive field in archaeological research.

Despite the different perspectives adopted, from investigating use-wear formation (e.g. Tringham et al., 1974; Brink, 1978; Diamond, 1979; Kamminga, 1979; Mansur-Franchomme, 1983; Unger-Hamilton, 1984; Fullagar, 1991; Yamada, 1993; Stemp and Stemp, 2003; Ollé and Vergès, 2008) to improving methods to quantify it (e.g. Grace, 1989; González and Ibáñez, 2003; Lerner, 2007, 2014a, 2014b; Evans and Donahue, 2008; Stemp et al., 2013), the general methodology of lithic use-wear analysis has been primarily set upon the study of industries mainly composed by flint/chert. Although valuable information has been obtained from these researches, resulting in the acknowledgment that a valid functional study always requires solid experimental bases, functional interpretations are still problematic. For instance, the functional analysis of non-flint/chert specimens is usually performed with little or very little regard for the specific characteristics of the different lithic raw materials. The few detailed experimental studies that have focused on the mechanical properties of lithologies chosen for the production of artefacts have highlighted the need for caution when making functional assessments with use-wear traces found on flint/chert as the only reference (Greiser and Sheets, 1979; Lerner et al., 2007; Braun et al., 2009; Delgado-Raack et al., 2009; Pargeter, 2013).

As different lithologies present specific intrinsic characteristics that influence their mechanical behaviour when subjected to stress, it would be appropriate to describe use-wear patterns related to all of them, emphasising their differences and similarities. It appears to be clear now that in a controlled situation (maintaining some variables constant) wear patterns differ depending on the type of lithic raw material being used, resulting in a use-wear panorama which is more varied than previously thought. In our opinion, in

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order to provide a more accurate interpretation of archaeological use-wear evidence, each type of rock should be treated individually, with the utmost attention paid to establishing precise parameters for describing surface modifications.

Many authors have suggested that the main source of the development of use-wear traces is the detaching of rock microchips from the surfaces of stone tools (Cotterell and Kamminga, 1979; Fedje, 1979; Kamminga, 1979; Lawn and Marshall, 1979; Levi-Sala, 1996; Ollé and Vergès, 2008). Consequently, an increase in fractures results in more intense use-wear, assuming that the internal structure of the different lithologies might influence use-wear formation to some degree. Material sciences have shown that cracks and fractures initiate from flaws in the raw material, as weak boundaries between grains (Lawn and Marshall, 1979). Therefore, in areas of high local tension, some cracks may develop into a well-defined propagating fracture. We aimed to assess which parameters could influence the propagation of those cracks, and as a consequence, use-wear formation.

With our primary goal being to contribute to the understanding of the general processes involved in use-wear formation, we chose quartzite as the investigated lithology in this study. The use of petrography as an auxiliary discipline in the characterisation of lithic raw materials prior to any use-wear description is regarded to be a fundamental step in reaching a reliable use-wear interpretation. We characterised two of the most common quartzite varieties in some of the Atapuerca lithic assemblages in detail, providing petrographic descriptions. We then attempted to evaluate the incidence of several parameters in the use-wear formation process for these varieties, such as their metamorphic grade, grain size and compaction, the presence of relict sedimentary features, and chemical composition. For each variety, we created a use-wear reference collection, which will be fundamental for comparison with the archaeological material. We noted differences in the relationship between material variation and fracture during the process of use-wear formation and sought to evaluate the degree of interference caused by these in the future functional interpretations of the archaeological material. As the main aim of this paper was to reach a major understanding of the properties of these two quartzite varieties, the use-wear data presented here are of qualitative nature.

2. The Utrillas facies and its role in lithic raw material procurement at Atapuerca

The Sierra de Atapuerca is an NNW–SSE oriented anticline located in northern Spain, near the city of Burgos. It contains archaeological sequences covering a period of more than one million years, which contributed to the reconstruction of human evolution in Euroasia. The Trinchera del Ferrocarril is an area where a railway trench was dug at the end of the 19th century, exposing several infilled karstic cavities. Gran Dolina, Galería and Sima del Elefante are the most important of these (Fig. 1). Details on the site characteristics, stratigraphy, chronology, and the archaeo-paleontological record can be found elsewhere (Rodríguez et al., 2011; Carbonell et al., 2014, and references therein). These sites have yielded a relatively continuous sequence of lithic technology going from mode 1 assemblages dated to the Early Pleistocene (Carbonell et al., 1999, 2008; Ollé et al., 2013; de Lombera-Hermida et al., 2015) to the Full and Late Acheulean, where some early Middle Palaeolithic features were identified (García-Medrano et al., 2014, 2015).

All of the lithic raw materials identified in the Atapuerca Lower and Middle Pleistocene archaeological assemblages are local (García-Antón and Mosquera, 2008). Chert outcrops are located on the top of the Sierra de Atapuerca, near the sites. The most representative of these outcrops are located on the SW slope of the Sierra, in Neogene deposits. In terms of coarse-grain materials (quartz, quartzite and arenite), two main detritic formations were documented: the Early Cretaceous "Arenas de Utrillas" facies (Cabrera et al., 1997; Pineda and Arce, 1997) and the Miocene "Pedraja" facies (García-Antón, 2010). Secondary deposits close to the Atapuerca sites (fluvial terraces) had been previously sampled and these samples underwent morphological and metric analyses. Comparing the quartzite varieties present in the Gran Dolina and Galería assemblages with those sampled, we were able to identify the areas associated with raw material procurement. They are located 5–10 km from the Atapuerca sites.

The two quartzite varieties analysed (V1 and V2) in this study belong to the Arenas de Utrillas facies formation (Fig. 2). This is a detrital formation containing sands and feldspathic clays with concentrations of iron oxides. The upper levels of this formation are conglomerates containing quartzose cobbles and pebbles (quartz and quartzite). Quartzite cobbles of the two varieties were collected in modern sand and gravel quarries located near the village of Olmos de Atapuerca (8 km NW from the Atapuerca sites). Among the cobbles coming from the Utrillas formation, we found a predominance of ellipsoidal and sub-spheroid morphologies suitable for knapping activity breaking through the quartz grains and giving rise to conchoidal fractures. Because of its origin, this type of quartzite is the most clearly recognisable in the Atapuerca lithic archaeological record. In fact, it was specifically identified at the Gran Dolina site (TD6 level, Mallol, 1999; and TD10.1 level, García-Antón and Mosquera, 2008; García-Antón, 2010) as well as at the Galería site (Gabarró et al., 1999).

Considering the entire lithic assemblage, V1 and V2 are not very abundant at the Atapuerca (Trinchera) sites, ranging from less than 1% in TD6 to less than 5% in Gran Dolina-TD10.1 (Table 1). If only non-chert raw materials are considered, representation increases; these two varieties are found mostly at TD10.1 (10%). The most significant differences involve the occurrence of the two quartzite varieties in the Lower Pleistocene (Gran Dolina-TD6) and Middle Pleistocene assemblages (Galería-GII and Gran Dolina TD10.1). In fact, their presence is inversely proportional, meaning that V1 is the most commonly used type in the Lower Pleistocene assemblages, while a preference for V2 was found in the Middle Pleistocene (Fig. 3).

Ongoing research is examining the degree to which differences in the abundance of these rocks in the sequence can be attributed to behavioural preferences in raw material management, or if it simply reflects availability (visibility of these rocks in the secondary deposits during the Lower–Middle Pleistocene). For this reason, a more in-depth characterisation of the varieties employed in knapping activities is needed in order to better understand the properties of these materials. This will contribute to making the results of further use-wear analyses on the Atapuerca quartzite assemblages more reliable.

3. Methodology

3.1. Quartzite terminology

Quartzite is a basic term normally used to define a wide variety of different rocks, from cemented sandstones to well metamorphosed specimens. Technically speaking, the term quartzite should be used only to define metamorphic types. Silica cemented sandstone should be distinguished (although its knapping behaviour might be similar), as it generates conchoidal fractures that break through grains and not around them (as in the case of regular sandstone). Therefore, quartzite or metaquartzite is a metamorphic rock very rich in quartz content. Its protolith is generally a quartz

Fig. 1. Location of the Sierra de Atapuerca on the Iberian Peninsula and position of the three archaeological sites in the Trinchera del Ferrocarril.

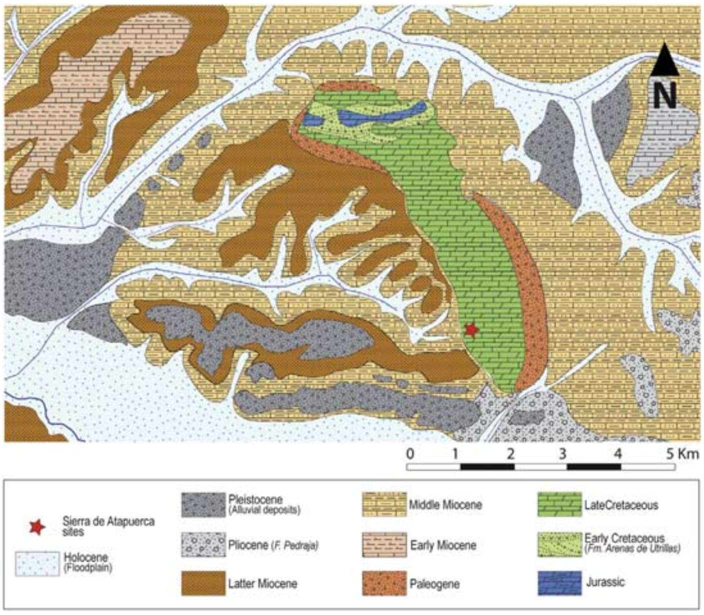

Fig. 2. Location of the Trinchera del Ferrocarril archaeological sites, the facies Arena de Utrillas formation and other main raw material sources in the surrounding of the Sierra de Atapuerca.

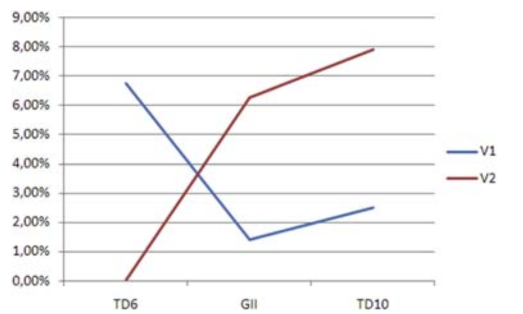

Fig. 3. Graph showing the inversely proportional quantitative patterns of the two analysed quartzite varieties from the most ancient chronologies (GD-TD6) to the most recent ones (GD-TD10).

arenite, which is sandstone containing less than 15% of matrix (the finer fraction) and at least 95% quartz grains (Tucker, 2001). This type of rock is also known as orthoquartzite. Orthoquartzite also originates from sandstone, but it did not suffer any metamorphic process. Sometimes individual quartz grains are interlocked by a siliceous cementing solution that hardens around the grains (Pettijohn et al., 1987; Blatt et al., 2006). When this occurs, fracture is more homogeneous and, as in metaquartzites, it breaks across quartz particles.

After undergoing heat and pressure deformation phases, quartz arenites achieve a stable crystalline structure. Quartzite can reach this stability after different phases of the metamorphic process and may sometimes display relict features of previous phases (banded structures, replaced porosity). The identification of the metamorphic phase of quartzites and the presence of accessory minerals allow distinctive varieties to be differentiated (Blomme et al., 2012). Besides, when studying archaeological material, it is important to understand how different rocks varieties fracture, since each

Table 1
Representation of the two Utrillas quartzite varieties (V1, V2) from selected samples of the Gran Dolina site (levels TD6 and TD10.1) and the Galería site (unit GII). The table shows the relative weight of these varieties as compared to the total selected sample (including chert, quartzite, quartz, sandstone and limestone), and also compared only to raw materials recovered from detrital deposits such as fluvial terraces, dismantled conglomerates and other similar formations (García-Antón, 2010).

		V1	V2	Total
TD6	Quartzite record	19	3	22
	% of the detrital record (total n° 281)	6.762%	0.011%	6.772%
	% of the selected sample (total n° 620)	1.068%	0.002%	1.069%
GII	Quartzite record	2	9	11
	% of the detrital record (total n° 144)	1.39%	6.25%	7.64%
	% of the selected sample (total n° 431)	0.46%	2.09%	2.55%
TD10.1	Quartzite record	11	35	46
	% of the detrital record (total n° 443)	2.48%	7.90%	10.38%
	% of the selected sample (total n° 991)	1.11%	3.53%	4.64%

crystalline phase gives quartzite different mechanical characteristics. According to Fig. 4, we see that in phase 1 the border of the grains is deformed plastically, creating imbricate grains and great mechanical coherence. Knapping activity converts percussive energy into a wave which is uniformly distributed across the rock's surface, producing a perfect conchoidal fracture and leaving a flat flaked surface. During the recrystallisation process of phase 2, the mechanical wave is discontinuously distributed. Conchoidal fracture is observable, although the flaked surface reflects some discontinuities in the wave amplitudes, resulting in a more irregular topography. Micro-discontinuities are produced when the energy wave passes through the neoblast fraction (newly formed grains) and changes its intensity due to variations in granulometry. The result is a macroscopic granular and inhomogeneous flaked surface, resembling fine grain quartz arenites. Phase 3 quartzites are well-developed metaquartzites and were not found in the cobbles recovered; however, the expected fracture for phase 3 quartzites would be similar to that one described for phase 1 varieties. The polygonisation or annealing process generates an isotropic material characterised by an equidimensional mosaic grain structure. The wave energy is distributed uniformly and the flaked surface is even flatter and more regular than phase 1 quartzites. In fact, the physical properties of isotropic solids do not vary with direction. The isotropic attributes of metaquartzites are due to the unimodal grain structure acquired at the end of an ideal metamorphic sequence (Kornprobst, 1996).

3.2. Experimental activity

An extensive experimental programme was established to create a reference collection of use-wear traces on quartzite in order to provide an empirical foundation for subsequent functional analyses of quartzite archaeological assemblages (Pedergnana and Ollé, 2014). However, to correctly evaluate the role of the different lithological structures in use-wear formation processes, different quartzose rock types should be included (from quartz arenites/orthoquartzites to well-developed metaquartzites) in the experimentation. Before adding other lithologies, we decided to focus on quartzite in order to first determine if minor divergences in the same rock type are recognisable. Therefore, this study only

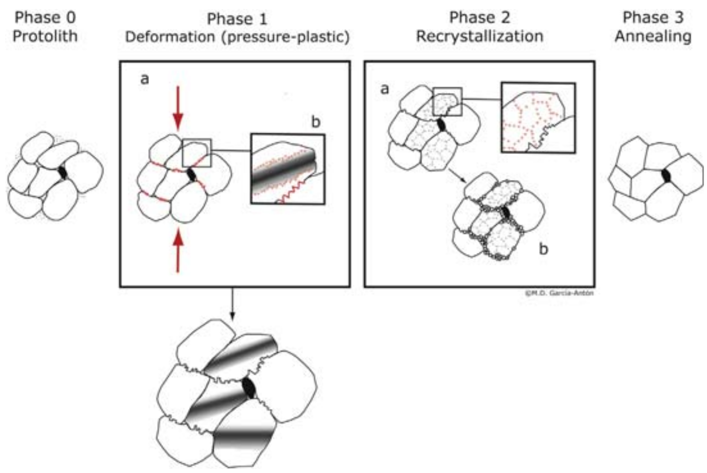

Fig. 4. Illustration of the various phases of metamorphism that quartzite can undergo. Phase 0) The parent rock, quartzarenite or orthoquartzite, exhibits rounded grains. Clasts are distinguishable from matrix. Siliceous cement, binding the clasts and matrix together, may or may not be present. Porosity is generally very high. Phase 1) Two deformation types occur in this phase: a) deformation through pressure among the grain borders, which is perpendicular to compressive forces, b) plastic inter-crystalline deformation, where the exerted forces provoke dislocations in the grains interior. The effort to stabilise these forces results in a regular, oriented pattern. Phase 2) Recrystallisation processes: a) the inter-crystalline dislocations are now grouped together generating lines which are the subgrain borders, b) when the grain edges are deformed, new crystals or neoblasts form (dynamic recrystallisation). Two different grain size populations are visible. Phase 3) Annealing or polygonisation process: a crystalline restructuring takes place, in which the smaller grains are eliminated, giving rise to more stable grain borders. Relict features related to the porosity of the protolith are no longer visible and the grain fabric is described as having a perfect equigranular appearance.

examines two metaquartzite varieties (V1 and V2), both coming from modern sand and gravel quarries near the village of Olmos de Atapuerca (Burgos), containing quartzites pertaining to the Utrillas facies. We included simple flakes (ten flakes for each variety) obtained from a single cobble of each variety through hand-held direct percussion. Different worked materials (wood, hide, meat, bone, plant matter) and actions (cutting, sawing, scraping) were considered. All the experiments were performed under laboratory conditions, maintaining some chosen variables fixed, as the type of movement, the contact angle, and the elapsed time (Table 2). The experiments were sequential (different use phases of the same artefacts, maintaining the same variables and microscopic observations of some selected control points) in order to monitor the development of use-wear in each variety. The control points (i.e. the specific areas on the tools' edges were wear is monitored through various stages) were selected after the first stage of use, taking in account the degree of development of the use-wear traits observed and their position with regard to the edge. Moulds and casts were taken to examine the appearance of the edges prior to utilisation, following the protocol of Ollé and Vergès (2008, 2014) and Pedergnana and Ollé (2014). This enabled us to compare wear resulting from use with the rocks' original surfaces.

3.3. Microscopic analyses

Macroscopic characterisation is the first step in the analysis of the physical properties of the materials from which stone tools are made. A general description of the two quartzite varieties was obtained by means of visual inspections and low-power (stereoscope, with magnifications from 5× to 60×) observations. Afterwards, samples of the same inspected lithologies were scanned with a scanning electron microscope (SEM) to obtain better resolution of the morphological, textural, topographic, structural, and chemical features of the samples. When available, back-scattered electron detector and energy-dispersive x-rays spectroscopy (EDX) can be used to investigate the composition of the rock (Krinsley and Manley, 1989; Krinsley et al., 1998; Braun et al., 2009). However, the most reliable analysis for studying the structural heterogeneity of rocks is the examination of thin-sections. Structural features, both deriving from original chemical composition and from diagenesis, were described applying conventional petrographic observations (Klein and Hurlbut, 1996; Blatt et al., 2006; Winter, 2010). The petrographic study of thin-sections of the Utrillas quartzites was conducted using an Olympus BH-2 microscope with polarised light.

Use-wear analysis on the same experimental varieties focused on high-power techniques, resorting to optical light microscopy (Zeiss Axio Scope A1) with magnifications from 50× to 500×, and two SEMs (an SEM JEOL JSM-6400 and an ESEM FEI Quanta 600), where the maximum magnification used was 3000×. Both SEM microscopes were equipped with an Oxford Instruments INCA system for digital image acquisition and microanalysis by electron probe (EDX).

All the experimental pieces were submitted to a rigorous cleaning procedure aimed to eliminate the organic residues. The chemical products involved were hydrogen peroxide, a neutral phosphate-free detergent (Derquim®) and acetone (for more details see Byrne et al., 2006; Ollé and Vergès, 2014; Pedergnana and Ollé, 2014).

4. Results

4.1. Petrographic study

After an initial macroscopic screening, thin-sections of the most representative varieties were obtained and later analysed.

The first quartzite variety analysed is an initial metamorphic form (Fig. 4, phase 1), displaying a colour ranging from brownish and greyish tones to reddish and greenish ones. Its finer granular texture was observable with the naked eye, and it clearly fractured conchoidally. Lustre is greasy, vitreous, and sometimes translucent. It consists almost completely of angular to sub-angular quartz grains. In addition to quartz grains (95%), some iron oxides, sericite, muscovite, zircon, rutile and clinochlore were randomly distributed throughout the samples (Fig. 5: A; Fig. 7: A; Fig. 8: D, E, F). Muscovite and iron oxides sometimes present a regularly oriented pattern (Fig. 5: B). The crystalline texture is granular, exhibiting a granoblastic bimodal quartz mosaic (Fig. 5: C) (Yardley et al., 1990). Grain size ranges from 50 to 100 μm and the quartz grain borders are sutured. An initial formation of neoblasts is also observed. Quartz grains rarely exhibit undulatory extinction.

Table 2
Use experiments performed with unretouched flakes knapped from cobbles of the two varieties collected from secondary deposits. Summary of the main controlled experimental variables.

Ref. Code	Edge angle	Worked material	Worked material type	Action	Movement	Contact angle[a]	Time (minutes)
V1-01	40°	Fresh wood	*Quercus ilex*	Sawing	Long-Bid	60° <α < 90°	15 + 15 + 30
V1-02	60°	Shed antler	*Cervus elaphus*	Sawing	Long-Bid	90°	15 + 15 + 30
V1-03	40°	Skin	*Cervus elaphus*	Cutting (skinning)	Long-Bid	80° <α < 40°	35 + 26 + 20
V1-04	45°	Fresh hide	*Cervus elaphus*	Scraping	Transv-Uni	60°<α < 30°	15 + 15 + 15
V1-05	50°	Fresh bone	*Cervus elaphus/Bos taurus*	Sawing	Long-Bid	80° <α < 90°	15 + 15 + 30
V1-06	25°	Fresh bone	*Cervus elaphus/Bos taurus*	Scraping	Long-Bid	90°	15 + 15 + 30
V1-07	60°	Dry hide	*Cervus elaphus*	Scraping	Transv-Uni	40°	15 + 15 + 15
V1-08	30°	Fresh wood	*Quercus ilex*	Scraping	Transv-Uni	40°	15
V1-09	45°	Dry hide	*Cervus elaphus*	Cutting	Long-Bid	90°	15 + 15
V1-10	35°	Giant cane	*Arundo donax*	Sawing	Long-Bid	90°	15 + 30
V2-01	55°	Fresh wood	*Quercus ilex*	Sawing	Long-Bid	90°	15 + 15 + 30 + 30
V2-02	50°	Shed antler	*Cervus elaphus*	Sawing	Long-Bid	80° <α < 90°	15 + 15 + 30 + 30
V2-03	35°	Flesh, tendons bone	*Cervus elaphus*	Cutting (defleshing)	Long-Bid	80° <α < 90°	35 + 50
V2-04	45°	Fresh hide	*Cervus elaphus*	Scraping	Transv-Uni	80°	15 + 15
V2-05	45°	Fresh bone	*Cervus elaphus/Bos taurus*	Sawing	Long-Bid	80° <α < 90°	15 + 15 + 30
V2-06	40°	Fresh bone	*Cervus elaphus/Bos taurus*	Scraping	Transv-Uni	45°	15 + 15 + 30
V2-07	40°	Dry hide	*Cervus elaphus*	Scraping	Transv-Uni	40°	15 + 15
V2-08	50°	Fresh wood	*Quercus ilex*	Scraping	Transv-Uni	45°	15 + 15
V2-09	35°	Dry hide	*Cervus elaphus*	Cutting	Long-Bid	90°	15 + 15 + 15
V2-10	45°	Giant cane	*Arundo donax*	Sawing	Long-Bid	90°	15 + 15

[a] The contact angle is defined as the angle between the contact face of the lithic tool and the worked material.

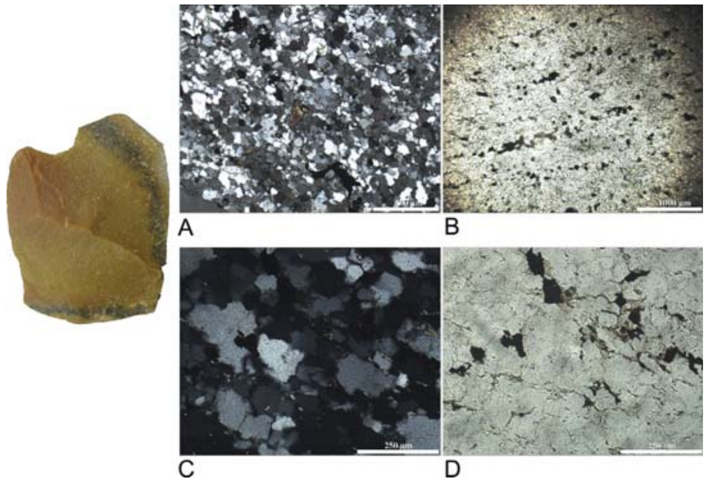

Fig. 5. Thin-section microphotographs of Utrillas Facies Variety 1 quartzite (V1): A) heterogranoblastic quartz texture with chlorite, muscovite and hematite minerals (crossed-polarised image, scale bar: 500 μm); B) relict sedimentary structure showing an oriented pattern with opaque banded hematite minerals (plain light image, scale bar: 1000 μm); C) quartz grain irregular granoblastic texture with intergranular hematite and sericite minerals (crossed-polarised light image, scale bar: 250 μm); D) detail of the granular hematite minerals and non-opaque intergranular iron oxides (plain light image, scale bar: 250 μm). C and D show the same imaged point.

The second variety is an intermediate metamorphic form. It differs from V1 in colour, which ranges from whitish, greyish tones to yellowish ones. It is a metaquartzite presenting conchoidal fracture with a tendency towards schistosity and the macroscopic texture is defined as saccharoid or granular. The lustre is waxy or greasy. Besides a quartz content of 98–99%, secondary minerals such as zircon, clinochlore, sericite, iron oxides, and rutile are present (Fig. 6: C; Fig. 7: E, C).

It exhibits a well-developed granoblastic structure with no porosity (Fig. 6: D). There is strong neoblasts growing, observed among the elongated quartz crystals. The neoblasts appear as subgrains among the quartz grain borders, pushed together by pressure forces (Fig. 6: B) (Kornprobst, 1996). The metamorphic process is advanced, placing it within phase 2 (Fig. 4), with consistent recurrence of undulatory extinction on the quartz grains. The deformed grains exhibit oriented lamination (Fig. 6: A), reflected in the fracturing pattern. Grain size in this variety is heterogeneous with quartz grains ranging from 100 to 200 μm, while neoblasts measure ~25 μm.

Both varieties were defined as metaquartzites, although characterised by a different metamorphic grade. V1 is less metamorphosed than V2, and has lower quartz content, with little

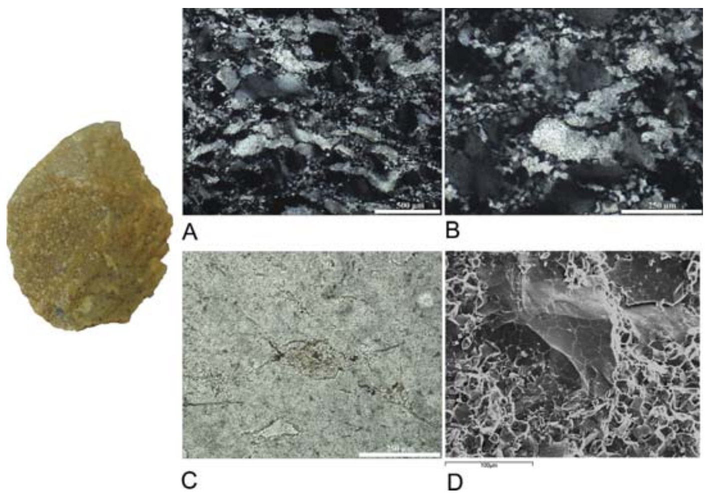

Fig. 6. Thin-section microphotographs of Utrillas facies quartzite Variety 2 (V2): A) granoblastic texture in quartz banded tectonic structure (crossed-polarised image, scale bar: 500 μm); B) undulatory extinction in deformed quartz crystals and neoblasts growing among grain boundaries. On the top of the image it is possible to distinguish some subgrains in the quartz grains (crossed-polarised image, scale bar: 250 μm); C) intergranular non-opaque iron oxides and muscovite-sericite minerals (plain light image, scale bar: 250 μm); D) detail of the neoblasts growing and metamorphic subgrains formed in the quartz grains (SEM microphotograph, mag.: 500×, scale bar: 100 μm).

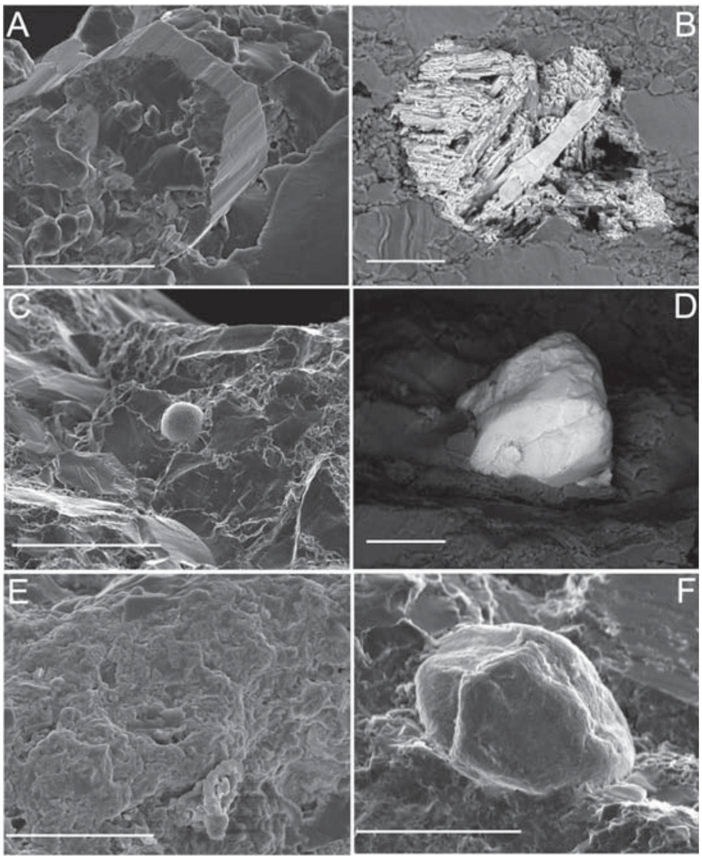

Fig. 7. SEM micrographs of some of the accessory minerals identified on the two varieties of quartzite from the Utrillas facies. A rutile crystal documented on V1 (A) and on an archaeological artefact (Gran Dolina-TD.10) (B). A zircon crystal embedded in a V2 sample (C) and on an archaeological sample (Gran Dolina-TD.10) (D). A hematite spot (E) and an identified crystal (F) showing Ca and Fe in its composition, both on V2 specimens. Crystals are imaged through a secondary electron detector showing their specific morphologies, except for images B and D, which were taken with a back-scattered electron detector. Therefore, the bright appearance of the elements with a high atomic number (Ti: z = 22; Zr: z = 40) is appreciable. A) mag.: 2000×, scale bar: 50 μm; B, D) 1300×, scale bar: 50 μm; C) mag.: 250×, scale bar: 200 μm; E) mag.: 1000×, scale bar: 50 μm; F) mag.: 700×, scale bar: 80 μm.

presence of undulatory extinction of quartz grains. V1 presents very well-sutured quartz grains, while V2 has a very notable presence of neoblasts between quartz grain borders generating two granulometric populations, which influence wave propagation during knapping activity. The main structural and compositional attributes of both quartzite varieties are summarised in Table 3.

4.2. Scanning electron microscopy

Secondary electron and back-scattered electron detectors were employed to analyse the surface topography and compositional contrast of the samples. Examined with SEM, quartzite fracture surfaces exhibit conchoidal fracture which occurs across the quartz grains (Fig. 13: A), and sometimes obstructed by the presence of neoblasts in V2 (Fig. 13: C). SEM also proved very useful in detecting the presence of accessory minerals in the samples, especially by means of back-scattered electron detectors. X-ray analysis (SEM-EDX) was applied to single crystals to detect their elemental composition. It has been shown that it is possible to differentiate quartzite varieties on the basis of differential amounts of accessory minerals (Blomme et al., 2012). For this purpose, a more precise mineral characterisation is needed in order to better localise the varieties employed at the Atapuerca sites.

Several minerals were identified in our samples based on their morphology and chemical composition. Zircon and rutile are the most abundant, and are also found in Gran Dolina artefacts (Fig. 7: B, D). The characteristic appearance of those minerals, different from that of quartz crystals, is more easily identifiable using a SEM-back scattered electron detector, but their structural traits are also distinctive using a SEM secondary electron detector. Generally, secondary minerals are integrated into the rock micro-structure, embedded among quartz grains (Fig. 7). All of this information is normally missed when only an optical microscope is used, though we were able to locate a square-shaped crystal with a metallographic microscope after having found it with the SEM (Fig. 8: B, C). The relative EDX spectrum revealed the presence of Ca, Fe and P. A similar crystal, but slightly smaller, was found on a thin-section of

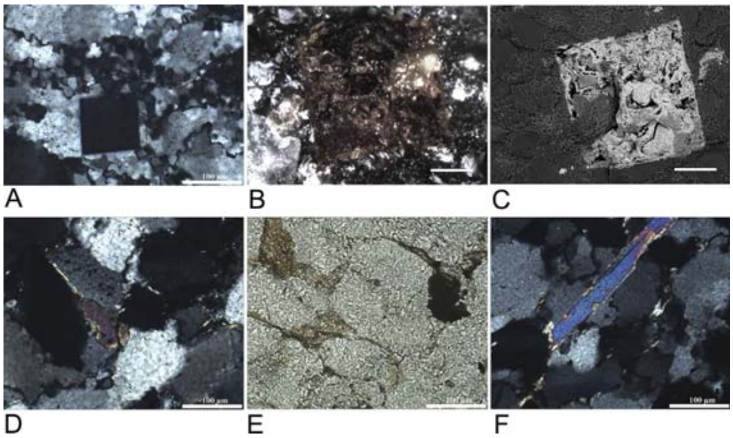

Fig. 8. An identified square-shaped crystal seen under optical microscopes (A = crossed-polarised light image, scale bar: 100 μm; B = incident light image, mag.: 200×; scale bar: 100 μm) as well as with SEM secondary electron detector (C = mag.: 510×, scale bar: 100×). The crystal depicted in images B and C is the same. The associated EDX spectra identified the presence of Fe, Ca and P. Other secondary minerals documented on V1 specimens: zircon (D = crossed-polarised light image, scale bar: 100 μm), hematite oxide (E = plain light image, scale bar: 100 μm) and muscovite mica (F = crossed-polarised light image, scale bar: 100 μm).

Table 3
Summary of the general and petrographic characteristics of the two analysed quartzite varieties.

	V1	V2
Naked eye texture	Regular fine	Coarser and saccharoid
Fracture	Conchoidal	Conchoidal with schistosity episodes
Mineralogical composition	95% quartz	98–99% quartz
Accesory minerals	Zircon, rutile, muscovite-sericite, clinochlore and hematite	Rutile, clinochlore and hematite
Grains shape	Sub-angular	Angular
Texture	Granoblastic	Well-developed granoblastic
Grain size	50–100 μm	100–200 μm
Porosity	Very low	Zero
Cohesion	High	High

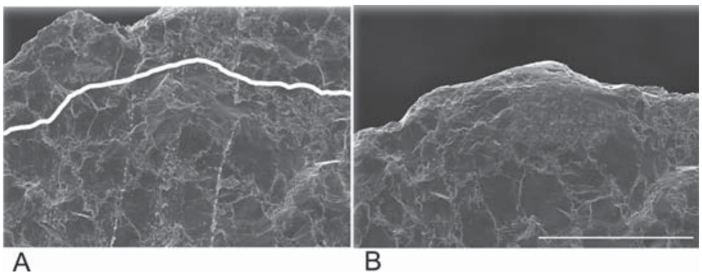

Fig. 9. Comparison of a V1 edge portion used for 15 min to cut (bi-directionally) a fresh cane stalk (B) with the same point prior to use (A). Edge was reduced by ca. 100 micron, and later polish developed on the highest topographical points. Fracture seems to have initiated through the larger grain boundaries and then eventually crosses to some smaller grains (on the right). Loss of material characterises the initial phase of all brittle materials subjected to stress (knapped rocks in general). In the case of quartzite, it accompanies the entire process of use, though having a noticeably stronger impact when the edge first comes into contact with the worked material. A, B) mag.: 100×, scale bar: 500 μm.

V2 (Fig. 8: A). Intergranular iron oxides were also occasionally documented (Fig. 7: E; Fig. 8: E).

4.3. *Experimental use-wear description*

The documented petrographic characteristics allowed us to identify both rock varieties as metaquartzites. Based on their similar internal structure and chemical composition, we would expect a relatively equivalent mechanical behaviour. Indeed, use-wear patterns are very similar and few differences were documented. Divergences between the two varieties include micro-fracturing behaviour and, in some cases, use-wear distribution and invasiveness on edges to which the same experimental variables (action, worked material, elapsed time, etc.) were associated.

4.3.1. *Fracturing: loss of material*

As already observed (Clemente Conte and Gibaja Bao, 2009), the first stage of quartzite edge modification is fragmentation (Fig. 9).

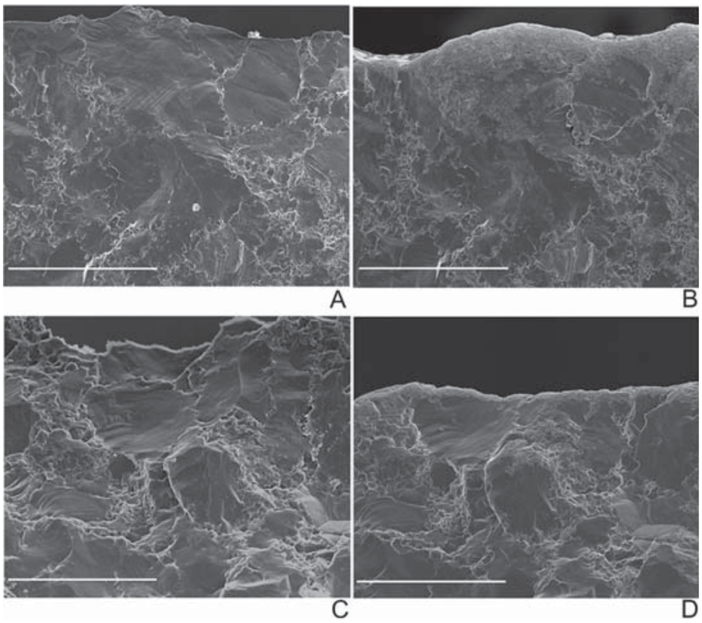

Fig. 10. Rough polish appearance observed on a V1 flake (B) and on a V2 flake (D) with SEM after 15′ of scraping fresh hide. The same edge portions were observed before utilisation to detect the points where wear initially develops. Although both control points are located on the face that has the most contact with the worked material, the distribution of the polish is different. The rim shows continuous abrasion which penetrates for ca. 100 μm from the original surface topography (B). On extremely irregular portions, wear is only visible on the highest points and the lower crystals are intact, having avoided any durable contact with the worked material (D). The evaluation of the surface roughness is based on qualitative assessments. A, B) mag.: 100×, scale bar: 500 μm; C, D) mag.: 250×, scale bar: 200 μm.

This brittle behaviour was also seen in other lithic raw materials, although to differing degrees. Vitreous materials, such as obsidian and rock crystal, are much more fragile than quartzite (Sussman, 1985, 1988; Knutsson, 1988; Hurcombe, 1992). However, fracturing at an early use-stage seems to occur more frequently in quartzite than in flint/chert for example, probably because of the larger quartz grain size (leading to larger detached particles). In fact, when two bodies are in relative motion across one another, the first mechanical event is the detachment of particles from both bodies (depending on the hardness of the material). What concerns us is the fragmented rock particles which cause an evident decrease in edge volume and play a crucial role in the formation of wear (Semenov, 1964; Cotterell and Kamminga, 1979; Kamminga, 1979).

During our experiments, we noticed that V1 exhibited a more fragile macroscopic behaviour, meaning that it tended to break more frequently than V2 when processing hard materials. The observed fragmented micro-chips of V2 were larger than those detached from V1 flakes. The slightly different structure of the two varieties seems to influence the fracturing process in that V1 displays more isotropic attributes than V2, and therefore homogeneously fractures when force is applied. V2, on the other hand, offers more resistance to the applied stress and fragments made up of several quartz grains are detached, typically creating micro-schistosity planes.

4.3.2. Edge damage: rounding and scarring

Any use tends to leave the working edge very rounded, as a consequence of the continuing load (and relative stresses) to which it is subjected. Depending on the worked material involved in the action, this process leaves behind more or less evidence. When treating very abrasive materials (hide), a longer and generally continuous portion of the edge becomes rounded, and this wear feature is already appreciable after only 15 min (Fig. 10: B; Fig. 11: L). When diverse experimental variables (other worked materials or actions) are considered, the abraded part is concentrated only on a small portion of the edge, usually on the border of a quartz grain (Fig. 11: M).

We found that V1 tends to develop edge rounding faster than V2, although the differences are negligible. This might be related to the size of the detached rock particles, which in the case of V1 are smaller and more numerous. Being smaller, they can easily remain embedded in the interfacial medium created between the tool and the worked material. These particles act as indenters during the process of wear formation (Lawn and Marshall, 1979; Ollé and Vergès, 2008). Meanwhile, the relatively larger fragments detached from V2 pop off the flake, and very few of them are likely to remain embedded within residues. This might be why edge rounding develops faster and to a relatively higher degree in V1. At any rate, edge rounding is always due to erosional processes, starting on the higher parts of the micro-topography and then expanding to the lower parts.

Edge scarring as defined in the use-wear literature (Tringham et al., 1974; Odell, 1981) is not a reliable criteria to analyse quartzite, first of all because its occurrence is rare and it does not display a characteristic pattern with regard to different worked materials and actions (Gibaja et al., 2002; Leipus and Mansur, 2007). Scars are usually caused by contact with hard materials (Tringham et al., 1974) and they normally result from bending and compressive stresses (Cotterell and Kamminga, 1979). We confirmed their scarcity on both of the Utrillas varieties and found

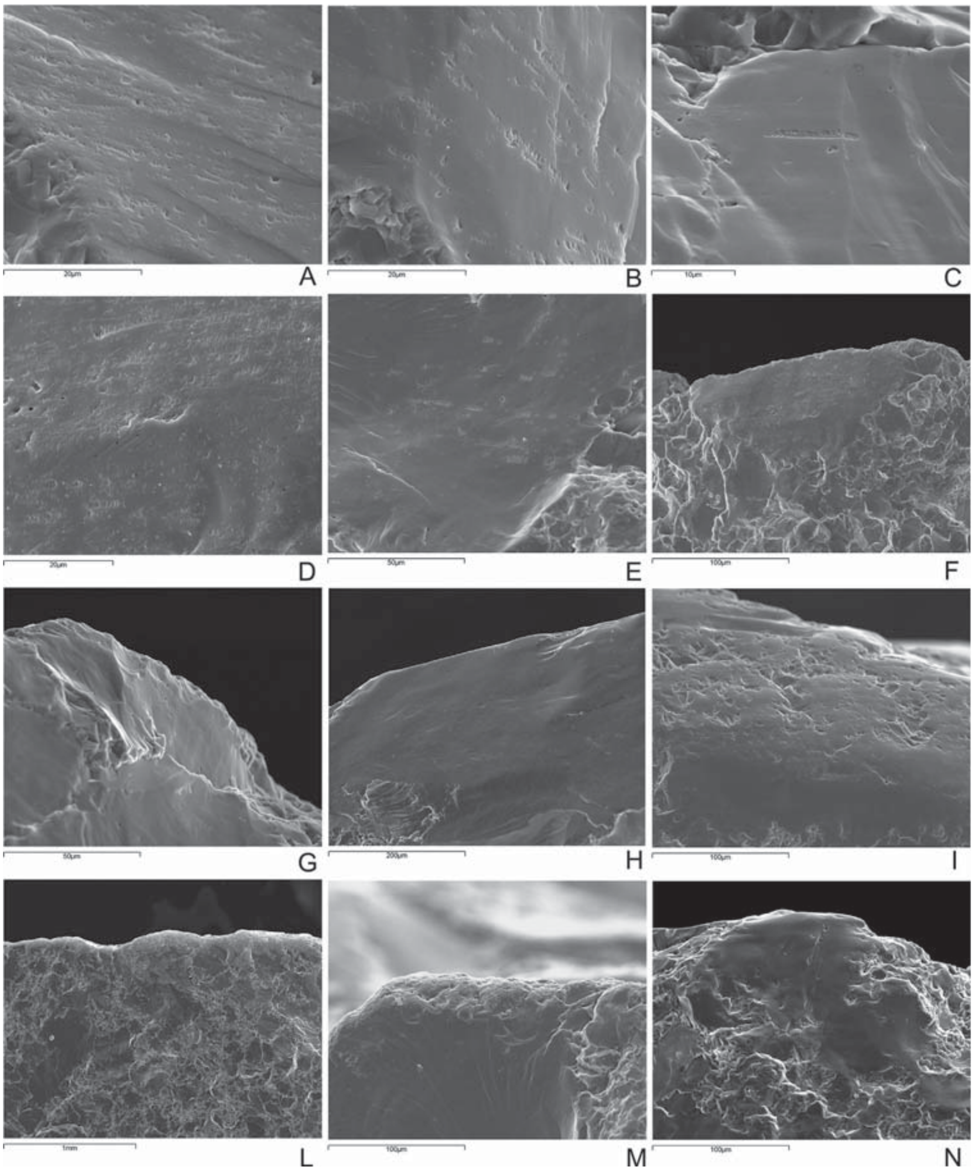

Fig. 11. Main use-wear features observed on Utrillas quartzites formed after processing wood (*Quercus ilex*) (A–G; M), giant cane (*Arundo donax*) (H, I, N) and animal hide (*Cervus elaphus*) (L). Striations occur on the flat surfaces of quartz grains presenting the same morphological traits on V1 (A–C) and V2 (D–F). Micro-scarring is only detectable at high magnifications on the upper border of the quartz grains (V1: G, V2: H). Edge rounding tends to invade the interior part of the edge, extending to the finer quartz granular portion (V1: L), or it is limited only to the extremities of the larger quartz grains (V2: M). Polish on quartzite is not generally distributed over large portions, but depending on several parameters it can smooth entire quartz crystals (V1: I) or only a part of them (V2: N). SEM micrograph magnifications: A, B, D) 2500×; C) 3000×; E, G) 1000×; F,I, M, N) 500×; H) 250×; L) 50×.

they were sometimes indistinguishable from fresh irregular rock surfaces.

Micro-scarring occurred more regularly on the borders of quartz grains, where the rim morphology favours this kind of breakage (Fig. 11: G, H). In fact, depending on how the quartz grains are bonded together and the diversity of the absolute size of the grains (granulometry), they might be entirely detached instead of being medially fractured after the stress caused by use. This phenomenon is also the main cause of the loss of edge portions with use-wear traces on them, which are replaced by fresh portions after

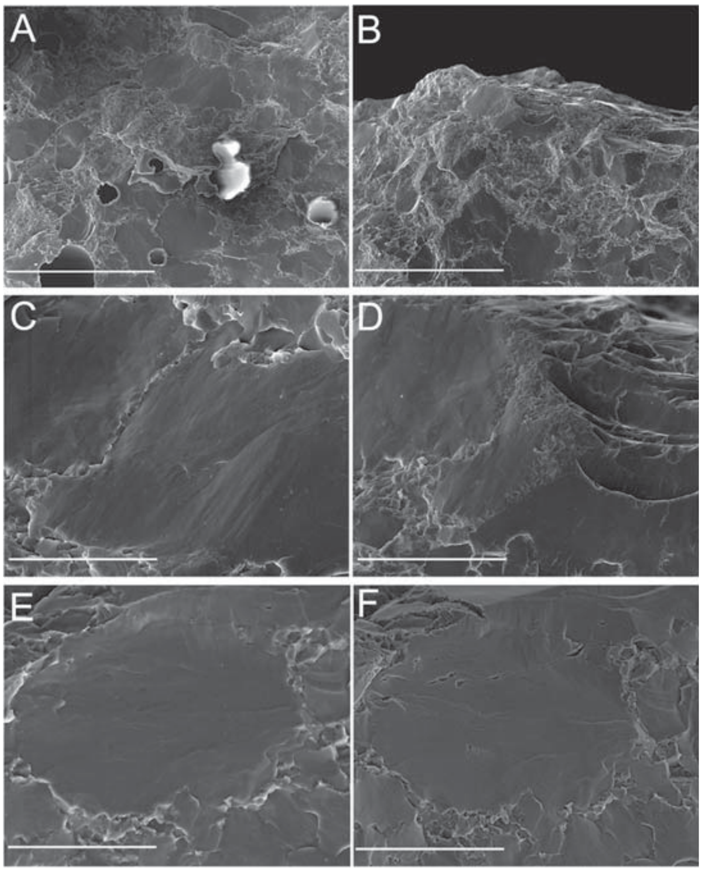

Fig. 12. SEM micrographs of fracturing episodes on edges used to saw a shed antler. Loss of material on V2 is visible through comparison with the edge prior to utilisation (A, B). Holes are flaws in the resin cast. Close-up of the same controlled point, showing the micro-scarring located on the upper extremity of a quartz grain leaving an unusual multilayer substrate appearance (D). Smooth polish later develops on the higher points of the same crystal, exposed by the previously occurring micro-fracture. Initiating micro-fracture in the interior of a V2 quartz grain (F), which is not present on the recently flaked surface (E). This type of fracture was occasionally documented, always prompted by the disposition of the original quartz grains. A, B) mag.: 100×, scale bar: 500 μm; C–F) mag.: 500×, scale bar: 100 μm.

fragmentation episodes (Clemente Conte and Gibaja Bao, 2009; Pedergnana and Ollé, 2014). When the grain bonds are strong enough, only the portions of the quartz grains forming the edge itself undergo micro-scarring (Fig. 11: G, H) and, very rarely, internal micro-cracking (Fig. 12: F). Both micro-scarring (only observable with SEM) and macro-scars are uncommon in both quartzite varieties and no clear differences were noted.

4.3.3. Polish

Polish is a very important use-wear attribute, normally used to differentiate among diverse worked materials (among others Keeley, 1980; Vaughan, 1985; Levi-Sala, 1996). On quartzite, polish distribution responds to different criteria than on flint/chert, so specific polish textures documented on the latter material and traditionally associated with various worked materials are much harder to identify on quartzite artefacts. Since the rock reflectivity hampers the detection of polish brightness using light microscopy, we distinguished two different polish textures, resorting to SEM observations alone. A general distinction can be made between smooth (Fig. 11: I, N; Fig. 13: B, D) and rough (Fig. 10: B, D; Fig. 11: L, M) polishes, although efforts to provide a more detailed classification on the basis of micro-relief features (such as evenness, wavy appearance, convex contours, etc) would probably require some quantitative approach.

Polish is essentially formed by the same process that causes edge rounding. In fact, edge rounding can be defined as a "polished surface", normally with a rough appearance. Abrasive erosion plays a major role in the development of this feature (OCED, 1969), maximising its effects when the edge is used for a long period of time or when it processes hard or very abrasive materials. Obviously, harder worked materials lead to more particles becoming detached and, in turn, more extensive wear. Similarly, the longer a flake is used, the more the particles can interact with the rock surface and the higher the possibility of generating wear. As discussed elsewhere (Ollé and Vergès, 2008, 2014), polish in general involves both fracturing and plastic (compression and translocation) deformations, and the combined effects of these phenomena during the working process lead to worn surfaces on the tool due to attrition and smoothing.

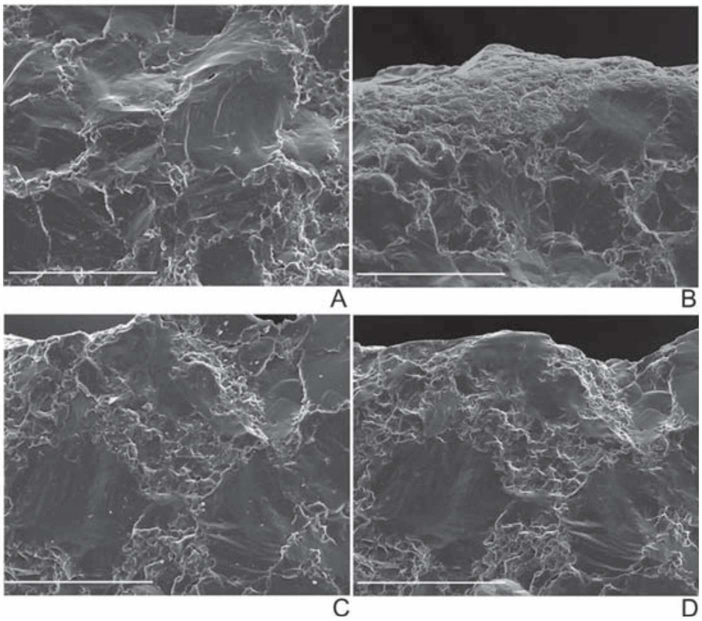

Fig. 13. SEM micrographs of smooth polish occurring after processing a giant cane stalk (*Arundo donax*) for 15'. On a V1 control point, rapid polish development on several crystal surfaces occurred (B). Comparing it with the original surface (A), it is clear that the lower parts of the topography were also levelled. On a second point on a V2 sample (D) some small crystals have visibly weak sutures with the others were extracted (C). After that, a slight polish developed on the upper border of the central larger crystal. Polish may have different distributional patterns on pieces characterised by the same experimental variables, depending on the arrangement of the original crystals. The evaluation of the surface roughness is based on qualitative assessments. A–D) mag.: 250×, scale bar: 200 μm.

As in the case of edge rounding, polish on V1 seems to develop faster and on broader micro-surfaces. It always starts from the highest points, on protruding junctions or on changes in the surface angle of quartz crystals. The rim is usually more affected by polish because it has more contact with the worked material.

4.3.4. Striations

Striations were found on both varieties. They are a very characteristic trait in functional analyses, and usually contribute to determining the directionality of tool use (transversal or longitudinal). They are so important in the sphere of use-wear analysis, that most analysts paid close attention to their recognition and classification (Del Bene, 1979; Keeley, 1980; Mansur, 1982; Mansur-Franchomme, 1983).

The most common striations found on quartzite are categorised as furrow striations (Kamminga, 1979; Fullagar, 2006). No notable differences in the morphology or distribution of the striations were identified between the two varieties.

Striations were found mainly on the relatively flat crystal surfaces (Fig. 11: A, D) and none were identified on the smaller grain fraction. In fact, they cannot develop on extremely irregular planes such as those interlocking the small quartz grains or neoblasts. Any change in the angle topography acts as an obstacle, stopping or changing the trajectory of the rock particle responsible for the formation of the linear feature. Obstacles were seen in different angulated faces of the same crystal (Fig. 11: B), in transitions from a large grain to smaller grains (Fig. 11: F), slopes, holes, protruding grains, crests and valleys. For this reason, striations only form on regular portions of the surface (Fig. 11: A–F), resembling those found on macro-crystalline quartz (Knutsson, 1988; Sussman, 1988).

The differences between V1 and V2 may be associated, again, with the number and size of the detached particles. Evaluation is done in a qualitative way, based on experimental observations. The precise number of striations related to each artefact is not provided because of the enormous presence of micro-striations on some crystals which rendered this task almost impossible. Again, striations are more numerous on V1 than V2, although their distribution is very similar on both varieties due to physical constraints, as discussed above.

5. Discussion

5.1. Petrographic study as a previous step to use-wear analysis

Experimentation has always been an essential step prior to use-wear analyses in order to carefully monitor and document use-wear distribution with regard to specific actions, kinematics and worked materials. The "raw material variable" has not always been regarded as playing an important role in use-wear formation processes.

Quartzite is a coarse rock normally fracturing across their crystals through conchoidal waves and although its chemical composition is mainly comprised of quartz (SiO_2), its structure is quite different from macrocrystalline quartz. Furthermore, a wide range of intermediate forms of quartzite exists, defined by differing metamorphic grades (implying a series of physical differences).

From material sciences, we have borrowed the concept that each material has its own properties (chemical composition and structure) and that those properties greatly influence any physical or chemical process that it is subjected to. Assuming that use-wear formation is basically a physical process, we are conscious of the importance of precisely characterising the rock itself before attempting to analyse the wear traces that may be on it. This is why petrographic studies are so important: they provide an understanding of the mechanical behaviour of the rock when subjected to stress (during experiments or during prehistoric use). Thus, a prior petrographic study is regarded as a fundamental step to describe the intrinsic structural characteristics of the rocks, which may influence use-wear formation and development. If no detailed petrographic description is provided, analysts may face some problems in interpreting the artefacts' functions, underestimating variation in use-wear distribution based on the structure of the rock.

Furthermore, terminological problems may also occur when describing use-wear on quartzite. In some cases, use-wear descriptions are not sufficiently clear because of the adoption of imprecise terms that sometimes depart from general and well-established geological terminology. In order to avoid this in our study, we adopted slightly different terminology than some other authors, who distinguished use-wear occurring on quartz crystals from that found on the matrix (Mansur, 1999; Hroniková et al., 2008; Clemente Conte and Gibaja Bao, 2009; Lemorini et al., 2014). After not having detected any matrix on our specimens (matrix as a sedimentary relict cannot be present on meta-quartzites), we preferred not to employ this nomenclature. It is possible that the distinction between matrix and crystals was drawn on the basis of the bimodal aspect of some quartzites (also observed in our varieties). We noticed the presence of two grain size populations in the two analysed Arenas de Utrillas facies varieties, one larger and one smaller. In V1, this is due to the original granulometry of the protolith, whereas in V2 it is derived from an abundant presence of new-formed crystals (neoblasts). Therefore, some authors may believe they have observed relict matrix in quartzites when what they observed was actually a difference in grain size, and not sedimentary relicts. In any case, if matrix is indeed present on the analysed varieties, we should then refer to them as quartz arenites or orthoquartzites, according to geological definitions (Pettijohn et al., 1987; Tucker, 2001; Blatt et al., 2006).

Finally, terminological confusion might also be due to the type of microscope employed in this kind of study. Generally, light microscopes do not have high enough resolution to examine rock structure by directly analysing flaked surfaces. Problems in documenting use-wear with OLM have also been noticed (Del Bene, 1979; Grace, 1990; Igreja, 2009; Borel et al., 2014). The use of the Nomarski prism might help to better discern some rock types surfaces (Pignat and Plisson, 2000; Igreja, 2009), but regarding quartzite we noticed that this is not always the case. The extreme surface irregularities render some edge portions impossible to be focused with a regular optical microscope, even with the addition of the DIC (Differential Interference Contrast). Conversely, SEM images allow structural differences to be perfectly distinguished, from absolute grain size, grain cohesion and sutures (Welton, 1984). So, in addition to overcoming the limitations of OLM when observing highly reflective rocks, the employment of SEM in use-wear analyses offers clear imaging advantages, such as high magnification power and resolution. Even the tiniest surface modifications (either due to use or to post-depositional processes) can be imaged with this equipment (Tsirk, 1979; Anderson, 1980; Mansur-Franchomme, 1983; Knutsson, 1988; Yamada, 1993; Ollé and Vergès, 2014; Pedergnana and Rosina, 2015).

5.2. Use-wear on Utrillas quartzites

Since we did not apply a methodology suitable for quantifying wear on lithic surfaces, we restricted our research to qualitative features. The analysed quartzite varieties displayed a clear brittle behaviour normally described for quartzose rocks (Knutsson, 1988; Clemente Conte and Gibaja Bao, 2009; Aranda et al., 2014). Knowing that fracturing is a process involving different types of forces (mainly compressive and tensile stresses), the brittle character of quartzite is determined by its reaction to those forces. Quartzite is an anisotropic material, therefore fracture cannot propagate homogeneously. Generally, the grains were not found to be medially fractured, even if occasionally this kind of feature was documented (Fig. 12: F). Fracturing tends to occur around the grains, breaking the weakest bonds between them. The compaction bonds of the grains are either strong enough to withstand compressive stresses or their weakness allows the grains to be entirely extracted. When subjected to high compressive stress (for example sawing a very hard material such as antler), crystals may fracture transversally and open up on several layers (Fig. 12: D).

When quartz crystals are characterised by two different size populations, micro-fracture is even less homogeneous because changes in grain size and morphology cause changes in the material's response to the above-cited stresses. A massive presence of neoblasts, for example, can improve the anisotropic character of quartzites, leading to a macroscopic preferred cleavage as in our V2. Apart from the obvious macroscopic evidence, we demonstrated that its particular structure (derived principally from the metamorphic phase to which it belongs) has a strong influence on wear formation and extension.

Concerning all of the observed use-wear features, V2 displayed a lower degree of wear development in almost all combinations of worked materials and actions. This can be explained by its physical structure. As fracture and wear in general are determined by the geometrical characteristics of both the indenter and the specimen (Lawn and Marshall, 1979), knowledge of the surface structure of rocks makes it possible to predict the way they fracture at a microscopic level as well.

On the other hand, the role of the indenter is a little more variable. Considering only fracture, the indenter is identified through the worked material. Hence, the harder the worked material (bone, antler), the more intense the material loss is. For example, sawing a giant cane stalk does not result in as deep a material loss in relation to the original edge (Fig. 13) as might occur when processing harder materials. With regard to micro-wear itself, we consider the indenters that cause the most diverse wear patterns to be rock particles (Cotterell and Kamminga, 1979; Fedje, 1979; Kamminga, 1979; Lawn and Marshall, 1979). Hence, rock particles detached after fracturing and becoming embedded in the residue of the worked material act like abrasives capable of flattening the flake surfaces. Wear appearance and frequency are therefore dependent upon the size and morphology of the detached particles.

V1 appears to produce more rock fragments and, although smaller, they generate more, and more developed, use-wear than that documented on V2. Apart from a few documented differences, wear distribution responds to a standardised pattern. Striations always occur only on the larger quartz grain surfaces, and do not involve the smaller grain fraction. Conversely, rough polish and, to a lesser extent, smooth polish both give rise to edge rounding and can be associated with both granulometric fractions. Polish always starts on the higher topographic points and it spreads throughout the lower points, depending on the activity performed (Fig. 13: B).

Generally speaking, the extension of wear on quartzite seems to heavily depend on the micro-fracturing behaviour of the rock. More anisotropic materials break more irregularly (V2) but form less wear, while more homogenous varieties (V1) give rise to more regular use-wear patterns.

6. Concluding remarks

The convergence of lithic use-wear and petrographic analyses is an example of the fruitful interaction of different disciplines in order to obtain a more confident interpretation of the archaeological record. The potential and reliability of this combined approach was experimentally evaluated. We developed petrographic characterisations of two quartzite varieties pertaining to the Arenas de Utrillas formation, collected in an outcrop located near the Sierra de Atapuerca (Northern Spain), defining them as metaquartzites with an initial to intermediate metamorphic grade. We compared use-wear patterns connected with the two quartzite varieties and recorded differences between them to improve the accuracy of the use-wear analyses applied to the Atapuerca lithic assemblages. Nevertheless, more research is needed by means of expanding the experimental programme, adding new quartzite varieties (from other known outcrops) and including innovative microscopic techniques in order to quantify use-wear (such as laser scanning confocal microscopy) (Evans and Donahue, 2008; Stemp et al., 2013) or digital image analysis for quantitative analysis of edge modifications (Lerner et al., 2007; Lerner, 2014a, 2014b).

Although the data provided in this paper are limited to the study of the Utrillas varieties, we are conscious that a petrographic characterisation of the entire Atapuerca quartzite corpus would allow us to reach a more confident use-wear interpretation of the archaeological lithic record. Based on our results, we consider a petrographic study to be fundamental prior to any functional analysis.

Adding to the body of knowledge regarding the different types of quartzite (different knapping properties, functional behaviour, etc.) will contribute to the understanding of their differential representation throughout the Atapuerca sequence. The addition of archaeological use-wear information will allow us to assess the degree to which functionality influenced the selection criteria for the different quartzite types. We will then have more data with which to interpret the role of functional needs in the criteria for raw material selection throughout the entire Atapuerca sequence.

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4.2 Moulding and casting

Although the process of moulding and casting of the fresh edges before their utilisation is extensively described within the publications cited throughout the text, a brief introduction is necessary here in order to connect the various sections of the methodology.

Having access to replicas of the original edges is important in order to be able to observe the original aspect of the surface before use throughout the entire experimental process (after each stage of use). In this manner, modifications due to use are distinguishable from those already present before use (lines, pits, ambiguous textural aspects, etc.). Also, the gradual changes of the used edges are always available for comparison with the original surface of the tools.

With this purpose in mind, casts of the edges thought to be used were obtained for all the 46 tools, which were given the same references of the tools to which they correspond. Silicon moulds are obtained by mixing equal parts of the two components, a base and a catalyst (*Provil® novo Light)* and then applied onto the edges (Fig. 4.10: a). The negative moulds are then filled with a rigid polyurethane resin (*Feropur PR55®*) to obtain the positive replicas of the edges (Fig. 4.10: b). The time required for the resin to solidify is very short (3-4 minutes).

Additional details on this cleaning process and more comprehensive descriptions of the different products used are provided in Ollé 2003: 30-40.

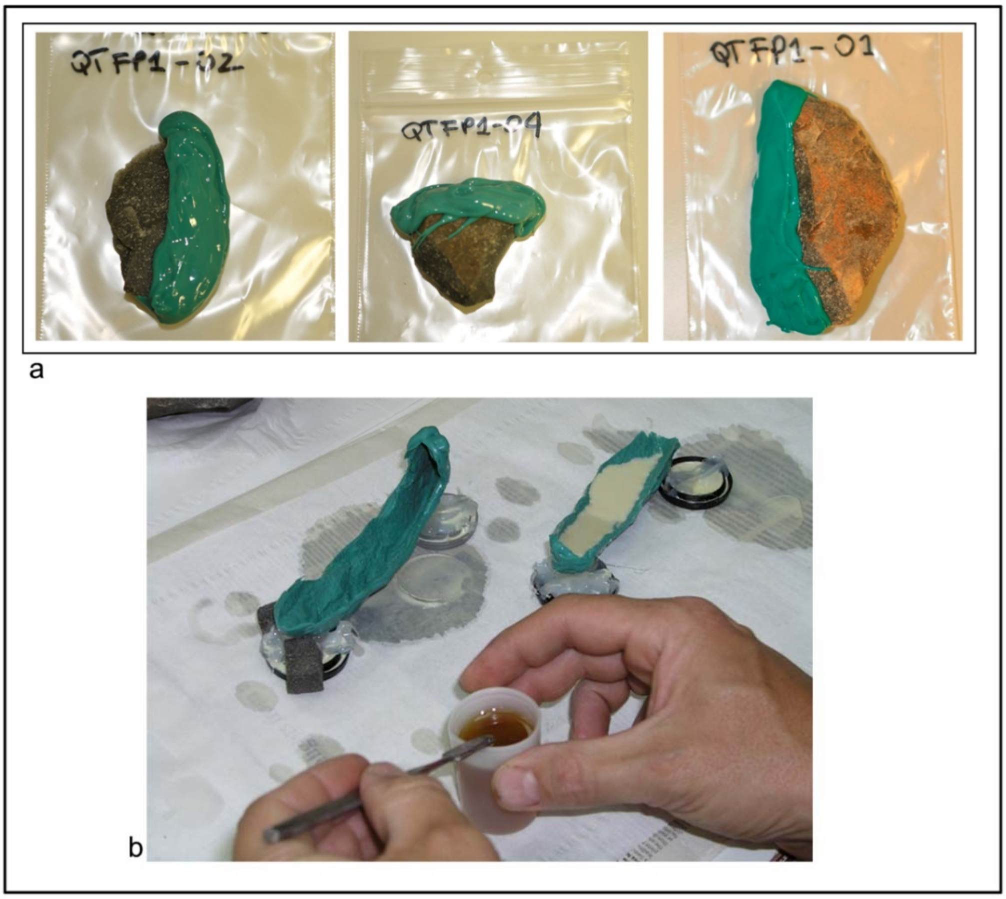


Fig. 4.10: *a) Silicon moulds before their removal from the experimental tools; b) preparation of the resin mixture to be poured into the moulds.*

4.3 Cleaning

We adapted the cleaning process found in Ollé and Vergès (2014). This process was maintained consistent for both experimental and archaeological materials. The only difference is that for archaeological tools presenting high degree of sediment concretion, baths in a 20% Hydrochloric acid solution were employed.
The main steps are as follows:

1. Removal of the ink and varnish used to mark the archaeological tools;

2 15-30min baths in a 10-20% HCl solution (only for archaeological pieces);

3.15min ultrasonic baths in H_2O_2 to remove organic matter;

4.15min ultrasonic baths in a 2% neutral soap solution (Derquim®);

5. Removal of soap residues under running water;

6. 2-5 min ultrasonic baths in pure acetone (only prior to SEM analysis).

Several issues might be raised by debating the best option to clean the samples before microscopic observation, amongst which the most important is that of contamination. This topic will be treated in detail in the 4.6 section of this manuscript, which constitutes the fourth publication presented in this thesis. In fact, one should learn how to discriminate modern contaminants, which might be mistaken for ancient residues or wear related to use (*e.g.* dots, lines).
The specific procedure we selected is mainly devoted to rendering the samples as clean as possible, since SEM is capable of very high magnification and resolution and consequently, to image even tiny particles (such as dust, tissue fibers, skin flakes, etc.). Therefore, the cleaning products to be used before observations need to be carefully selected considering the employed microscopic technique. In fact, the detection of modern residues is strongly correlated with the selected equipment (and obtained magnification). Microscopic particles which are invisible under a stereo-microscope, can be easily detected by a metallographic microscope. Those invisible under this microscope, can be distinguished under a SEM, therefore a stronger cleaning procedure is necessary.
After the SEM analysis (high vacuum only), the samples are dismounted from the microscope steps, the gold layer is removed with an acid mixture containing ¼ concentrated nitric acid (HNO_3) and ¾ hydrochloric acid (HCl). This substance does not damage siliceous rocks, but has to be avoided for carbonate materials (Ollé and Vergès, 2008). The samples are carefully cleaned under running water and then left in a water-only bath for 20-30min to remove all the acid residues. In the final stage they are marked and stored in plastic bags.
For pieces analysed under low-vacuum conditions (SEM-low vacuum), no further cleaning is required, as the samples do not need to be covered by any conductive materials (i.e. carbon,

gold). Only a short bath in acetone is needed to remove the ink landmarks on the surface of the samples (used to locate the wear observed).

4.4 Microscopy

While the functioning of both optical and electron microscopy is introduced in Chapter 2, here we only list the equipment we employed for this study.

For rapid microscopic scanning, a conventional metallographic microscope (Zeiss-AXIO Scope1) was used (Fig. 4.11: a). Images were taken with a 5MP DeltaPix digital camera (Invenio 5SII model) and multi-focused images were usually obtained using the DeltaPix Insight and the Helicon Focus software. Since it has been suggested that this is not the most adequate technique for coarse and reflective raw materials (Grace, 1989, 1990), most of the documentation has been collected using a Scanning Electron Microscope (Fig. 4.10: c (3), d).

Although the use of the Differential Interference Contrast (DIC) and the Nomarski Interference Contrast (NIC) (Heath, 2005) can be very useful to avoid light reflection of flat reflective materials such as quartz (Igreja, 2008, Knutsson et al., 2015), it did not produce similar results when observing quartzite (Ollé et al., 2016b; Pedergnana and Ollé, 2017a).

The observations were carried out mainly with a high-vacuum mode (JEOL JSM-6400), which implied covering the samples with a thin layer of gold (30mA) through the use of a Sputter coater (Fig. 4.11: c, 1-2). An ESEM FEI Quanta 600 was used when the samples were not metallised (Fig. 4.11: d). In fact, this equipment works at a low vacuum mode as well, which does not require the samples to be covered with a conductive material (*e.g.* gold, carbon). The observations of the experimental material were done almost entirely with high-vacuum conditions (metallizing the samples), while the archaeological material was most frequently analysed under low-vacuum conditions. The mean reason for this was that SEM-low-vacuum images, although presenting less resolution than those taken under high vacuum conditions, were clear enough to show use-wear on quartzite. At the same time, not covering the archaeological samples with anything, guarantees a better preservation of the lithics, avoiding the use of aggressive acid mixtures to remove them. In addition, this procedure was also far less time-consuming than using a high-vacuum-SEM (avoiding the metallization process and the removal of the thin gold layer afterwards. The samples were normally observed with a working distance of 10-15 mm and at a 15-20 kV.

Both OLM and SEM provide qualitative data only. We aimed at describing use-wear on quartzite based on the comparison of images taken during OLM and SEM observations (Borel et al., 2014). However, to provide quantitative data, a Laser Scanning Confocal Microscope (Olympus-LEXT 3100) (Fig. 4.11: b) was used on an experimental sample (6 pieces) made of a quartzite variety other than those described in this work. Details about this variety (VSH-4) and related results are found as supplementary material (Annex 1). Although this specific variety analysed is different, it is also a metaquartzite and comes from the same region (Northern Spain) as three of the varieties included in this study. Therefore, results are pertinent as they constitute the only quantitative data provided in this study and will be used to articulate the final discussion of this thesis.

A digital microscope (3D Hirox KH-8700) was occasionally used to image particular details (such as residues), or when it was not possible to use SEMs (because of technical problems).

The details of the lenses of the digital microscope are:

1. Low Range High Resolution Zoom Lens, MXG-2016Z. 20x to 160x;
2. Dual Illumination Revolver Zoom Lens, MXG-5000REZ; with a Low-range objective (35x to 250x), a Mid-range objective (140x to 1000x) and a High-range objective (700x to 5000x).

It is also coupled with multiple lighting modes (Co-axial, ring light, mixed).

Stereo-microscopes have also been sporadically used during the pre-selection of the material (at least in the case of the Payre material).

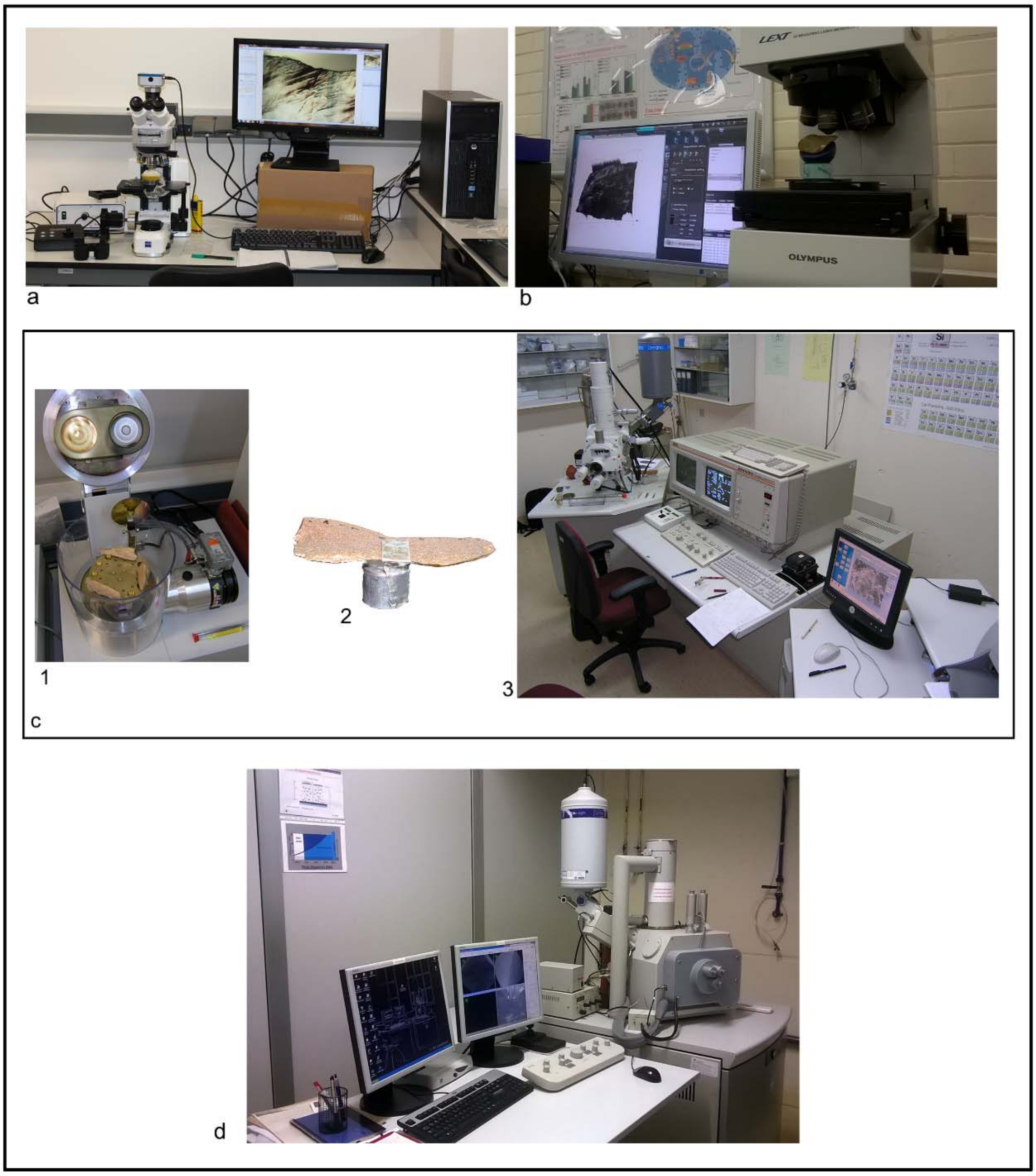


Fig. 4.11: *a) Zeiss AXIO Scope1 at the Lithic Laboratory of the IPHES; b) Olympus-LEXT 3100 at the Engineering Department of the University of Bradford (UK); c) Preparation of the sample before SEM analysis: 1) Gold layer depositing in a sputter coater (EMITHEC K575X); 2) Sample mounted on a metal stub; 3) Scanning Electron Microscope (JEOL JSM-6400); d) Scanning Electron Microscope ESEM FEI Quanta 600. Both SEMs were made available by the Servei de Recursos Científics i Tècnics of the University of Tarragona (URV). (Image a: courtesy of J.L. Fernández-Marchena).*

4.5 Use-wear recording

All of the use-wear traits observed were plotted on sketches or photographs of the analysed tools. Data was then elaborated a second time and digital forms were filled in. An example of the empty form is provided in Table 4.4. On this form, the information of the experiments connected to the experimental tool is summarised and an image of the tool is provided with the used edge indicated by a circle. Finally, any development of use-wear is briefly described. The 46 forms pertaining to the present thesis have been made available in digital format (Annex 3).

A different form was used for the analysis of the archaeological tools (Table 4.5). On this form, we provide the possibility to show the location and intensity of any observed surface modifications. Use-wear, post-depositional modifications, and fresh parts of the tool are recorded. Tools are divided into 10 different segments, allowing to specifically localise the detailed descriptions of surface modifications (Fig. 4.12) (based on Lombard, 2008:29, Fig.3). Additionally, the basic characteristics of the retouch and the possible attribution to a techno-type are also included (previously identified during the techno-functional analysis). Schematic descriptions of the portions of the used edges (angle, linear and sagittal views, morpho-type) are made available and these are helpful for a rapid data consultation. The archaeological forms of all the analysed tools, provided in digital format only, are grouped in Annex 4 of this thesis.

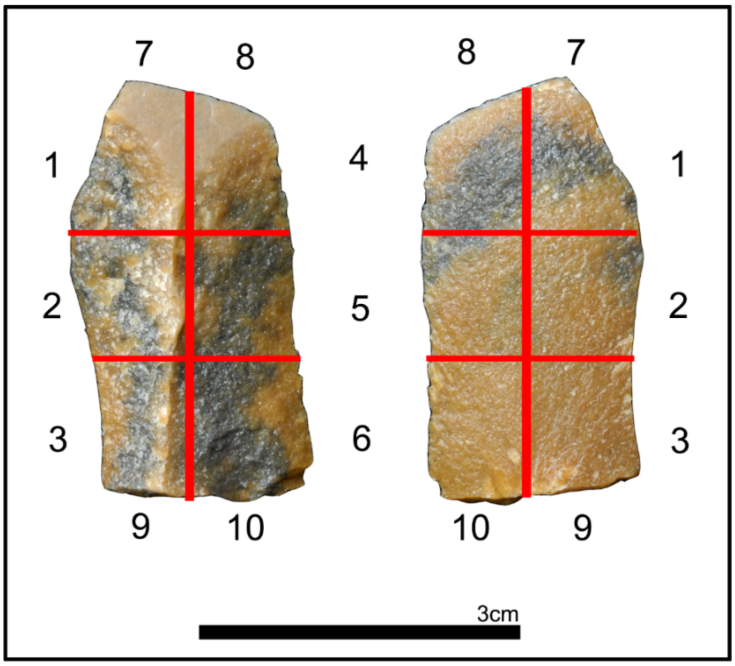


Fig. 4.12: *Schematic illustration of the virtual division of tools into 10 different segments. The recording of use-wear on archaeological tools follows this scheme.*

USE-WEAR ANALYSIS OF EXPERIMENTAL TOOLS

Artefact reference:

Experiment reference:

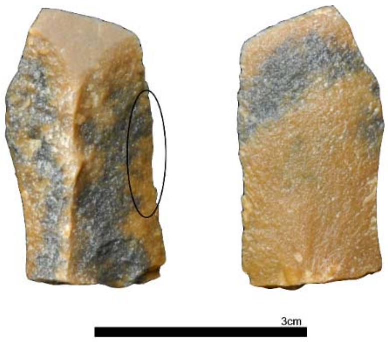
Action:

Movement:

Worked material:

Approximate number of strokes:

Used part: indicated by a circle



Edge damage:

Striations:

Grain edge rounding and scarring:

Polish (and edge rounding):

Table 4.4: *Example of use-wear recording form for experimental tools.*

USE-WEAR ANALYSIS OF ARCHAEOLOGICAL TOOLS				
Reference number:	**Raw material:**	**Granulometry:**	**Technological category:**	**Laplace type:**
Techno-type:	**Analysed faces:**	**Applied analyses:**	**Analyst:**	**Date:**

PHOTOGRAPH OF THE ARCHAEOLOGICAL TOOL

USE-WEAR RECORDING

PORTIONS / **USE-WEAR TYPE**	**Portion 1**	**Portion 2**	**Portion 3**	**Portion 4**	**Portion 5**	**Portion 6**	**Portions 7-8 9-10**
Edge damage							
Edge Rounding Development							
Grain edge Rounding develop.							
Striae on quartz crystals							
Striae types							
Striae direction							
Polish							
Polish texture							
Undulations							
Pits							
Fresh part:							
PDSM:							

USED SIDE PORTIONS

PORTION 1	**Horizontal delineation:** linear/convex/ concave / denticulate	**Sagittal delineation:** linear/curve/sinusoidal	**Edge angle:**	**Morpho-type:**
PORTION 2	**Horizontal delineation:** linear/convex/ concave / denticulate	**Sagittal delineation:** linear/curve/sinusoidal	**Edge angle:**	**Morpho-type:**

INTERPRETATION

PARAMETER	**RESULT**	**DEGREE OF RELIABILITY (0-5)**
Hardness of the worked material	soft/hard/very hard	
Worked material type:		
Performed action:		
Movement:	transverse/longitudinal Unidirectional/bidirectional	
PDSM:		
OBSERVATIONS:		

Table 4.5: *Example of use-wear recording form for archaeological samples (reduced size compared to the original). The wear attributes are localised with an X on the portions of the tool where they have been observed. Intensity is expressed by the number of x (x= low; xx= medium; xxx= high). The division of the tool into 10 portions or segments is based in the Figure 4.11.*

4.6 Publication 4:

Pedergnana, A., Asryan, L., Fernández-Marchena, J.L., Ollé, A., 2016. **Modern contaminants affecting microscopic residue analysis on stone tools: A word of caution.** *Micron* 86, 1-21.

This publication discusses problems relating to the contamination of stone tools when they are analysed under a microscope. Mistaken identification of ancient residues or wear because of contamination can falsify the results of both use-wear and residues analyses of archaeological material. Because of that, it is important that analysts become familiar with modern contaminants that may be present as residues on lithic tools. This will permit them to eliminate the 'background noise' related to them and, as a consequence, to provide more reliable results.

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Modern contaminants affecting microscopic residue analysis on stone tools: A word of caution

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ABSTRACT

Residue analysis is a method frequently used to infer the function of stone tools and it is very often applied in combination with use-wear analysis. Beyond its undeniable potential, the method itself has several intrinsic constraints. Apart from the exceptional circumstances necessary for residues to survive, the correct identification of the residue type is a very debatable topic. Before attempting to recognise ancient residues, a proper method should allow analysts to identify possible modern contaminants and exclude them from the final interpretation. Therefore, analysts should not underestimate the presence of modern contaminants and might learn how to discriminate the background noise due to handling.

The main aim of this research is to provide some methodological improvements to residue analysis through the characterisation of some modern residues often present on the surface of stone tools (e.g. skin flakes, modelling clay). This characterisation was done by using both optical light microscopy (OLM) and scanning electron microscopy (SEM).

Finally, a special care in the post-excavation treatment of stone tools is claimed in order to avoid major contamination of the samples.

1. Introduction

Residue analysis has been used with improving frequencies within lithic studies to gain more information about the function of ancient stone tools. Very often this method has been applied in combination with use-wear analysis to improve the accuracy of the functional interpretation (Högber et al., 2009; Rots and Williamson, 2004; Rots et al., 2015). In spite of the great progress of residue analysis, a number of methodological problems have been detected by analysts especially connected to the identification of the residue type (Langejans, 2010). In fact, out of all the artefacts composing the archaeological record, the micro-residues possibly present on the lithic surfaces suffered taphonomic alterations to a variable extent. Decay and post-depositional processes inevitably modify the original morphology of fresh micro-residues. Colour, one of the most frequently adopted parameters in residue identification, is also subjected to relatively extensive changes.

As for use-wear analysis, optical light microscopy (OLM) has been the most adopted technique to analyse residues (among others, Hardy et al., 2001, 2013; Hardy and Moncel, 2011; Lombard, 2008, 2011; Lombard and Wadley, 2007; Rots et al., 2015). In those studies, the identification of the residue type was generally done through the comparison of the visual appearance of the residues found on the archaeological artefacts with those experimentally generated.

In other words, the assertion of the capability of recognising residues is based on the observed morphological and colouring traits, exactly what seems to change most after being buried (Langejans, 2010). Indeed, those studies do not properly consider the occurrence of taphonomic processes which altered the aspect of micro-residues.

On the other hand, some recent studies seem to be much concerned with the effort to provide a more secure interpretation of residues and adopted multi-analytical approaches including chemical analyses of the substances found on stone tools or on

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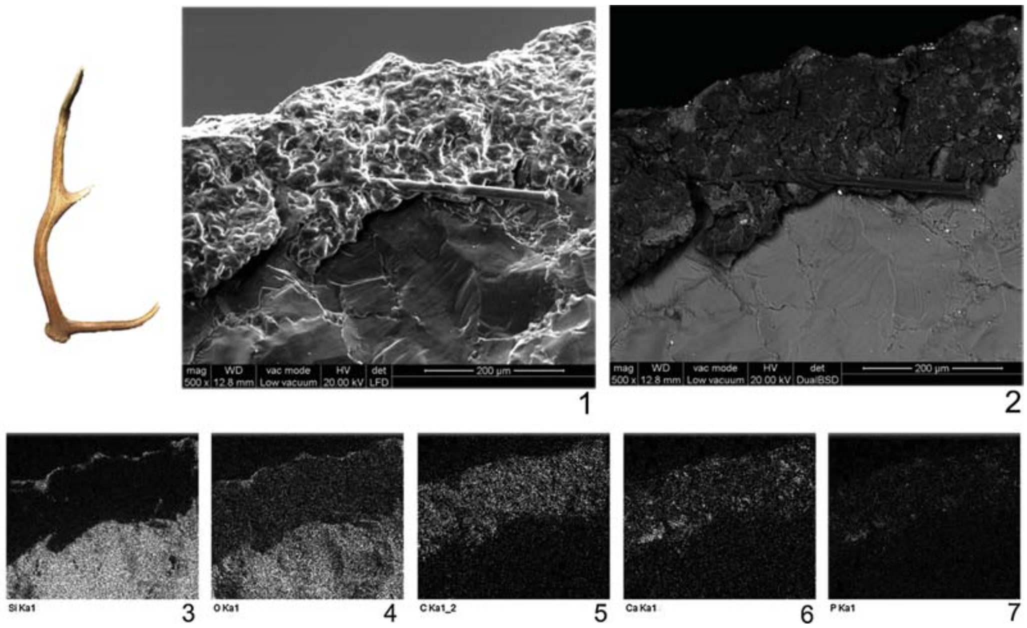

Fig. 1. An example of our experimental comparative collection showing an antler residue imaged by the SEM-secondary electron detector (1) and the SEM-backscattered electron one (2) (orig. mag.: 500×). 3–7 Mapping of the same residue showing the elements detected through EDX and connected with the residue (3 = Si; 4 = O; 5 = C; 6 = Ca; 7 = P).

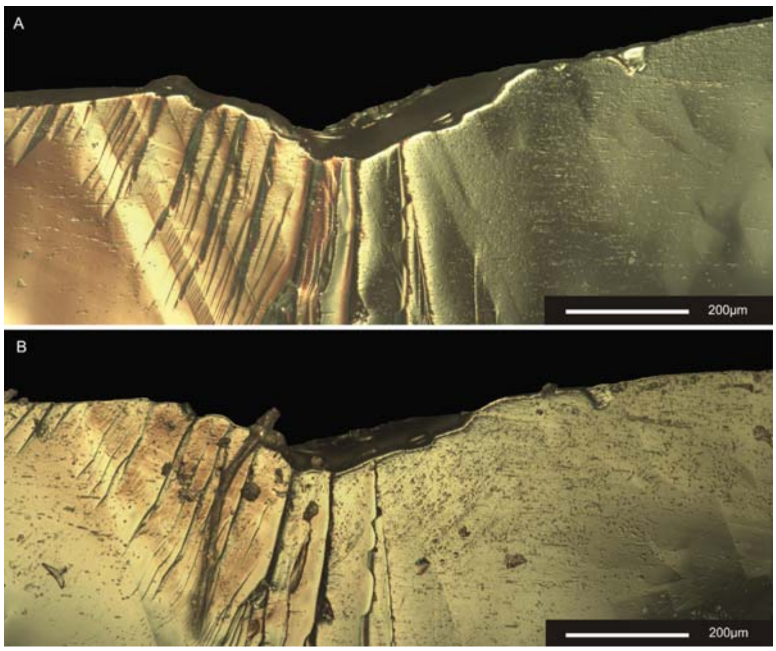

Fig. 2. An experimental rock crystal surface bearing use-wear traits before (A) and after hand contact (B). Skin flakes are randomly distributed on the edge and on the interior of the piece and a linear residue with the same texture of skin flakes is also present. A greasy layer in the centre of the image in the form of linear oblique lines covers the real striations parallel to the edge. We see how this could also interfere with the use-wear analysis. Photo stitching of 7 extended focus images (orig. mag.: 200×).

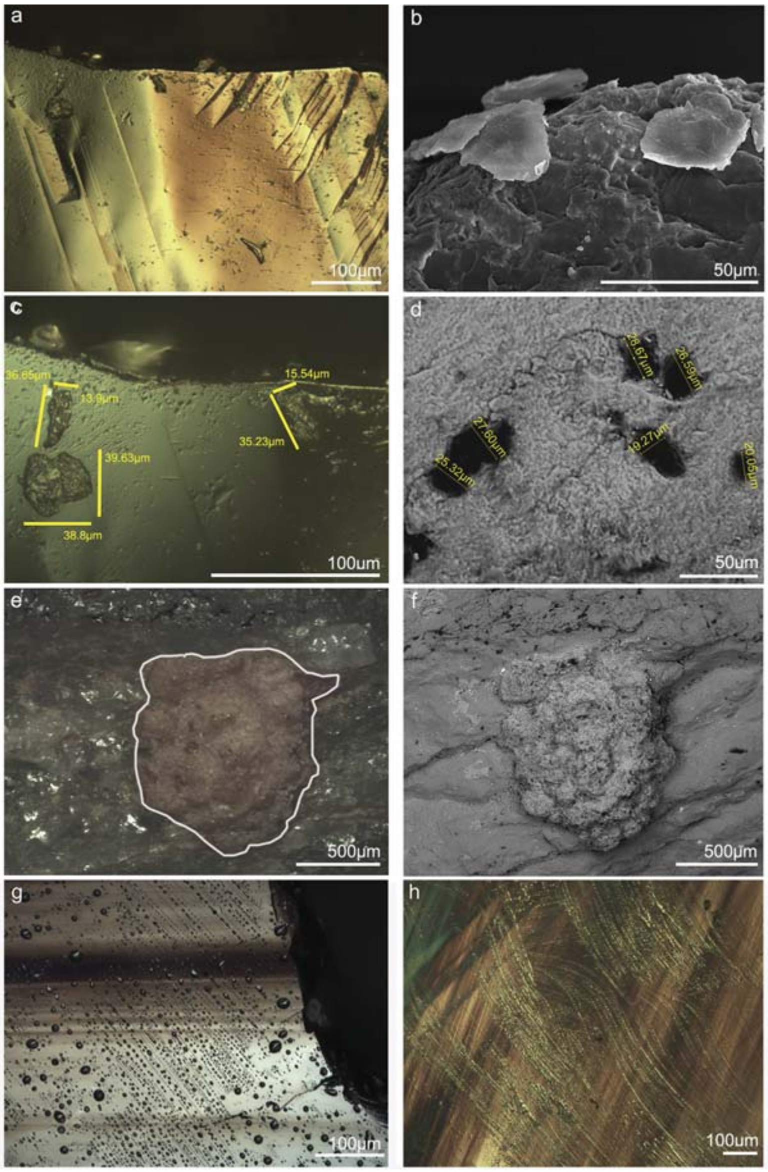

Fig. 3. Handling residues on different raw materials. Skin flakes imaged with OLM (a, c) appear as circular whitish transparent particles, while in SEM micrographs they are dark when using backscattered electron detectors (d) and light grey exhibiting topographical details with secondary electron detectors (b). Sometimes skin flakes are not visible under the OLM: all over the sediment concretion found on archaeological pieces (surrounded in white) (e), skin flakes do not appear, while under the SEM-backscattered electron detector they are in the form of small black particles (f). Grease on rock crystal in the form of circular spots (g) or lines (h). a, g) orig. mag.: 200×; b) orig. mag.: 1000×; c) orig. mag.: 500×; d) orig. mag.: 1250×; e) orig. mag.: 50×; f) orig. mag.: 135×; h) orig. mag.: 100×.

other artefacts (Cârciumaru et al., 2012; Charrié-Duhaut et al., 2013; Cristiani et al., 2009, 2014; Dinnis et al., 2009; Hauck et al., 2013; Helwig et al., 2014; Jahren et al., 1997; Monnier et al., 2013; Pawlik, 2004; Pawlik and Thissen, 2011; Prinsloo et al., 2014).

Since all substances are characterised by specific chemical composition, the application of methods to chemically characterise each residue type is an unavoidable step to reach a more faithful interpretation. Although analysts should be able to identify the presence of residues on stone tools under the optical microscope, lithic artefacts might be further subjected to other types of analysis for a better characterisation of the residues. For instance, Monnier et al. (2012) demonstrated that it is possible to improve the identification of residues by using scanning electron microscopy (SEM).

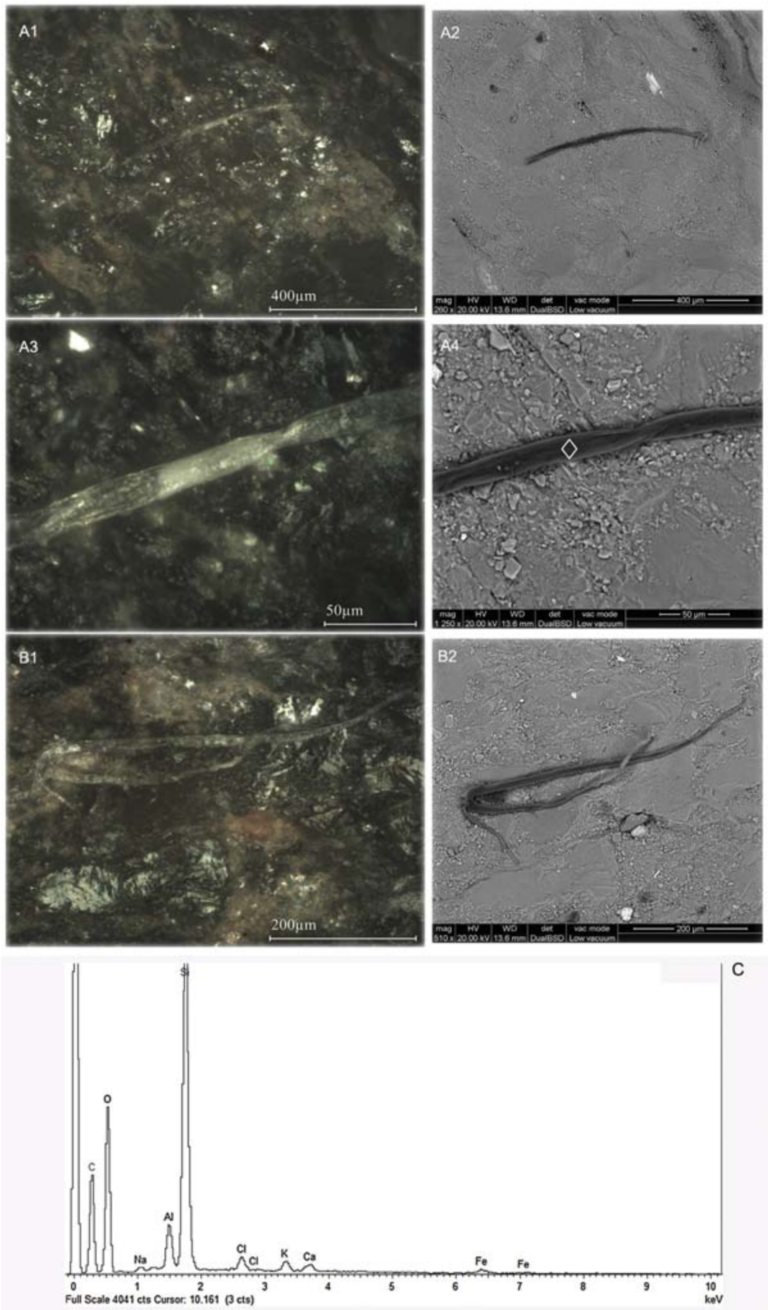

Fig. 4. Comparison of the appearance of two vegetal fibres probably originating from the laboratory paper used in the drying process of the artefacts by using OLM (A1, A3, B1) and SEM (A2, A4, B2). A3-4 are close-ups of A1-2, showing that the fibre is twisted. A1) orig. mag.: 100×; A2: orig. mag.: 260×; A3) orig. mag.: 500×; A4) orig. mag.: 1250; B1) orig. mag.: 200×; B2) orig. mag.: 510×. C) SEM-EDX analysis performed on the spot signalised by the white rhomb in A4, shows the presence of large quantities of Carbon (C) and Oxygen (O), and less Chlorine (Cl), Sodium (Na), Potassium (K) and Calcium (Ca) contents. Silicon (Si), Aluminium (Al) and traces of iron (Fe) correspond to the rock substrate, which is quartzite.

In fact, the comparison between images taken by OLM and SEM proved to be very useful to overcome the problem of identifying ambiguous experimental residues. Furthermore, SEMs are normally equipped with EDX detectors, which allow the elemental composition of the sample to be obtained.

Certainly, it is fundamental to construct an experimental comparative collection before analysing ancient residues on stone tools. Our photographic reference collection was constructed by collecting both OLM and SEM images as well as the elemental compositional analysis for each contact material (Fig. 1).

Beside a strong and suitable methodological approach, which permits the recognition of the residue type, a realistic interpretation should be also concerned with the capability of distinguishing between recent and ancient residues. Therefore, the first step in residue analysis is the exact recognition of the residue type; however, it is not sufficient to reach a correct interpretation, since the observed residues might be recent. For instance, a correct identification of hair fibres, pollen particles or phytoliths would not necessarily correspond to an ancient chronology and might be the result of modern accumulation (from the surrounding environment). Hence, the differentiation between ancient and modern residues would be a theoretical second step in residue analysis. A third step would be the capacity of describing the relationship of the supposed ancient residue with the lithic tool on which it is found (Barton et al., 1998; Langejans, 2011). This is, perhaps, the most challenging part of the process of reconstructing the stone tools' function through the analysis of micro-residues.

In fact, the capability to correctly identify micro-residues and to evaluate their ancient chronologies does not assure a correct interpretation of the relationship with the performed action. For example, the presence of blood or animal muscular fibres on a lithic artefact does not necessarily imply that it was used to butcher a carcass. It might have laid on the ground next to the place where the activity took place, had contact with the organic substances and trapped some particles on the surface, which eventually survived the burial processes. For all of those reasons, residue analysts should be cautious with their interpretations and always consider the impact of post-depositional processes.

This study focused on the recognition of modern residues or contaminants, which would be the theoretical second step of archaeological residue analysis. The handling of archaeological and experimental artefacts was considered to be one of the principal sources of modern contamination, potentially interfering with residue analysis. We think that before attempting to analyse ancient residues, one should be able to correctly identify residues due to handling, which are almost certainly present on all the archaeological artefacts. In fact, lithic artefacts are always subjected to a number of post-excavation treatments: cleaning, typological and technological analyses, drawing, refitting studies, microscopic observations, etc. Artefacts are then continuously handled, and suitable gloves are not contemplated in most of the laboratories.

In this framework, a rigorous documentation of some of the most frequently observed modern contaminants was undertaken, in particular small particles of human skin, modelling clay, drawing pencil and other particles coming from the surrounding environment (e.g. pollen spores, vegetal fibres).

In addition, in order to clearly characterise the dry (human) skin particles on the lithic artefacts, an experiment consisting of intensive handling of experimental flakes made of different raw materials (flint, basalt, quartzite and obsidian) was set up. A secondary objective was to understand the accumulation patterns of this residue on different types of rock and to be able to compare them with those seen on the archaeological artefacts. Modelling clay, which is frequently used in lithic laboratories all over the world, was also documented on the tools' surfaces.

Subsequently, different cleaning procedures were applied to the experimental tools in order to evaluate how aggressive they need to be in order to remove those modern contaminants. It emerged that some modern residues are very difficult to remove, therefore archaeologists should be concerned with the application of new handling protocols to the lithic assemblages.

In our opinion, the most feasible solution to avoid major modern residue contamination would be the selection in the field of some pieces to be microscopically analysed, collecting them using powder-free sterile gloves and directly putting them in clean zipped plastic bags. These tools should remain untouched until residue analysis is performed. Afterwards, other analyses (such as technological analysis, drawing and refitting analysis) could be applied.

2. Materials and methods

2.1. Microscopic documentation of residues

Both OLM and SEM were used to document modern residues directly on the surface of stone tools. Comparison of the same residues seen by means of different microscopes was also undertaken in order to emphasise similarities and differences of the residue appearance (Borel et al., 2014; Monnier et al., 2012).

A metallographic microscope (Zeiss Axio Scope-A1) with magnifications ranging from 50 to 500 times was used. Pictures were taken with a 5MP DeltaPix-digital camera (Invenio 5SII model) and extended focus images were obtained using the DeltaPix Insight software. An ESEM FEI Quanta 600 equipped with an Oxford Instruments INCA system for digital image acquisition and treatment was also employed. It was always used at low vacuum mode, which does not require the coating of the specimens with conductive materials (e.g. gold, carbon). Both secondary and backscattered electron detectors were used to image modern residues. The secondary electron detector (Large Field Detector, LFD) is useful to observe topographic and textural traits of the residues, while the backscattered electron detector (DualBSD) provides grey-scale images according to atomic number contrast, with brighter regions being generated from areas of higher average atomic number. For this reason, it is extremely useful to detect organic components (darker tones) on stone tools. Also, backscattered electrons are less affected by electric charge and are more suitable for imaging samples with thin insulating layers on their surfaces, which would interfere with LFD images.

Additionally, the SEM microanalysis system (energy-dispersive x-ray spectroscopy, EDX or EDS) was used to analyse the elemental composition of the residues (e.g. Byrne et al., 2006; Monnier et al., 2012, 2013; Pedergnana and Blasco, 2016; Pedergnana and Ollé, 2014; Vergès and Ollé, 2011).

2.2. Some common contaminants in residue analysis

Stone artefacts, along with all the objects composing the archaeological record, suffer post-depositional processes when they are

Table 1
Experimental flakes used to document modern human skin flakes.

Reference	Raw material	Provenance
SNF-SR-01	Flint	Norfolk (UK)
SN-SR-1	Flint	Northern Spain (Burgos)
Ish-bas-1-SR-1	Basalt	Nagorno Karabagh (Southern Caucasus)
QTFU1-SR-1	Quartzite	Northern Spain (Burgos)
HRZD-0bs-1-SR-1	Obsidian	Central Armenia (Caucasus)

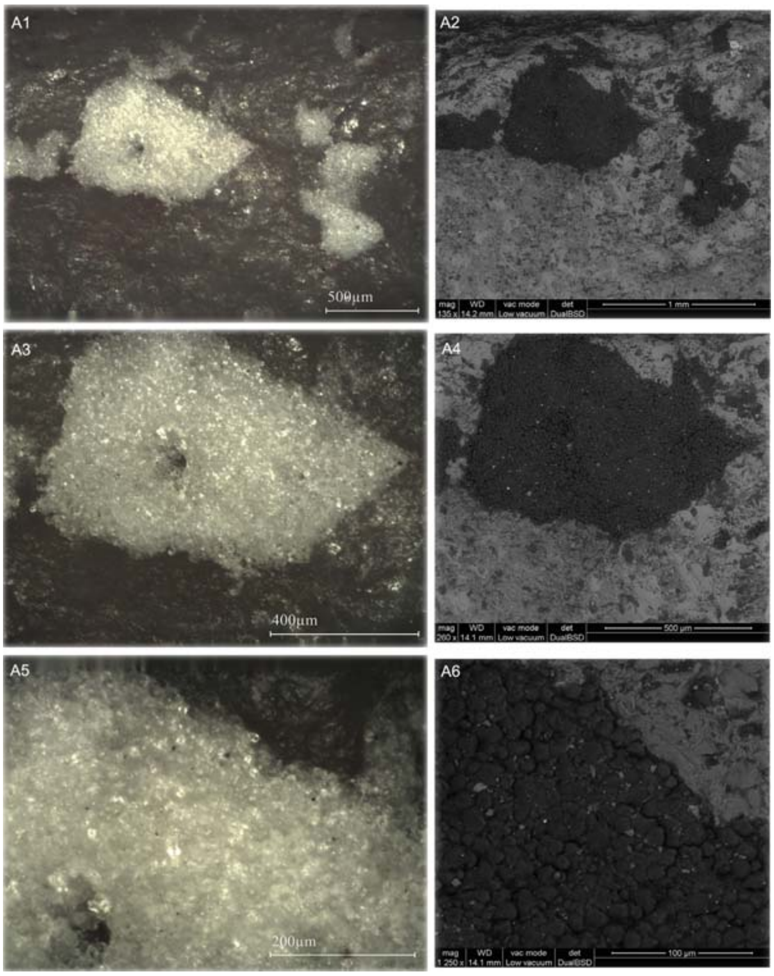

Fig. 5. Comparison of the appearance of modelling clay residue on a quartzite archaeological artefact by using OLM (A1-3-5) and SEM (A2-4-6). The residue is imaged at different magnifications to show details. A1) orig. mag.:50×; A2) orig. mag.: 135×; A3) orig. mag.: 100×; A4) orig. mag.: 260×; A5) orig. mag.: 500×; A6) orig. mag.: 1250×.

buried. From the moment they are collected from excavations, a new life begins. Specialists continuously handle the artefacts for cleaning, drawing and for further analyses. This simple and very general habit resulted in the generation of one of the most numerous and widespread modern residues detected when tools are analysed at high magnifications (e.g. Unrath et al., 1986) (Fig. 2).

While the manipulation affects the whole surface of the artefacts, a careless drawing procedure particularly affects stone tools by leaving pencil marks all over the edges. The modelling clay used for different purposes (e.g. refitting studies, fixing the tools while taking photographs, fixing the samples to the microscopic holders) can also be an important modern contaminant.

Furthermore, contaminants from the surrounding environment such as dust or pollen, as well as substances related to the direct manipulation in laboratories (e.g. starch grains of some medical powdered gloves, paper or clothing fibres, etc.) might deposit on the tools' surfaces (Langejans, 2011; Wadley and Lombard, 2007).

2.2.1. Skin flakes

One of the most common contaminants observed when lithic artefacts (both archaeological and experimental) are microscopically analysed are the small particles of human skin, resulting from their handling during and after excavation, experimentations or posterior studies. These small particles of human skin are often referred to as 'skin flakes' in the medical dictionaries (e.g. Rothenberg and Chapman, 2006) and are described to be the result of the drying of the skin outermost layer (*epidermis*). Following this description, the term 'skin flakes' will be used in this study to refer to those particles.

Skin flakes are usually small (25–40 μm), exhibiting oval or rhomboidal shapes (Fig. 3c and d). The appearance of these particles under the OLM is usually greasy, translucent and of whitish colour (Figs. 2 and 3a and c). If no other microscopes or methods of analysis are used, these particles can be very easily confused with other residues or they can even resemble the original rock structure (they are similar to crystals). It is easier

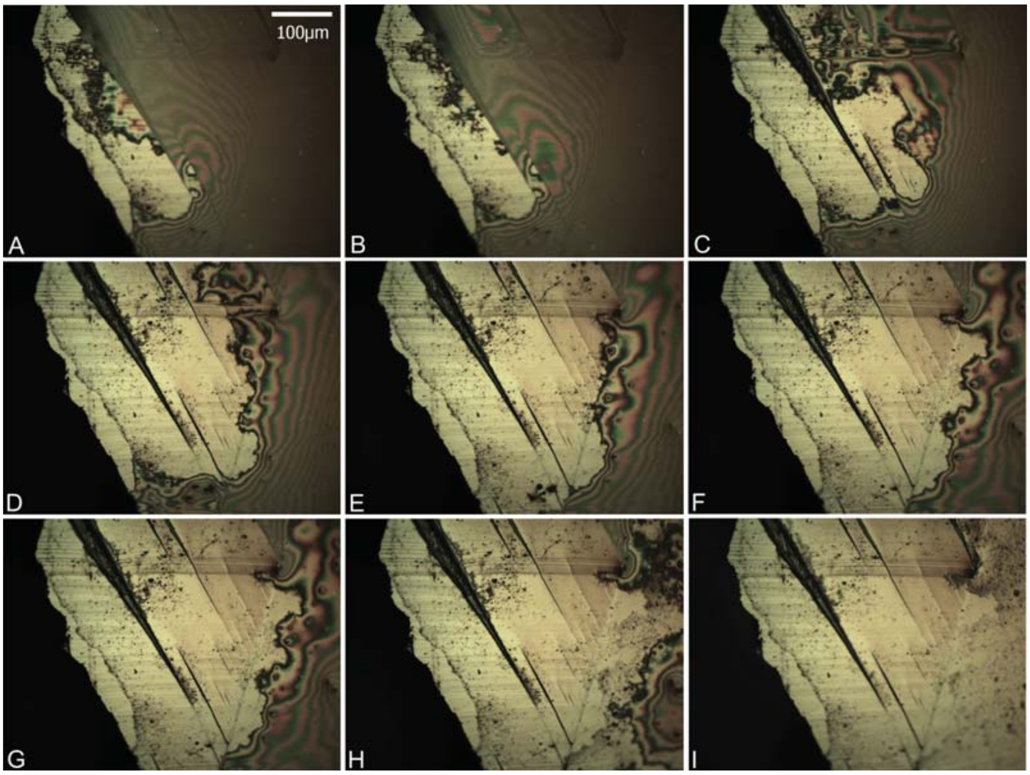

Fig. 6. The evaporating process of pure acetone used for cleaning purposes. This experimental rock crystal flake was cleaned with acetone and was analysed with the optical microscope after ca. 20 min. Apparently, some liquid spots were still present and the drying process was accelerated by the incident light of the microscope. Sometimes, this results in dirty lines or circular spots on the surface (G, H, I). Orig. mag.: 200×.

to distinguish these particles under the SEM particularly using the backscattered detector, where they appear dull and dark in colour (Fig. 3c and f). From secondary electron detector images, the morphology and texture of skin flakes are better observable (Fig. 3b).

On flatter surfaces (e.g. rock crystal), on cleaned specimens ready for analysis, greasy lines might appear after accidental manipulation (Fig. 3g and h) (Fernández-Marchena and Ollé, 2016). The hand grease resulted to be more evident on flat specimens; in fact, it was not documented on coarser raw materials.

2.2.2. Other possible contaminants

Laboratory conditions may also introduce modern contaminants in the form of fibres coming from clothing or from the laboratory paper towels used to dry specimens after cleaning (Fig. 4).

On coarse raw materials (such as quartzite or basalt), the detection of those fibres is easier with the SEM-backscattered electron detector thanks to the colour contrast of different elements (Fig. 4A2-4, B2). Fibres are found very frequently on the surfaces of stone tools and sometimes they appear to be twisted or bent (Fig. 4A3-4, B1-2), which is a feature commonly reported for ancient plant and animal fibres. Elemental microanalysis showed the presence of Carbon (C), Oxygen (O) and Chlorine (Cl) (Fig. 4C).

Another extremely contaminant material turned out to be the modelling clay used in some steps of lithic analysis, such as in the photographic documentation, refitting studies or use-wear analysis. Indeed, this substance is sometimes used to attach the analysed tools to the microscope specimen holder or to special supports (Pawlik, 1993; Unrath et al., 1986). Agglomerates of modelling clay are microscopically visible on the lithic surfaces due to its distinct colour (Fig. 5). Under the secondary-electron detector of the SEM it appears darker than the rock substrate, whereas with the OLM it is white (the colour obviously depends on the variety employed) and birefringent at high magnifications (Fig. 5A3). The granular textural appearance is more visible in the SEM micrographs (Fig. 5A6).

Water or acetone, normally used during the cleaning procedures, can generate unclear spots on flat surfaces (e.g. rock crystal), once dried. Darker shadows, which resemble pits, are also documented (Fig. 6). Although this was not recorded on all types of rock, we found the rigorous documentation of these spots to be important in order to avoid misleading interpretations during use-wear analysis.

Additionally, we noticed that also the pencils used for drawing the lithic artefacts can be the source of surface contamination. Not all the artefacts are properly cleaned from the graphite or other residues that can remain on the edges after a careless drawing. For this reason, residues of this activity can be found sometimes on the archaeological tools when they are microscopically scanned. Under the optical microscope, residues of graphite appear as glossy and

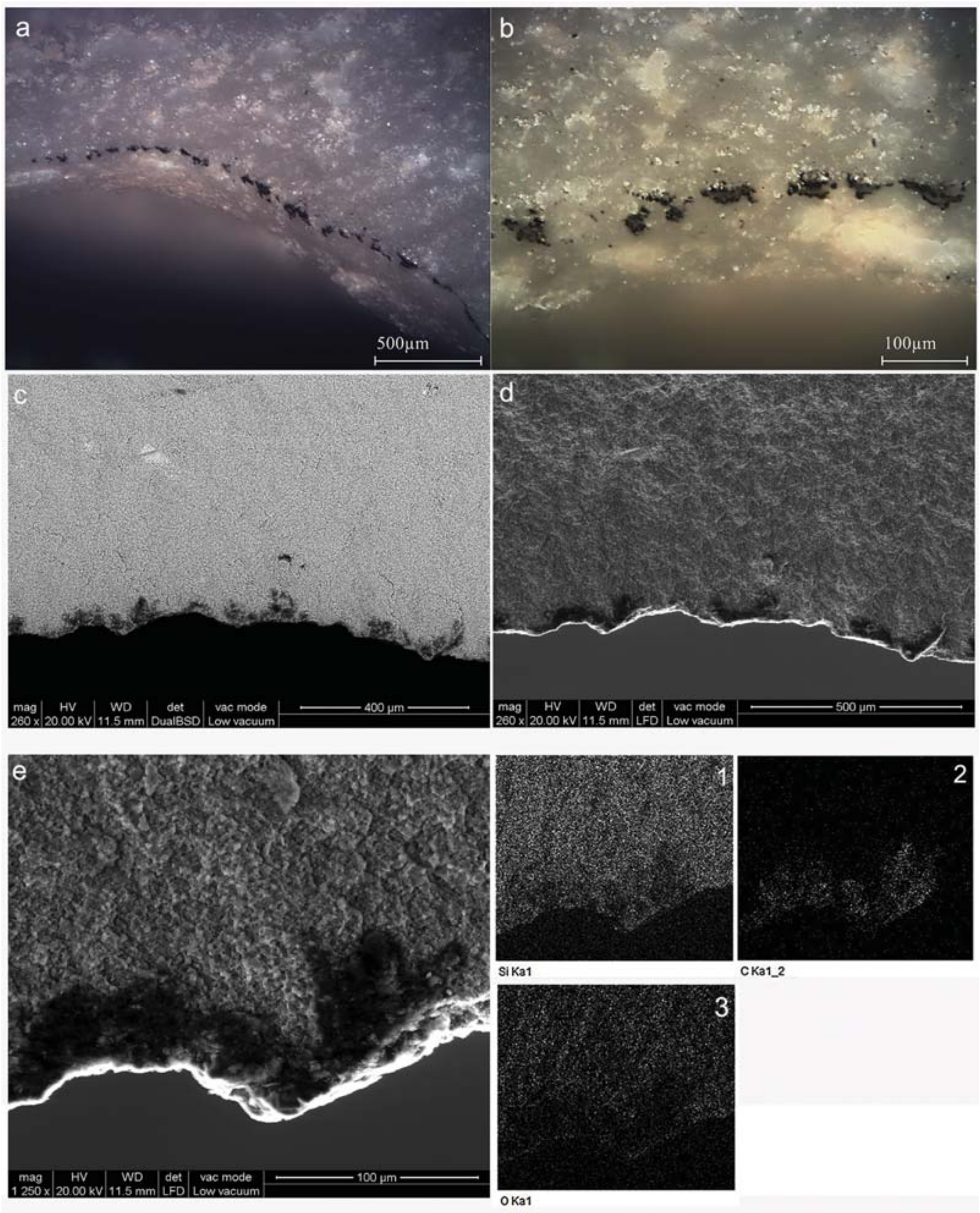

Fig. 7. Black pencil marks over the edges of a chert lithic artefact after drawing it. Black spots are present all over the edges. OLM pictures (a: orig. mag.: 50×; b: orig. mag: 200×); SEM-backscattered electron (c) and secondary electron detectors (d) images of the same portion of the edge are compared. The low atomic number of carbon (n = 6) generates less backscattered electrons and therefore the resulted image is darker (compared to silicon; n = 14). EDX was applied to the e picture and the mapping of the elements distribution correlates the carbon (2) content with the residue (e).

continuous 'black spots' along the edges (Fig. 7a and b). They show some similarities with the marks left by metal objects (Gutiérrez-Sáez et al., 1988). By only observing the optical micro-gaphs, it is possible to confuse these graphite remains with other commonly discussed 'black residues' (e.g. tar, resin, pitch, etc.) in the archaeological assemblages (Boëda et al., 2007; Charrié-Duhaut

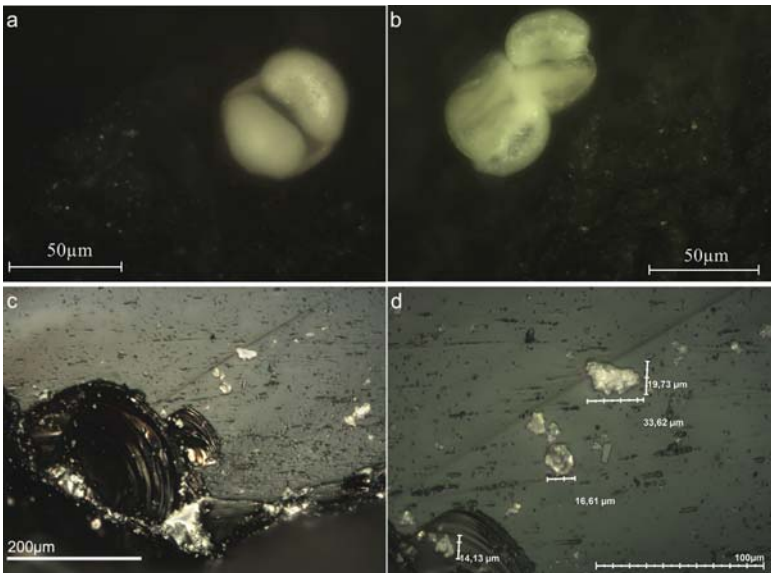

Fig. 8. (a-b) Conifer pollen particles observed on an archaeological artefact (orig. mag.: 500×). (c-d) Residues on the surface of a rock crystal experimental tool after being handled with medical starch grain-powdered gloves (c: orig. mag.: 200×; d: orig. mag.: 500×).

et al., 2013; Dinnis et al., 2009; Hauck et al., 2013). Under the SEM, the pencil marks appear in the form of accumulation of dark (dark grey or black) particles (Fig. 7c–e). EDX microanalysis clearly shows the almost exclusive presence of carbon on these spots.

Another kind of residue coming from the surrounding environment is pollen. Although not frequently registered, it might contaminate the results of residue analysis if not recognised as modern. The concentration of pollen grains in the environment increases in the period of active pollination of plants. Then, it can be easily transported and deposited everywhere. There are many ways in which pollen can enter the laboratories (the air system of the buildings, directly through an open window, adhered to the researchers' clothes...).

Pollen grains on lithic artefacts are usually difficult to be seen by naked eye, but they can be clearly detected with OLM. Indeed, they were registered on some archaeological artefacts. Under the OLM the observed pollen grains were 40–80 μm in size, of yellowish colour and usually of oval or circular shape with two *sacci*, very characteristic for conifers (Fig. 8a and b). Of course, in these cases we were sure that they were modern pollen grains (as they appeared on flakes already subjected to use-wear analysis, during which they were not detected), but in other contexts this might cause confusions.

Environmental particles can also originate from the laboratory facilities. For instance, disposable medical gloves sometimes used to manipulate the artefacts in order to avoid direct contact with hands, might also be the source of contamination. In fact, it is possible to microscopically detect residues of starch grains (usually of corn) on flakes, which originate from the talc used for the lubrication of the usual medical gloves (Loy, 1994; Wadley et al., 2004). This kind of contaminants was registered also on some of our samples (Fig. 8c and d). Therefore, a careful selection of powder-free gloves (made of vinyl, nitrile or latex) and detailed reading of the information about their composition are of great importance for the residue analysts.

2.3. *Handling experiment*

As skin flakes are very common residues observed on the stone tools' surfaces during microscopic analyses, we thought to set up an experiment aimed at their characterisation. Five unretouched flakes (Table 1) were knapped from different raw materials (flint, quartzite, basalt and obsidian) and were then extensively manipulated in order to mimic the prolonged hand contact that archaeological tools suffer after being excavated. Although rock crystal was not used in this specific experiment, observations done on this raw material contributed to the interpretation of the results.

The handling of all the experimental flakes lasted ten minutes. Subsequently, without any posterior cleaning, the flakes' surfaces were analysed combining OLM and SEM observations. Aggregates of skin flakes were localised and precisely plotted on photographs of the experimental flakes.

To better characterise this modern residue type, a thickened human skin portion (callus) of one of the authors was microscopically analysed to provide a morphological and chemical experimental reference.

3. Results

3.1. *Residues characterisation from the handling experiment: skin flakes*

The modern reference sample was analysed in order to compare the skin flakes found on the lithic surfaces with the particles composing the human epidermis. Skin flakes linked together were imaged at different magnifications with the SEM (Fig. 9). The particles composing the tissue showed the same dimensions documented for single skin flakes found on experimental and archaeological pieces during microscopic observations (Figs. 9d; 2: 3). Elemental analysis related to skin particles showed the presence of Carbon (C), Sodium (Na), Chlorine (Cl), Sulphur (S), Potassium (K)

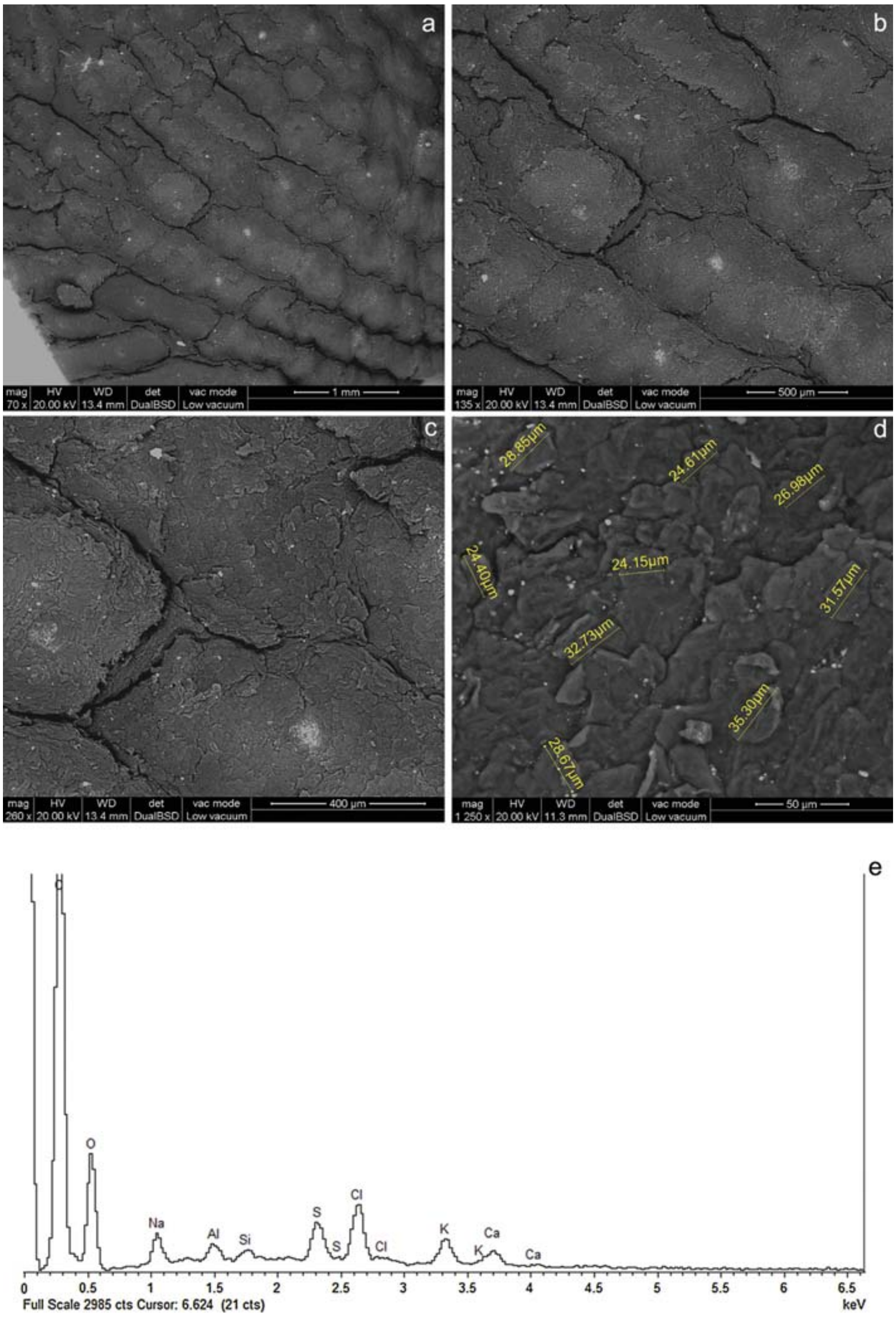

Fig. 9. SEM-backscattered electron detector images of a callus portion at different magnifications. Skin flakes appear to be cell accumulations linked together of the human skin outermost layer. Each particle have similar measurements with the skin flakes found on the surfaces of the lithic tools. (a) orig. mag. 70×; (b) orig. mag.: 135×; (c) orig. mag.: 260×; (d) orig. mag.: 1250×. (e) SEM-EDX applied on the residue showed in image d: there is the presence of C, Na, S, Cl, K, Ca and minor traces of Si. Al likely comes from the microscope stub.

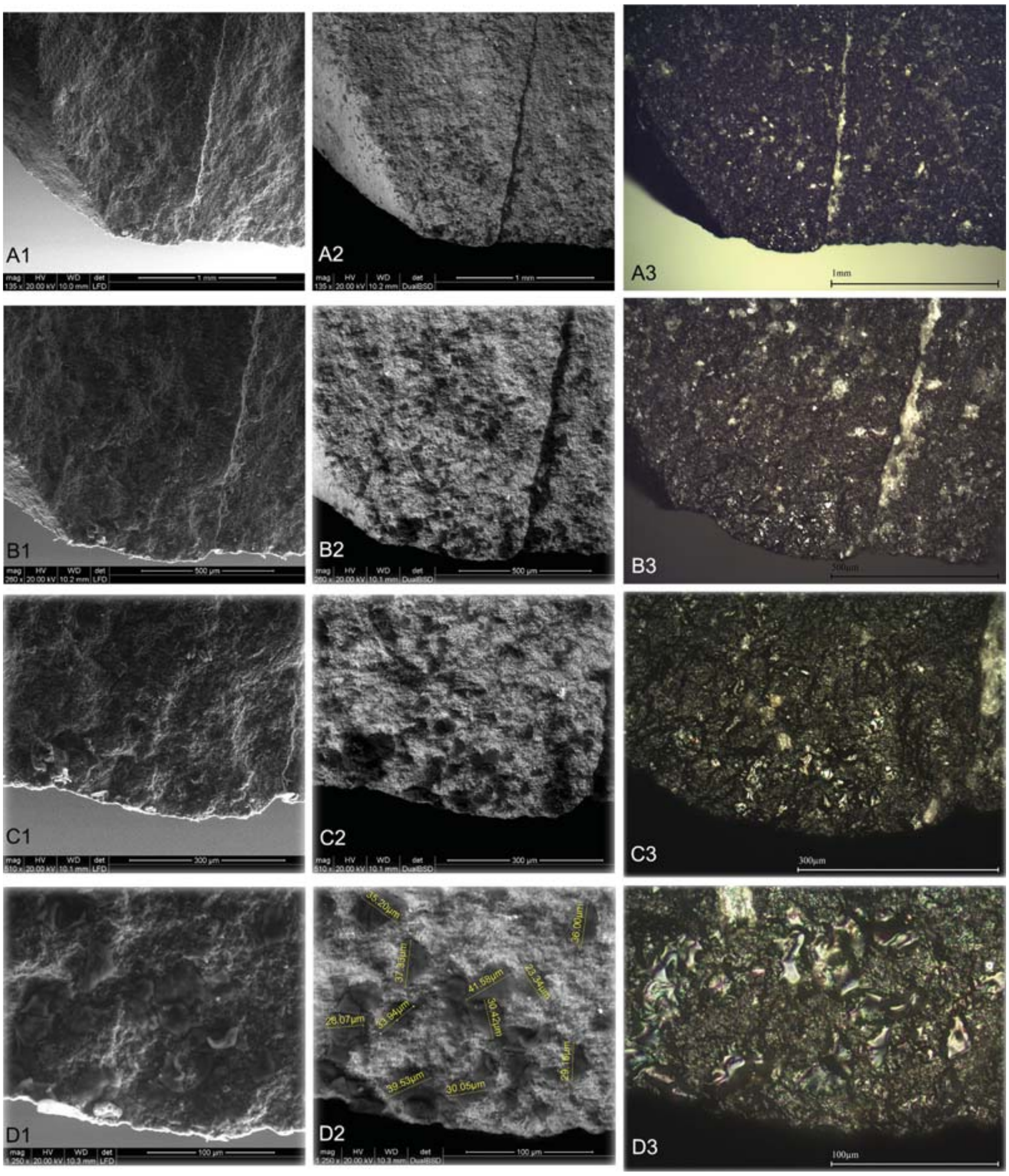

Fig. 10. Accumulations of skin flakes on a basalt tool after experimental handling. The same points are imaged at different magnifications by means of the two SEM detectors and OLM. Secondary electron detector (LFD) images are good to show textural details of the residue (A1, B1, C1, D1), while the backscattered detector (DualBSD) is useful to localise it thanks to the colour contrast (A2, B2, C2, D2). Skin flakes are also well visible under the OLM, being white and birefringent at low magnifications (A3, B3) and having a darker, "liquid" aspect at higher magnifications (C3, D3). Measurements of single skin flakes are visible in D2. OLM original magnifications: A3: 50×; B3: 100; C3: 200×; D3: 500×.

and Calcium (Ca) (Fig. 9e), as already observed in previous works (Ollé, 2003; Vergès, 2003).

Because of the intense manipulation, residues appeared to be extremely abundant on the experimental lithic samples and therefore, visible even at relatively low magnifications (Fig. 10A). They are better observable in images taken with the SEM-backscattered electron detector (Fig. 10, A2, B2, C2, D2), because they appear darker than the rock surface. The secondary electron detector is more useful to image the morphological traits of the residue, yet only clearly visible at higher magnifications (Fig. 10C1, D1). Under the lower magnifications of the OLM (e.g. 50×) (Fig. 10A3), depending of the rock type and quantity of particles, they look like the small

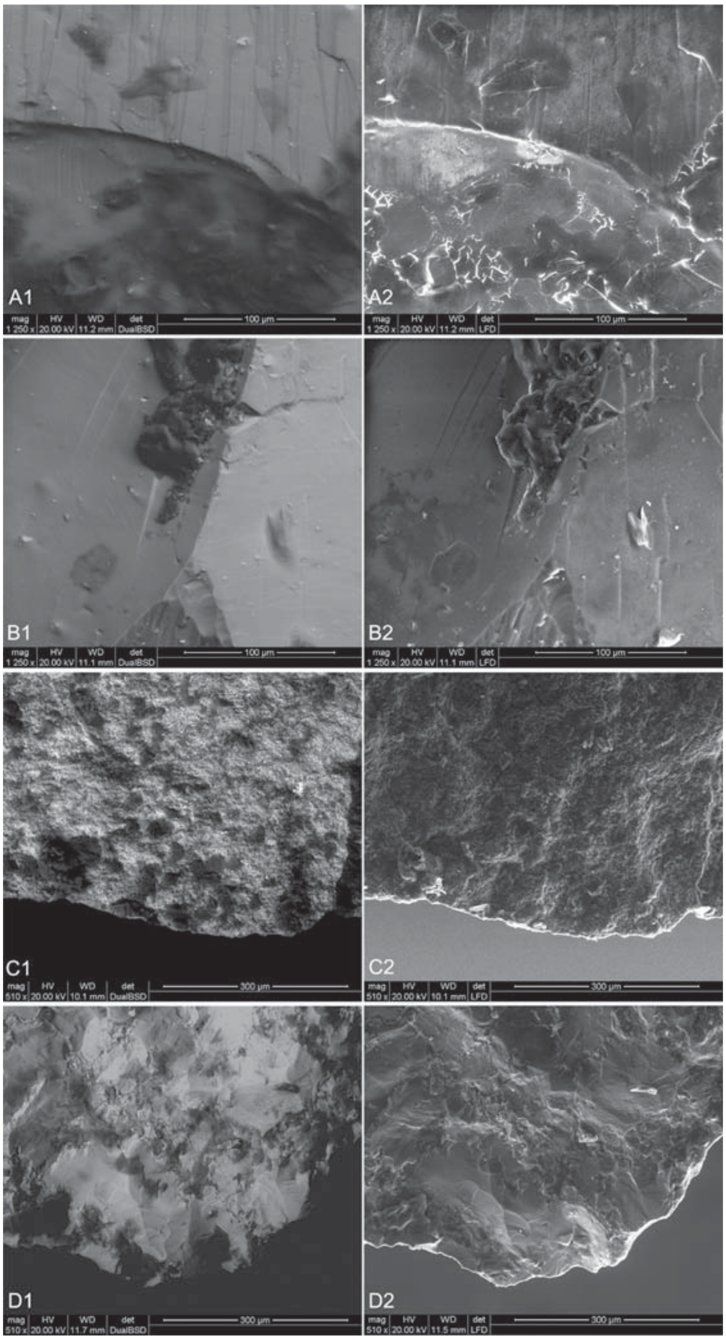

Fig. 11. Accumulations of skin flakes after experimental handling on tools made of obsidian (A1-2; B1-2), basalt (C1-2) and quartzite (D1-2). Comparison of the same points imaged through the DualBSD and the LFD detector.

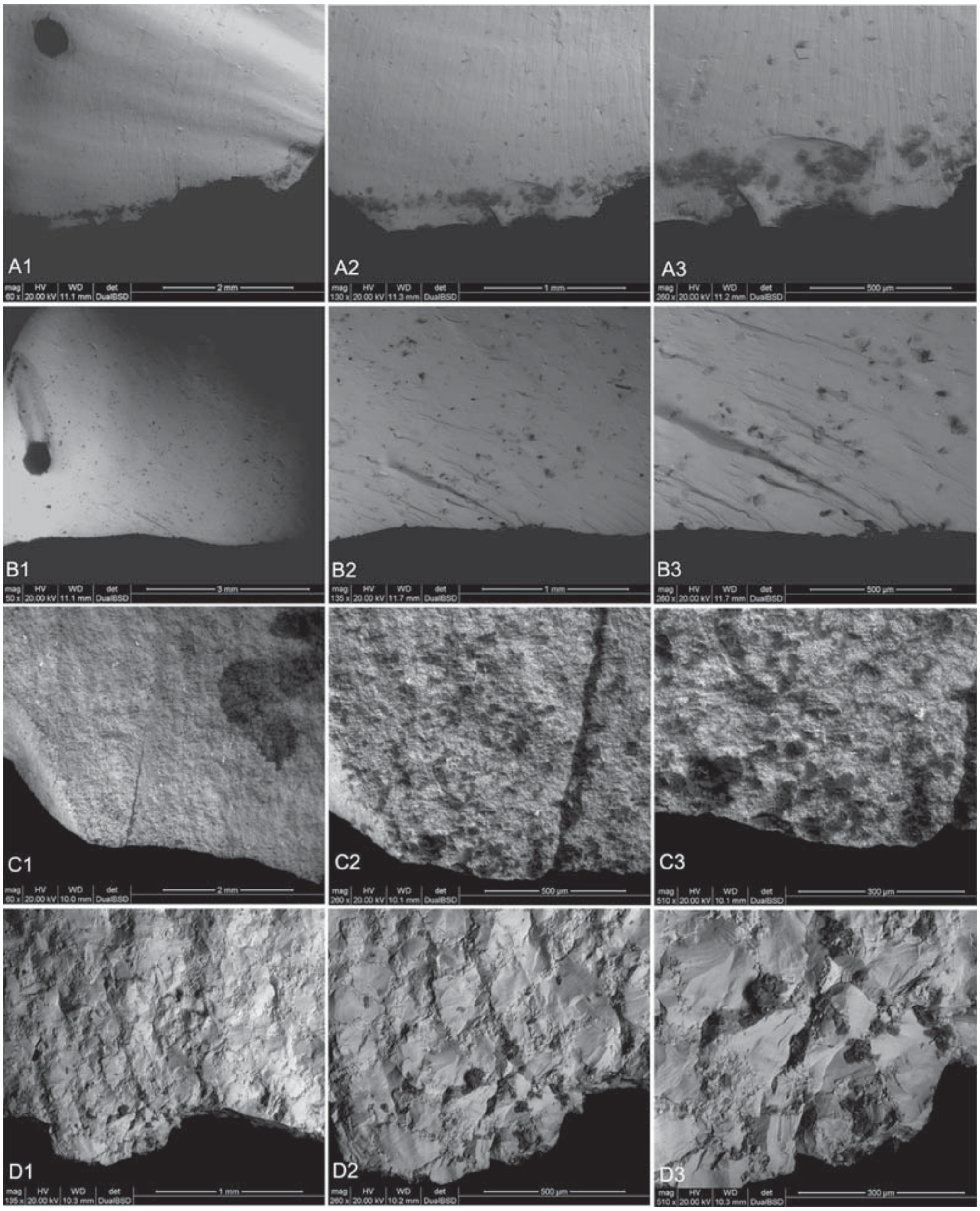

Fig. 12. Accumulations of skin flakes at different magnifications after experimental handling on tools made of obsidian (A1-2-3; B1-2-3), basalt (C1-2-3) and quartzite (D1-2-3). All the micrographs are obtained with the DualBSD detector.

crystals of the rock substrate, while at higher magnifications (e.g. 200×) they are similar to surface alterations (e.g. rounding, polish) (Fig. 10C3). Under the OLM, accumulations of skin flakes are seen as white and quite a birefringent substance at relatively low magnifications (Fig. 10A3, B3), while they appear as dark particles with a 'liquid' appearance at higher magnifications (Fig. 10C3, D3). This could complicate their identification, especially when the rock substrate is dark.

Apart from the colour of the rock substrate, which could facilitate or hinder the detection of skin flakes on the lithic surfaces,

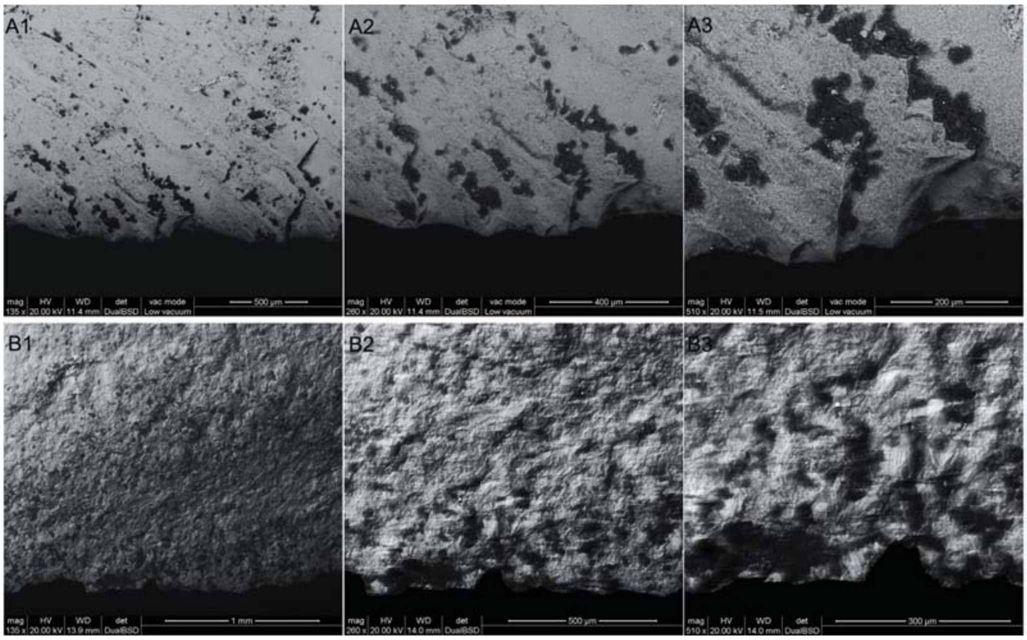

Fig. 13. Backscattered electron detector micro-graphs of skin flakes on experimental flint flakes. The same points are seen at different magnification. A1-2-3: Norfolk flint (UK); B1-2-3: Neogene chert (Burgos, Northern Spain).

we noticed that the particular micro-topography of the raw material may affect the distribution of skin flakes (and maybe other residues). In particular, skin flakes seemed to be more homogeneously distributed over the flat surfaces of fine-grained raw materials (e.g. flint, obsidian) (Fig. 11A and B; Fig. 12A, B). On flint artefacts, the smoother the surface the more regular the residue distribution. The main concentrations tended to appear just on the edges and often following the rippled surface created by the knapping lancets (Fig. 13a2, a3).

Conversely, on coarse materials (e.g. basalt and quartzite) skin flakes were more concentrated over the larger fissures and the irregularities of the surface, being in the form of scaled surfaces in the case of basalt (Figs. 11C; 12C). Concerning quartzite, skin flakes were more abundant near holes or elevations all around single quartz crystals (Figs. 11D and 12D).

These preliminary results inferred by analysing a small experimental sample are not sufficient for determining the particular patterns of skin-flake distribution related to different raw materials. Together with the rock texture, other variables could affect the actual distribution of skin flakes on the tool surfaces, such as the handling and environmental conditions (humidity, temperature, the time of exposure, etc.).

3.2. Modelling clay

Some of the studied artefacts were in contact with the modelling clay often used in laboratories for different studies. As we mentioned above, relatively big patches of this substance can adhere to the tools' surfaces after using it to hold them on the specimen holder for optical microscopic analysis (Fig. 14).

Under the OLM only the bigger portions of the residue are visible (Fig. 14A1, A3), while the SEM-backscattered detector is capable of detecting even the tiniest spot (Fig. 14A2, A4). In fact, even when the surface seemed to be free from this type of residue under the OLM (Fig. 14B1), the SEM micrograph of the same point showed a sparse distribution of it (Fig. 14B2). Elemental microanalysis showed the clear presence of Carbon (C), Sodium (Na), Chlorine (Cl), with minor concentration of Sulphur (S), Potassium (K) and Calcium (Ca) (Fig. 15). Obviously, the Silicon (Si) and Aluminium (Al) peaks come from the rock substrate. The mapping of one residue spot linked these three elements with the residue itself, contrasting with the Silicon present in the rock substrate (i.e. quartzite) (Fig. 16).

3.3. Cleaning procedures

The processing and cleaning of the samples (archaeological and experimental) is one of the most delicate moments prior to use-wear analysis. In the use-wear related literature, different authors suggested diverse methods and chemical products to clean the surfaces of stone tools (among others: Anderson, 1980; Anderson-Gerfaud, 1986; Evans and Donahue, 2005; Grace, 1989; Keeley, 1980; Mansur-Franchomme, 1983; Ollé and Vergès, 2008, 2014; Van Gijn, 1986). One of the most followed proposal of cleaning processes prior to use-wear analysis was made by Plisson (1982, 1986), with the introduction of acetone to eliminate animal or human grease. Although we normally cleaned our samples with pure acetone, generally regarded to be sufficient to remove handling dirt, the surfaces of the tools appeared very frequently full with this particular residue.

Consequently, we adapted a multi-step cleaning procedure used in previous works consisting of different steps, including different chemical products. After each step, some selected points on the artefacts were microscopically observed in order to evaluate if the products we used were effective or not.

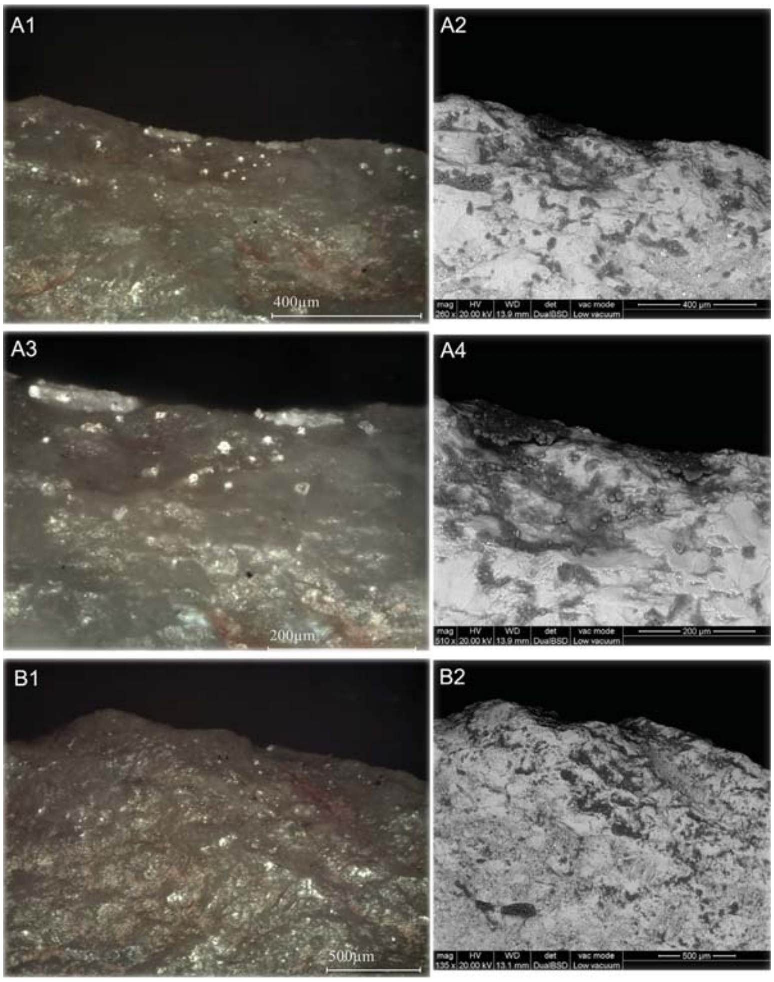

Fig. 14. OLM and SEM-DualBSD micro-graphs of the same points showing the presence of modelling clay accumulations on a quartzite archaeological tool. Under the OLM modelling clay is birefringent (A3), but at lower magnification it is not visible (B1), while the same point analysed at SEM shows the sparse occurrence of this residue (B2). At higher magnifications, the granular aspect is appreciable (A2-4). OLM original magnifications: A1: 100×; A3: 200×; B1: 50×.

We considered the following steps: (1) a sonic bath in pure acetone for 5 min (Fig. 17A1, B1, C1); (2) a sonic bath in a 2% neutral soap solution for 15 min, followed by a 5 min sonic bath in pure acetone (Fig. 17A2, B2, C2); (3) a sonic bath in oxygenated water (H_2O_2) for 10 min, followed again by a 5 min sonic bath in pure acetone. We observed that neither pure acetone alone, nor the neutral soap solution were enough to remove the residues, and also after the bath in oxygenated water some particles survived (Fig. 17A3, B3, C3).

The modelling clay we found on some artefacts appeared also to be very difficult to remove. We applied the same procedure described above. We observed that neither the pure acetone nor the acetone combined with the neutral soap are capable of removing this very sticky substance (Fig. 18: A1-2, B1-2, C1, 2). After the last sonic bath in oxygenated water (10 min), followed by the routine 5 min acetone bath, most of the residue disappeared. However, some particles survived this intense cleaning (Fig. 18C3). When there were large accumulations of modelling clay both on the edge or in the interior of the surface, relatively large particles survived after our cleaning process (Fig. 19).

Concerning other modern contaminants possibly found on the lithic artefacts, the documented graphite remains left by the pencils used for drawing appeared to be very difficult to eliminate. It normally required long sonic baths (20 min each) in neutral soap and pure acetone.

For non-sticky residues which accidentally are deposited on the surface of stone tools, such as fibres, pollen, or starch grains coming from powdered gloves, no particular cleaning procedure is needed. Generally, they can be easily removed using neutral soap baths and, when they are completely dry, simply with pressured air.

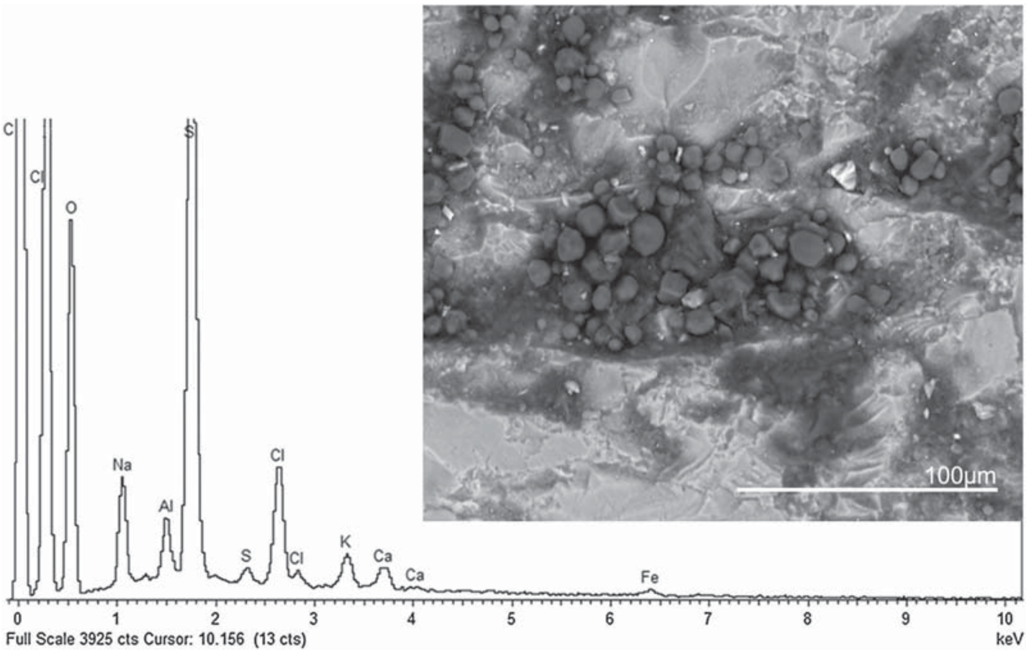

Fig. 15. SEM-EDX analysis of a modelling clay residue showing the presence of C, Na, S, Cl, K and Ca. Si, O, Al and Fe are components of the rock substrate, which is quartzite.

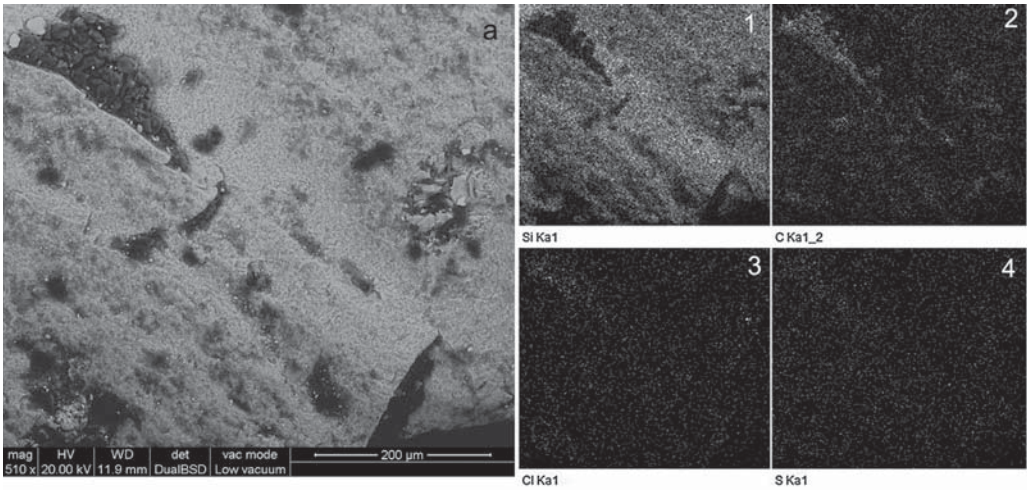

Fig. 16. SEM-EDX analysis of a modelling clay residue showing a mapping of the present elements. Carbon (C = 1), Chlorine (Cl = 3) and Sulphur (S = 4) are connected with the residue, while silicon (Si) is related to the rock substrate (quartzite).

4. Discussion

Besides the correct identification of the residues type, one of the most controversial problems one can face in residue analysis applied on lithic tools is the differentiation between ancient and modern material.

Dating methods were designed to directly determine the age of residues on stone tools and some studies already presented preliminary, but very promising, AMS results (Yates et al., 2014, 2015). However, at least for very ancient chronologies, those methods are not applicable, hence analysts should find a way to overcome this problem. Therefore, the first step to avoid confusion is being able to recognise modern contaminants.

The combination of the OLM and the SEM proved to be a feasible solution to improve the characterisation of micro-residues on stone tools (Borel et al., 2014; Monnier et al., 2012; Pedergnana

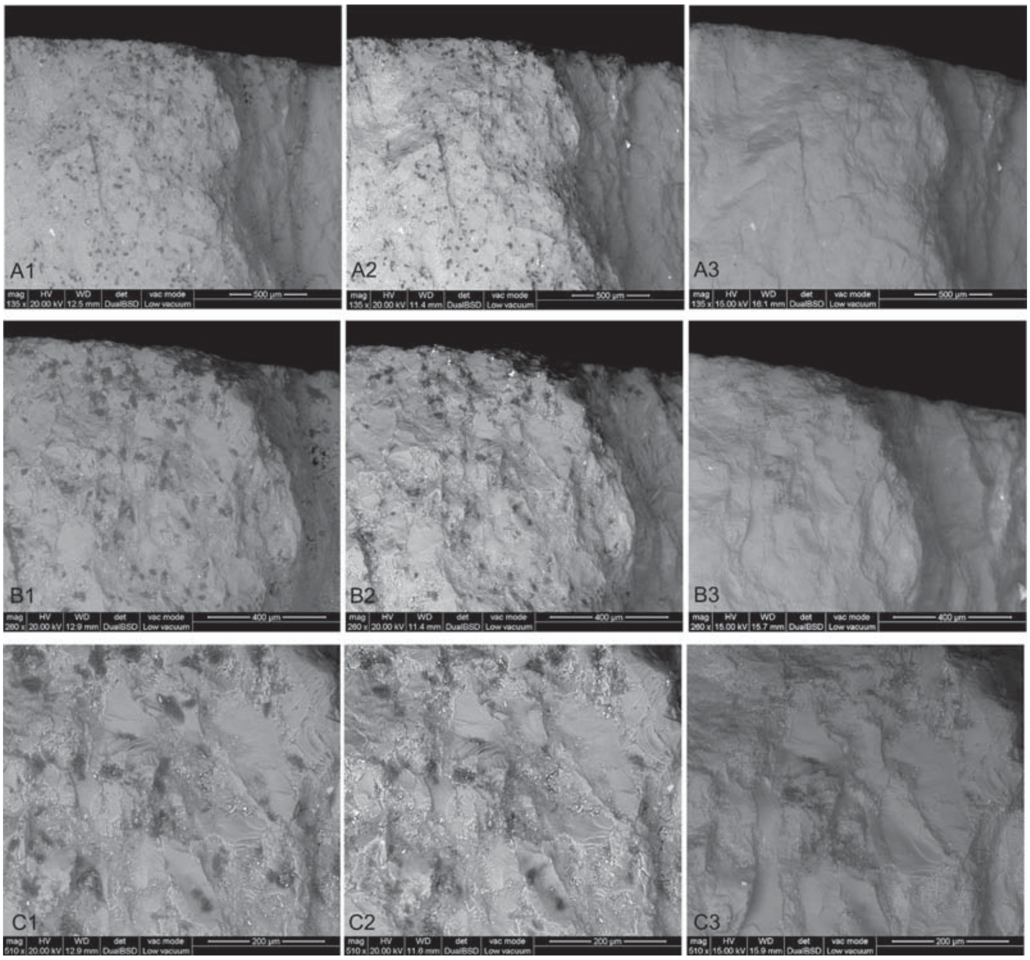

Fig. 17. Backscattered electron detector micro-graphs of the same point on a quartzite tool, imaged at different magnifications after sequential cleaning procedures aimed to remove modern skin flakes. The first step involved a 5 min sonic bath in pure acetone (A1, B1, C1); the second one comprised a 10 min sonic bath with a neutral soap solution at 2% (15 min) followed by a 5 min acetone bath (A2, B2, C2); and in a third phase a 10 min sonic bath in oxygenated water (H_2O_2) and a 5 min one in pure acetone were performed (A3, B3, C3). Some residue remains survived the entire cleaning procedure.

and Ollé, 2014). Particularly, the EDX microanalysis is an additional tool which instantly provides the elemental composition of the analysed residues, therefore it highly facilitates their assignation to the correct residue type. Other analytical techniques, employed to identify specific compounds (e.g. GC–MS-Gas chromatography-Mass Spectrometry, FTIR-Fourier Transform Infrared microscopy, Raman spectroscopy) are considered as a fundamental step to better characterise both ancient residues and contaminants (e.g. Cârciumaru et al., 2012; Evershed, 2008; Helwig et al., 2014; Prinsloo et al., 2014).

Certainly, the experience of the analyst plays an important role, and the availability of a complete experimental comparative collection is fundamental to reach a correct interpretation. Indeed, the characterisation of modern residues is part of this essential extensive experimental collection. When such a characterisation is missing, modern residues can be misinterpreted for ancient ones.

For instance, skin flakes are the most recurring modern residue on stone tools. Dozens of people may virtually work on the same assemblages and practically all kind of analysis (e.g. technological, refitting, raw material, functional) involves hand contact. Therefore, residue analysts should always be able to correctly recognise these particular residues. The combined imaging of the same points with both OLM and SEM allows to assess their possible different appearance when scanned with different microscopic techniques, and also to improve the analysts' capacities to recognise them. Thanks to our experiments, we showed that sometimes even after relatively intense cleaning procedures, modern skin flakes can survive. Furthermore, in some cases, remarkable morphological similarities are found between animal skin residues and our experimental skin flakes, therefore it might be difficult to differentiate them (Mansur-Franchomme, 1983). In this context, some ancient attribution to this kind of residue should be taken with extreme caution (Boëda et al., 2014).

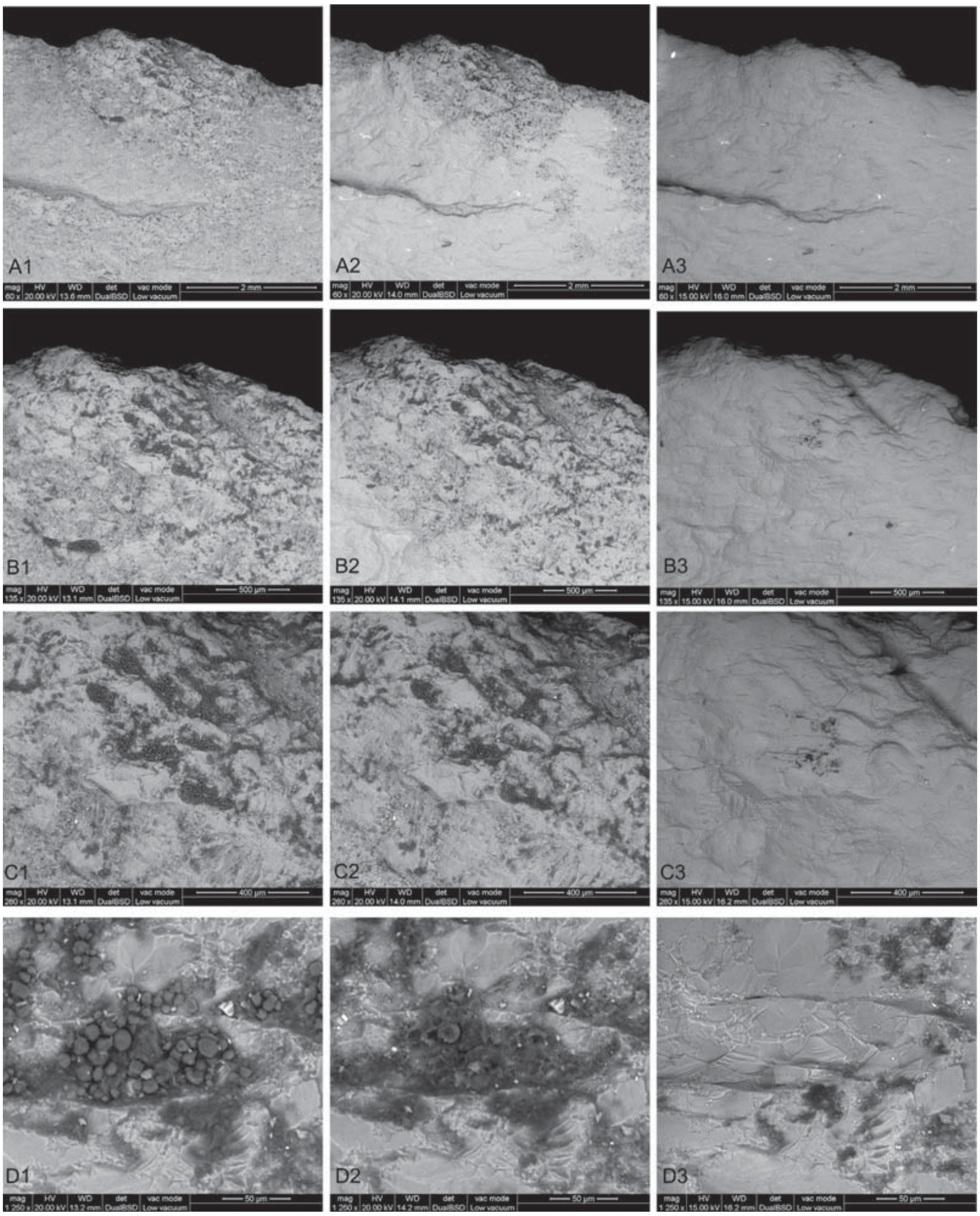

Fig. 18. Backscattered electron detector micro-graphs of the same point on a quartzite tool, imaged at different magnifications after sequential cleaning procedures aimed to remove modern modelling clay residues. The first step involved a 5 min sonic bath in pure acetone (A1, B1, C1, D1); the second one comprised a 10 min sonic bath with a neutral soap solution at 2% (15 min) followed by a 5 min acetone bath (A2, B2, C2, D2); and in a third phase a 10 min sonic bath in oxygenated water (H_2O_2) and a 5 min one in pure acetone were performed (A3, B3, C3, D3). Some residue remains survived the entire cleaning procedure.

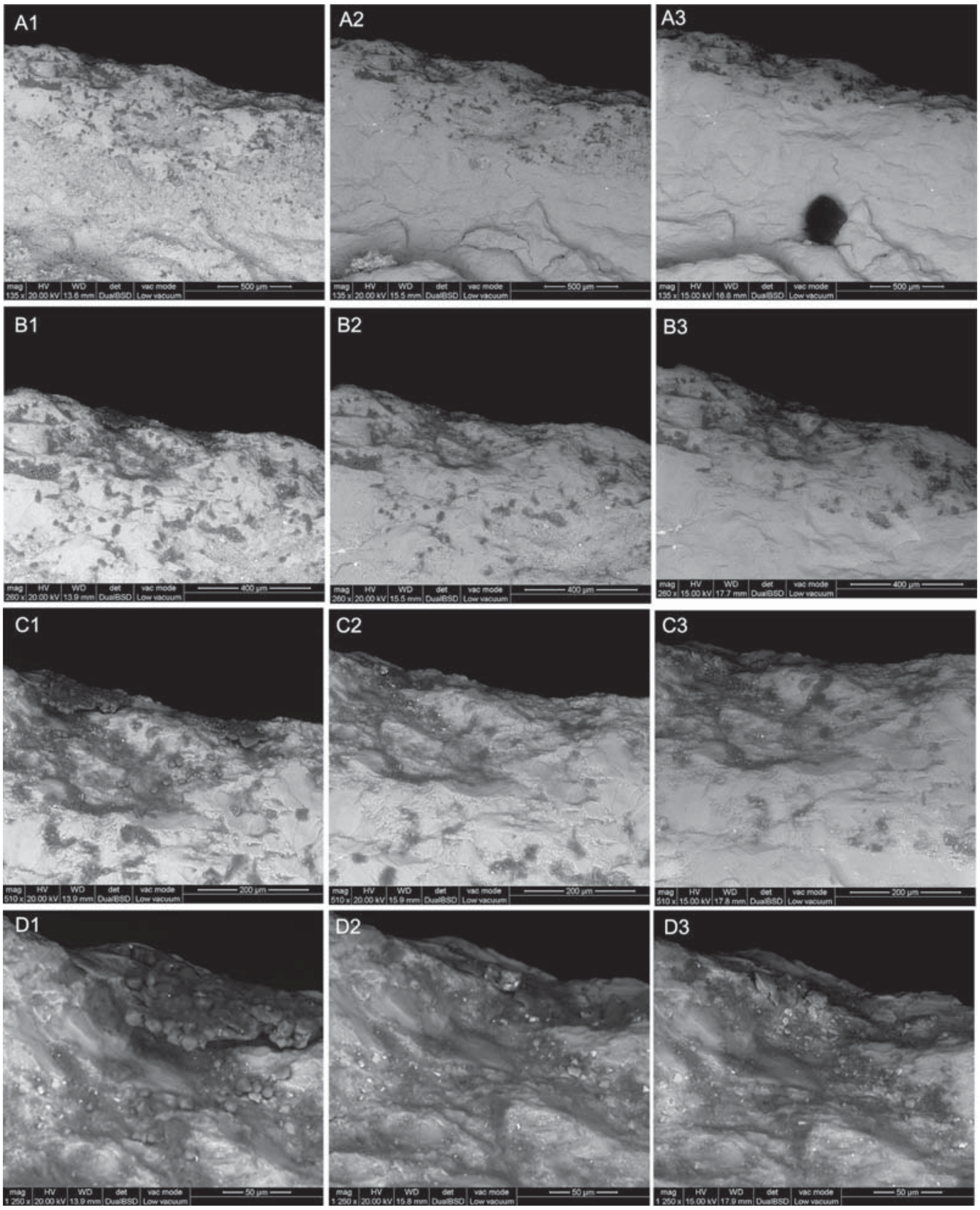

Fig. 19. Backscattered electron detector micro-graphs of the same point on a quartzite tool, imaged at different magnifications after sequential cleaning procedures aimed to remove modern modelling clay residues. The same cleaning procedures described for Fig. 18 were applied; first step (A1, B1, C1, D1); second step (A2, B2, C2, D2); third step (A3, B3, C3, D3). As the residue accumulations were quite large, more particles of the residue survived the cleaning procedure.

To be certain that the analysed residues are indeed ancient, it would be highly convenient to analyse the artefacts soon after they are excavated. The ideal of absolutely untouched archaeological collections is only occasionally feasible, even if sometimes this is done (Lombard, 2008). Evidently, it would be desirable that the analysed samples do not undergo any cleaning procedure so as not to destroy ancient residues which may be possibly present.

As this is not a realistic scenario, knowing that lithic assemblages need to be subjected to different kind of studies, we think that analysts should develop their own cleaning methods depending on their specific case-studies. The careful sampling of the artefacts to be analysed (possibly in the field) may reduce the incidence of modern contaminants. Those selected artefacts would not be washed nor drawn and contact with hands or modelling clay would be avoided.

Modern contamination depends also on the raw material type. When dealing with coarse materials (quartzite, basalt, granite, etc.) we must expect a higher incidence of modern residue (especially skin flakes), distributed all along the micro-topographical irregularities (Rodríguez-Rodríguez, 1998). Flat surfaces (obsidian) do not retain a large quantity of skin flakes but other kind of dirt can be more evident (Rots and Williamson, 2004). For instance, minor hand contact is enough to spread grease lines all over the surface (at least concerning rock crystal) (Fernández-Marchena and Ollé, 2016).

Blind tests were not contemplated in this study, since a previous evaluation of contamination was needed before testing the capacity of analysts to recognise contaminants. The potential of this kind of tests is undeniable to construct and perfect a specific method (Lombard and Wadley, 2007; Monnier et al., 2012; Wadley and Lombard, 2007; Wadley et al., 2004; Rots et al., 2016). In our opinion, blind tests should contemplate various techniques (OLM, SEM, Raman spectroscopy, staining procedures, etc.) to assess the best possible technique combinations to obtain the most reliable results. For instance, a valuable proposal come from a recent work where the authors, after a careful revision of their blind test results, describe a possible order to analyse residues on stone tools, including direct observation of residues by recording their exact position, extraction of the residues and subsequent application of staining reagents (Rots et al., 2016). Such studies are crucial for a proper development of the method and are also very useful to underline the limits of a discipline.

This work was not only addressed to residue analysists, but also to archaeologists handling lithic material and it was thought to call for caution in the post-excavation treatment of archaeological lithic collections. In short, handling should be kept to a minimum. We also demonstrated that particular substances normally used in laboratories such as the modelling clay can be extremely difficult to remove; thus they should be absolutely avoided when residue or use-wear analyses are expected to be later applied. Another easy option is to put plastic film between the analysed tool and the modelling clay to avoid direct contact.

Apart from handling, the environmental conditions of the laboratories where residue analysis is performed should also be considered in order to reduce sample contamination. In this sense, to check whether the micro-residues found on the stone tools are genuine prehistoric residues or not, systematic tests on the laboratory materials and facilities are crucial (Mercader, 2009).

However, as there is not a universal formula for all the contexts, analysts should always develop specific cleaning methods and laboratory protocols for the archaeological material they study.

5. Conclusion

The major aim of this research was to carefully analyse the most recurring modern residues on stone tools in order to be able to discriminate between them and the ancient residues when archaeological tools are analysed. For this purpose, both OLM and SEM were used.

The cleaning procedures we used to remove the modern contaminants, such as skin flakes or modelling clay, proved to be effective, although it would probably affect also the ancient residues, if present. For this reason, the best option is always to minimise the post-excavation handling of the artefacts meant to undergo microscopic residue analysis.

Acknowledgments

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4.7. Selection of the archaeological material

The selection of the artefacts to be microscopically analysed followed a number of criteria. First of all, given the different nature of the two assemblages (number of artefacts, preservation condition, quality of the raw material, etc.), we chose to apply different criteria in the process of selecting the tools to be analysed. Basic conditions were required for the tools to be selected, such as: good preservation of the surfaces, edges presenting significant angles and morphologies making them apt for intervening on both organic and inorganic materials (regular edges).

Other criteria are taken into consideration, such as: the presence/absence of retouch, metric features (compared with the average mean measures of the whole assemblages).

However, because of the high number of artefacts at Gran Dolina site-TD10.1 (3,608), we were required to adapt our selection process. Since focusing only on a specific tool-type (*e.g.* scrapers) or randomly selecting retouched and un-modified pieces would not be representative of the entire assemblage, we chose instead to apply a techno-functional approach and to analyse all quartzite flakes from GD-TD10.1 exceeding 20 mm in length as a first stage preceding an additional selection for microwear analysis.

Regarding the assemblage from the Payre site, the relative low number of artefacts (155 implements, 126 being flakes) (Moncel ed., 2008), did not require the application of the same selective criteria prior to submitting the tools to microscopic analysis.

4.7.1 Techno-functional analysis of the quartzite flakes from Gran Dolina, level TD10.1 and selection for use-wear analysis

The techno-functional approach (Lepot, 1993; Boëda, 1999, 2013) was adapted to fit the scope of this research project. Not all of the assemblage was submitted to analysis. Debris, unidentified angular fragments, cores and natural pieces have been systematically excluded. Cores, even if not included in the analysis, were analysed separately in order to understand the flake production systems. All of the whole flakes (retouched and non-modified) exceeding 20mm long were submitted to a technological analysis. Broken flakes (> 20mm) were also included for study. Smaller flakes were not included here because may more readily be attributed to simple knapping waste (likely unused). Out of 3,608 quartzite implements found in TD10.1 (Ollé et al., 2013), a total of 519 (14,4% of the assemblage) was technologically analysed in this work, mainly following Inizan et al. (1995, 1999).

Hence, cortical and semi-cortical flakes, pre-determinant and predetermined flakes and retouched flakes measuring at least 20mm on one axis were analysed. For each artefact, the basic information (raw material colour, granulometry, cortex presence and distribution, length, width and thickness measurements and morphology) was recorded. The ‘symmetry’ of the artefacts was documented based on the flaking axis orientation of the piece and not on the morphological one. Type, measurements and angle of the striking platform were also recorded. Episodes of accidental breakage were documented as well (hinged, plunging, Siret or abrupt fractures). The number and orientation of the previous removal (negative scars) visible on the dorsal surfaces were also recorded (Dauvois, 1976). The Laplace typology (1962) was used for the adscription of the tools to catalogue types.

As the main objective of this analysis was to try to understand the functional potential of the edges prior to microscopic analysis, and then make an additional selection amongst a smaller number of tools, additional analytical criteria have been extrapolated from the French techno-functional approach (Lepot, 1993; Boëda, 2013). Considering the exponents of both the techno-functional approach and the analysis of the functional potential of the tools, new variables are introduced in our study (among others, Airvaux, 1987, Bourguignon, 1997, Lourdeau, 2010; Soriano, 2010; Rocca, 2013).

The criteria considered were:

- The 'cutting plane' (*dièdre de coupe*) of all the regular edges was analysed (Fig. 4.13: 1). The angle was measured (Fig. 4.13: 2), the convergence of the dorsal and ventral surfaces is described as being: plane/plane (Fig. 4.13: 3, b), plane/convex (Fig. 4.13: 3, b), plane/concave (Fig. 4.13: 3, c), convex/plane (Fig. 4.13: 3, d), convex/convex (Fig. 4.13: 3, e), convex/concave (Fig. 4.13: 3, f), concave/plane (Fig. 4.13: 3, g), concave/convex Fig. 4.13: 3, h), concave/concave Fig. 4.13: 3, i).
- The frontal view of the edges is described as concave, denticulate, rectilinear, or convex (Fig. 4.13: 4), while the sagittal view can be rectilinear, curved or sinusoidal (Fig. 4.13: 5).

A database including technological data as well as the techno-functional characters for each piece was then compiled (Annex 6). After the collection of data, statistical analysis allowed to identify techno-functional groups on the basis of recurrent characters in both backed edges and active parts (retouched or not). Pieces pertaining to the same techno-functional group share a similar volumetric structure and techno-functional potential (Boëda, 1997).

Subsequently, use-wear analysis was performed on a selection of artefacts per techno-group with the main aim to evaluate the coherence of the techno-groups identified.

Not all the artefacts analysed pertain to techno-groups, as use-wear analysis started before the conclusion of techno-functional analysis. Also, technological categories not included in the techno-functional analysis, have been sometimes considered for use-wear analysis due to the potential of concrete tools (biface-like, hachereau-like). Other few pieces were not technologically analysed, but yet included in the selection of samples for use-wear analysis (for example artefacts compositing refits).

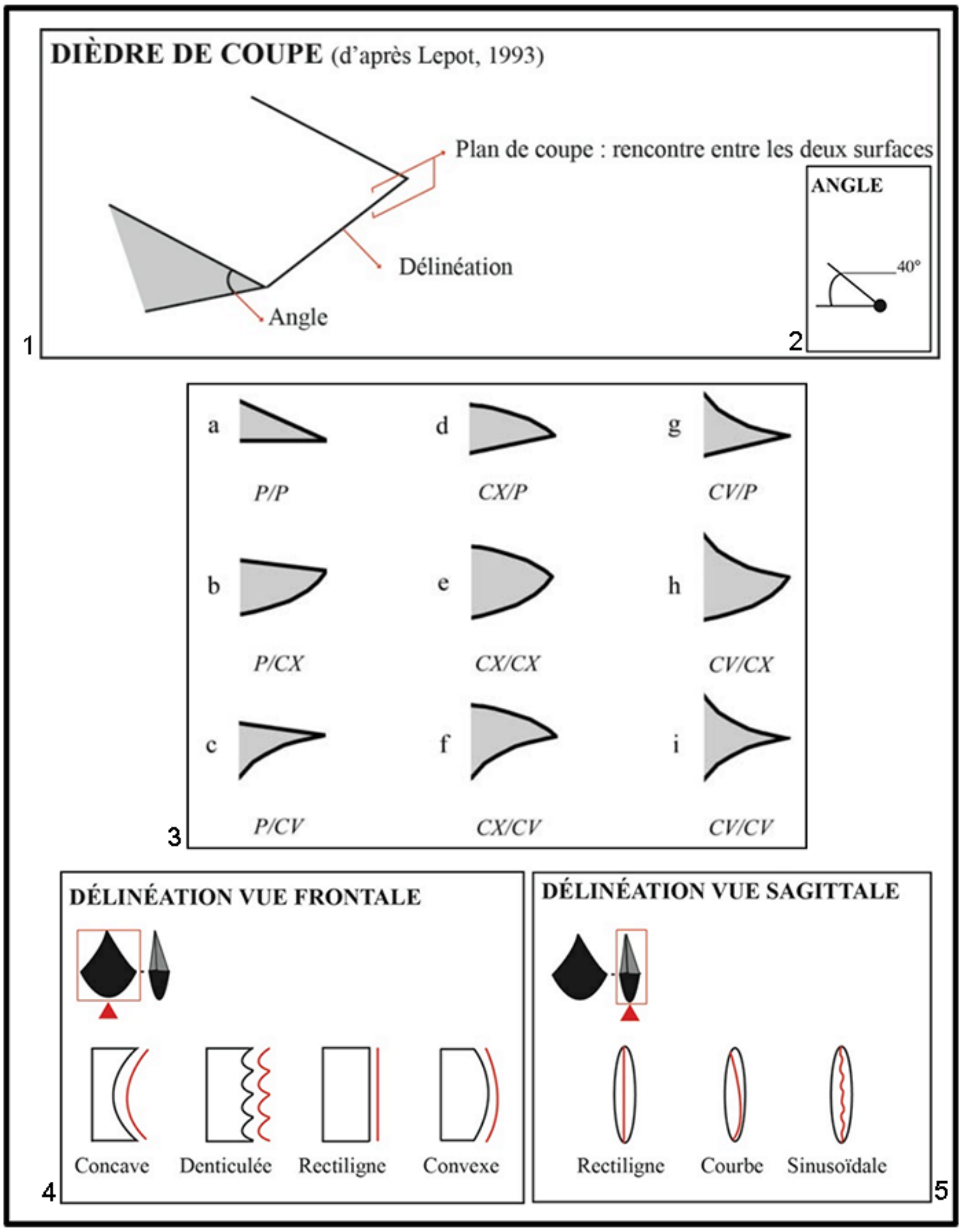


Fig. 4.13: *Criteria for the techno-functional analysis: 1) 2) 3) 4) 5). Schemes are modified from Rocca, 2013:66, except number 3 which is taken from Bonilauri, 2010:47.*

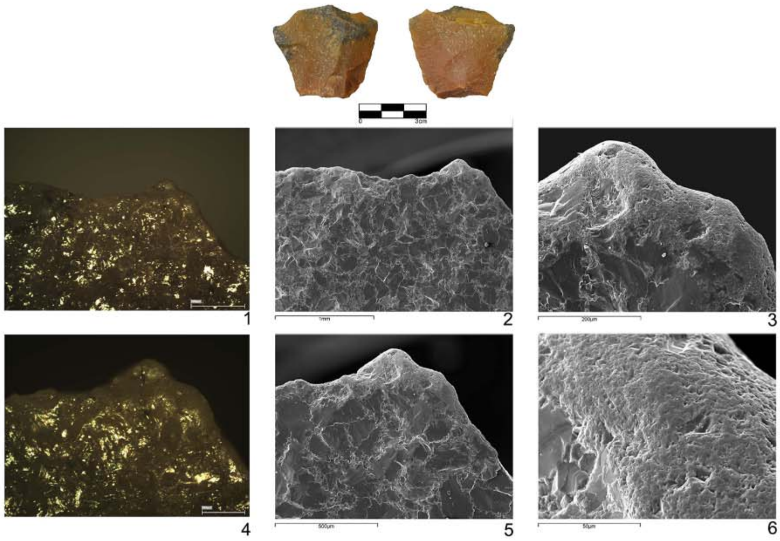
4.7.2 Payre site: selection of the analysed sample

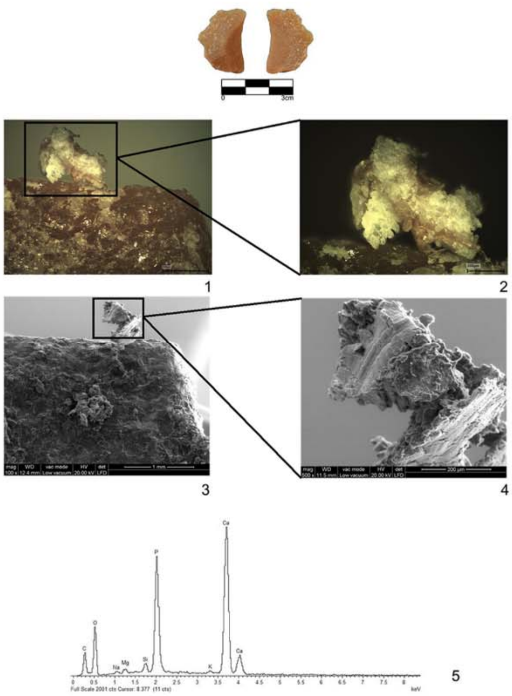
Concerning the quartzite assemblage of the Payre site, no additional technological data were obtained. The selection of the artefacts to be analysed microscopically follows the available technological data (Moncel ed., 2008). Different technological supports were included, from un-modified flakes to large cutting tools.

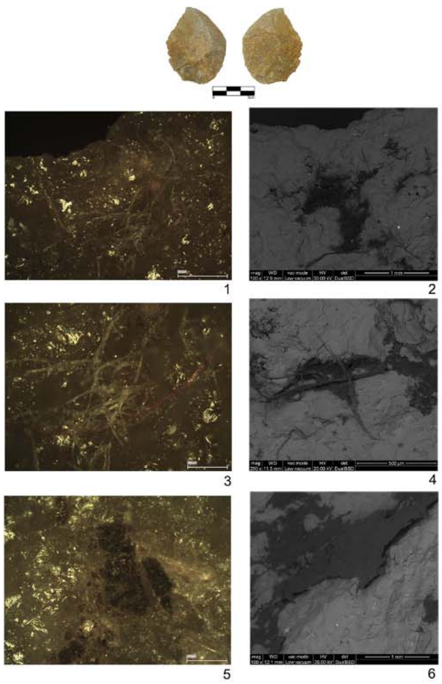
All the small to medium-sized flakes were subjected to low power observations in order to make an initial selection. The main parameter considered at this stage of analysis was the presence/absence of any macroscopic post-depositional modifications.

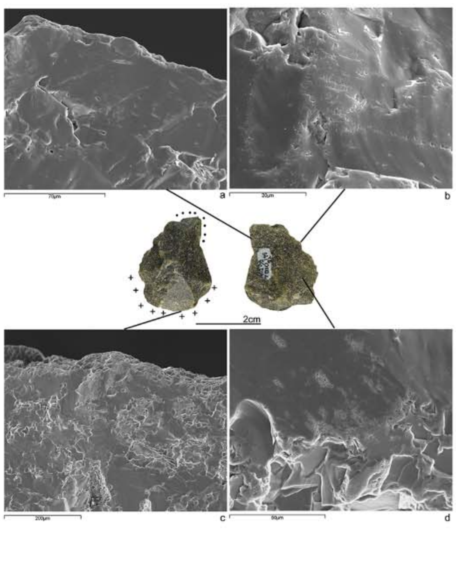
Afterwards, some of the artefacts which presented good preservation were submitted to high-power observations (metallographic and electron microscopes). At first, the idea was to analyse the totality of the artefacts by means of high-power devises, but in a more advanced phase of the work we had to reformulate our objectives, mainly because of limited amount of time.

Hence, a limited number of artefacts was analysed, trying to respond to several questions, such as: defining differential preservation of the quartzite artefacts throughout the stratigraphic sequence, understanding the reasons for the presence of significant differences in the size of the implements, etc. Because of all of these reasons, it was important to comprise different technological categories in the sample selected for use-wear analysis.









PART III: RESULTS

Chapter 5: Experimental results

In this chapter are exposed in detail the results of the experiments carried out within this thesis. These comprise the observations from both experimental use-wear and residues of the worked materials.

First, a published work is presented, containing the use-wear results on the four varieties of quartzite used in the experiments.

Second, wear on quartzite is discussed taking into consideration its formation processes. Details of the appearance of traces on each variety are summarised and similarities and divergences are described.

Third, experimental results from the study of micro-residues are presented in the form of a published work.

5.1 Publication 5:

Pedergnana, A., Ollé, A., 2017a. **Monitoring and interpreting the use-wear formation processes on quartzite flakes through sequential experiments.** *Quaternary International* 427 (Part B), 35-65.

In this publication, the experiments included in this thesis are described in detail. The methodology employed is also summarised as well as the technical equipment used.

Results are dedicated to show the visual appearance of wear on quartzite, as originating from contact with different materials. An effort to systematise this rich information was accomplished.

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Monitoring and interpreting the use-wear formation processes on quartzite flakes through sequential experiments

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A B S T R A C T

Sequential experiments were performed with quartzite flakes with the main purpose of monitoring use-wear formation processes. The two main objectives of this research were the construction of a wide reference collection to serve for future functional interpretations of the archaeological material and to achieve a better comprehension of the mechanical behaviour of quartzite when subjected to the stress applied in determined prehistoric tasks (e.g., sawing, scraping bone, wood, etc.).

The two objectives are strictly related because the appearance of wear on the tool edges resulting from those tasks would be dependant on the mechanical behaviour of the rock in question. Concepts from tribology were used to provide an explanatory framework. As mechanical behaviour of solid materials always depends on their mechanical proprieties which are unique, each raw material should be treated individually in use-wear analysis. For this reason, there is an urgent need to create a reliable and objective system to identify and interpret wear due to use on quartzite. For data recording, we resorted to both optical and electron microscopes (OLM and SEM) to present a wide photographic documentation and to compare the adequacy and complementarity of those microscopic techniques for microwear studies.

Furthermore, both the experimental residues of the worked materials and the rock particles detached from the active edges were analysed to understand their role as interfacial medium affecting use-wear formation. EDX (Energy- Dispersive X-ray spectroscopy) was used to document the presence of rock particles detached from the tools edges and then embedded in the residues of the worked materials.

The results from analysing the experimental flakes allowed us to infer more closely the mechanical behaviour of quartzite. As a final point, the potential of OLM and SEM for analysing quartzite surfaces was evaluated and it emerged that the combination of the two techniques in an integrated approach is a feasible choice, though the application of SEM is always desirable in order to get more trustworthy results.

1. Introduction

Although use-wear analysis has been largely applied to determine stone tools' functionality, not so many efforts have been done to improve the methods for the analysis of non-chert/flint raw materials (from now on referred to as non-flint raw materials). Despite sporadic studies which provided specific methodologies to recognise use-wear on non-flint raw materials (Richards, 1988; Knutsson, 1988a; Sussman, 1988a; Hurcombe, 1992; Kononenko, 2011), as pointed out by Leipus and Mansur (2007:182), most of the contributions regarding lithic use-wear analysis have focused on the study of flint (Tringham et al., 1974; Keeley, 1980; Vaughan, 1985; Grace, 1989; Van Gijn, 1990; González Urquijo and Ibáñez Estévez, 1994; Levi-Sala, 1996). Nevertheless, non-flint raw materials have been occasionally considered for functional analysis, recently being the central object of sessions in international conferences (Clemente-Conte and Igreja, 2009; Sternke et al., 2009).

Quartzite, as other "secondary raw materials" like rock crystal (Alonso and Mansur, 1990; Pignat and Plisson, 2000; Plisson, 2008; Lombard, 2011; Fernández-Marchena, and Ollé, 2016) and rhyolite (McDevit, 1994; Clemente-Conte and Gibaja-Bao, 2009), have

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always received less attention by use-wear analysts compared to other lithic raw materials from which stone tools were produced in prehistory. In fact, basalt (Richards, 1988; Rodríguez-Rodríguez, 1997–1998; Asryan et al., 2014), obsidian (Mansur-Franchomme, 1988, 1991; Hurcombe, 1992; Kononenko, 2011), and vein quartz (Beyries and Roche, 1982; Sussman, 1985, 1988a, 1988b; Fullagar, 1986; Knutsson, 1988a, 1988b; Pant, 1989; Bracco and Morel, 1998; Derndarsky and Ocklind, 2001; Jaubert et al., 2005; Igreja et al., 2007; Derndarsky, 2009; Eigeland, 2009; Taipale, 2012; Taipale et al., 2014; Venditti, 2014; Knutsson et al., 2015) are much more known regarding use-wear appearance.

Although functional analyses involving quartzite have been previously performed by archaeologists, practically no specific experimentation focusing on this lithology has ever been undertaken on a systematic basis. We assume that within the framework of use-wear analysis, occasionally a reduced number of experiments on quartzite implements had been performed to provide data comparable with the archaeological record. Nevertheless, the resulting implications of such experiments were hardly ever investigated. Indeed, we noticed that as a prevailing attitude to deal with this methodological weakness (limited published experimental referential data concerning use-wear on quartzite), analysts generally applied the classical methodology developed for flint artefacts (either based on low or high power microscopy as well as on the combination of the two) (among others, Plisson, 1986; Alonso and Mansur, 1990; Pereira, 1993, 1996; Igreja et al., 2007; Leipus and Mansur, 2007; Hroníková et al., 2008; Igreja, 2008; Aubry and Igreja, 2009; Cristiani et al., 2009a; Gibaja et al., 2009). However, in few cases the intrinsic peculiarities of this rock were investigated, trying to evaluate the role of intra-raw material variability in use-wear formation and appearance (Beyries, 1982; Gibaja et al., 2002; Ollé, 2003; Vergès, 2003; Leipus and Mansur, 2007; Clemente-Conte and Gibaja-Bao, 2009; Ollé et al., 2016).

The extreme surface irregularities of quartzite, mainly due to its microcrystalline structure and the differential orientation of crystal surfaces, have always been regarded as a major obstacle by use-wear analysts (Grace, 1990; Mansur, 1999; Clemente-Conte and Gibaja-Bao, 2009). This difficulty was sometimes overcome by the use of DIC (Differential Interference Contrast) (Igreja, 2008, 2009; Cristiani et al., 2009b) and by the observation of the negative silicone moulds (Lemorini et al., 2014; Venditti, 2014) or of the positive resin casts of the artefacts' edges (Banks and Kay, 2003).

Some authors have also pointed out the advantages of Scanning Electron Microscopy (SEM) to avoid the light reflectivity of the rocks' surfaces and the problems of depth of field of irregular samples (Hayden, 1979; Grace, 1990; Borel et al., 2014). In fact, when this microscopic technique was applied on quartzose raw materials, results were characterised by an improved quality of the photographic documentation, resulting in a better comprehension of the use-wear appearance (Sussman, 1988a; Knutsson, 1998a; Carbonell et al., 1999; Márquez et al., 2001; Ollé, 2003; Vergès, 2003). This technique was also employed to monitor use-wear formation processes thanks to its high resolution capacities (Mansur-Franchomme, 1986; Yamada, 1993; Ollé and Vergès, 2008, 2014; Pedergnana and Ollé, 2014).

Some pioneers in use-wear analysis already highlighted the importance of well characterising the specific raw-material types related to one's assemblage (Keeley, 1974; Odell, 1975), probably because they had observed differences in the appearance of use-wear on the distinct lithologies. In fact, knowing that the mechanical behaviour of quartzite differs from that of chert and other lithic raw materials (because of structural differences) (Greiser and Sheets, 1979; Lerner et al., 2007; Lerner, 2014a, 2014b; Pedergnana et al., 2016), we recognised the need to provide a comprehensive use-wear experimental collection for this rock type.

Therefore, a large-scale experimental programme focused on the formation, identification and possible interpretation of use-wear traits on quartzite was initiated. The entire experimental programme was designed to monitor the processes of use-wear formation and the development of wear over time. Sequential experiments involving the use of replicas of the fresh edges were performed (Yamada, 1993; Ollé, 2003; Vergès, 2003; Ollé and Vergès, 2014).

Experiments were thought to serve as a reference for the study of the Middle Pleistocene sites of Gran Dolina (Sierra de Atapuerca, Burgos, Spain) (Ollé et al., 2013) and Payre (Southern France) (Moncel, 2008) and therefore comprising quartzite varieties coming from the surroundings of those archaeological sites.

The involvement of different quartzite varieties, exhibiting slightly different structural characteristics, allowed us to highlight analogies and divergences related to their mechanical behaviour when a force is applied and, as a consequence, to document differences in the wear appearance. The evaluation of the variability of the use-wear appearance is thought to increase the capacity of analysts to interpret use-wear on quartzite. The expected results are thought to improve the knowledge of the mechanical behaviour of this rock, and to make the experimental collection available for other use-wear analysts. With this main purpose in mind, particular attention was devoted to providing satisfactory photographic documentation, an aspect considered very important to allow other researchers to interpret the proposed data (Newcomer et al., 1986; Grace, 1996).

As a latter point, concerning the microscopic techniques employed within this study, we resorted to both optical light microscopy (OLM) and scanning electron microscopy (SEM). By a systematic comparison of these techniques their potential and reliability to record use-wear on quartzite was evaluated. As already demonstrated (Monnier et al., 2012, 2013; Borel et al., 2014), an integrated methodology commonly results in the best option, as the advantages of one technique overcome the disadvantages of the other. However, in the case of quartzite, SEM proved far better in fulfilling the need of the analysts providing images with a much higher resolution and higher magnifications. The limitations of optical microscopy in focusing high depth of fields when analysing very coarse materials such as quartzite are overcome by SEM.

1.1. A tribological approach

Perhaps the most evident obstacles in microwear analysis are the subjectivity of the analysts' observations and the scarcity of standards in terminology and methodology (Keeley, 1974). Those obstacles are frequently accompanied by a lack of interest in reaching a comprehensive understanding of wear formation processes. If the behaviour of the different lithologies is misunderstood, how can microwear analysts attempt to discern and interpret modifications of lithic micro-topographies? This concern was shared by some authors who incorporated concepts from fracture mechanics (Cotterell and Kamminga, 1979; Kamminga, 1979) and tribology (Knutsson, 1988a; Fullagar, 1991; Levi-Sala, 1996; Sala et al., 1998; Burroni et al., 2002; Ollé, 2003; Vergès, 2003; Anderson et al., 2006; Ollé and Vergès, 2008, 2014; Adams, 2014) in their researches.

Tribology (from the Greek *tribos*, rubbing) is defined as the study of contacting surfaces in relative motion and it deals with different aspects of materials' behaviour, such as lubrication, friction and wear (OECD, 1969). Although lubrication (when the two solids are separated by a lubricant) and friction (rubbing of one surface against another in dry conditions) intervene in stone tools' use, the main concern of the functional analyst is indeed "wear" (Semenov, 1964), which is defined as "the progressive loss of substance from the operating surface of a body occurring as a result of a relative motion at the surface" (OECD, 1969:64). Generally speaking, mechanical wear processes are sorted into abrasion, erosion, adhesion and surface fatigue, while corrosive wear is regarded to be a chemical process frequently acting in conjunction with mechanical processes (Hutchings, 1992; Kato, 2006). Abrasion or abrasive smoothing (Kamminga, 1979:151) has a predominant role in tribological systems, giving rise to different kinds of surface modifications (polished areas, linear features, grooves, pits, etc.). Abrasive wear occurs when there is displacement of material generating plastic deformations on one or on both surfaces (Ludema, 1996).

In experimentation involving lithic implements and generally softer worked materials, the displacement of material on the lithic surfaces is caused by their protuberances or asperities, which may remain stuck between the two surfaces and eventually accelerate wear (scratching or grooving the surface) (Hayden, 1979). When hard abrasive particles are present between the surfaces in relative motion, the intervening process is called scouring abrasion (OECD, 1969). When those particles are incorporated in a liquid and swept along in its flow, the process implicated in the formation of wear is called abrasive erosion. It is worth reminding that the interaction between sediments and lithic fragments produces surface modifications very similar to those due to use and sometimes it is very difficult to differentiate them (Keeley, 1974; Levi-Sala, 1986, 1996; Burroni et al., 2002; Pedergnana and Rosina, 2015). If we assume that abrasion is the principal process causing wear on lithic implements during use (being aware that other processes, such as surface fatigue and adhesion also intervene but with lower impact), the nature of the two materials taking part in the tribological system may affect the degree of wear development. In fact, this assumption would explain the clearly different visual appearance of wear on different lithologies.

Knowing that the rocks generally employed in knapping activity vary both in terms of chemical composition and structure, we may think that the effects of abrasion change in both extension and intensity. This is why, differences in use-wear appearance were documented also on different varieties of the same raw material (e.g. Levi-Sala, 1996). A possible explanation for that is that different rock types as well as different varieties of the same rock are characterised by distinct structures, which implies different physical properties and therefore a propagation of forces in different ways. The most important properties of solid materials are toughness, resilience, ductility and malleability. Resilience is measured by the quantity of energy a material can absorb when it is deformed elastically, while ductility and malleability are values connected to the ability of materials to deform under tensile or compressive stress respectively. Toughness is a combination of the latter two, which are both aspects of plasticity, the extent to which a solid material can be plastically deformed without fracture, and it seems to be more important than hardness in wear formation. In fact, as material hardness (i.e. the measurement of how resistant a solid matter is to plastic deformation or to fracture when a compressive force is applied) is basically the same for rocks mainly composed of quartz (Mohs scale hardness: 7); what changes instead is toughness, seen as the material's resistance to fracture when stressed (Hayden, 1979; Tiryaki, 2006). What defines the toughness value in crystalline materials is their crystal structure. Thus, it is connected to the disposition, orientation and compaction degree of crystals and it highly defines the way those materials fracture (Lawn and Marshall, 1979).

It emerges that, with the same amount of compressive stresses (e.g. during activities involving a knapped implement and a softer worked material) applied to lithic tools, the way of fracturing of different lithologies changes. Different fracturing models imply different amounts of fragmented particles, and as a consequence, more or less available abrasive material between the two surfaces. Thus, the less abundant the abrasive material, the slower the formation of wear is. The presence of external particles embedded in the residues would act as a "third body" of the tribo system (Hutchings, 1992; Ludema, 1996; Williams, 2005) and the quantity and size of those particles would be a crucial parameter to predict wear extension. Certainly, the hardness of the particles plays a very important role in this context. In fact, particles must have a hardness value equal to or higher than the surface they scratch. In a tribo-system composed by lithic surfaces and lithic micro-chips the hardness of the scratched surface and of the active particles is exactly the same.

Adhesive wear is also present, but to a much lesser extent, and it takes place when one surface removes material from the other, generally at the junction points between the two surfaces. The removed material normally adheres to the harder surface. An example of this wear can be seen in the presence of organic residues on tools' surfaces.

Finally, surface fatigue is another process described specifically to explain the formation of linear features (e.g. furrows, grooves, sleeks) on brittle materials as quartz (Knutsson, 1988a) and it is defined as surface or subsurface fracturing when a material is subjected to cycling loading.

2. Materials and methods

2.1. Quartzite varieties

Quartz, rock crystal and quartzite are different lithic raw materials but all are formed by macrocrystalline quartz crystals. Generally, the term quartz is employed in the literature to refer to vein (or milky) quartz, but sometimes it includes also the purest form of quartz (rock crystal), which technically is to be considered as a mineral and not as a rock.

Quartzite, in turn, is a very general term used to refer to siliceous rocks very rich in quartz content (at least 95%). The gradations of this lithology span a wide range of physical characters, including different varieties. A basic difference exists between orthoquartzites and metaquartzites considering the genesis of those rocks. Metaquartzites are true metamorphosed sandstones, while orthoquartzites have a sedimentary origin, being silica cemented sandstones (Tucker, 2001). The visual similarities between these two groups are shown in their mineral composition and the way they fracture. In fact, fracture propagates through the grains rather than around them (as in regular sandstones) (Andrefsky, 2006).

The flakes included in our experiments were manufactured from four different cobbles, with the major aim to limit the intra-raw material variability. Two varieties were subjected to petrographic analysis and the results defined them as metaquartzites (Pedergnana et al., 2016). Those varieties pertain to the Utrillas facies and were collected in the area surrounding the Sierra de Atapuerca (Burgos, Spain) (Fig. 1: 1, 2). The third variety (Fig. 1: 3) included in the experiments pertains to another facies, called Pedraja, and also comes from the same geographical region. It is a fine-grained quartzite, very likely a metaquartzite exhibiting a well-developed conchoidal fracture. A fourth variety (Fig. 1: 4) was collected in the Ardèche region (Southern France). Grain size is bigger compared to the other varieties and knapping fractures sometimes produce more irregular flaked surfaces. Although no specific petrographic study is available for this variety, it displays a high degree of grain compaction resembling that of metaquartzites.

The selection of these varieties is explained by the necessity to create a wide experimental referential collection to be later

Fig. 1. The quartzite varieties included in the study. 1–2) Fine-grained and coarse grained metaquartzites pertaining to the Utrillas facies (Burgos, Northern Spain); 3) Fine-grained metaquartzite pertaining to the facies Pedraja, 3) Coarse-grained metaquarzite coming from the Ardèche region, Southern France.

comparable with the Middle Pleistocene lithic assemblages coming from the Gran Dolina (Sierra de Atapuerca, Northern Spain) (Ollé et al., 2013) and the Payre (Rhône Valley, Southern France) (Moncel, 2008) sites.

2.2. Experimental protocol

A comprehensive experimental programme was carried out using these four different varieties of quartzite. The programme comprised both controlled and sequential experiments, although the majority of them pertains to the latter category (Table 1). 42 experimental flakes were used in 78 actualistic experimental sessions. All the experiments were performed under strictly controlled conditions and the majority of them was conducted by one of the authors (A.P.) in order to minimise changes in those variables which normally are the less measurable (e.g. the pressure exerted, the personal way of holding the instrument, etc.). The other author (A.O.) performed the tasks involving the treatment of large carcasses (skinning, eviscerating and separating the proximal parts of the animals from the axial ones). When exploiting this range of tasks, we were sometimes assisted by under-graduated students. Specific experiments were intended to mimic the movement of lithic artefacts in sediments and water. We reproduced similar conditions with the use of a mechanical tumbling device with the main aim to assess the aspect of microwear due to post-depositional events. The resulting wear has not been thoroughly analysed yet and compared with the appearance of wear due to use.

All of the experiments were recorded using photographic as well as videotape documentation. The latter was very

Table 1
Sequential experiments including different varieties of quartzite. Summary of the main controlled experimental variables. The number of strokes is always expressed in terms of real contact episodes with the worked material (one bidirectional stroke counts as two unidirectional strokes). The artefacts subjected to tumbling have not been yet microscopically analysed, but are included in the list of the experiments.

Experimental flake	Edge angle	Contact material	Type of contact material	Action	Contact angle[a]	Stages	Total time min	Approx. n. strokes
qtfu1-01	40°	Fresh wood	*Quercus ilex*	Sawing	60° < α < 90°	3	60	18000
qtfu1-02	45° < α 60°	Shed antler	*Cervus elaphus*	Sawing	90°	3	60	24000
qtfu1-03	40°	Skin, flesh, tendons, bone	*Cervus elaphus/Sus scrofa*	Cutting (uni)-Skinning/ Dismembering	80° < α < 40°	3	80	4000
qtfu1-04	45°	Fresh hide	*Cervus elaphus/Sus scrofa*	Scraping	60° < α < 30°	3	45	6000
qtfu1-05	50°	Fresh bone	*Cervus elaphus/Bos taurus*	Sawing	80° < α < 90°	3	60	18000
qtfu1-06	25° < α 30°	Fresh bone	*Cervus elaphus/Bos taurus*	Scraping	90°	3	60	7200
qtfu1-07	60°	Dry hide	*Cervus elaphus*	Scraping	40°	3	45	6000
qtfu1-08	30°	Fresh wood	*Quercus ilex*	Scraping	40°	2	30	3000
qtfu1-09	45°	Dry hide	*Cervus elaphus*	Cutting (bi)	90°	2	30	6000
qtfu1-10	35°	Giant cane	*Arundo donax*	Sawing	90°	2	45	13500
qtfu1-11	35°	Fresh hide	*Cervus elaphus*	Cutting (uni)	90° < α < 70°	1	10	1200
qtfu1-12	60°	Flesh, tendons, bone	*Cervus elaphus*	Cutting (uni)-Defleshing	90° < α < 80°	1	20	1600
qtfu1-13	40° < α < 30°	Wood	*Quercus ilex*	Multiple actions (sawing, scraping, whittling)	40° < α < 90°	1	25	2000
qtfu1-14	35°	Shed antler	*Cervus elaphus*	Cutting (uni)	90°	1	15	1000
qtfu2-01	55°	Fresh wood	*Quercus ilex*	Sawing	90°	4	90	27000
qtfu2-02	50°	Shed antler	*Cervus elaphus*	Sawing	80° < α < 90°	3	60	24000
qtfu2-03	35°	Flesh, tendons, bone	*Cervus elaphus*	Cutting (uni)-Defleshing	80° < α < 90°	2	85	6800
qtfu2-04	45°	Fresh hide	*Cervus elaphus*	Scraping	80°	2	30	4000
qtfu2-05	45°	Fresh bone	*Cervus elaphus/Bos taurus*	Sawing	80° < α < 90°	3	60	18000
qtfu2-06	40°	Fresh bone	*Cervus elaphus/Bos taurus*	Scraping	45°	3	60	7200
qtfu2-07	40°	Dry hide	*Cervus elaphus*	Scraping	40°	2	30	4000
qtfu2-08	50°	Fresh wood	*Quercus ilex*	Scraping	45°	1	15	1500
qtfu2-09	35°	Dry hide	*Cervus elaphus*	Cutting (bi)	90°	3	45	9000
qtfu2-10	45°	Giant cane	*Arundo donax*	Sawing	90°	2	30	9000
qtfu2-11	45°	Fresh hide	*Cervus elaphus*	Cutting (uni)	90° < α < 70°	1	10	1200
qtfp1-01	40°	Skin, flesh, tendons, bone	*Cervus elaphus/Sus scrofa*	Cutting (uni)-Skinning/ Dismembering	50° < α < 40°	2	60	3000
qtfp1-02	55°	Fresh bone	*Cervus elaphus*	Sawing	90°	2	30	9000
qtfp1- 03	75°	Fresh bone	*Cervus elaphus*	Scraping	60°	2	30	3600
qtfp1-04	70°	Fresh hide	*Cervus elaphus/Sus scrofa*	Scraping	80° < α < 70°	2	30	4000
qtfp1-05	45°	Wood	*Quercus ilex*	Scraping	60°	1	11	1000

Table 1 (*continued*)

Experimental flake	Edge angle	Contact material	Type of contact material	Action	Contact angle[a]	Stages	Total time min	Approx. n. strokes
qtfp1-06	45°	Wood	*Quercus ilex*	Cutting (uni)	90°	1	15	1000
qtfp1-07	30°	Shed antler	*Cervus elaphus*	Cutting (uni)	90°	1	13	1000
pay1-01	45°	Wood	*Quercus ilex*	Cutting	90°	1	13	1000
pay1-02	45°	Wood	*Quercus ilex*	Sawing	90°	1	6	500
pay1-03	45°	Wood	*Quercus ilex*	Scraping	$70° < \alpha < 60°$	1	6	500
pay1-04	50°	Wood	*Quercus ilex*	Planning	$50° < \alpha < 30°$	1	5	500
pay1-05	45°	Fresh hide	*Cervus elaphus*	Scraping	$70° < \alpha < 60°$	1	30	4000
pay1-06	45°	Fresh bone	*Cervus elaphus*	Scraping	$70° < \alpha < 60°$	1	7	500
TUMBL1-qtfu1	–	Sediment, water	-	Rolling	–		1800 (30 h)	–
TUMBL2-qtfu1	–	Sediment, water	-	Rolling	–		1200 (20 h)	–
TUMBL3-qtfu2	–	Sediment, water	-	Rolling	–		1800 (30 h)	–
TUMBL4-qtfp1	–	Sediment, water	-	Rolling	–		1800 (30 h)	–

[a] The contact angle is defined as the angle between the contact face of the lithic tool and the worked material.

useful for later review, including the counting of the strokes executed by lithic implements, a variable very difficult to monitor in the field. Therefore, an estimated number of strokes was provided for each tool, rather than only presenting the duration of use.

All the used edges were free from retouch and all tools were hand-held during the experiments. The possible motions were those commonly designated as basic modes of utilising tools (Tringham et al., 1974; Keeley, 1980; Grace, 1989), longitudinal (cutting, sawing) or transverse (scraping, whittling) to the tool's edge and the movement could be either unidirectional (one movement forward or backward resulting in a unique stroke) or bidirectional (reciprocal movements resulting in two strokes per movement). Cutting is used for unidirectional longitudinal movements (on any kind of material) and for uni or bidirectional movements when the worked material is soft (Keeley, 1980), while sawing always implies bi-directional movements but only on harder materials (bone, wood and antler).

The contact materials involved in the experiments were muscular tissue, dry (Fig. 2: c, d) and fresh hide (Fig. 2: a, b), fresh bone (Fig. 2: e, f), dry antler (Fig. 2: g), fresh wood (*Quercus ilex*) (Fig. 2: h, i) and reed (*Arundo donax*) (Fig. 2: l). All the animal remains were mainly obtained from ungulate carcasses (*Cervus elaphus*), although occasionally diverse species were included (*Bos taurus, Sus scrofa*). Butchering, defleshing and hide processing activities were performed at outside locations not only because of practical issues, but also considering that use-wear might develop differently within natural environments than under strict laboratory conditions. In fact, we neither attempted to hinder nor to induce the natural intrusion of extraneous particles such as sand grains between the tools and the contact material especially during hide scraping activities, which happened always on the ground.

Each tool was used on a single worked material and the angle to which it was held remained as constant as possible throughout the duration of the experiments. The angle recorded is the contact angle, that is to say the angle between the lower face of the tool (the contact face) and the contact material. The working angle instead is always dependant on the angle of the used edge itself (González Urquijo and Ibáñez Estévez, 1994:21). Therefore, we decided not to consider it in order to avoid confusion. We maintained all the aforementioned variables as consistent as possible throughout the entire experimental programme in order to obtain a certain degree of uniformity of wear production, which would later facilitate comparisons of use-wear traits recorded on the various quartzite varieties.

As previously stated, the experimental tools were mostly used sequentially, meaning through various stages of use (Márquez et al., 2001; Stemp and Stemp, 2003; Ollé and Vergès, 2008, 2014; Lerner, 2014b; Pedergnana and Ollé, 2014; Evans et al., 2014). Sequential stages consisted of activities carried out during 15, 30, 45 or 60 min, even though not all the experimental flakes reached the third stage of use. The duration of the activity may differ from those fixed intervals when for instance strokes were counted during the experiments. The combination of the stages may also vary (e.g. 15 + 30; 15 + 15+15 min; 15 + 15+30 + 30 min). Subsequently, all the implements were microscopically observed after each time interval as well as before use.

2.2.1. Moulding and casting

We moulded and casted the edges before using them to provide a record of their original appearance. After the first stage of use, we selected the control points to be monitored throughout the various stages of use, with a base on the wear traces observed there. Control points are defined as portions of the used edges which are observed before use and after the various stages of use with the major aim of understanding the dynamics of use-wear formation (Ollé and Vergès, 2014). Moulds (or negative imprints) (Rose, 1983) of the edges which were going to be subsequently used were prepared with silicon-based dental impression material (*Provil® novo Light*), by mixing the two components, a base and a catalyst in a ratio of 50%. Then the mixture was applied onto the tools using a metal spatula and left to dry for a few minutes (Fig. 3: a). Subsequently, casts (or positive imprints of the edge) were obtained filling the moulds with a bicomponent rigid polyurethane resin (*Feropur PR55®*) (Fig. 3: b).

Casts were analysed at the same magnifications used for the experimental pieces in order to compare exactly the same points before and after use. The systematic monitoring of some selected points allows us to observe the gradual changes of the edge due to use and, as a consequence, how use-wear forms and develops (Fig. 4). One of the advantages of having replicas of the fresh edges is that we are able to observe the original appearance of the edges and to add more control points, if needed, at any stage of use.

2.3. Cleaning protocol and sample preparation

Following each stage of use, each experimental flake was subjected to several procedures to remove all the adhering organic residues. The general cleaning protocol consisted in initially soaking each tool for 15–20 min in a bath of oxygenated water (H_2O_2)

Fig. 2. Different experimental activities to produce our reference collection: a) scraping fresh ungulate hide at an outside location; b) skinning an ungulate carcass; c) unidirectional scraping (backward) dry hide; d) cutting (unidirectional) dry hide; e) unidirectional scraping (backward) of an ungulate limb bone; f) sawing (bidirectional) an ungulate limb bone; g) sawing (bidirectional) dried antler; h) whittling (backward) a wood branch; i) sawing (bidirectional) a wood branch; sawing (bidirectional) a fresh giant cane stalk.

using a sonic cleaner (Fig. 3: d). This was followed by another sonic bath in a 2% neutral phosphate detergent solution for an additional 15 min. Residues of the detergent solution were removed by rinsing the tools under running water.

Flakes were soaked in a bath of 10–20% of hydrochloric acid (HCl) to finally remove any surviving residue, since some residues (e.g. bone, antler) proved to be quite resistant to cleaning. This last procedure is particularly necessary prior to SEM analyses because the surfaces analysed need to be thoroughly cleaned when observed at the high magnifications reached by this equipment. Finally, tools bathed for 30 min in water in order to remove any remaining traces of HCl. Following this long cleaning procedure and until the analysis took place, flakes were kept in clean zipped plastic bags and from this moment on contact with hands was avoided. Nevertheless, tools were subjected to a last sonic bath using pure acetone for a few minutes immediately before entering the SEM chamber, in order to remove modern grease due to accidental handling.

Furthermore, as most of the SEM observations were undertaken at high vacuum mode, samples needed specific preparation in order to enhance conductivity. With this purpose, tools were coated with a 30A thick gold layer by placing them in a sputter coater (EMITECH-K575X) for 3 min (Fig. 3: f). This was followed by the creation of a colloidal silver path from the metal stub to the upper surface of the tool. Alternatively, an adhesive aluminium tape specific for SEM sample preparation was used (Fig. 3: c).

2.4. Microscopy

For microscopic observations we combined the use of optical and electron microscopes, choice dictated by the coarseness degree of our samples. An outline drawing was made for each tool on which the position of any features and the location of any photographs were recorded.

Regarding optical observations, we employed a metallographic microscope (Zeiss Axio Scope A1) equipped with a Differential Interference Contrast (DIC) mounted with Nomarski prisms, using magnifications from 50 to 500×. Images were taken with a 5 MP DeltaPix digital camera (Invenio 5SII model) and multi-focused images were usually obtained using the DeltaPix Insight and the Helicon Focus software.

For reaching higher magnifications a JEOL JSM-6400 (Fig. 3: g) and an ESEM FEI Quanta 600 were used (from 15× to 5000×). Both of these microscopes were normally employed in high vacuum conditions. The SEM microanalysis system (energy-dispersive x-ray spectroscopy, EDX or EDS) was only used to analyse the elemental composition of residues or of the rock substrate. Both SEMs are

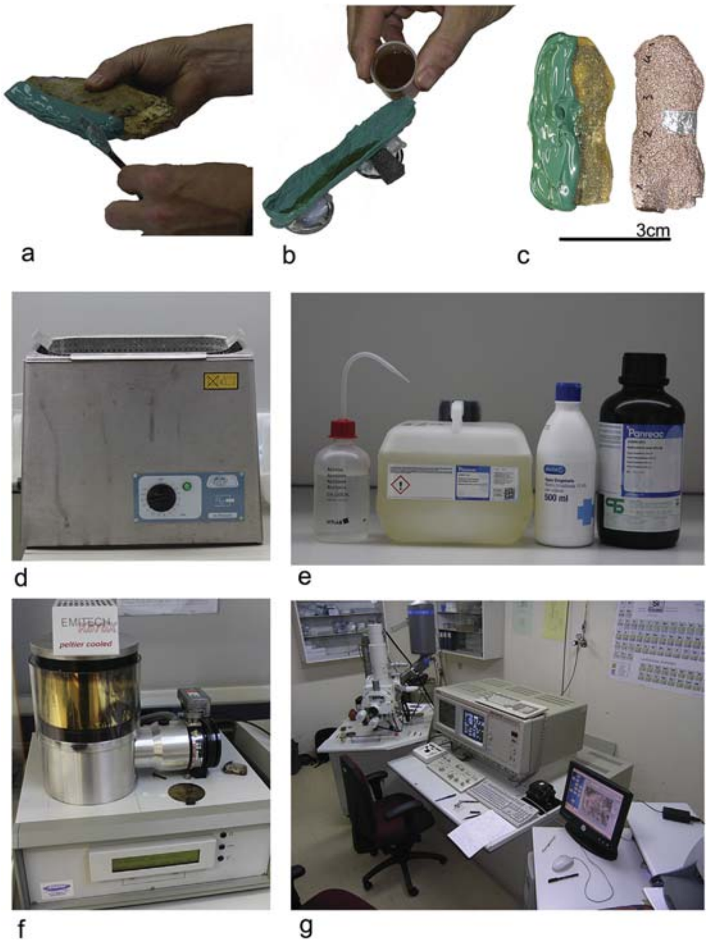

Fig. 3. The methodology employed: a) moulding the tools' edges before use with a silicon-based product; b) the obtaining of a positive cast from the mould employing a polyurethane resin; c) an experimental quartzite flake before removing the silicon mould from the edge and the same flake with the gold coating, as a preparation for high vacuum SEM observation. The aluminium tape enhances the conductivity of the sample; d) The ultrasonic tank used during the cleaning protocol; e) the chemical products employed in the cleaning processes: acetone, a neutral-phosphate soap, oxygenated water, and hydrochloric acid; f) the Sputter coater (EMITHEC K575X) used to coat the lithic surfaces with a gold layer, as part of the SEM sample preparation; g) one of the Scanning Electron Microscopes employed in this study (JEOL JSM-6400). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

equipped with an Oxford Instruments INCA system for digital image acquisition and treatment.

3. Results

3.1. Use-wear attributes on quartzite

Modifications of the micro-topography of lithic tools are defined as microwear. Those traits related to the activities carried out by prehistoric groups are called use-wear. Use-wear evidence has been traditionally described by means of different categories: edge damage, striations and polish (Semenov, 1964; Keeley, 1980; Kamminga, 1982). The variables associated to each of those categories can vary depending on the terminology adopted by individual analysts. All the use-wear features recorded in this study are principally due to abrasion and surface fatigue. The appearance of use-wear on quartzite is different from that on chert and its formation also differs, as a result of its specific mechanical properties. Quartzite is a tougher material compared to chert, which implies that it can absorb more energy before fracturing (Heiniö, 1999). Granulometry also plays an important role in the detachment of micro-chips, which in quartzite are normally bigger than those detached from chert artefacts.

3.1.1. Edge fracturing: micro-scarring

Micro-flaking, micro-scarring or micro-chipping are general terms to define small removals found all along the edge of the tools. They are conchoidal fractures which initiate from the margin of the tools and propagate on the surface resulting in specific shapes (Cotterell and Kamminga, 1979). More specifically,

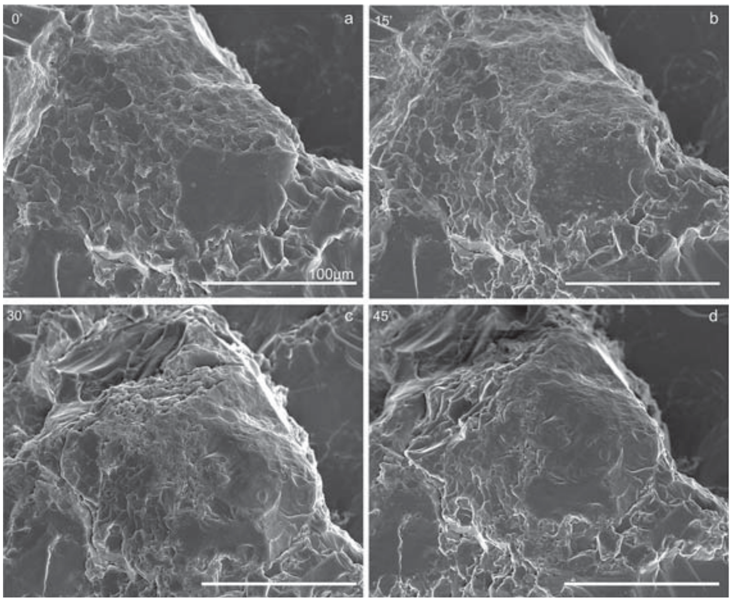

Fig. 4. An example of a four-stage sequential experiment after sawing a wood stick (Quercus ilex). The comparison of the same selected control points before use and after each stage of use permits the identification of the modifications that occurred and the observation of their development over time. A set of linear features parallel to the used edge developed on the smooth surface of a quartz grain (b) and then disappeared almost completely because of some grain ruptures (c). The surviving striations are then erased by the continuous friction with the worked material (d) (orig. mag.: 500×, scale bar: 100 μm).

edge scarring is defined as "any retouch on a stone artefact which is not the result of deliberate effort by man to modify the morphology of the implement" (Moss, 1983:231). Distinctions are generally based on the relative outline morphology, size and distribution of the scars (Tringham et al., 1974; Kamminga, 1982). They were considered good indicators of the relative hardness of the contact material and they are usually recorded with low power equipment (up to 40×) (Tringham et al., 1974; Odell, 1975, 1981; Moss, 1983).

Fracture mechanics help in understanding how cracks form. It seems that in isotropic materials (e.g. chert, obsidian, rock crystal) cracks propagate more homogeneously creating negative scars with distinct patterns (clear morphologies and terminations) (among others, Tringham et al., 1974; Kamminga, 1982; González-Urquijo and Ibáñez-Estévez, 1994; Rots, 2010) while in anisotropic materials (e.g. coarse-grained materials) cracks follow suitable paths through or around the grains (Lawn and Marshall, 1979).

This use-wear attribute is not regarded to be sufficient to distinguish among different worked materials, because of the high variability of the micro-scars' morphology as well as the overlapping of the same morphologies. Moreover, micro-chips are similar to both intentional retouch and to scars resulting from post-depositional processes, like trampling (Keeley, 1974; Levi-Sala, 1986, 1996:15; Mansur-Franchomme, 1986; Burroni et al., 2002). Scars formed after ancient and modern handling and transporting of the artefacts may cause confusion as well. Besides, if conchoidal fractures develop differently on chert and on coarse-grained materials, a direct comparison between scar morphologies found on those lithologies is not possible. Therefore, the raw material type can affect the extent and the shape of scars.

Furthermore, it has been stated that on quartzite the appearance of this particular feature is rare and occurs mainly when processing hard materials (Mansur, 1999; Gibaja and Carvalho, 2005; Leipus and Mansur, 2007; Igreja, 2008; Clemente-Conte and Gibaja-Bao, 2009). Comparing the edges before and after use, it is possible to infer that the processing of soft materials (e.g. reeds, hides) also causes a considerable loss of material, but without generating clear scars (Fig. 5).

An additional problem is the technical difficulty in documenting this attribute on coarse-grained surfaces with optical microscopes (Gibaja et al., 2002; Igreja et al., 2007). The detection of scars seems to be hindered by the particular structure of quartzite, particularly its considerably large and differently oriented quartz grains. Furthermore, micro-scars on quartzite are frequently found on the border of single grains, which are typically in the order of a few microns (thus only visible at high magnifications) (Fig. 5, f).

For all of those reasons, we marginally integrated this attribute within this study; however, an effort was made to evaluate the degree to which it is present on quartzite.

3.1.2. Linear features or striations

Striations are linear features appearing both on the original surface of the tools and on polished areas. They are regarded to be the most important indicator of tool use or kinematics (Semenov, 1964:17). A basic qualitative distinction between furrows and sleeks was made first by Kamminga (1979:192; 1982:12), even though terms such as scratches, lines, grooves and furrows appeared as early as the beginning of traceology (Semenov, 1964:15). The differentiation proposed by Kamminga was later adopted by other researchers and then maintained with slight terminological differences over time (Mansur, 1982; Mansur-Franchomme, 1986; Hurcombe, 1992; Levi-Sala, 1996; Derndarski and Ocklind, 2001; Fullagar, 2006).

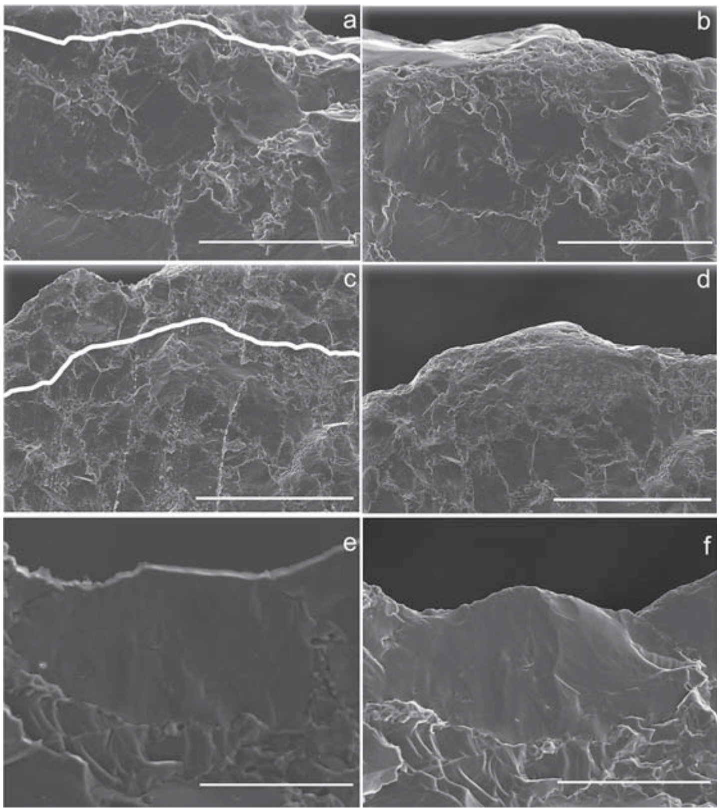

Fig. 5. Loss of material after edge fracturing. Three control points before use (a, c, e) and after 15min of use (b, d, f). White lines on the replicas' pictures mark the outlines of the edges after use. This helps in visually determining the loss of material from the edges themselves. Giant cane cutting: a, b) orig. mag.: 250×, scale bar: 200 μm; c, d) orig. mag.: 100×, scale bar: 500 μm; Fresh hide (*Cervus elaphus*) cutting: e, f) orig. mag.: 500×, scale bar: 100 μm.

This general differentiation is also adopted in this work. Regarding quartzose materials, striations are not so abundant (Semenov, 1964:15; Mansur, 1999:12; Leipus and Mansur, 2007:186) and they are generally better observed with SEM (Sussman, 1985, 1988a; Knutsson, 1988a; Levi-Sala, 1996). This is particularly true when analysing quartzite implements, mainly because of the irregularity of the surfaces. Secondly, striations appear to be quite shorter and narrower when compared to other raw materials with flatter surfaces (chert, obsidian, rock crystal): therefore, higher magnifications are needed to image them. The main observed linear traits on quartzite are defined as furrows (Fig. 6: a, c, d, h; Fig. 7: B, D). Furrows, also called linear grooves, are well described by Sussman (1988a: 13–14) as being "gouges or rough bottom striations, forming partial hertzian cones of percussion". The formation of those cracks on the flat surfaces of the rocks follows the same rules described for glass (Lawn and Marshall, 1979:70). Extraneous particles or fragments of the rock itself (Semenov, 1964:15; Brink, 1978; Fedje, 1979: 183; Kamminga, 1979:147; Kamminga, 1982:13; Mansur, 1982:216: Mansur-Franchomme, 1983:224; Sussman, 1988a:14; Levi-Sala, 1996:67) scratch the rock surface and lead to its inelastic deformation through a combination of compression and incident forces.

The models derived from ceramic behaviour (Evans, 1979; Hutchings, 1992; Ludema, 1996; Williams, 2005; Kato, 2006) provide an explanatory framework for understanding linear features found on lithic material. Cracks tend to initiate in the wake of the quartzite debris dragged along the flat surface of the quartz grains (Lawn and Marshall, 1979). This process is particularly evident in furrow striations and it is combined with surface fatigue, where lateral cracks form under the surface (Fig. 7: A). Those cracks trigger the detachment of small ellipsoidal fragments, whose negative marks determine the irregular aspect of furrows described in the literature (Mansur, 1982; Knutsson, 1988a; Taipale, 2012) (Fig. 6: c, d). Sleeks or regular striations (Hayden, 1979: XVIII; Fedje, 1979:1863; Kamminga, 1982:12; Levi-Sala, 1996:12; Fullagar, 2006:222) are seen very rarely on quartzite, predominantly on polished areas and exclusively by using SEM (Fig. 6: b, f). Sometimes, they appear as previous furrows which are then erased through a process of polishing. What survive are lineal scratches resembling what are described as sleeks in the literature.

To sum up, quartzite debris are regarded to be the main source for the formation of linear features on lithic surfaces. They participate in the tribo system as the third body, compressed between the

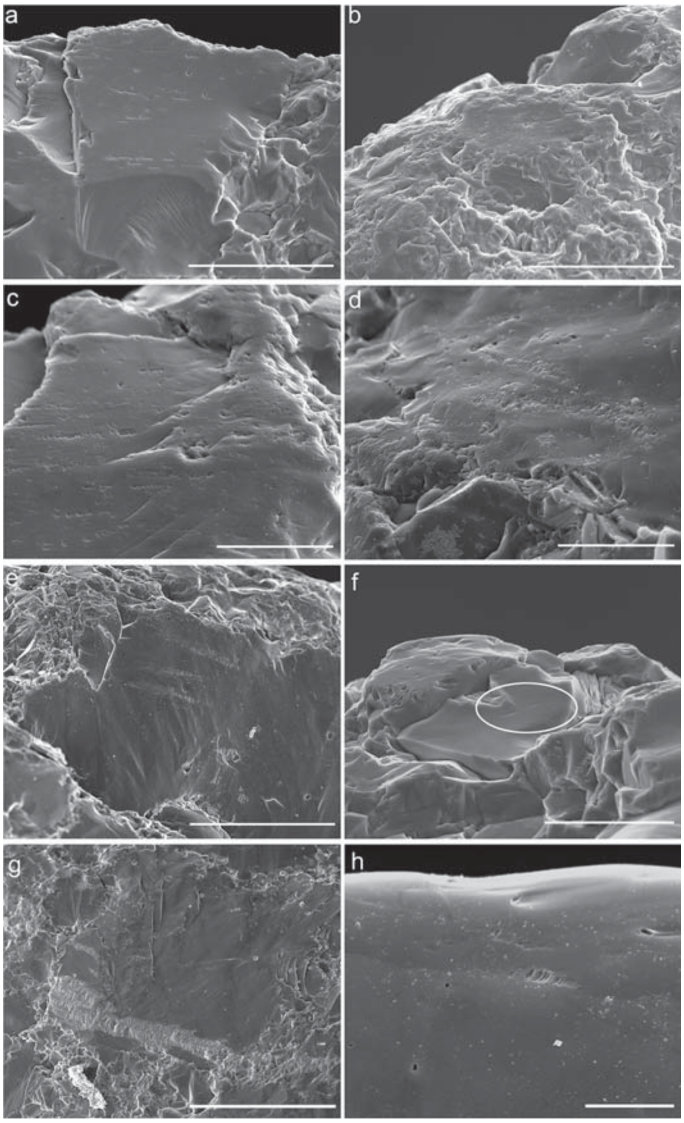

Fig. 6. Linear features on flat surfaces of quartz grains. Furrow striations found after processing wood (a, c), antler (e), butchering activity (d) and bone sawing (h); sleeks after working wood (b) and bone (f); irregular striation after sawing antler (g). a, b, e) orig. mag.: 1000×, scale bar: 50 μm; c, d) orig. mag.: 2000×, scale bar: 20 μm; f) orig. mag.: 1500×, scale bar: 30 μm; g) orig. mag.: 250×, scale bar: 200 μm; h) orig. mag.: 3000×, scale bar: 10 μm.

lithic tool and the softer material. They were frequently observed in all the organic residues included in the experiments (Levi-Sala, 1996). In one case, on one of our experimental flakes used to scrape fresh hide, they were documented through SEM-EDX (Fig. 8). Spectra of the micro-chips showed clearly the presence of Si and O (the main components of quartzose rocks), contrasting with the chemical composition of the residue.

3.1.3. Polish

Polish is defined as an alteration of the original structure of stone tools' surfaces (Keeley, 1974, 1980; Keeley and Newcomer, 1977). This alteration normally appears in the form of shiny or dull spots when using incident light microscopes. The visual differences of the appearance of polished surfaces led analysts to qualitatively distinguish between two main categories, smooth and

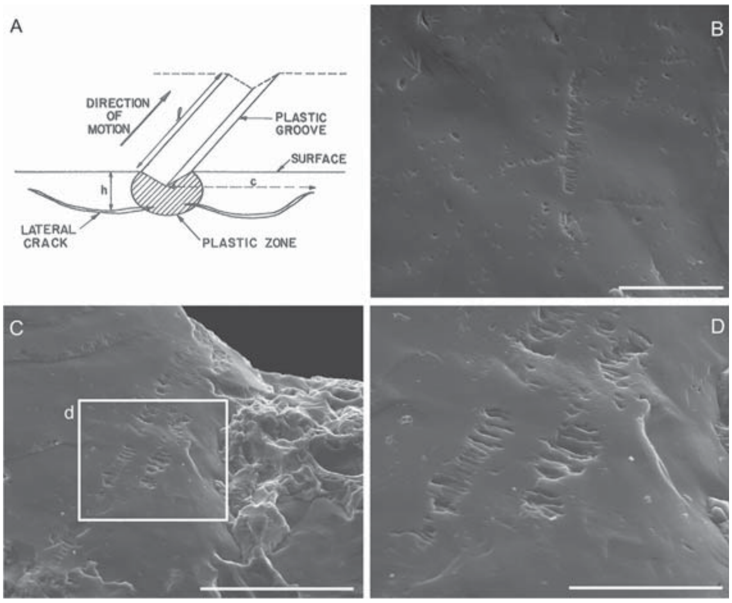

Fig. 7. Formation processes of striations. A) a general model developed within ceramic studies showing the formation of sub-surface lateral cracks in the wake of the particles dragged along the surface (after Evans, 1979); B) regular appearance of furrow striations at SEM, with the characteristic half-moon morphology of the superficial extractions; C) Particularly intermittent furrow striation found on a sample employed in butchering activities, probably formed after contact with bone. The transversal trend of this striation is explained by the use of the experimental tool to perform rare transverse strokes to remove the skin from the thorax. The tool was mainly use longitudinally; D) close-up of the same furrow striation showing the characteristic ellipsoidal micro-removals corresponding to the width of the sub-surface cracks previously formed. B) orig. mag.: 3500×, scale bar: 10 μm; C) orig. mag.: 1000×, scale bar: 50 μm; D) orig. mag.: 2500×, scale bar: 20 μm.

rough polish (Keeley, 1980; Vaughan, 1985; Mansur-Franchomme, 1986; Van Gijn, 1990; González-Urquijo and Ibáñez-Estévez, 1994; Juel Jensen, 1994; Rots, 2010).

Based on the variation in texture, extension, brightness and linkage of the polish areas on a tool, analysts claimed to be capable of distinguishing among different worked materials (Keeley and Newcomer, 1977; Newcomer and Keeley, 1979; Keeley, 1980; Vaughan, 1985). At the same time when polish was becoming the preferred attribute to discriminate the worked material, there were other scholars who considered the adoption of this single criterion to be a questionable solution (Holley and Del Bene, 1981; Newcomer et al., 1986; Grace, 1989, 1996). Particularly Grace (Grace, 1989) suggested caution when attributing distinct polish types to specific work materials, considering the fact that there are, as in the case of micro-scarring, significant overlaps in textural, reflectional and distributional patterns affecting polishes formed after the intervention on different worked materials. In fact, the same polish features would appear on tools after having been in contact with different materials and having been used during different time periods. Therefore, polish would be a continuum and would be indistinguishable. This is also confirmed by some authors whose functional interpretations normally relied on the polish characteristics. They stressed that polish areas in their primary stage of development are always quite similar, presenting no clear diagnostic character for the identification of the worked material (Vaughan, 1985; Mansur-Franchomme, 1986; Levi-Sala, 1996).

Polish on quartzite is not so well known and detailed descriptions of its appearance are hard to find in the literature. Nevertheless, it has been observed that the very irregular topography of quartzite does not allow polish to develop on very large areas, even after a long period of utilisation (Clemente-Conte and Gibaja-Bao, 2009; Stemp et al., 2013; Pedergnana and Ollé, 2014). This makes the conventional classification of different polish appearances quite useless. At any rate, polish is likely to be formed at least on the highest points of the micro-topography depending on the performed action, the elapsed time and the contact material (Mansur, 1999; Gibaja et al., 2002), even if in some cases polish was not documented on any of the analysed experimental pieces (Hroníková et al., 2008:356).

The conventional differentiation between smooth and rough polish is maintained in this work, but their definitions are based on SEM micrographs and therefore they do not consider the degree of reflectivity (Fig. 9; Fig. 10). On quartzite polished areas appear as extremely worn-out spots. This has been interpreted as the product of actual plastic deformation which is part of the general abrasive processes involved in surface polishing (Levi-Sala, 1996; Ollé and Vergès, 2008). That is why edge rounding (Fig. 9: b, d), frequently considered as a separate use-wear attribute, is here considered as a sub-unit of polish (Kamminga, 1982:17). Thus, the foremost visual parameter used to describe polish areas is surface roughness, which permits the differentiation of rougher and smoother areas.

Generally, rough polish is described as being dull when imaged with OLM and it is generally linked to hide processing, mainly in its dry state. Fresh hide generates flatter and brighter surfaces, similar to meat polishes (Keeley, 1980; Beyries and Rots, 2008). It is also characterised by the presence of pits, being topographical depressions (possibly the residual natural topography). By observing tools which were used sequentially, it is clear that polish always starts on the surface asperities and later flattens the lower parts (Fig. 9: c, d). Apart from the irregular topographical aspect of rough polish areas (Mansur-Franchomme, 1983), some spots of plastic

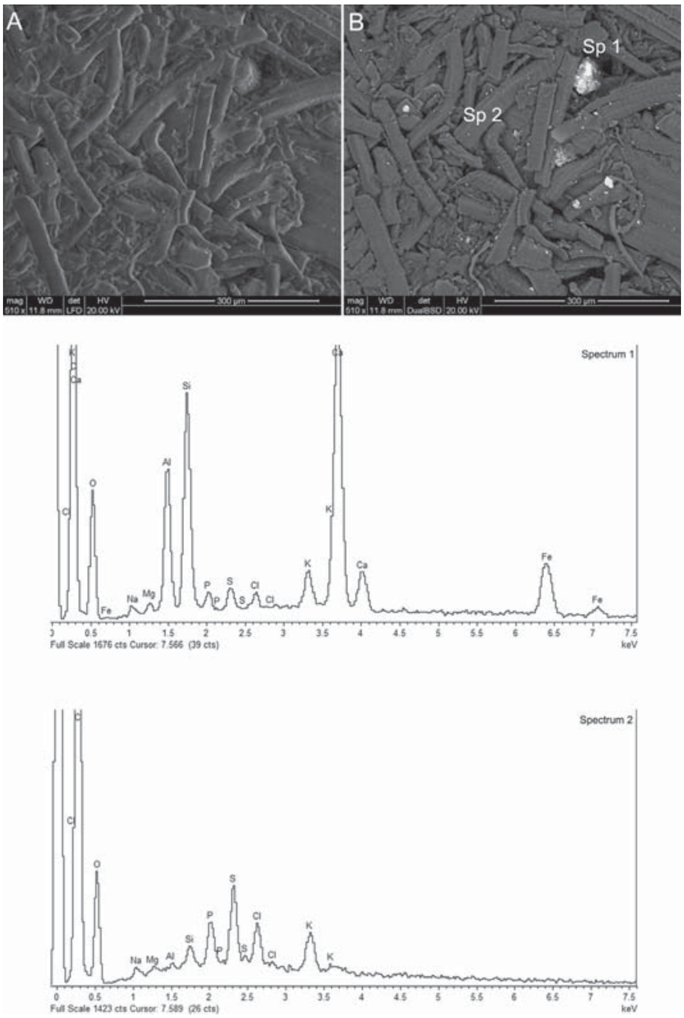

Fig. 8. Documentation of quartzite micro-chips within experimental residue of cervid dry hide (*Cervus elaphus*). These particles are thought to be responsible for abrasive wear production on lithic stone tools, acting as indenters. EDX-Spectrum 1 is related to one rock particle, exhibiting silicon (Si) and Oxygen (O) peaks. Aluminium (Al) sometimes appears as an accessory element of the quartzite variety to which the sample analysed pertains and luckily this element was recorded when analysing this rock particle. EDX-Spectrum 2 shows the elemental composition of the organic residue, including Sulfur (S), Phosphorus (P), Chlorine (Cl), and Potassium (K). Low-vacuum SEM micrographs taken with A) secondary electron detector and B) back-scattered electron detector (orig. mag.: 510×, scale bar: 300 μm).

deformation (displacement of material) are visible at the highest magnifications (SEM observations) (Fig. 10: a, b). Those areas display very flat surfaces and are a type of abrasive wear resulting from a three-body system including the friction between the lithic implements, the contact material and the micro-chips in between them.

Conversely, evenness is the main character of smooth polish surfaces. The original topography of the rock is erased by the flattening of all the asperities (Fig. 9: e, f). Additional characters, such as undulations (Fig. 10: c; Fig. 15), pits (Fig. 10: e; Fig. 14: d) or domes (Fig. 13: e, f) are seen within the smooth polished areas. This wavy or rippled feature on chert is associated with contact with wood (Vaughan, 1985; Mansur-Franchomme, 1986; Knutsson, 1988a), antler (Vaughan, 1985; Rots, 2010), bone (Vaughan, 1985) or even shell (Ollé, 2003; Vergès, 2003); while in our experiments on quartzite it was only found on polish formed after the contact with bone or wood.

The abrasive character of polish is evident from the observation of its development on flakes used sequentially. The modified areas firstly cover the highest parts of the original surface, levelling it out (Fig. 11: a, b). Over time, roughness decreases and the polished areas tend to be more even. However, the smoother character of the quartz grains highly contrasts with the rougher topography of the abrasive wear in LCSM images (Fig. 11: e, f).

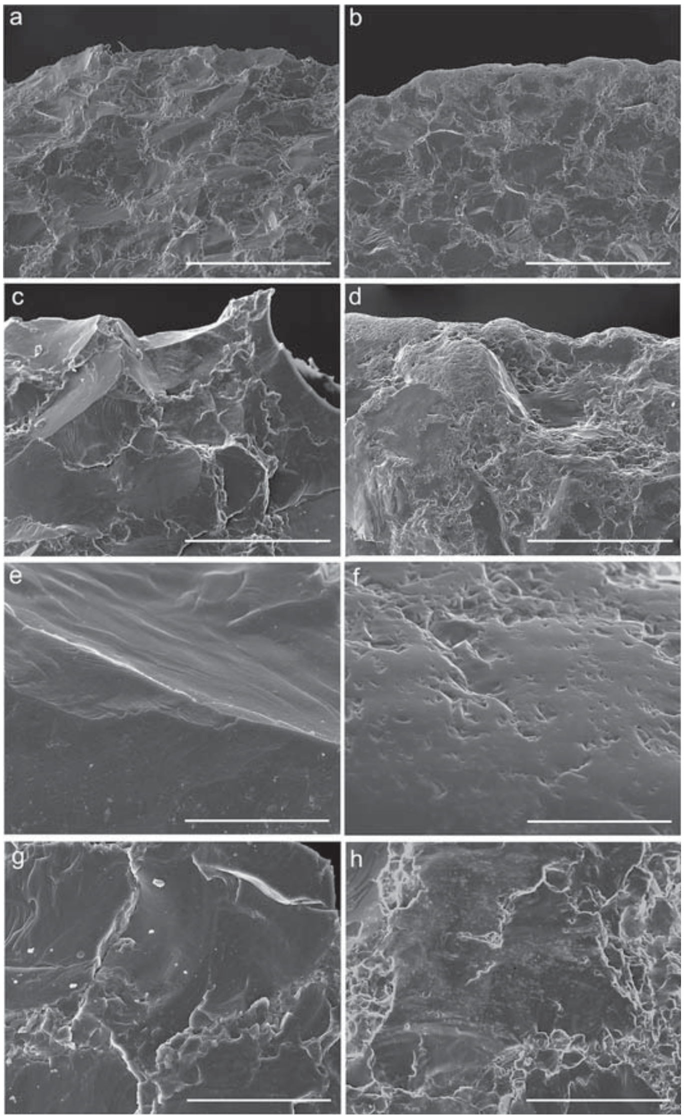

Fig. 9. Abrasive wear on quartzite flakes after working different worked materials (b, d, fresh hide; f, fresh cane and h, hard wood). Pictures on the right (b, d, f, h) reproduce some control points after the first stage of use (15min), while those on the left show exactly the same points found on the replicas of the edges and therefore, prior to any utilisation (a, c, e, g). Flaws in the form of striae or holes on the resin replicas are sometimes observable (c). All micrographs were taken at high vacuum mode and with a secondary electron detector: a, b) orig. mag.: 100×, scale bar: 500 μm; c, d) orig. mag.: 250×, scale bar: 200 μm; e, f) orig. mag.:1000×, scale bar: 50 μm; g, h) orig. mag.: 500×, scale bar: 100 μm.

3.2. Use-wear attributes and contact materials

3.2.1. Butchering activities

Butchering activities in the experimental programme included skinning, dismembering and defleshing medium-size animal carcasses (adult red deer and wild boar). To perform those activities the selected implements were naturally backed flakes or flake knives (one cortical side to facilitate prehension) (Fig. 2, b).

The skinning process was generally performed by one experimenter (expert or non-expert), assisted by two other people, and took no more than 20–30 min. Movements were mainly longitudinal and unidirectional, being bidirectional when needed (presence of tendons or ligaments). Sporadic transverse movements were also performed for instance when attempting to remove the skin from the lateral sides of the thorax. The activity always started by opening up the thoracic cavity of the animal from the sternum region to the hipbone. It continued with the removal of the skin

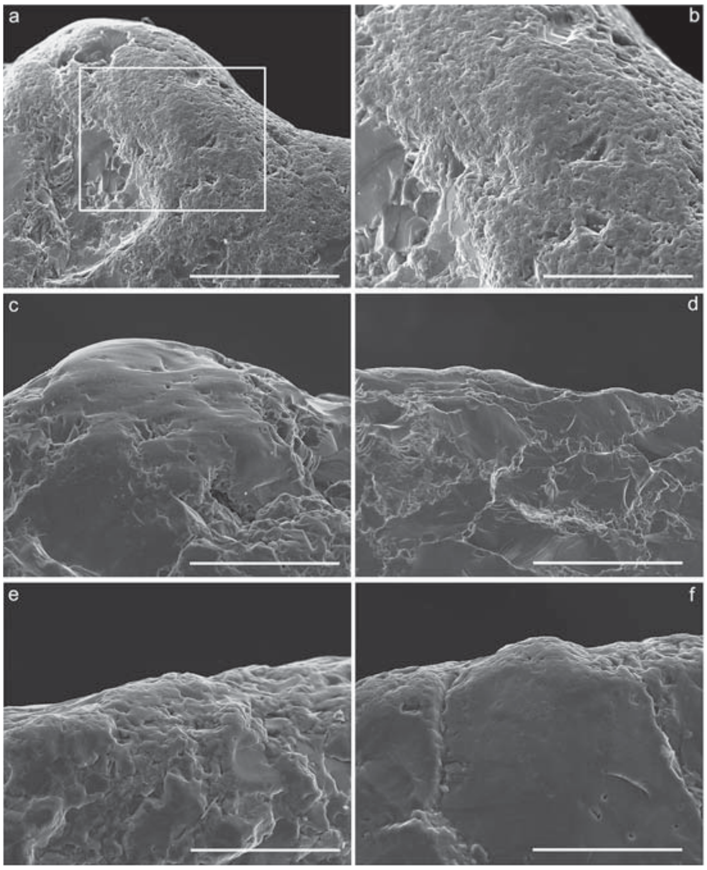

Fig. 10. Different visual appearances of abrasive wear on quartzite. Rough and pitted polish after scraping fresh hide (a). Detailed close-up of the topography showing small flat plastic deformations on the polished surface (b). Flat, smooth, non-pitted polished crystal after sawing bone distributed all along the edge (d). Undulations parallel to the edge are visible at high magnifications (c). Very smooth polish after scraping hard wood (e, f). a, c) orig. mag.: 500×, scale bar: 100 μm; b, e, f) orig. mag.: 1000×, scale bar: 50 μm; d) orig. mag.: 250×, scale bar: 200 μm.

from the upper and lower limbs and the back of the animal. The wider gestures, also implying a higher pressure on the lithic implement, were the strokes exerted to remove the skin from the joints of the extremities (hindlimb, forelimb). Before proceeding to dismember the animal, the internal organs were removed. Then, the four limbs and the head of the animal were separated from the trunk.

Subsequently, the limbs were defleshed by trying to separate the muscular tissue from the long bones. Movements were unidirectional, except for removing harder tissues such as sinew, when flakes performed occasional bidirectional strokes. The elapsed time to remove the muscles from one limb ranged between 20 and 50 min.

The average number of strokes per min was 50–55 for the skinning process and 80–100 for defleshing activities. During all the butchering procedures lithic tools had recurrent contacts with bone, which certainly increased the detachment of micro-chips from the used edges.

3.2.1.1. Skinning and dismembering: skin, muscles, sinews and occasionally bone. Use-wear is much more developed on the implements used for skinning than for cutting meat. After 15 min the loss of material was documented both in the form of micro-scars on the grains' borders (ca. 20–30 μm in length) and bigger scars (ca. 100–200 μm in length) (Fig. 13: b). The outline of the used edges was sinuous, typical of longitudinal activities.

No polished areas were detected after the first stage of use. After 30 min of use, a smooth textured polish begins to form on the topographical protruding asperities. The rim was slightly rounded, again displaying this particular smooth appearance. At the last stage of use, after more than one hour of use, polish was better developed, sometimes extending to lower topographical parts (Fig. 12: d). At the highest points, where polish started to form before and maintained the longest contact with the worked material, the aspect of polish changed, resembling very much that one formed on implements used to process hide. In fact, the texture of polish became quite rough, maintaining some tiny smooth spots,

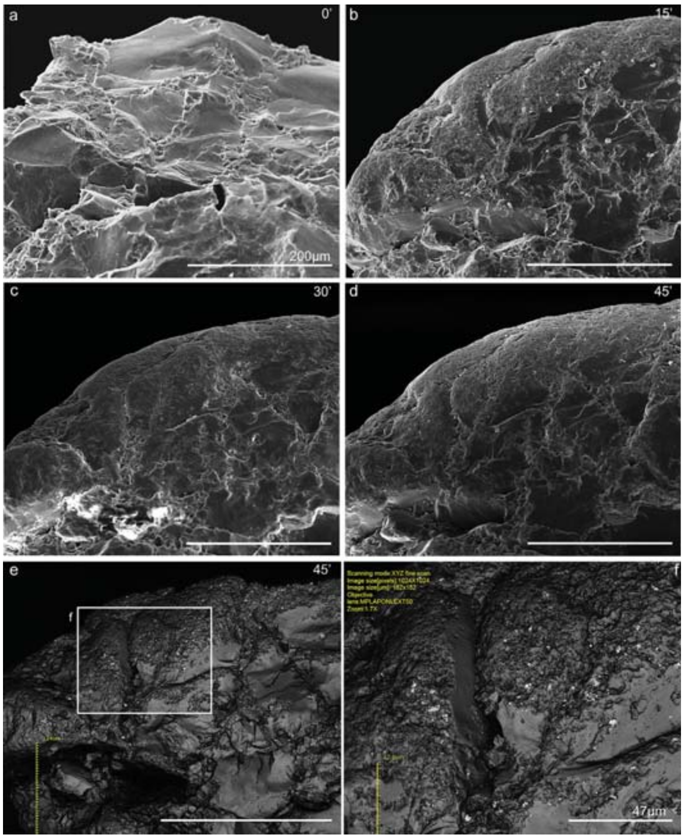

Fig. 11. The formation and development of abrasive wear on one control point after scraping fresh hide (qtfu1-04). Rough polish formed very rapidly after 15min of use (b) and then slowly expanded on lower areas (c, d). From this sequence it is evident that polishing is an abrasive process which tends to flatten the original surface of the rock from the higher points to the lower ones. A laser confocal microscope was used to obtain a better resolution of the roughness aspect of the polished area at 45min of use (e). A close-up of the same point displays the contrast between the original flat surface of quartz crystals and the modified surface (f). a–e) orig. mag.: 250×; scale bar: 200 µm; f) orig. mag.: 1000×; scale bar: 47 µm.

formed by plastic deformation (Fig. 12: e, f). Polish was relatively continuous on the rim of the used portion of the edges, sometimes becoming indistinguishable from that related to hide cutting activities.

Linear features were extremely rare and bore similar characters throughout the various stages of use, resembling those found on implements used to cut muscular tissue or hide, which is to be expected. They displayed the same morphological traits, but lengths were slightly longer (10–20 µm), reaching sometimes notable values (Fig. 13: d–f, 80.5 µm).

3.2.1.2. Defleshing: muscles, sinews and occasionally bone. Use-wear on implements used for removing muscles from bones is poorly developed. No polished areas were documented. Material removal is deduced by comparing used edges with their original replicas, measuring something between 50 and 100 µm. Striations are very rare. Only few superficial cracks with furrow-like morphologies, ranging from 1 to 5 µm and sometimes reaching 10 µm, were imaged.

3.2.2. Hide working

3.2.2.1. Scraping: grease, animal tissue residuals. Unidirectional scraping is intended to cleaning the internal side of fresh skins, removing remnants of flesh or subcutaneous layers which may be present depending on the butchered animal. When the skin is already dried, an additional scraping phase is needed to remove the sub-cutis to be able to further processing it. The average number of strokes per min in scraping actions on both fresh and dry hides was 130.

Use-wear on scraping implements was very similar regardless the state of material. Both fresh and dry hides appear to be highly abrasive materials. Fresh hide retained more quartzite particles than dry hide, which is harder, and therefore generated the detachment of more numerous chips from the edges of the tools but it was not capable of holding them. There are no significant differences in the distribution of the abrasive wear on the edges depending on the state of the worked material. For hide scraping activities, wear is found all along the used part of the edges, extending 150–200 µm at the points with more contact with the worked material (Fig. 9: b; Fig. 14: b; Fig. 21: a, b).

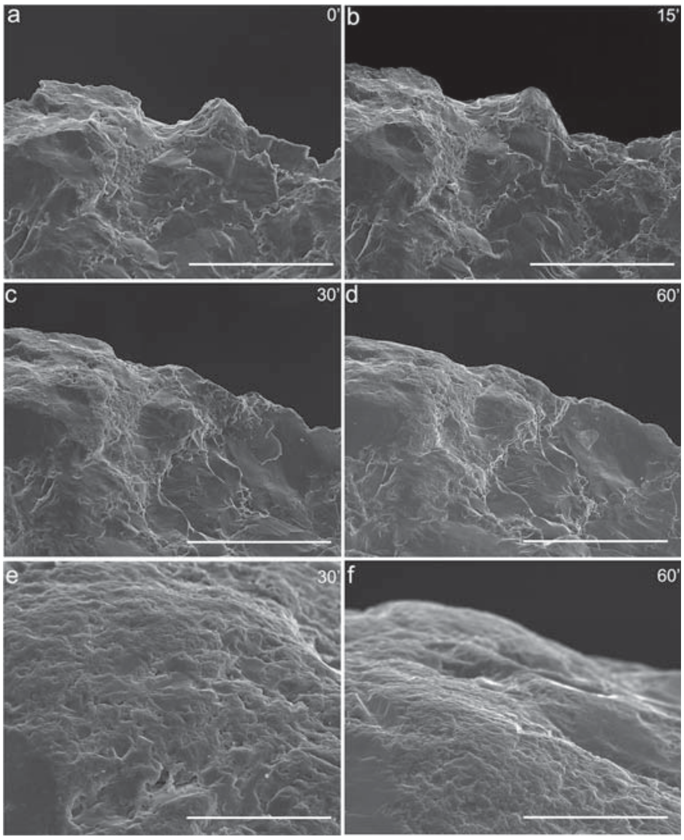

Fig. 12. Microwear related to butchering activities, sequential monitoring of the same point from the time zero (a) until the last stage of use (d). Micro-fracturing of the most protrusive areas led to a regularisation of the edge (c). Polish is poorly developed and present only on the highest topographical points after the first stage of use (b), slightly extending on larger areas at the end of the sequence (d). e, f) Details of the polish texture at higher magnifications and the loss of an edge's portion with wear on it due to fracturing (f). a-d) orig. mag.: 250×, scale bar: 200 μm; e, f) orig. mag.; 1000×, scale bar: 50 μm.

The only difference between "fresh and dry polishes" was texture. Both polishes were rough and pitted, but polish from dry hide seemed to be flatter, exhibiting more numerous and wider plastic deformations (Fig. 14: d), while plastic deformations on implements used to scrape fresh hide were evident only at very high magnifications (Fig. 10: b). It is curious that the appearance of "dry hide polish" closely resembles that related to butchering activities, while "fresh hide polish" slightly departs from it. One would expect the opposite situation.

We observed the progressive development of wear through sequential stages (Fig. 17: A, B). It seems that sometime after 30 min of use the edge stabilised and fracturing decreased. Therefore, we documented that most of the fractures causing the loss of previously formed wear (e.g. Fig. 17: A, 3) happened at middle stages of use. In fact, the increasing rounding gives the edge more stability, abrading the borders between grains, where fracture seems to normally propagate. Striations were extremely rare and when present they were shorter that 5 μm.

Additionally, apart from the characteristic edge rounding, lines of polish are sometimes present. Those worn areas are located on the higher parts of the relief and they are parallel to the used edge, being linked to the polish found on the rim (Fig. 17: A, 4).

3.2.2.2. Cutting: hide and fur. This specific task (unidirectional cutting of fresh hide and bidirectional cutting on dry hides) did not produce extensive wear. The average number of strokes per min was 120 for activities performed on fresh hides and 200 for dry hides.

Micro-scars (30–40 μm) on the borders of grains were documented, especially after dry hide cutting. Half-moon morphologies dominated. Striations are rare, as in other activities involving soft materials, though exceptionally big furrows were observed (dry hide, Fig. 22: e–h). Polish is found only on implements which cut dry hide and they were located on very tiny and prominent spots. Polish texture was smooth, not pitted, and resembled that found on implements used to remove muscular tissues from bones.

3.2.3. Bone working

3.2.3.1. Scraping. Use-wear is less extensive on flakes used to scrape bones than on implements used for the same activity on hide. The average number of strokes per min when scraping long bones was 120. The resulting polish was very smooth and it appeared only on high topographical parts of the surfaces. Hence, polished spots were sporadic and not linked together. They were mainly located on flat crystals and rarely were found on the smaller

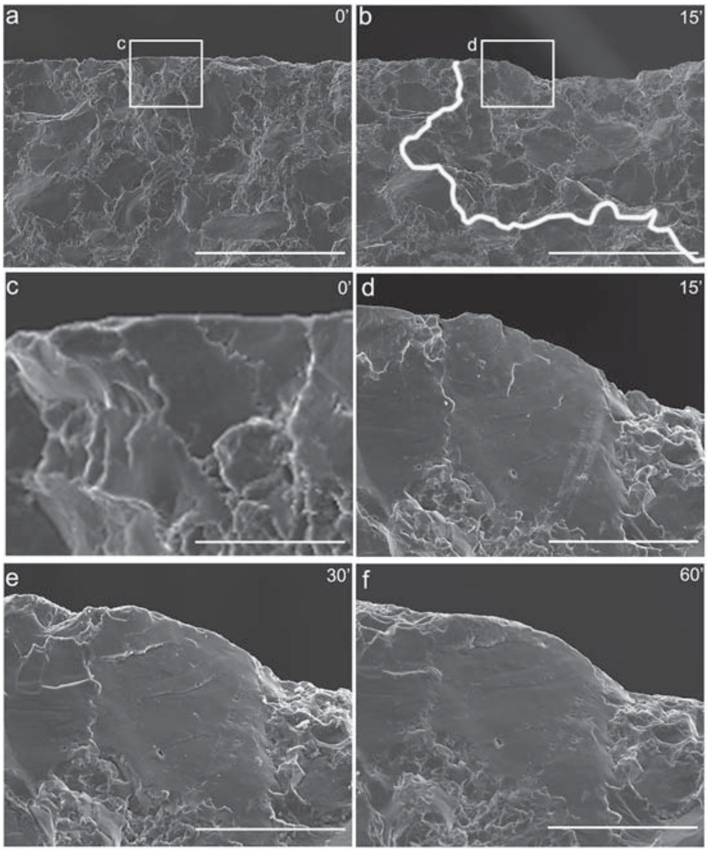

Fig. 13. Microwear related to butchering activities on a control point after three stages of use (15, 30, 60min). The documentation of micro-scars on the edge is only possible through a direct comparison with the edge's replica. A white line marks the outline of the scar (b). Because the fracture occurred in an early stage of use, a new quartz grain was revealed (d) and was not obviously present on the replica (c). This crystal suffered some modifications due to use, from the appearance of a quite large furrow (e) and the polishing of its upper border (f). a, b) orig. mag.: 100×, scale bar: 500 μm; c-f) orig. mag.: 500×, scale bar: 100 μm.

fraction (quartz grains measuring 10–30 μm). At a later stage the topography of the polished areas could be pitted. Striations were almost absent and very short, as in all scraping actions.

3.2.3.2. Sawing. Flakes used for sawing fresh bone displayed a typical asymmetrical edge profile. The average number of bidirectional strokes per min was 150 (300 real strokes). The zig-zag pattern of the edges is due to the micro-scars detached on both ventral and dorsal sides of the edge. The harder the worked material, the more pronounced this feature is. The high impact of micro-cracking was macroscopically visible during the experiments through the observation of the detachment of several microflakes. Under the microscope, edges appeared as vertically scaled off, as a result of the continuous formation of scars. Therefore, polished spots were preferentially distributed on the portions of the edges which escaped fracturing. Those parts are in fact the most prominent ones with respect to an imaginary rectilinear edge line. Polish was very smooth but not so well-developed; it was found only on the protruding crystals (Fig. 17: C). A characteristic trait found on pieces used to perform this activity is a wavy aspect of the polish topography. Longitudinal undulations, following the kinematics of the gesture, formed in early stages of use (15 min) and became more pronounced over time (Fig. 10: c; Fig. 15). The few striations were also observed on those prominent spots. Generally, striations were not present in the first stages of use and they were irregular and very short furrows (3-10mr) (Fig. 6: h). Some sleeks were also found sometimes on the polished areas (Fig. 6: f), but they seemed to be previous furrow striations which suffered a later process of polishing and then looked like grooves incised in the polished areas.

3.2.4. Antler sawing

Flakes were only used on dry antler; therefore, edge micro-fracturing was significant. The average number of bidirectional strokes per min was 200 (400 real strokes). Polish was not observed until after the first 30 min of work and occurred in very tiny spots when compared to bone working. It developed mainly on irregularities, as ridges or curves of the crystals, rather than on the flat surfaces. Texture was also different, resembling that related to wood working (smooth and pitted). Linear features can be very long (50–200 μm) (Fig. 6: g), but shorter (10–20 μm) and less irregular ones were also present (Fig. 6: e). The largest ones differed

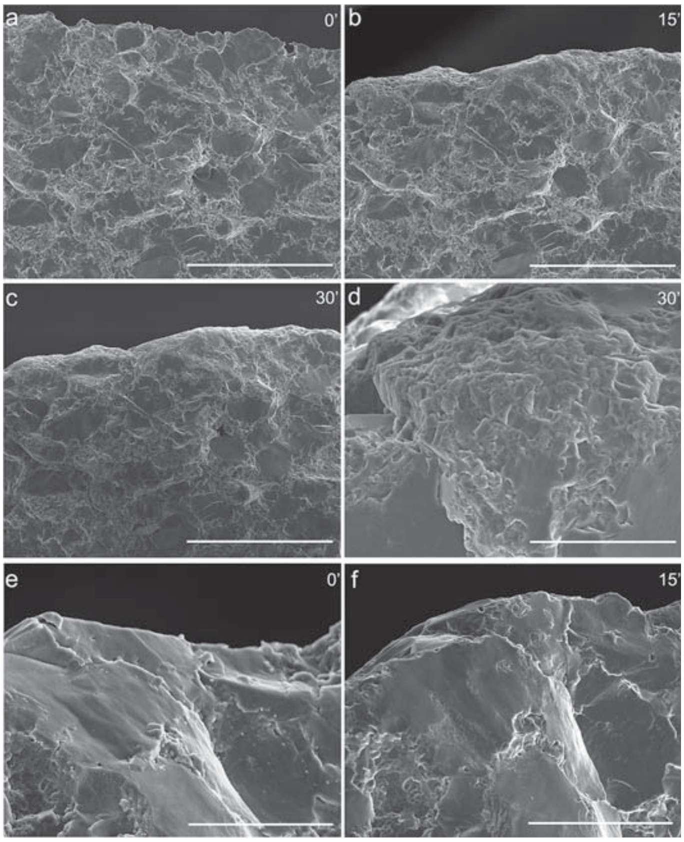

Fig. 14. Microwear related to dry hide scraping (b–d) and cutting (f). Fracturing does not significantly affect the general outline of the edge. The distribution of the rough polish is very continuous on the edge at an early stage of use (b), being more smooth-like after 30min of use (c). A detail exhibiting the presence of several smooth areas inside the general rough topography of the polish (d). a-c) orig. mag.: 100×, scale bar: 500 μm; d) orig. mag.: 1000×, scale bar: 50 μm; e, f) orig. mag.: 500×, scale bar: 100 μm.

in morphology from regular furrows, being more abrasive in nature. It is as if abrasion strongly affected the natural surface of crystals, possibly erasing cracks previously formed along the wake of the abrasive particles. No visible modification was found on the only experimental tool used to perform unidirectional strokes on antler (qtfu1-14). One possible explanation for both the poorly developed wear and also the large abrasive linear features is the state of the worked material. Dry antler is much harder than soaked, producing bigger rock chips detached during use. Bigger quartzite particles would produce longer and wider striations, like those observed on our experimental samples, and would have more difficulty in taking part of the tribo system as a third body. That means that they would flake off the edge almost simultaneously and would not be trapped in the antler residue; then they would not contribute to the formation of extensive abrasive wear.

3.2.5. Wood working

3.2.5.1. Scraping. Scraping hard wood (*Quercus ilex*) branches appeared to be more complicated than other materials. In fact, the average number of strokes per min was much lower (ca. 100). It is possible that this was due to the irregularities of the bark due to the presence of smaller branches and also to the difficulty encountered in holding the implements (not suitable morphologies).

Use-wear was poorly developed on implements used to perform this task. A very smooth and slightly pitted polish was present on tiny points on the very rim of the used edges (Fig. 10: f). This feature did not extend into the interior of the surface. Striations were almost completely absent; the few documented examples always measured less than 10 μm.

3.2.5.2. Sawing. Sawing hard wood branches is the action that absolutely created more linear features, mainly furrow type striations parallel to the used edges. After only 15 min of use striations covered entire flat surfaces of quartz grains (Fig. 16). On more irregular surfaces striations were less abundant and shorter. This abundance of linear features could be explained because the average number of strokes per min was very high compared to transverse actions (ca. 300).

After 30 min of use striations continue to form, but also a polishing process starts, erasing previously formed linear features. It is for this reason that abraded furrow striations, as in the case of wear connected to bone sawing, can be described as sleeks (Fig. 6: b).

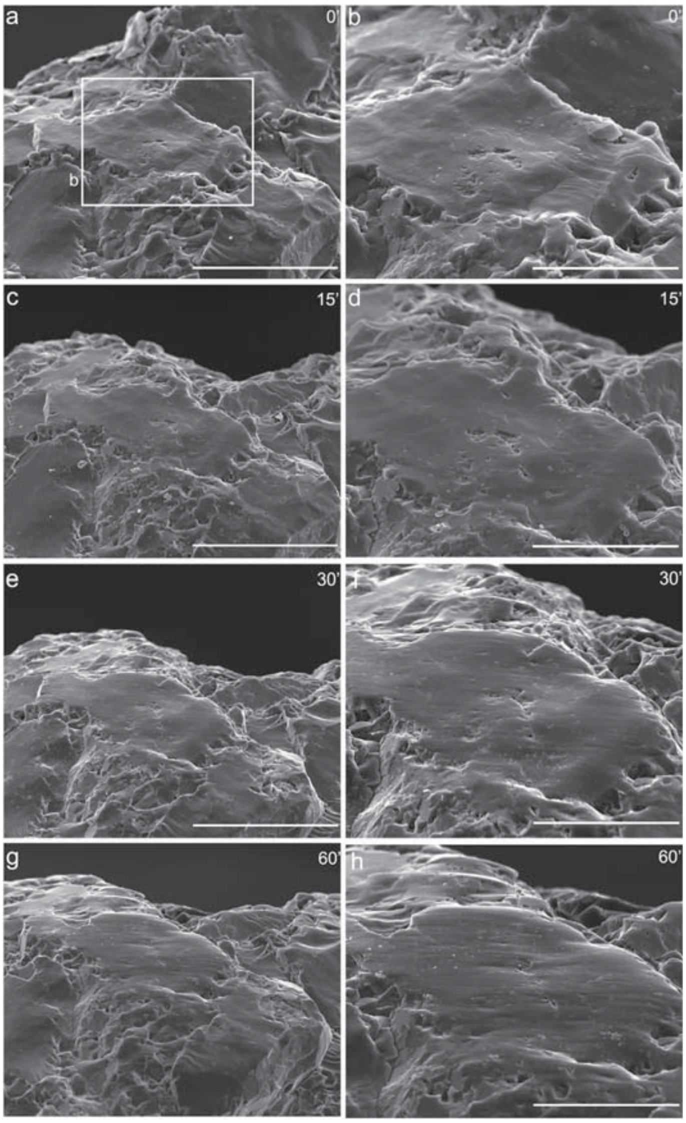

Fig. 15. Microwear related to bone sawing; sequence of the same control point imaged at two sets of magnifications. Depressions in the middle of the quartz grain are present before use. The progressive flattening of the surface is evident. Tiny furrows were documented after 15min of use (d), but were then erased by the abrasive process resulting in a very smooth polish characterised by clear undulations indicating the kinematics of the gesture (f, h). a, c, e, g) orig. mag.: 500×, scale bar: 100 μm; b, d, f, h) orig. mag.: 1000×, scale bar: 50 μm.

Very few flat polished spots were observed throughout the various stages of use and only after 45/60 min of use was polish well-developed. Texture was very smooth and non-pitted. Occasionally, after a prolonged action, some barely visible undulations (as those found on “bone polish”) were documented.

3.2.6. Reeds

3.2.6.1. Cutting. Bidirectional cutting giant cane stalks (*Arundo donax*) was the action which produced the most extensive wear in the shortest periods of time. The average number of strokes per min was 150 (real strokes 300). Non-woody plants have been registered

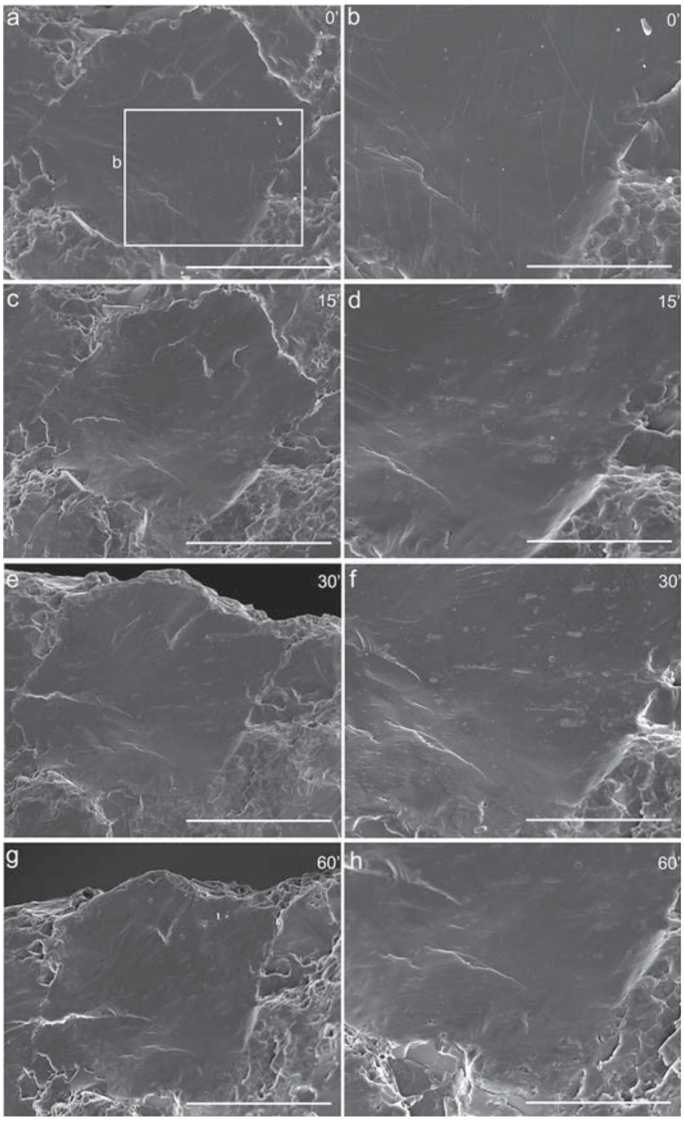

Fig. 16. Microwear related to wood sawing; sequence of the same control point imaged at two sets of magnifications. Fracturing of the edge and formation of linear features on the flat surface of a quartz grain (a, c, e, g). A white square (a) encompasses the portion showed in the detailed images of the same point at higher magnification (b, d, f, h). Furrow striations are very abundant and are always found on the surfaces of the quartz grains (d) and always tend to disappear due to continuous abrasion (h). a, c, e, g) orig. mag.: 500×, scale bar: 100 μm; b, d, f, h) orig. mag.: 1000×, scale bar: 50 μm.

as extremely abrasive materials, probably as a response to a high silica content (Mansur-Franchomme, 1986; Juen Jensen, 1994). In fact, on our experimental tools polish was extremely well-developed after the first stage of use (15 min) and it was distributed all along the used edges. Polish topography was very smooth and pitted. On the extremities of the used parts of edges polish was, in some cases, indistinguishable from “bone polish”, being very flat with no pits in it. Conversely, polish was always extremely pitted on the most used parts of edges (Fig. 9: f).

Notable differences of the appearance of polish were found on the different quartzite varieties. For instance, on fine-grained varieties polish was more developed and invasiveness was higher (200–500 μm), while on coarse-grained specimens it was mainly located on the borders of grains; it was very flat, non-pitted and not invasive at all (50 μm).

Striations were rare, short and the morphology of cracks composing furrows was generally more rounded and less ellipsoidal than those found on flakes used to work other materials.

Table 2
Presence (+) and absence (−) on the experimental tools of different use-wear features with regards to the worked material types. The number of crosses indicates the relative abundance of each trait: not abundant (+), abundant (++) and very abundant (+++). The determination of those categories is not based on a real quantification of the use-wear.

Use-wear features		Fracturing	Micro-scars on the borders of quartz grains	Furrow striations	Sleeks	Rough polish	Smooth polish	Pits on the polished areas	Undulations on the polished areas
Butchering	Skinning	++	+	+	−	−	+	−	−
	Muscles cutting	++	++	+	−	−	−	−	−
Fresh hide	Scraping	+	−	+	−	+++	+	+++	−
	Cutting	+	+	+		−	−	−	−
Dry hide	Scraping	+	−	+		+	++	+	−
	Cutting	+	++	+	−	+	++	+	−
Bone	Scraping	+	−	+		−	+++	−	−
	Sawing	+++	+	++	+	−	+++	−	+++
Antler	Sawing	+++	−	++	−	−	+	+	−
Wood	Scraping	+	−	+	−	+	++	+	−
	Sawing	++	+	+++	+	+	++	+	+
Reed	Cutting (bi)	++	−	++	−	−	+++	++	−

The visual grouping of the various features is intended only to facilitate the visual comparison among the different worked materials and will help for the future comparison with the archaeological evidence.

3.3. *Summary of the experimental results*

Evidences of the experimental use-wear were systematically sorted into the traditional main categories used in lithic functional studies. We attempted to group all the recorded features related to each contact material (Table 2). Grain size and grade of compaction of the different varieties of quartzite can produce slightly different use-wear, with distinct appearance and distribution (Pedergnana et al., 2016). What we noted is that the individual characteristics of the three varieties (grain size, morphology, etc.) might influence the wear pattern (considering the same variables, such as the elapsed time and activity). We assume that some of those slight differences would be measurable with quantitative methods, as demonstrated elsewhere (Lerner et al., 2007; Lerner, 2014a, 2014b).

However, the differences documented on the studied varieties were not so dissimilar to require individual descriptions. For this reason, the processes of use-wear formation on the different varieties can be explained as a whole. In fact, the use-wear traits related to each contact material appeared to be qualitatively consistent, despite the variety of quartzite analysed.

Although scars were difficult to observe and to image, the fracturing process of the edges was documented thanks to the comparison with the replicas of the unused edges. As other analysts pointed out (e. g., Tringham et al., 1974; Gibaja et al., 2002) the hardest contact materials (bone, wood) produce more fractures than the softest ones (animal tissues). We did not find any correlation between scar morphology and action performed or worked material. Moreover, micro-scars found on the border of quartz grains were unexpectedly associated to soft materials (hide, muscular tissue).

Linear features were present on tools intervened on all the contact materials included in the experiments. What differed was their morphology, length, and abundance. They can be all described as furrows, with a reduced incidence of sleeks, which were only found after sawing hard materials (bone and wood). However, the sporadic presence of sleeks cannot be indicative of the worked material because they might simply be abraded furrows.

Furrows are extremely abundant and relatively long on tools used for sawing wood branches and remarkably less abundant when sawing other hard materials (bone and antler). In all the performed activities, either longitudinal actions on soft materials or transverse movements (both on soft and hard materials), furrow striations are exceptionally rare and short. For transverse actions, the kinematics are generally better deduced from the linear patterns of the polish areas then from the few visible striations.

The other significant category is polish. Abraded areas can be described through the distinct visual aspect of topography. Rough polish is mainly found on implements which scraped fresh or dry hides, while smooth polish is characteristic of bone, antler and reed working. An intermediate state of the polish topography is described as being rough, but having numerous smooth spots on it and it is found in correlation with wood working and sometimes with dry hide. A particular trait, only found on tools which performed longitudinal actions on bone and more rarely on wood, is seen as an undulating pattern on polished areas.

Since the polishing process always implies a levelling out of the surface starting on the highest asperities (Levi-Sala, 1996:68), it is logical to think that at early stages of use polish is only found on the upper parts and only after a prolonged use it expands to the lower ones. We confirmed this after our sequential observations.

A very difficult aspect of polish to estimate is the presence of pits. However, some considerations can be done. Pits are very abundant and large on polished areas formed after the contact with hide and reeds and less abundant after the contact with wood and antler. Morphology and size of those pits might vary and their formation is not yet understood. For instance, it was proposed that they are the surviving parts of the original topography of the rock, after having suffered a polishing process (Levi-Sala, 1996). Although these descriptive attributes might help in the characterisation of different polish types, they are unlikely quantitatively measurable.

3.4. *Quartzite mechanical behaviour under stress*

Quartzite is an anisotropic material, characterised by a relatively high resistance to abrasion due to its extreme hardness and toughness. From this assumption, it is possible to infer that wear takes more time to form on it than on other less tenacious rocks as chert or obsidian (conceiving the tenacity as resistance to breaking or being deformed). The knowledge of the mechanical properties of quartzite let us to better understand how use-wear forms on it and it also allowed us to evaluate a possible life-cycle of quartzite tools. It is logical to think that, at similar external conditions (same contact material, the contact area, the applied load, number and direction of strokes, the slide speed, etc.) wear formation can manifest at different degrees of magnitude depending on the lithic material the tools is made of. Assuming that quartzite is tougher

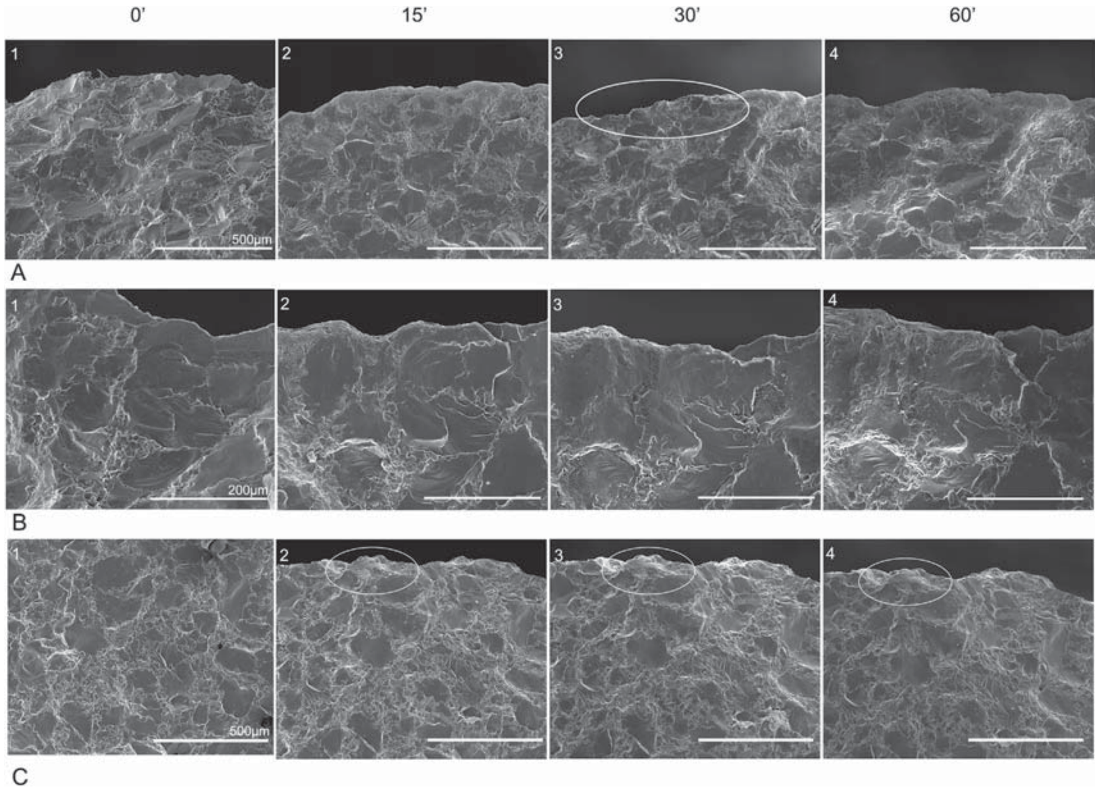

Fig. 17. Sequence of the same control points on flakes used for scraping fresh hide (A, B) and sawing bone (C). Fracturing is especially evident in the first stage of use (after 15min) and it is more invasive when working hard materials such as bone (C: 2). Fracturing is a constant throughout all the stages of use and sometimes affects wear previously formed (A: 3; B: 3). After losing the portions of edges bearing wear traces, the freshly exposed parts start to be worn out themselves at the next stage of use (A: 4; B: 4). White circles indicate the loss of wear from one stage or another (A: 3), or the undulations on bone polish only on the most protruding points of the topography (C). A, C) 100×, scale bar: 500 μm; B) 250×, scale bar: 200 μm.

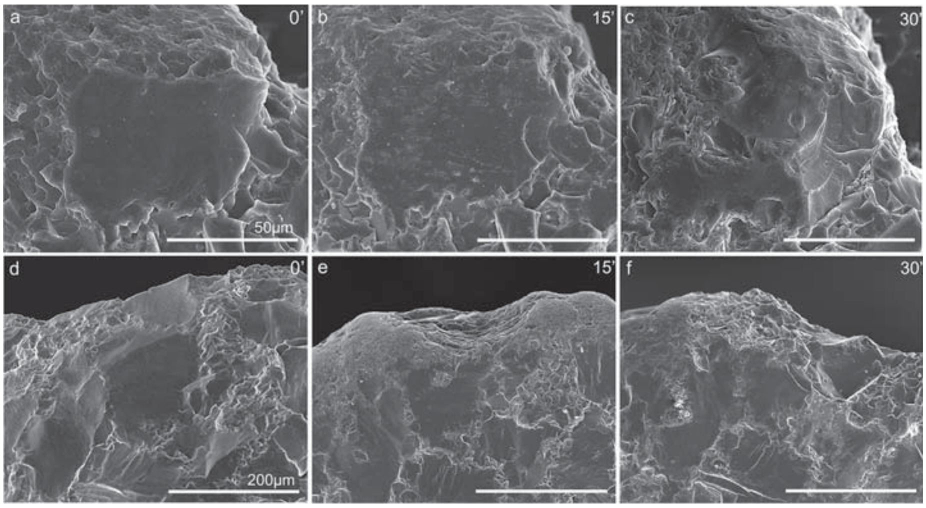

Fig. 18. Loss of edge portions bearing use-wear during the process of use. Invasive modifications of two control points happening during use render the points themselves barely recognisable (and only at low magnifications). From the second stage of use (15min) (b, e) to the third one (30min) (c, f) extended parts of the control points are lost due to quartz grain extractions. All micrographs were taken at high vacuum mode and with a secondary electron detector: a, b, c) wood working, orig. mag.: 1000×, scale bar: 50 μm; d, e, f) fresh hide scraping, orig. mag.: 250×, scale bar: 200 μm.

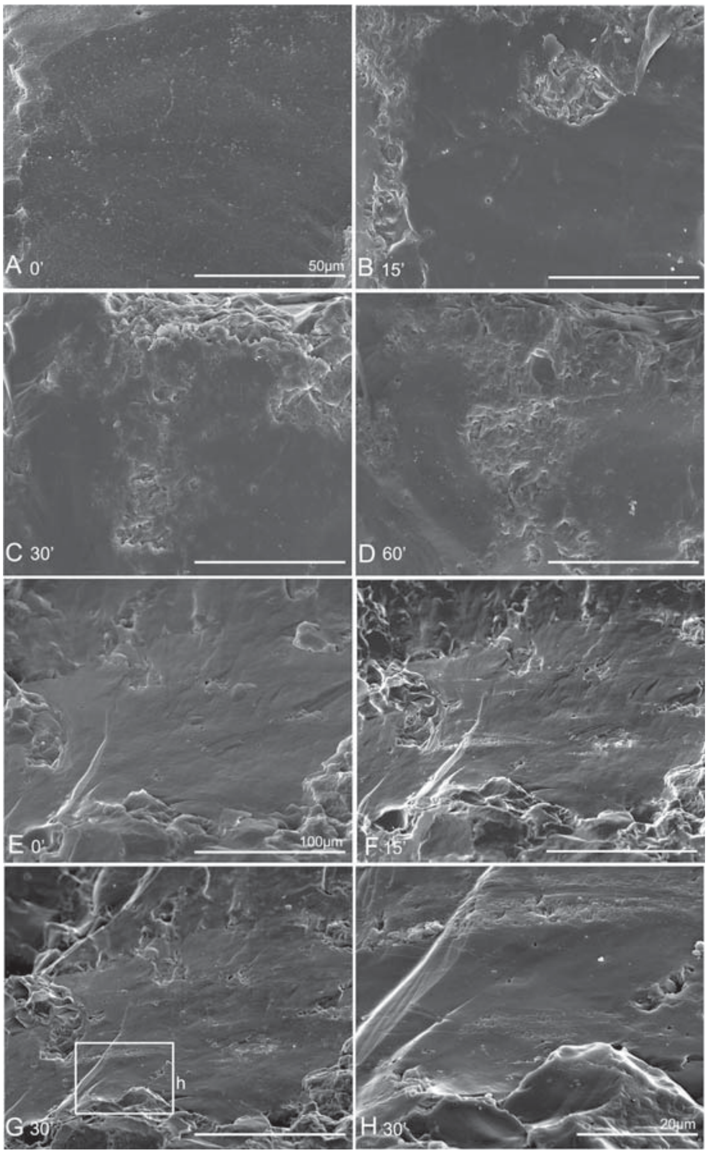

Fig. 19. A–D) a flat quartz grain observed after three stages of use (15, 30, 60min), after scraping fresh hide (orig. mag.: 1000×, scale bar: 50 μm). A possible impact removed a circular portion from the surface which created some short furrows, perpendicular to the used edge (B). Abrasion erased the linear features and circular removal (C), significantly extending the polished, flattened areas after one hour of use (D). E, F, G) another quartz grain observed after two stages of use (15min, 60min) for sawing a shed antler portion (orig. mag. 500×, scale bar: 100 μm). The depression in which this quartz crystal is located facilitates the preservation of the wear on it. H) Close-up of the striations showed in the G picture after the second stage of use (30min), being irregular furrows with linear grooves inside them (on the bottom) and very pronounced and wide abrasion line (on the top) (orig. mag. 2000×, scale bar: 20 μm). All micrographs were taken at high vacuum mode and with a secondary electron detector.

than chert, we can assume that a quartzite tool has a theoretically longer use-life than a chert one. Generally speaking, wear rates seem to be controlled by different factors: material loss, particle generation and the amount of retained particles (in the residues). The latter might be influenced by the state of the worked material, that is to say the amount of water present in the organic portions detached from it (and therefore being capable of retaining more or less lithic micro-chips).

However, as in any brittle material, the first appearing mechanical wear is edge fracturing. Entire portions of quartz grains are extracted as soon as friction with the worked material starts. Although present, scars are not easy to observe and document

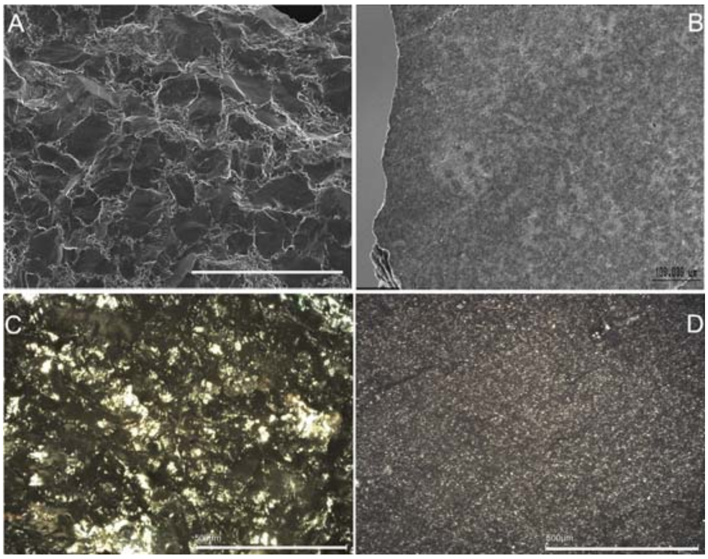

Fig. 20. Comparison of unused surfaces of quartzite (A, C) and chert (B, D). Differences in the granulometry are easily visible in the SEM pictures (A, B). Quartz grains in quartzite specimens range from 30 to 100 μm, while in chert they are quite smaller (3–10 μm). OLM pictures show not only the visual differences of textures, but also the high degree of surface light reflection of quartzite. This is due to the different orientation of the quartz grains. A–D) orig. mag.: 100×, scale bar: 500 μm (A, C, D), 100 μm (B).

because of the extreme irregular original surface of quartzite (Mansur, 1999:11; Gibaja et al., 2002; Clemente-Conte and Gibaja-Bao, 2009). Traces of this fracturing may not be so evident, but the progressive loss of material due to micro-fracturing is noticeably appreciable when the appearances of the edges prior and post utilisation are compared (Fig. 5).

Besides the aspects of the used edge (delineation and angle) (Moss, 1983), the impact of fracturing is directly dependent upon the hardness of the contact material. The harder the worked material the more fractures are produced on the edges. Compressive as well as tensile stresses contribute to the formation of scars (Lawn and Marshall, 1979). The singularity of scars present on quartzite edges is that they are not only propagating horizontally (producing flat conchoidal scars as in chert), but also vertically, thinning the edge by the continuous exposure of new surface areas. This was the cause of the loss of some experimental control points on our specimens (Fig. 18). Consequently, it is important to note that more developed wear on quartzite does not always indicate longer use times and that less developed or less abundant wear (see the erasing of striations) does not always relate to shorter usage. Also, worn areas related to some contact materials seem to provide equivalent roughness measurements despite the elapsed time (Evans et al., 2014). This would imply an additional problem, when it comes to the effort of providing functional reconstructions of the tools' life.

Afterwards, as friction continues, abrasion provokes edge rounding and polishing. Indeed, the rounding degree and wear texture depend upon the worked material, being highly developed in the case of hide processing (both fresh and dry hides).

In general terms, having noted that some use-wear features on quartzite tend to disappear or become less noticeable, and that this “vanishing” is directly proportional with the elapsed working time, we noted that use-wear formed during the various stages of use might eventually be replaced by fresh edge portions, due to the mechanics of quartzite (Fig. 18: b, d) (Clemente-Conte and Gibaja-Bao, 2009).

The original topography of the micro-surfaces is another important variable influencing the survival of use-wear traits. Flat crystals are very often subjected both to furrows and polish formation (Fig. 19, A–D). The subsequent surface smoothing might sometimes erase previously formed linear features, compromising the kinematic interpretation. Conversely, when linear features form in concave depressions, the chance of surviving after several stages of use increases (Fig. 19, E–H).

The hardness of the worked material is a very interesting variable related to the number and size of the rock particles detached from the tools' edges. The state of the worked material also seems to influence the wear rate by the capacity of including those particles in the third body of the tribo system, composed of the residues of the worked material itself plus the aforementioned rock chips.

Humid contact materials have higher adhering tendencies, therefore the more humid the worked material the more numerous the particles included and the more developed the resulting wear. This is why fresh hide, despite being a soft material, produces very well-developed wear in a short period of time (Fig. 11).

As the sliding motion goes on, the extreme edge rounding (which also increases the edge angle) might be responsible for the decrease in the effectiveness of the edge and the eventual abandonment of the tool. Extreme rounding also stabilises the edges, inhibiting the development of fractures. Clearly, in an ideal cycle of the lithic tool's use-life, the contact with the worked material and subsequently the mechanisms of wear production are suspended whenever the edge is no longer usable and loses its effectiveness.

4. Discussion

Since the foundation of use-wear analysis until the present, the most frequently applied technique to document microwear on lithic surfaces has been optical light microscopy. Despite the

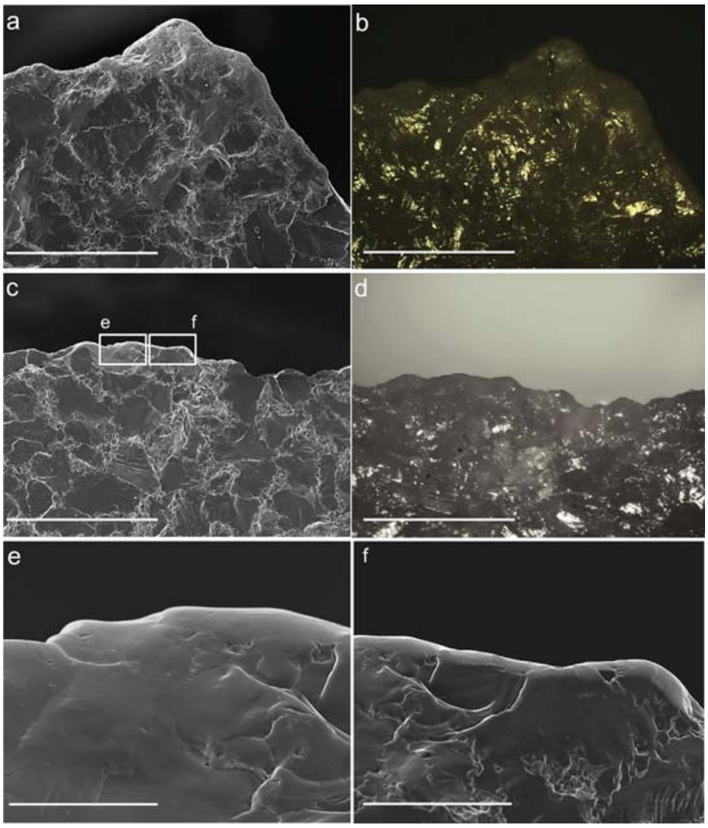

Fig. 21. Comparison of the same polished areas on quartzite imaged by means of two microscopic techniques. The texture and extent of a very well-developed rough polish spot (qtfu1-04, 15min of fresh hide scraping) are more visible with SEM (a) than with OLM (B). We face the same conditions analysing smooth polish due to bone scraping (qtfu1-06, 60min). The outline of the edge is not totally in focus even using the extended focus option (d). The SEM provided sharper details of the edge (c) and details of the smooth texture are appreciable at higher magnifications (e, f). For image b and d 15 slides were stitched together. a, b, c, d) orig. mag.: 100×; scale bar: 500 μm; e) orig. mag.: 1000×; scale bar: 50 μm; f) orig. mag.: 500×; scale bar: 100 μm.

previous works used to select one specific approach (low or high power approaches), more recent trends are dealing with a combination of low-power (stereo-microscopes) and high-power (metallurgical reflected-light microscopes) microscopy (e.g. Rots, 2010; Claude, 2012; Lemorini et al., 2015; Marreiros et al. (eds.), 2015). Sporadically, SEM was used to document both use-wear (e.g. Del Bene, 1979; Fedje, 1979; Anderson-Guerfaud, 1981; Kamminga, 1982; Masson, 1982; Meeks et al., 1982; Mansur-Franchomme, 1983; Unger-Hamilton, 1984; Sussman, 1985; D'Errico and Espinet-Moucadel, 1986; Knutsson, 1988; Longo, 1994; Levi-Sala, 1996; Christensen, 1998; Sala et al., 1998; Jardón Giner, 2000; Ollé and Vergès, 2008, 2014; Morales and Vergès, 2014) and residues (Clouse, 1979; Anderson, 1980; Tankersley, 1994; Shanks et al., 2001; Pawlik, 2004; Byrne et al., 2006; Cristiani et al., 2009a; Pawlik and Thissen, 2011; Vergès and Ollé, 2011; Monnier et al., 2012, 2013; Smith et al., 2015; Pedergnana and Blasco, 2016) on lithic implements. Although SEM was included in use-wear studies since the beginning of the discipline, its capabilities remained relatively under-utilised by lithic analysts and therefore its evident advantages were somehow underestimated.

In this work we evaluated the suitability of both techniques for the microscopic observation of quartzite surfaces. It emerged that, despite the fact that OLM is the most frequently used technique in use-wear analysis, this is not the best option when the tool analysed is made of quartzite. Differences in structure between chert and quartzite are evident at first glance (Fig. 20). Quartz grains in quartzite are bigger in size (50–150 μm) than those found in chert (few μm). General differences in grain size and morphology can be documented with the two microscopes, but the most evident difference is the way quartz grains reflect the light when observed with an optical microscope, as they have different orientations (Fig. 20: C). This significantly affects the detection of wear features, especially of polish. Polish was traditionally defined as a modified surface reflecting the light when scanned with OLM, but curiously polished surfaces do not shine at all when they are scanned with an optical microscope (Fig. 21: b, d). The most brilliant surfaces on quartzite are connected to those crystals whose orientation angle reflects the most incident light back to the microscope.

Texture of polish is much more discernible in SEM micrographs, thanks to its higher depth of field and magnifications compared to

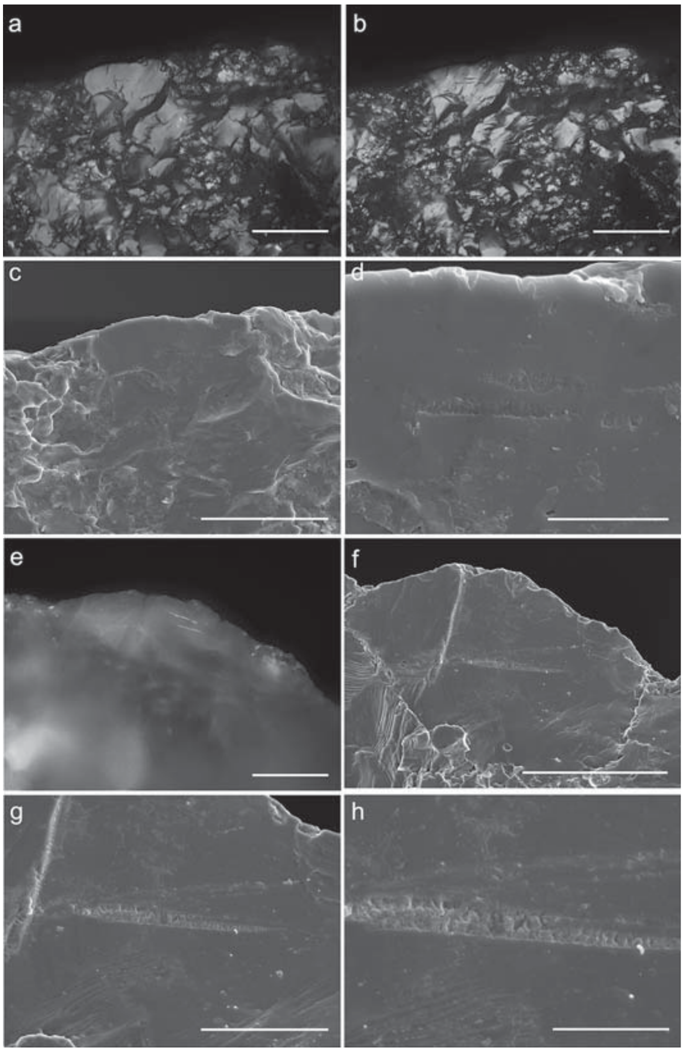

Fig. 22. Comparison of the same linear features on quartzite imaged by means of two microscopic techniques. One striation due to unidirectional wood cutting is documented through a reflective light microscope, with (b) and without (a) the employment of a DIC (Differential Interference Contrast), and with a SEM (c, d). Only through SEM observations is the furrow character of the striation visible (c, d). Another furrow striation measuring ca. 60 μm formed after bi-directionally cutting dry hide was imaged both with OLM (e) and SEM (f, g, h). Again, the detailed morphology of the striation is only noticeable in SEM pictures. All the SEM micrographs were taken at high vacuum mode and with a secondary electron detector. For images a and b, 8 slides were mounted and only 7 slides were used to compose the image e. a, b, e) orig. mag.: 500×, scale bar: 50 μm; d) orig. mag.: 4000×, scale bar: 10 μm; f) orig. mag.: 500×, scale bar: 100 μm; g) orig. mag.: 1000×, scale bar: 50 μm; h) orig. mag.: 2000×, scale bar: 20 μm.

OLM. Zoomed-in images of selected points allow the observation of detailed topographical traits of the polished surfaces, usually not seen through a conventional metallographic microscope (Fig. 21: e, f). SEM is therefore regarded as the best technique to image polished surfaces without losing important information, such as texture appearance, pits, undulations, etc.

Linear features are also better imaged through SEM, since higher magnifications permit better observation of those morphological traits. In fact, it has been stressed that a furrow can be misidentified as a sleek depending on the employed magnification (Fedje, 1979:181). For this reason, a thorough description of the appearance of sleeks and furrows together with the specification of the employed microscopic technique is necessary. The use of Nomarski prisms might help to avoid much light reflection (Igreja, 2009) and to better document this use-wear in some cases, but still the resolution obtained is not satisfactory (Fig. 22: a, b). Also, we noticed that several linear features on experimental flakes previously documented with SEM are not visible when the same implements are scanned with a regular reflected light microscope (max. magnification 500×). That means that only relying on OLM might negatively impact the results of use-wear analysis by underestimating the number of linear features (Borel et al., 2014). This difficulty is probably due to the optical limitations of this equipment when analysing coarse surfaces. In fact, light microscopes are designed to scan flat samples, therefore the higher the depth of field of the sample the smaller its in-focused portion. However, this obstacle is frequently overcome by the use of some software designed to combine several images, with different topographical regions in focus, of the same point of interest.

Only using OLM for use-wear analysis of quartzite could be misleading because of the light reflection of the samples and because of the technical problems discussed above. The use of SEM, even if it is better suited for the analysis of quartzite, may not be always possible given how time-consuming it is and its considerable cost; therefore, the combination of SEM and OLM could be a feasible solution (Borel et al., 2014). The selection of at least limited samples to be analysed with SEM would certainly improve the accuracy of functional interpretations.

The only analytical problem is that inferences based on both OLM and SEM images are always made through qualitative statements. Especially for the definition of polish, subjectivity plays a major role in its attribution to specific categories, such as “poor, medium, highly” developed, or rough and smooth.

Some researchers already tried to improve methods to quantify wear, from using digital image analyses (Grace et al., 1985; Grace, 1989; González-Urquijo and Ibáñez-Estévez, 2003; Lerner, 2014a, 2014b; Lerner et al., 2007; Mansur, 2009) to laser confocal microscopy, focus variation microscopy and laser profilometry (Evans and Donahue, 2008; Stemp et Chung, 2011; Evans et al., 2014; Macdonald, 2014; Stemp, 2014). Those techniques proved to be a valuable source of descriptive information. Especially for polish, defined as an improvement in surface smoothness (Williams, 2005:865), roughness measurement would be theoretically capable of providing numerical values of the amount of diminishing roughness compared to natural surfaces. The quantification of the roughness variation of the modified areas associated with specific contact materials would allow us to better characterise distinct polish types, which, until now, have only been visually described. Wear quantification will also help in the definition of the distinct use-wear appearance on the different varieties of quartzite. Even the thorough description of wear on each variety was not the main scope of this paper, qualitative observations were done in relation to the distribution of the worn areas, reaching deeper details concerning the varieties pertaining to the Utrillas facies (Pedergnana et al., 2016). Polish on the Pedraja variety seems to take more time to form and generally covers smaller areas, compared to the others; whereas on the French variety polish differences were more difficult to document, possibly due to the very irregular micro-topography.

It is clear that the resulting wear always depends on the original micro-surface of the different quartzite varieties. This is why, even if quantitative data are here missing, a good knowledge of the raw material is a fundamental step to provide a more reliable functional interpretation when archaeological collections are analysed. Nevertheless, characteristic traits related to each contact material are present on all the varieties analysed, which permit to provide a global description of use-wear on quartzite.

5. Conclusion

Sequential observations proved to be very useful and indispensable to investigate use-wear formation processes (Kamminga, 1979: 193; Yamada, 1993; Ollé and Vergès, 2008, 2014).

We believe that each raw material should be treated individually within functional studies, as different chemical composition and structures correspond to specific physical properties. Adopting concepts from material sciences seems to be the only path to comprehend the mechanical behaviour of rocks. In fact, tribology proved to be fundamental to deduce how quartzite surfaces are worn out when they are subjected to different stresses.

As quartzite is a brittle material and fractures often remove portions of the edge bearing use-wear traits, the absence of wear on archaeological implements is not always related to the absence of use. Also, as many use-wear features (especially striations) are subjected to a process of polishing until they eventually are completely erased, the limited presence of this feature is not a reliable parameter to infer the duration of use. This assumption is particularly valid for longitudinal actions, because for transverse ones the incidence of linear features is always very low (at least for actions without the addition of external abrasive agents).

Furthermore, quartzite not only differs from other rocks in the processes of wear formation (different development of the same use-wear attributes related to the same duration of the sliding motion between the tool and the same contact materials), but also in the appearance of wear. This happens because the original topographical structure of the rock strongly determines the aspect of wear. For this reason, quartzite requires specific descriptions of use-wear (other than the classical ones having chert as a main reference). Through the construction of an extensive use-wear reference collection including four different quartzite varieties we set up the basis for a reliable, analytical comparative method to interpret evidence on the archaeological materials. Hence, we categorised use-wear features associated to the various contact materials included in our experimentation by comparing SEM and OLM micrographs. The choice to employ both optical and electron microscopes was justified by the difficulties we encountered in the identification of use-wear using only optical devices, due to the physical limitations of this technique when applied to coarse-grained and highly reflective materials (like quartz and quartzite).

SEM seems to be the most effective technique to observe microscopic wear on quartzite, but as it could be difficult to have wider access to this equipment due to economic reasons, it is more conceivable to search for alternative solutions. A combination of different microscopic techniques, which could provide a compromise between effectiveness and efficiency would be preferable. Therefore, it would be desirable that analysts have a basic knowledge of various microscopic techniques to be able to select the most appropriate ones for each case study.

Furthermore, to improve the resolution capacity of functional analysis on quartzite, it would be worthwhile to include in the future new methods to quantify use-wear on quartzite (trying to correlate for example the roughness degree with the worked material), especially to try to differentiate distinct topographies of polish areas, which, until now, have only been described through qualitative features.

At any rate, we believe that as a first step the extensive photographic documentation we provided should be sufficient to limit subjectivity and to make our results beneficial to other analysts and comparable with other studies.

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5.2 Wear on quartzite

While there might be slight differences depending on the type of lithic raw material, the basic wear formation processes are very similar, as the solid bodies involved in such processes are analogous. First, the mechanics are evidently the same. The two solid bodies which always take part into the processes of wear formation on stone tools are evidently the stone tools themselves, activated by human force (both during Prehistoric and modern times) and the worked material. Stone tools are made by different kinds of raw materials, all being harder than the materials intervened with them (except when rock is worked).

The third solid body involved in what is normally defined as 'tribo system' in engineering sciences, is less obvious to think of. It consists of the small rock particles detached from the tool during use, due to the friction with the worked material. Although this is a quite acknowledged fact by use-wear researchers (*e.g.* Hayden ed., 1979), it has never been experimentally proven.

Within the frame of our sequential experiments, we aimed at documenting the said particles, claimed as the main causes of the formation of wear on stone tools (Brink, 1978; Kamminga, 1979; Mansur-Franchomme, 1983). During our systematic observations of the experimental residues, we succeeded in documenting some particles of the rock substrate, embedded into the residues (Fig. 5.1: a, b; Fig. 8 in Publication 5). By characterising these particles with the same methodology employed in the characterisation of the experimental residues, we have been able to visually isolate the rock particles and assess more clearly their role in the processes of wear formation. The EDX element maps correlate Silicon (Fig. 5.1: c) and Oxygen (Fig. 5.1: d) with quartzite, being therefore evident that the light particles observed comes from the rock substrate.

In fact, only a material having the same hardness as the rock itself would be able to scratch it, modifying inelastically. Because of this, if the only recognised explanation for the formation of use-wear is of mechanic nature (for more details, see section 5.1 and Publication 5), then the rock particles found mixed with the residues are the only possible agents to start and enhance the formation of wear on the surfaces of stone tools.

Having said so, obviously different rocks act differently, depending on their own intrinsic characteristics. Physical characters such as the hardness, toughness, disposition of grains, granulometry, etc. have a significant role in the development of wear (among others, Lerner et al., 2007). Thus, not only the type of rock, but also the different varieties would influence the formation and disposition of wear. In the case of quartzite, the role of such a variability seems to be a crucial point and deserves a deeper understanding. We think that, by only having a deep knowledge of the rock studied, can one truly assess the way wear forms, develops and distributes over the surfaces of stone tools. In this way, the functional interpretations of the archaeological evidence could only increase their degree of reliability.

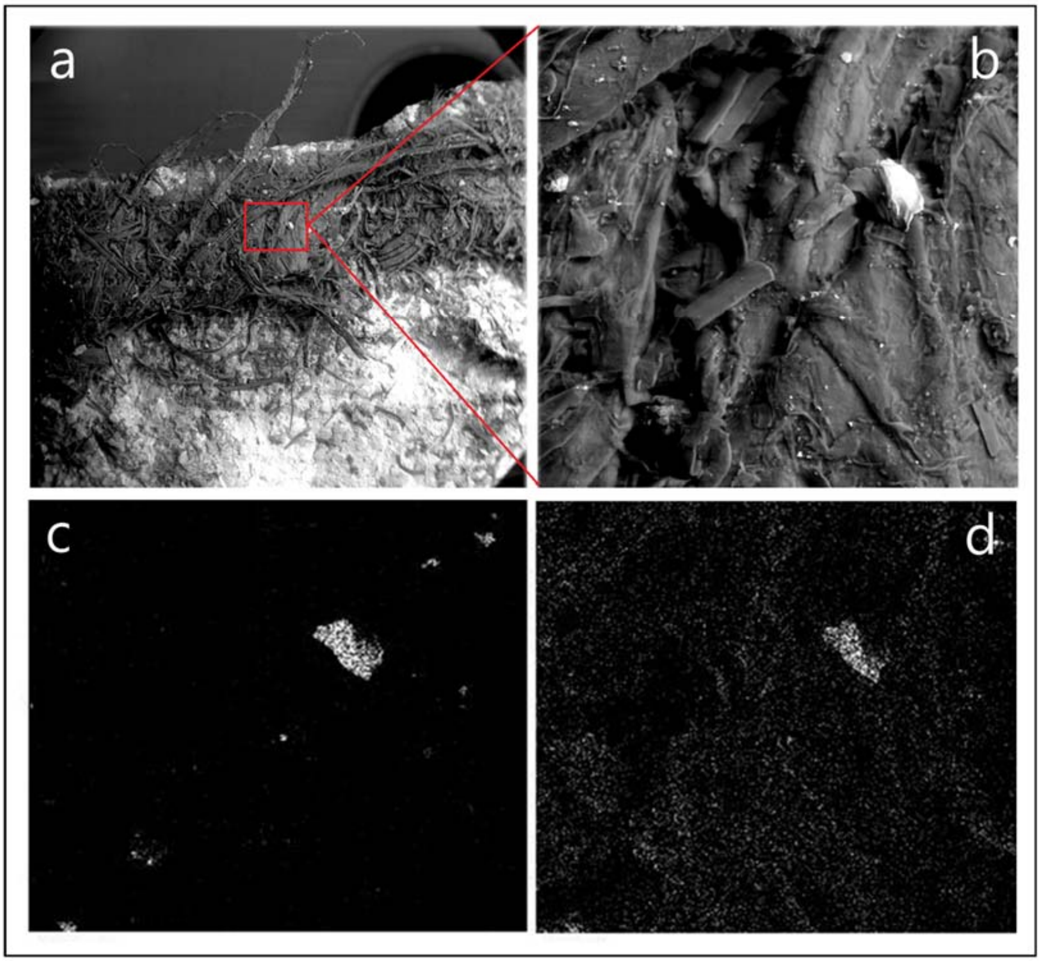

Fig. 5.1: *Rock particles embedded into experimental residues of dry skin and elemental map of one of such particles. a) General view of the residues, 60x; b) Close-up of the red rectangle in picture a, 510x; c) Si content; d) O content. Silicon and Oxygen are concentrated on the particle showed in picture b, contrasting with the surrounding residue.*

The quartzite varieties analysed in this study are quite similar, since all of them show comparable degrees of metamorphism. Very diverse degrees of metamorphism result in a huge diversity of topographical traits. As all the varieties analysed consist of metaquartzites, they are widely comparable.

We will then provide some general insights about the development and distribution of wear on the four quartzite varieties included in our experiments. All observations are subjective, and are based upon general visual estimations after the analysis of all experimental pieces. No specific analysis aimed to measuring the physical extent of wear, nor to monitoring the quantity of mass removal during wear formation has been carried out. Such measurements would require sophisticated equipment, generally found at engineering or physics departments.

However, we believe that general descriptions, though being subjective, are necessary for reaching a deeper understanding of the materials analysed. Moreover, they are useful to researchers dealing with coarse-grained material as a matter of comparisons of their own observations with those of other researchers.

The first two varieties considered come from the same geological formation, the Utrillas facies, and they show comparable features (Publication 3). The third variety comes from a different formation, named Pedraja, from the same region. A thorough petrographic characterisation is not available for this variety, so as for the fourth one (southern France). General descriptions are provided in Chapter 4, part 4.1.1.
Further descriptions of use-wear on each variety are provided, in order to discuss the intra-variability of wear of the same lithology.

5.2.1 QTFU1 (Facies Utrillas 1)

Wear on this variety was clearly distinguishable, depending on the different actions performed. The regular micro-topography helped in the slightly quicker development of wear (compared to other quartzite varieties). Polish was found sometimes on large areas; clear features were also described as connected to distinct worked materials. When non-ligneous vegetal species were worked, the development of polish was incredibly high. Polish originated from the working of bone sometimes displayed the characteristic wavy aspect which is traditionally considered as a diagnostic trait of bone working. Striations were particularly abundant on this variety, frequently allowing the determination of the kinematics. This is due again to the relative uniformity of the micro-topography of this variety.

5.2.2 QTFU2 (Facies Utrillas 2)

This variety displays a more irregular micro-topography, compared to the other Facies Utrillas variety (qtfu1) and to the Facies Pedraja one (qtfp1). This slightly influences the distribution of wear. Despite the types of wear observed, they are the same than on the other varieties, and their morphological characters are also very similar. Some differences especially in the frequency of wear, were also documented.
Polish is not as abundant as in the Facies Utrillas 1 and striations are present to a lesser degree. Even when reed was worked, the areas of the polish originated were more reduced.
Furrows are always the predominant type, but for some activities (bone and antler working) striations are practically absent.

5.2.3 QTFP1(Facies Pedraja 1)

This variety is a very well-sorted rock; quartz grains display very similar measurements and high compaction. Thus, the micro-topography is quite regular. Nevertheless, the general development of wear was not higher than that of the other varieties studied. On the contrary, wear was under-developed on tools used to perform transversal actions. Normally, polish was always restricted to the edges, with no or very limited development towards the interior surface. Striations were extremely rare and, when present, shorter than those observed on the other quartzite varieties.

5.2.4 PAY1 (Payre variety)

The French variety, collected in the surroundings of the Payre site shows features which clearly depart from most of the other varieties analysed. The main structural difference is that this variety is characterised by two, clearly distinguishable, granulometric fractions. Large quartz crystals and smaller crystals compose a very irregular micro-surface. There is strong neoblasts growing, which contribute to the presence of micro-picks and valleys. Striations are particularly rare on this variety and clear smooth polish has not been observed. A higher abundance of striations (furrows) was observed when the worked material was wood. In this case, and only on flat and big quartz grains, striations were found. Polished areas mostly present a rough texture, and clear distributional patterns were not documented. The limited and slow development of polish is explained by the presence of major prominent portions, where wear normally starts to form. A minor degree of fragmentation was also observed, but the size of the chips detached when the worked material was hard or very hard, were bigger than those of the other varieties analysed and generally observable macroscopically (during the experiments). This could depend on the nature of the rock, possibly derived from less sorted sediments compared to the other three varieties (more varied granulometry of the original sand sediments).

5.3 Publication 6:

Pedergnana, A., Ollé, A., 2017b. **Building an experimental comparative reference collection for lithic micro-residue analysis based on a multi-analytical approach.** *Journal of Archaeological Method and Theory* (in press), DOI: 10.1007/s10816-017-9337-z

This article comprises all the experimental data collected from the characterisation of the residues obtained from the experiments carried out during the construction of the reference collection of microwear on quartzite.

A wide bibliographic review is the backbone of this work, which allowed us to identify both strong and weak points of the method. The characterisation of residues first aims on improving our capacity of recognising and distinguishing the different types of residues. The results obtained allowed us to formulate a preferred method to analyse residues and to increase the reliability of the interpretations. Such method combines the use of OLM and SEM equipped with the EDX analysis system, which allows to investigate the elemental composition of residues.

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Building an Experimental Comparative Reference Collection for Lithic Micro-Residue Analysis Based on a Multi-Analytical Approach

A. Pedergnana [1,2,3] · **A. Ollé** [1,2]

Abstract Residue analysis applied to stone tools is a useful aid for better understanding their past function and, by extension, reconstructing early human behaviour. However, if the nature of residues found on the lithic tools is misinterpreted, so will be our understanding of their archaeological context. As a consequence, correctly identifying residues in the domain of lithic studies is of paramount importance. With this main goal in mind, we analysed different experimental materials likely to have been involved in daily tasks in the prehistoric context (*e.g.* bone, wood, meat). Microscopic analyses were then carried out using two (comparable) techniques: Optical Light Microscopy and Scanning Electron Microscopy. Also, energy dispersive X-rays spectroscopy (EDX or EDS) was applied to the experimental samples to determine their elemental composition. Advantages and disadvantages of both microscopic methods and their implications for correct residue identification are discussed. The distribution of residues on lithic surfaces is also considered. This study resulted in the construction of a data-set including both photographic material and EDX spectra for each residue analysed. The main result is that, compared to OLM scanning, SEM analyses highly improves the accuracy of residue identification.

Keywords Micro-residue analysis · Optical light microscopy · Scanning electron microscopy · Energy dispersive X-rays spectroscopy · Stone tools

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Introduction

Functional analysis of archaeological stone tools has traditionally relied upon the microscopic study of both surface modifications and organic residues. Two different disciplines made their appearance in the domain of archaeology as early as the second half of the XXth century. On the one hand, use-wear (or microwear) analysis aims to recognise stone tool function through the interpretation of traces left on their surfaces (Semenov 1964). On the other hand, residue analysis involves the direct observation of micro-residues (organic and inorganic) potentially left behind by the materials worked or by substances used in the manufacture of composite tools (*e.g.* hafting techniques).

Residue analysis involves both chemical and microscopic analyses and it may be applied to different kinds of archaeological artefacts. Its main purpose is to identify and define the nature of micro-residues, in order to respond to technological or behavioural inquiries. For example, when applied to historical or prehistoric pottery, generally the main purpose is to reconstruct human diet and trade (*e.g.* Barnard and Eerkens 2007; Barnard et al. 2007; Eerkens 2005, 2007; Evershed et al. 1990; Oudemans et al. 2007), whereas in the case of Palaeolithic stone tools, it is often aimed at determining their past functions. For this reason, residue analysis is often applied in conjunction with use-wear analysis (*e.g.*, Dinnis et al. 2009; Fullagar et al. 2015; Rots et al. 2015; Rots and Williamson 2004). It was precisely during the pioneer microscopic analyses on stone tools that organic residues were first documented (Anderson 1980; Briuer 1976; Hurcombe 1986; Shafer and Holloway 1979). Although the application of this kind of analysis to the lithic record is relatively recent, its great potential has now been recognised. This is reflected by an abundance of publications and contributions in international conferences treating this topic (*e.g.* Fullagar et al. 2015; Hardy and Garufi 1998; Haslam 2005; Haslam et al. 2009; Lemorini and Nunziante 2014; Lombard 2005; Marreiros et al. 2014; Ollé et al. 2017; Rots and Williamson 2004; Seeman et al. 2008; Sobolik 1996; van Gijn et al. 2015).

Essentially, two different methods have been designed to analyse residues on lithic surfaces (for both knapped stone tools and grinding stones):

1) Direct microscopic observation of residues preserved on the surfaces of the archaeological artefacts (with incident light microscopes);
2) Mechanical extraction of the residues to be analysed, usually through ultrasonic methods or using a pipette. Extraction is followed by appropriate preparation of the samples before they are mounted onto microscope slides (*e.g.* Fullagar 2006; Fullagar et al. 2015; Haslam et al. 2009).

Large residue samples made available by exceptional preservation conditions can be manually extracted and mounted onto microscopic slides (Dove and Peurach 2002; Dove et al. 2005). This procedure allows them to be observed with transmitted light microscopes and, when coupled with biochemical tests, is very useful for identifying diagnostic anatomical features. These consist of the employment of chemical reaction agents to detect the presence of specific protein groups (*e.g.* Barnard et al. 2007; Fullagar et al. 2015; Högberg et al. 2009; Kooyman et al. 1992; Loy 1983; Loy and Dixon 1998; Stephenson 2015; Yohe and Bamforth 2013). In the case of identifying blood with chemical reagents, it has been claimed that some confusion may arise in particular situations, possibly even yielding false positive reactions (Custer et al. 1988; Gurfinkel and Frankling 1988; Manning 1994). Therefore, results obtained using only this method should be taken with adequate caution.

Regardless of the method selected, the technique most often adopted to characterise residues has traditionally been optical light microscopy (OLM). Moreover, the few available residue blind tests were set up using this technique (Lombard and Wadley 2007; Wadley et al. 2004) and only recently other techniques, such as staining, have been incorporated (Rots et al. 2016). While in other disciplines (*e.g.* art history, prehistoric rock art, conservation) sophisticated analytical techniques have always been used (*e.g.* Gomes et al. 2014; Smith and Clark 2004), surprisingly, archaeologists dealing with micro-residues on stone tools have only extensively explored the advantages of OLM. Hence, interpretations have essentially been based upon the morphological features and colour of the observed residues (Hardy et al. 2001, 2013; Hardy and Garufi 1998; Hardy and Moncel 2011; Lombard 2008; Lombard and Wadley 2007; Wadley et al. 2004; Wadley and Lombard 2007). Scanning Electron Microscopy (SEM) has sometimes been included in the studies, principally to provide high-resolution topographical details and to determine the elemental composition of the residues (Anderson 1980; Byrne et al. 2006; Hortolà 2005; Jahren et al. 1997). Only recently, image comparison of some organic residues observed with different microscopes (OLM-SEM) was published (Borel et al. 2014; Monnier et al. 2012), a contribution that has significantly enriched the photographic information available in the literature.

Remarkable finds dealing with outstanding archaeological topics, such as the question of adhesive substances related to hafting (Cârciumaru et al. 2012; Charrié-Duhaut et al. 2013; Dinnis et al. 2009; Hauck et al. 2013; Helwig et al. 2014; Mazza et al. 2006; Monnier et al. 2013; Pawlik 2004a; Pawlik and Thissen 2011), ornaments, portable art or pigments (Cristiani et al. 2009, 2014; d'Errico et al. 2010; Fiore et al. 2008; Rifkin et al. 2016) have been subjected to chemical analyses to improve the reliability of the interpretations. Certainly, when precise chemical characterisations are incorporated, functional interpretations are more reliable, compared to studies where only morphological details are presented.

Some techniques recently introduced or re-incorporated in the field of residue analysis are: SEM-EDX analysis (Dinnis et al. 2009; Pawlik 2004a, b; Pawlik and Thissen 2011), Raman spectroscopy, Fourier Transform Infrared microscopy-FT-IR (Luo et al. 2012; Monnier et al. 2013, 2017; Prinsloo et al. 2014; Solodenko et al. 2015) and gas-chromatography-mass spectroscopy (GC-MS) (Boëda et al. 2008; Cârciumaru et al. 2012 Charrié-Duhaut et al. 2013; Eerkens 2002, 2005; Evershed et al. 1990; Hauck et al. 2013; Helwig et al. 2014; Perrault et al. 2016).

Assuming that the main challenges of residue analysis are sample contamination and decomposition (Eerkens 2007; Langejans 2010), we argue that providing parallel chemical characterisations of the observed residues powerfully strengthens the final interpretations. Therefore, once warned about the possible misunderstandings originating from modern contaminants (Pedergnana et al. 2016), one should refine the methods so as to provide the most faithful interpretation of the residue type.

Following these premises, this study is designed to provide an experimental, comparative reference collection of common organic residues, observed with both OLM and SEM.

The photographic documentation generated not only provides important data with which to compare between lithic assemblages, but also for performing viable new studies. This catalogue would also be useful for analysts in order to avoid confusion when they are not familiar with images taken with SEM. In addition, it will be a valuable resource for researchers who wish to consult additional comparative images other than those obtained from their own observations. Hence, the systematic comparison of micro-graphs obtained through OLM and SEM observations improves the analyst's skills to correctly identify residues. The enhancement of the average rates of correct residue interpretations of experimental samples is a necessary bridge towards gaining a more reliable interpretation of the archaeological evidence.

Finally, to provide a better characterisation of the selected materials, energy dispersive X-rays spectroscopy (EDX or EDS) is applied to one sample for each of the materials presented here.

Methods

A catalogue composed of microscopic images and elemental spectra of different organic materials was elaborated over a period of approximately two years. The residues to be analysed are considered to be some of the most likely used materials in the daily tasks performed by prehistoric peoples (*e.g.* soft animal tissues, bone, wood).

More specifically, this catalogue is destined to serve as a basis for comparing with archaeological residues that may be present in the Middle Pleistocene lithic assemblage from the Sierra de Atapuerca's Gran Dolina site (TD10) (Northern Spain) (Carbonell et al. 1999, 2014; Ollé et al. 2013; Rodríguez-Hidalgo et al. 2015).

Experiments

The residues analysed here originate from our extensive experimental activity involving different varieties of quartzite which aimed at the construction of a use-wear reference collection for this rock type (Pedergnana and Ollé 2017). Specifically, the experimental flakes included in this work were obtained by free-hand knapping of three different fluvial quartzite cobbles from the Utrillas and Pedraja formations (Pedergnana et al. 2017). Two additional flint samples coming from a different experimental programme were also included (Pedergnana and Blasco 2016). All of them were kept in plastic zipped bags until the performance of the experiments. Since the main objective of this work was to observe the appearance of fresh residues, no specific experiments including post-depositional processes have been carried out.

The activities generally included in this type of study were carefully selected (*e.g.* butchery, hide scraping, wood and bone sawing) (Fig. 1). All of the experiments were performed under strictly controlled conditions. As a result, residues of the contact materials found on the tools' surfaces were made available for additional studies (Fig. 2).

The residues included in this study are: soft tissue and hard animal materials (muscle, fat, fresh and dry skins, fresh bone/shed antlers (*Cervus elaphus*), feathers (*Gyps fulvus, Circaetus gallicus*); wood (*Quercus ilex*) and cane (*Arundo donax*). They were observed on 23 experimentally manufactured stone tools (Table 1).

Because of the wide diversity of wood species, each characterised by distinct properties, we began our analysis using a single taxon: *Quercus*; a type of wood that

Fig. 1 Some of the controlled experiments: a) removal of a deer skin (qtfu1–03); b) removal of the muscular tissue from long bones of a deer (qtfu2–03); c) scraping a fresh red deer skin (qtfu1–04); d) scraping a dry red deer skin (qtfu2–07); e) sawing a hardwood branch (qtfu1–13); f) sawing a fresh long bone (qtfu1–05)

is often attested in the palaeo-ecological setting of our study region (Rodríguez et al. 2011). Although it is not represented in the European prehistoric register, the reed species *Arundo donax* was also included in our study. It serves as a reference for non-woody materials residue distribution on stone-tool surfaces. However, deeper investigation on residues left by different vegetal species would require very detailed experimental sessions focusing more specifically on such materials, like the one recently carried out by Xhauflair (2014).

The state of the residues generated was generally fresh (wood collected the same day of the experiment, bones used the day after the butchering of an animal, *etc*...). Two well-dried deer skins were scraped, respectively 1 and 2 years after the animals were butchered. Antlers were worked dry as well, and were not soaked in water prior the performance of the experiments. No other treating additives or abrasive substances were used at all (ash, ochre, *etc*.).

The macroscopic distribution for each residue type was systematically recorded immediately after the experiments. The experimental flakes were then stored in clean,

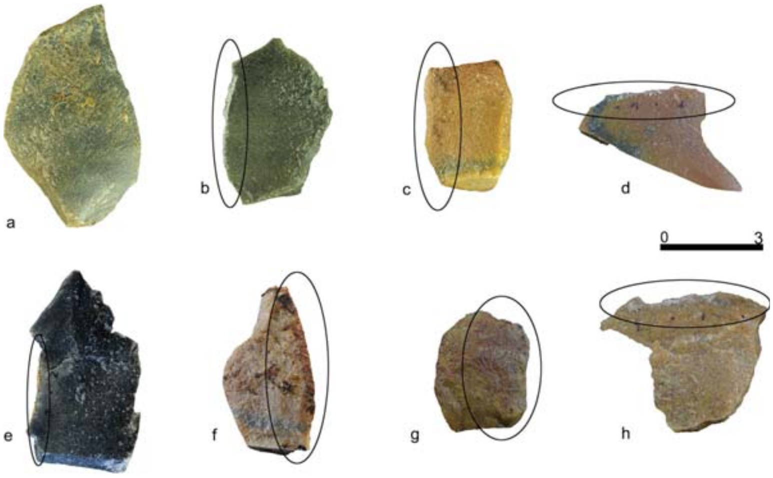

Fig. 2 Some of the experimental flakes just after the experiments: a) skinning and dismembering (qtfp1–01); b) sawing bone (qtfp1–02); c, g) cutting fresh skin (qtfu1–11; qtfu2–11); d) cutting reeds (qtfu1–10); e) sawing wood (qtfp1–05); f) butchering (qtfu1–12); h) scraping dry skin (qtfu2–07). Note the macro distribution of the organic residues

zipped plastic bags, until they were subjected to microscopic observations (a few days-weeks after the conclusion of the experiments). Neither cleaning procedures nor any other sample preparation were applied to the lithic surfaces before the performance of the residue analysis so as to preserve the original distribution and appearance of the residues. The bags where the experimental flakes were stored were not opened until the residues were analysed, thus keeping the risk of contamination to a minimum.

Microscopy

Residues were first analysed with a metallographic microscope Zeiss Axio Scope A1 (with magnifications ranging from 50 to 500 times) and multi-focused images were usually obtained using the DeltaPix Insight software. Images were taken with a 5 MP DeltaPix digital camera (Invenio 5SII model).

The same residues were then analysed with a variable pressure scanning electron microscope (ESEM-FEI-Quanta 600) to image details at higher magnifications and resolution. When using the latter, the Oxford INCA system for image acquisition was used. The SEM was equipped with two detectors: the secondary electron detector (Large Field Detector, LFD) and the backscattered electron detector (DualBSD). The former allows the observation of topographic and textural traits of the residues, while the latter provides grey-scale images according to atomic number contrast, with brighter regions being generated from areas of higher average atomic number. All observations took place in low vacuum conditions, so that the samples did not need to be covered with thin carbon or gold layers, which enhance sample conductivity in high-vacuum observations. A detailed discussion on the microscope settings can be consulted in previous papers (Borel et al. 2014; Ollé and Vergès 2014).

Table 1 Experimental flakes with residues analysed in this study. The main experimental variables are summarised

NUM.	EXPERIMENTAL FLAKE	EDGE ANGLE	CONTACT MATERIAL	TYPE OF CONTACT MATERIAL	ACTION	CONTACT ANGLE*	TOTAL TIME min	Approx. n. strokes
1	qtfu1–01	40°	Fresh wood	*Quercus ilex*	Sawing	60° < α < 90°	60	18,000
2	qtfu1–02	45° < α 60°	Shed antler	*Cervus elaphus*	Sawing	90°	60	24,000
3	qtfu1–03	40°	Skin, flesh, tendons, bone	*Cervus elaphus/ Sus scrofa*	Cutting (uni)-Skinning/ Dismembering	80° < α < 40°	80	4000
4	qtfu1–04	45°	Fresh skin	*Cervus elaphus/ Sus scrofa*	Scraping	60° < α < 30°	45	6000
5	qtfu1–05	50°	Fresh bone	*Cervus elaphus/ Bos taurus*	Sawing	80° < α < 90°	60	18,000
6	qtfu1–06	25° < α 30°	Fresh bone	*Cervus elaphus/ Bos taurus*	Scraping	90°	60	7200
7	qtfu1–07	60°	Dry skin	*Cervus elaphus*	Scraping	40°	45	6000
8	qtfu1–08	30°	Fresh wood	*Quercus ilex*	Scraping	40°	30	3000
9	qtfu1–10	35°	Giant cane	*Arundo donax*	Sawing	90°	45	13,500
10	qtfu1–11	35°	Fresh skin	*Cervus elaphus*	Cutting (uni)	90° < α < 70°	10	1200
11	qtfu1–12	60°	Flesh, tendons, bone	*Cervus elaphus*	Cutting (uni)-Defleshing	90° < α < 80°	20	1600
12	qtfu1–13	40° < α < 30°	Wood	*Quercus ilex*	Multiple actions (sawing, scraping, whittling)	40° < α < 90°	25	2000
13	qtfu2–03	35°	Flesh, tendons, bone	*Cervus elaphus*	Cutting (uni)-Defleshing	80° < α < 90°	85	6800
14	qtfu2–07	40°	Dry skin	*Cervus elaphus*	Scraping	40°	30	4000
15	qtfu2–11	45°	Fresh skin	*Cervus elaphus*	Cutting (uni)	90° < α < 70°	10	1200
16	qtfp1–01	40°	Skin, flesh, tendons, bone	*Cervus elaphus/ Sus scrofa*	Cutting (uni)-Skinning/ Dismembering	50° < α < 40°	60	3000
18	qtfp1–02	55°	Fresh bone	*Cervus elaphus*	Sawing	90°	30	9000
19	qtfp1–05	45°	Wood	*Quercus ilex*	Scraping	60°	11	1000
20	qtfp1–06	45°	Wood	*Quercus ilex*	Cutting (uni)	90°	15	1000

Table 1 (continued)

NUM.	EXPERIMENTAL FLAKE	EDGE ANGLE	CONTACT MATERIAL	TYPE OF CONTACT MATERIAL	ACTION	CONTACT ANGLE*	TOTAL TIME min	Approx. n. strokes
21	qtfp1–07	30°	Shed antler	*Cervus elaphus*	Cutting (uni)	90°	13	1000
22	flint 1	35°	Flesh, Feathers, tendons, bone	*Gyps fulvus*	Cutting (uni)-Skinning/ Dismembering	80°-90°	50	-
23	flint 5	30°	Flesh, feathers, tendons, bone	*Circaetus gallicus*	Cutting (uni)-Skinning/ Dismembering	40°-80°	40	-

Comparison of micro-graphs imagining exactly the same selected details of the residues and taken by means of the two microscopes, was performed in order to provide a thorough description of each sample.

Furthermore, energy dispersive spectroscopy (SEM-EDX or EDS) was applied for each residue type and relative spectra and element maps were obtained. X-rays are generated by primary electron bombardment of the sample, the analysis of which gives quick elemental information. SEM-EDX, used in "spot" mode, is capable of providing a full elemental spectrum of the analysed spot in only a few seconds. This makes it a very useful tool for obtaining preliminary inferences about the chemical composition of the residues.

Results

Animal Residues

Muscle Tissue

Muscle tissue refers to the flesh of butchered animals and is basically composed of water, proteins, fat and carbohydrates. Muscles are made up of elongated cells (myocytes) interlocked by a connective tissue, whose main component is collagen. Collagen is one of the most abundant proteins in an animal's body and is found in practically all of the structural tissues (bones, ligaments, tendons, skin). It is described to be fibrous in nature (Mescher and Junqueira 2013). Muscles are in contact with adipose layers, whose function is to store energy in the form of fat. Generally, it is possible to visually differentiate fat from muscle because of its whitish-yellowish colour (during butchering activity, for example).

Microscopically, fibres are often visible on implements used to butcher animals. Collagen has sometimes been reported to be opaque and not birefringent (Fullagar 2006); while elsewhere a birefringent value similar to that of vegetal fibres was documented (Lombard 2008). Particularly, this feature was said to cause confusion when trying to differentiate between plant and animal residues (Lombard and Wadley 2007).

According to our observations, white fibres are birefringent, but only at high magnifications (Fig. 3: f). Normally, white, birefringent fibres are thought to be collagen (Lombard 2008), but without further histological analyses or biochemical staining, this is very difficult to confirm (Stephenson 2015). Bluish tones, usually reported for bone (Lombard 2008), were also associated with collagen residues (Lombard 2004); a fact which might cause some confusion.

Brownish-reddish fibres were also observed within animal residues possibly being composed of muscular tissue (Fig. 3: a) or blood vessels (Fig. 3: c). Even if SEM micrographs do not allow to observe colours, the fibres' morphology is scanned with higher resolution (Fig. 3: b), thus allowing to differentiate them from similar materials (animal hair, feathers). Fat, in the form of adipose cells, has been claimed to be visible under OLM (Lombard and Wadley 2007; Lombard 2008). Again, with no extraction of the residues and further proper laboratory sample preparation, cells cannot be seen (because of optical limitations of the device). In fact, biological samples are normally cut into thin layers, attached to a microscope slide and then observed with transmitted light microscopes (Mescher and Junqueira 2013). This procedure allows the observation of the internal

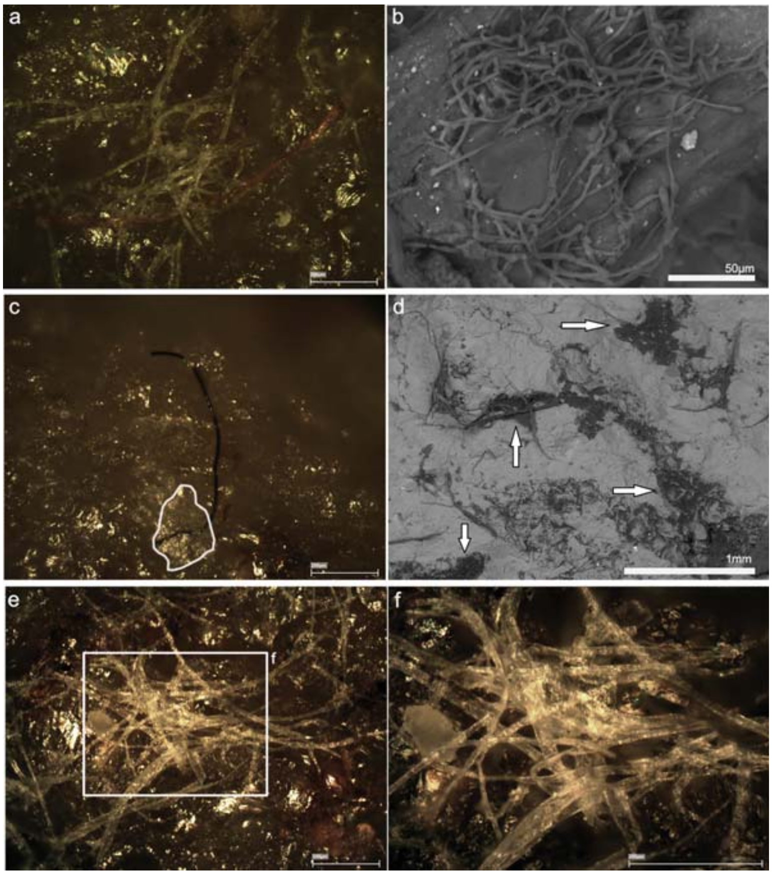

Fig. 3 Residues obtained during butchering activity (qtfu2–03). Tissue fibres imaged under the OLM (a, e, f) and SEM (b): colour and structure are compared. Fat residues are sometimes visible (c, circumscribed by the white outline). The brownish fibre is thought to be a blood vessel. Fat residues are clearly visible under the SEM (d, white arrows point to the residues). With higher magnifications, white animal fibres are birefringent (f). Orig. mag. And scale bars: a, c, e) 100×, 200 μm; b) 1250×, 50 μm, (BSD); d) 100×, 1 mm, (BSD); f) close-up of image e, 200×, 200 μm

cellular structure of tissues. Indeed, by directly observing smashed residues on the tool surfaces, we were not able to document single cells nor to clearly differentiate collagen fibres. Fat is thought to leave very greasy spots on the experimental tools. Under the OLM, depending also on the rock substrate, such stains might be more or less visible (Fig. 3: c, within the white line). In fact, fat residues seem to be more difficult to image on highly reflective rocks such as quartz or quartzite (Lombard and Wadley 2007:161). These fat residues are more clearly discernible under the SEM-backscattered electron detector (Fig. 3: d), thanks to their darker grey colour derived from the different chemical composition of the fat with respect to the silica substrate.

More compact residues composed of large portions of muscular tissue, were also imaged (Fig. 4). The linear trend of the fibres was visible even to the naked eye. Clusters of white and red fibres appeared to maintain their original anatomical structure (*i.e.* fibres are still compacted into portions of tissue). While under the OLM it is possible to see light and dark fibres (Fig. 4: a, c, e), the best tri-dimensional details are obtained with SEM (Fig. 4: b, d, f). Smeared residues were

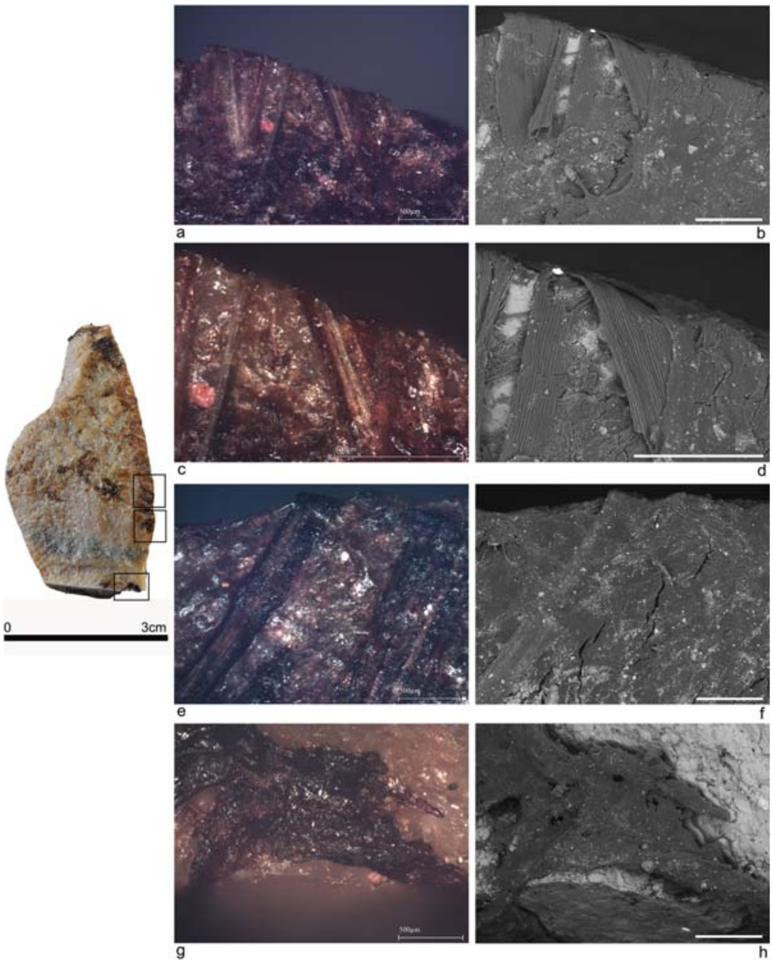

Fig. 4 Muscle tissue residues with the relative position on the experimental flake (qtfu1–12). OLM (a, c, e, g) and back-scattered electron detector (b, d, f, h) graphs of exactly the same details. Muscular fibres are clearly visible, exhibiting an ordered pattern (c, d), or are mixed with fat and blood in amorphous masses of tissue (g, h). Orig. mag. And scale bars: a, e) 50×, 500 μm (13 slides); b, f, h) 135×, 500 μm; c) 100×, 500 μm (13 slides); d) 260×, 500 μm; g) 50×, 500 μm (40 slides)

also documented (Fig. 4: g, h), where sparse fibres were seen underneath a layer of fat and blood.

Skin

Animal skins (or hides) are the outermost layer of vertebrates. They function as a barrier between the internal organs and systems and the environment with which they continuously interact (*e.g.* protection, thermoregulation, metabolic reactions).

Although "hide working" is a very general term, usually employed to indicate the processing of both hides and skins, a differentiation between hides and skins should be made. For large animals (cattle, horse, bison), the correct term is hide, while skin refers to smaller-sized animals (*e.g.* sheep, goat, deer, rabbit) (Rots 2010; Wiederhold 2004). Skins are formed by two main layers, the epidermis and the dermis, mainly composed of connective tissue. Beneath the dermis, there is a subcutaneous layer, the hypodermis, which contains also adipocytes (fat cells) (Mescher and Junqueira 2013).

Scraping activities are normally exerted either on the outermost layer (to remove fur), or on the inner layer (to remove organic residues, such as remnants of meat, greasy substances, *etc.*), while cutting actions intervene upon all the hide layers.

The experimental tools used to cut portions of fresh skin (Fig. 2: c) were covered with blood and no large accumulations of tissue were found. Conversely, scraping activities resulted in wide accumulations of residues (meat, greasy particles) to be stuck on the surfaces of the used tools, mainly near the tool edges (Fig. 2: h; Fig. 5: a-c). Clusters of white fibres were found all around the used edges, sometimes occurring as loose residues on the rim (Fig. 5). At OLM high magnifications, such accumulations were birefringent (Fig. 5: d). Under the SEM, the structure of the residue is more easily discernible, and some fibres were detected embedded in a fat layer (Fig. 5: e, f).

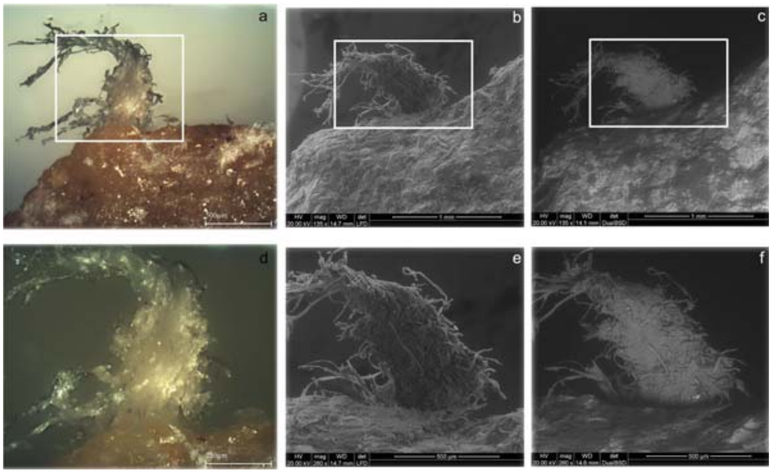

Fig. 5 Dry skin residue on the very edge of a tool (qtfu1–09), imaged with OLM and both SEM detectors. Orig. mag. And scale bars: a) 50×, 500 μm; b, c) 135×, 1 mm; d) 100×, 250 μm; e, f) 250×, 500 μm. Extended focus pictures were obtain for images a and d (11 slides)

When dry skins are worked, some organic components are thought to be removed (due to the activity of the bio-organisms and perhaps previous scraping/de-fleshing). Therefore, no greasy substrate is expected to enclose the connective tissue, as in fresh residues. In fact, accumulations of dry skin-fibres appear to be greater, and these fibres intersect with one another, composing intricate patterns (Fig. 6: a, c, d). Single fibres appeared wider and flatter, compared to those observed on fresh residues (Fig. 6: b, e, f) and they did not exhibit birefringent features even under high magnifications (Fig. 6: b). The average width documented for those fibres was 20-40 μm (Fig. 6: f).

SEM-EDX analysis of skin residues showed the presence of carbon (C), sulphur (S), chlorine (Cl), phosphorus (P) and potassium (K) (Fig. 6: g); the same elements documented for human skin particles (Pedergnana et al. 2016).

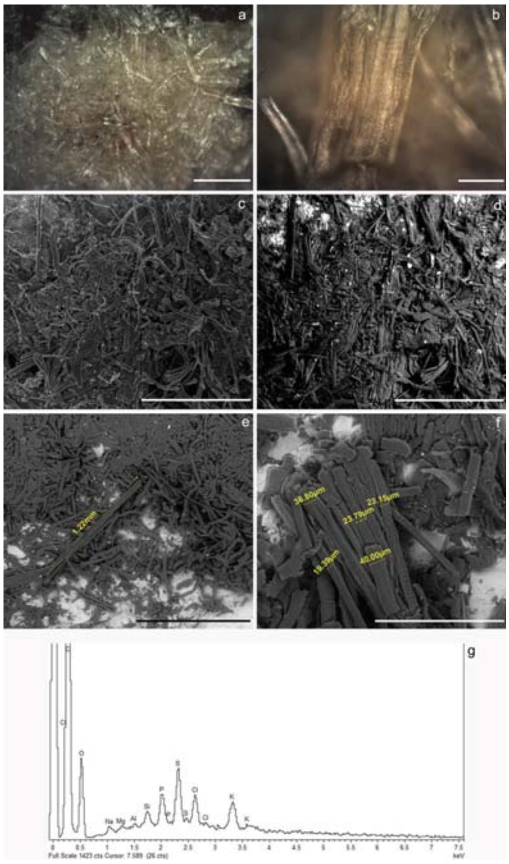

Fig. 6 Scraping dry skin residues: accumulations of fibres (qtfu1–07, qtfu2–07). OLM graphs orig. Mag.: a) 100×, 500 μm b) 200×, 100 μm. SEM images orig. Mag.: c) 130×, 1 mm (LFD); d, e) 130×, 1 mm (BSD); f) 510×, 300 μm. g) EDX-spectrum showing the presence of C, S, Na, Mg, P, S, Cl and K. Si and Al are related to the rock substrate (quartzite). No extended focus images were obtained

Bone

Bone is the rigid skeletal tissue composing the firm internal structure of the vertebrates. It is composed of an inorganic part (70%, mainly hydroxyapatite) and an organic part (30%, mainly collagen fibres, other proteins and water). In the archaeological record, bone remains composing the archaeo-faunal record is conserved when it has undergone the fossilisation process. Thanks to their high mineral content, fossilised bones constitute one of the best preserved materials in archaeological contexts. In studies dealing with residue analysis, fragments of bone were extensively described as being amorphous and compact, sometimes greasy, white masses (due to the experimental processing of fresh bones) (Lombard 2005, 2008).

Amorphous crystalline masses were indeed present on our specimens (Fig. 7: a, c, d). In some areas, remnants of the periosteum were visible in reddish-pinkish tonalities, and sometimes red fibres were also present (Fig. 7: b, c; Fig. 8: c). The greasy residue is better imaged under the SEM, where its darker colour (due to the high C presence) clearly contrasts with the light tonalities of the bone (due to the higher atomic number of Ca, P, and Mg) (Fig. 7: e; Fig. 8: b). A distinctive layered pattern is visible under higher magnifications (Fig. 7: f). Although these observations were not made with a rotated polariser, the bluish tones described in the literature for this method (Fullagar 2006; Lombard 2008) were sometimes recorded on our specimens (Fig. 7: b). The main components of bone apatite (Ca_{10} $(OH)_2$ $(PO_4)_6$) were documented by EDX analysis (Fig. 8: d) (Jahren et al. 1997).

Antler

Antlers are bony and branched structures, characteristic of the family *Cervidae*. They are present only in males (with the exception of reindeer) and are shed annually. Their main function is related to the adults' behaviour during the breeding season, when males compete for females. The structure of antlers is built up in order to resist high-loaded impacts (Launey et al. 2010). Antlers, as bone in general, are mainly formed by osteoblasts, collagen fibres and hydroxyapatite (Chen et al. 2009). Antler has been a widely used material throughout most of Prehistory, predominantly from the Upper Palaeolithic onwards, and therefore it is important to be able to recognise it in the archaeological record when its micro-residues have been preserved.

Due to its equivalent chemical composition with bone, the EDX-analysis showed the same elemental peaks (C, Na, Mg, P, K, Ca) (Fig. 9: e). Under the microscope, compact accumulations may be present (Fig. 9: a, b), but a characteristic background granular aspect is always visible (Fig. 9: a; Fig. 14: d) (Monnier et al. 2012: Fig. 14a). The organic parts are darker than the inorganic ones when antler residues are scanned with the SEM-BSD detector (Fig. 9: d).

Feathers

Details of the experiments carried out with flint (instead of quartzite) on avian species are found in Pedergnana and Blasco (2016). Feathers of griffon vulture (*Gyps fulvus*) and short-toed snake eagle (*Circaetus gallicus*) were analysed microscopically. Tissue residues of these avian species were also analysed, revealing that

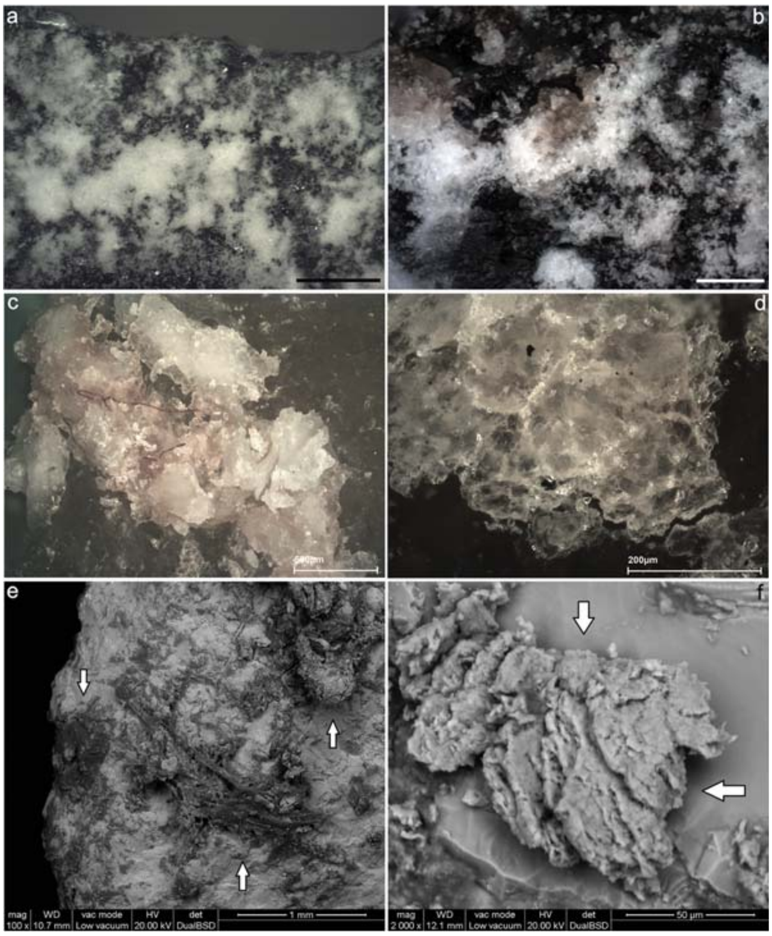

Fig. 7 Fresh bone residues (scraping and sawing activities, qtfu1–06, qtfp1–02). OLM orig. Mag. and scale bar: a) 50×, 500 μm; b) 200×, 100 μm; c) 50×, 500 μm; d) 200×, 200 μm. BSD-SEM images orig. Mag. and scale bar: e) 100×, 1 mm; f) 2000×, 50 μm. Extended focus pictures were obtained for images: a (25 slides), b (30 slides), c (30 slides) and d (20 slides). White arrows point to the residues imaged with SEM

they greatly resemble visually those obtained from deer (Fig. 10: d). In the archaeological context, it is very hard to find the diagnostic traits of feathers on tiny micro-fragments. Surprisingly, this is also true of experimental residues. Secure identification is easier to accomplish by observing entire barbs (Fig. 10: a, b). Unfortunately, relatively large sized feather portions are unlikely to have been preserved on archaeological samples where evidence generally consists of only single barbules (Hardy et al. 2001, 2013). In fact, relatively large samples only occur under very extraordinary preservation conditions. In such cases, samples are

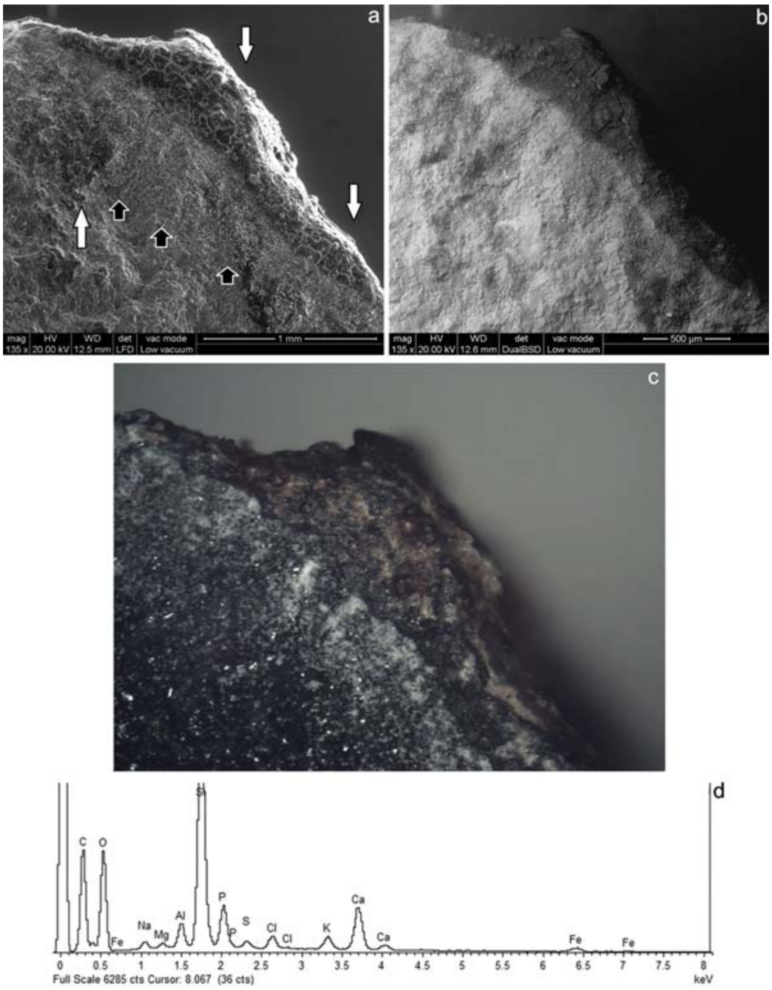

Fig. 8 Residue on a tool used to saw fresh bone (qtfp1–02). Both organic and inorganic parts are visible. a, b) LFD and BSD SEM pictures, 135×, 500 μm. c) OLM image of the same point showing white bone particles and an organic brown film, 50×, 500 μm (20 slides); d) SEM-EDX spectrum of the residue showing the presence of C, O, P and Ca, related to bone. White arrows in image a point to the organic parts of the residue, while black arrows indicate the inorganic part

carefully prepared to undergo transmitted light microscopic observation and therefore, identification can reach the order or even species level (Dove and Peurach 2002; Dove et al. 2005).

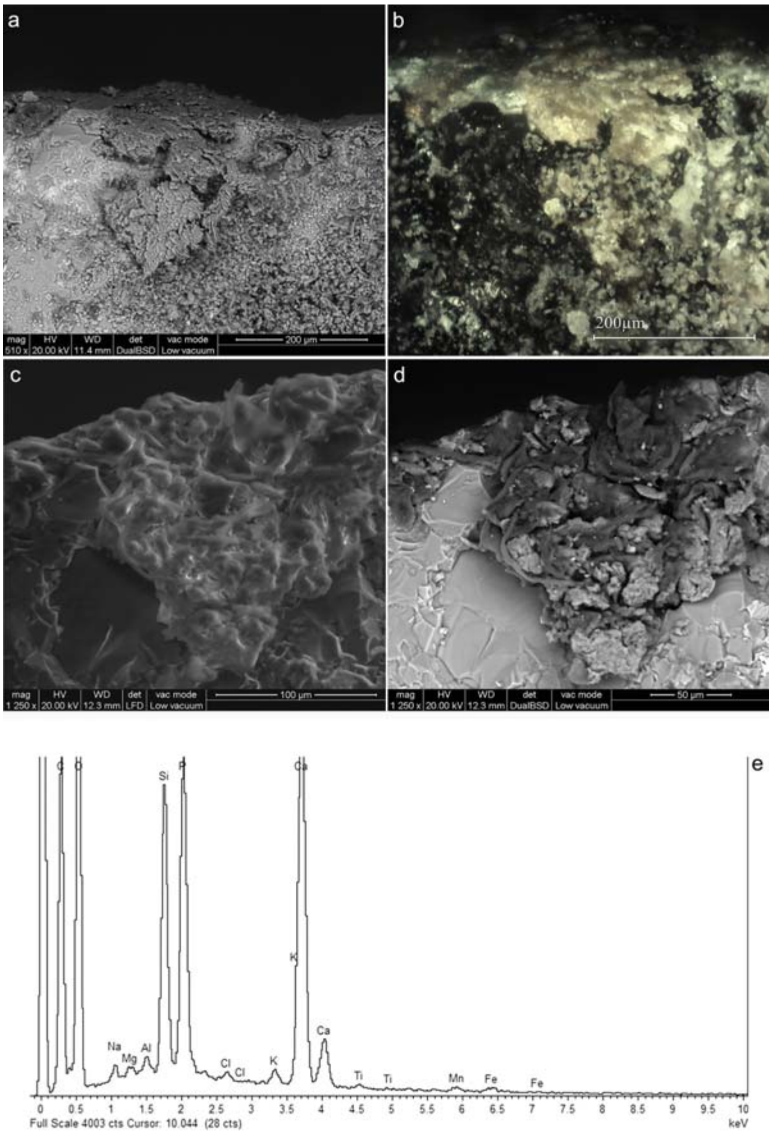

Fig. 9 Residue on a tool used to saw deer antler (qtfp1–07). SEM images orig. Mag.: a) 510×, 200 μm (BSD); c, d) 1250×, 100 μm (LDF and BSD respectively). OLM image: b) the same point imaged in: a, 200×, 200 μm. e) SEM-EDX spectrum of the residue showing the main peaks of C, Ca, P, O and minor concentrations of Na, Mg, K, Mn and Fe. Si, Al, Ti and part of the O are connected to the rock substrate

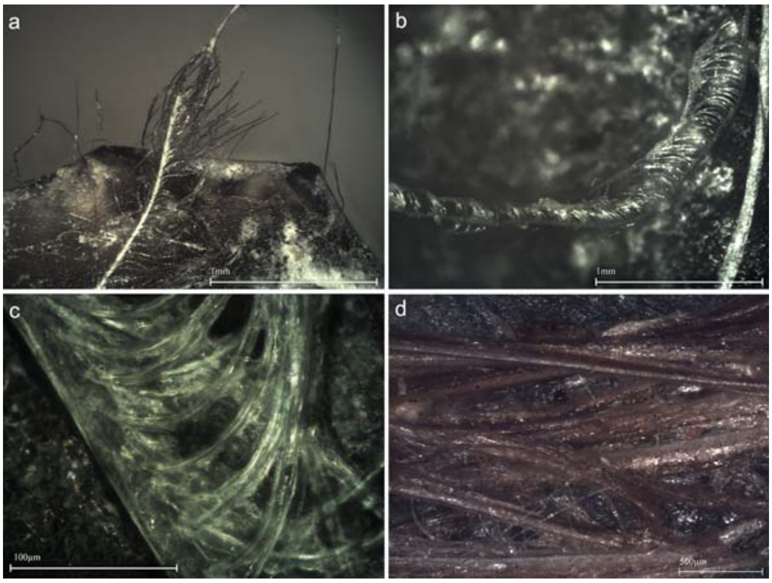

Fig. 10 Feathers and residue of avian tissue on flint experimental tools. Number of slides of the extended-focus pictures and scientific name of the avian species: a) 28 slides, *Circaetus gallicus*; b) 17 slides; *Gyps fulvus*; c) 6 slides, *Gyps fulvus*; d) 30 slides, *Gyps fulvus*. The original magnification of all images is 50×

Barbs do not appear birefringent even at high OLM (reflected light) magnifications (Fig. 10: c). Single diagnostic barbs (plumulaceous barbs) were not observed on our experimental flakes, meaning that their presence on stone tools might not be very common. Consequently, in the rare cases that diagnostic barbs were trapped on the surface of prehistoric tools, they would probably have much lower preservation rates than those of other residues.

SEM observations proved useful for the morphological characterisation of this residue type, allowing differentiating it from fibres with analogous colour and morphology under the optical microscope, such as animal hair. Moreover, elemental composition underlined the presence of the main compounds found in keratin (C, O, S) (Pedergnana and Blasco 2016).

Vegetal Residues

Wood

Wood is an organic material composing the structural parts of trees and other woody plants. Although the chemical composition of wood is complex and varies from species to species, three structural polymeric components, namely, cellulose, hemicellulose, and lignin, are common to all woods. The inorganic content, also referred to as ash, comprises various elements, (*e.g.* K, Mg, Fe, Al, Na, Cu) (Rowell 2005). The most studied vegetal micro-residues are phytoliths, which are tiny siliceous particles (50mμ) produced by plants. Although phytoliths are difficult to observe on stone-tools, their identification can be useful to understand what the tools were used for (Anderson 1980; Anderson-Gerfaud 1986; Domínguez-Rodrigo et al. 2001; Kealhofer et al. 1999; Piperno 1984; Shafer and Holloway 1979).

Strictly regarding wood, what is most likely to be preserved on archaeological stone tools are residues of the outermost layer of trunks or branches and the first inner layer (the "living wood" responsible for the transport of sap and the storage and synthesis of biochemicals), respectively known as bark and sapwood (Rowell 2005). It is very ambitious to hope to observe microscopically the main cells of the woody tissue (*e.g.* tracheids) on archaeological residues. This is basically for two reasons: firstly, because it is reasonable to think that the morphological traits which make them recognisable have suffered extensive modifications over time due to residue decay; therefore, direct comparisons with the anatomic cell structure found in modern atlases are not recommended (Monnier et al. 2012:3285). Secondly, because of technical, optical limitations, this kind of observation is impeded when specimens are not prepared and mounted on proper microscope slides (like in Shafer and Holloway 1979). In most archaeological studies, residues are not extracted from the lithic surfaces, but rather are observed directly. Thus, the direct observation of wood cells in the archaeological context is a rare event and it is generally restricted only to experimental (not buried) residues (Lombard and Wadley 2007).

Our experiments with wood were carried out using both transversal and longitudinal gestures, on a hardwood species of holm oak (*Quercus ilex*). *Quercus* residues appeared as amorphous accumulations of brownish-reddish woody tissue (Fig. 11: c). Sometimes, a mud-cracking aspect was visible on the surface of these accumulations (Fig. 11: e).

The colour is possibly related to the outermost woody layer of branches and trunks: the bark. In fact, when the interior layer is scraped, the colour of the resulting residue might be significantly lighter than the bark (Fig. 11: g). Obviously, colour also depends on the species of the selected wood (see differences between *Fraxinus* and *Picea* in Monnier et al. 2012). Fibres are very rare and usually whitish in colour (Fig. 11: c, f, i). At higher magnifications, plant fibres are reported to be birefringent (*e.g.* Langejans 2012; Lombard 2008). The mapping of one *Quercus* residue imaged by means of the two SEM detectors exhibited the presence of O, C and Ca, contrasting with Si and a Ti inclusion; both referring to the rock substrate (Fig. 12).

Reeds

Giant reed (*Arundo donax*) is the only graminaceous plant used in our experiments. *Arundo donax* is a species native to the Middle East (Herdion et al. 2014). It dispersed westerly and was at least partially introduced intentionally by humans when they colonised the more temperate areas of the Mediterranean basin. It is a reed-like grass, reaching 8 m in height. Its hollow stems resemble those of bamboo. The culms have a diameter of 1–4 cm and their thickness can vary from 3 to 7 mm. They are quite hard and dry (low moisture degree) and are separated by nodes, which are harder than the interior of the stems.

Residues of only longitudinally oriented activities (cutting) were analysed. They appear as whitish-yellowish compact spots of plant tissue (Fig. 13: a, c). Although they

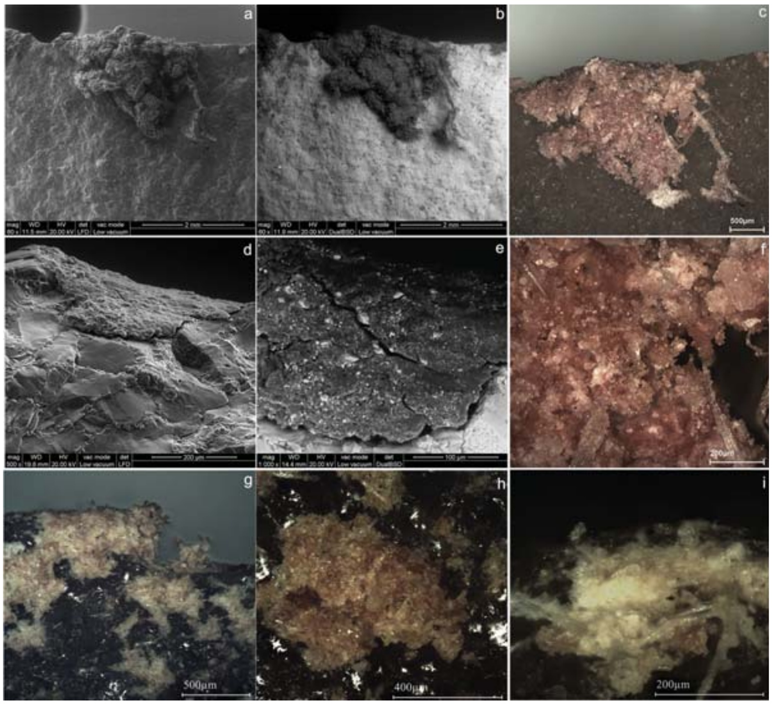

Fig. 11 Residues found on implements used to scrape and saw holm oak branches (qtfu1–08, qtfu1–13). SEM images orig. Mag.: a, b) 60×, 2 mm (LFD and BSD respectively); d) 500×, 200 μm (LFD); e) 1000×, 100 μm (BSD); OLM images orig. Mag.: c) same residue imaged in: a and b, 25×, 500 μm (30 slides); f) 100×, 200 μm (28 slides); g: 50×, 500 μm (20 slides); h) 100×, 400 μm (13slides); i) 200×, 200 μm (14slides). Image c was obtained through a stereo-microscope

are not fibrous in nature, some linear trends were visible (Fig. 13: b, d). Fibres seen under the OLM showed interesting similarities with those found within wood residues (Fig. 11: f, i). It is possible that reed residues were flattened because of a high content of water, as fresh cane stems were used. It has been demonstrated that the residue distribution of different vegetal species is dependent upon their water content. In fact, residues with higher water content show a more widespread distribution on stone tool surfaces (Xhauflair et al. 2017).

Giant cane is comparable with bamboo, for which a general low to medium water content was assigned by Xhauflair et al. (2017). The overall distribution of this kind of residue was observed to be very close to the used edge (Fig. 2: d). Elemental analysis and mapping of cane residues revealed the presence of C, O, S, Cl and K.

Overlapping Morphologies

It has frequently been stated that one of the most challenging obstacles in residue analysis is the morphological and colour similarities found on different types of residues

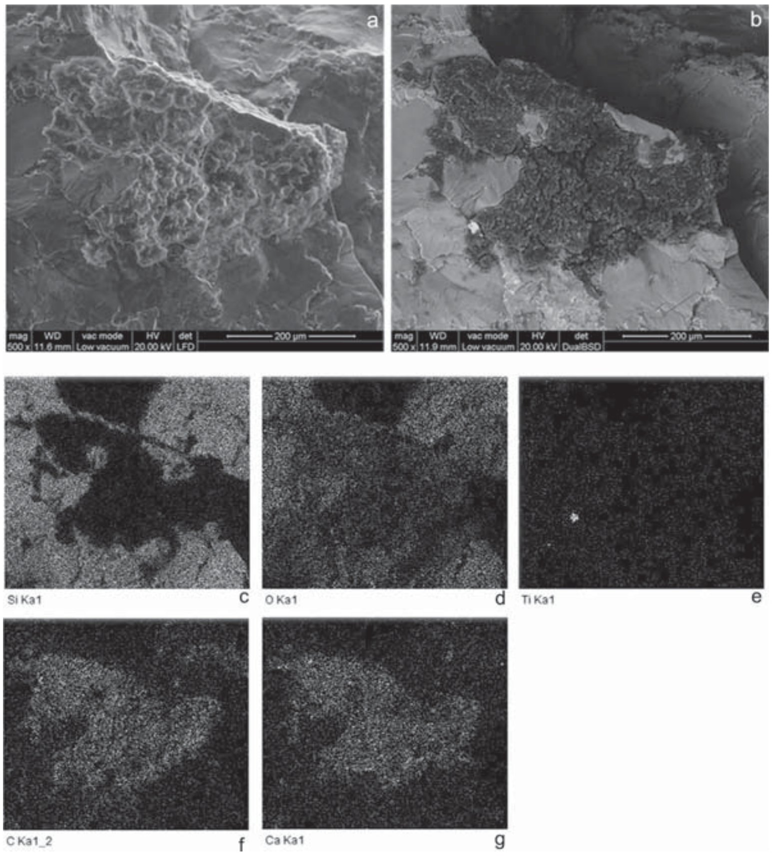

Fig. 12 SEM-EDX analysis of a wood residue imaged with the two SEM detectors: a, b) 500×, 200 µm. Elemental map of the residue: c = Si; d = O; e = Ti; f = C; g = Ca

(Langejans 2010; Lombard and Wadley 2007; Monnier et al. 2012). Although analysts have eventually been able to manage some of the problems after several years of practice, there are still some issues which need to be resolved, even on an experimental level. One of these is the high resemblance (both visual and chemical) of bone and antler, and the second is the visual appearance of general fibrous materials.

Bone vs. Antler

Two of the most ambiguous residues detected in this study are bone and antler (Fig. 14). The reasons for this are easy to explain. First of all, they are basically the 'same material'. They share a very similar chemical composition (organic material, mainly collagen and an inorganic part, mainly hydroxyapatite). Consequently, the analysis of the elemental composition, which might clarify some equivocal

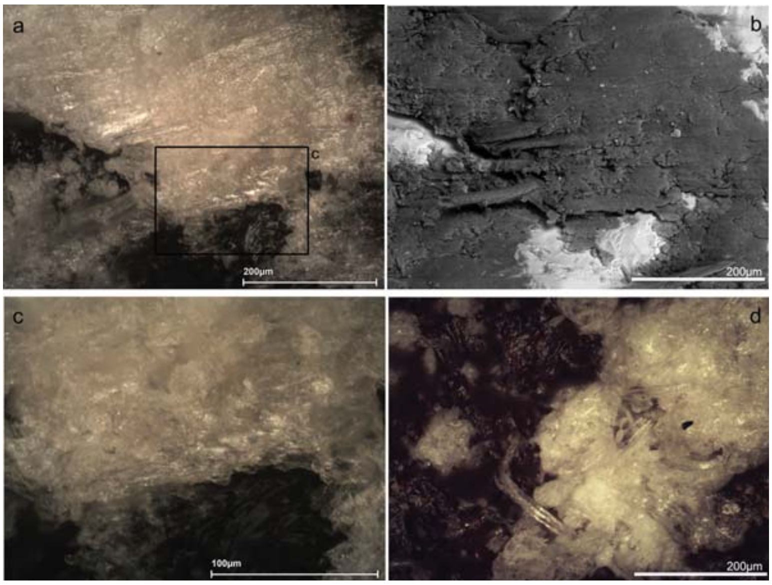

Fig. 13 Giant cane residues on a tool actioned with longitudinal movements (qtfu1–10). Orig. mag.: a, d) 200×, 200 μm; b) 510×, 200 μm (SEM-BSD); c) enlarged image of the area delimitated by the square in: photo a, 500×, 100 μm

circumstances, appears to be useless in this case. In fact, exactly the same elemental peaks were documented on spectra of both experimental bone and antler (Fig. 15).

Additionally, several visual analogies were documented for both residues: similar distribution on the used edges (Fig. 14: a, b), analogous granular aspect (Fig. 14: c, d), birefringence and transparency at high magnifications (Fig. 14, e, f). When carefully observed, slight differences are indeed present and, therefore, the experience of the analyst is crucial for a more secure identification. For instance, a reddish tinge is found on bone residues only (Fig. 14, a), though at higher magnifications some darker spots were documented also on antler residues (Fig. 14: d). Furthermore, the granular aspect is more pronounced on antler residues, while the texture of bone remains appears to be saccharoidal (Fig. 14: d, c). The most ambiguous characteristic is clearly the transparent-like feature, typical of bone, but also present on antler residues. In fact, some optical images of antler residues (Fig. 14: b, f) astonishingly resemble some published bone fragments (*e.g.* Lombard 2008, Figs. 13, 14). The latter feature seems to make it impossible to discern between these two residue types by only resorting to optical microscopy imaging. Moreover, bone residues might also be misidentified for other materials, such as hide (Monnier et al. 2012). Analogies between compact portions of bone and other materials, such as antler, cane and skin, were also documented in this study (Fig. 18: a-c).

In general, SEM proved to be extremely effective to improve the capacity of discernment between residues bearing both morphological and chemical analogies. For instance, SEM was successfully employed to discriminate between animal hair and feathers

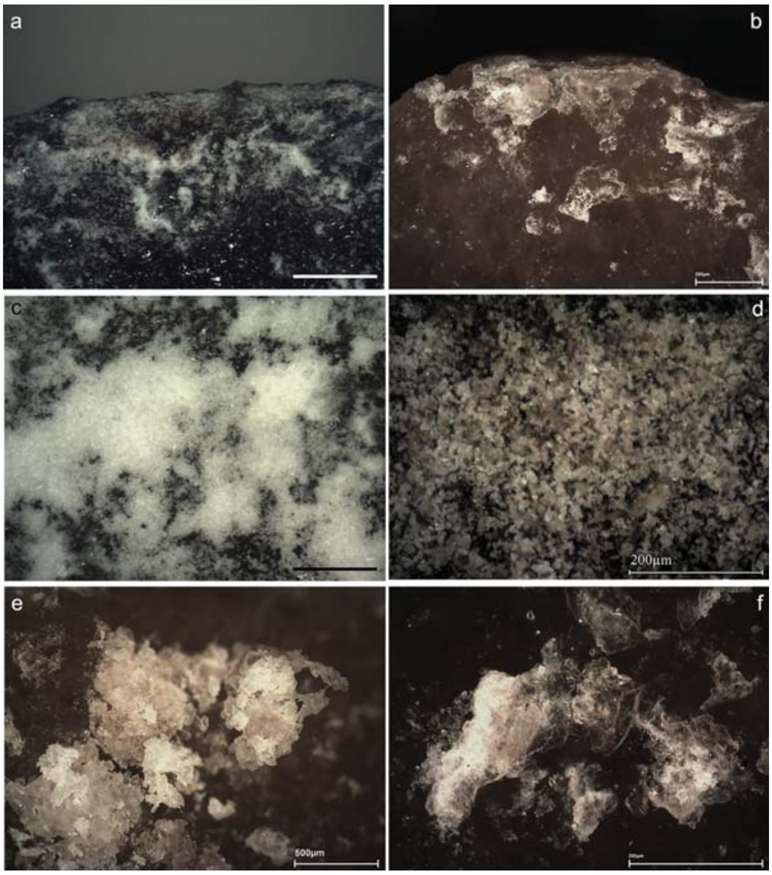

Fig. 14 Overlapping morphologies of bone (a, c, e) and antler (b, d, f) residues. Orig. mag. And scale bar: a) 50×, 500 μm (25 slides); b) 100×, 200 μm (20 slides); c) 100×, 250 μm (25 slides); d) 100×, 200 μm (11 slides); e) 50×, 500 μm (20 slides); f) 100×, 200 μm (20slides)

(Pedergnana and Blasco 2016). Hence, resorting to SEM imaging may be a feasible solution also to distinguish antler from bone remains. When comparing bone and antler residues, one can see that bone is always related to a lot of greasy material (since the used bones were fresh) (Fig. 16: a, b). Larger bone compact masses usually exhibit regular trends (lines, tiny holes). Antler always has a very pronounced granular aspect (Fig. 16: c, d) and, even when large antler accumulations appeared to be very similar to bone, the characteristic granular aspect is always present on the dispersed residues (Fig. 9: a).

It should be noted that this consideration was not confirmed by Monnier et al. (2012), where SEM was not useful for a correct identification of bone. Conversely, the identification of other residue types (such as *Fraxinus*) was clearly improved by the use of SEM (Monnier et al. 2012: 3298).

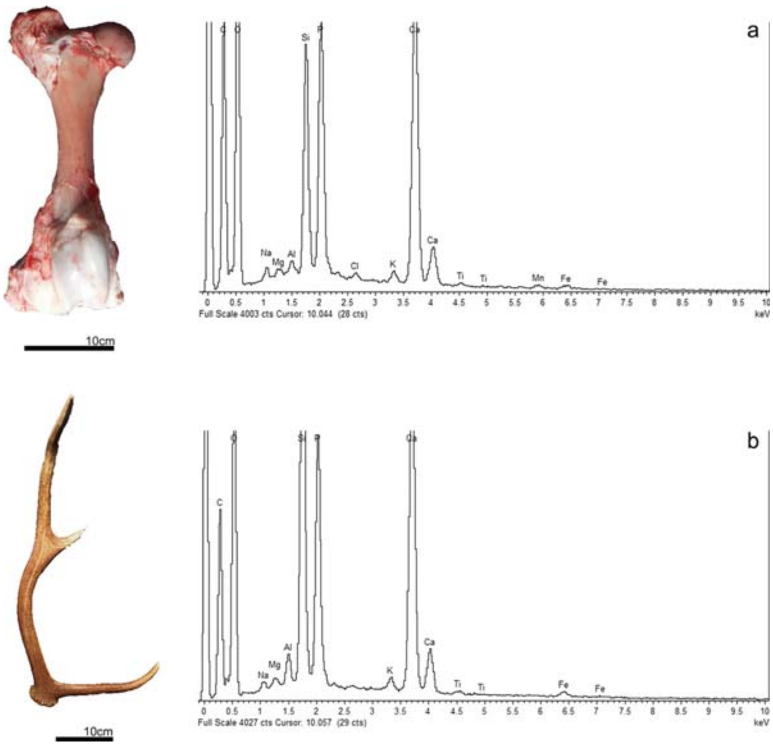

Fig. 15 EDX spectra of experimental bone (a) and antler (b), showing an almost identical composition. The main peaks are C, O, P and Ca, with minor concentrations of Na, Mg and K. Si, Al and Ti are attributed to the rock substrate (quartzite)

By combining these two methods, a bone fragment was successfully identified on a quartzite side-scraper (ATA04-TD10-I20–82) from level TD10 of the Gran Dolina site. The fragment appeared to be covered by sediment and diagnostic features of bone were visible with both OLM and SEM (Fig. 17: a-f). A more secure attestation was provided by the application of the elemental analysis (Fig. 17: g). The elemental maps of the residue correlated Ca and P contents to the bone fragment. Ca (red) is also present in the sediment (Fig. 17: c).

Fibres

Fibrous materials are perhaps the most unclear matter to identify. Firstly, fibres are documented both in faunal and vegetal materials (*e.g.* Fullagar 2006; Lombard 2004; Monnier et al. 2012). Secondly, fibres in general bear no particular distinctive traits permitting their attribution to a specific material. Perhaps different measurements or colours can be observed (*e.g.* reddish-brownish fibres for muscular or woody tissues, blood vessels), but fibres found on stone tools are mostly whitish in colour, often

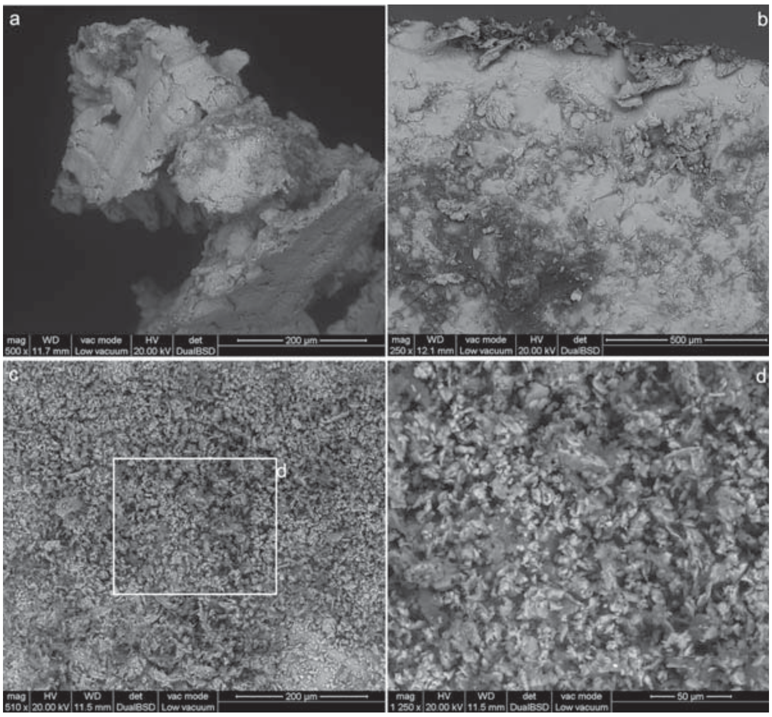

Fig. 16 Comparison of bone (qtfu1–06, scraping) and antler (qtfp1–07, cutting) residues under the SEM: a) compact mass of bone, 500×, 200 μm; b) bone micro-residues and greasy patches, 250×, 500 μm; c) granular aspect of antler residue, 510×, 200 μm; d) close-up of the same spot, 1250×, 50 μm

transparent and sometimes highly birefringent (Fig. 18). The birefringence value itself cannot be considered diagnostic, as it is related to several different materials (plant tissue, resins, muscle tissue, collagen, *etc.*).

Single fibres (observed at similar magnifications) are clearly more difficult to identify than bunches of fibres. Animal hairs, if not showing the characteristic cuticular layer, are not identifiable (Fig. 18: d) and might easily be mistaken for feather barbules (Fig. 18: e) or even for antler fibres (Fig. 18: f). A very analogous fibre was documented for bone residues as well (Monnier et al. 2012, Fig.12: f). Confusion is also encountered when accumulations of fibres are considered. Barbs (Fig. 18: g) strongly resemble woody and dry skin fibres, although they may appear straighter. Meanwhile, wood fibres (*Quercus*) contain some tiny pigmented spots (Fig. 18: h) and dry skin fibres appear to be more transparent (Fig. 18: i).

All of the aforementioned descriptions could only depend on the optical characteristics of the microscope used, or on a differential rotation of the prisms mounted on the microscope, *etc.* Colour identification also might be tricky. If white balance is not adjusted at the beginning of each observation and then kept constant throughout the entire working session, the real colour of the sample is not recorded (Fig. 13: d; Fig. 18:

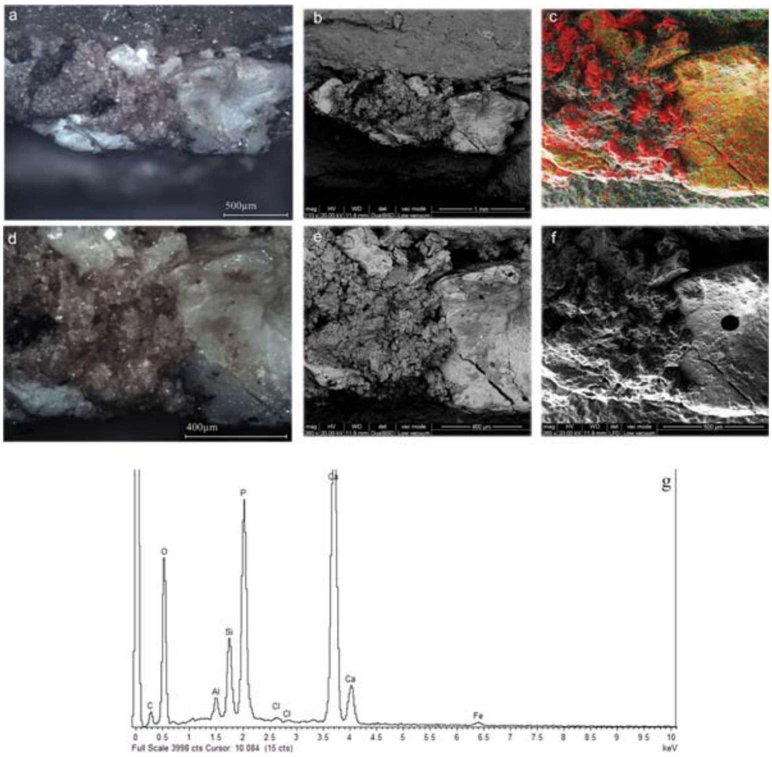

Fig. 17 Bone fragments on an archaeological quartzite tool (ATA04-TD10-I20–82). The fragments are embedded in the sediment concretion on one edge of the tool. The EDX mapping connects Ca (red) and P (green) to the bone; Ca is also present in the sediment concretion. Orig. mag. And scale bar: a) 50×, 500 μm, 24 slides (OLM); b) 110×, 1 mm, (BSD); c, f) 260×, 500 μm, (LDF); d) 100×, 400 μm, 24 slides (OLM); e) 260×, 400 μm, (BSD); g) EDX spectrum obtained from the residue. The black spot on image f indicates the exact point where the analysis was performed

f, n). Long, dry skin fibres, feather barbs and animal fibres again show astonishingly similar traits (Fig. 18: l-n). Hence, we should be extremely careful in defining parameters that are too rigid for identifying each material. Bearing in mind that we are observing experimental residues that have not undergone any burial processes, we understand that the identification of archaeological fibres is even more problematic.

The higher quality of SEM images helps to differentiate fibrous materials (Fig. 19). Topographical details of the residues are best imaged with SEM, improving the capacity of finding diagnostic traits for each material (Fig. 19: b, c, d, f).

In addition, EDX analysis provides instantaneous elemental composition of single points. Animal hairs always show high content of S and lesser Na, Cl and K percentages (Fig. 19: Sp1). Skins show similar composition, but with a lower average of S content (Fig. 19: Sp2). *Quercus* residues show a mixed composition (Al, K, Ca, Al) (Fig. 19: Sp3). Associated spectra for each worked material provide a better

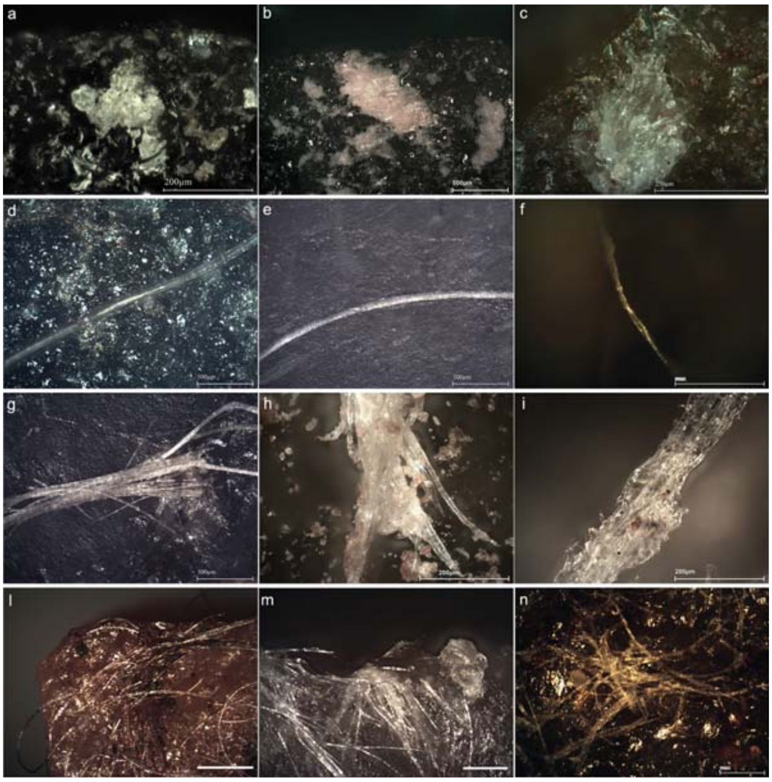

Fig. 18 Overlapping morphology of fibrous and compact materials. Each line comprises residues thought to be extremely similar. Fibres appear to be the most ambiguous ones. Orig. mag., scale bar, number of slides (when available), and material: a) 200×, 200 μm, 10 slides, (antler); b) 50×, 500 μm, 10 slides, (giant cane); c) 200×, 250 μm, 13 slides, (dry skin); d) 50×, 500 μm, (deer hair); e) 50×, 500 μm, (vulture barbule); f) 200×, 250 μm, (antler fibre); g) 50×, 500 μm, (agglomerate of vulture barbules); h) 50×, 500 μm, 27 slides, (wood accumulation of fibres); i) 200×, 200 μm, 3 slides, (dry skin fibres); l) 50×, 500 μm, 15 slides, (dry skin fibres); m) 100×, 200 μm, (vulture barbules); n) 100×, 200 μm, 20 slides, (animal fibres, possibly collagen)

characterisation of fibres in general. Although results of the elemental composition of fibres are hardly conclusive, they can lead closer to a correct interpretation. In fact, the presence and absence of some particular elements may be mutually exclusive. For instance, the occurrence of sulphur can be associated either to skin, hair or feathers, but never to wood (Fig. 19).

Residues Distribution Patterns on Stone Tools

Distribution of micro-residues is generally recorded during analyses at least when residues are observed *in situ* (directly on the tools). Certain distribution patterns have been recognised on archaeological artefacts. For example, animal residues were

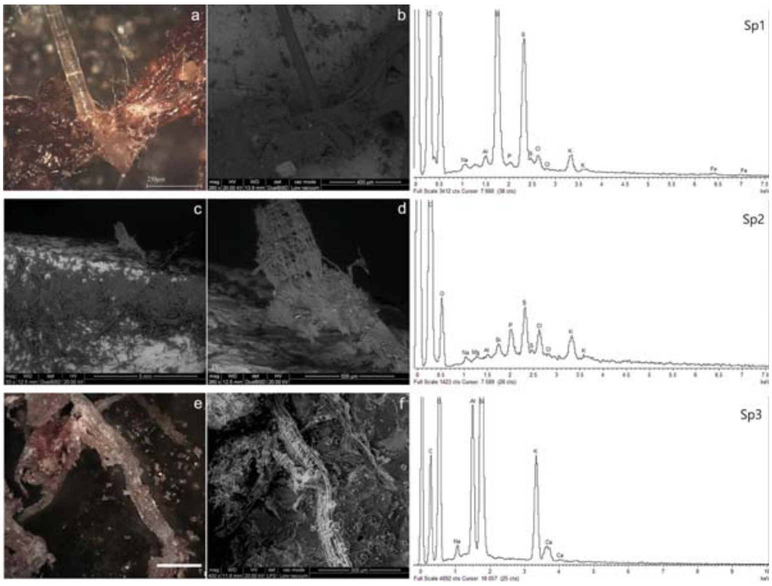

Fig. 19 Appearance of three fibrous materials and their relative spectra. Red deer hair: a) 100×, 250 μm, (OLM); b) 260×, 400 μm, (BSD); Dry skin: c) 50×, 3 mm, (BSD); d) 260×, 500 μm, (BSD); Wood: e) 100×, 200 μm, (OLM); f) 400×, 300 μm, (LFD)

concentrated on the distal tool edges, while vegetal residues were found on proximal and mesial parts of the artefacts (Lombard 2004). Contaminants are described to appear in isolated spots, while use-related residues are seen as relatively large accumulations along the used edge (Langejans 2010; Wadley and Lombard 2007).

Experimentally, residues seem to have distributional patterns related more to the type of movement (longitudinal, transversal, rotational, *etc.*) than to the residue type (Rots et al. 2016) or to the relative water content (Xhauflair et al. 2017).

In fact, residues visible macroscopically on experimental tools before washing are most often concentrated on the active edges (Fig. 2). Microscopically, these residues show certain directionalities (diagonal from the edge for transversal actions, parallel for longitudinal ones). Sometimes it is even possible to discern directionality by observing muscle fibres which preserve the original organisational pattern of the muscular tissue (Fig. 4). The major problem in such observations is that they can only be made in relation to experimental tools (as macro-residues are generally available). However, even on experimental tools, directionality is not always deducible at a macro-scale. For instance, residues with a higher adherence capacity (watery residues) tend to cover wider areas on the stone tool surfaces. Tools employed in the butchering activity present an aleatory pattern of residue distribution, perhaps due to the presence of grease and blood, which contribute to a larger dispersal of more solid residues (muscle and fat fibres) (Fig. 2: a). When tools are in contact with blood and the activity is performed outdoors, particles from the ground such as earth or plant fibres might be deposited onto the tool surfaces (Fig. 2: c, f, g). Therefore, residue distribution seems to be a more complicated topic than previously thought. Hard materials (bone, antler, wood) indeed show clear concentrations relatively near to the tool's used edges. Nevertheless, when surfaces are observed under the SEM, micro-residues are present on different areas of the tools, often reaching the opposite (un-used) edge (Fig. 20: a, b, d, f). The most

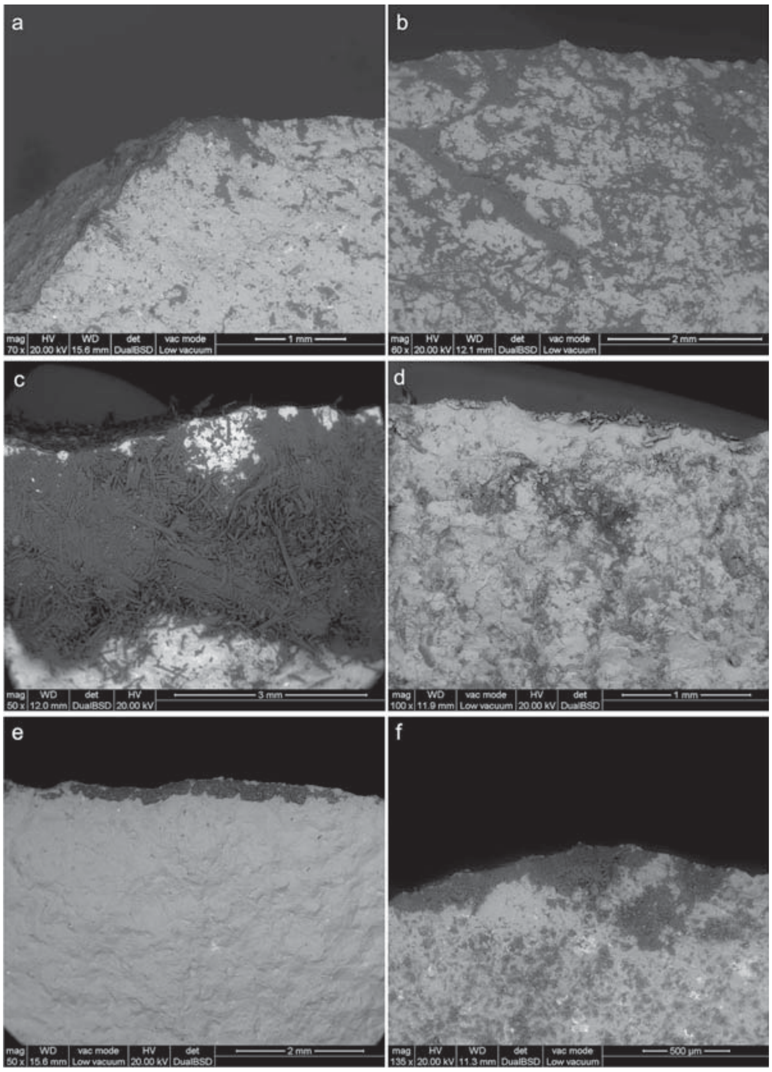

Fig. 20 Distribution pattern of some residues: Butchering: a) 70×, 1 mm; b) 60×, 2 mm; Scraping dry skin: c) 50×, 3 mm; Scraping bone: d) 100×, 1 mm; Sawing antler: e) 50×, 2 mm; Sawing wood: f) 135×, 500 μm. All the images were taken with a SEM-backscattered-electron detector (BSD)

coherent distribution (residues only found on the used edge with no sparse micro-residues) was observed on tools used to saw dry antler (Fig. 20: e). We might expect a different situation when the antler is soaked prior utilisation (because of higher water content).

As pointed out by Langejans (2010), differential preservation residue indexes might lead to false interpretations. In the same way, the inference of the used edge by only plotting the location of micro-residues on archaeological surfaces might lead to interpretation bias. If residues of different materials have differential preservation rates (depending on the soil conditions, among other factors) (Langejans 2010), decay may also affect residues differently with regard to their position. Therefore, residues found on the used edges might even be more poorly preserved than those found on unused parts of the tools.

However, even if meaningful patters are usually interpreted as products of use and contaminants are found to have a random organisation (Lombard 2004, 2005; Lombard and Wadley 2007), it has been proven that experimental burial seriously compromises the legibility of specific residue spatial organisations (Langejans 2011). We are forced to consider that this phenomenon happens with much higher frequencies in an archaeological context. Moreover, the scenario may worsen: modern contaminants may also present clear distributions along the edges of the tools (Pedergnana et al. 2016). This adds serious problems when the only criterion adopted to identify active edges is based on the residues' distributional patterns.

An additional issue, so far not extensively discussed in the contributions on residue analysis, concerns the rock substrate. It is reasonable to think that rock topography may influence the distribution of residues. Flat and smooth surfaces (*e.g.* flint, obsidian, rock crystal) are likely to present a more widespread distribution of residues in general, while rough and irregular rocks (*e.g.* basalt, quartzite, rhyolite) would more easily trap residues into micro-holes and depressions. In fact, it has been experimentally observed that this happens at least with some contaminant substances (skin flakes, moulding clay) (Pedergnana et al. 2016).

In any case, the recording of residue location, as well as of use-wear, are important, even if they entail functional meaning only when the analysed residues exhibit excellent preservation.

Discussion

One of the most problematic issues taken into account in the attempt to identify residues on archaeological tools is that of preservation. Taphonomic processes obviously induce important structural changes to all kind of residues. Because of this, direct visual comparison of smashed residues with laboratory prepared specimens is not recommended.

As the most frequently used technique to observe micro-residues up to now has been optical light microscopy, interpretation has relied only upon visual features (morphology and colour). Surely, OLM is a valuable tool for an initial approximation, but we propose that it cannot provide exhaustive results, not only because of the post-depositional changes, but also because of the numerous ambiguities recorded on experimental fresh residues.

For instance, the compact aspect of osseous accumulations, that are sometimes transparent (Fig. 7: c, d; Fig. 14: e; Monnier et al. 2012 Fig. 12: a, b, d; Lombard and Wadley 2007, Fig. 4: e; Lombard 2008: Figs. 13, 14) is also found on antler residues (Fig. 14: b, f). In some cases, the presence of greasy films together with whitish bone fragments (Fig. 7: b, e; Fig. 8: c; Fig. 14: a) or tissue fibres (Fig. 7: c) are found on experimental tools and can be used to differentiate bone from antler. Nevertheless, brown pigmentations are rarely seen also within antler residues (Fig. 14: d).

While the birefringent character was sometimes claimed to cause general confusion in distinguishing animal from plant residues (Lombard and Wadley 2007) and overlaps were also described for hide and bone (Monnier et al. 2012), the main ambiguous residues underlined in this study were bone and antler. Generally, most of the similarities considered to be tricky were found mainly while using OLM. SEM, in general, overcomes the main obstacles by reaching higher magnifications and thus obtaining greater topographical details.

Nevertheless, SEM is not always thought to improve the quality of results. For instance, in the Monnier et al. study (Monnier et al. 2012: 3298), SEM did not significantly improve the capacity of distinguishing bone from either hide or wood. Furthermore, the same work also describes difficulties in recognising bone residues with SEM. We did not face such problems during our observations. On the contrary, bone micro-residues appeared quite distinctive under the SEM (Fig. 7). Both compact bone and bone fragments mixed with grease and collagen exhibited characteristic traits which cannot be misinterpreted (Fig. 7: f, e).

Additionally, we suggest that SEM observations are crucial to better characterise fibrous materials and to distinguish bone from antler residues, which are extremely ambiguous under the OLM. Also, elemental analysis cannot solve the problem, since both materials have very similar chemical composition. Therefore, as their appearance under the SEM is quite distinctive, it is recommended to provide SEM micro-graphs alongside the classic optical images when analysing archaeological residues identified as bone. This should be sufficient to permit correct residue identification.

Hence, the systematic combination of OLM and SEM observations in the direct analysis of residues found on stone tools is a valuable choice to improve identification confidence (Borel et al. 2014; Monnier et al. 2012). However, SEM proved to be more reliable in providing specific diagnostic traits, we think thanks to its higher resolution. For instance, collagen (or animal fibres in general) exhibit flat and compact textures, contrasting with the cuticular layer of hairs. Feathers can be recognised if entire barbs or characteristic anatomical features of barbules are present. A general granular aspect is always observed for antler residues, while, although present also in bone residues, it is not so pronounced. Bone is also generally accompanied by organic, greasy portions (very easily detectable under the SEM).

However, for a better characterisation of residues in general, especially when combined OLM and SEM microscopy together with EDX data acquired are insufficient to identify a specimen, more sophisticated analytical techniques are available. In this kind of studies, the concept of biomarker is fundamental (Evershed 2008), being the characteristic chemical finger-print of each substance. It is clear that, to be able to access the biochemistry of residues, the technique employed has to be capable of reaching a high resolution; up to the molecular level.

Among the available techniques providing characteristic vibrational spectra of materials, Raman spectroscopy is broadly used in art history and conservation (Smith and Clark 2004), while it is somewhat problematic when the analysed samples are naturally florescent (organic materials). These kinds of problems are generally avoided by FT-IR, which is more suitable for analysing organic compounds. In fact, FT-IR has been proved to generate characteristic spectra for materials likely to be found on lithic tools (Prinsloo et al. 2014).

Gas chromatography (GC), associated to mass spectroscopy (MS) also proved useful to investigate molecular bonds of organic residues (Evershed et al. 1990; Evershed 2008). Although this technique has been extensively applied to ceramic material to analyse lipidic compounds (see inside Barnard and Eerkens 2007), it can be very promising for future research applied to other classes of artefacts, such as stone tools. GC-MS, despite being a destructive technique, it seems to be a valid choice especially to analyse waxes, resins and petroleum bitumens, as demonstrated in recent works (Boëda et al. 2008; Cârciumaru et al. 2012; Hauck et al. 2013; Helwig et al. 2014; Mazza et al. 2006).

GCxGC-TOFMS (comprehensive two-dimensional gas chromatography-time-of-flight mass spectrometry) differs from the traditional GC-MS for being a non-destructive technique. A first test on experimental samples was recently carried out and results are indeed promising (Perrault et al. 2016). It appears to be a valuable source of information regarding adhesive compounds, while it did not provide strong spectra for regular organic materials involved in tasks performed with the aid of stone tools (e. g. bone, hide, meat), possibly because there were no volatile compounds within those samples.

However, when these biochemical analytical techniques are not available, SEM imagining alone seems to be a fast solution for the analysis of residues, providing reliable insights. Moreover, at least for a first approximation, the use of SEM micro-analysis helps in giving some clues about the composition of the residues (Dinnis et al. 2009; Pawlik 2004a), while more sophisticated techniques can be applied subsequently.

Residue distribution has also been considered in this study and it demonstrates that, although identifying the exact location on stone tools is a valuable criterion, it cannot provide reliable functional interpretations. In fact, depending on the activity performed, fresh experimental residues might be very sparse or concentrated near the used edges. Residue distribution seems to be dependent upon several causes, such as the kinematics, edge morphology, water content, the hardness of the worked material, *etc.* Moreover, considering that soil conditions might selectively preserve some specific categories of residues (not necessarily connected to the worked material) (Langejans 2010), we can assume that residue patterns found in the archaeological record can hardly correspond to the original distribution (soon after the conclusion of the tasks). Additional implications are given by the stone tools' life after excavation: innumerable contaminants can be superimposed to the ancient residues, puzzling even more the analysts (Pedergnana et al. 2016; Rots et al. 2016; Xhauflair et al. 2017). For all of these reasons, caution always needs to be used in order to avoid erroneous interpretations.

Aiming mainly to shed light on the question of interpreting residue distributional patterns on archaeological materials, a good approach would be to set up long-term experiments involving the monitoring of the decay of residues after burying them in different environments (*e.g.* dry, humid, acid-basic soils) and types of sites (caves, open-air locations such as fluvial terraces, lake shores, mountainous camps) (*e.g.* Croft et al. 2016; Langejans 2010; Rots et al. 2016). This would also help us to understand the real impact that burial has on the organic substances and in evaluating to what extent morphological and chemical changes can compromise the correct identification of the residue type.

Conclusion

A first approach to building a residue comparative collection is provided, including morphological and elemental characterisation of 7 materials. Much effort has been given to providing a reliable photographic catalogue, intended to be useful for comparisons in future residue analyses of archaeological collections.

SEM observations improve the identification confidence of residues, compared to the only OLM scanning. Knowing that residues change their aspect after being buried, we insisted on the necessity of searching for the chemical/elemental composition of residues.

Moreover, SEM-EDX is considered a relatively rapid, non-destructive and very valuable tool to provide preliminary data about the composition of the observed residues. Although no techniques other than EDX were applied to the experimental residues, we think that its great advantage with respect to other widely used techniques (such as GC-MS), is that it does not destroy the residues analysed. Also, fragile objects can be analysed with this technique as samples do not require specific preparation.

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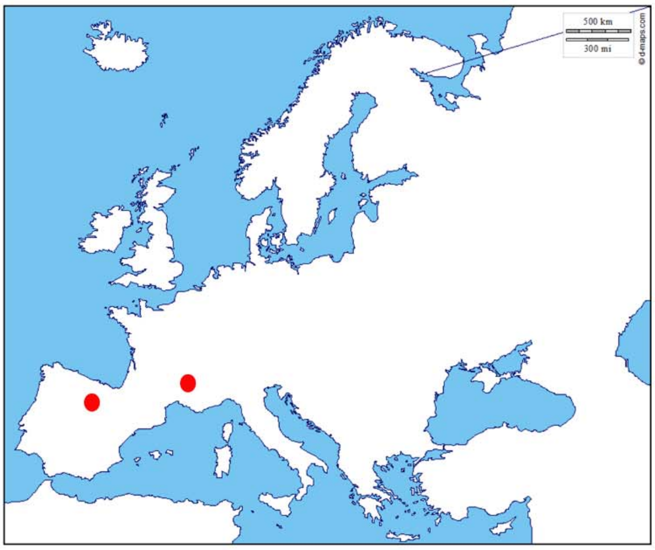

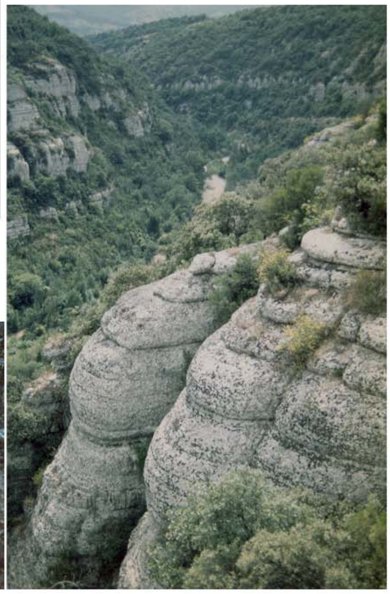

PART IV: THE CASE STUDIES

Chapter 6: The Gran Dolina site

Gran Dolina is one of the most known sites pertaining to the complex of the Sierra de Atapuerca. First, a brief contextualisation of the Sierra is provided for then focusing on the studied site.

6.1 The complex of the Sierra de Atapuerca

The *Sierra de Atapuerca* (the Atapuerca Range) is an anticline (NNW-SSE) located in the northern part of the Iberian Peninsula (Fig. 6.1: a), in the Duero Basin, at an approximate elevation of 1000 m.a.s.l. (Fig. 6.1: b-e). The nearest city is Burgos (*ca.* 15 km), one of the main city of the Spanish province of *La Castilla y León*.

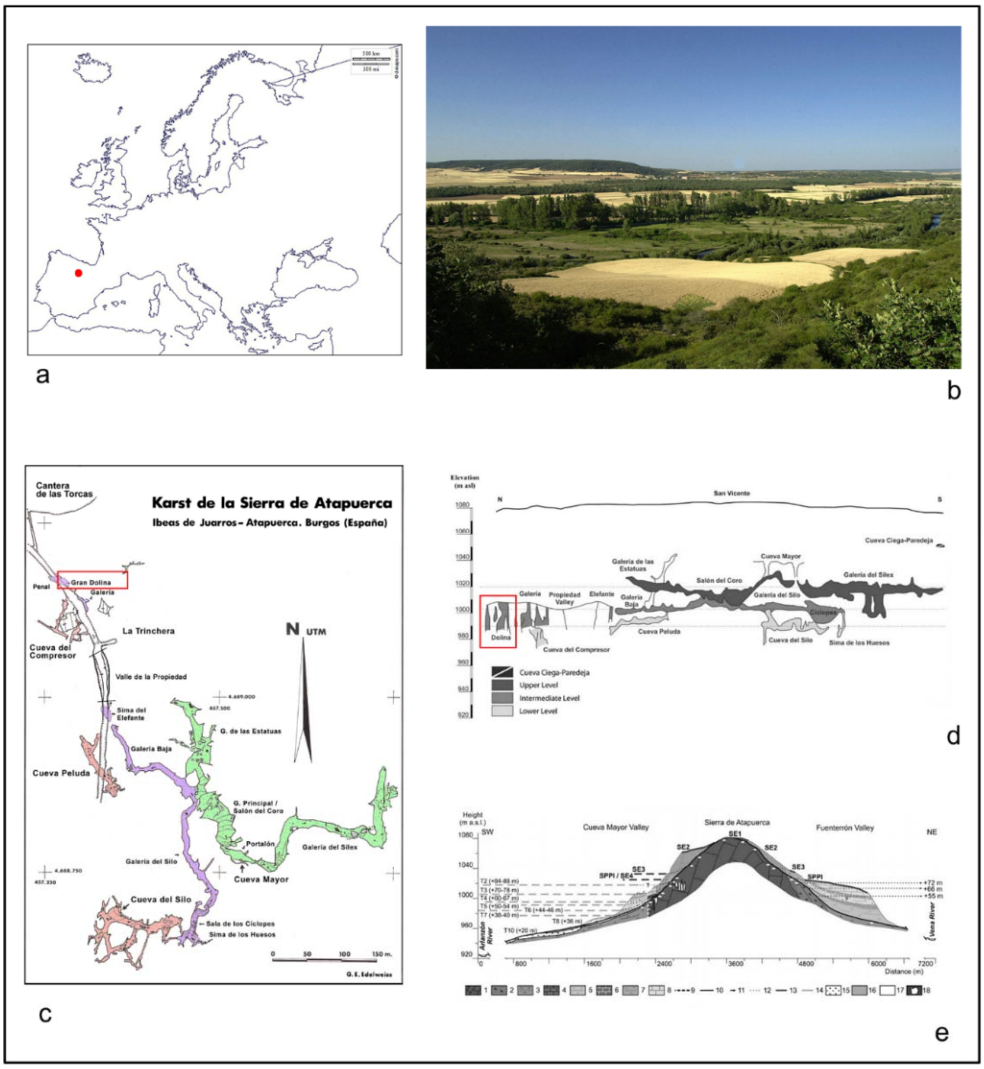

Fig. 6.1: *a) Location of the Sierra de Atapuerca in the European continent; b) general view of the Sierra at the back and of the Arlanzón River at the front (IPHES-Atapuerca Research Team-A. Ollé); c) map of the Atapuerca karst system with the three sub-horizontal levels underlined by different colours. The Gran Dolina site is signalised by a red rectangular; d) longitudinal view of the karst system; e) altitude of the Sierra. Images c, d and e are modified from Ortega et al., 2015.*

The Sierra de Atapuerca is a karst range, formed during the Mesozoic (Cretaceous), connected with the Arlanzón and Vena river valleys (Fig. 6:1: c). Many archaeological sites have been identified both into the karst system, in the form of infilled cavities, and outside, as open-air sites (Carbonell et al., 2014). The archaeo-paleontological evidence coming from those sites covers more than one million years, spanning from the Lower Pleistocene to the Holocene (Rodríguez et al., 2011; Ollé et al., 2013). Due to the importance of these findings, some of them being keys in the reconstruction of human evolution of the European continent, the archaeological sites at Atapuerca were declared a World Heritage Site by UNESCO in 2000.

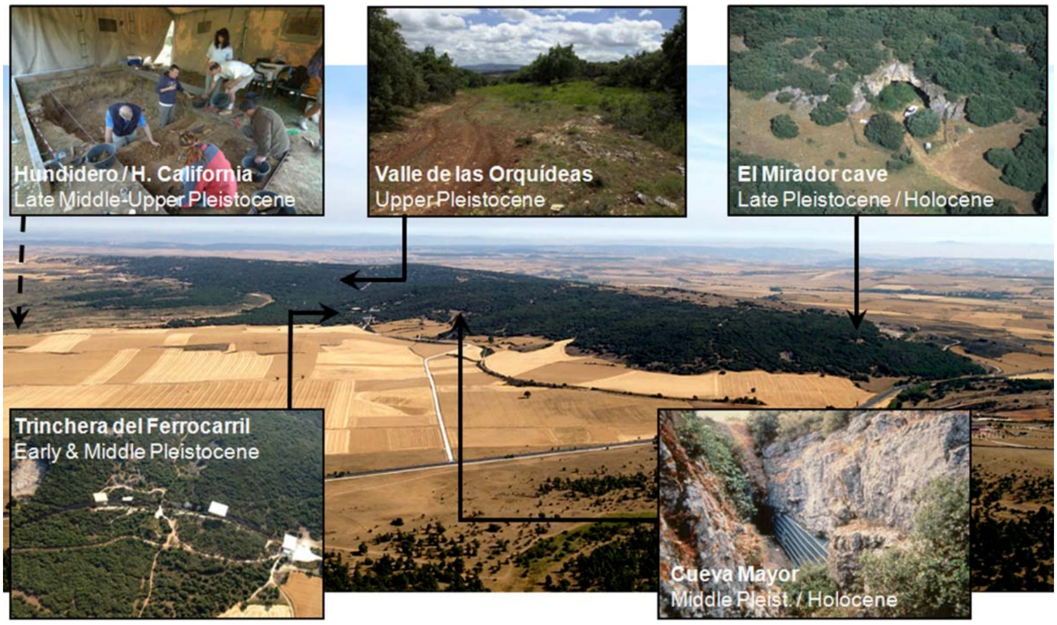

Fig. 6.2: *General view of the Sierra de Atapuerca and the disposition of the main sectors where the archaeological sites are located (IPHES-Atapuerca Research Team).*

Four distinct sectors, comprising several sites, were defined: The Trinchera del Ferrocarril, Cueva del Mirador, Cueva Mayor-Cueva del Silo complex and the open-air sites area. The cave sites are: Sima del Elefante, Galería, Gran Dolina, Sima de los Huesos, Portalón de Cueva Mayor y Cueva del Mirador; while the open-air sites are Hotel California, Hundidero, Fuente Mudarra and Valle de las Orquídeas (Fig. 6.2).

The Sierra has a strategic position with regard to the location of the lithic raw materials apt for the knapping activity (Fig. 6.3). Neogene (Final Miocene) chert outcrops are found in the immediate surroundings of the Atapuerca Ridge, especially on the southwestern slope, while Cretaceous chert (Upper Cretaceous) is found on the top of the Ridge as well as at the interior of one of the cavities (*Galería del Sílex*) (García-Antón et al., 2002; García-Antón, 2016).

Fluvial raw materials (quartzite, sandstone and quartz) are commonly found at the primary geological sources (Fig. 6.3) as well as on the banks of the Arlanzón River. The Middle-Upper Pleistocene terrace (which chronologically corresponds to TD10 level) is found 1.2 km from the site. The primary geological sources are identified as different locations in the

Paleozoic ridge of La Demanda (located 17 km east of the Sierra). The Utrillas facies, containing fine-grained quartzites, is found in the Atapuerca ridge (García-Antón et al., 2002; García-Antón, 2016).

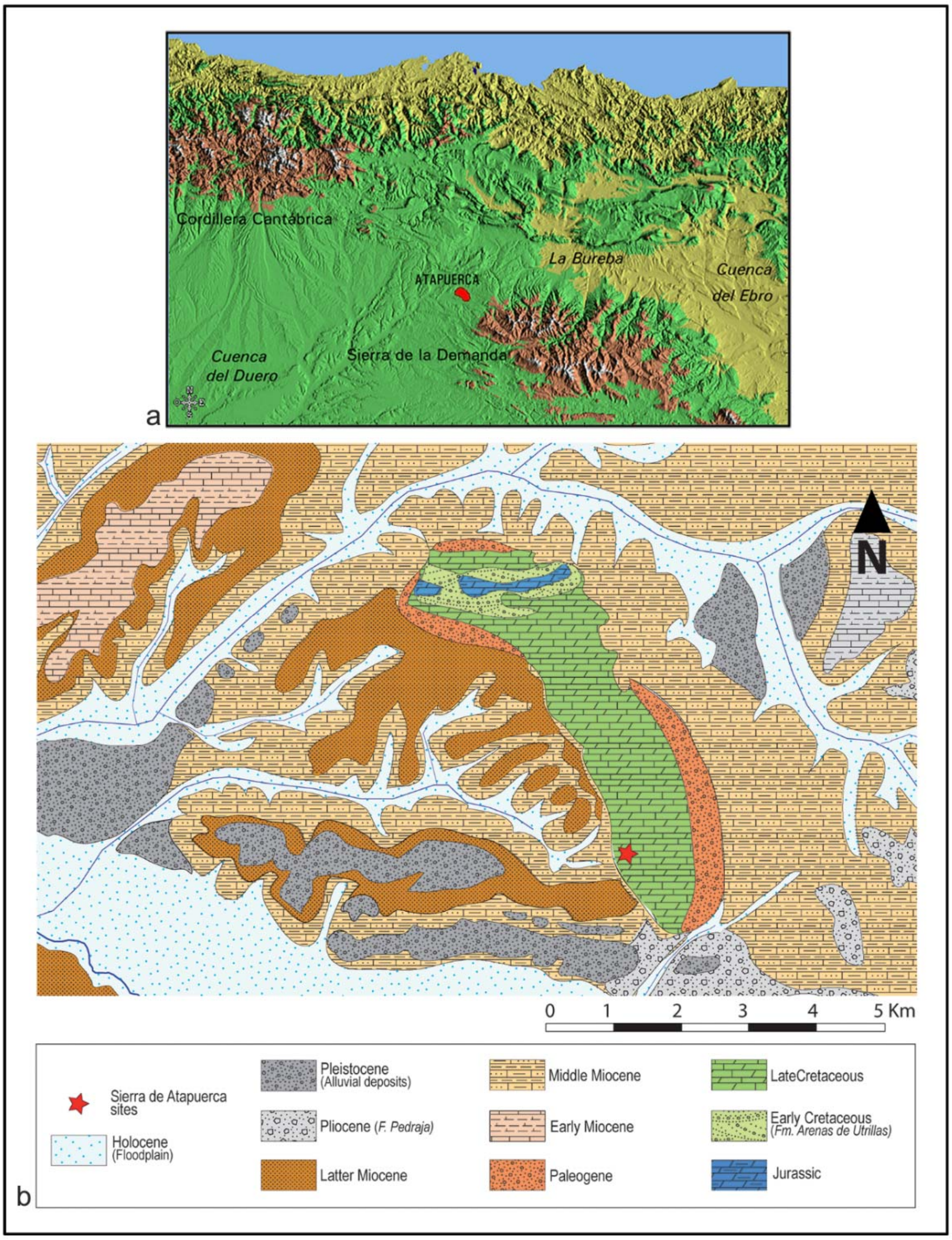

Fig. 6.3: *Geological settings of the Sierra de Atapuerca: a) its location in the Bureba corridor, in between the basins of the Duero River and the Ebro River; b) geological map of the Sierra showing the main areas of raw material procurement (note: the Arenas de Utrillas formation in light green and the Pedraja facies formation in light grey, both connected to the experimental quartzites used in this study. Floodplains are also important for the fluvial cobbles procurement (Miocene and Pleistocene deposits). (Modified from Pedergnana et al., 2017).*

6.1.1 Cueva Mayor-Cueva del Silo complex

The entrance of the Cueva Mayor-Cueva del Silo complex is found at approximately 1 km from the Trinchera del Ferrocarril. It includes many sedimentary sequences, the main ones being the Portalón and the Sima de los Huesos sites.

The Portalón cave

The Portalón site is one of the current entrances to the Cueva-Mayor-Cueva del Silo karst system. Its long stratigraphic sequence (over 9 m) is divided into a Pleistocene and a Holocene unit. Human presence is more attested in the Holocene layers, which span from the Mesolithic until the historical period (Medieval and Roman times). Ceramics, bone tools and personal items were found in the Bronze Age levels, revealing an intense occupation of the cave. The main function related to the site is domestic animal livestock shelter (Carretero et al., 2008). The identified animals are cow, sheep, goat, pig and horse. The great quantity of horse remains as well as the systematically associated cut marks, are indexes of domestication (Galindo-Pellicena et al., 2014).

A collective burial is dated to the Chalcolithic and an intact burial of a child was found in 2012. Anthropological analysis revealed two diseases affecting the bones of this young individual, rickets and scurvy (Castilla et al., 2014).

Sima de los Huesos

The Sima de los Huesos site is found at c.a. 500 m from the entrance to Cueva Mayor, it belongs to the third level of the karst system and it is found at the bottom of a 13m vertical shaft (Arsuaga et al., 1997). It yielded a remarkably numerous and well preserved Middle Pleistocene human fossil record, corresponding to at least 28 individuals. The skeletal representation includes all anatomical parts and the average age study of the fossils identified many adolescents or young adults, with very few children or senile individuals. After a previous ascription of the fossils to the *H. heidelbergensis* species (Arsuaga et al., 1997), recent studies proposed to remove those from this taxon, due to extreme differences between the SH record and the holotype of *H. Heidelbergensis* (the Mauer mandible, Mounier et al., 2009). In fact, the SH record shows more similarities with the Neanderthal lineage than the Mauer mandible does. Nevertheless, recent genetic studies showed that the analysed sample from SH was closer to the Denisovan population living in Siberia 40kya that to the Neanderthals (Meyer et al., 2014). Luminescence dating provided a minimum age of 427±12ka for the layer underlying the accumulation of hominin fossil (Arnold et al., 2014).

Different hypotheses were put out to explain this astonishing accumulation of human bones, but after detailed taphonomic and stratigraphic analyses (Aranburu et al. 2017; Sala et al., 2012, 2015), there was only one stratigraphic event connected to the hominin deposition. Secondly, there is a low incidence of carnivore activity on the hominin bones. These studies also underlined that the fossils are in primary position, contrasting to the hypotheses which wanted the bones to have suffered long transportation events.

The only stone tool recovered from the site is an Acheulean handaxe made of a high quality fine-grained metaquartzite (Fig. 6.4). This tool was made on a fluvial cobble, probably collected on one of the floodplains of the Arlanzón River. The presence of this unique tool can be due either to a fortuitous fall together with one of the humans or to an intentional throw in some unidentified mortuary practices (Carbonell and Mosquera, 2006).

Fig. 6.4: *The only lithic tool recovered at the Sima de los Huesos site, a quartzite amygdaloidal handaxe (Ollé et al., 2016a:320).*

6.1.2 The Mirador cave

The Mirador cave is located on the southernmost slope of the Sierra de Atapuerca. Its present appearance is that one of rock-shelter due to the partial collapse of the roof. Archaeological excavations began in 1999 and have continued until the present. A 6m^2 test pit excavated between 1999 and 2008 identified 24 Holocene and two Pleistocene units (Vergès et al., 2016). The Pleistocene sequence is composed of 14m of blocks fallen from the roof and only two sedimentary units. One of them showed evidence of human presence and it was dated to 13 580-13 420 cal BP. Directly above the last unit containing limestone blocks, a 6m Holocene sequence was identified, corresponding to a chronological framework spanning from the Early Neolithic (4th millennium BC) until the Bronze Age (2th millennium cal. BC) (Vergès et al., 2016). The Holocene sedimentary package is mainly composed of alternating layers of burned livestock dung and partially burned or unburned layers, producing a singular colour fluctuation (Fig. 6.5). This sedimentary pattern is known as "*fumiers*" and it is typical for sites whose primary function is animal husbandry. The regular combustion of the animal dung aims at reducing its volume and at eliminating parasites (Angelucci et al., 2009). The use of the cave as a livestock pen for domestic animals, mainly goats and sheep, is confirmed by the high number of fetal and neonatal individuals (Martín et al., 2016). Along the consumption of domestic animals, ungulates and small preys were hunted. Moreover, the sporadic consumption of carnivores was also attested (Martín et al., 2014).

From 2009 to the present two new sectors (sectors 100 and 200) have been excavated, identifying three units exhibiting continuity in the use of the site as domestic animal livestock (sector 100). In the same sector the last occupational phase of the cave is embodied by a single burial of a young male, whose radiocarbon age is 3 670-3470 cal. BP. The excavation of the sector 200 revealed the presence of a Chalcolithic collective burial (*ca*. 4 500 BP), where 23 individuals have been identified up to present. Human remains are found in a small chamber and are not in anatomical connection.

Another episode of human burials is dated to the beginning of the Bronze Age (in the final third of the 3rd millennium), where the remains of six individuals were found in a small hole (Vergès et al., 2016). The taphonomic analysis of the human bones highlighted the presence of cut marks and other evidences pointing to cannibalism (Cáceres et al., 2007).

The material culture includes both lithic and ceramic implements. Lithic tools were collected in all the excavated levels (Palaeolithic, Neolithic and Bronze Age). Laminar technology is clearly present and recycling episodes were also observed. The material shows strong thermal alteration, due to the frequent burning events. The most used raw material is chert throughout all the sequence, followed by quartzite. Retouched implements are not very abundant, being denticulates, notches and circles. Ceramic remains are more abundant in the Neolithic levels than in the Bronze Age ones. Also, a higher number of remains are attested in the Early Neolithic levels. Differences in morphology, volume and decorations have also been described, suggesting differences in the intensity of occupation of the cave (Vergès et al., 2016).

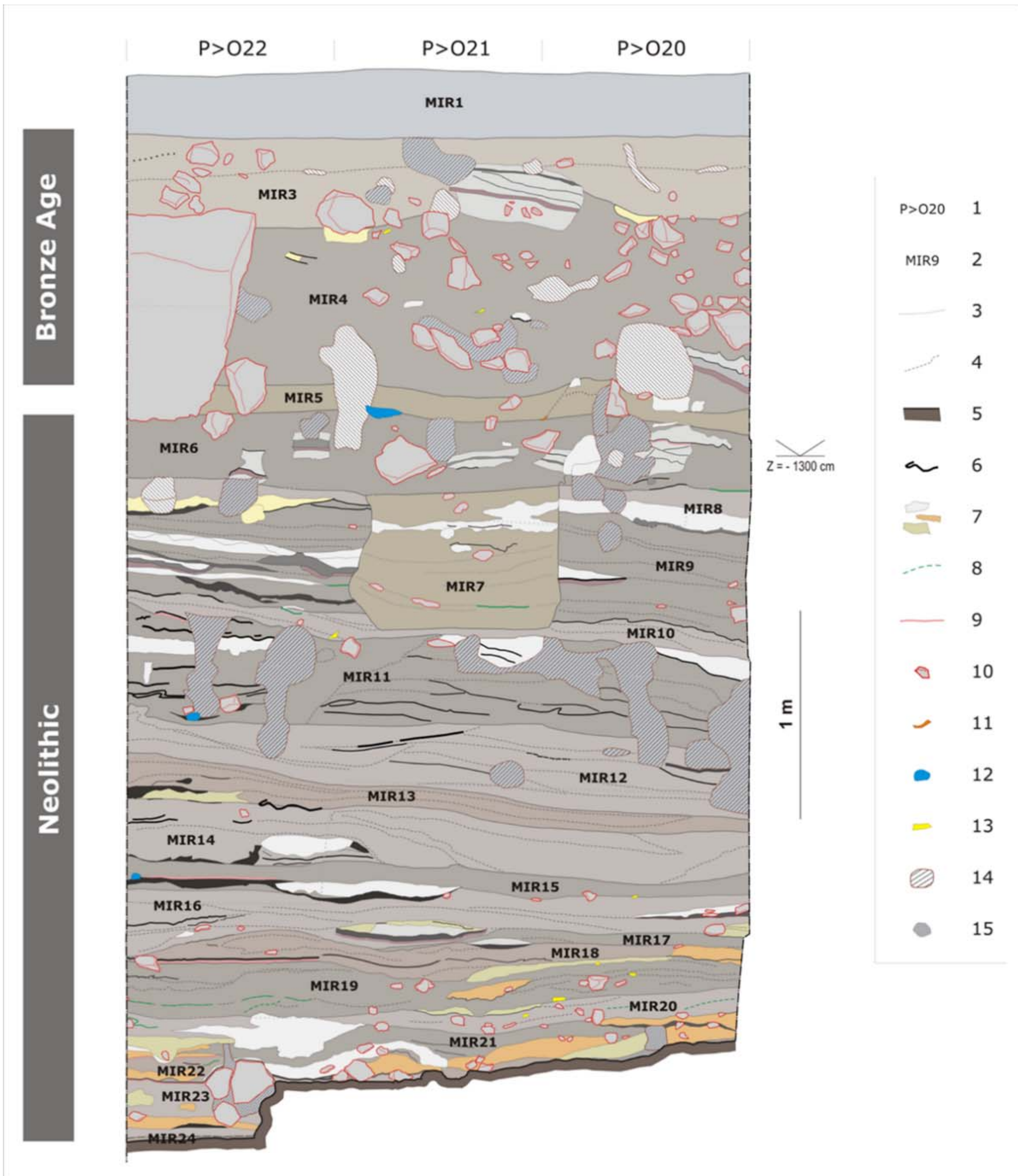

Fig. 6.5: *Stratigraphic sequence of the Mirador cave, with the typical structure of "fumiers" (adapted from Vergès et al., 2016).*

6.1.3 Open-air sites

Several open-air sites attested the human presence in the Upper Pleistocene at the Atapuerca Range. Archaeological surveys discovered 31 open-air site and some promising localities were excavated (Navazo, 2006). The excavated sites are Valle de las Orquídeas, Hotel California and Fuente Mudarra. They provided several occupational layers and dates span from *ca.* 70 to 27 ka BP. Typical Middle Pleistocene lithic implements were recovered, mainly denticulates, sidescrapers and notches. The most used raw material was the Neogene chert, followed by the Cretaceous variety and quartzite. Reduction strategies include in all cases centripetal and unifacial methods (Carbonell et al., 2014, Navazo and Carbonell, 2014).

6.1.4 Trinchera del Ferrocarril

The Trinchera del Ferrocarril (railway trench) was constructed at the end of the XIX century and is 500 metres long, located in the southern part of the Sierra (Fig. 6.6: g). The cutting in the limestone walls exposed several cavities infilled with sediments of different age. In three of the cavities archaeological and paleontological remains were found. From north to south, Gran Dolina, Galería and Sima del Elefante are found in the Trinchera (Fig. 6.6: a).

Gran Dolina

The Gran Dolina site is the northernmost site in the Trinchera. The stratigraphic sequence is composed of 11 litostratigraphic levels, TD1 to TD11 (from the base to the top) (Parés and Pérez-Gonzáles, 1999; Pérez-González et al., 2001) (Fig. 6.6: b, e). The sequence corresponds to a very long chronological frame, which is divided into two main blocks by the Matuyama-Brunhes boundary identified in TD7 (Parés and Pérez-González, 1999). The reversal in Earth's magnetic pole is known to have occurred 780,000 years ago. The record below this boundary is ascribed to the Lower Pleistocene, while evidence coming from the above levels have a Middle Pleistocene age. A more detailed description of each level is provided below (paragraph 6.2).

A conjunct of human fossils was uncovered in the TD6 layer (*Aurora stratum*), during the 1994 and 1995 campaigns (Carbonell et al., 1995b). More than 80 fragments corresponding to at least six individuals slightly older than 780 000 were ascribed to a new species, *H. antecessor* (Bermúdez de Castro et al., 1997). The new ascription was based on the remarkable characteristics of the fossils, which combined primitive and modern traits. After more than 15 years of studies, this combination of primitive and derived features is confirmed, underlying similarities with both modern and Neanderthal lineages (Bermúdez de Castro et al., 2017).

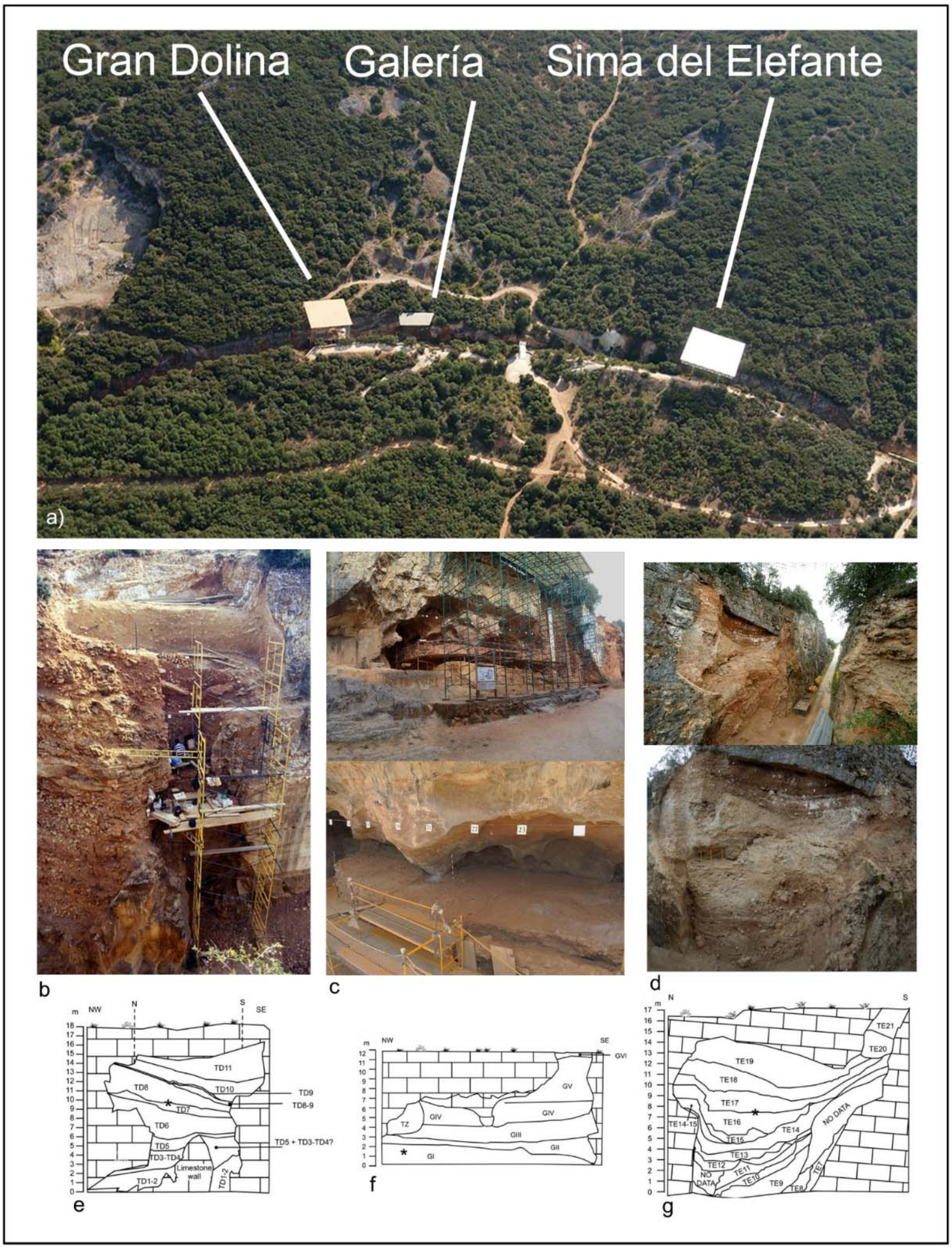

Fig. 6.6: *The Trinchera del Ferrocarril (railway trench) sector. a) Aerial view of the Trinchera with the three archaeological sites signalised; b, e) The Gran Dolina site and stratigraphy; c, f) The Galería site and stratigraphy; d, g) The Sima del Elefante site and stratigraphy. (a-d, IPHES-Atapuerca Research Team). Pictures e, f and g are modified from Rodríguez et al., 2011.*

Galería

The Galería site is found in between Gran Dolina and Sima del Elefante (Fig. 6.5: a). The cave is c.a. 14m high and 18 m wide (Fig. 6.5: c). It is characterised with sporadic and intermitted occupational episodes, whose main activity was connected with the exploitation of large herbivore carcasses which had fallen into the natural trap (Huguet et al., 2001; Ollé et al, 2005; Cáceres et al, 2010).

The stratigraphic sequence was divided into six main levels (Pérez-González et al., 2001) (Fig. 6.05: f) and are as follows (from bottom to top):

-GI sterile layer;

-GII (sub-units GIIa and GIIb) dated between 450 and 350 ka;

-GIII (sub-units GIIIa and GIIIb) dated between *ca.* 466 and 220 ka;

-GIV to GVI; the top of the deposit is dated *ca.* 177 ka and the base 185 ka.

(dates are extracted from Berger et al., 2008 and Falguères et al., 2013).

Two human remains were recovered in the levels GIII and GII, both ascribed to the same species found at Sima de los Huesos site (Arsuaga et al., 1999).

The lithic assemblage is composed of uncomplete reduction sequences and shows typical traits which led to its adscription to the Acheulean techno-complex (Carbonell et al., 2001; Ollé et al., 2005). Handaxes and cleavers are found all along the stratigraphic sequence (Fig. 6.06). Evolutionary patterns have been identified in terms of changes in both morphology and percentages of bifaces (García-Medrano et al., 2014, 2015). Cleavers are found as typical cleavers (Tixier, 1956) and cleaver-like pieces, shaped on cobbles. Retouched tools appear to be highly standardised and the main identified types comprise denticulates, scrapers and points. The dominant flaking methods are multipolar centripetal, unipolar longitudinal and multipolar orthogonal.

Among the raw material types employed in the knapping activity, chert always dominates, followed by quartzite and sandstone. Nevertheless, concerning large tools, a clear over presence of quartzite is found in the basal layer (SubUnit GIIa), used to obtain both large flakes and large tools. The use of quartzite decreases through time, and the introduction of other raw materials (limestone and chert) is attested (from SubUnit GIIb upward).

The large cutting tools were imported into the site, as lithic material waste was not recovered in any of the different units. However, handaxes are more numerous in the middle Unit GIIb and their presence highly decreases in the upper level (Unit GIII), where the modification of flakes into tools (denticulates, scrapers and points) increases. The raw materials provenance, as at all the Atapuerca's sites, was restricted to the surroundings of the Sierra (García-Antón et al., 2002; García-Antón and Mosquera 2007).

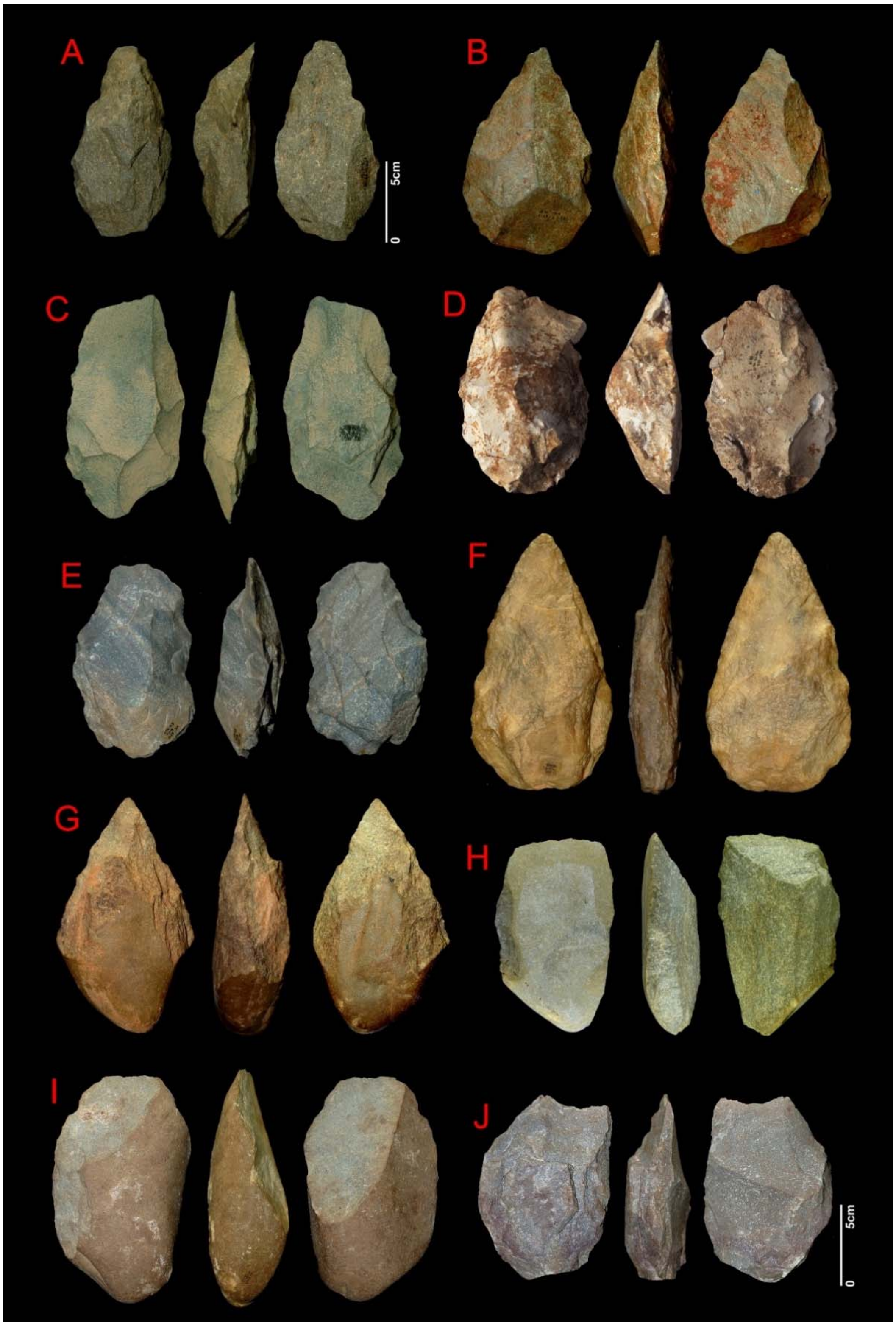

Fig. 6.7: *Selection of handaxes and cleavers from Galería: A) Handaxe, quartzite (ATA90-TG10A-G21-90); B) Handaxe, quartzite (ATA-90-TN07-E29-1); C) Cleaver, sandstone (ATA88-TG10A-G17-83); D) Cleaver, Neogene chert (ATA85-TG11-GSU11-G21-48-); E) Handaxe, quartzite (ATA95-TN05-G25-30); F) Handaxe, quartzite (ATA92-TG10B-H20-25); G) Cleaver-like, quartzite, (Ata94-TG07-F20-4); H) Cleaver, quartzite (Ata93-TN05-F25-32); I) Cleaver, quartzite (ATA94-TN2B-F27-2); J) Cleaver, quartzite (ATA94-TN2B-F22-3) (from Ollé et al., 2016a: 319).*

Sima del Elefante

The Sima del Elefante site is the southernmost cave of the railway trench. The stratigraphic sequence is 25m thick and 15m wide and it is divided into 16 different lithostratrigraphic units (from bottom to top, TE7 to TE21) (Fig. 6.6: g, d) and provide archaeo-faunal record from both Early and Middle Pleistocene levels (Rosas et al., 2001, 2006). The lower levels (TE7-TE16) show a reversal of the Earth's magnetic pole and they are attributed to the Matuyama Chron (Parés et al., 2006). Cosmogenic nuclide dating shows that the level TE9c has a burial age of 1.22 ± 0.16 Ma (Carbonell et al., 2008).

Eight levels contain archaeological material (TE8, TE9, TE11, TE12, TE13, TE14, TE18 and TE19), but the richest level is undoubtedly TE9. The faunal record is composed by small animals, such as birds, lagomorphs and castorids (Cuenca Bescós et al., 2010) as well as carnivores and mammals, such as *Cervidae* and bison, some of them presenting anthopogenic modifications (Huguet et al., 2013).

The lithic assemblage comprises 127 artefacts, 86 coming from the Early Pleistocene levels and 41 from the Middle Pleistocene ones (de Lombera et al., 2015). The richest level is TE9, which yielded 71 implements. The lower record is composed mainly by Neogene flint, whose preservation is sometimes compromised. Only three quartz flakes were recovered. Technological data describes the occurrence of short and uncomplete reduction sequences, characterised by simple technical adjustments. The application of the direct percussion with hard hammers has been attested as well as a preference for unipolar longitudinal knapping (Ollé et al., 2013; de Lombera et al., 2015). Cores are not fully exploited and the products are quite small (ranging from 20 to 70mm in length). Retouched pieces are absent in the lowest levels and only appear in the TE13 (two notches and one marginal sidescraper) and the TE14 (one sidescraper) levels (Fig. 6.8: 8, 9, 10).

The main difference of the lithic assemblage coming from the Middle Pleistocene sequence (TE18-19, TEsup) is the raw material use. Most of the implements are composed of quartzite, followed by sandstone and chert. Out of the 41 pieces ascribed to the upper sequence, 36 comes from the TE19 level. Knapping methods are not well represented. Only four cores are available, three of them are on flakes. They all show preliminary reduction stages, with a preference for unifacial and unipolar strategies. More rarely, centripetal products are identified. Blanks are mostly cortical, reiterating the expeditious character of lithic production. Retouched flakes are mostly found in unit TE19 and are sidescrapers and denticulates. They show a higher degree of standardisation, compared to the modified implements found in the Lower Pleistocene levels. A quartzite point, a sandstone handaxe and a quartzite cleaver have also been identified.

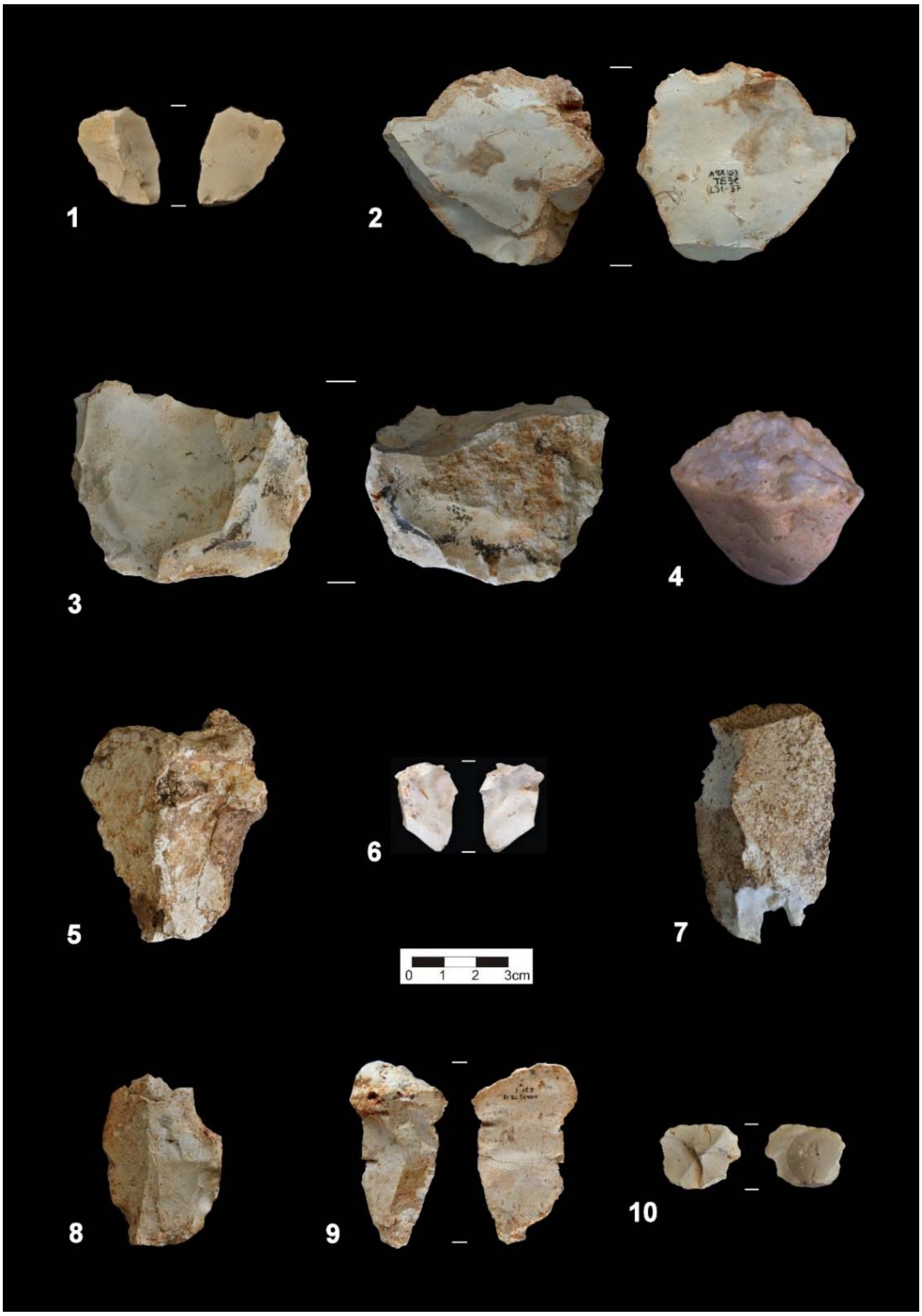

Fig. 6.8: *Selection of lithics from Sima del Elefante: 1) Flake (Unit TE8, Cretaceous chert), 2: Flake (Unit TE9, level TE9c, Cretaceous chert), 3) Core (Unit TE9, level TE9c, Cretaceous chert), 4) Small fractured pebble (Unit TE9, level TE9c, Quartz), 5) Flake (Unit TE9, level TE9c, Neogene chert), 6) Flake (Unit TE9, level TE9c, Cretaceous chert), 7) Flake (Unit TE9, level TE9c, Neogene chert), 8) Retouched flake (Unit TE13, Neogene chert), 9) Retouched flake (Unit TE13, Cretaceous chert), 10) Retouched flake (Unit TE13, Cretaceous chert) (from de Lombera et al., 2015: 101).*

6.2 The Gran Dolina site

The Gran Dolina site was a cave which was progressively filled with exokarstic and endokarstic sediments during the Lower and Middle Pleistocene. The cave, being home to both carnivores and hominins (when there was at least one aperture to the outside), yielded numerous archaeological and paleontological evidences. A long sequence (18m) composed of 11 lithostratigraphic units was identified and later, through the excavation of a test pit (9 square meters), these units were described (Pérez-González et al., 2001) (Fig. 6.9). At the same occasion, paleontological remains were identified in all the levels, except from TD1-2 (Carbonell et al., 1999a).

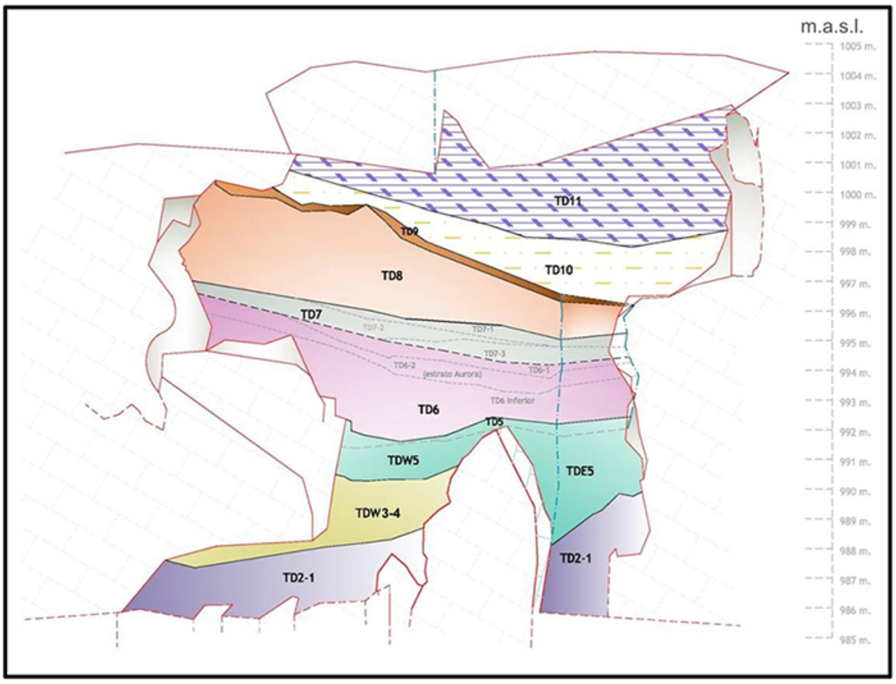

Fig. 6.9: *Stratigraphic sequence of the Gran Dolina site (drawing by R. Pérez).*

Specifically, human presence has been ascribed from both lithic tools and paleo-anthropological remains. The formers are found all along the sequence, with the exceptions of TD8, TD8/9 (exhibiting a hiatus in terms human occupational events), and TD11. TD7 level also does not correspond to a phase of intense occupation, since the only lithic tool recovered was a small-sized quartz flake.

165 human remains were found in TD6 level (popularly known as the *Aurora stratum*), and were assigned to a new species, *Homo antecessor* (Bermúdez de Castro et al., 1999; 2008, 2017; Carbonell et al., 2010).

TD10 level is the richest level of the Atapuerca sites in terms of lithic and paleontological remains (Ollé et al., 2013). The cave entrance eventually collapsed during the sedimentary deposition of TD10 (Middle Pleistocene). Although no human fossils have been found in TD10 level, the hominin species connected to the occupational phases identified was first ascribed to *H. heidelbergensis*. A recent revision of the Atapuerca human fossils contemporaneous to TD10 (Sima de los Huesos) has inserted these hominins in the direct lineage to Neanderthals (Arsuaga et al., 2014).

The sequence of the site is closed by the sterile TD11 level, which reaches the only survived parts of the roof, located at the eastern part of the site.

6.2.1 History of research

After the construction of the railway trench between the end of the XIX and the beginning of the XX century and after the subsequent exposition of the cave, the first scientific studies began to take place at Gran Dolina (Fig. 6.10: a). The first systematic excavations were carried out between 1981 and 1989 on a surface of 30 square meters (TD10 level). Afterwards, a $9m^2$ test pit was initiated in 1993, uncovering paleontological material throughout the entire sequence (with the only exception of TD1-2) (Carbonell et al., 1999a) (Fig. 6.10: b). From 1996 onward, horizontal excavations took place on different levels of the site (TD10-TD4). Presently, two excavations are carried on at Gran Dolina: TD10.3 is being excavated on a total area of *ca*. 90 m^2 (Fig. 6.10: c, d) and TD4 on a total area of *ca.* $15m^2$.

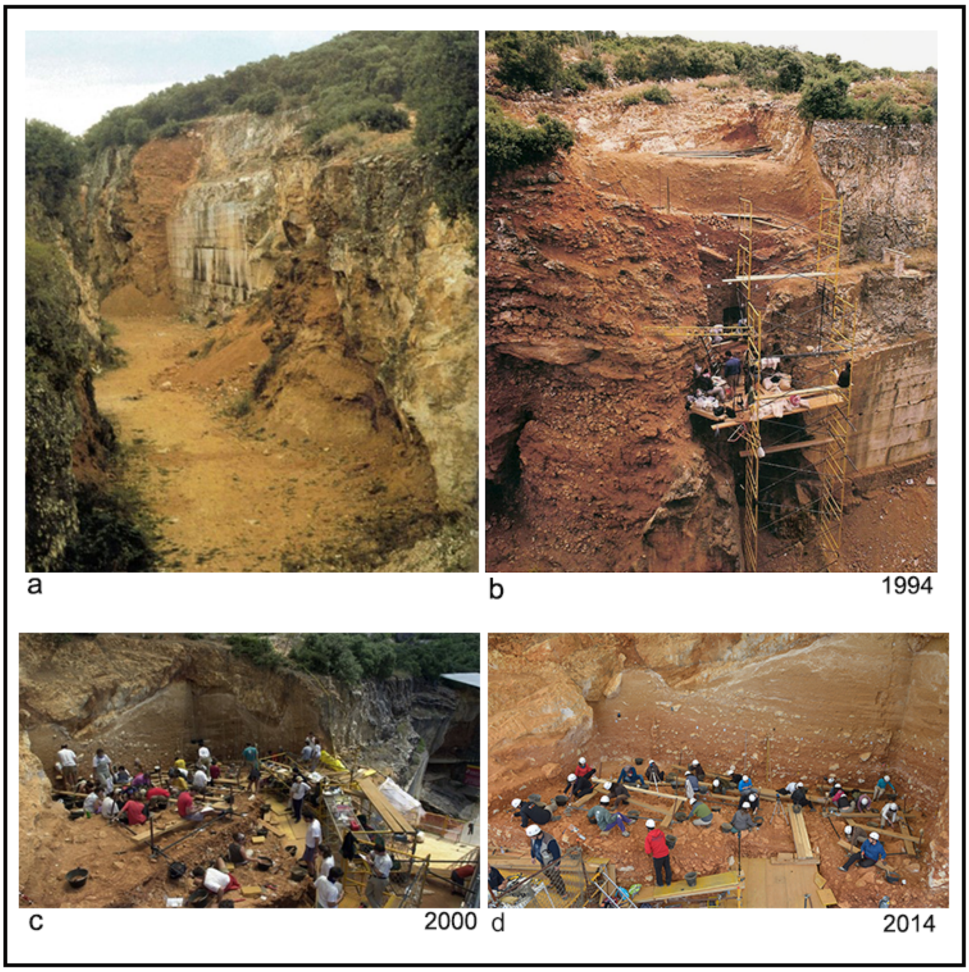

Fig. 6.10: *Gran Dolina site seen at different historical moments: a) Before the beginning of systematic excavations; b) During the excavation of the test pit; c, d) Recent extension excavation of the TD10.1 level: TD10.1 in photo c, TD10.3 in photo d. (IPHES-Atapuerca Research Team).*

6.2.2 Stratigraphy

The excavation of the test pit allowed the identification of 11 different lithostratigraphic units, denominated from the base to the top TD1-TD11 (Gil et al., 1987; Parés and Pérez-González, 1999; Pérez-Gonzáles et al., 2001; Rodríguez et al., 2011). Geochronological studies put the infilled sediments of Gran Dolina into a chronological frame corresponding to *ca.* MIS25-3 (Falguères et al., 1999, 2013; Berger et al., 2008; Moreno et al., 2015). Although three levels are archaeologically sterile (TD1, TD2 and TD11), the remaining ones contain a high number of archaeo-paleontological artefacts.

The lithostratigraphic units bearing paleontological evidence are described as follows (Fig. 6.11, 6.12) (Pérez-Gonzáles et al., 2001; Rodríguez et al., 2011; Vallverdu i Poch, 2016):

- TD3-4: levels composed by exogenous sediments, mainly gravels with rare matrix. Silty sediments are also present. This layer has been interpreted as a den used for hibernation, due to the high number of bear remains (*Ursus dolinensis*). The cave was also a trap for macro-mammals, which were later consumed by carnivores (Rosell, 1998). Human presence is attested by few cut marks on animal bones and by a small lithic collection, exhibiting simple reduction strategies (Carbonell et al., 2001);

- TD5: level composed by exogenous sediments, mainly gravels with rare matrix. Silty sediments are also present. Different uses of the cave were described, from a den used alternatively by hyenas and ursids, to a place where human occupation took place;

- TD6: 20cm unit, mainly formed by reddish-yellowish silt with gravels. The faunal assemblage has an anthropogenic origin (Saladié et al., 2011). Human remains allowed to describe a new species, *H. antecessor* (Bermudez de Castro et al., 1997). Some of these remains bore cut mark evidence, pointing out for the most ancient cannibalism event known to date (Saladié et al., 2012). Paleomagnetic data identified the age of TD6 as 800ka (Parés and Pérez-González, 1999), while TL dates pointed to an older age of 960±120ka (Berger et al., 2008). Numerous lithic tools were also recovered, highlighting a variability in the reduction strategies new to Atapuerca. Unipoilar, bipolar and centripetal strategies were documented (Carbonell et al., 1999b, 1999c). Blanks were also systematically retouched into denticulates, notches and sidescraper;

- TD7: mainly formed by calcarenites. the Matuyama-Brunhes palaeomagnetic boundary is located At the top of this layer. Remains of *Stephanorhinus etruscus* and *Praeovibos* were found in anatomical connection, that means that the natural trap in the roof was reactivated;

- TD8: 3m unit of yellowish-reddish sandy mud in bed of breccia. The breccia of sandy mud is finely stratified. There is a diverse range of ungulates and carnivores, dominated remains of fallow dear. Carnivore consumption of the ungulate remains is attested, and no evidence of human activity is documented;

- TD9: thin unit of yellowish-reddish sandy mud with altered fine gravels. It is the first Middle Pleistocene unit bearing evidence of human occupation. In fact, four lithic implements were recovered and the layer is dated by TL at 480±130ka (Berger et al., 2008);
- TD10: It is a 2-3 m thick deposit, beds of bed of breccia, supported by clast or by sandy mud. Divided into four lithostratigraphic units, it is the richest level of the site and of all the Atapuerca sites, yielded an approximate number of 120,000 faunal remains and 35,000 lithic items;
- TD11: 4m deposit of breccia and terra rossa soil on the top. The stratigraphic succession of Gran Dolina finishes with this archaeologically sterile unit, dated between 240±44 and 55±14ka (Berger et al., 2008).

The results of several paleoclimatic proxies indicate no major changes along the Gran Dolina sequence, pointing to an absence of very rigid-cold conditions (Blain et al., 2009; Cuenca-Bescós et al., 2010, 2011; Rodríguez et al., 2011).

Regarding TD10 level, there are some contradictions between the micro-mammals and the amphibian and squamate proxies (Cuenca-Bescós et al., 2005; Blain et al., 2008). While the micro-mammals results pointed out to open-dry environments, with the absence of woods, the herpetofauna distribution indicate humid conditions all along the TD10 level and the presence of more wooded environments than in the rest of the Gran Dolina sequence. The only bottom of TD10 level would present warm-temperate conditions, while the upper sub-levels would be the result of mild-summers and very arid and cold winters (Blain et al., 2008, 2009). The differences between the micro mammals and herpetofaunal results can also have taphonomic reasons.

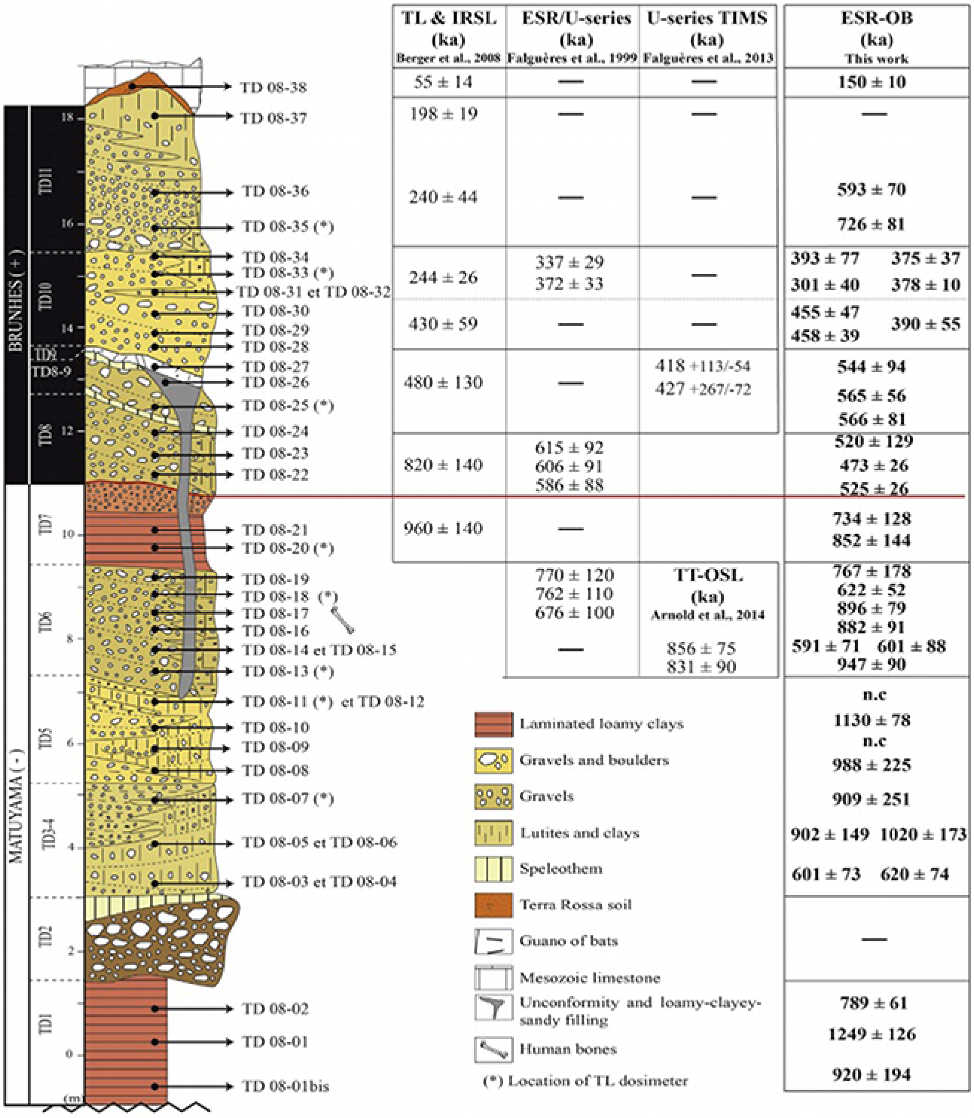

Fig. 6.11: *Stratigraphy of the Gran Dolina site and a sum-up of the dates obtained for each level and by different geochronological methods (Moreno, et al. 2015).*

6.3 TD10 level

TD10 is the most recent level of the site (MIS 11-9) (Berger et al., 2008; Falguères et al., 2013), bearing evidences of human occupation. It is a three meters thick deposit, mainly composed of limestone blocks coming from the degradation of the cave, and a finer reddish-brown clayish matrix. Four litho-stratigraphic sub-units were identified, named from the top to the base, TD10.1 to TD10.4 (Fig. 6.12) (Rodríguez et al., 2011).

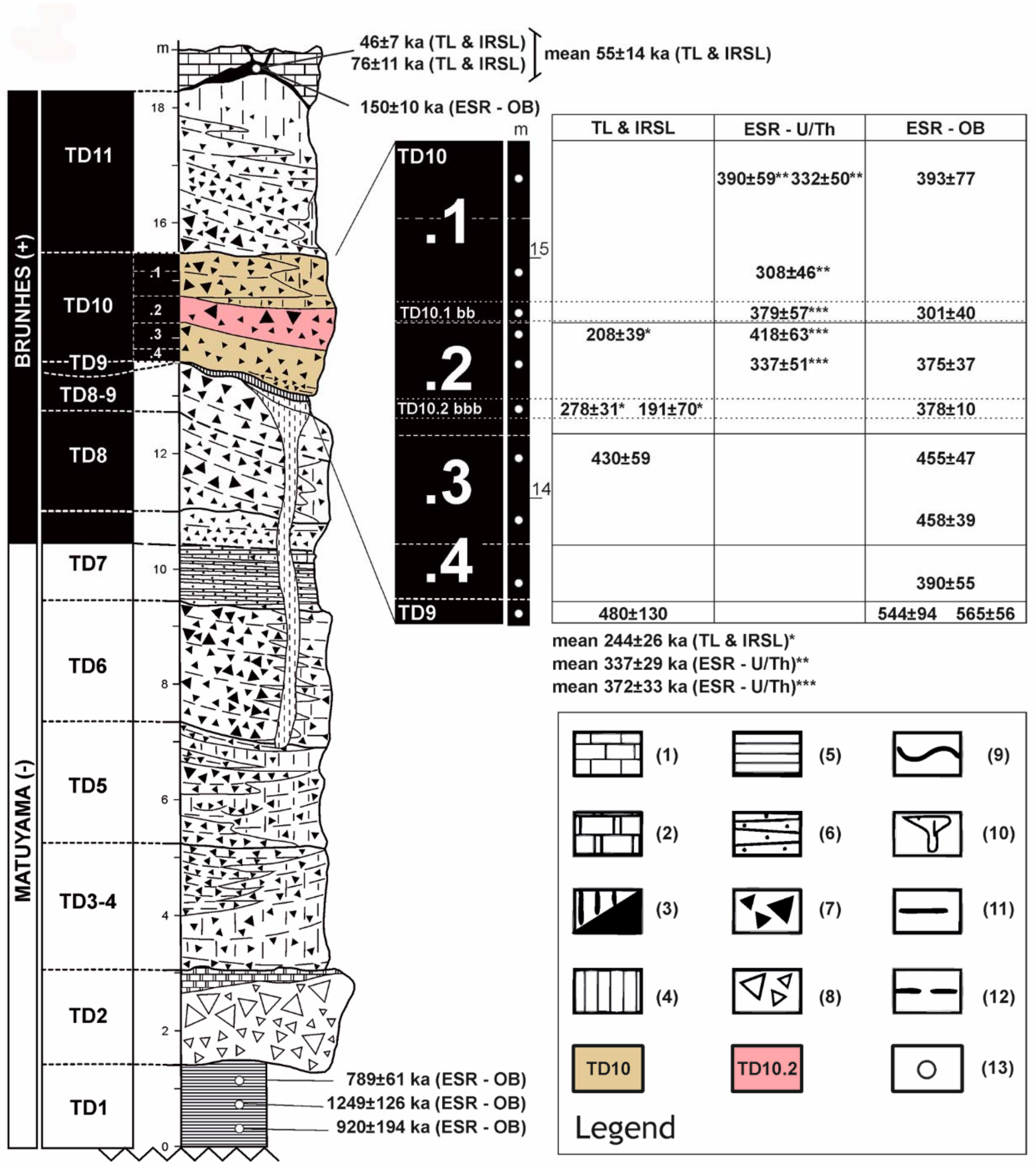

Fig. 6.12: *Location of the TD10 level in the sequence of the Gran Dolina site and the main dates obtained with different methods (modified from Rodríguez-Hidalgo et al., 2017).*

Extension excavations cover an approximate surface of 95-100 m^2 (Ollé et al., 2013, 2016). Units TD10.1 and TD10.2 have been completely excavated and the ongoing excavations are focusing on the TD10.3 unit (Fig. 6.13: a).

The micro-morphological studies of the whole package are still ongoing; the presently available studies refer to the only upper level (Mallol and Carbonell, 2008). The cave entrance was identified at the west sector of the site and a gradient of 15-20° to the northeast is described.
The archaeo-levels study divided the TD10 level into 8 main sub-units (from the top to the base, A-H), based on the vertical disposition of the recorded artefacts (Obregon, 2012).

TD10.1

TD10.1 is the richest level of the Gran Dolina site (and all the Atapuerca sites), having yielded approximately 21,000 lithic artefacts and 80,000 faunal remains, all three-dimensionally recorded (Ollé et al., 2013). It was excavated during the 1996-2005 campaigns on a surface of *ca.* 80 m^2.
Two sedimentary blocks compose the sub-unit: TD10.1sup (from now onward called upper TD10.1), which is denominated TD11 in older publications and the basal unit. Archaeo-paleontological material is found all over the surface, exhibiting a high concentration near the eastern section of the surface. As this concentration is located on the opposite side of the entrance of the cave, it could be the result of the sedimentary infilling by following the natural slope of the original pavement of the cave,
The bottom of the level was denominated ‘bone bed’, due to the remarkably high concentration of lithic and faunal remains (Fig. 6.13: b; Fig. 6.14) It coincides with the archaeo-level H (Obregon, 2012). A total of *ca.* 48,000 faunal remains is associated with this sub-level (Rodríguez-Hidalgo, 2015).

Fig. 6.13: *Appearance of the TD10 level at the end of the 2015 field season: a) borders among the three sub-units are drawn in different colours, dividing from the top to the bottom Upper TD10.1, TD10.1 and Td10.2; b) detailed picture of the east stratigraphic sequence (red square in photo: a). The two bone beds are visible and signalised by the lateral placard (IPHES-Atapuerca Research Team-A.Ollé; modified by A. Pedergnana).*

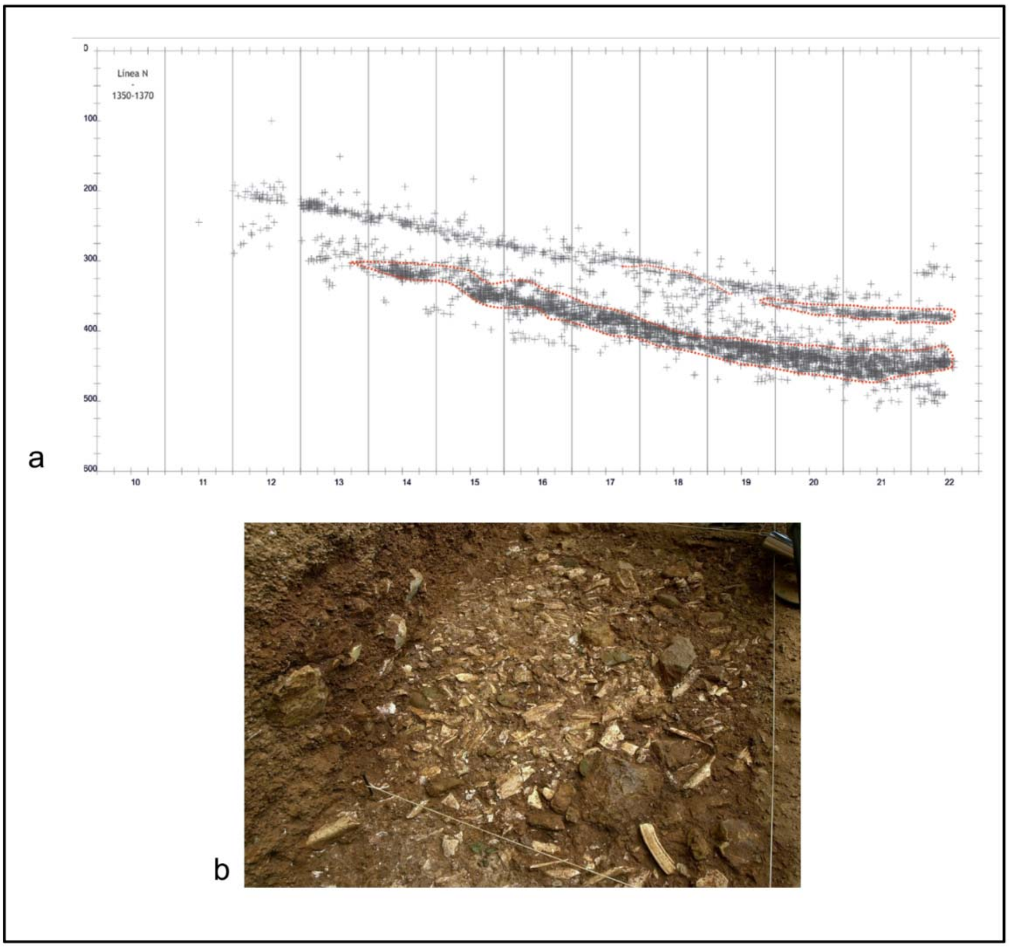

Fig. 6.14: *a) Vertical projection of all the coordinated bones until 2014. The cross concentrations correspond to the two bone beds, the upper one being the TD10.1 bone bed, the lower one being the bison bone bed in TD10.2 (Rodríguez-Hidalgo, 2015); b) Photograph of the TD10.1 bone bed during the 2002 field season (IPHES-Atapuerca Research Team-A.Ollé).*

Sedimentary rates seem to be very slow and the presence of organic matter and high moisture explained the diagenic processes of this unit (Mallol and Carbonell, 2008). The relative sedimentary stasis is confirmed by several studies, which highlighted *in situ* knapping activities (López-Ortega et al., 2011; 2017) and butchering events (Rosell, 2001; Blasco, 2010; Blasco and Rosell, 2010; Rodriguez-Hidalgo et al., 2015). Extraordinary events, as the butchering of a lion carcass (*Panthera leo*) was identified in this sub-unit (Blasco et al., 2010). Bone-tools production has also been attested (Rosell et al., 2011; Rodríguez-Hidalgo et al., 2015).

All of those evidences suggest the presence of multiple occupational events, when the cave was used as a camp-site (Ollé et al., 2013). From the archaeo-stratigraphic study, only the bottom of this sub-unit (archaeo-level H) should be considered as an evidence of long-term hominin use of the site (Obregon, 2012). This data has been confirmed by the analysis of the faunal record, which contributed to the understanding of this sub-unit as a residential base-camp (Rodríguez-Hidalgo et al., 2015). The bone bed was dated through electron spin resonance (ESR/U-series), giving a date of 379±57ka for the bottom and a date of 337±29ka for the top (Falguères et al., 1999) (Fig. 6.11). A recent ESR date obtained from quartz grains gave a date of 301±40 ka (Moreno et al., 2016).

TD10.2

The second sub-unit of TD10 level was excavated during the years 2006-2013.

The archaeological record corresponds to *ca*. 51,000 faunal remains and *ca*. 9,800 lithic remains (Ollé et al., 2013). Chert dominates, while the faunal record is almost exclusively composed of bovid (Rodríguez-Hidalgo et al., 2016, 2017). This highly standardised patterns of prey selection led to the interpretation of this unit as a highly-specialised butchering site (Rodríguez-Hidalgo et al., 2016; Rodríguez-Hidalgo et al., 2017).

This sub-unit was dated by means of different methods and the available dates are two electron spin resonance/uranium-series (ESR/U-series) dates of 418±63ka and 337±51ka (Falguères et al.,1999). Two recent ESR dates on quartz grains have been also obtained (375±37ka and 378±1ka) (Moreno et al., 2015). A discordant date was also obtained through optical stimulated luminescence (OLM) (244±26ka) (Fig. 6.12) (Berger et al., 2008; Rodríguez-Hidalgo et al., 2017).

TD10.3

The TD10.3 level is currently being excavated on a surface of *ca*. 100 m^2. From field observations and from the analysis of the materials collected from the test pit, it is possible to say that it is significantly poorer in terms of lithic and archaeo-faunal remains compared to the upper sub-levels. Several large cutting tools made of quartzite and flint were collected and the incidence of carnivores seems to be higher than in the upper sub-levels. A TL date of 430±59 ka is available for this sub-level (Berger et al., 2008).

6.3.1 The TD10 lithic record

Since the excavation of the TD10 level is still ongoing, the available information comes from the entirely excavated Upper TD10.1, TD10.1 and TD10.2 (Fig. 6.12). Published numerical data refers to the material excavated before 2010, therefore they do not comprise the entire record excavated in TD10.2 (Ollé et al., 2013). Regarding the lower sub-levels (TD10.3 and TD10.4) data was extrapolated from the study of the material recovered during the excavation of the test pit and some projecting areas of the south section.

The richest sub-level is TD10.1, which yielded more than 21,000 lithic artefacts (Upper TD10.1= 834, basal TD10.10= 21 050) (Table 6.1). A total number of 9,799 artefacts were collected from TD10.2, the test pit and projected areas revealed the presence of 206 artefacts for TD10.3 and only 20 artefacts for TD10.4. Consequently, a detailed characterisation of the lithic assemblage is only available for the three upper layers (Upper TD10, TD10.1, TD10.2). However, a simple updating of the counts up to the 2016 season (unpublished and subjected to possible stratigraphic readjustment), gives a number of 12,918 lithic artefacts for TD10.2, 653 for TD10.3 and the above-mentioned 20 for sub-unit TD10.4.

Clear differences derived from a differential use of the cave are visible based on technological data. The assemblage coming from the top of the layer (Upper TD10.1) yielded uncomplete reduction sequences, while complete reduction sequences are found in the rest of the TD10.1 unit. Regarding TD10.2, complete production sequences have been documented for chert, with relatively scarce cores (Ollé et. al., 2013).

The exploitation of lithic resources shows similar trends, though some differences are present. Neogene and Cretaceous cherts, found on the top of the Sierra, dominate the three assemblages (more than 50%). A far greater percentage of this is found in TD10.2, where it represents *ca*. 95% of the total, defining a high specialisation, also confirmed by the analysis of the fauna assemblage (Rodriguez-Hidalgo et al., 2016, 2017). Quartzite and sandstone were used with an average similar intensity, followed by poorly represented materials such as quartz and limestone.

Limestone was available directly at the site or in the near vicinity, while quartzite, sandstone and quartz were found on the fluvial terraces 5 to 20km from the site (García-Antón, et al., 2002; García-Antón and Mosquera, 2007). Specific selection strategies have been also identified in the preference of good quality varieties coming from the alluvial planes of the Arlanzón River and the Vena River for the production of tools (García-Antón and Mosquera, 2007).

Knapping strategies change throughout the sequence, being more expedite in Upper TD10.1, highly standardised in TD10.1, and less specialised in TD10.2. In fact, unipolar and multipolar strategies dominate in the upper part of the layer. In TD10.1, unipolar, multipolar, orthogonal methods are attested, but centripetal strategies dominate all over the others. A certain hierarchy of core faciality within the centripetal production is also noted. However, clearly predetermined products are not frequent. In TD10.2 no centripetal hierarchised cores have been recognised and simple flake production predominate. Centripetal reduction is found mainly through bifacial strategies.

In general, cores are not abundant in the TD10 lithic assemblage, being less than 1,6% of the total (Upper TD10.1= 1,56%, TD10.1=0,89%; TD10.2=0,60%) (Ollé et al., 2013).

Furthermore, shaping of large tools is attested in all three units with the presence of handaxes, cleavers and choppers. Large cutting tools (LCT) appear in extremely low proportions also in the upper two levels (Upper TD10.1=4, TD10.1=13) (Fig. 6.16). The tools (bifaces or cleavers) were usually obtained from cobbles and coarse-grained raw materials were generally preferred (Upper TD10.1=3, TD10.1=10) over chert (TD10.1=2) and limestone (Upper TD10.1=1, TD10.1=1) (García-Medrano et al., 2015; Ollé et al., 2016a). Until the 2010 season, only one chert biface was known from the TD10.2 sub-level (Ollé et al., 2013), while at present 8 bifaces (3 on sandstone, 3 on quartzite, 2 on flint) and 4 cleavers (3 on sandstone and 1 on quartzite) are counted.

Among the retouched pieces, sidescrapers, denticulates, points and notches are documented, but with different percentages. In Upper TD10 and TD10.1 the same proportions of sidescrapers and denticulates are attested, while in TD10.2 sidescrapers are predominant, even if points and denticulates are also present (Fig. 6.15) (Ollé et al., 2013).

In sum, a much higher number of artefacts coming from the lower part of sub-level TD10.1 and roughly complete operational sequences pointed out *in situ* knapping activities and defined the function of the site as a base-camp. Additionally, refitting studies identified *in situ* knapping events both in Upper TD.10.1 and TD10.1 (López-Ortega et al., 2011, 2017).

Despite analogous dates and the vicinity to the Galería site, the two lithic assemblages show unexpectedly different technological traits (García-Medrano et al., 2015). TD10.1 has a general less incidence of large shaped tools, a more standardised retouch, and similar percentages of denticulates and sidescrapers compared to Galería, where a relatively high presence of bifaces and cleavers and less retouched and smaller implements are found at Galería.

	Raw material	Flakes	Flake fragments	Retouched flakes	LCT	Cores	Shaped tools/cores	Fragments	Natural bases	Indet.	TOT.	TOT.
Upper TD10.1	Chert	180	49	31	-	6	4	44	-	233	547	**834**
	Quartzite	72	40	12	1	7		10	17	1	160	
	Sandstone	29	12	9	2	-		20	7	30	109	
	Quartz	2	2	1	-	-		1	1	-	7	
	Limestone	1			1			2	5		9	
Tot. Upper TD10.1		**285**	**103**	**53**	**4**	**13**	**4**	**78**	**30**	**264**	**834**	
TD10.1	Chert	5 072	2 274	355	2	118	29	596	4	4 428	12 878	**21 050**
	Quartzite	1531	1530	204	6	38	4	172	82	41	3 608	
	Sandstone	1442	1040	83	4	29	7	268	48	875	3 796	
	Quartz	294	234	23	-	3	1	130	8	8	701	
	Limestone	11	3	1	1	-	-	22	5	5	48	
Tot. TD10.1		**8 352**	**5 081**	**666**	**13**	**188**	**41**	**1 199**	**147**	**5 363**	**21 050**	
TD10.2	Chert	3 487	1 601	306	1	57	10	277	1	3 631	9 371	**9 799**
	Quartzite	79	46	11	-	1	-	19	16	4	176	
	Sandstone	56	25	2	-	1	-	28	6	46	164	
	Quartz	23	9	3	-	-	-	16	-	-	51	
	Limestone	4	2	-	-	-	1	2	9	-	18	
	Others	7	1	-	-	-	-	6	-	2	16	
Tot. TD10.2		**3 656**	**1 684**	**322**	**1**	**59**	**11**	**348**	**32**	**3 683**	**9 799**	**31 683**

Table 6.1: *The TD10 lithic assemblage divided into technological categories and raw materials and relative presence in the three sub-levels (modified from Ollé et al., 2013).*

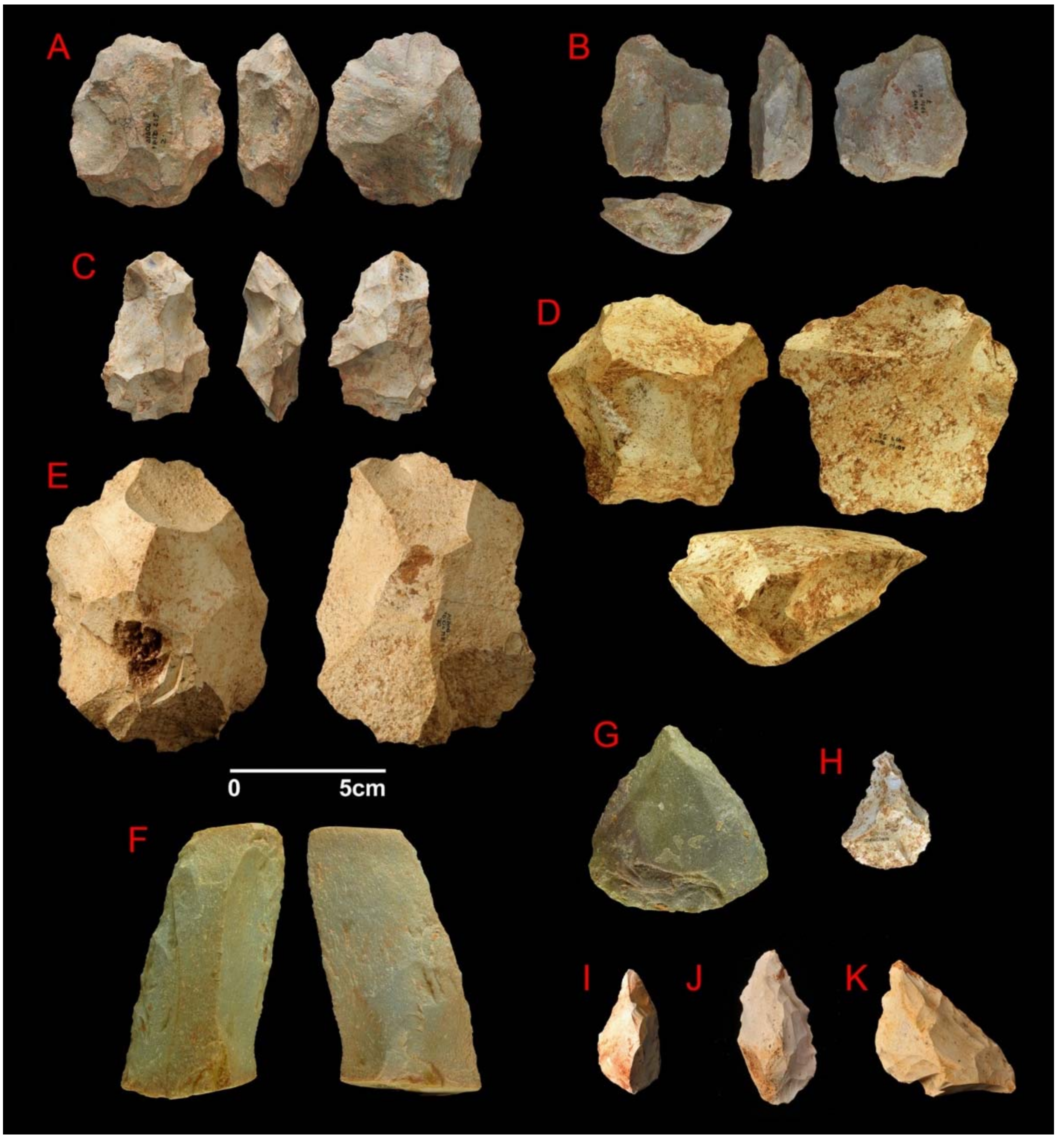

Fig. 6.15: *Selection of cores and tools from the sub-levels TD10.1 (A, B, C, F, G, H) and TD10.2 (D, E, I, J, K). A-B) quartzite centripetal cores; C, D, E) Chert centripetal cores; F, G) Quartzite sidescrapers; H, I, J, K) Chert sidescrapers (Ollé et al., 2016a).*

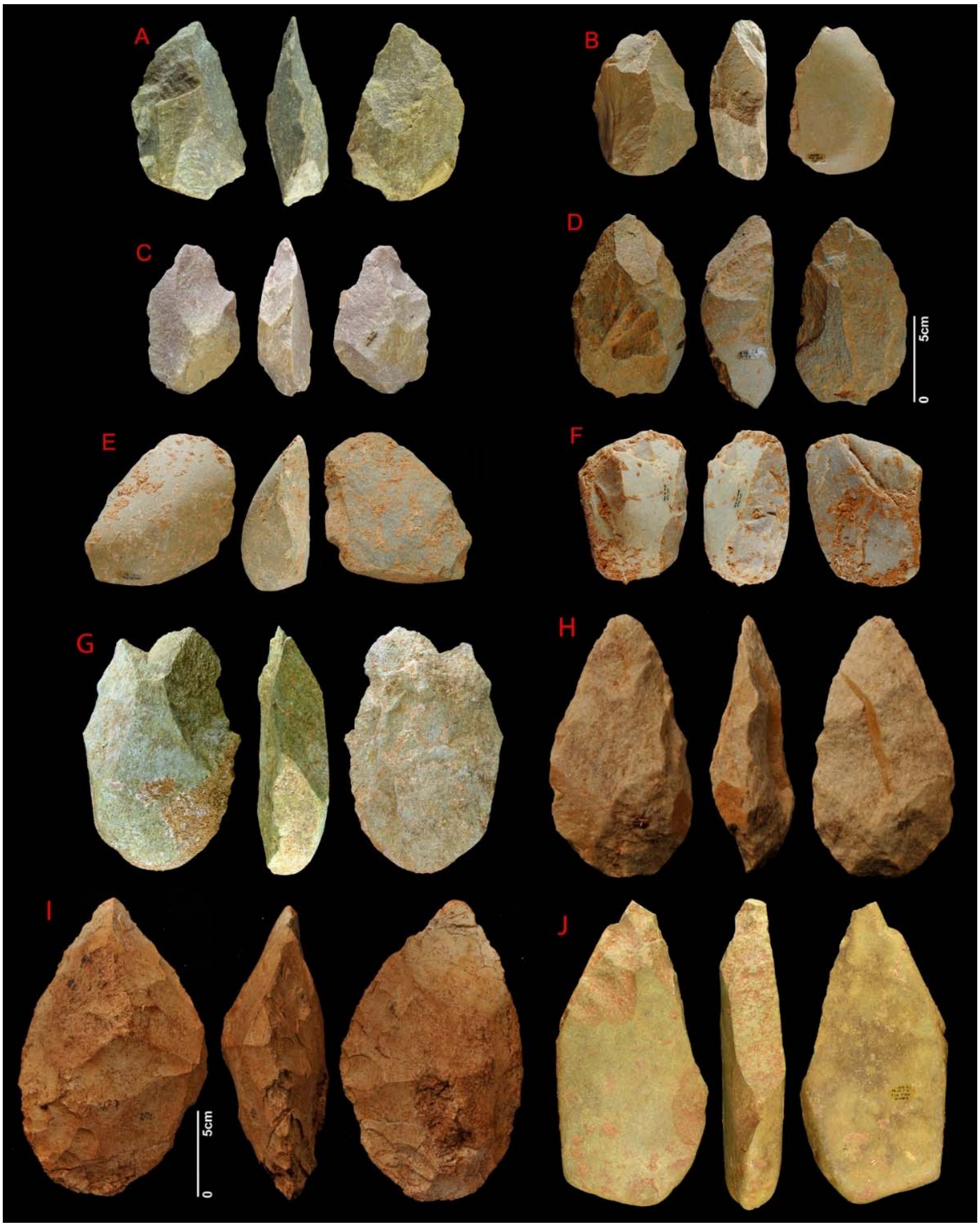

Fig. 6.16: *Selection of large cutting tools from the TD10.1 (A-F), TD10.2 (G-I) and TD10.3 (J) units. A, B, C, D, G) Quartzite bifacial tools; E) Large cortical quartzite flake; F) Quartzite tool with cleaver-like features; H) Quartzite handaxe; I) Chert handaxe; J) Quartzite pick (Ollé et al., 2016a).*

6.3.2 The TD10 archaeofaunal record

Different zooarchaeological analyses were performed on the faunal material coming from the TD10 unit. Specifically, the unit TD10.1 (upper TD10.1 and the rest of TD10.1) was studied by Rosell (2001), Blasco (2011) and Rodríguez-Hidalgo (2015). Regarding the lower unit (TD10.2), the entire faunal record was analysed by Rodríguez-Hidalgo (2015).

Data about the lowest sub-levels are not available yet, as the analysis of the TD10.3 material is still ongoing and the TD10.4 level has not been excavated yet.

Rosell (2001) studied the material recovered in the 1998-99 campaigns, while Blasco (2011) analysed the material coming from the following archaeological campaigns (2000-2001). Despite the vertical continuity of the two studies and the underlined similarities in the treatment of carcasses, divergent interpretations of the assemblages were proposed. In fact, while Rosell saw in the standardisation of the butchering activities, a reflection of long-term occupations by groups with similar strategies of animal resources exploitation, Blasco interpreted the data as products of short-terms and expedite events. Both interpretations might be correct, considering that both studies deal with material pertaining to different archaeo-levels (Obregon, 2012) and therefore, they might be the result of a number of occupations characterised by very different economical strategies, performed by different hominin groups. This would explain the differences with the more ancient occupations of the cave (TD10.1 bone bed). In fact, the most evident difference regards the prey selection strategies. Although the most exploited animals were adult deer, equids and bovid, the mesovertebrates (leporids and avian species) were consumed in the middle sub-levels of TD10.1 (Blasco, 2011), while at the bottom of the level they do not show any anthropogenic modification (Rodríguez-Hidalgo, 2015).

The anatomical skeletal representation of the upper and medium sub-levels is similar, suggesting differential strategies of carcasses transportation. For large-sized animals (weighting more than 100kg), only the appendicular elements with high nutritional value were transported into the site and then processed, while entire carcasses of small-sized animals were butchered directly at the site (Rosell, 2001; Rosell and Blasco, 2009; Blasco, 2011).

Furthermore, although most of the exploited carcasses showed a primary and immediate consumption by humans, sporadic secondary access to large-sized animals was identified (Blasco, 2011). Anyhow, low competence rates with carnivores are identified by very low carnivore-induced modifications on bones as well as by at least two distinct butchering events involving two carnivore individuals (a lion and a fox) (Blasco et al., 2010).

The very low presence of carnivores and their incidence in the formation of the TD10.1 faunal record is confirmed by more recent studies, focusing on the bottom of the level (Rodríguez-Hidalgo study, 2015). However, while the previous studies saw in small-sized carnivores the main taphonomic agent other than humans (Rosell, 2001; Rosell and Blasco, 2009; Blasco, 2011), the analysis of the TD10.1 bone bed identified the presence of large carnivores, based on the gnawing marks as well as the extreme damage found on epiphysis of large-sized ungulates (Rodríguez-Hidalgo, 2015; Rodríguez-Hidalgo et al., 2015).

The main difference underlined by the study of the material from the bottom of the TD10.1 unit regards pray selection and butchering strategies. The faunal record is mainly composed by ungulates (deer, bison and horses) (Table 6.2). Other taxons correspond to less than 1% of the total (comprising rhino, caprids, roe deer, birds and rabbits). Carnivores are also present with a lion, a wolf and a wild cat. An estimated MNI (minimum number of individuals) of 34 emerged, with a prevalence of deer, fallow deer, horses, rabbits, bison and birds. The mortality patterns showed a specific preference towards prime adult individuals (Table 6.2). The anatomical skeletal representation is virtually the same considering medium and large-sized animals, reflecting an analogous carcass treatment with no regard to the animal size (Rodríguez-Hidalgo, 2015; Rodríguez-Hidalgo et al., 2015).

There is a high incidence of anthropogenic modifications (16,3%), most of them occurring as cut-marks (12,9%). Cut-marks are found prevalently on deer remains, followed by bison and horse (table 6.3).

<table>
<tr><th>Taxa</th><th>NISP</th><th>%</th><th>MNI</th><th>Young</th><th>Prime</th><th>Old</th></tr>
<tr><td>Cervus/ Dama sp. indet.</td><td>1,484</td><td>79.6</td><td>8</td><td rowspan="3">4</td><td rowspan="3">12</td><td rowspan="3">1</td></tr>
<tr><td>Cervus elaphus priscus</td><td>22</td><td>1.2</td><td>7</td></tr>
<tr><td>Dama dama clactoniana</td><td>9</td><td>0.5</td><td>2</td></tr>
<tr><td>Bison schoetensacki</td><td>136</td><td>7.3</td><td>2</td><td>0</td><td>2</td><td>0</td></tr>
<tr><td>Equus sp. indet.</td><td>132</td><td>7.1</td><td>1</td><td rowspan="3">1</td><td rowspan="3">3</td><td rowspan="3">0</td></tr>
<tr><td>Equus ferus</td><td>2</td><td>0.1</td><td>2</td></tr>
<tr><td>Equus cf. hydruntinus</td><td>1</td><td>0.1</td><td>1</td></tr>
<tr><td>Stephanorhinus cf. hemitoechus</td><td>46</td><td>2.5</td><td>1</td><td>1</td><td>0</td><td>0</td></tr>
<tr><td>Hemitragus bonali</td><td>3</td><td>0.2</td><td>1</td><td>0</td><td>1</td><td>0</td></tr>
<tr><td>Capreolus priscus</td><td>5</td><td>0.3</td><td>1</td><td>0</td><td>1</td><td>0</td></tr>
<tr><td>Panthera leo</td><td>1</td><td>0.1</td><td>1</td><td>0</td><td>1</td><td>0</td></tr>
<tr><td>Canis/Cuon sp. indet</td><td>2</td><td>0.1</td><td>1</td><td>1</td><td>0</td><td>0</td></tr>
<tr><td>Felis silvestris</td><td>1</td><td>0.1</td><td>1</td><td>0</td><td>1</td><td>0</td></tr>
<tr><td>Oryctolagus sp. indet.</td><td>16</td><td>0.9</td><td>3</td><td>0</td><td>3</td><td>0</td></tr>
<tr><td>Ave</td><td>4</td><td>0.2</td><td>2</td><td>0</td><td>2</td><td>0</td></tr>
<tr><td>Total NISP-MNE-MNI</td><td>1,864</td><td></td><td>34</td><td>7</td><td>26</td><td>1</td></tr>
<tr><td>Very Large size</td><td>12</td><td></td><td></td><td></td><td></td><td></td></tr>
<tr><td>Large size</td><td>1,119</td><td></td><td></td><td></td><td></td><td></td></tr>
<tr><td>Medium size</td><td>1,759</td><td></td><td></td><td></td><td></td><td></td></tr>
<tr><td>Small size</td><td>148</td><td></td><td></td><td></td><td></td><td></td></tr>
<tr><td>Very small size</td><td>0</td><td></td><td></td><td></td><td></td><td></td></tr>
<tr><td>Indeterminate</td><td>2,092</td><td></td><td></td><td></td><td></td><td></td></tr>
<tr><td>Total</td><td>6,994</td><td></td><td></td><td></td><td></td><td></td></tr>
</table>

Table 6.2: *Taxonomic determination, NISP, (%), MNE, (%), MNI and age size weight categories of the fauna from the TD10.1 bone bed level of Gran Dolina (Rodríguez-Hidalgo et al., 2015).*

	Cut marks	Anthropogenic Breakage
Cervus/Dama sp. indet.	397 44%	167 51.9%
Bison schoetensacki	40 4.4%	11 3.4%
Equus sp. indet.	27 3%	8 2.5%
Stephanorhinus cf. *hemitoechus*	3 0.3%	2 0.6%
Hemitragus bonali	0	0
Capreolus priscus	1 0.1%	0
Canis/Cuon	1 0.1%	0
Very large size	2 0.2%	1 0.3%
Large size	167 18.5%	58 18%
Medium size	208 23.1%	68 21.1%
Small size	12 1.3%	2 0.6%
Indeterminate	44 4.9%	5 1.6%
Total	902	322

Table 6.3: *Taxonomic distribution of the TD10.1 bone bed specimens (NISP/NSP) with anthropic induced modifications (Rodríguez-Hidalgo et al., 2015).*

One *Canis/Cuon* specimen was also found bearing cut-marks. The analysis of cut-marks and the relative position and frequency with respect to the anatomical elements shows a very similar pattern considering medium and large prays (Fig. 6.17: a, b). Morphological traits and distributional patterns of cut-marks permitted the identification of several actions connected to the butchering activity, such as skinning, eviscerating, dismembering, disarticulating, defleshing and periosteum removal (Fig. 6.17: d).

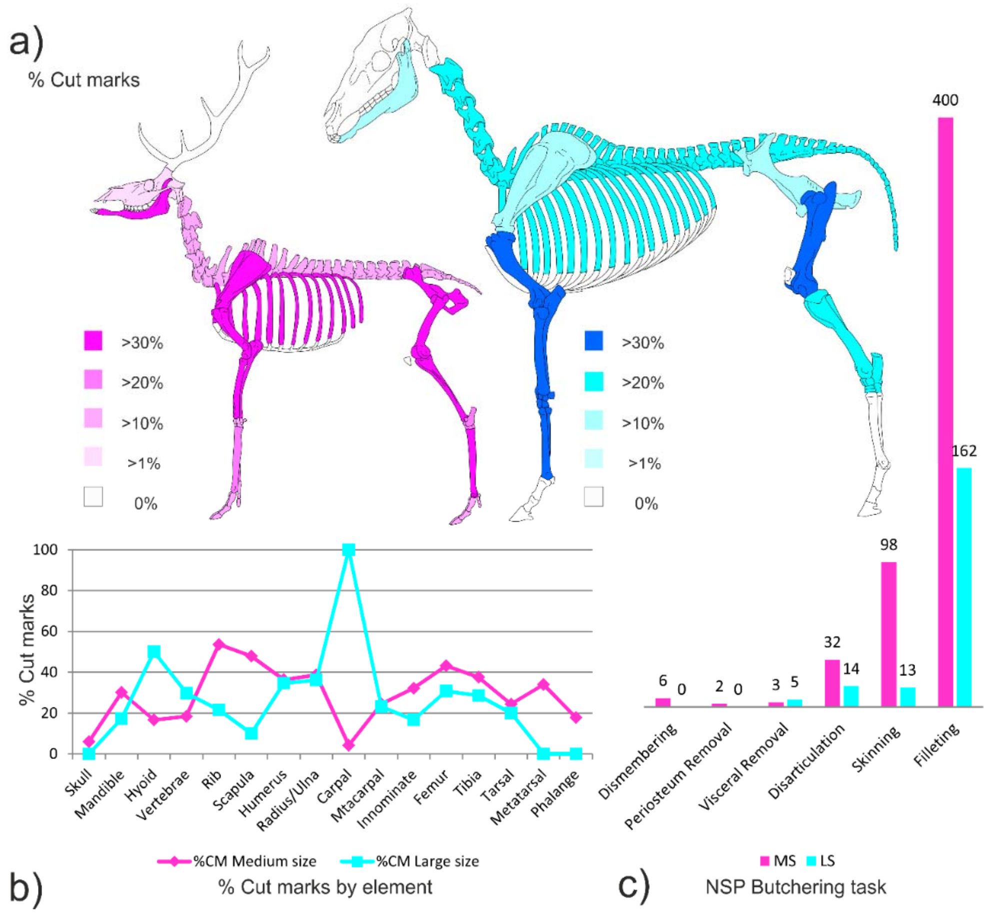

Fig. 6.17: *Summed-up graphs of the taphonomic results on the archaeofaunal record of the TD10.1 bone bed: a) Frequency distribution of cut marks on different anatomical elements in medium-sized animals (represented by red deer) and in large-sized animals (represented by horse); b) Detailed information by anatomical elements of cut marks in medium and large-sized animals; c) Bar chart of the number of cut marks referring to specific butchering tasks (Rodríguez-Hidalgo et al., 2015).*

Moreover, hominin tooth marks were identified on 66 specimens, while carnivore tooth marks are present on 306 specimens. The low incidence of modifications related to carnivores reinforced the hypothesis of this level reflecting long-terms human occupations. The faunal accumulations is seen as having anthropogenic causes and directly deriving from systematic hunting. An intensive selection of type of prey and age is shown by the high frequency of primary-adult red deer. Finally, complete butchering sequences define the site as a residential camp during the formation of the TD10.1 bone level (Rodríguez-Hidalgo, 2015; Rodríguez-Hidalgo et al., 2015).

The faunal record of the TD10.2 unit is composed of more than 60 000 elements, 45,000 of them being comprised in the same archaeo-level (the bison bed bone). A sample of *ca.* 25,200 corresponding to the sector where this archaeo-level showed more uniformity (N-E) was studied and showed a taxonomic composition of almost a unique species, *Bison* sp. (Rodríguez-Hidalgo, 2015; Rodríguez-Hidalgo et el., 2016, 2017). In fact, more than 98% of the analysed elements is associated to this species and pertain to a minimal number of 60 individuals. The stratigraphic unit (bison bed bone) has been named after this unique composition. Apart from bison, other taxa were identified, namely horse, deer, lion, wolf, lynx, fox (table. 6.4). Mesovertabrates (leporids and birds) are also present.

Taxa	**NISP**	**%NISP**	**MNI**			
			Young	**Prime**	**Old**	**Total**
Bison sp. (small)	22,532	98.4	21	36	3	**60**
Equus sp.	55	0.2	3	2	0	**5**
Cervus elaphus/*Dama dama clactoniana*	48	0.2	1	2	1	**4**
Capreolus priscus	4	0.02	1	1	0	**2**
Panthera leo spelaea	12	0.05	1	1	0	**2**
Canis lupus	7	0.03	0	3	0	**3**
Cuon alpinus europaeus	3	0.01	0	1	0	**1**
Canidae indet. *Canis*/*Cuon* cf.	51	0.2	1	3	0	**0**
Lynx sp. *pardinus* cf.	8	0.03	1	1	0	**2**
Vulpes vulpes	29	0.1	0	3	0	**3**
Mustelidae indet cf. *Meles meles*	4	0.02	0	1	0	**1**
Mustela putorius	1	0.001	0	1	0	**1**
Carnivora indet.	9	0.04	0	0	0	**0**
Castor fiber	16	0.07	1	1	0	**2**
Hystrix sp.	2	0.01	0	1	0	**1**
Marmota marmota	5	0.02	0	1	0	**1**
Oryctolagus sp.	58	0.2	2	4	0	**6**
Erinaceus europaeus	3	0.01	0	1	0	**1**
Testudo hermanni	1	0.001	0	1	0	**1**
Ave	41	0.2	0	4	0	**4**
Total	24,216	-	**32**	**68**	**4**	**104**

Table 6.4: *Number of Specimens (NSP), Number of identified specimens (NISP), Minimal Number of Elements (MNE) and Minimal Number of Individuals (MNI) by taxonomic group for the bison bone bed level of TD10.2 Gran Dolina site (Rodríguez-Hidalgo et al., 2016).*

Cut marks were identified on 4,5% of the elements and have been related mainly to de-fleshing. Skeletal part representation, presence of cut-marks and the mortality profile very close to a living population structure allowed the identification of the main activity of the site as the hunting-butchering of bison. Evidences of communal hunting are inferred by the large number of prey and the systematic seasonal occupation of the site. In fact, seasonality was identified through the analyses of dental eruption and dental microwear (Rodríguez-Hidalgo, 2015; Rodríguez-Hidalgo et al., 2016). A bimodal occupational pattern during the year (late spring and early autumn) was proposed and this would be the result of high mobility rates of the hominin groups visiting Gran Dolina site at this moment.

All the analyses of the faunal remains of the upper sub-levels of TD10 coincide in the principle statement that the level is an archaeological palimpsest, formed by different occupational phases. Although the activity of carnivores was identified and is important to understand the assemblages as a whole, the human agent had a unique importance in the formation of the faunal accumulations. More specifically, at least in the TD10.1 bone bed, which coincides with the archaeo-level H, the cave functioned as a residential camp. Conversely, the bison bed bone provides evidences of great specialisation in the exploitation of one single animal resource. Specialisation, which is reinforced by the analysis of the lithic assemblage, which is almost exclusively composed by chert and show evidences of expedite knapping strategies.

6.4 The chronological framework: The European Late Acheulean

Although one of the current most lively debates related to the Acheulean is about its early phase and origin, its last phase and transition to the Early Middle Palaeolithic are also topics of much interest. The Acheulean techno-complex covers a period of *ca* one and a half million years. It is present over wide geographical areas, in three different continents. Obviously, it is characterised by a large variability in terms of technology, raw materials and types composition. Its first evidence was found in Africa, in West Turkana (Kenya) (Roche, 1995) and it is dated to 1.8 Ma, but the complex was originally named after a European site located in the Somme Valley, France. St. Acheul was then the first locality where bifaces were catalogued as a new type of prehistoric instrument.

The Acheulean has been described as a unity displaying particular characters which permit the adscription of all assemblages ranging from 1.8 Ma to *ca.* 300ka to the Mode 2 (Clark, 1969). Such characters comprise the presence of large flakes (longer than 10cm) and LCTs (Large cutting tools made on large flakes, mainly handaxes and cleavers), and the application of centripetal methods.

At Konso Gardula, the oldest Acheulean assemblage identified up to now (KGA6-A1) shows already typical Acheulean traits such as large flakes, some of them unifacially or bifacially modified into picks, handaxes or cleavers. The main raw material employed is local basalt and a clear bifacial component is lacking (Beyene et al., 2013).

The chronological subdivision of the African Acheulean was mainly constructed based on the archaeological record from Olduvai Gorge in Tanzania (Leakey, 1971, 1975). A recent revision on the origins of the Acheulean made by de la Torre (2016) provided a thorough descriptions of the early Acheulean industries in Africa. He underlined the main problems connected with the emergence of the Acheulean, from the few direct association of lithics and human fossils to the presence of lithic industries lacking handaxes after the emergence of the Acheulean. Moreover, there is not a clear ascription of Acheulean industries to a univocal human species and cultural mechanism which led to the technological shift between Oldowan and Acheulean industries is still not completely understood.

In the Levant, Acheulean assemblages date back to 1.5 Ma. Reference sites, such as Ubeidiya (Bar Yosef and Goren-Inbar, 1993) and Gesher Benot Ya'aqov (Goren Inbar and Sharon, 2006) allowed the reconstruction of the first phases of the Acheulean techno-complex of the middle-east, from the Early Acheulean until the Late Acheulean (Sharon and Barsky, 2016). Typical characters of the early phase at Ubeidiya are the high presence of picks, handaxes and spheroids, while cleavers are absent. Coarse raw materials, such as quartz, quartzite, basalt and limestone dominate the assemblages. The Large Flake Acheulean observed at GBY is the intermediate phase, where the main lithic production generally aims to obtaining large flakes (>10cm), which are generally modified into bifacial tools. There is a high abundance of handaxes and cleavers (Sharon, 2007). The Late Acheulean sees changes in the production strategies, raw material use and percentages of types. The most used material at many sites is flint, handaxes continue to be abundant, while cleavers are practically absent. Moreover, the production of large flakes is not the main technology applied anymore. While in Africa and in the Levant the Acheulean assemblages display amazingly high concentrations of LCTs in some sites, in Europe the presence of these types often shows low percentages.

The origins of the European Early Acheulean are difficult to track. It has been proposed, among others, that early African migrations through the Gibraltar Strait or the Levant might be the cause of the early onset of this techno-complex in the European continent (*e.g.* Santoja et al., 2016; Sharon and Barsky, 2016). The main evidence to sustain one of these hypotheses is the similarities observed between the Levantine and the European Early Acheulean. Other theories see a Western European or Asian origin for the raise of the Acheulean in the European continent, based on the pre-Acheulean series of Dmanisi (Baena et al., 2010; Mgeladze and Moncel, 2016; Lordkipanidze, 2017), or a local development (Carbonell et al., 2017).

Contacts with the Levantine Acheulean would explain the technological similarities such as the presence of large flakes, found in early sites in the Iberian Peninsula (Mosquera et al., 2016). The recent discovery of La Boella in Spain (Tarragona) is a further step in the comprehension of the worldwide phenomenon which is the Acheulean (Vallverdú et al., 2014). The two bifacial tools found at this site are among the oldest ones of Europe, together with Notarchirico in Italy and La Noira and Arago in France (Pereira et al. 2015; Moncel et al. 2016; Mosquera et al., 2016).

Although the presence of handaxes is necessary to consider an assemblage Acheulean, based on traditional definitions (Leakey, 1971), apparently assemblages without handaxes are frequent at the same time both in Africa and in the Levant since its early phases (Sharon, 2007). The absence of bifaces is also documented in Central Europe, where assemblages with Acheulean handaxes are not found (Rocca, 2016).

Because of the presence of contemporary assemblages with and without handaxes and cleavers and of the Acheulean's high internal variability recorded worldwide as soon as it appears in the archaeological record, a recent conference was held in Paris to discuss important topics such as the definition of the term Acheulean, its possible change or elimination, dating, variability, etc. (Quaternary volume 411 and references therein). It emerged that, despite of the maintenance or deletion of the term, the concept of the Acheulean considered as a whole is very difficult to maintain in the light of recent discoveries and debates.

In fact, it has been suggested the presence of several 'Acheuleans' or different traditions in the Western Europe assemblages (Nicoud, 2013; Moncel et al., 2015, 2016). Moreover, the mere presence of bifacial tools does not seem to be diagnostic of a unique tradition. The decomposition of bifacial artefacts demonstrated that several different tools might be present in the traditional concept of the handaxe and therefore, different traditions may have played a much major role than previously thought in the development of lithic industries between MIS16-11 (Boëda, 2001, 2013; Nicoud, 2013). Core technologies and land-use patterns must be also considered when trying to define these traditions.

The enigmatic character of this techno-complex is found throughout its origin and development, until its transition to the Middle Pleistocene industries. The early phase of the Acheulean in Europe (until MIS16) is not very well known because of a lack of data. There are few sites with stratigraphically excavated and dated layers, there is the problem of defining its origin, if this happened through the Gibraltar Strait, from Asia (Kuhn, 2013) or from migrations from the Levant. Assemblages pertaining to this phase are better known in Southern Europe and evidences from Northern regions are scarce. La Noira site certainly represents an exception (Moncel et al., 2013, 2016).

The scant frequency of sites in Europe during this early phase and is generally interpreted as the results of several depopulation events, probably caused by rapid and intense climatic changes (Mosquera et al., 2013).

From MIS11 onwards sites are more and more frequent and a well-developed bifacial technology is visible in Western and Southern Europe. The high frequency of sites between after the MIS12 is interpreted as a re-population of the continent due to new migration waves (Mosquera et al., 2013)

In comparing the industries from different Acheulean sites, it is difficult to understand the significance of the presence/absence of bifaces, given that these tools are often seen to be so emblematic of Acheulean culture. It is unclear whether discrepancies in this feature can be explained by differences in traditions or perhaps in the function of the sites. However, long and well-dated archaeo-stratigraphic sequences (*e.g.* Galería-Atapuerca, García-Medrano et al., 2014) with exceptionally abundant artefacts (Roberts and Parfitt, 1999) allow to better comprehend local evolutionary trends in relation to bifaces.

True Acheulean assemblages are absent in Central Europe, lacking bifaces and comprising small *débitage* and small retouched flakes. These industries and generally ascribed to *pre-Mousterian* traditions (*e.g.* Doronichev and Golovanova, 2010; Rocca, 2013). Vertesszölös in Hungary, Bilzingsleben and Shöningen in Germany, Korolevo VI in Ukraine are further examples of this "*facies"* (Rocca, 2013). These industries might be the key towards the understanding of the rise of early Middle Palaeolithic traditions in Western Europe during MIS10-7 in Europe.

6.4.1 The late Acheulean and the transition to the early Middle Palaeolithic

The mosaic character of the early and middle phases of the Acheulean complex is also present in its last phase in the European continent. The late phase coincides with the transition to the early Middle Palaeolithic, during MIS10-7. The transitional phase leading to the Middle Palaeolithic in Western Europe is characterised by the appearance of new traits in early Middle Palaeolithic sites: more diversified technological behaviours reflected in more complex knapping methods, predetermined strategies and an increasing standardisation of tool types. The regular use of fire, hafting techniques, more complex hunting strategies and an increasing evidence of symbolic behaviour are constant in the definition of the transitional phase from the Lower to early Middle Palaeolithic (Moncel et al., 2012; Kuhn, 2013; Rots, 2013).

These traits may occur very rapidly, suggesting a break with the previous traditions, or they can happen progressively on geographically wide regions. The vision of progressive change is preferred as, in many cases, links with the previous Late Acheulean traditions are evident. Examples of traditions linked to the Acheulean substratum dated to MIS9-8 (400-250ka) are the Acheulo-Yabrudian in the Levant, where the presence of bifaces in some of its facies is attested (Barkai et al., 2003), and the Micoquian in Europe with various bifacial tools (Kozlowski, 2014). These Late Acheulean traditions are contemporaneous with other traditions, possibly leading to the development of the formal Middle Palaeolithic.

At Vértesszöllös in Hungary, Bilzingsleben, or Schöningen in Germany (MIS9/MIS11), reduction sequences focus on the production of small tools and bifaces are absent Rocca, 2013, in press) (Fig. 6.18).

Also in Western Europe, several traditions connected either with the Acheulean techno-complex or to the Middle Palaeolithic one, are thought to coexist during MIS11-8 (Santonja and Pérez-González, 2010; Malinsky-Buller, A., 2014, 2016; Mathias, C., 2016; Santonja et al., 2016). Generally, in assemblages lacking bifaces, an increasing degree of tool standardisation is noted. Flake-tool-dominated assemblages present high predetermination degrees in the reduction strategies and high standardisation of tool types, as well as re-sharpened tools

La Cueva de la Bajada site in Northern Spain, where the presence of small-sized tools, re-sharpening and exhausted cores are well attested (Santonja et al., 2014), provides one example of advanced Acheulean traditions.

The industries from level TD10 at Gran Dolina (MIS11-9) shows analogous features, such as a high standardisation of small-sized tools, few bifacial tools and the application technological strategies with a high degree of morphometrical predetermination (Ollé et al., 2013)

In France, Orgnac 3 displays the same characters ascribed to transitional industries: long and complex reduction sequences, highly standardised tools and the normalised production of Levallois blanks (Moncel et al., 2012).

Moreover, bone retouchers are also another key element to define modern behaviour, found at both Orgnac 3 and GD-TD10.1 (Moncel et al., 2012; Rodríguez-Hidalgo, 2015).

The upper levels of the Caune de l'Arago (levels G, D) are another example of transitional industries, where small-sized tools (mainly side-scrapers) are found alongside few handaxes and choppers. Quartz dominates, composing the almost entire industry. Evidence of the application of discoid methods as well as a limited presence of the application of the Levallois method have been documented (Barsky, 2013).

Fig. 6.18: *European map with the. early middle Palaeolithic sites mentioned in the text.*

The possibility that the variability encountered in the assemblages dated from MIS 11 to MIS 8 in the European continent is perhaps due to the presence of two distinct or more traditions explains the extremely different patterns and technological features found within them (Santonja et al., 2016). This view is tempting when considering assemblages located at relatively large distances one from another, but it is more difficult to sustain where the assemblages presenting significant technological differences are place next to each other. This is the case of Galería site and Gran Dolina sites, at Atapuerca (Ollé et al., 2016a). After the new dates of the Galería's layers (GII, GIII), it appeared that the technologically full Acheulean found at Galería is slightly younger that the transitional assemblage recognised at TD10.1 (Ollé et al., 2013). Therefore, alternative explanations are to be found to better understand the transitional shift to the Middle Pleistocene productional systems. Regarding this specific example, as the two sites are found next to one another, it is plausible to think that the lithic assemblages were produced by human groups with similar, if not the same, traditions. If this is correct, the differences observed in the two assemblages may be due also to functional reasons (Ollé et al., 2016a).

Obviously, at a continental scale one can see a clear distinction between two different technological traditions, but only a detailed study of the lithic assemblages allows to assess the role played by the Acheulean substratum to the development of such traditions.

It is known that technological features associated to the transition from the Lower Palaeolithic to the Middle Palaeolithic mark a technological change and this is reflected in the composition of lithic assemblages. Characters such as the decrease of large tools and the increase of standardisation of small-sized tools are constantly found in the so-called transitional assemblages. Although the roots of technological changes are usually explained as traditional shifts, we cannot underestimate the role that functional needs may have had on the composition of lithic assemblages.

From MIS7 onward, as in other regions of the World, the situation appears more stable: Mousterian Middle Palaeolithic assemblages rapidly dominated, replacing all remaining evidence of the Lower Palaeolithic traditions. The site of Payre, studied in this thesis, is inserted in this chronological framework, where Middle Palaeolithic features dominate.

6.5 Techno-functional analysis of the quartzite assemblage of GD-TD10.1

Out of all the artefacts analysed technologically (N= 519), 129 have been ascribed to four techno-functional groups (Table 6.5; Annex 6). The inclusion or exclusion of the artefacts from the adscription to techno-functional groups was done based on their general structure plus on the presence/absence of particular features on both the usable and prehensile areas, respectively the active and prehensile techno-functional units. Metrical features were considered as well and artefacts shorter than 30mm were not included. Therefore, those artefacts presenting no features related to usable edges and to edges apt to prehension (here mostly intended to be manual), were excluded from the counting of techno-functional groups.

Regular edges (possibly being usable) are identified when the same characters (for example angle, frontal delineation) are found on a considerable length. The prehensile parts are also identified when particular features (for example obtuse angles, rectilinear or convex frontal delineations, locations opposite or adjacent to the cutting edges) are found on significant lengths. Techno-functional groups are usually identified based on the recurrence of the same volumetric structures and technical characters on artefacts.

First, a brief description of the main groups identified after the application of the techno-functional analysis is provided, while each techno-functional group and related sub-categories are further described in detail. The main groups have been identified based on the volumetric structure of the blank (morphology and measurements), location of the abrupt and the cutting edges and their relative position on tools.

For a better understanding of the text some definitions of the main terms used in the description of the four techno-groups are provided:

- Edge or dihedral: are general terms used to define the convergence of two surfaces in a rim, characterised by certain angles and delineations;
- Cutting edge: it is recognised for the regularity of the same criteria used to define an edge and its presence on a certain length (Lepot, 1993). For example, regular edge delineations, constant angles apt to modify the matter (not excessively acute nor obtuse), etc.);
- Convergence or trihedral: are terms used to indicate a junction of two edges into a triangular tip. Both edges are characterised by their own cutting plane, angle, frontal and sagittal delineations;
- Back: is an abrupt edge, natural (cortical) or not, which, due to its technical features, cannot be used to modify the matter. The identification of banked edges is crucial to understand the possible prehensile portions present on tools.

Within each group, sub-categories have also been identified. Aspects such as the angle and frontal delineation of the edge, number and location of the transformative (t-TFU) and prehensile (p-TFU) techno-functional units participated in the adscription of each piece to techno-functional sub-types.

6.5.1 Volumetric structure of the products

Based on the combination of technical characters, resulting in similar geometries, four main groups have been identified (Fig. 6.19). The four groups act like wide categories, comprising similar general features (measurements, morphology, position of edges and backs).

1. Techno-functional group I. is composed of a dihedral opposed to a back. The morphologies associated are then quadrangular-rectangular and half-moon-like (when the backed edge has a convex frontal delineation);
2. Techno-functional group II. is formed of two opposed dihedrals connected by a third distal dihedral. Only quadrangular-rectangular pieces form this group;
3. Techno-functional group III. is a convergence (trihedral) of two dihedrals and it has a basal abrupt edge. Triangular morphologies dominate this group.

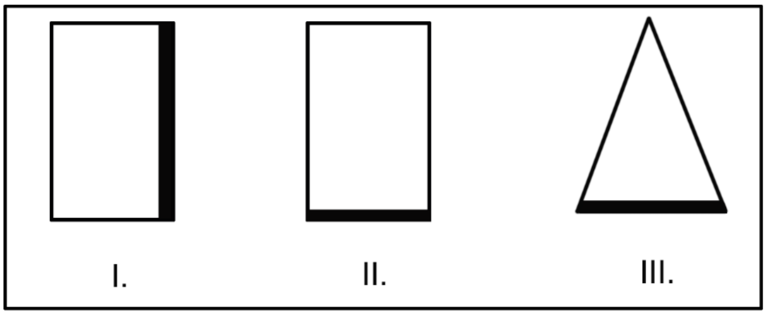

Fig. 6.19: *Three main techno-functional groups identified from the techno-functional analysis of the quartzite assemblage of TD10.1. I.) a dihedral opposed to a back; II.) two usable dihedrals divided by a distal dihedral; III.) a convergence of two dihedrals. Edges in bold represent backed edges.*

After defining the number of artefacts ascribed to each type, their relative abundance has been made visible on a table (Table 6.5). A higher abundance of techno-functional group I. is evident with 91 artefacts pertaining to this class. Techno-functional groups II. and III. present equivalent values (n= 19).

The sub-categories of each techno-functional group have been defined based on the characteristics of the transformative and prehensile techno-functional units.

Techno-functional group	Sub-types	Number	Total
I.	I.a	30	**91**
	I.b	18	
	I.c	22	
	I.d	15	
	I.e	6	
II.	II.a	5	**19**
	II.b	12	
	II.c	2	
III.	III.a	12	**19**
	III.b	4	
	III.c	3	
			129

Table 6.5: *Relative presence of each sub-group. In bold is the total number of artefacts per techno-functional group and at the bottom the total number of artefacts ascribed to techno-functional groups (129).*

6.5.2 Techno-functional Group I.

This group of instruments is the most represented in the assemblage with 91 samples (Table 6.5). It is formed by a unique t-TFU opposed to a back (Fig. 6.19: I.).

The analysis of the main measurements is showed in a box-plot: the mean value for the length ranges between 30 and 50mm, while the mean value for the width ranges between 30 and 45mm. The more frequent morphologies observed are rectangular and half-moon, followed by trapezoidal and quadrangular ones (Table 6.6).

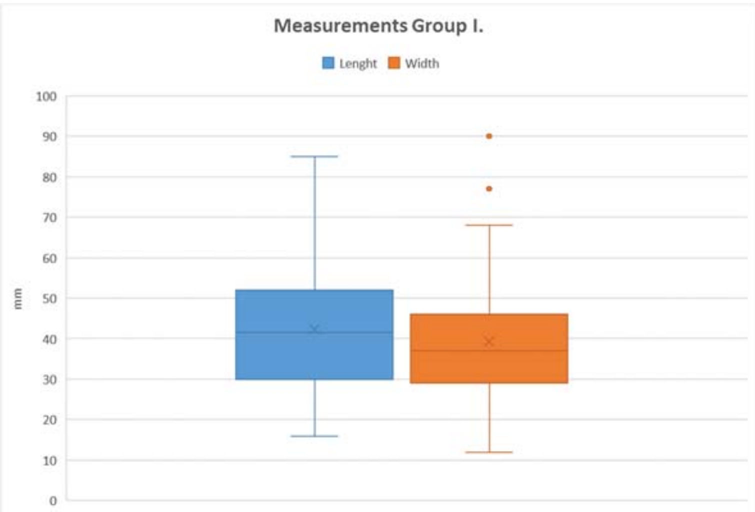

Fig. 6.20: *Box-plot showing the mean measurements of the artefacts pertaining to the Group I.*

Blanks are mostly non-cortical (n=54), with a lower presence of the semi-cortical ones (n=36) (Table 6.7). There is a high presence of cortical platforms (n=45), followed by flat (n=29) and prepared (n=7) ones. The number of retouched (n=52) and unretouched (n=38) blanks is approximately the same.

BLANK MORPHOLOGY GROUP I.			
	Description	**Number**	**Tot.**
Morphology	rectangular	33	**91**
	half-moon	22	
	trapezoidal	17	
	quadrangular	16	
	irregular	3	

Table 6.6: *Main morphological features of the artefacts ascribed to techno-functional Group I.*

BLANK TYPE GROUP I.			
	Description	**Number**	**Tot.**
Blank type	semi-cortical flake	37	**91**
	non-cortical flakes	54	
Retouch	retouched flake	53	**91**
	unretouched flakes	38	
Platform	flat	29	**81***
	cortical	45	
	prepared	7	

Table 6.7: *Blank features of the artefacts ascribed to Group I.*
**The total number of the platforms is not 91 as some of the samples are broken and have no platform.*

6.5.2.1 Techno-functional units of Group I.

Techno-functional group I. is characterised by one lateral cutting edge and an opposite back. The dihedral may have been retouched, while the bank may be cortical or not. Different sub-types are recognised within this group by the association of different frontal delineations and angles of the t-TFUs and the p-TFUs. Therefore, there are five different sub-types pertaining to Group I, which are presented in detail in the following sections (Fig. 6.21).

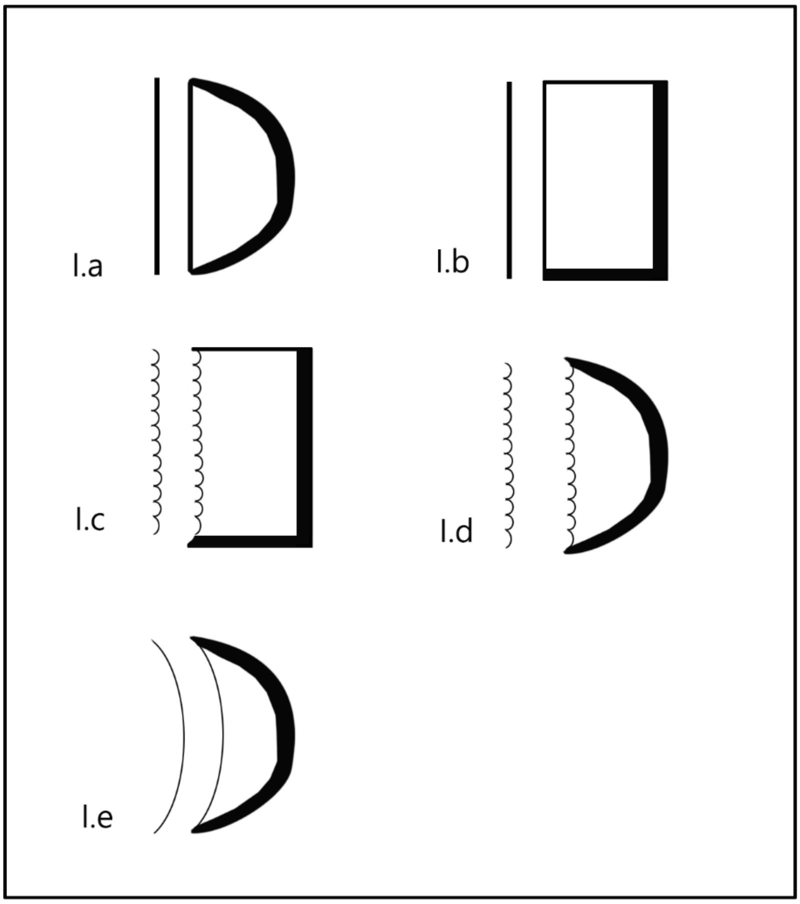

Fig. 6.21: *Schematic representation of the five sub-types of Group I. Edges in bold represent backed edges.*

6.5.2.1.1. Sub-type I.a

Sub-type I.a is composed by a dihedral opposed to a backed edge (Fig. 6.21: I.a; Fig. 6.24, 1-2, 4-7, 10). The angles of the t-TFUs are comprised between 30° and 70°, while the cutting plane is in the majority of cases plane/plane (n=18) (Table 6.8). Their frontal delineation is generally rectilinear (n=20) (Fig. 6.22).

The p-TFUs are located always on the opposite edge of the t-TFUs; their angles are mostly obtuse (n=22) and the cutting plane is generally plane/plane (n=18) (table 6.9). Their frontal delineation is found to be regularly convex (n=29) (Fig. 6.22). In several cases (n= 17), the p-TFUs are found on cortical backs.

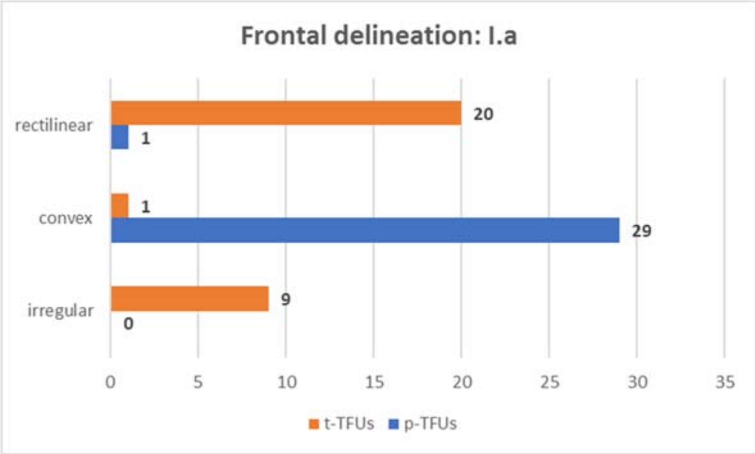

Fig. 6. 22: *Frontal delineation of t-TFUs and p-TFUs of Sub-type I.a.*

6.5.2.1.2. Sub-type I.b

Sub -type I.b is described as having a retouched edge on one of the lateral edges, an opposed un-modified edge and a distal irregular edge (broken distal part, oblique edge, etc.) (Fig. 6.21: I.b; 6.24: 3, 8-9; 6.28: 5). 18 artefacts are ascribed to this sub-type (Table 6.5).

The angle of the transformative units ranges between 30° and 70° and the cutting plane is mostly plane/plane (Table 6.8). The frontal delineation is mostly rectilinear (n=9), followed by denticulate and convex ones (n=3) (Fig. 6.23). The p-TFUs are always found on the edge opposite to the retouched one. They are always rectilinear and the angle is always obtuse (α≥70°) (Table 6.9; Fig. 6.23).

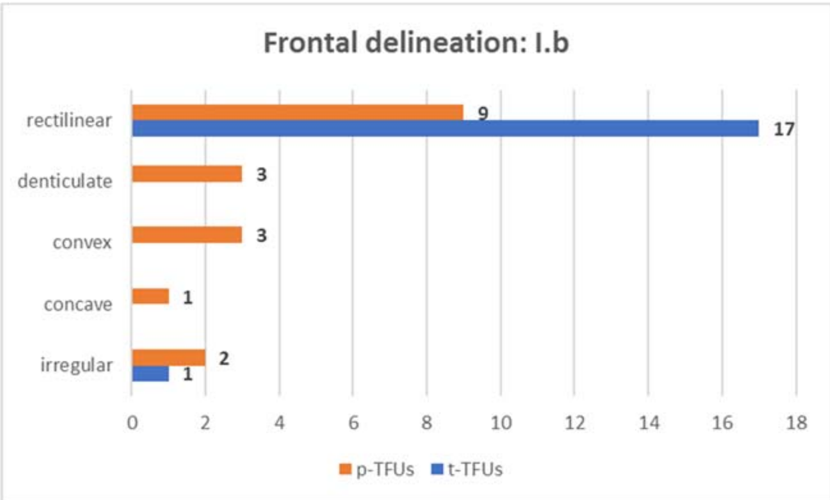

Fig. 6.23: *Frontal delineation of t-TFUs and p-TFUs of Sub-type I.b.*

t-TFU Group I.				
Sub-type		**Description**	**Number**	**Tot.**
I.a	**Angle**	<30°	0	**30**
		30<x≤50°	10	
		50°<x<70°	16	
		>70°	4	
	Cutting plane	plane/plane	18	**30**
		plane/concave	2	
		concave/plane	9	
		concave/concave	1	
I.b	**Angle**	<30°	0	**18**
		30°<x<50°	0	
		50°<x<70°	18	
		>70°	0	
	Cutting plane	plane/plane	10	**18**
		plane/concave	2	
		concave/plane	5	
		convex/concave	1	
I.c	**Location**	proximal	1	**22**
		distal	7	
		lat. right	4	
		lat. left	10	
	Angle	<30°	0	**22**
		30°<x<50°	4	
		50°≤x<70°	15	
		>70°	2	
	Cutting plane	plane/plane	8	**22**
		plane/concave	2	
		concave/plane	10	
		convex/concave	1	
		concave/convex	1	
I.d	**Angle**	<30°	0	**15**
		30°<x<50°	2	
		50°<x<70°	12	
		>70°	1	
	Cutting plane	plane/plane	6	**15**
		concave/plane	8	
		concave/convex	2	
I.e	**Angle**	<30°	0	**6**
		30°<x<50°	1	
		50°≤x<70°	5	
		>70°	0	
	Cutting plane	plane/plane	2	**6**
		concave/plane	1	
		concave/convex	3	

Table 6.8: *Technical characters of the transformative-TFUs of the artefacts pertaining to the 5 sub-types of Group I.*

Sub-type		p-TFU Group I. Description	Number	Tot.
I.a	**Angle**	<30°	0	30
		30°<x<50°	4	
		50°≤x<70°	4	
		>70°	22	
	Cutting plane	plane/plane	18	30
		plane/concave	2	
		concave/plane	1	
		convex /concave	1	
		convex/plane	8	
I.b	**Angle**	<30°	0	18
		30°<x<50°	0	
		50°<x<70°	0	
		>70°	18	
	Cutting plane	plane/plane	15	18
		concave/plane	2	
		plane/convex	1	
I.c	**Angle**	<30°	0	22
		30°<x<50°	1	
		50°<x<70°	2	
		>70°	19	
	Cutting plane	plane/plane	20	22
		plane/concave	1	
		plane/convex	1	
I.d	**Angle**	<30°	0	15
		30°<x<50°	3	
		50°<x<70°	1	
		>70°	11	
	Cutting plane	plane/plane	13	15
		concave/plane	1	
		plane/convex	1	
I.e	**Angle**	<30°	0	6
		30°<x<50°	0	
		50°<x<70°	1	
		>70°	5	
	Cutting plane	plane/plane	5	6
		concave/plane	1	

Table 6.9: *Technical characters of the prehensile-TFUs of the artefacts pertaining to the 5 sub-types of Group I.*

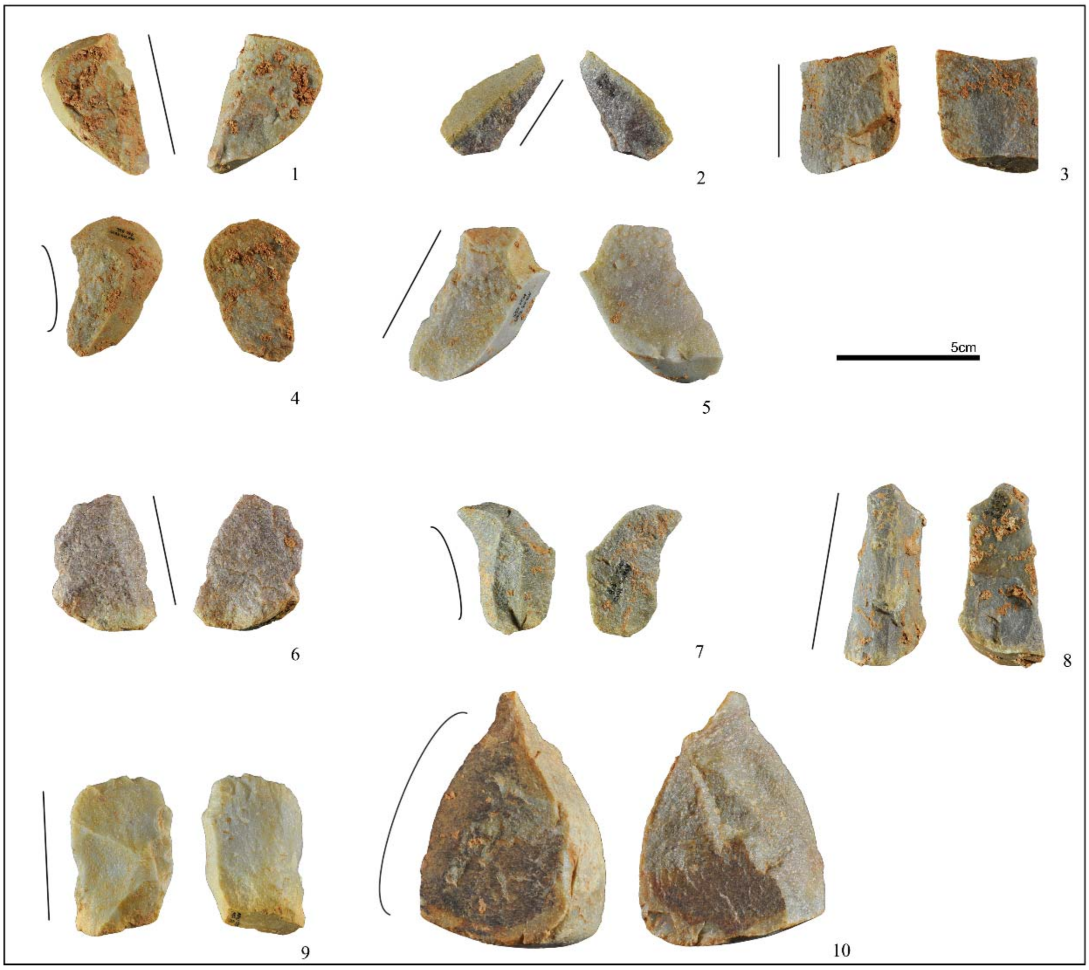

Fig. 6.24: *Some of the archaeological artefacts ascribed to the techno-functional Group I. (sub-type I.a: 1, 2, 4, 5, 6, 7, 10; sub-type I.b: 3, 8, 9).*

6.5.2.1.3. Sub-type I.c

22 artefacts have been ascribed to sub-type I.c. Sub-type I.c is formed by a retouched t-TFU, which usually has a denticulate frontal delineation (Fig. 6.21: I.c; 6.25; Fig. 6.28: 1-4), and two backed edges. One backed edge is opposed to the retouched one and the other is located on the basal part. The angle of the t-TFUs varies between 50° and 70°. The cutting plane of the t-TFUs is concave/plane on several artefacts (n=10), a direct consequence of the retouch. The frontal delineation of t-TFUs is mostly denticulate (n=20), while p-TFUs are generally rectilinear (n=16) (Fig. 6.25). The angle of p-TFUs is always obtuse and (Table 6.9) and their cutting plane is plane/plane in most of the cases (n=20).

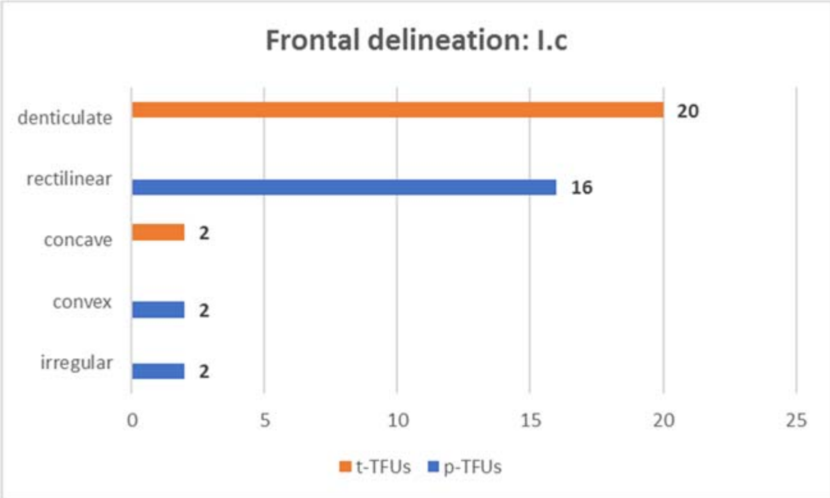

Fig. 6.25: *Frontal delineation of t-TFUs and p-TFUs of Sub-type I.c.*

6.5.2.1.4. Sub-type I.d

15 artefacts pertain to sub-type I.d. Sub-type I.d is composed of a t-TFU with a denticulate frontal delineation, opposed to a convex p-TFU (Fig. 6.21: I.d; Fig. 6.26; Fig. 6.28: 6-7, 9-10). Almost all angles are of the t-TFUs are comprised between 50° and 70° and the cutting plane can be either concave/plane or plane/plane. (Table 6.8).

p-TFUs are always found on the edge opposed to the retouched one, frequently on cortical surfaces and normally present very obtuse angles. The cutting plane of the p-TFUs is generally plane/plane (Table 6.9).

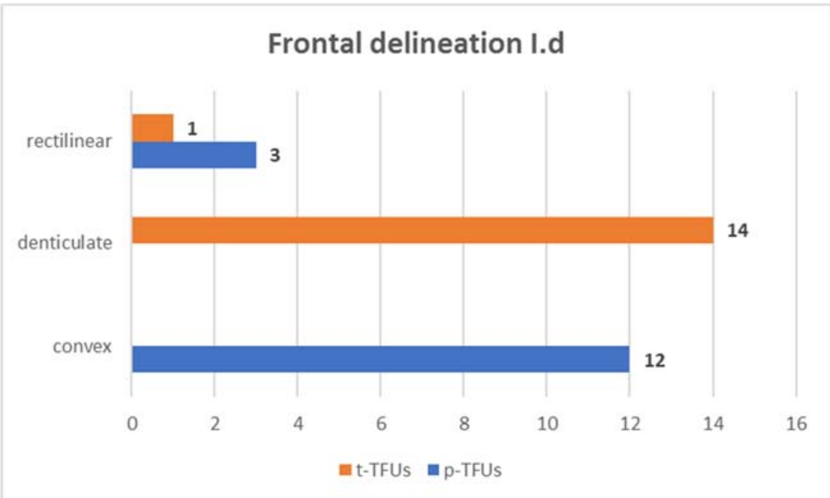

Fig. 6.26: *Frontal delineation of t-TFUs and p-TFUs of Sub-type I.d.*

6.5.2.1.5. Sub-type I.e

Only 6 artefacts have been ascribed to sub-type I.e. It is described as having a concave t-TFU, opposed to a convex, backed edge (Table 6.5; Fig. 6.21: I.e; Fig. 6.28: 8). The t-TFUs may be retouched or not. The angle is quite obtuse in all cases and the frontal delineation is, as already said, concave (Fig. 6.27). The p-TFUs are always found on the edge opposed to the t-TFUs and have a convex frontal delineation, obtuse angles and plane/plane cutting planes (Table 6.9).

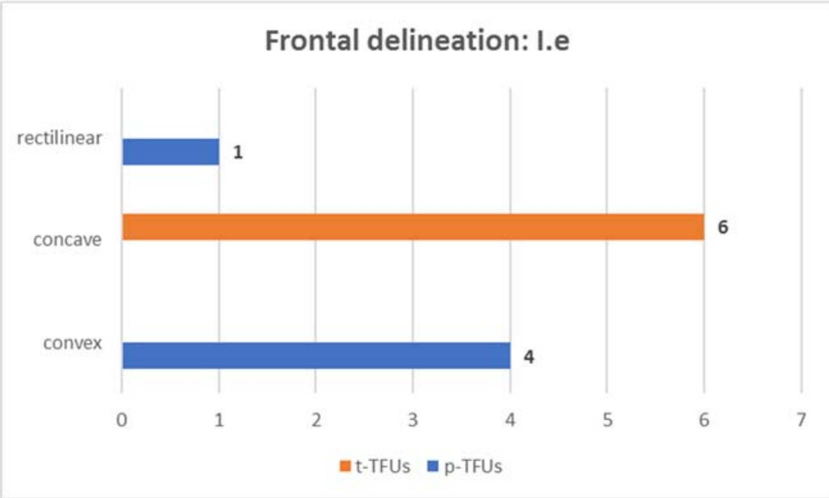

Fig. 6.27: *Frontal delineation of t-TFUs and p-TFUs of Sub-type I.e.*

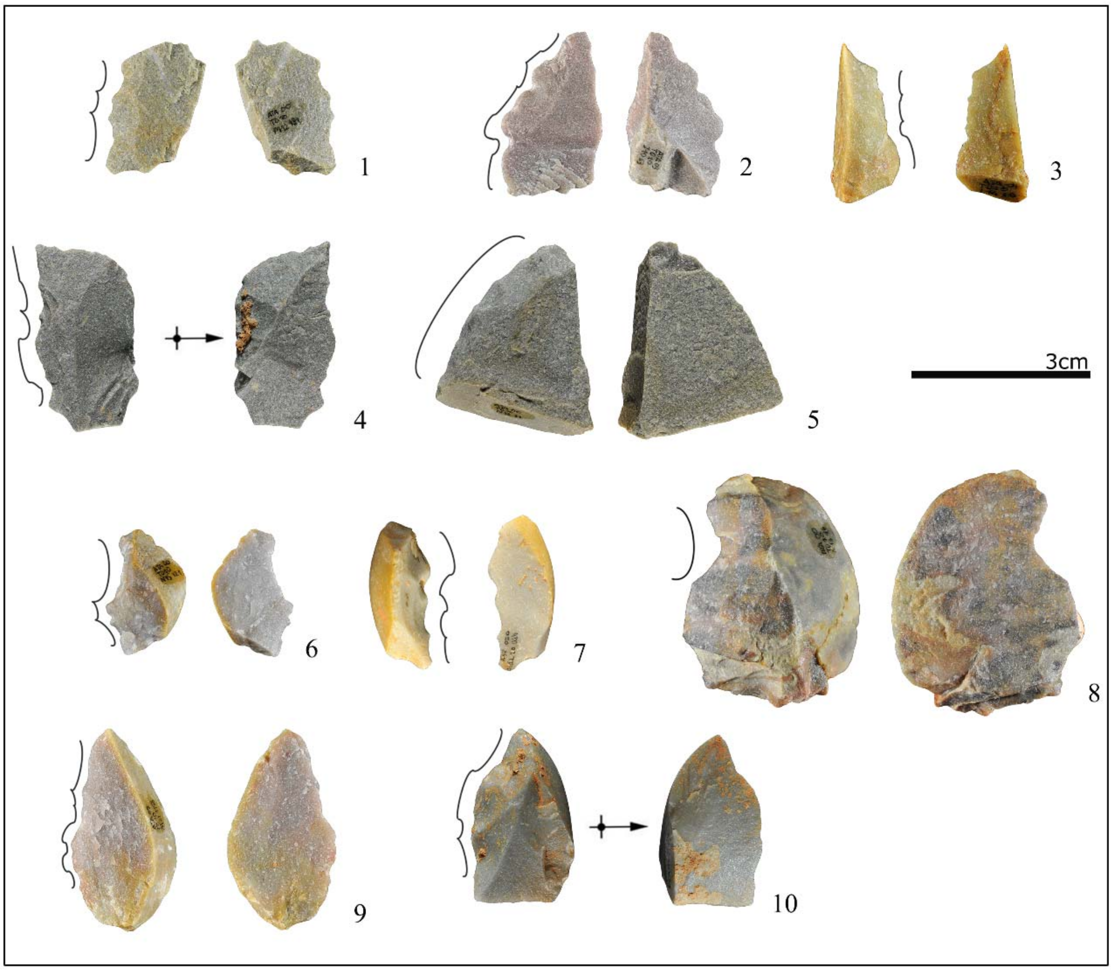

Fig. 6.28: *Some of the archaeological artefacts ascribed to the techno-functional Group I. (I.c: 1-4; I.b: 5; I.d: 6-7, 9-10; I.e: 8).*

6.5.3 Techno-functional Group II.

Techno-group II. is characterised by two lateral dihedrals and a distal dihedral (Fig. 6.19: II.). The lateral dihedrals may or may not be retouched. 19 artefacts compose this group (Table

6.5). Three different sub-types have been described (Fig. 6.30). Mean measurements see a length comprised between 35 and 50mm and a width comprised between 25 and 45mm (Fig. 6.29). Rectangular morphologies predominate (Table 6.10).
All the artefacts are non-cortical flakes and mostly unretouched (Table 6.11). Few platforms are cortical (n=3). Most of them are flat (n=13) and only two are prepared.

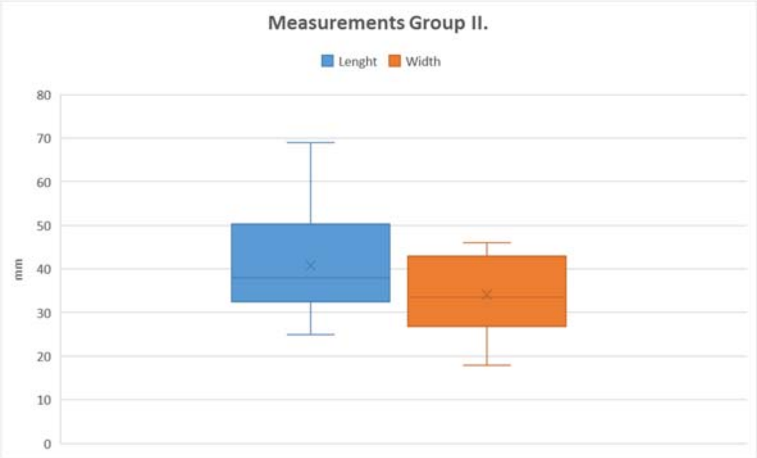

Fig. 6.29: *Box-plot showing the mean measurements of the artefacts pertaining to the Group II.*

BLANK MORPHOLOGY GROUP II.			
	Description	**Number**	**Tot.**
Morphology	rectangular	13	**19**
	half-moon	2	
	trapezoidal	1	
	quadrangular	3	
	irregular	0	

Table 6.10: *Main morphological features of the artefacts ascribed to the techno-functional Group II.*

BLANK TYPE GROUP II.			
	Description	**Number**	**Tot.**
Blank type	semi-cortical flake	0	**19**
	non-cortical flakes	19	
Retouch	retouched flake	5	**19**
	unretouched flakes	14	
Platform	flat	13	**18***
	cortical	3	
	prepared	2	

Table 6.11: *Blank features of the artefacts ascribed to Group II.*

6.5.3.1. Techno-functional units of Group II.

Three different sub-types have been identified within Group II., depending on the number of t-TFUs present and their disposition (Fig. 6.30). The sub-type II.b is the most numerous one with 12 artefacts, followed by sub-types II.a (n=5) and II.c (n=2) (Table 6.5). They usually present a basal backed edge, but p-TFUs can be identified also with one of the lateral edges, often being irregular and so less adapted to regular use. The distal trihedral very rarely present a combination of characters to be considered functional. This is why, only two artefacts have been ascribed to sub-type II.c.

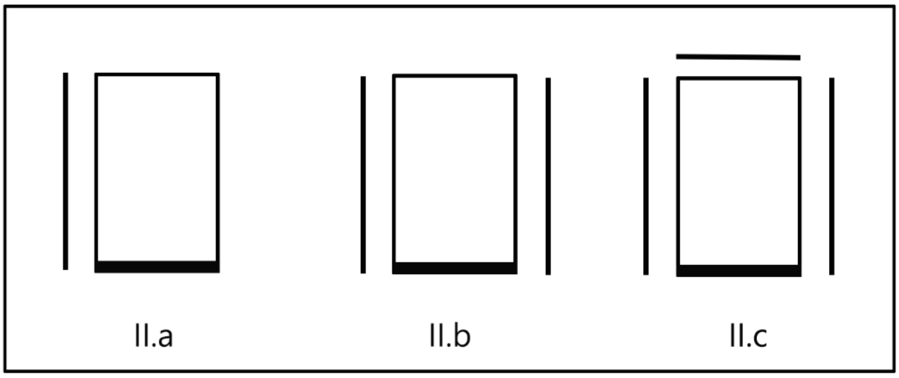

Fig. 6.30: *Schematic representation of the three sub-types of Group II. Edges in bold represent backed edges.*

6.5.3.1.1. Sub-type II.a

Sub-type I.a is composed of a unique lateral t-TFU. They usually present very irregular opposite edges, which are interpreted to be the p-TFUs, together with the basal part.

t-TFUs can be rectilinear, convex or denticulate, while the opposite edge is either rectilinear or irregular (Fig. 6.31). The basal part is always rectilinear. t-TFUs present angles ranging from 30° to 70° and the cutting plane has generally plane/plane surfaces (Table 6.12). p-TFUs always present plane/plane cutting plane and angles ranging between 50° and 70° (Table 6.13)

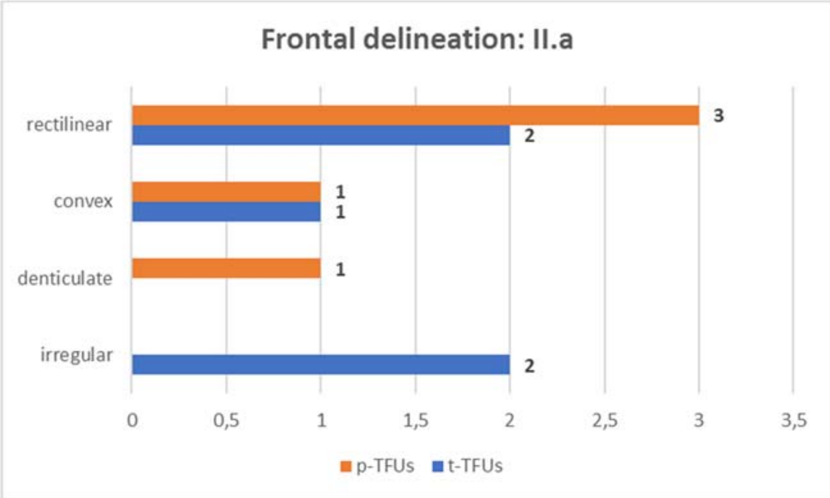

Fig. 6.31: *Frontal delineation of t-TFUs and p-TFUs of Sub-type II.a.*

6.5.3.1.2. Sub-type II.b

Sub-type II.b is formed by two lateral t-TFUs and a basal backed edge (Fig. 6.30: II.b; Fig. 6.33, 1-3). p-TFUs are thought to be the basal part plus the edges opposed to the used ones.

t-TFUs are mostly rectilinear (n=12), followed by denticulate (n=5), convex (n=3) and concave (n=2) ones (Fig. 6.32). t-TFUs present angles ranging from 50 to 70 and cutting planes can be plane/plane or concave/plane (Table 6.12).

p-TFUs are always proximal and possibly also the edge opposed to the one being used, which is hardly to be identified at this stage of analysis. Angles and cutting planes vary a lot (Table 6.13).

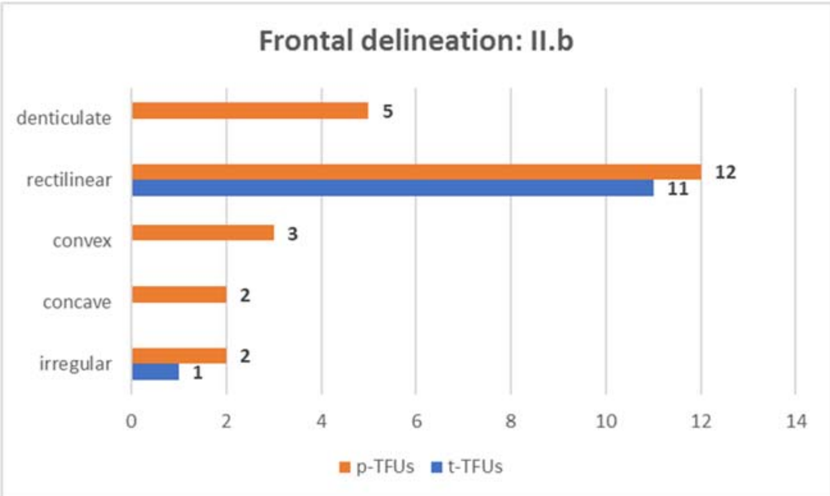

Fig. 6.32: *Frontal delineation of t-TFUs and p-TFUs of Sub-type II.b.*

t-TFU Group II.				
Sub-type		**Description**	**Number**	**Tot.**
II.a	**Angle**	30°<x≤50°	1	**5**
		50°<x<70°	4	
	Cutting plane	plane/plane	4	**5**
		concave/plane	1	
II.b	**Angle**	30°<x<50°	4	**24***
		50°<x<70°	8	
	Cutting plane	plane/plane	7	**24***
		plane/convex	1	
		concave/plane	4	
II.c	**Angle**	30°<x<50°	5	**6***
		50°≤x<70°	1	
	Cutting plane	plane/plane	4	**6***
		concave/plane	2	

Table 6.12: *Technical characters of the transformative-TFUs of the artefacts pertaining to the 3 sub-types of Group II. * The number of t-TFUs is not the same of the number of artefacts (II.b=12; II.c=2) because multiple TFUs are present (2 for II.b and 3 for II.c).*

p-TFU Group II.				
Sub-type		**Description**	**Num.**	**Tot.**
II.a	**Angle**	<30°	0	**5**
		30°<x<50°	1	
		50°<x<70°	4	
		>70°	0	
	Cutting plane	plane/plane	5	**5**
II.b	**Angle**	30°<x<50°	0	**12**
		50°<x<70°	5	
		>70°	7	
	Cutting plane	plane/plane	4	**12**
		plane/concave	6	
		plane/convex	2	
II.c	**Angle**	50°<x<70°	2	**2**
	Cutting plane	plane/plane	2	**2**

Table 6.13: *Technical characters of the prehensile-TFUs of the artefacts pertaining to the 3 sub-types of Group II.*

6.5.3.1.3. Sub-type II.c

Only two artefacts have been ascribed to sub-type II.c. It is composed of three rectilinear t-TFUs and one rectilinear p-TFU (Fig. 6.30: II.c; Fig. 6.33: 4).

t-TFUs are located on the lateral edges and on the distal one, while p-TFUs are always on the basal parts. As for sub-type II.b, p-TFUs can be also the lateral edges opposed to those being used. The angles of the t-TFUs are comprised between 30° and 70°, while the cutting plane can be plane/plane, plane/concave or plane/convex (Tables 6.12). p-TFUs have more obtuse angles and a plane/plane cutting plane (Table 6.13).

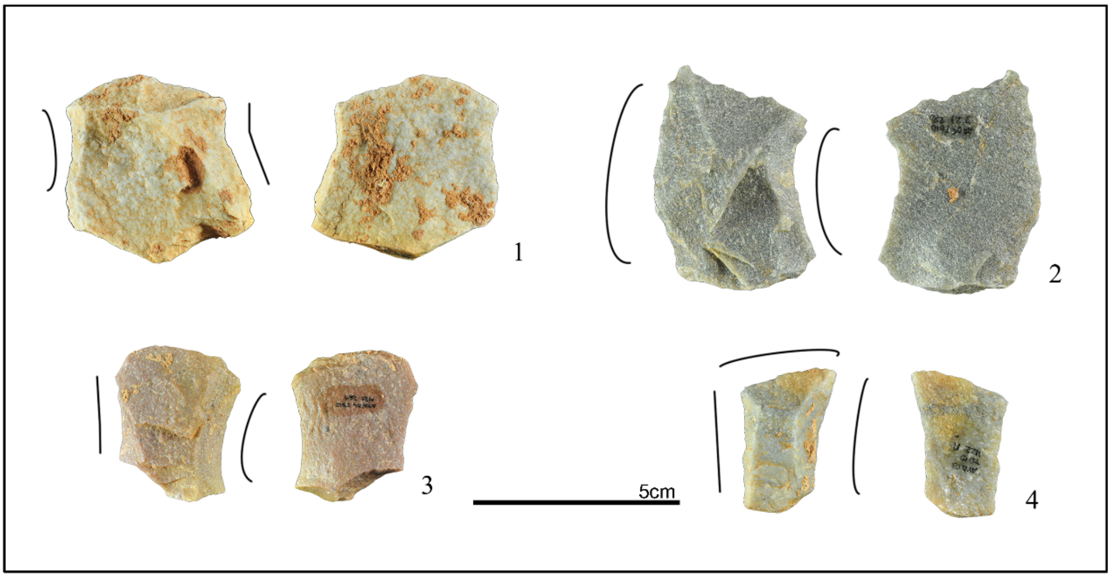

Fig. 6.33: *Some of the archaeological artefacts ascribed to techno-functional Group II.*

6.5.4 Techno-functional Group III.

Techno-functional group **III**. comprises 19 artefacts (Table 6.5). Artefacts of this group present two convergent dihedrals (Fig. 6.19: **III**). t-TFUs can be either the two lateral edges or the tip resulting from the convergence (trihedral) of them. Mean measurements are similar to those observed for the other groups, although artefacts seem to be slightly wider. Lengths are comprised between 25 and 45mm, while widths range from 35 to 50mm (Fig. 6.34).

All the artefacts present triangular morphologies. Artefacts are mostly retouched and they normally do not present any cortical surfaces (5 semi-cortical flakes out of 19) (Table 6.14). Platforms are generally flat (n=12), followed by cortical (n=4) and prepared (n=3) ones.

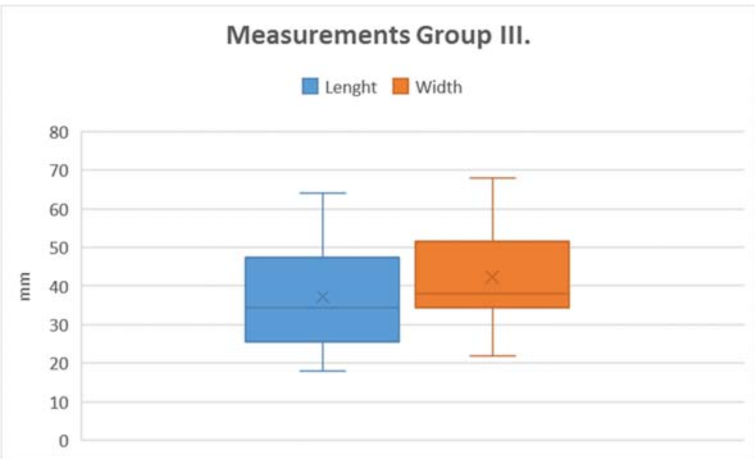

Fig. 6.34: *Box-plot showing the mean measurements of the artefacts pertaining to Group III.*

BLANK TYPE GROUP III.			
	Description	**Number**	**Tot.**
Blank type	semi-cortical flake	5	**19**
	non-cortical flakes	14	
	retouched flake	13	**19**
	unretouched flakes	6	
Platform	flat	12	**19**
	cortical	4	
	prepared	3	

Table 6.14: *Blank features of the artefacts ascribed to Group III.*

6.5.4.1. Techno-functional units of Group III.

Three different sub-types have been recognised based on the presence and combination of t-TFUs (Fig. 6.35). The most abundant sub-type is III.a with 12 artefacts, followed by sub-types III.b (n=4) and III.c (n=3) (Table 6.5).

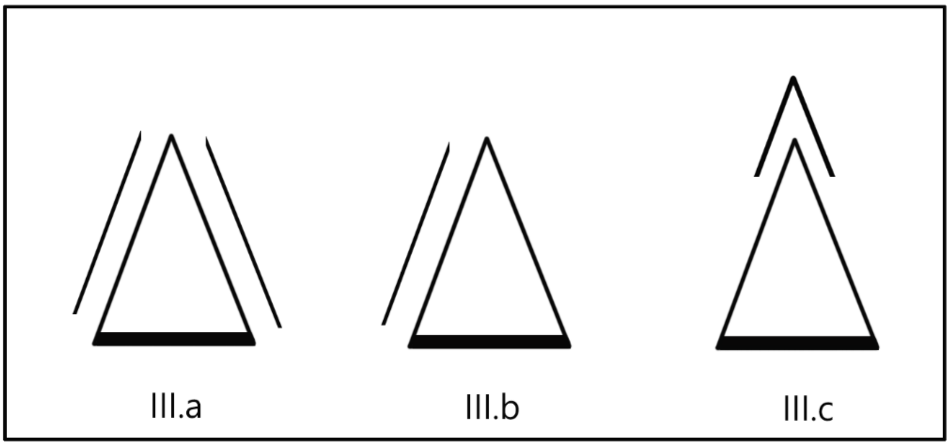

Fig. 6.35: *Schematic representation of the three sub-types of Group III. Edges in bold represent backed edges.*

6.5.4.1.1 Sub-type III.a

Sub-type III.a presents two t-TFUs on the lateral edges (Fig. 6.38: 1, 2, 4, 6, 7, 8). The angles of the edges are comprised between 50° and 70° and cutting plane is normally plane/plane (n=13) or concave/plane (n=5) (Table 6.15). Frontal delineations of the t-TFUs are mostly rectilinear, with low presence of denticulate, convex, concave and irregular ones (Fig. 6.36).

p-TFUs are always found on the proximal edge, with angles comprised between ≥50°. Cutting planes are mostly concave/plane (Table 6.16) and frontal delineations rectilinear (Fig. 6. 36).

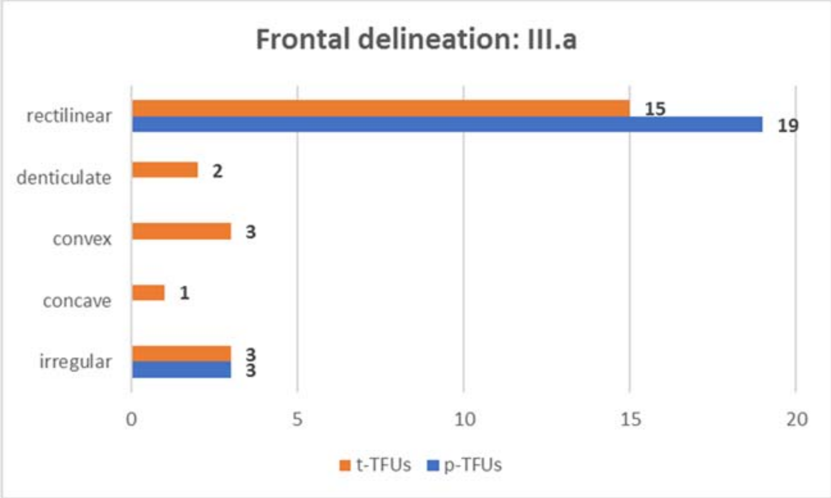

Fig. 6.36: *Frontal delineation of t-TFUs and p-TFUs of Sub-type III.a.*

6.5.4.1.2 Sub-type III.b

Sub-type III.b comprises only four artefacts and it is composed by a lateral t-TFU, opposed to an irregular edge. The basal part is always backed.

Angles of t-TFUs are comprised between 50° and 70° and cutting plane is always plane/plane (Table 6.15). Frontal delineation can be rectilinear, denticulate or convex (Fig. 6.37). p-TFUs are always proximal and lateral (the basal part and the edge opposed to the t-TFU). Angles are obtuse and cutting planes mostly plane/plane (Table 6.16).

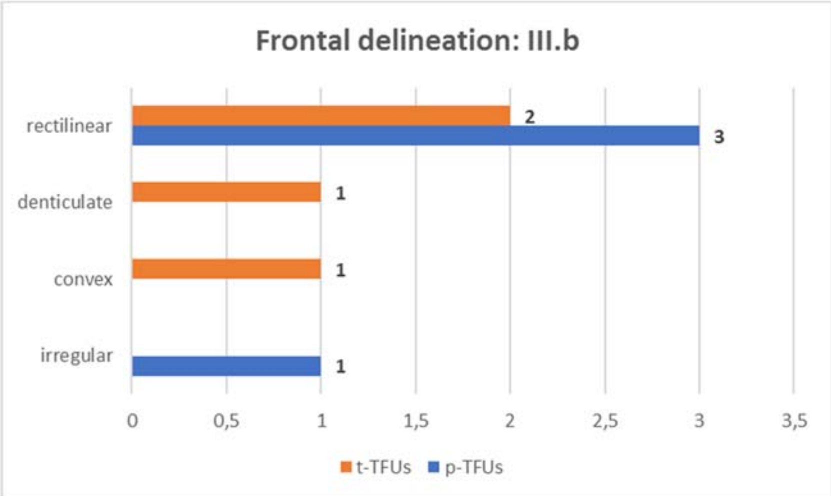

Fig. 6.37: *Frontal delineation of t-TFUs and p-TFUs of Sub-type III.b. Only proximal p-TFUs are imaged in this graph.*

6.5.4.1.3 Sub-type III.c

Three artefacts have been ascribed to sub-type III.c. t-TFUs are identified as the tip, while p-TFUs are the proximal edge plus one or both lateral edges (Fig. 6.38: 3, 5). Only the proximal p-TFUs are described due to the uncertainties related to the identification of the lateral ones. In fact, depending on the prehension mode, p-TFUs may change. They can comprise both proximal lateral edges or only one of them. Lateral edges are normally irregular, creating concavities near the distal part through retouch.

t-TFU Group III.				
Sub-type		**Description**	**Number**	**Tot.**
III.a	**Angle**	50°<x<70°	24	**24***
	Cutting plane	plane/plane plane/concave concave/plane	13 6 5	**24***
III.b	**Angle**	30°<x<50°	4	**4**
	Cutting plane	plane/plane	4	**4**

Table 6.15: *Technical characters of the transformative-TFUs of the artefacts pertaining to the sub-types of Group III.*

p-TFU Group III.				
Sub-type		**Description**	**Number**	**Tot.**
III.a	**Angle**	50°≤x<70° ≥70°	4 8	**12**
	Cutting plane	plane/plane concave/plane concave/plane	2 7 3	**12**
III.b	**Location**	proximal and lateral	4	**4**
	Angle	≥70°	4	**4**
	Cutting plane	plane/plane concave/plane	5 1	**4**
III.c	**Location**	proximal	3	**3***
	Angle	50°≤x<70°	3	**3**
	Cutting plane	plane/plane	3	**3**

Table 6.16: *Technical characters of the prehensile-TFUs of the artefacts pertaining to the sub-types of Group III. * Only the proximal p-TFUs are described.*

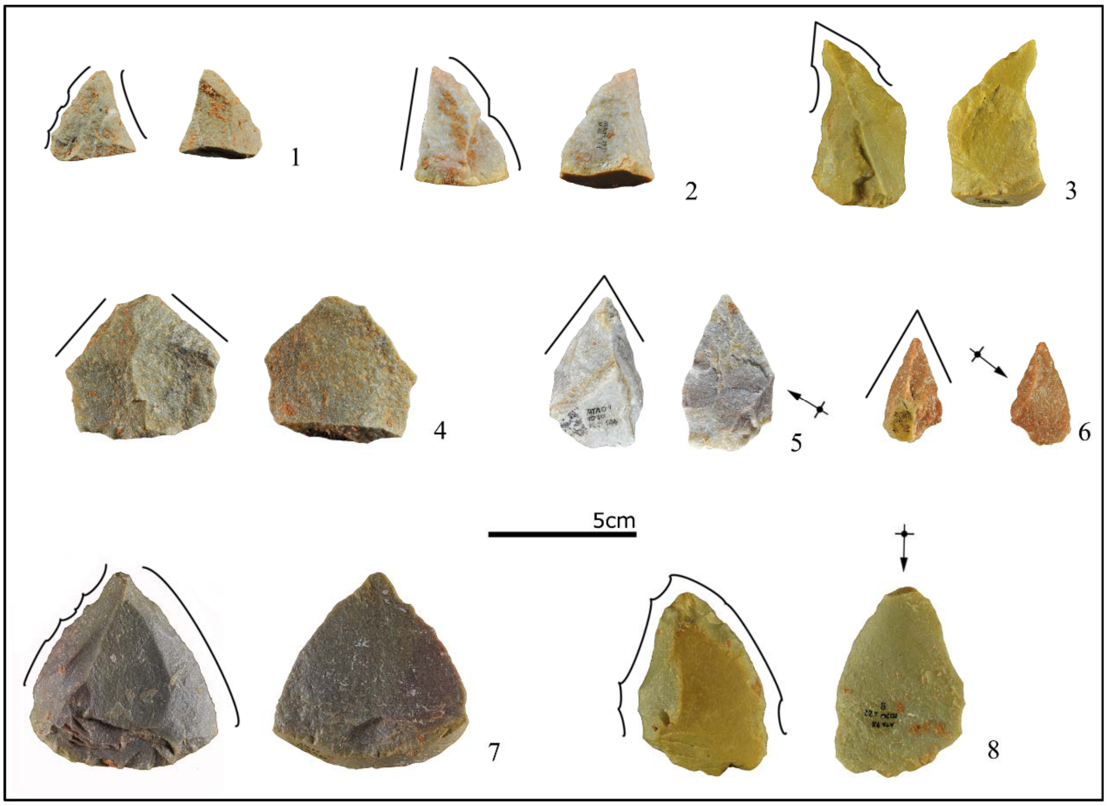

Fig. 6.38: *Some of the archaeological artefacts ascribed to the techno-functional Group III. (sub-type III.a: 1, 2, 4, 6, 7, 8; III.c: 3, 5).*

6.5.5 Summary of the techno-functional analysis results

The three techno-functional groups identified after the performance of the techno-functional analysis are compared in this section to discuss the implications of such organisation of the flake assemblage.

Techno-functional group I. is the most abundant with 91 specimens, while groups II. and III. present equal values (n=19) (Fig. 6.39). The five sub-types identified (I.a to I.e) have rectangular or half-moon morphologies and they only have one t-TFU, which may have different frontal delineations. Retouched edges are mostly present in I.c and Id. types, while the others are mainly composed of un-retouched edges. They always present a backed edge, opposed to the t-TFU, which is thought to be the p-TFU. Interestingly enough, when the p-TFU frontal delineation is convex (sub-types I.a, I.d, I.e), it is often cortical (Table 6.17). Corticality may be a very important factor for the constitution of the p-TFUs. The presence of cortex surely facilitates the grip, as cortical edges are less sharp than non-cortical ones and often present high angles. The artefacts pertaining to these types were produced during early phases of lithic production, and probably by taking advantage of the natural convexities of cobbles. This resulted in highly standardised structures, which were probably one of the main objectives pursued. Since diverse t-TFUs can be associated to this structure, it is understandable that at different edge characteristics, would correspond different capacities of incising matter (and so, different functions).

Techno-functional sub-type	p-TFU cortical	p-TFU not cortical	t-TFU retouched	t-TFU not retouched	p-TFU retouched
I.a	18	-	4	14	1
	-	12	4	8	-
I.b	2	-	-	2	-
	-	16	8	8	-
I.c	4	-	3	1	
	-	18	18	-	-
I.d	11	-	11	-	-
	-	4	3	1	-
I.e	3	-	2	1	-
	-	3	1	2	-
II.a	-	5	1	4	-
II.b	-	12	5	7	-
II.c	-	2	-	2	-
III.a	2	-	2	-	-
	-	10	5	5	-
III.b	-	4	2	2	-
III.c	3	-	3	-	-

Table 6.17: *Presence/absence of cortex and retouch on the p-TFUs and t-TFUs.*

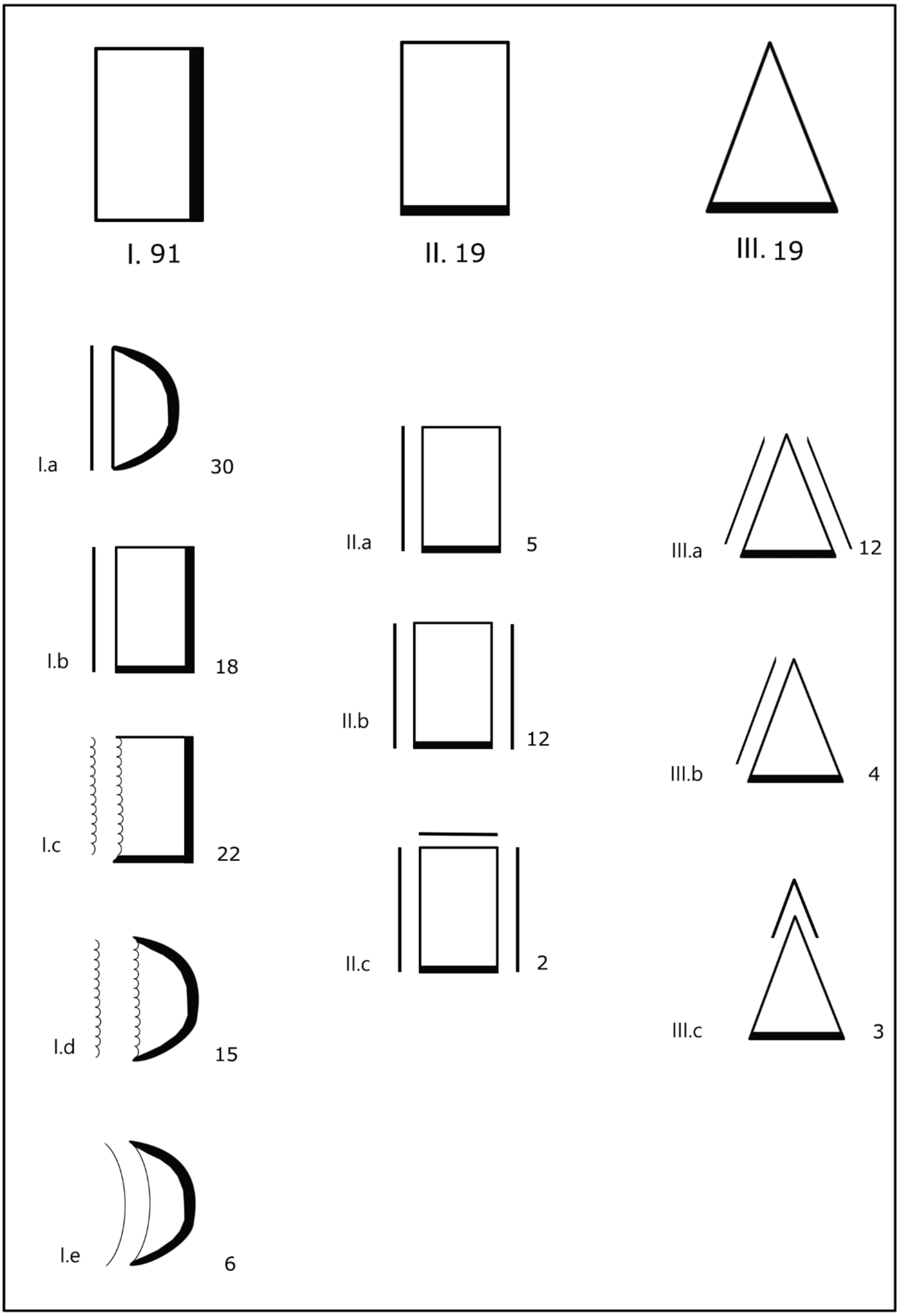

Fig. 6.39: *Schematic illustration of the three techno-functional groups and sub-types and relative number of artefacts. Edges in bold represent backed edges.*

Retouch is applied whenever a change in the technical characters (angle, frontal or sagittal delineations) of an edge is desired. We see that whenever denticulate frontal delineations are present (sub-types I.c, I.d), retouch was often applied, while the obtaining of other delineations (rectilinear, convex) did not always required a secondary modification of edges (Table. 6.17). Moreover, t-TFUs units were parallel to the technological axis in most of the cases, and only on 9 artefacts this does not occur (Table 6.18). In one case, retouch was applied on the distal part of a backed edge, probably to adjust the gripping mode.

The cutting edges of sub-type I.a are normally found on one of the lateral edges (n=22) and are then parallel to the technological axis of the pieces. However, they are found on distal (n=7) and proximal (n=1) edges of a few pieces (Table 6.18).

Regarding sub-type I.b, only in two cases the t-TFU is found on the distal end of the tools, while in most of the cases it is located either on the lateral right or left edge (Table 6.19).

The t-TFUs of sub-type I.c are mostly lateral (parallel to the technological axis) (n=14), and

4 t-TFUs of sub-type I.d are found on the distal edge, while the others 11 are all lateral (Table 6.19). On 4 artefacts pertaining to sub-type I.e, the t-TFU is found on a lateral edge, while on 2 of them it is located on the distal one.

Regarding the second techno-functional type (II.), edges were retouched on *ca.* half of the artefacts (Table. 6.17). Considering that more than one t-TFUs may be present (sub-types II.b, II.c), we see that retouch has a minor impact on this group. t-TFUs are mostly lateral and parallel to the technological axis of artefacts (Table 6.19).

We have a similar situation for the third group (III.). Large cortical surfaces are not present and retouch is present on the half of the artefacts. Retouch can be applied to create a more regular edge (by changing angle and delineations). A clear significance of retouch is found in sub-type III.c, where all the lateral edges (at least partially) are retouched (Table 6.17). The retouch here aims at creating a trihedral by modifying the lateral portions of the edges directly found below the convergence of the same edges.

Techno-functional sub-type	Location	Number	Tot. numb.
I.a	proximal	1	30
	distal	7	
	lat. right	10	
	lat. left	12	
I.b	proximal	0	18
	distal	2	
	lat. right	9	
	lat. left	7	
I.c	proximal	1	22
	distal	7	
	lat. right	4	
	lat. left	10	
I.d	distal	4	15
	lat. right	3	
	lat. left	8	
I.e	proximal	0	6
	distal	2	
	lat. right	0	
	lat. left	4	
II.a	proximal	0	5
	distal	1	
	lat. right	1	
	lat. left	3	
II.b	proximal	1	24*
	distal	1	
	lat. right	11	
	lat. left	11	
II.c	proximal	0	6*
	distal	2	
	lat. right	2	
	lat. left	2	
III.a	proximal	2	24*
	distal	2	
	lat. right	10	
	lat. left	10	
III.b	lat. right	2	4
	lat. left	2	
III.c	distal trihedral	3	

Table 6.18: *Number of t-TFUs and their location on the artefacts.*
** The number of t-TFUs does not coincide with the number of artefacts because in these cases there are multiple t-TFUs on the same artefact.*

6.6 Use-wear Analysis of the GD-TD10.1 quartzite artefacts

After having technologically analysed the sample of quartzite artefacts bigger than 20mm, a selection for use-wear analysis was made. Fifty-one artefacts were analysed with the methodology presented in this thesis. Nine of them had not previously ascribed to any techno-functional type. Out of these, two of them were handaxe-like tools and were not technologically analysed (as we selected only flakes and retouched flakes). Hence, the rest of the analysed material, being 42 pieces, are assigned to one of the three previously defined techno-functional groups.

Regarding the significance of the sample, we provide a distribution graph based on length and width of the implements (Fig. 6.40). Considering the entire quartzite assemblage of unit TD10.1, one can see that the samples analysed are representative of the general dimensional features of the quartzite implements.

The sample analysed represents around 3% of the entire products (non-retouched and retouched flakes, n= *ca.* 1700), but, when consider only the products larger that 20mm, the percentage is higher (9,8%, out of 519 implements). Therefore, these 519 implements are considered as the material on which the application of use-wear analysis may provide positive results. Because of that, the percentages discussed below will take as a reference this selected sample and not the entire quartzite assemblage, comprised of cores, natural bases, fragments and indeterminate fragments (n=3,608).

Table 6.19 shows the implements analysed sorted into different technological categories. More than 50% of the specimens analysed are retouched flakes, while around 37% is composed of unmodified flakes. A minor percentage (3,9%) relates to large tools, namely a handaxe-like tool and a cleaver-like one.

Technological category	Number of pieces	%
Non-retouched pieces	19	37,2%
Retouched pieces	30	58,8%
Large tools	2	3,9%
TOT	51	100%

Table 6.19: *The sample analysed from GD-TD10.1, sorted into different technological categories.*

In the following paragraphs, the modifications due to use as well as the micro-residues found on the sample analysed will be described in detail. After presenting the functional results of the 51 implements in detail, a last paragraph will be dedicated to the considerations arising from the combined application of techno-functional and use-wear analyses. In other words, functional data will be compared with the previous techno-functional ascriptions and parallels or discordances will be highlighted.

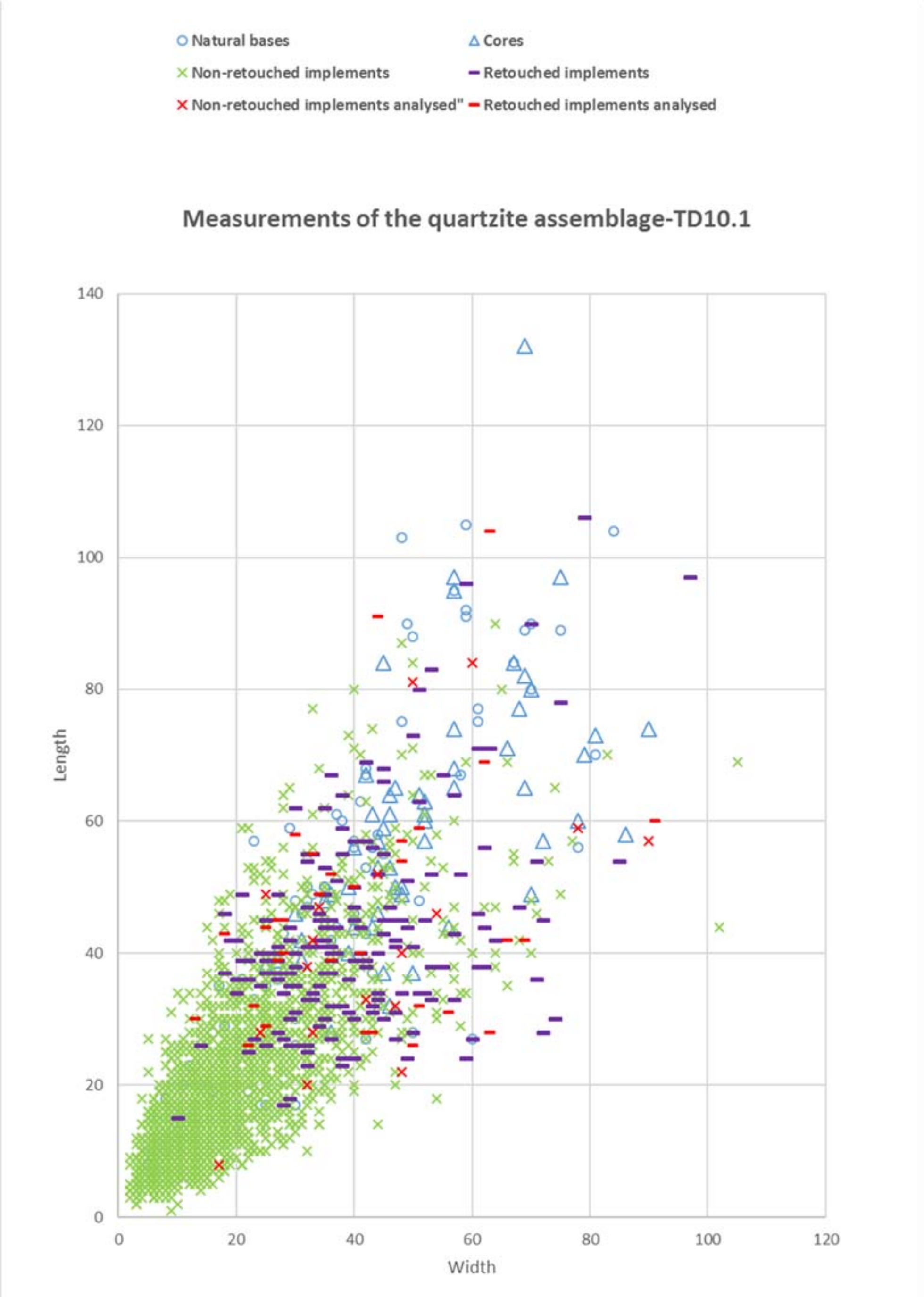

Fig. 6.40: *Distribution through length and width of the quartzite implements of the TD10.1 assemblage. In red, the samples analysed. Note their dimensional significance with respect to all assemblage.*

6.6.1 Analysis of residues

The samples were systematically submitted to a first microscopic screening with the aid of the metallographic microscope. This first observation had as a major aim the detection of the possibly present micro-residues and because of that, it was done previously to any cleaning.

On 7 samples (ATA00-TD10-L12-11, ATA03-TD10-J20 114, ATA03-TD10-N22 42, ATA03-TD10-L20-868, ATA04-TD10-L22-152, ATA04-TD10-I20-82) relatively big white residues, possibly linked to bone, were observed. The residues observed were systematically imaged with optical devises before cleaning the samples prior to the application of use-wear analysis (Fig. 6.41). Although it was necessary to resort to microscopic observation to detect residues, in some cases residue accumulations were big enough to be seen to the naked eye (Fig. 6.42).

To achieve a more secure attestation of the residues, a number of samples were analysed with a SEM under low vacuum conditions and EDX was applied in order to detect their elemental composition (Fig. 6.43; 6.44). Low-Vacuum conditions are preferable, as the samples do not need any covering. The covering materials (carbon or gold) would interfere with the EDX analysis.

The performance of the elemental mapping, which visually locates each element on the SEM images, allows to correlate the elements detected by the EDX analysis with the correspondent residue (Fig. 6.45: b; 6.37: b). In this way, it is possible to demonstrate the combined presence of different elements characterising a specific substance on the same spot (such as Phosphorus and Calcium in the case of bone). The contrast with other elements not present on the residues observed (such as the major elements of the rock substrate, as Silicon) allows to better visualise the results of the EDX analysis (Fig. 6.45: b).

Even if it was very tempting to relate the presence of small bone fragments on our specimens to specific activities, we have been very cautious because of several reasons. First, we are aware that the sole presence of residues is not enough to infer specific functions. Second, the residues observed were not organised in recurrent distributional patterns, they were not always on the very edge of the implements analysed and they were always embedded in sediment concretions. All of these factors contributed to our decision of not proposing clear correlations between these quartzite artefacts with the processing of bone.

Besides, we need also to consider the specific conditions of the excavation. When excavating the bottom of TD10.1 unit, bones were everywhere and bone fragments were mixed with sediments, often being attached to the lithics. This archaeo-level was actually named after this fact (*bone bed*). Knowing this, we understand that the observed residues are more likely to be connected to the specific formation processes of the layer, than to anthropogenic activities carried out at the site.

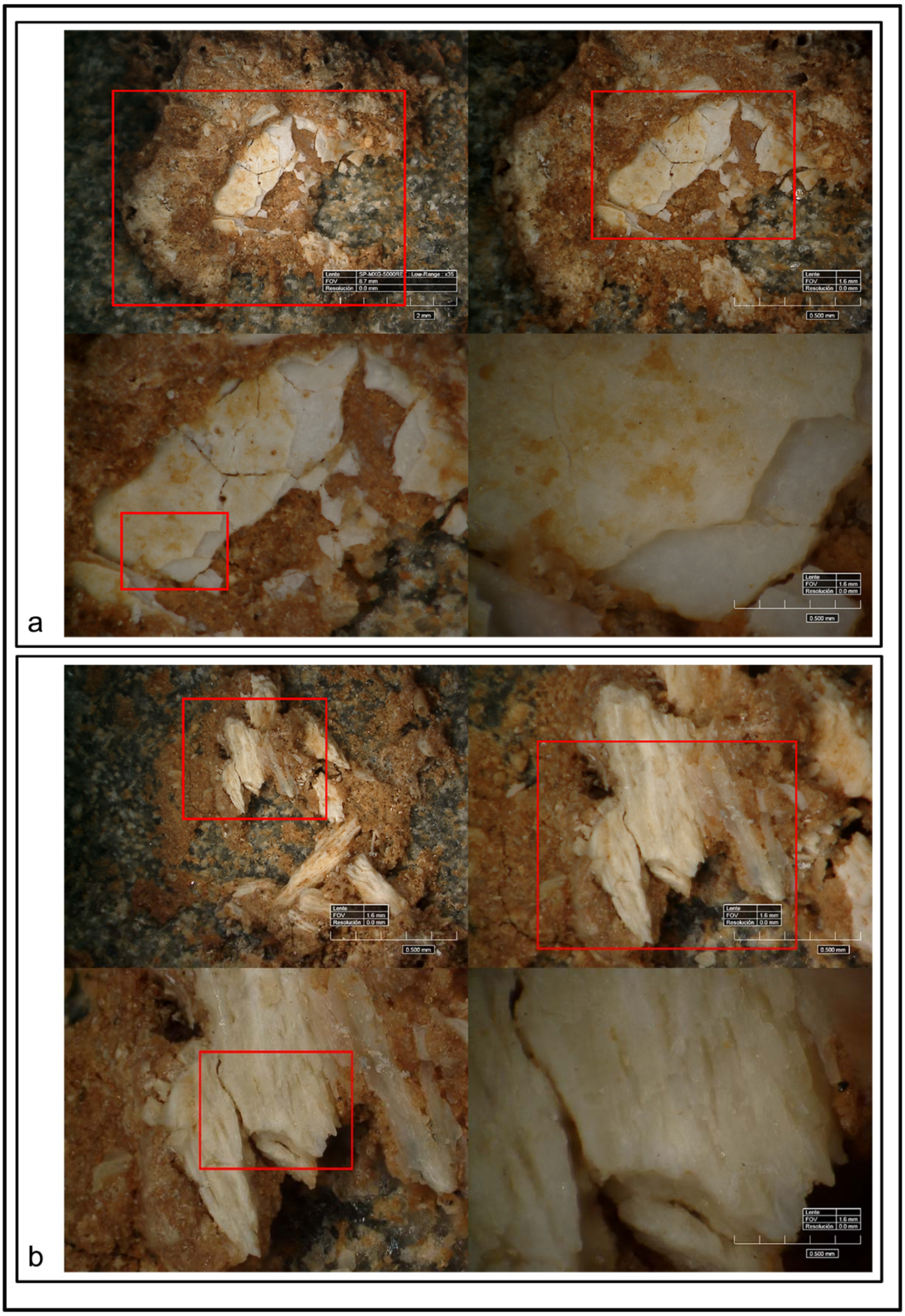

Fig. 6.41: *Mosaics of two bone residues observed on the same quartzite flake (ATA04-TD10-L22-152) being embedded in the sediment concretion. Both residues are imaged at different magnifications: red squares indicate the portion of the image magnified in the following one. (Hirox-Low range lens).*

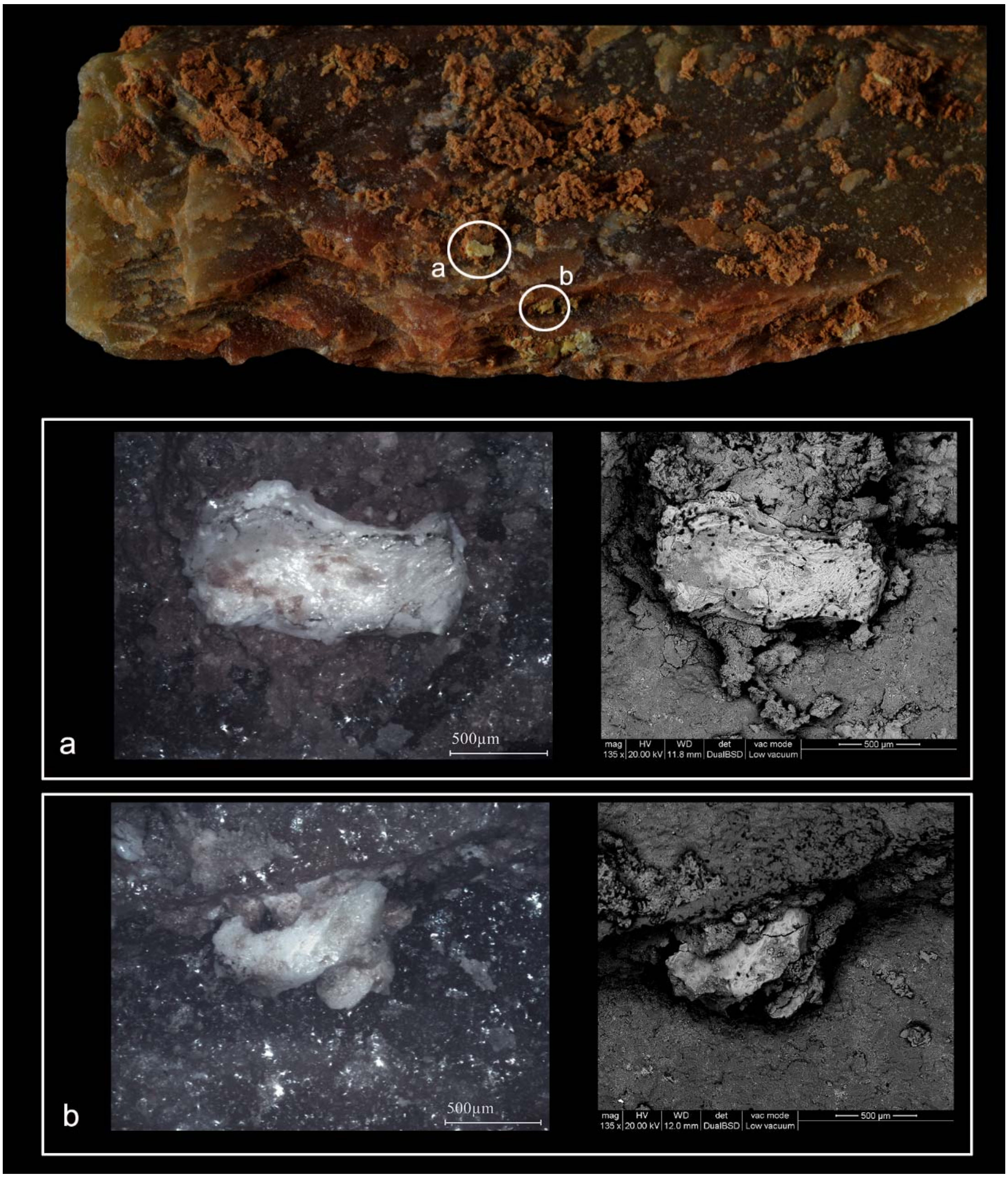

Fig. 6.42: *Dorsal face of ATA04-TD10-I20-82 prior to cleaning. Macro-residues of bone are visible in the photograph (panorama of three photographs taken with a NIKON600 camera; Lens: MACRO 440mm). a, b) Examples of the systematic use of OLM and SEM to compare the same residues' portions. Note the presence of skin flakes (black dots) on both the residues and concretions visible in the SEM images and being invisible under the optical microscope. (Optical images: mag.= 5x, scale bar= 500µm, multi-focused image composed of 42 slides; SEM-LV-Back-scattered electron detector images: mag. = 135x, scale bar= 500µm).*

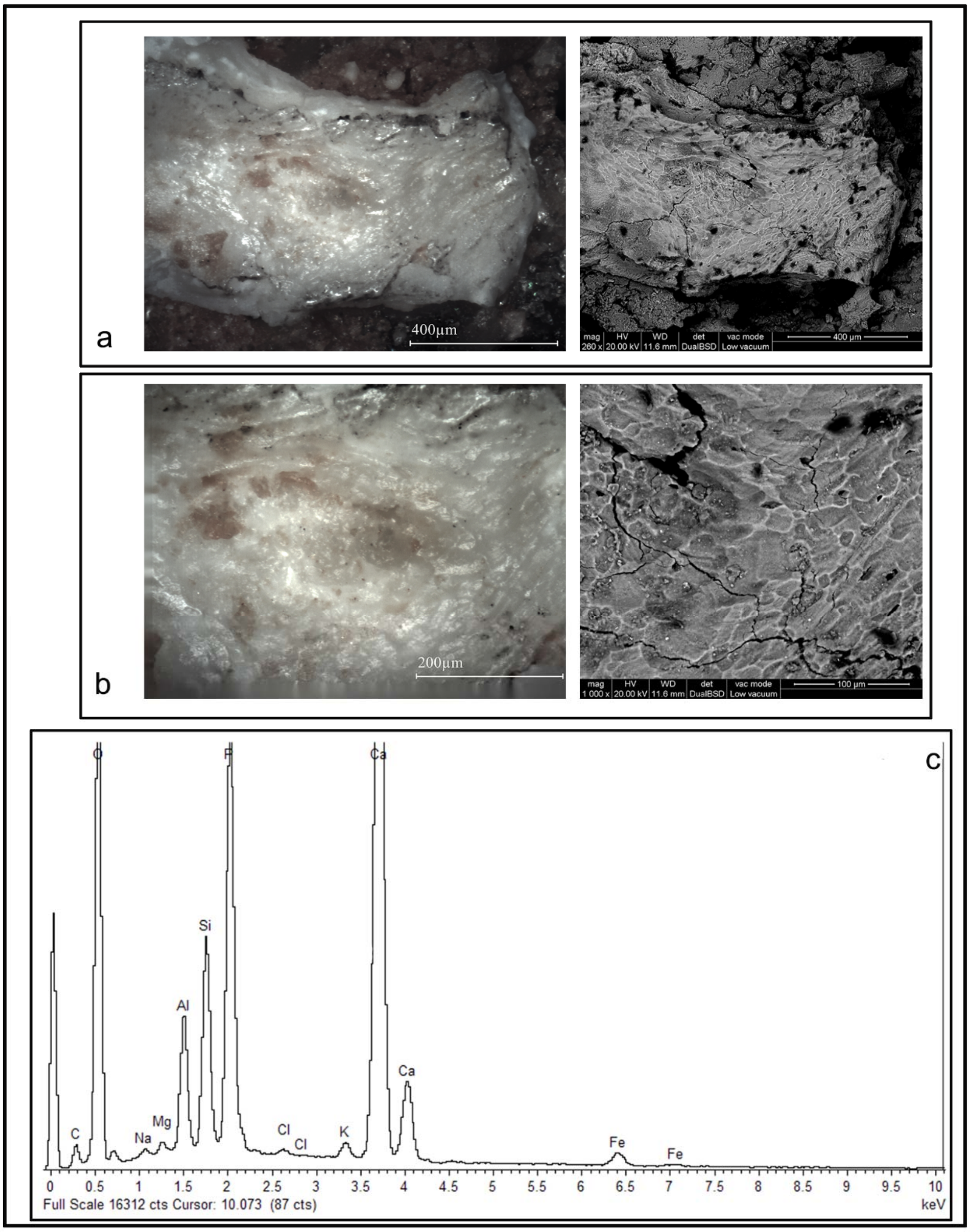

Fig. 6.43: *Details of the bone residue "a" in Figure 6.42. a) OLM picture: mag.= 10x, scale bar= 400µm, multi-focus image composed of 26 slides; SEM-LV, Back-scattered electron detector image: mag.= 200x, scale bar= 400µm; b) OLM picture: mag. 20x, scale bar 200µm, multi-focus image composed of 15 slides; SEM-LV, Back-scattered electron detector image: mag.= 1000x, scale bar= 100µm; c) EDX spectrum of the residue taken from a spot on the SEM micrograph b: Carbon, Oxygen, Potassium and Calcium are related to the bone composition.*

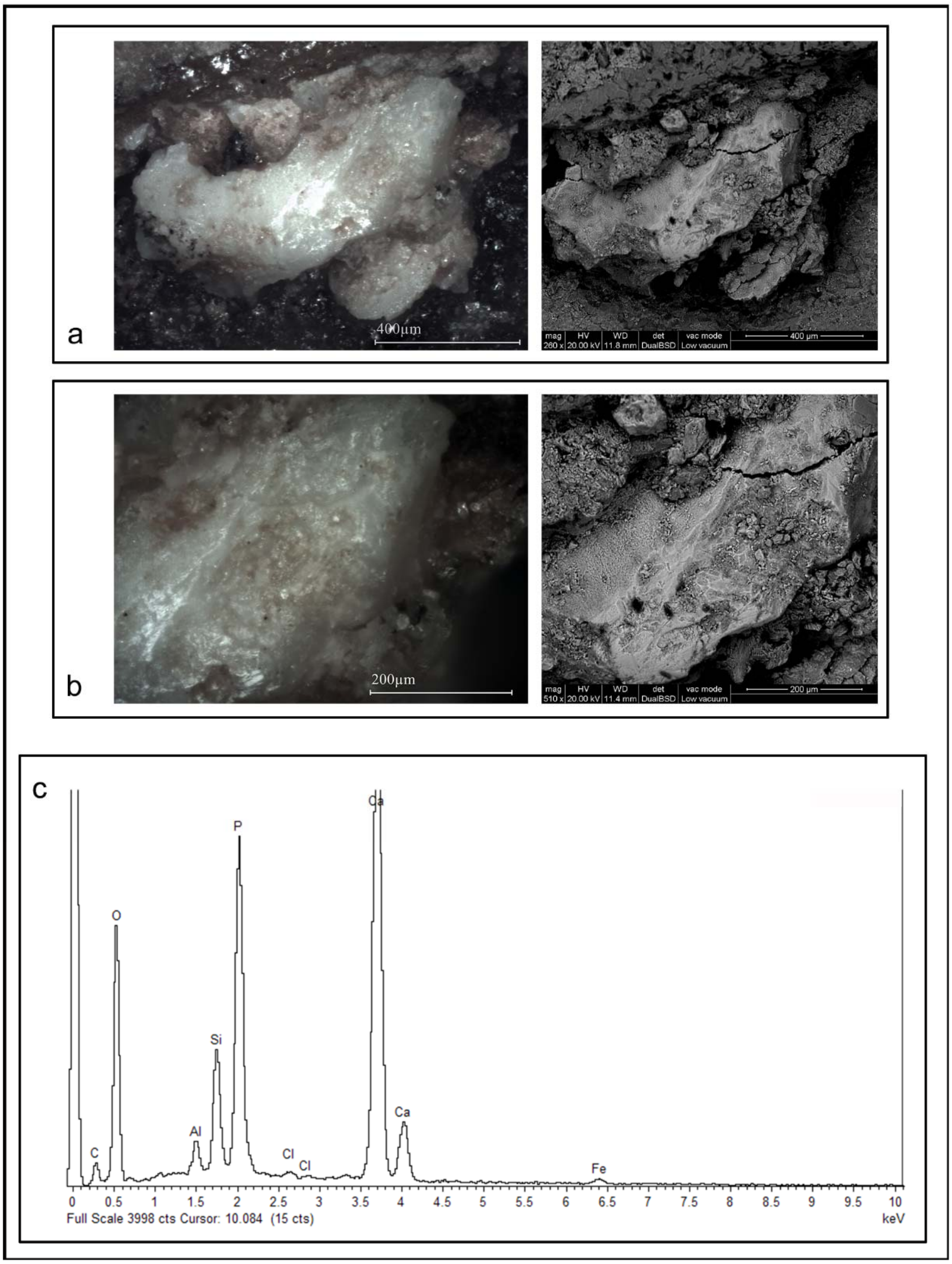

Fig. 6.44: *Details of the bone residue "b" in Figure 6.42. a) OLM picture: mag.= 10x, scale bar= 400µm, multi-focus image composed of 34 slides; SEM-LV, Back-scattered electron detector image: mag.= 200x, scale bar= 400µm; b) OLM picture: mag.= 20x, scale bar= 200µm, multi-focus image composed of 22 slides; SEM-LV, Back-scattered electron detector image: mag.= 510x, scale bar= 200µm; c) EDX spectrum of the residue taken from a spot on the SEM micrograph b: Carbon, Oxygen, Phosphorus and Calcium are related to the bone composition.*

While in most of the cases it is very difficult to infer functions based on a few number of micro-residues, in others it seems to be more feasible. A bone residue was found very near the edge of a quartzite side-scraper (ATA04-TD10-I20-82) and was not embedded in the sediment nor it was underneath it (Fig. 6.45: a). This residue appeared to be smashed onto the surface, resembling the experimental residues found on flakes having been retouched with bone hammers (Fig. 6.46: a). In both cases (archaeological and experimental), the EDX analysis correlated Calcium and Phosphorus to the white smashed residue (Fig. 6.45: b; 6.46: b).

Experiments were conducted with the main aim of understanding whether residues from an organic hammer used to retouch flakes are trapped onto the surface of the lithics or not. Moreover, if it this happens, their disposition on the edges was important to be recorded. Lithic residues on the bone hammers were also found on at least one of the bone retouchers identified in TD10.1 (Rodríguez-Hidalgo et al., 2013, in preparation). Bone hammers in different states (fresh, defatted and dry) were used to retouch both flint and quartzite flakes.

The retouching process was carried out through different gestures: different knapping angles were associated with different blow intensities (weak and strong) and different hand used (right or left) (Rodríguez-Hidalgo et al., 2013; in preparation). The whole experiment aimed at the reproduction of a type of retouch present in the TD10 lithic assemblage by using bone retouchers. This specific retouch is present on side-scrapers, it is quite abrupt, scaled and generally made of different generations (two or three) (Fig. 6.48).

Residues of bone hammers on flint and quartzite edges were compared and first assumptions were done, though results have not been completely revised yet (Fig. 6.47). Fresh and defatted hammers leave greasy residues on the surfaces of the lithics, while dry hammers apparently do not leave any residue. Due to the preliminary character of these observations, results here are not conclusive. The organic particles appear darker than the inorganic ones under the SEM-backscattered electron detector (Fig. 6.47: a, c, d). The presence of the main components of hydroxyapatite are inferred by the EDX element maps and spectrum (Fig. 6.47: b, e), both used for comparison with the residues observed on archaeological samples.

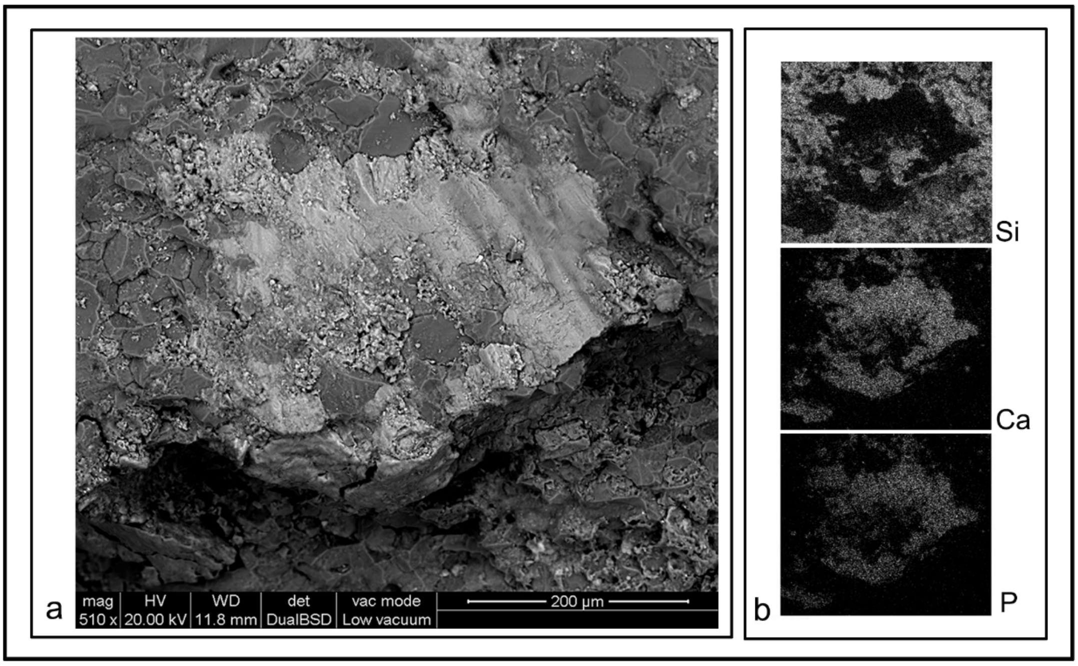

Fig. 6.45: *Archaeological bone residue observed near the rim (ATA04-TD10-I20-82). a) SEM-LV, back-scattered electron detector image (mag. 510x, scale bar 200µm). The appearance of this residue contrasts with that of the residues embedded in the sediment. This residue is smashed upon the surface, on a ridge created by the retouch. b) SEM-EDX, elemental map showing the combined presence of Ca and P in the residue (in white), contrasting with the Si of the rock substrate. The relative amount of the elements is visualised in a light colour, while the absence of the same elements results in darker colour.*

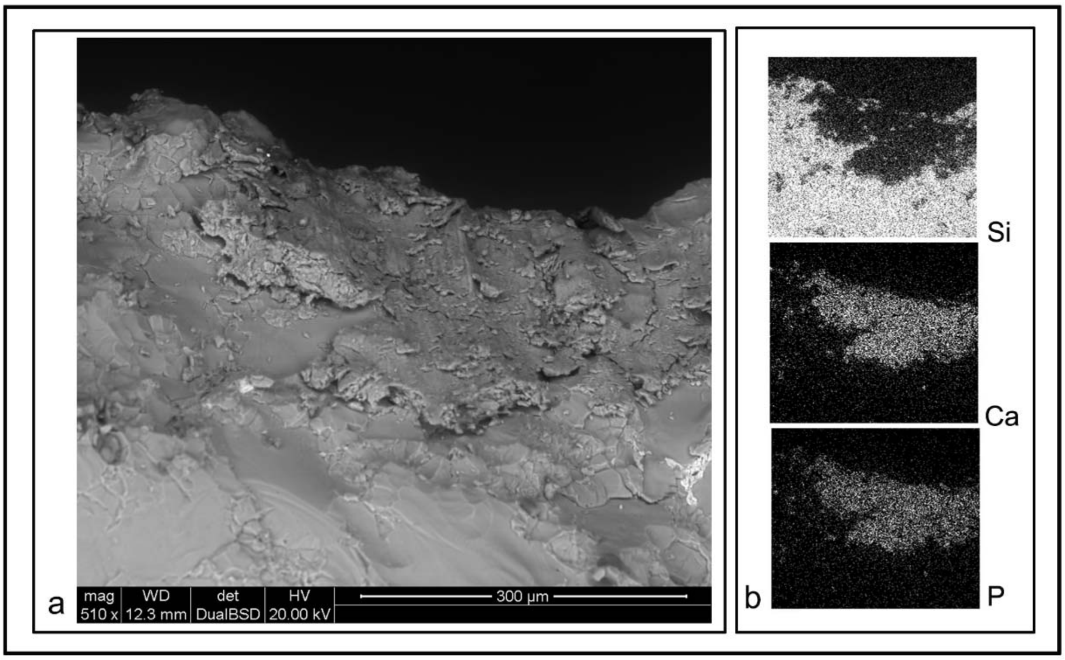

Fig. 6.46: *Experimental bone residue on a quartzite flake whose edge was retouched by a fresh-bone hammer (BRF-6). a) SEM-LV, back-scattered electron detector image (mag. 510x, scale bar 300µm). b) SEM-EDX, elemental map showing the combined presence of Ca and P in the residue (in white), contrasting with the Si of the rock substrate. The relative amount of the elements is visualised in a light colour, while the absence of the same elements results in darker colour.*

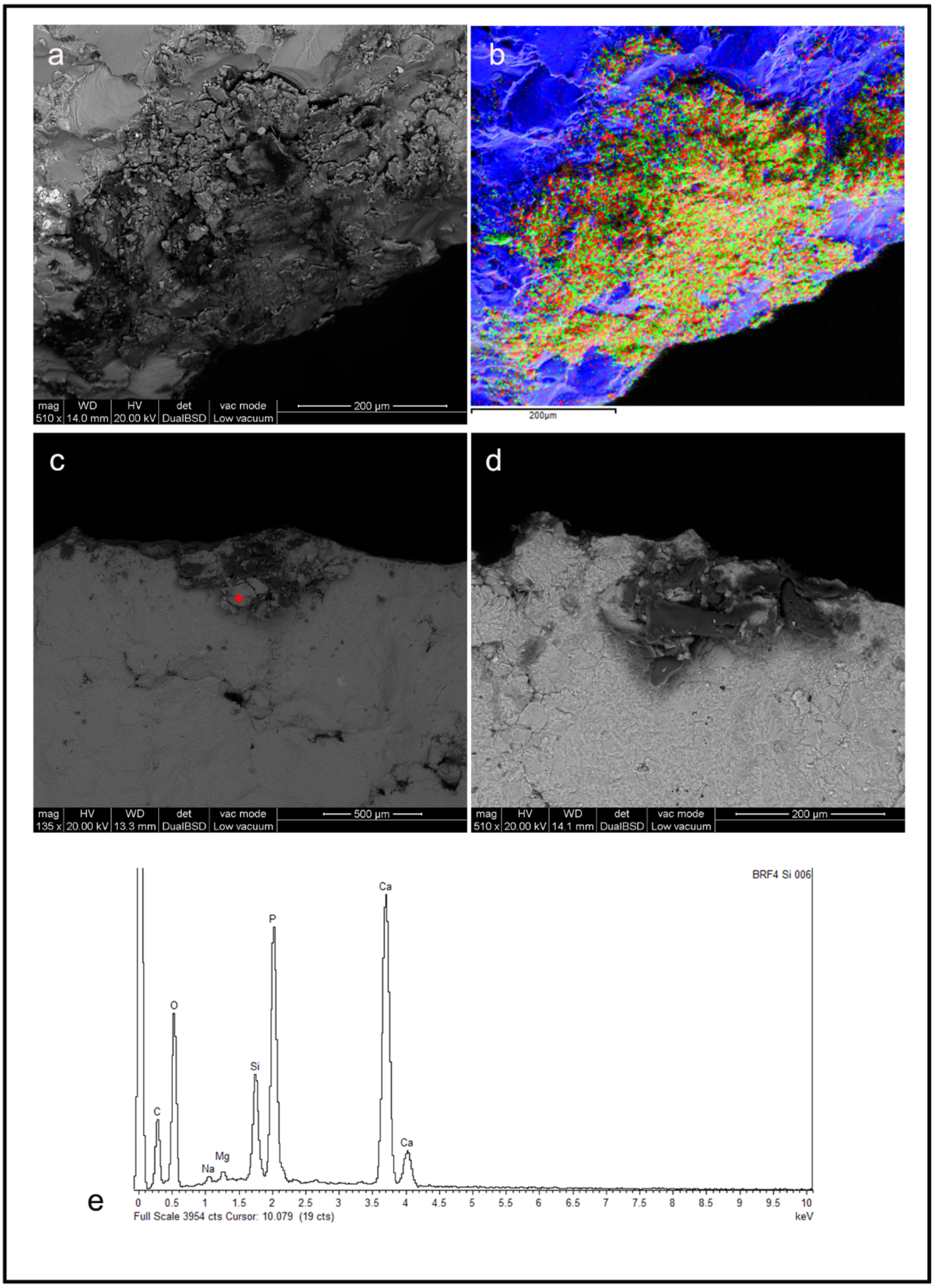

Fig. 6.47: *Bone residues on experimental quartzite (a, b) and flint (c, d) flakes. The residues originated by the contact with bone hammers during the retouching of the edges. In both cases the state of the bone retoucher was fresh. This is why the amount of greasy matter is considerable. Both the residues maps (b) and spectra (e) have been systematically used to characterise bone residues. On the residue map (b), blue colour is linked to the Silicon content, while green and red stand for Calcium and Potassium respectively. The Spectrum e is taken on the micrograph c (red dot).*

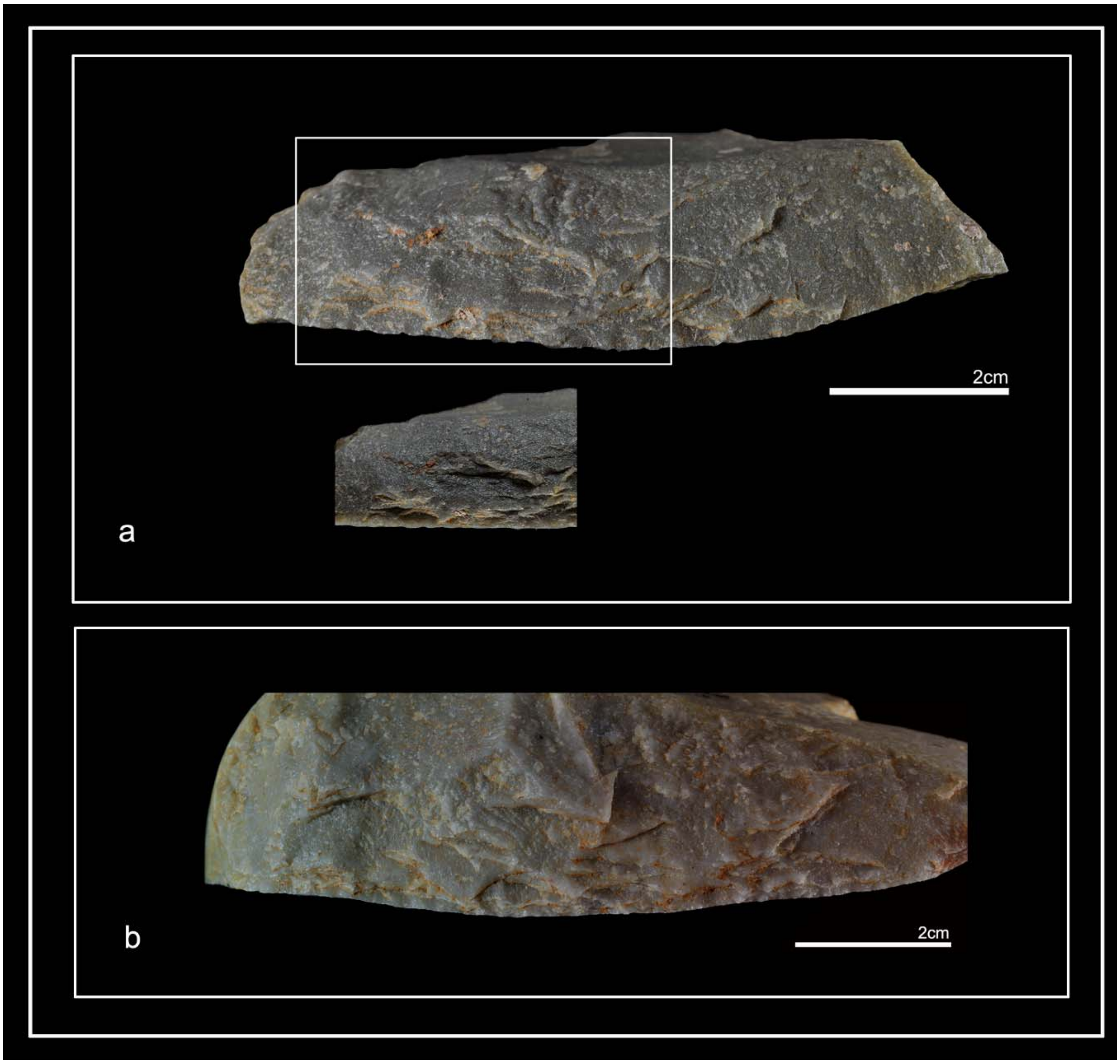

Fig. 6.48: *Macro-photographs of the retouch type taken as a reference in the experiments described in Rodríguez-Hidalgo et al., 2013 and in preparation. Two archaeological side-scrapers presenting an abrupt, scaled retouch, made of several generations: a) ATA00-TD10-N20-66. The portion included in the white square is showed below the general photograph and it reproduces its sagittal view to better appreciate the stepped-scaled character of the residue. b) ATA01-TD10-N14-320. Both photographs are panoramas obtained through sticking three different photographs (camera: NIKON600, lens: MACRO 440mm).*

6.6.2 Microscopic wear and its attribution

Use-wear analysis provided functional results on 68,6% (n=35) of the sample analysed (Tables 6.20, 6.21). For 16 implements, no functional interpretation is available. On 13 of them post-depositional modifications are present, although to different degrees (from low to very high).

The level of identification varies a lot, depending on the preservation of wear and of its degree of development. Sometimes, only the used portion of the edge is identified, while no additional information about the action is available. In other cases, the kinematics is quite clear due to a relatively large presence of linear indicators. The identification of the worked materials, as in most studies, is more challenging as many details are missing on archaeological specimens. Therefore, in most of the cases, the interpretation of the worked materials falls into broad categories of relative hardness (soft, medium, hard, very hard). In few cases, however, the presence of diagnostic characters of wear allowed more in-depth identifications of the worked material's type (Table 6.21).

Functional results on different technological categories							
Technological category	***Use-wear***	***%***	***Wear non-related to use***	***%***	***Fresh surfaces***	***%***	***Tot.***
Non-retouched pieces	12	23,5	6	11,8	1	1,9	19
Retouched pieces	22	43,1	6	11,8	2	3,9	30
Large tools	1	1,9	1	1,9	-		2
Total	35		13		3		**51**

Table 6.20: *Summary of the number and percentages of the artefacts analysed, displaying or not use-wear. Percentages are calculated considering the entire sample analysed (51 pieces).*

Num.	Reference	Retouched	Position used edge	Angle	Movement	Action	Material hardness	Material type	PDSM	Observations
1	ATA98 TD10 L22 8	yes	lat. left	60°	longitudinal	-	-	-	low	re-sharpening
2	ATA99 TD10 I11 107	yes	lat. left	60°	transversal	-	-	-	absent	REM_1.3 (refits)
3	ATA99 TD10 I15 92	yes	lat. left	55°	longitudinal	sawing	hard	-	low-absent?	hafting traces
4	ATA00 TD10 J16 183	no	lat. left	45°	longitudinal	-	soft	animal matter	absent	
5	ATA00 TD10 N13 71	yes	lat. left	65°	transversal	scraping	very hard	bone	absent	
6	ATA00 TD10 N15 121	yes	lat. left	70°	transversal	scraping	hard	wood	absent	
7	ATA00 TD10 N13 46	yes	lat. left	60°	transversal	scraping	hard	wood	absent	
8	ATA 00 TD10 N20 66	yes	lat. left	60°	transversal	scraping	soft	skin	low	possible hafting traces
9	ATA01 TD10 N21 251	yes	lat. left	60°<α<50°	transversal	-	-	-	absent	REM_3.21 (refits)
10	ATA01 TD10 N14 320	yes	lat. right; distal	90°<α<80° 60°<α<50°	longitudinal transversal	sawing chopping	hard	wood/bone	absent	
11	ATA01 TD10 L14 60	yes	lat. right	60°	transversal	whittling	hard	wood	absent	
12	ATA01 TD10 N16 190	no	lat. right	45°	longitudinal	-	-	-	low	
13	ATA01 TD10 K21 144	yes	lat. left	65°	transversal	-	-	-	absent	
14	ATA02 TD10 O20 248	yes	lat. right	65°	-	-	-	-	high	

15	ATA02 TD10 L13 77	yes	lat. left	60°	transversal	scraping	soft	skin	medium	
16	ATA02 TD10 L16 55	yes	distal	55°	longitudinal	sawing	hard	wood	absent	
17	ATA02 TD10 M22 520	no	distal	35°<α<40°	longitudinal	butchery	soft	meat, skin	absent	
18	ATA02 TD10 N22 20	no	lat. left	55°	longitudinal	butchery	soft	tendons	absent	
19	ATA02 TD10 O21 279	yes	distal	60°	longitudinal	-	-	-	absent	
20	ATA03 TD10 J10 63	yes	lat. left	65°	transversal	scraping	very hard	bone	absent	
21	ATA03 TD10 N22 42	no	lat. left	60°	longitudinal	cutting	soft	meat	absent	
22	ATA04 TD10 L22 738	no	lat. left, distal	55°, 55°	transversal	whittling	very hard	bone	absent	
23	ATA04-TD10-K21-132	yes	lat. left	40°	longitudinal	sawing	very hard	bone	low	Hafting traces.
24	ATA04 TD10 K22 206	yes	lat. left	55°	transversal	scraping	very hard	bone	low	
25	ATA04 TD10 L22 151	yes	lat. left	65°	longitudinal	sawing	hard	wood	low	
26	ATA04 TD10 L21 235	yes	lat. left	65°	transversal	scraping	soft	greasy matter, meat	absent	
27	ATA04 TD10 L22 152	no	lat. right	50°	longitudinal	-	hard	-	absent	
28	ATA04 TD10 N21 566	no	lat. left	45°	transversal	-	-	-	absent	
29	ATA04 TD10 M20 548	yes	lat. left	50°	longitudinal	cutting	soft	meat	low	
30	ATA04 TD10 N18 4	no	lat. right	-	rotational	boring-like	hard	-	absent	
31	ATA04 TD10 K21 68	yes	lat. right lat. left	70°, 80°	rotational	boring-like	hard	-	low	
32	ATA05 TD10 N20 97	no	lat. left	60°	transversal	whittling	-	-	very low	REM-1.3 (refits)

33	ATA05 TD10 L21 105	no	lat. right	45°	longitudinal	sawing	very hard	bone	absent
34	ATA05 TD10 M21 1158	yes	lat. left	50°	longitudinal	cutting/sawing	-	-	absent
35	ATA05 TD10 M21 273	yes	lat. right	65°	transversal	scraping	hard	wood	absent
36	ATA05 TD10 L22 323	no	lat. right	45°	transversal	scraping	hard	wood	absent

Table 6.21: *Use-wear results on the sample analysed. The location of use-wear, the type of movement, action and the worked material type, when known, are listed. The presence/absence of PDSM is also specified.*

6.6.2.1 Post-depositional surface modifications and technical wear

We opted for presenting the data related to post-depositional surface modifications (PDSM) and technical wear before giving additional details about the use-wear. In this way, we are able to first assess the surface preservation conditions of the sample analysed and also to understand the impact that PDSM had on the identification of use-related wear.

PDSM were documented with different degrees on 47,05% of the sample analysed (Table 6.22). Among these cases, on 13 artefacts it was not possible to propose any functional interpretation because of the high degree of PDSM as well as the absence of use-wear. On 11 artefacts (21,6%), despite the presence of PDSM, it was possible to differentiate clear traces due to use.

Therefore, 47,05% of the sample presents use-related wear and no PDSM (Table 6.23). 21,6% of the artefacts showed a combination of use-wear and PDSM, while on 25,5% only PDSM were documented. 5,9% of implements presented fresh surfaces.

Surfaces affected by PDSM presented characteristic traits, as randomly oriented, irregular and very deep striations (Fig. 6.49) or extremely polished areas (Fig. 6.50).

Post-depositional modifications			
Degree	***Number***	***Hinder use-wear identification***	***Allow use-wear identification***
Very low	2	1	1
Low	13	4	9
Medium	4	3	1
High	5	5	-
Total	24 *47,05%*	13 *25,5%*	11 *21,6%*

Table 6.22: *Degree of post-depositional modifications on the sample analysed and number of cases where they hindered or allowed the identification of use-wear. Percentages consider the total number of artefacts analysed (n=51).*

Combination of use-wear and PDSM		
Use-wear, no PDSM	24	*47,05%*
Use-wear + PDSM	11	*21,6%*
PDSM, no use-wear	13	*25,5%*
Fresh	3	*5,9%*
Total	51	*100%*

Table 6.23: *Combination of the presence/absence of use-wear and PDSM on the sample analysed.*

Irregular linear features can be very diverse: from deep furrows (Fig. 6.49: a, e, i) and grooves (Fig. 6.49: g, h), to partial Hertzian cones ((Fig. 6.49: b, c).

Polished areas are also indicative of PDSM when they are extremely developed, present on large areas with irregular patterns, or on areas far from the edges. Often, these polishes areas are accompanied by linear features, often grooves or sleeks (Fig. 6.50: a, b, c) or irregular scratches (Fig. 6.50: d).

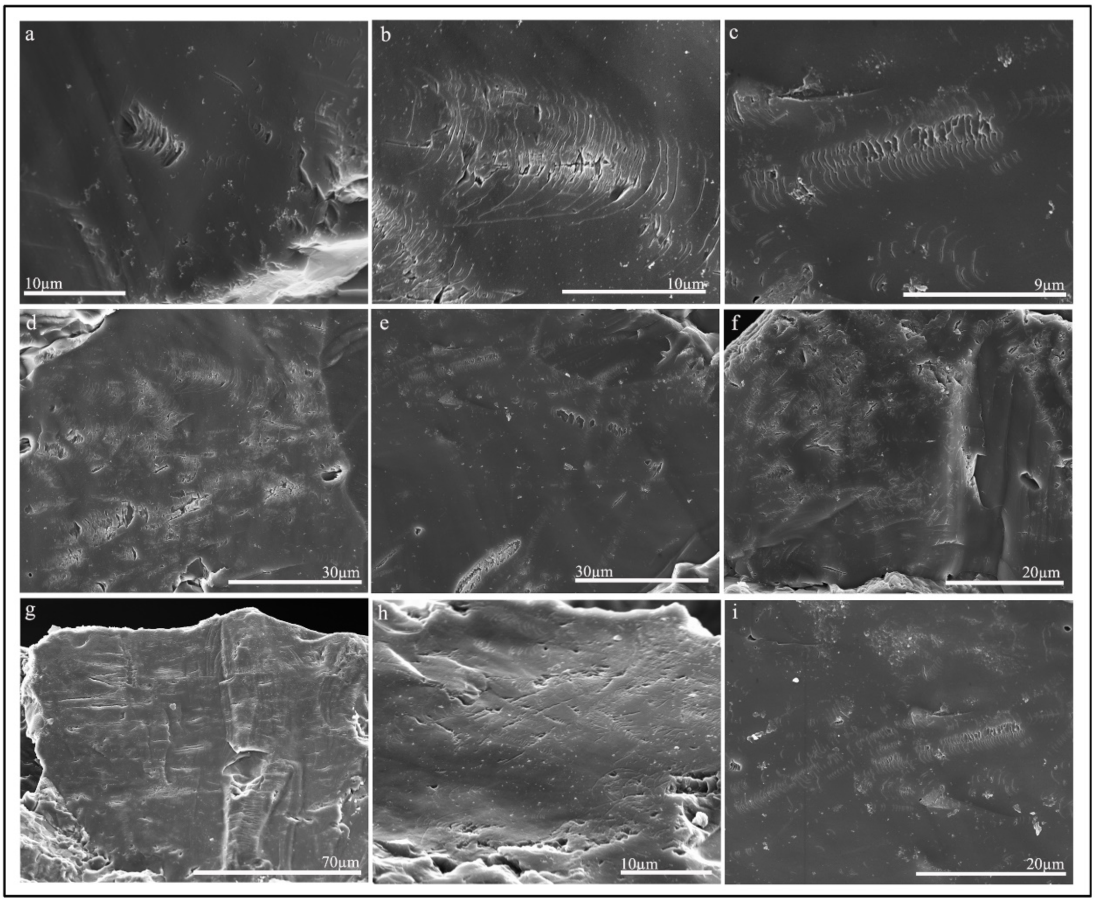

Fig. 6.49: *Examples of post-depositional surface modifications on archaeological samples. Different types of striations: a) irregular furrow; b) partial Hertzian cones; c) furrow and partial Hertzian cones; d) furrows and irregular scratches; e) furrows and scratches; f) furrows and grooves; g) furrows and grooves; h) grooves; i) furrows. Magnifications range between 800x and 3000x.*

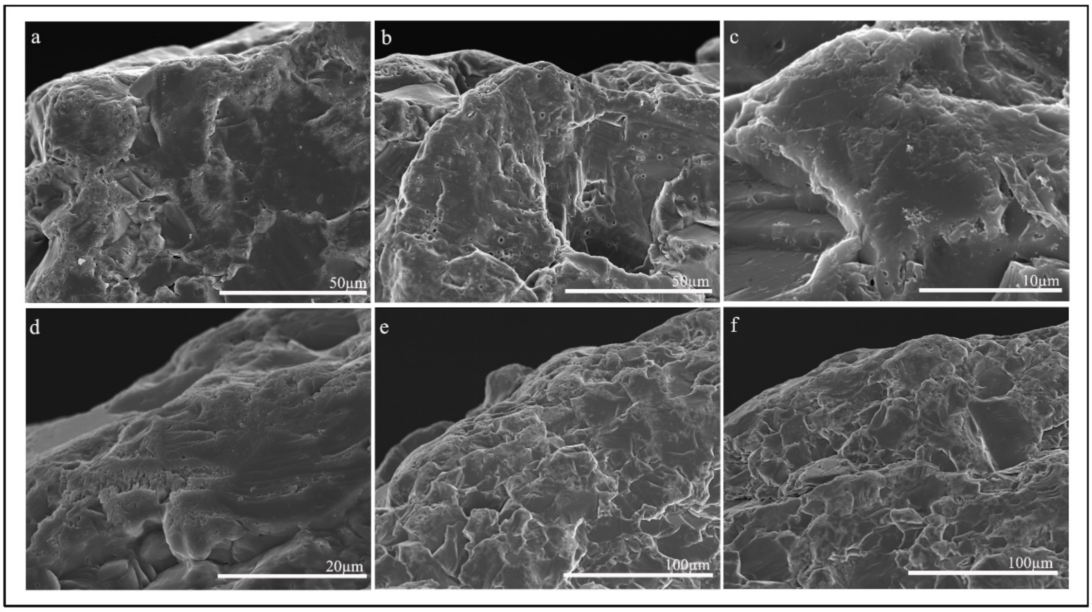

Fig. 6.50: *Examples of post-depositional surface modifications on archaeological samples. Extremely polished surfaces. Magnifications range between 500x and 2000x.*

For comparison, we analysed wear originated after tumbling experiments and found some analogies with the archaeological evidence. While there were no significant differences regarding the appearance of polished/abraded areas (Fig. 6.51), clear matches with the extremely irregular linear features found on some archaeological samples were not recorded. We need to remind that only two experimental artefacts subjected to tumbling experiments were microscopically analysed (Volume II, Annex 3), therefore this may not be enough to successfully assess the impact that soil movements have on quartzite surfaces. However, it is interesting that no linear features were observed after 20 and 30h of tumbling, especially considering the high amount of them on some archaeological samples. This may suggest that in some cases, soil movements conditions at Gran Dolina were more intense than what we reproduced at the laboratory. To better assess this, more experiments are needed.

Technical wear was also documented, although not systematically. By technical wear, we mean any macro or microscopic sign related to the production of stone tools. For instance, striations near the percussion point, crushing of the percussion platform, macro-fractures on retouched edges, incipient fractures on the very rim, etc…

Sometimes these technological features are easily observed and it is useful to document them microscopically to learn how not to confuse them with use-wear.

The most recurrent and more easily discernible technical wear on quartzite is the incipient fracture usually found on retouched edges, originated by the contact with a hammer used to retouch the edge. These fractures are reminders of blows aimed to retouch the edge, but which failed by not having enough force. They usually have a v-shaped outline, indicating where the blow was stroke (Fig. 6.52: 1-4). More rarely, tiny striations were observed near the impact point, on the ventral surface (Fig. 6.52: 5).

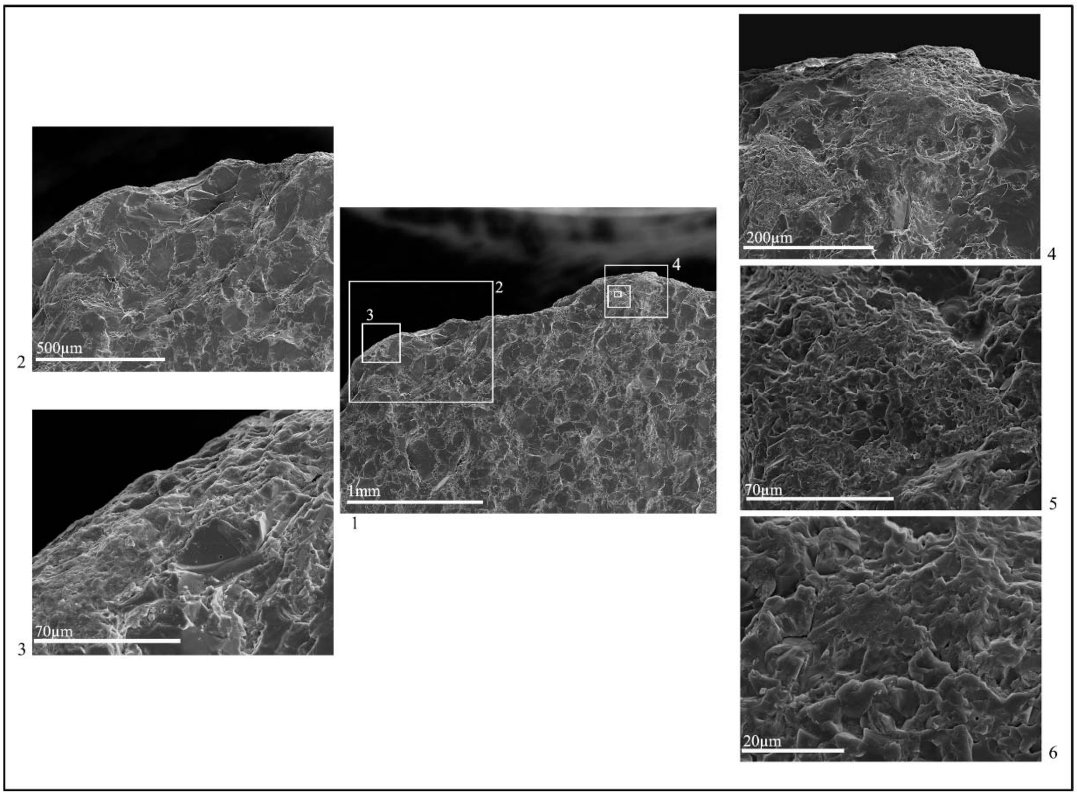

Fig. 6.51: *Experimental PDSM on a quartzite sample (xx). Macro-scars (2, 4) and extremely polished surfaces (4, 5, 6) are the main features observed. Magnifications: 1) 40x; 2) 100x; 3, 5) 800x; 4) 250x; 6) 1000x.*

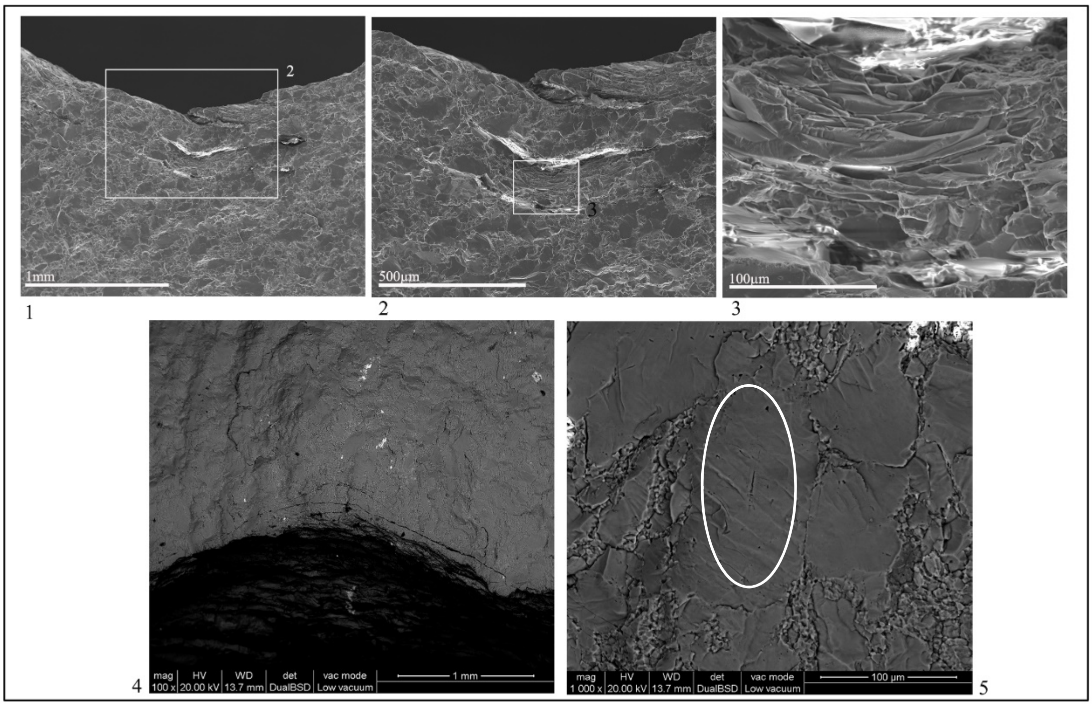

Fig. 6.52: *Technical traces: incipient fractures on retouched edges (1-3) and on the knapping surface (4). Micro-striation on the edge near the knapping surface (5). Magnifications: 1) 40x; 2) 100x; 3) 500x; 4) 100x; 5) 1000x.*

6.6.2.2 Morpho-potential units

38 used edges were identified on the 36 artefacts with use-wear traces. Two artefacts showed two used portions. 23 used portions were modified by retouch, while 12 of them are unretouched.

For all of them, the morpho-potential characters are considered (Carbonell et al., 1992, 1995a; Ollé, 2003; Vergès, 2003) and data are presented dividing the sample into retouched and unretouched artefacts.

The criteria considered are similar to those included into the techno-functional analysis of the entire flake assemblage, therefore at the end of the chapter a direct comparison with data coming from this analysis will be done.

All the used portions can be described as geometric dihedrals, except from two of them, which are identified as trihedral. For these two, no angle measurements are available.

As shown in Figure 6.53, the angle amplitude of the dihedrals ranges between the average values of 40° and 70°, with a minimal presence of more obtuse ones. This is valid for both retouched and un-retouched implements.

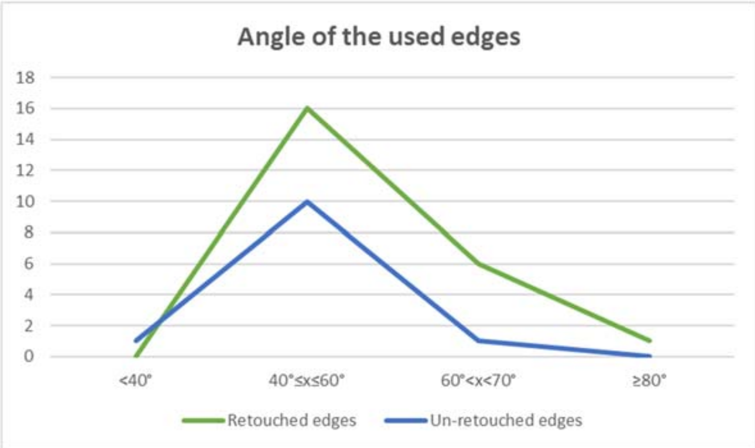

Fig. 6.53: *Average values of angle amplitude of the used edges.*

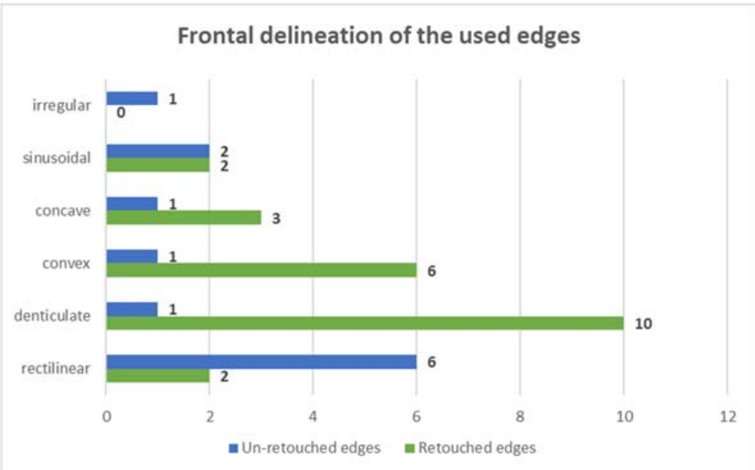

Fig. 6.54: *Frontal delineation of the used edge portions.*

Frontal delineations of the retouched artefacts are clearly mostly denticulate, followed by convex, concave, rectilinear and sinusoidal ones (Fig. 6.54). The un-retouched edges showed a prevalence of rectilinear frontal delineations and minor presence of the others.
Considering the sagittal delineation, the retouched edges have a prevalence of curve outlines, although rectilinear ones are also present. Conversely, rectilinear sagittal delineations predominate the un-retouched edges (Fig. 6.55).

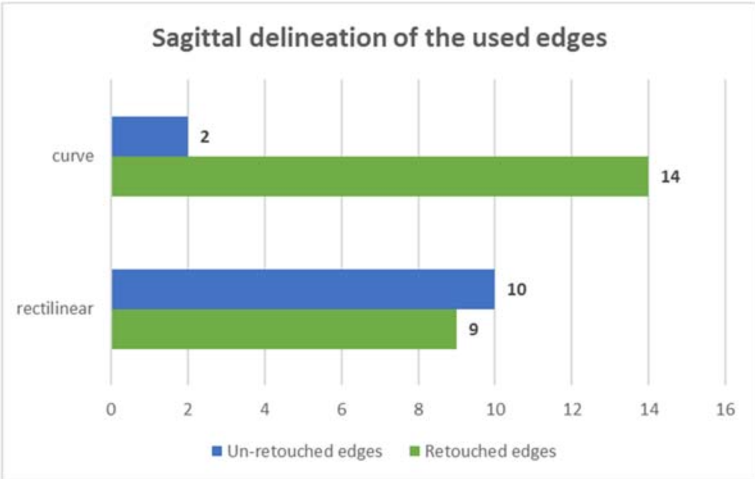

Fig. 6.55: *Sagittal delineation of the used edge portions.*

6.6.2.3 The kinematics and the actions performed

The kinematics of the action was identified for 35 artefacts presenting use-wear traces (for one of them it was not possible to deduce the kinematics and only the used portion was identified). For 7 of them, it was the only information provided by use-wear analysis, while for the other 27 more data is available (type of action, type of worked material, etc.).
37 used edges are then present on 35 artefacts. 18 of them were used to perform transversal actions, 17 to perform longitudinal actions and only two are related to rotational movements (Fig. 6.56).
An example when only the information related to the kinematics was obtained comes from the analysis of the most complete refit of TD10.1 (Fig. 6.57, REM1_3 refit). All the refitted implements were microscopically analysed, including the core, but only two of them showed some microwear evidence.
The first interesting insight comes from the conjoined pieces showed in Figure 6.58. The cortical flake broke probably during knapping and the larger piece was subsequently retouched by partially removing the cortex and by creating a denticulate frontal delineation (Fig. 6.58: a). While on the distal broken extremity of the original cortical flake (Fig. 6.58: b) no use-wear was observed, it is precisely on the retouched edge of the larger flake that some use-wear evidence was documented.

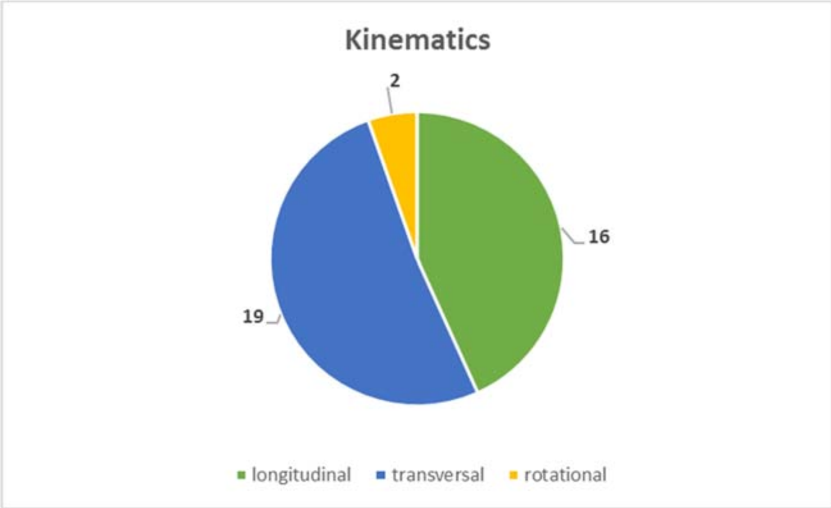

Fig. 6.56: *The kinematics of the used edges identified on 36 artefacts (38 used edges).*

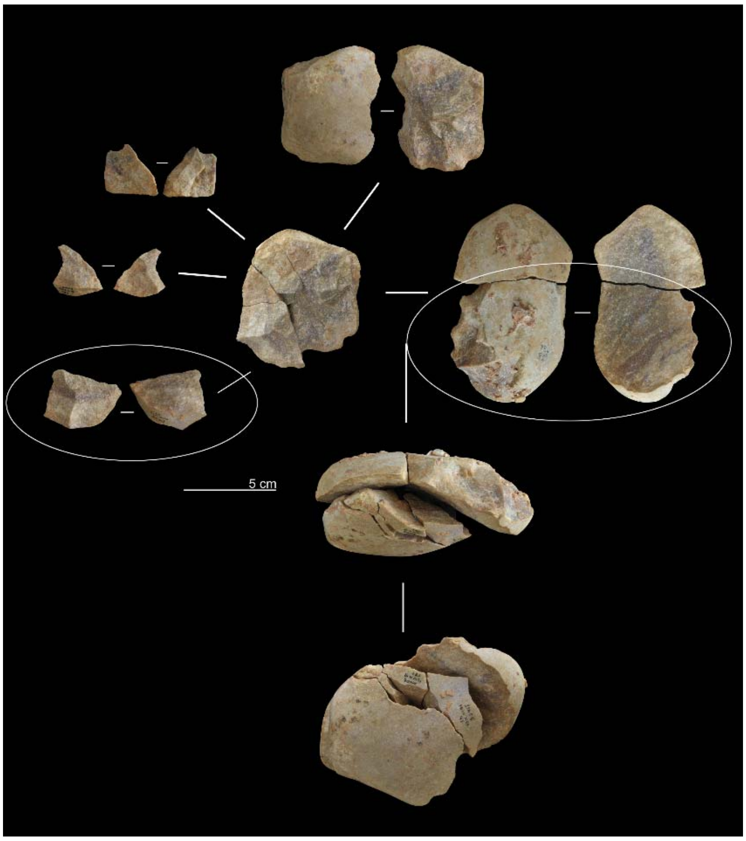

Fig. 6.57: *REM1_3 refit, the longest sequence refitted at TD10.1 unit (courtesy of E. López-Ortega). All the implements composing the refit were microscopically analysed. Only two pieces showed microwear evidence (white circles).*

However, use-wear was not enough developed to allow to identify the worked material. Only macro and micro-scars (Fig. 6.58:c, f) and a few polished areas (Fig. 6.58: e) were observed on this edge. Scars were present only on the dorsal face and showed evidences of transversal direction.

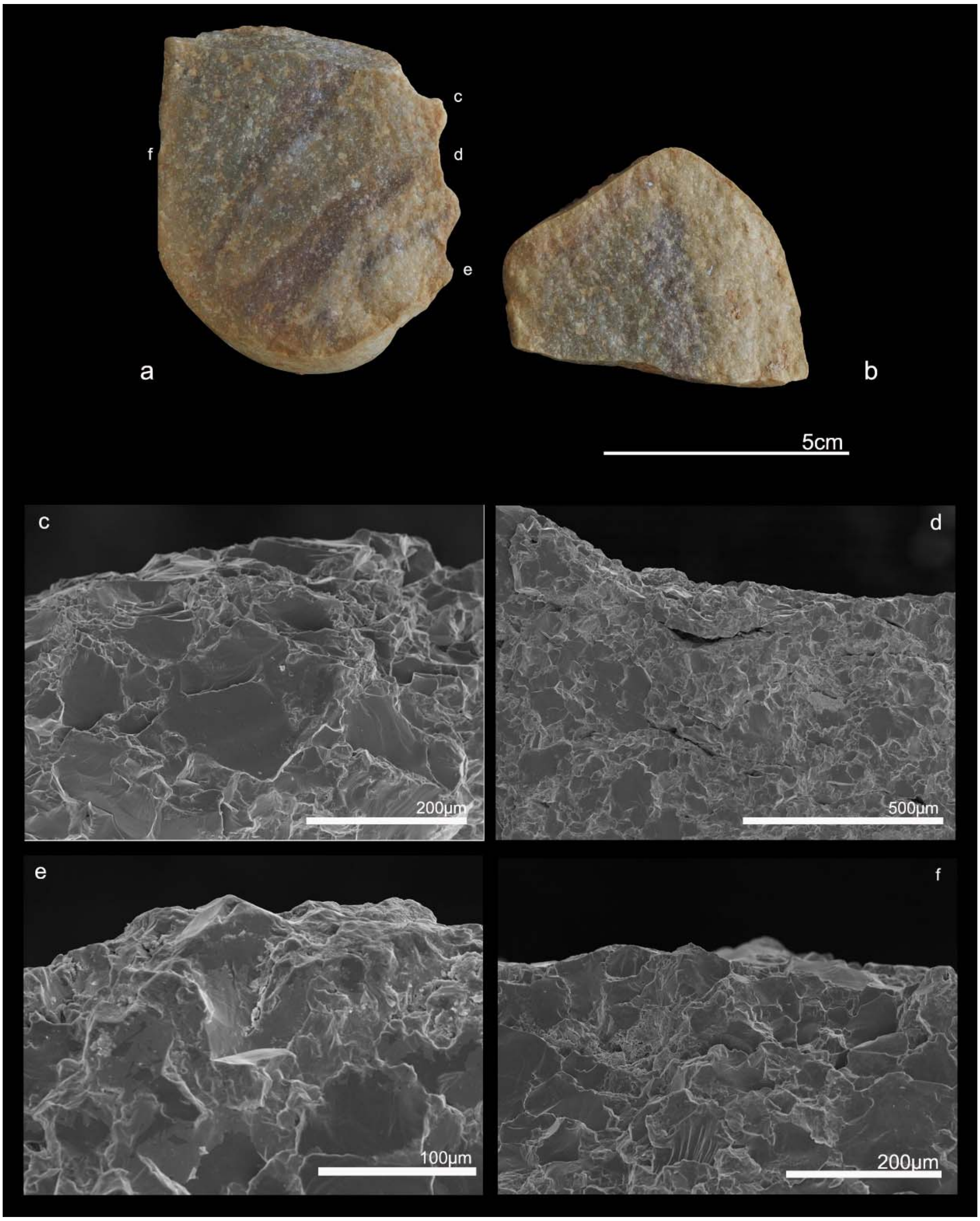

Fig. 6.58: *ATA99-TD10-I11-107: lateral denticulate on quartzite. The edge was retouched after the entire flake broke into two pieces; b) ATA03-TD10-N22-571: distal part of the flake that joins with I11-107. SEM micro-graphs showing evidence of scars (c, f: 200x), incipient fractures due to retouching (d: 65x) and a slightly rounded edge (e: 400x) (López-Ortega et al., in preparation).*

Moreover, incipient fractures due to the retouching of the edge were also observed (Fig. 6.58: d).

A second flake composing the REM1-3 refit also displayed use-wear traces (Fig. 6.59). Wear was less clear than on the first artefact and it is composed of mostly macro and micro-scars (Fig. 6.59: b). Very rare and under-developed polish was sometimes observed (Fig. 6.59: c).

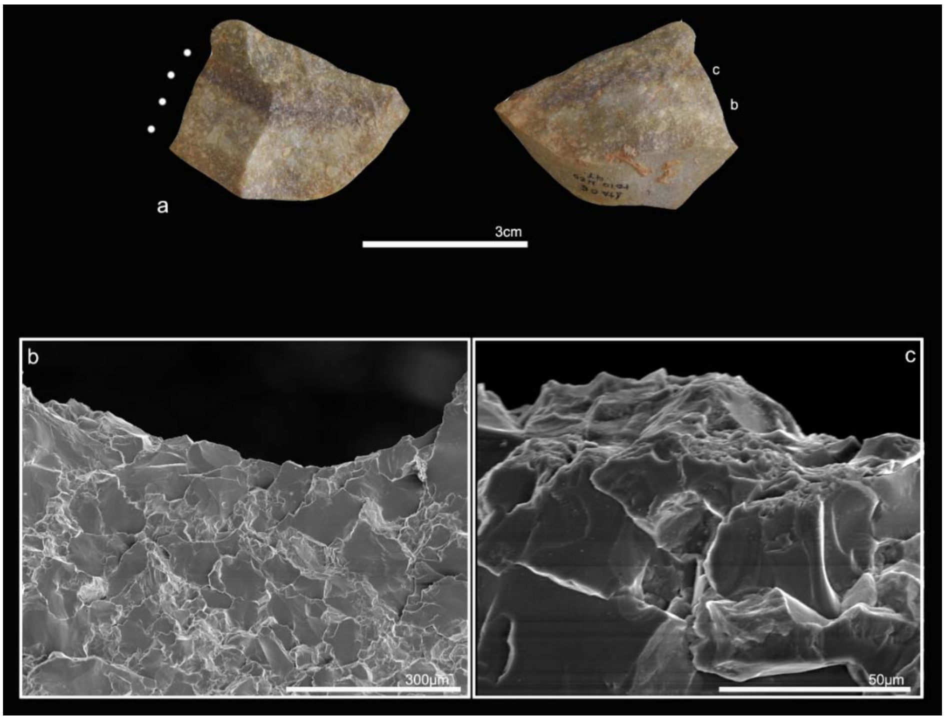

Fig. 6.59: *a) ATA05-TD10-N20-97: SEM micro-graphs showing evidence of scarring on the lateral left edge and reduced polished areas (magnifications: b) 150x; c) 1000x). Although use-wear evidence is poorly developed, it points to a transverse action (López-Ortega et al., in preparation).*

The interior surfaces of both artefacts were free from wear, fact which strengthens the interpretation that the edges showing under-developed wear were actually used to perform transversal actions.

The indicators of the kinematics can be multiple, but the most reliable ones are definitely linear features or striations. 'Linear features' is probably the most correct term, as it comprises a larger set of possible wear types (lineal distribution of polish or pits, scratches, etc.). The only presence of isolated striations is not sufficient to hypothesise about kinematics, as this requires more detailed information. For instance, the relative quantity and frequency of striations are important to evaluate the type of movement, as well as their relation to the edge (vicinity and orientation). Because of this, it is more useful to show images of striations within a certain context (for example by showing the extremity of the edge), than to provide only highly magnified images of them.

Then, the orientation with respect to the edge and the frequency of similarly oriented patterned are considered to provide reliable interpretations. At the same time, the absence of randomly oriented striations on the whole surface of artefacts is an additional criterion which is important to always specify. Therefore, striations parallel to the edge usually indicate longitudinal actions (Fig. 6.60), while linear features perpendicular to the edge are indicative of transversal movements (Fig. 6.61). Striations slightly oblique to the edge are also possible to form during longitudinal actions, mainly when performing uni-directional, forceful strokes (like in the butchering activity) (Fig. 6.60: e).

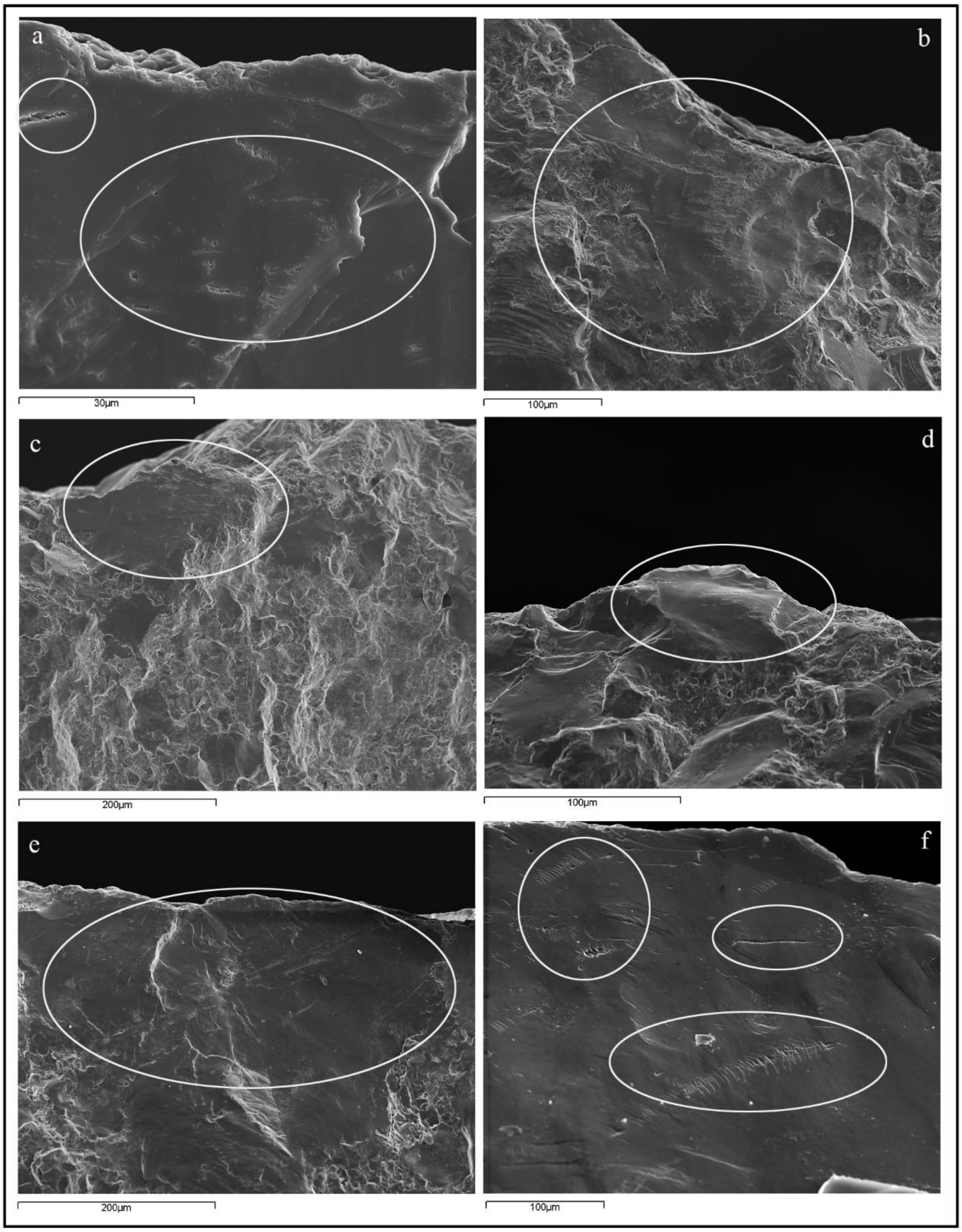

Fig. 6.60: *Microwear related to longitudinal actions. All linear features are parallel to the used edges. a) Relatively short furrows, 1500x; b) Several furrows having as a starting point a prominent crest, 300x; c) Numerous furrows on a flat crystal, 500x; d) A bunch of furrows on a linear depression of quartz crystal; e) Long furrows, oblique to the edge. The large flat and regular area of this crystal allowed the formation of particularly large furrows, 250x; f) Partial Hertzian cones and a groove (the linear, deep line), 3000x.*

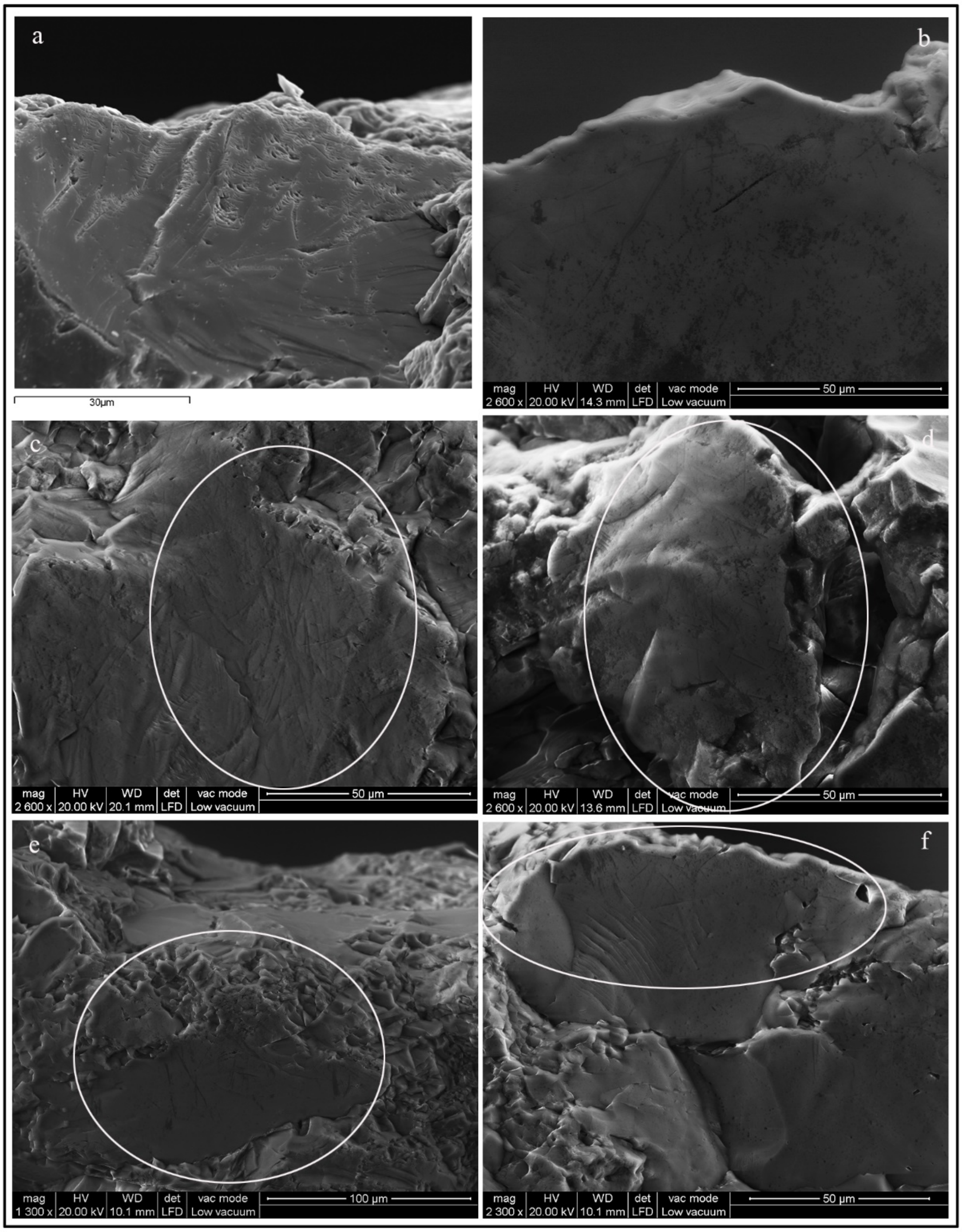

Fig. 6.61: *Microwear related to transversal actions.*

Generally, linear features related to transverse actions are shorter and somehow narrower than those formed during longitudinal activities. Moreover, their initial point is frequently located on the very edge (Fig. 6.60: a, b, e, f).

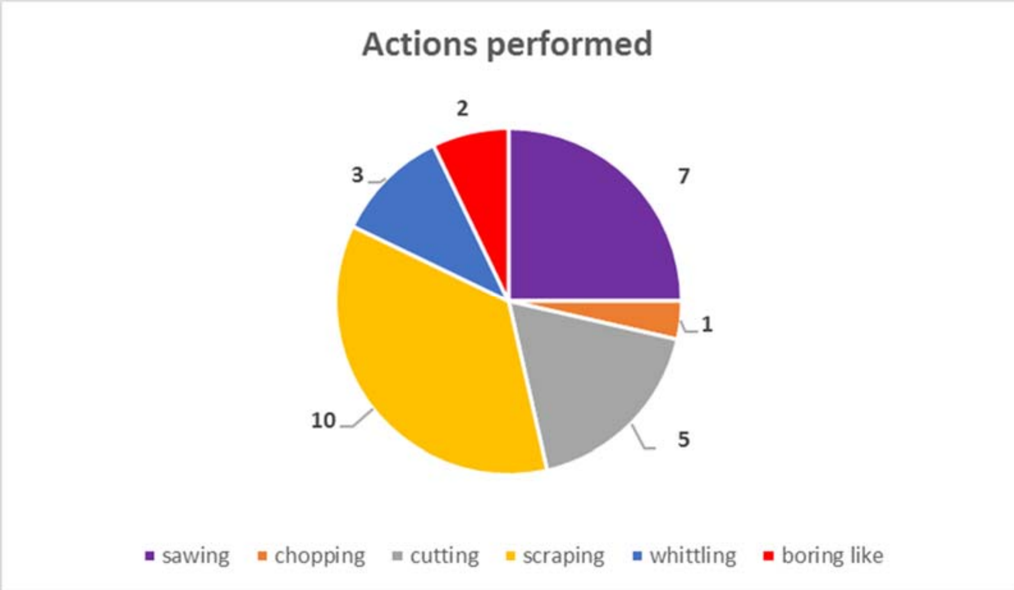

Fig. 6.62: *Main activities performed identified on 26 artefacts (two of them have two used edges).*

The main actions identified on the sample analysed comprise scraping and sawing, with respectively 10 and 7 artefacts. 5 artefacts are connected with cutting actions, while minor evidence of chopping, whittling and boring-like actions were also documented (Fig. 6.62).

Only two artefacts showed evidence of rotational-like movements. Use-wear is localised on the trihedral formed by the convergence of the two edges (tip) (Fig. 6.63, 6.64). The first artefact (ATA04-TD10-N18-4, Fig. 6.63) showed a naturally pointed lateral extremity, therefore the convergence is formed by the distal and proximal edges. On this extreme, use-wear connected to a boring-like action was found. Large macro-scars were observed as having a regular distribution on both the portions of the convergent edges, near the pointed extreme (Fig. 6.63: 1-3, 5). Micro-scars were also documented on the same portions (Fig. 6.63: 4). Rare striations, mostly oblique to the edges, were imaged (Fig. 6.63: 6).

The other artefact displaying evidence of rotational movements is ATA04-0TD10-K21-68 (Fig. 6.64). Again, wear is located on the trihedral originated by the convergence of the two lateral edges. The distal portions of these edges were slightly retouched, probably to emphasise the lateral concavities, which eventually made the apical point stand out. In fact, the potential of incising matter with the pointed extremity is increased by the presence of the two lateral concavities. Wear is found on the tip as well as on the distal lateral edges in the form of large macro-scars (Fig. 6.64: 1-4).

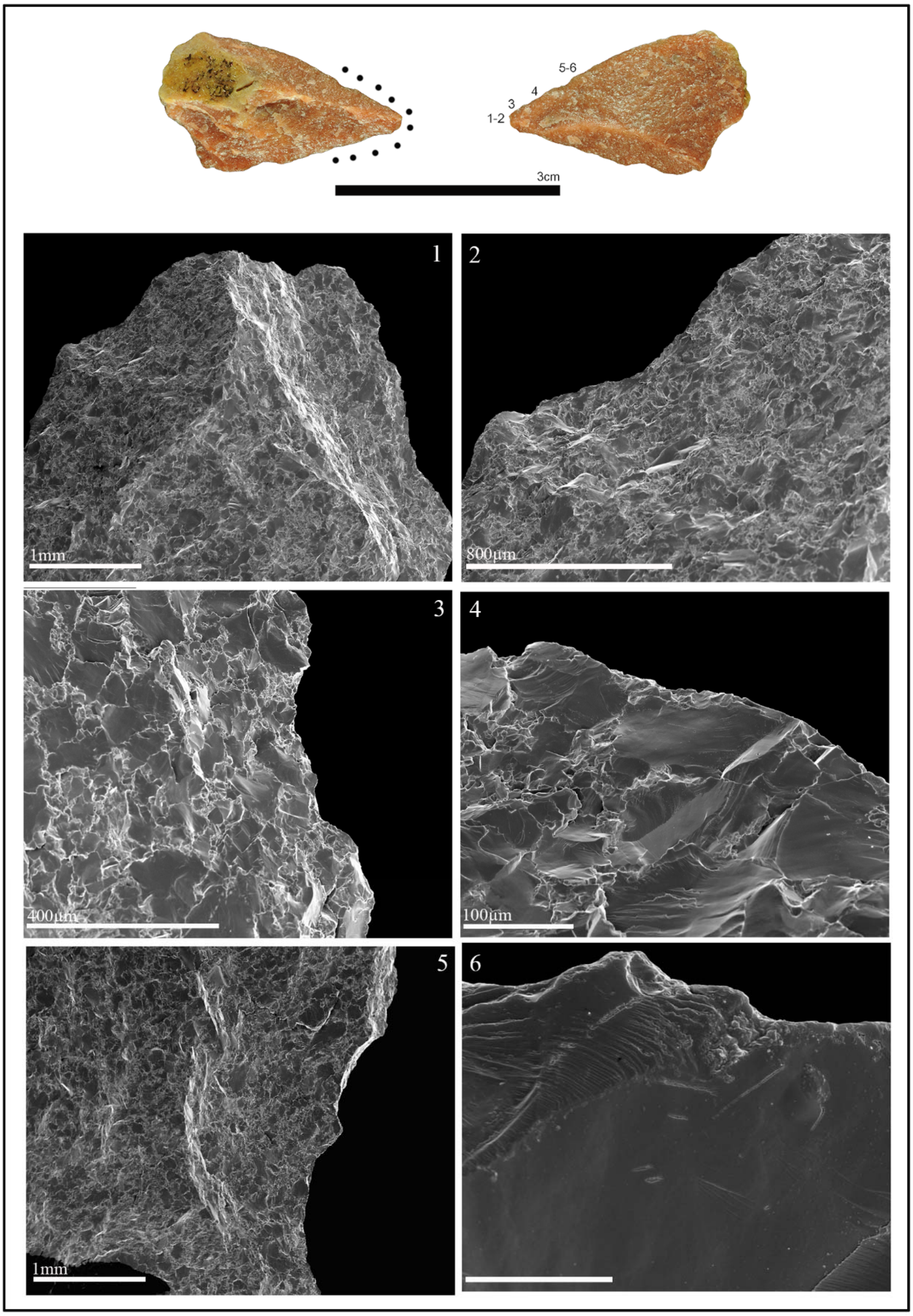

Fig. 6.63: *ATA04-TD10-N18-4: use-wear related to boring-like actions located on the convergence of the proximal and distal edges. Macro (1-3, 5) and micro (4) scars, and striations (5). Original magnifications: 1, 5) 30x; 2) 70; 3) 130x; 4) 300x; 6) 2000x.*

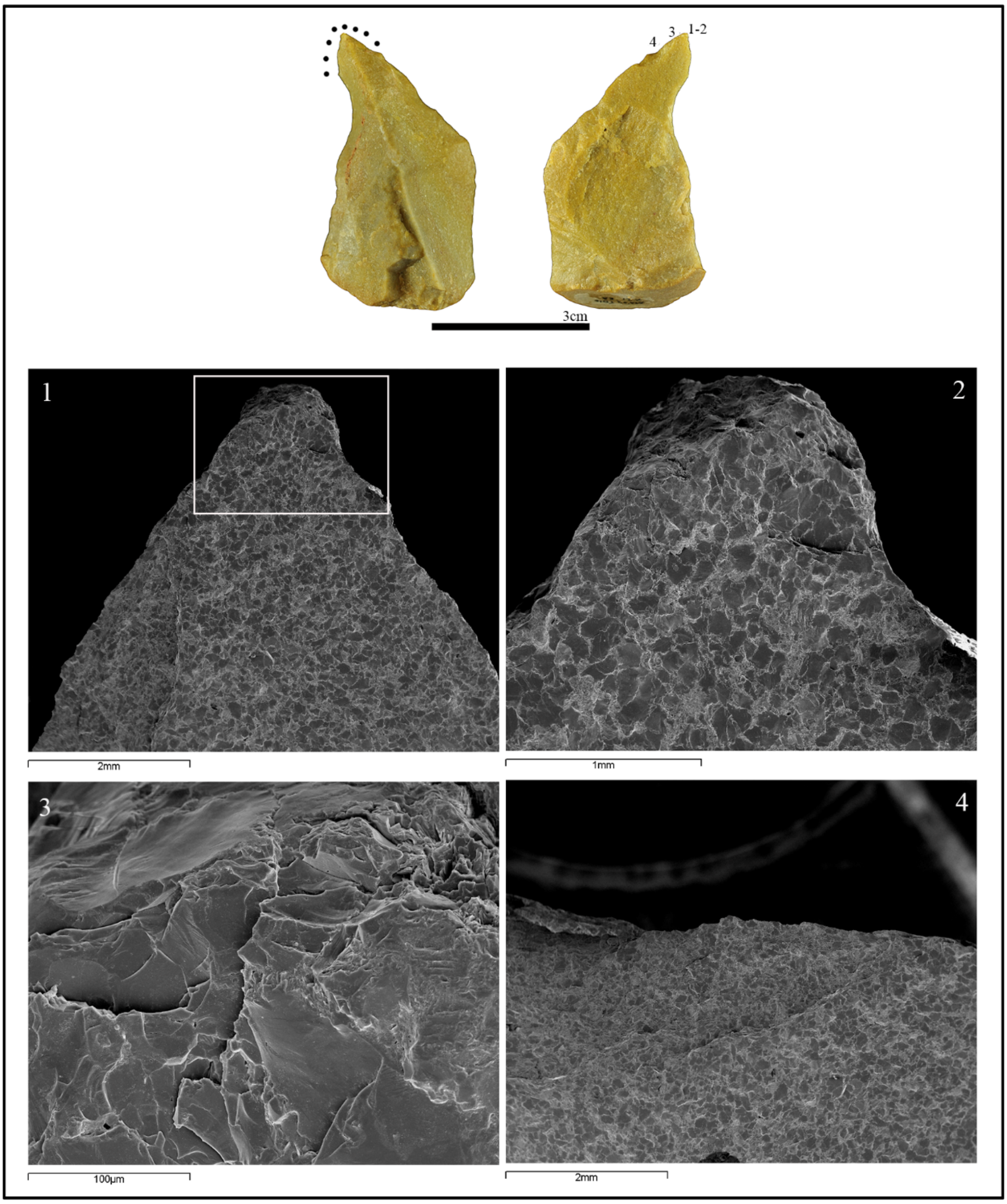

Fig. 6.64: *ATA04 TD10 K21 68: use-wear related to a boring-like action (rotational movement). Large macro-scars on the tip and on the lateral edges (1-4). Original magnifications: 1, 4) 20x; 2) 50; 3) 400x.*

One of the two artefacts characterised by two used edges is a cleaver-like object (ATA01 TD10 N14 320, Fig. 6.65). It a large semi-cortical retouched flake. The lateral right edge was retouched with a very abrupt and invasive retouch. In fact, this could also lead to consider it as a Quina side scraper. Evidences of longitudinal and transversal actions were observed on this artefact.

Use-wear related to a longitudinal action were found on the retouched edge. The second used edge is the distal one, where evidence of a chopping action is macroscopically visible: large macro-scars are present on both faces. On the dorsal face, impact points are probably the result of lithic percussion activities (Fig. 6.65, white squares).

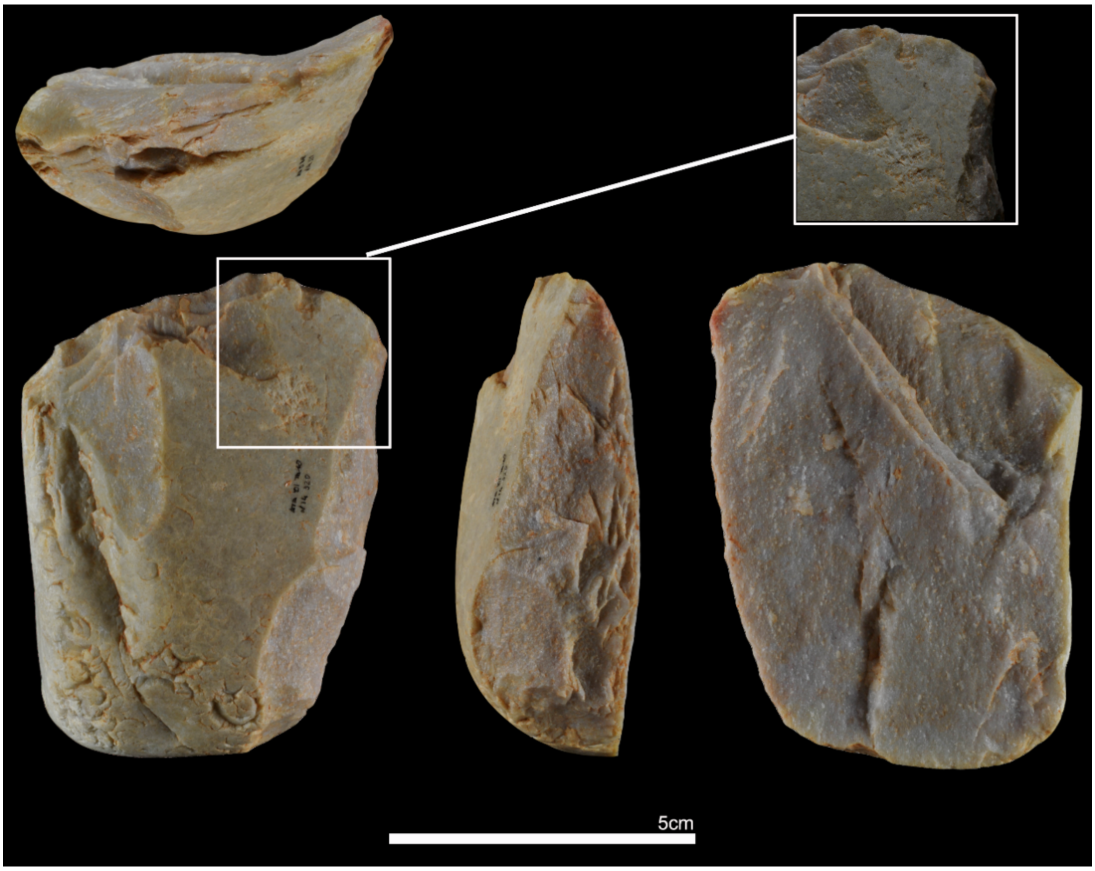

Fig. 6.65: *ATA01-TD10-N14-320. Hachereau-like artefact with a lateral abrupt retouched edge. The apical part show macro-detachments, related to chopping activities. An invading scar, possibly derived from the chopping activity, is visible on the ventral side of the artefact. On the above right corner, a detailed photograph of the impact points.*

6.6.2.4 The worked materials

The type of worked material was identified on 28 transformative units. From now on, data will relate only to this sample and not to the entire sample analysed (n=51).

More than half of the edges analysed showed evidence connected to woodworking (n=9) and to the processing of bone (n=8). A lower number of edges showed traces which were interpreted as originated from contact with soft animal matter (n=5), skin (n=2). The wear found on the rest of the edges was ascribed to more general categories, such as hard material (n=3) and greasy material (n=1). The broad category of animal matter is split into meat alone (n=2) and meat/skin (n=3), when traces refer to both materials and clearly point to butchering activities (Fig. 6.66).

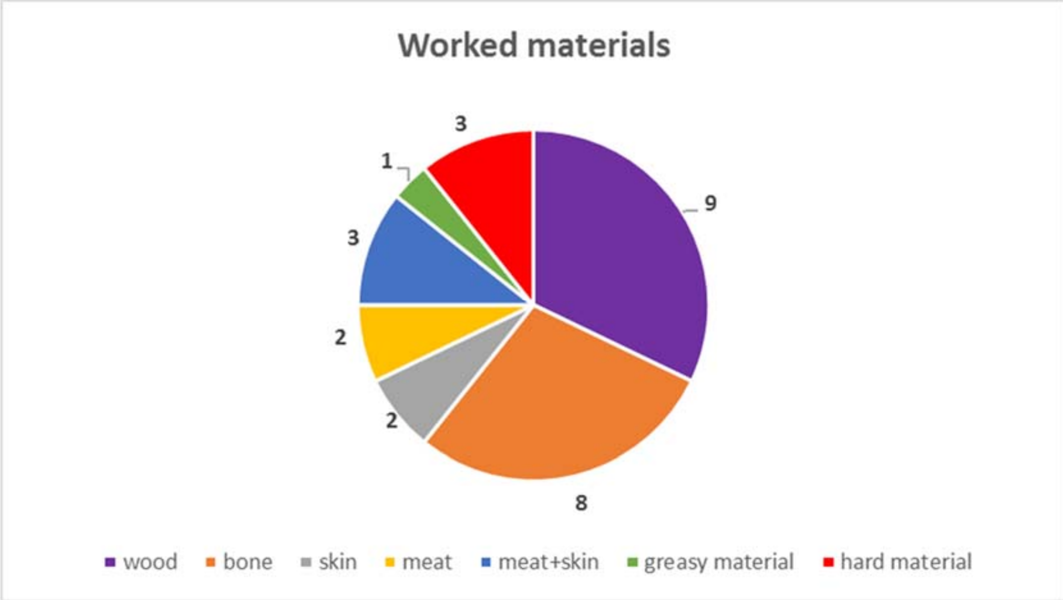

Fig. 6.66: *Worked materials identified on 26 artefacts (28 used edges).*

Traces related to the processing of wood displayed characteristics similar to those observed on the experimental record. Polish has the same visual appearance which has been observed on experimental implements used on wood. The texture is essentially very smooth (Fig. 6.67: c, d), although in some cases, a mixture of smooth and rough polishes might occur (Fig. 6.67: a). When polish is under-developed, it is mostly found on high prominences of the micro-surface (Fig. 6.67: b) or it only affects the very rim (Fig. 6.67: f). Typical furrows associated to woodworking were also recorded (Fig. 6.67: e), although not with the same developmental degree than those observed on experimental tools.

Wear connected to bone also displayed features recognisable thanks to comparison with the experimental referential data. Furrows are less numerous than on implements used on wood and are generally shorter (Fig. 6.68: a). Polish is very smooth, but polish areas are very limited and always found on protruding zones of the micro-surface (Fig. 6.68: c, e, f, g). Scarring can affect large macro-portions of the edge (Fig. 6.68: d) or be restricted to the edge of single crystals (micro-scars) (Fig. 6.68: b).

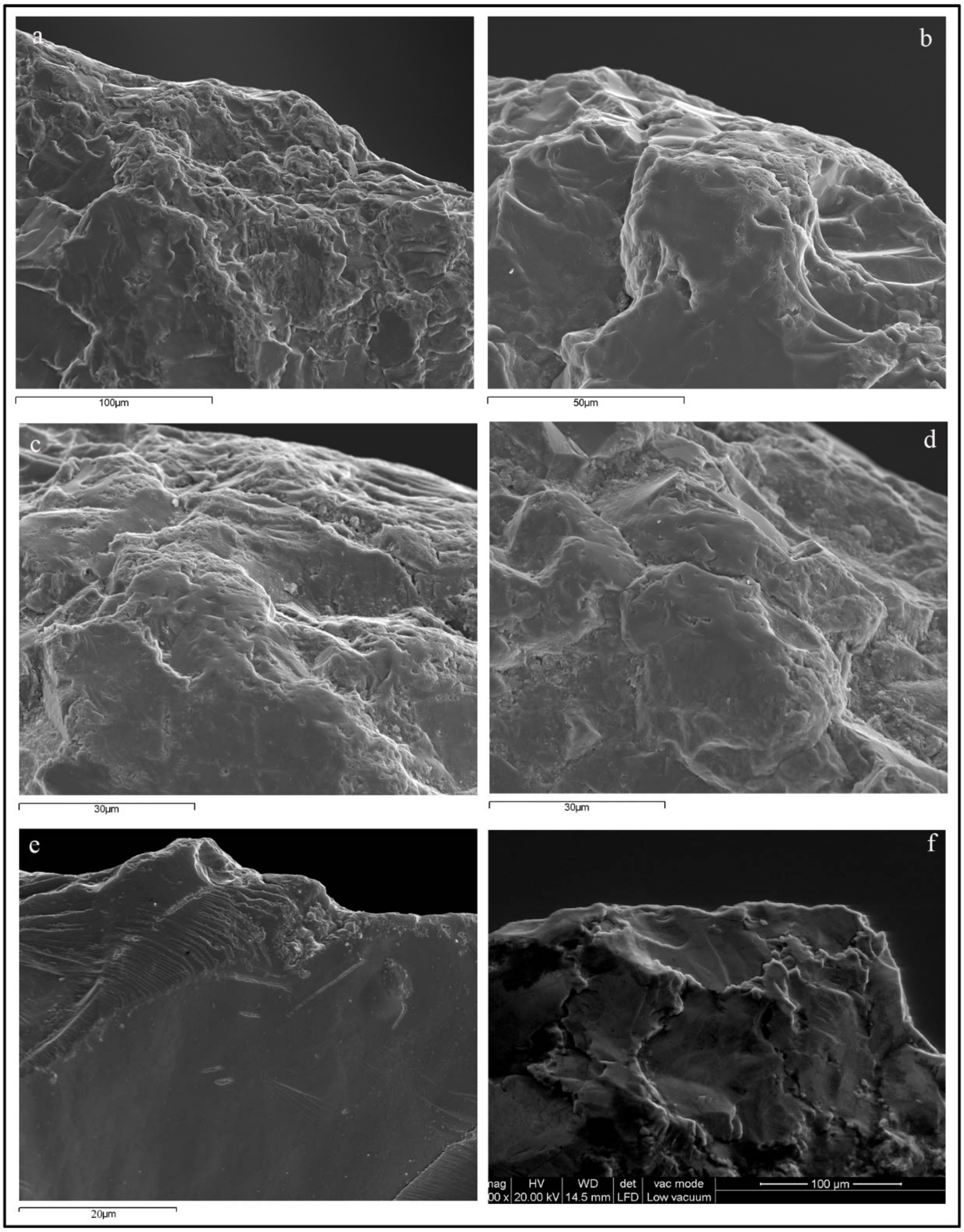

Fig. 6.67: *Use-wear traces connected to woodworking. a) Extensive polished area, 500x; b) Small polished area on a high micro-topographical point, 1000x; c) Smooth polished area and furrows on a flat crystal, 1500x, 1500x.*

Traces related to the butchering activity, therefore to the processing of meat and skin, comprise very well-developed polished areas and very rare striations. Polish is often restricted on the high parts of the micro-topography, but they frequently display directionality marks. Polished areas are often found oblique to the edge, as a consequence of repeated unidirectional, longitudinal movements, characteristic of the actions necessary to remove the skin of an animal and to de-flesh long bones (Fig. 6.69: a-c, e). Protruding, angular zones are also suitable to be covered by rough polish during butchering activities (Fig. 6.69: d).

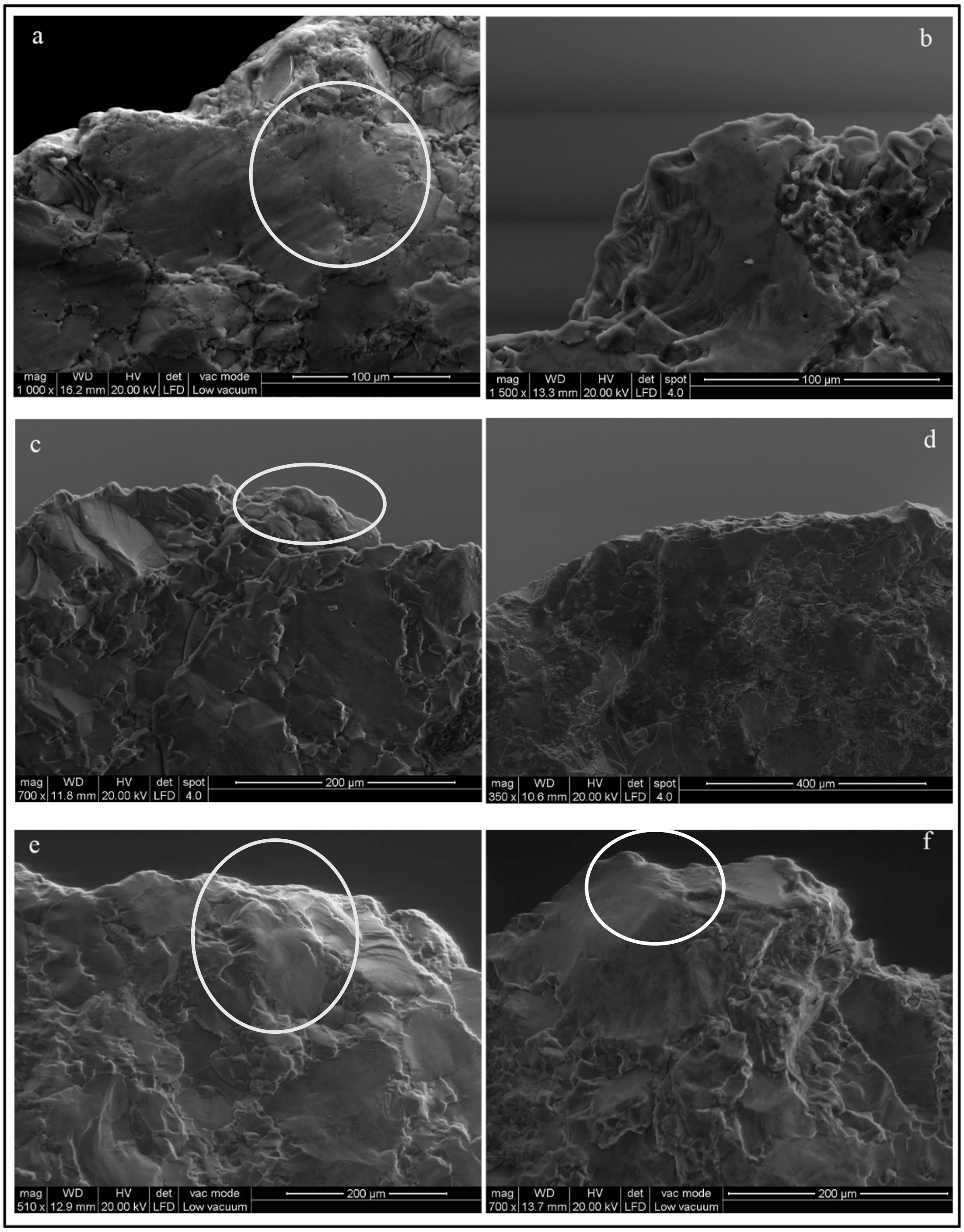

Fig. 6.68: *Use-wear traces connected to the processing of bone. a) Micro-rounding of the very rim, localised polished on the highest part of a quartz crystal and furrows perpendicular to the edge (circle); b) Micro-rounding of the edge of a quartz crystal and micro-scars on the very rim; c) Localised smooth polished area on a prominent spot; d) Continuous scarring of the edge; e) smooth polished and rounding; f) Smooth polished areas on the highest part of a quartz crystal.*

Striations are rare and normally very short (Fig. 6.69: f).

Wear formed after contact with hides or skins is very characteristic and normally develops on relatively large areas. Edge rounding is present on large portions of edges and it can be quite invasive when the action performed is transversal (Fig. 6.70: a, c). The polish texture is always rough and pits are sometimes visible on it (Fig. 6.70: e). Micro and macro-scars are also visible (Fig. 6.70: b, d). Striations are very rare, but when present, are always very short and narrow (Fig. 6.70: f).

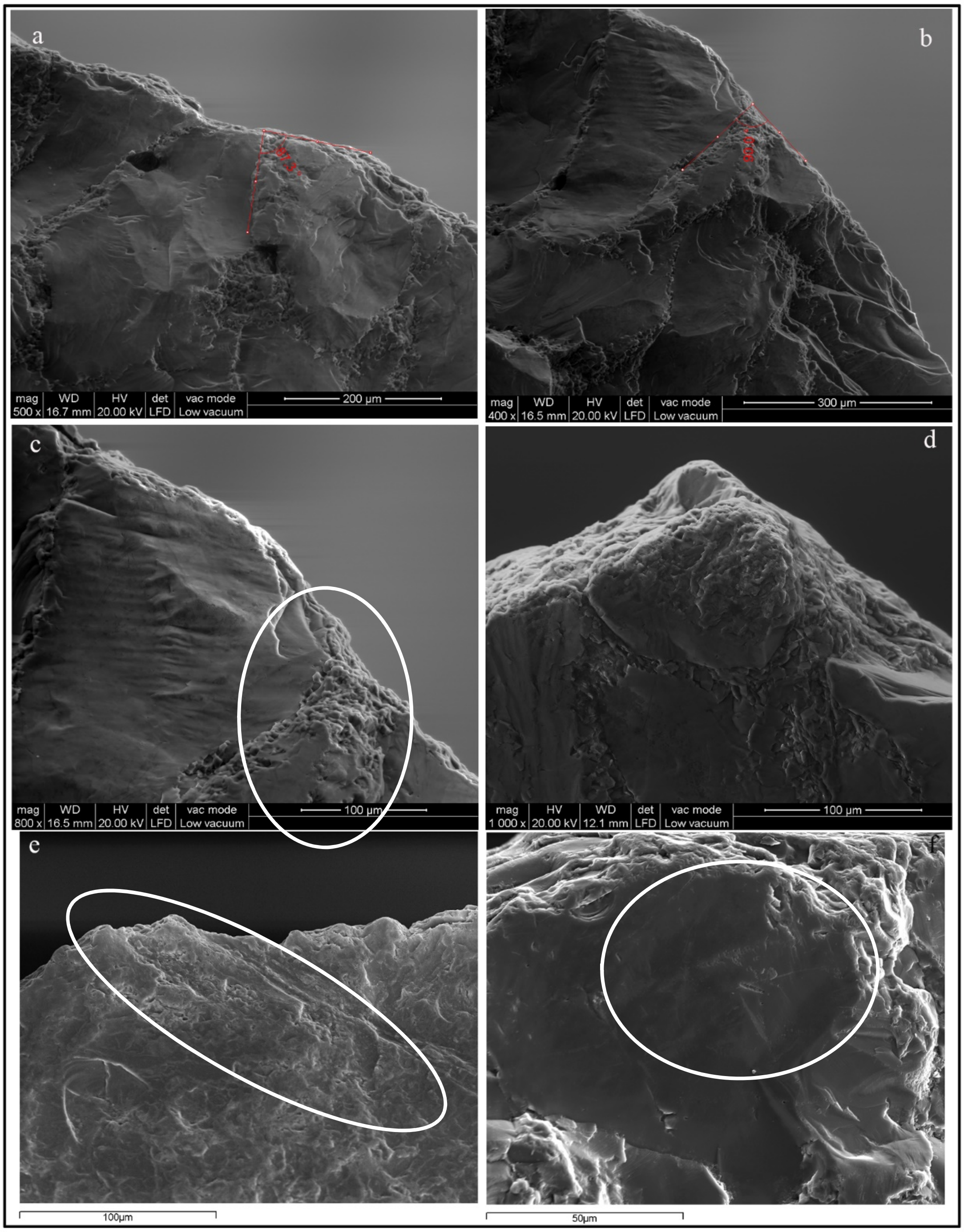

Fig. 6.69: *Use-wear traces connected to butchering activity. a, b) Lines of rough polish, distributed obliquely to the edge; c) Line of rough polish, distributed obliquely to the edge and micro-rounding of a single quartz grain; d) Polished area on a prominent, angular zone; c) Line of polish, disposed obliquely to the edge 500x; f) Short striations parallel to the edge located on a flat crystal and polish on the edge of the same crystal, 1000x. In figures a and b, the angle between the lines of polish and the edge is measured (red lines).*

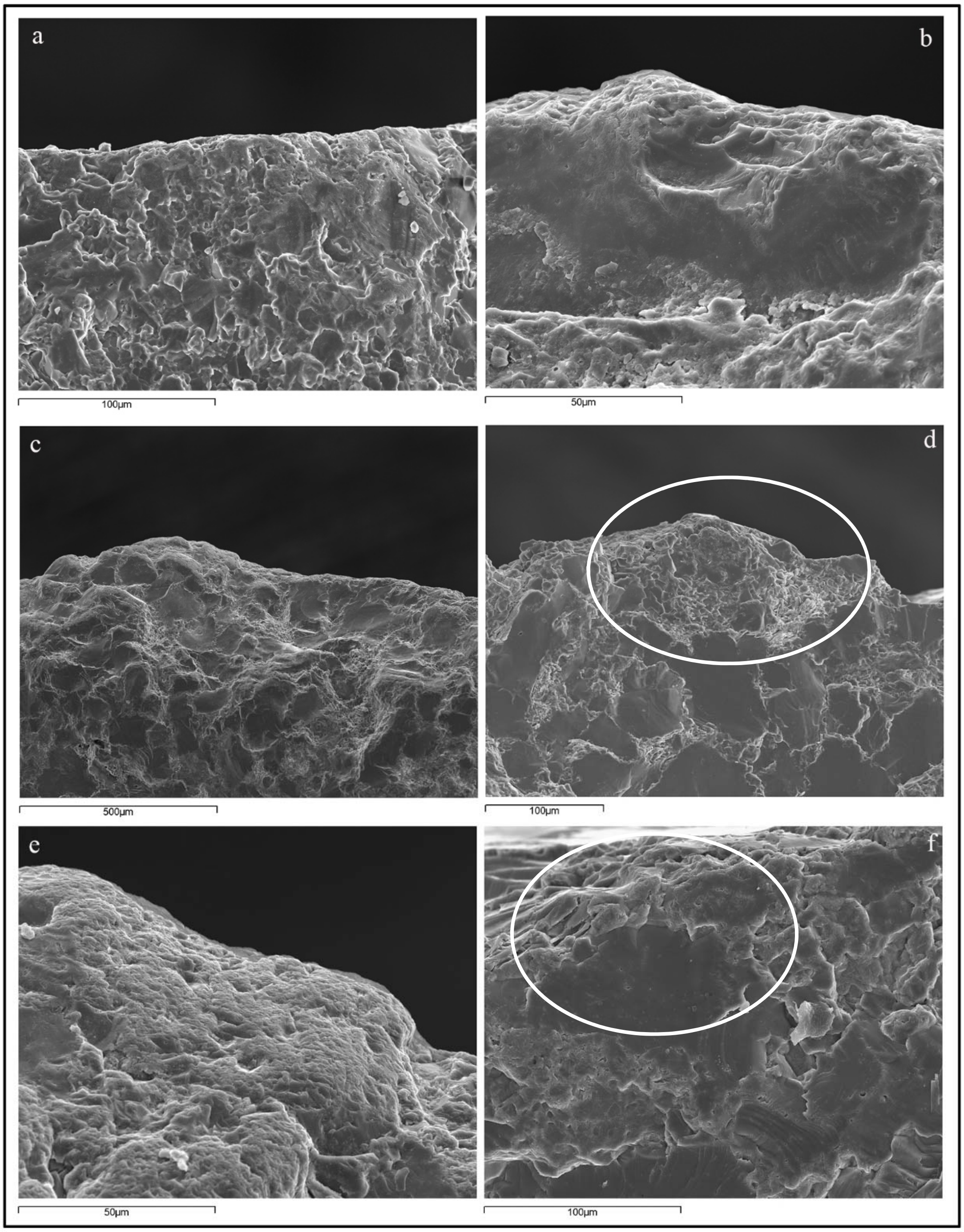

Fig. 6.70: *Use-wear traces connected to hide scraping. a) Continuous edge rounding, 500x; b) Edge rounding, micro-scars and micro-striations, perpendicular to the edge, 1000x; c) Continuous edge rounding, 100x; d) Large scar and small polished area, 500x; e) Detailed image of the rough texture of a polished area, 1000x; f) Polish on the rim and tiny striations, perpendicular to the edge, 500x.*

6.6.2.5 Hafting traces

Possible evidence of hafting was encountered on three artefacts. The inference of hafting traces was quite challenging, since no systematic hafting experiment was carried out during the previous stages of our research. The proposition of these three artefacts as having been hafted is based on several evidences, from the different characters of certain traces to their disposition on the surfaces of the tools. However, more data is necessary to corroborate these interpretations, mainly the performance of specific experiments with hafted quartzite implements.

The first tool on which hafting evidence was found is a triangular small-sized retouched flake (26x22x15mm). Both lateral edges are retouched, the left one by an invasive, semi-abrupt denticulated retouch and the right one by a unique invasive removal (*encoche*). Wear connected to a longitudinal action on a hard material was recorded on the lateral edges of the tip was observed, namely furrows parallel to the edges (Fig. 6.71: a, b) and micro-fractures on the rim. On the proximal part, crushing of the edge and abrasion of the surface were observed (Fig. 6.71: c). On the highest part of the dorsal face, polished areas were also documented. Moreover, furrows perpendicular to the edge were visible on crystals found at the extremity of elevated ridges (Fig. 6.71: d). This type of wear was only found on this area and the mesial part of the artefact was observed to be relatively fresh. Wear is very distinct based on its location on the artefact, therefore the clear pattern differentiation of wear on this artefact allowed us to propose the hypothesis of it having been hafted on the basal part.

A second artefact had different patterns of traces depending on their position. A triangular retouched flake (ATA00-TD10-N20-66) (61x63x18mm) displayed wear related to use on one of its lateral edges, precisely the non-retouched one (Fig. 6.72). Intense edge rounding and furrows perpendicular to the edge were indicative of a transversal action performed with the un-retouched edge (Fig. 6.72: a, b). On the basal part, there is macro-evidence of edge thinning, possibly to fit in a handle. Moreover, abrasion of the upper parts on the proximal zone as well as crushing on the highest ridges were observed (Fig. 6.72: c, d).

A third artefact (42x28x9mm) displayed evidences of hafting on its distal part. The distal portion of the left edge was retouched and a concavity was obtained, maybe with the purpose to fit a handle. Large polished areas ('bright spots') were observed on the lateral distal portions of the edges and on the distal edge (Fig. 6.79: c). On the same zones, large scars, having a perpendicular disposition to the edges were recorded (Fig. 6.73: d). Striations pointing to a longitudinal action were recorded on the proximal left edge (Fig. 6.73: b). On the same portion of the edge, a few polished areas resembled polish originated from the processing of experimental bone (Fig. 6.73: a).

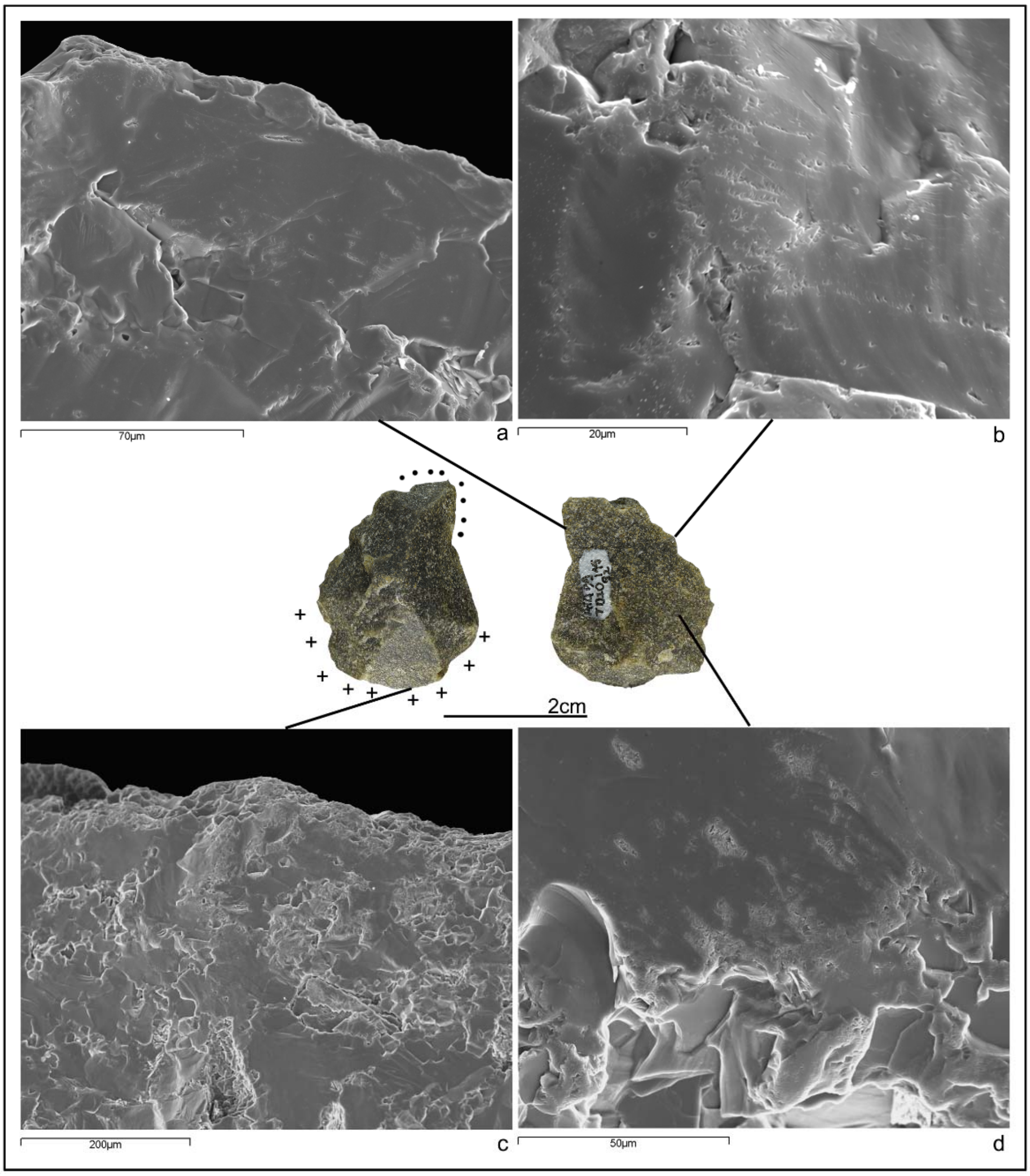

Fig. 6.71: *ATA99-TD10-I15-92: Evidence of hafting on a fine quartzite lateral denticulate. Use-wear associated with a longitudinal action (sawing) performed on a hard material was recorded exactly on the distal part. Striations are parallel to the lateral edges of the trihedral (a-b), whereas abrasion signs (c) are located on the proximal edge. Large and extremely marked striations were found on the proximal part of the tool and only on the most prominent portions of the ventral face (d). Dots indicate areas exhibiting use-wear, whereas crosses illustrate where the traces associated with the handle were recorded. Magnifications: a) 750x; b) 2000x; c) 200x; c) 1000x.*

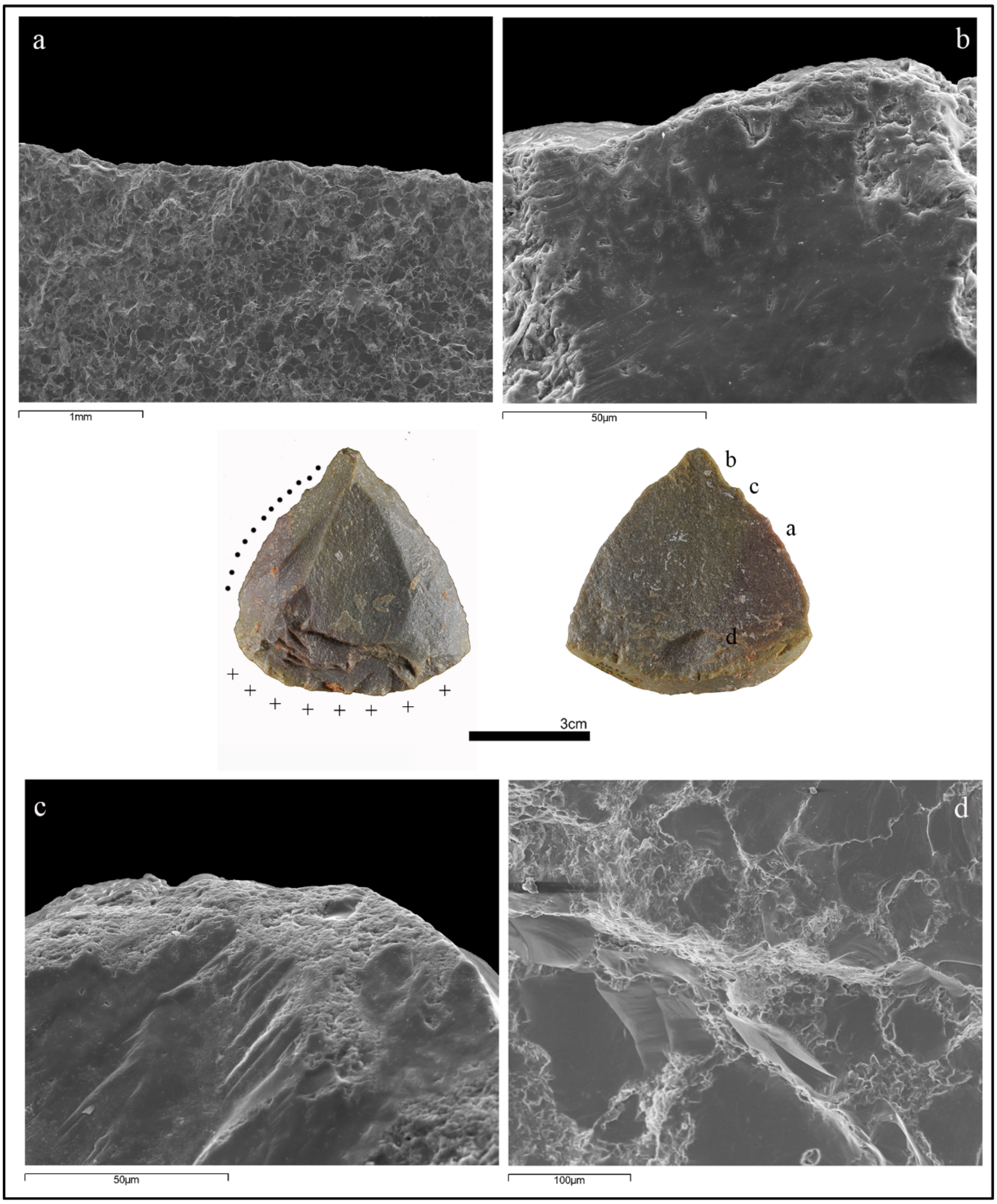

Fig. 6.72: *ATA00-TD10-N20-66. Use-wear related to hide scraping on one of the lateral edges (a, b) and evidence which points to hafting: thinning of the basal part and micro-abrasion and crushing on the proximal edge (c, d). Dots indicate areas exhibiting use-wear, whereas crosses illustrate where the traces associated with the handle were recorded. Magnifications: a) 40x; b, c) 1000x; d) 300x.*

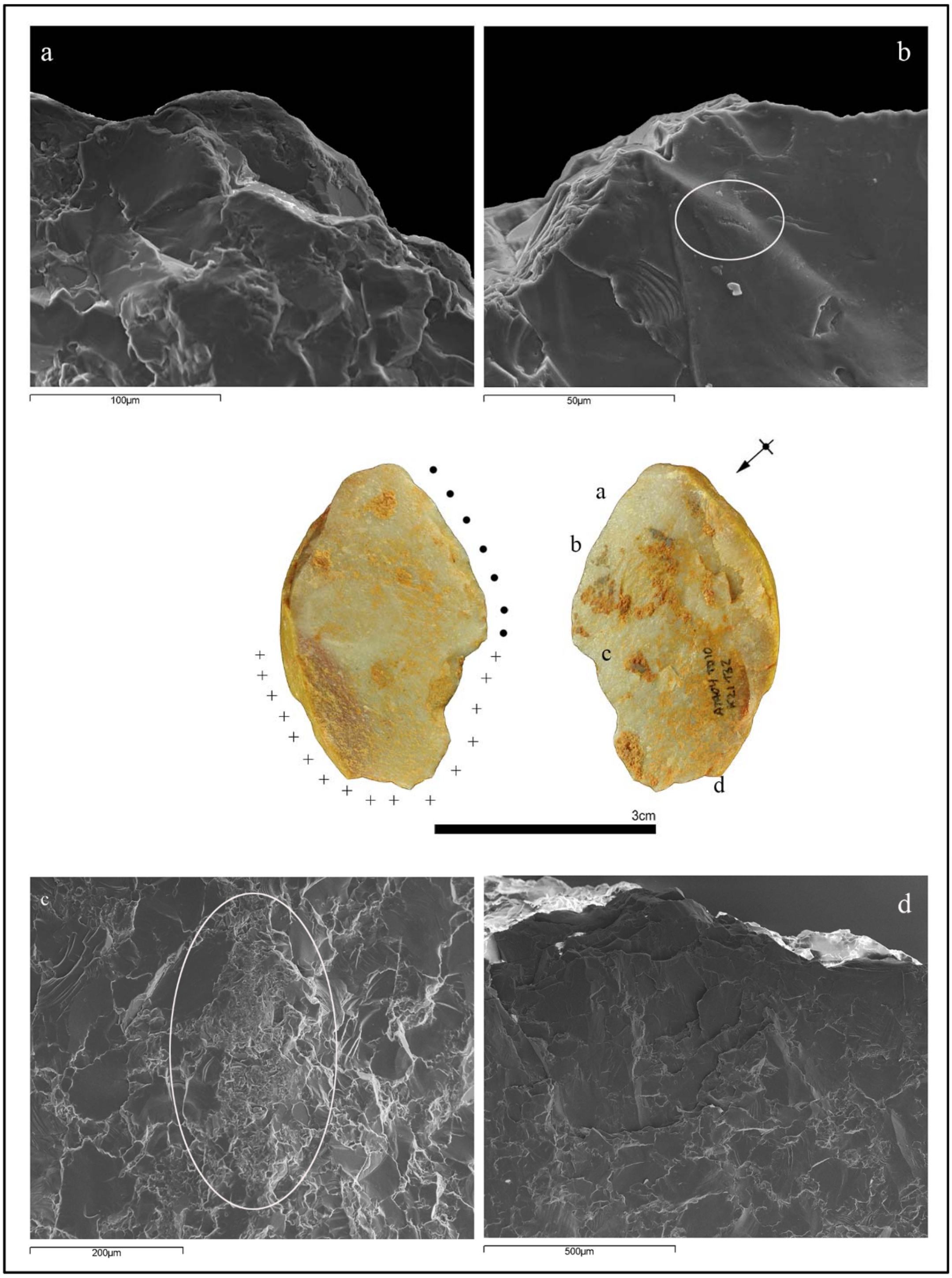

Fig. 6.73: *ATA04-TD10-K21-132. Use-wear evidence pointing to longitudinal action on bone and hafting traces. a) Smooth polish on the very rim, 500x; b) Furrows on a flat crystal, parallel to the edge, 1000x; c) Large bright spot, 200x; d) Large quadrangular scar, 250x. Dots indicate areas exhibiting use-wear, whereas crosses illustrate where the traces associated with the handle were recorded.*

6.6.3 Summary of use-wear results

6.6.3.1 Relation between actions and worked materials

Because the data is based on a limited number of artefacts analysed, we present our results in terms of numerical data and not of percentage. By comparing the kinematics and the material hardness, we see that only two artefacts were found bearing traces of rotational movements and all of them were used on hard materials (wood or bone). The processing of hard materials is displayed on more than half of the sample analysed with 18 artefacts, 11 of them used to perform transversal actions and 7 of them to perform longitudinal ones. 8 artefacts were used to perform transversal movements (n=3) and longitudinal ones (n= 5) on soft materials (Fig. 6.74).

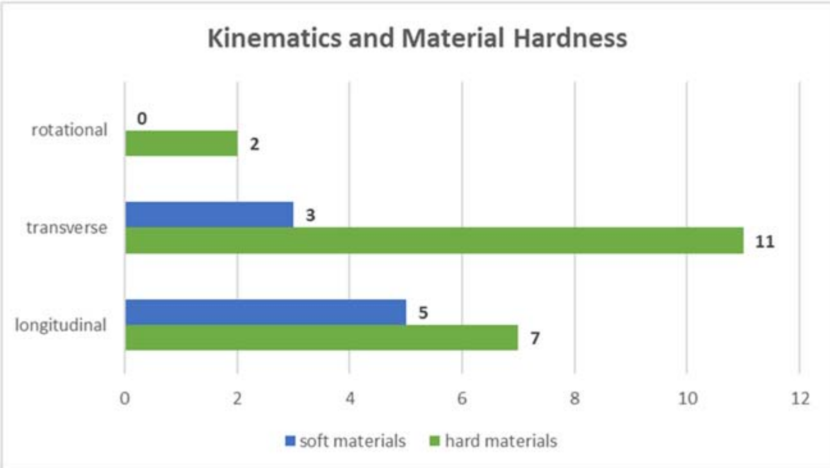

Fig. 6.74: *Kinematics and material hardness of the sample analysed.*

When analysing the activities identified (kinematics and worked materials), there is not a clear predominance of a specific action (Fig. 6.75). Scraping activities are slightly more frequent than the longitudinal ones and were performed on wood with 4 artefacts, on bone and on hide with respectively 3 and 2 artefacts. Scraping on greasy materials is intended as the possible removal of periosteum and meat remnants from bones and it is found on only 1 implement. Whittling is also present on wood and bone, with respectively 2 and 1 tools. Chopping is also included into transversal movements and it is present on only one artefact.

Longitudinal actions can be basically divided into cutting and sawing, based on the nature of the material worked. Cutting is used when the material which is modified is comprised in the general category of soft materials, while sawing is used when the worked material is hard or very hard. Cutting soft animal matter is identified on 5 artefacts, while sawing is found on 7 artefacts. For 6 of them, the type of worked material is identifiable: 3 of them were used on wood and the other 3 on bone.

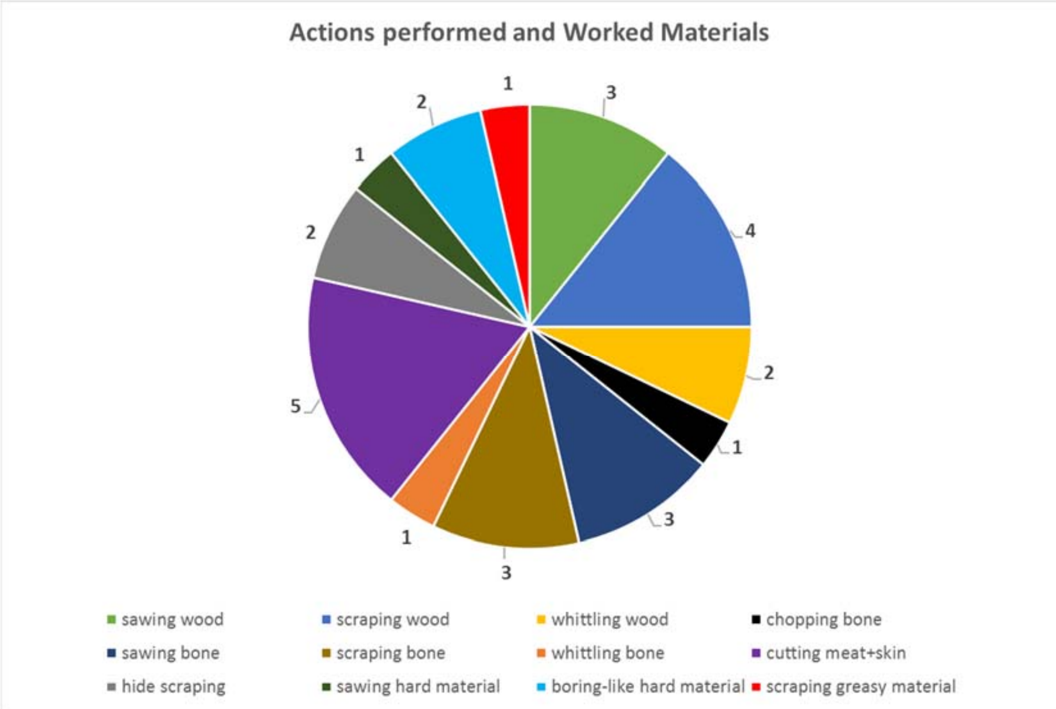

Fig. 6.75: *Kinematics and worked materials of the sample analysed.*

Regarding the morpho-potential characters of the used edges, angles and frontal delineations are considered separately on tools used on hard and soft materials. We can see that obtuse angles are not present, except from one case and that angles are normally comprised between 50° and 70°, despite the action performed (Fig. 7.76). Very acute angles are also not common. Frontal delineations of the active edges are varied, but no clear predominance of a particular feature is seen. Denticulate frontal delineations are present on a considerate number of edges used to modify hard materials. Concave, convex, rectilinear and sinusoidal delineations are present with similar values (Fig. 6.77).

The limited number of artefacts with clear traces does not allow us to provide deeper statistical significance to the data presented here. So far, no clear correlations between the morpho-potential characters of the used edges and specific functions can be made.

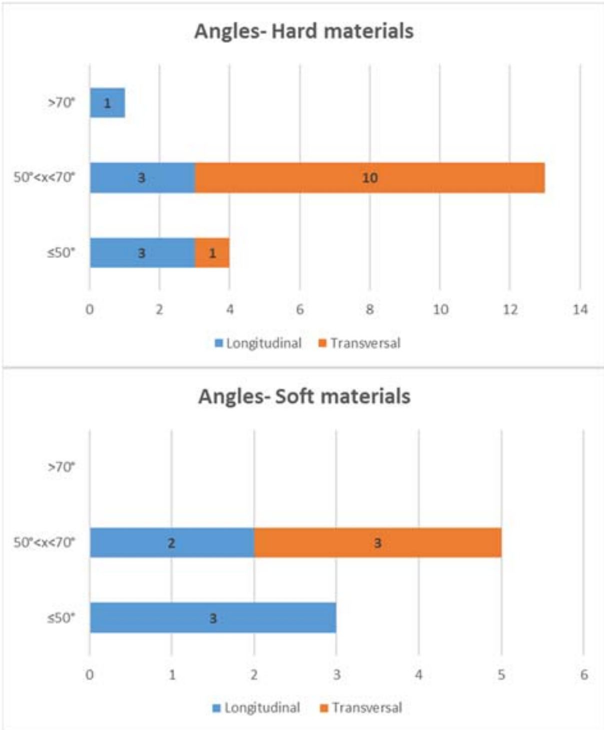

Fig. 6.76: *Angles of the active edges, used on hard and soft materials.*

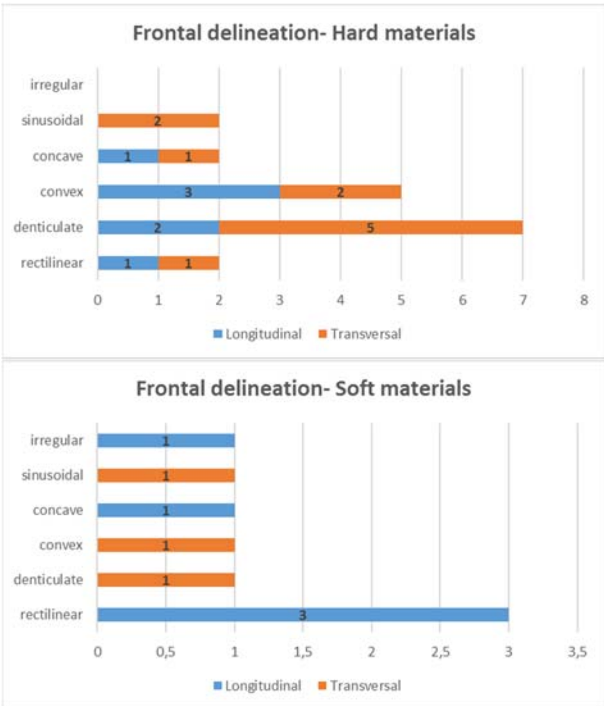

Fig. 6.77: *Frontal delineation of the active edges, used on hard and soft materials.*

6.6.3.2 Integration with techno-functional data

Among the artefacts which displayed use-wear on their surfaces, 30 were previously ascribed to one of the three groups identified during techno-functional analysis. Table 6.24 shows the number of artefacts per sub-type and the type of movement associated.

The use-wear observed was always found on the t-TFUs previously identified on artefacts pertaining to Group I. Regarding Groups II. and III., where several potential t-TFUs were identified (sub-types II.b, II.c, III.a), use-wear was generally found on only one transformative unit. More specifically: used edges were recognised on a later edge of the only II.b artefact analysed; on the two II.c artefacts analysed, used edges were found on a lateral and on a distal edge respectively; 5 artefacts pertaining to III.a sub-type displayed use-wear on a lateral edge, except from one which had use-wear on the distal trihedral (tip).

III.b and III.c had one lateral transformative-TFU and a distal convergent-TFU (trihedral) respectively. In both cases (2 artefacts), use-wear was found on the portions coinciding with the previously described t-TFUs.

Therefore, the only discordance underlined regards one artefact ascribed to sub-type III.a (therefore, having two potential lateral t-TFUs), on which use-wear was found on the convergence of the two lateral edges (rotational movement).

Techno-type	Longitudinal	Transversal	Rotational	Unclear	Tot. Numb.
I.a	5	1			6
I.b		1			1
I.c		3			3
I.d	4	2		1	7
I.e		2			2
II.b	1				1
II.c	1	1			2
III.a	3	2	1		6
III.b	1				1
III.c			1		1
no techno-type	2	5			7*
					37*

Table 6.24: *Number of artefacts per techno-functional sub-type and kinematics associated.*
** on one artefact 2 active edges were identified. Therefore, 36 actual artefacts compose the sample considered here.*

Only 20 artefacts ascribed to techno-functional groups displayed traces which allowed to infer the type of worked material. Not significant correlations between specific actions and techno-functional types was noticeable (Fig. 6.78). This data might be due to the limited numbers of artefacts of the sample considered. Longitudinal and transversal actions are distributed equally among the various techno-functional sub-types.

The two artefacts used to perform rotational movements are ascribed to group III. (1=III.a; 1= III.c). No preferences of definite sub-types to incise hard and soft materials was noticed.

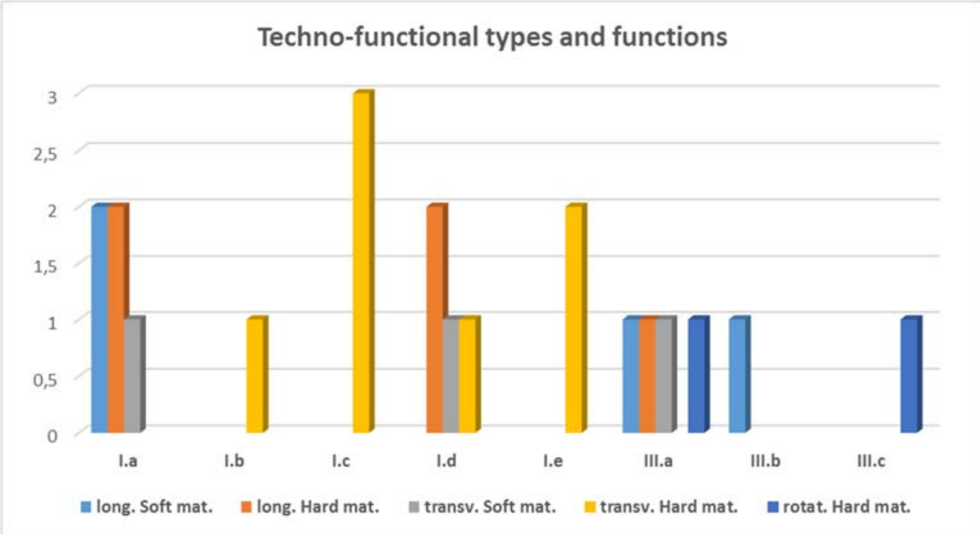

Fig. 6.78: *Correlation between techno-functional types, action performed and worked materials. Data is referred to the 20 artefacts ascribed to techno-functional sub-types whose traces allowed to infer the type of worked material.*

Chapter 7: The Payre site

7.1 Introduction

The site of Payre is located on the left side of the Rhône Valley, in Southern France (Fig. 7.1). It forms part of a karst complex of Jurassic and Cretaceous formations, near the Payre River, a tributary of the Rhône River, which is the biggest river of the Ardêche region. The first excavation of the site took place in the 1960s and systematic excavations began in 1990 (Moncel, ed., 2008). These extensive excavations, covering an area spanning from 30 to 70 meter square, yielded a 5 meter thick stratigraphic sequence comprising eight main units (G, F, E, D-C, B-A) (Moncel et al., 2009). Geological and stratigraphic studies revealed that the site was a cave originally, whose ceiling collapsed at the end of MIS 6.

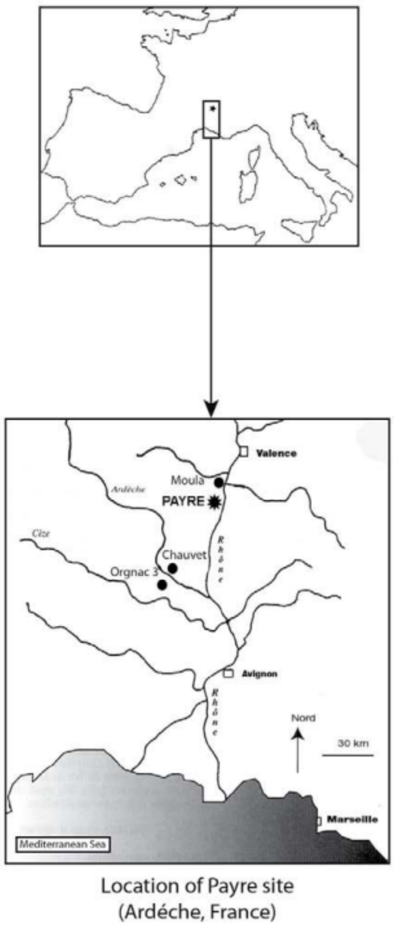

Fig. 7.1: *Geographic location of the Payre site (modified from Hardy and Moncel, 2011).*

The six units were characterised and several dates were obtained for each level. The stratigraphy has been described as follows, from bottom to top (Valladas et al., 2008):

- Unit G: composed of orange clays, numerous pebbles and blocks. It contains two main human occupational phases, lying on the stalagmitic floor;
- Unit F: formed by grey units intercalated with beds of rubble and clay. It contains four human occupations alternating with paleontological units dominated by bear remains;
- Unit E: formed by large blocks of limestone, connected to the partial collapse of the roof;
- Unit D-C: formed by brown clastic sediments;
- Units B-A: archaeologically sterile layers closing the sequence and composed of karst sediments.

Dates obtained through ESR, U-Th series, TL and TIMS methods provided a chronological span between MIS 8-5. More specifically, sub-levels Gb to Fa are dated to the end of MIS 8 and the beginning of MIS 7 and sub-levels E and D are dated to the end of MIS 6 and the beginning of MIS 5 (Valladas et al., 2008).

Human remains were found along the sequence, with main concentrations in units F and G. They have been identified as teeth and fragments of parietal bones and have been attributed to the Neanderthal lineage (Moncel and Condemi, 1997, 2007).

The analysis of the fauna assemblages revealed slight compositional variation between levels. It identified the presence of three main taxa all along the sequence, *Equus* sp, *Cervus elaphus* and *Bos primigenius* (Daujeard, 2008; Moncel et al., 2002) along with less abundant species (*e.g. Capreolus capreolus, Dama dama, Dicerorhinus* sp.). The faunal composition suggests a temperate climate and variability in the environmental conditions surrounding the site. These observations are confirmed by micro-faunal, avian and pollen analyses, which revealed the existence of semi-forest environments together with temperate and humid climate conditions (Moncel, ed., 2008; Daujeard and Moncel, 2010).

Among carnivores, the best represented species is *Ursus spelaeus,* especially in level F, which was interpreted as a bear den. Apart from the important presence of carnivores in this level, the paleontological and archaeological remains of the other levels suggest the presence short-term seasonal occupations by humans all along the sequence (Moncel, ed., 2008). Based on the animal bone representation and high presence of cut-marks on bones, the site could have been used as a butchering location. Besides, whole animal carcasses were brought to the site. The presence of several episode of in-situ knapping activity would suggest the presence of long or intense occupations.

The lithic assemblage is composed mainly of flint, followed by quartz, quartzite, basalt and limestone. Flint was collected from local and semi-local sources on the southern plateau, while quartz and quartzite could be found locally on the banks of the Rhône River. Discoid strategies were applied on flint, but no standardised products were obtained. All the stages of the *chaînes opératoires* on flint are present at the site. Retouched flint flakes comprised of scrapers and points (Moncel et al., 2009). Reduction sequences of other raw materials (quartz, quartzite and limestone) are incomplete and therefore they were only imported as pre-forms (unmodified items), cores or final products (large flakes, large-cutting tools) (Moncel et al., 2008).

The 155 pieces of quartzite form 5% of the entire assemblage. An interesting fact is that it is found through the entire sequence of the site and always as the final products (or blocks). Flakes are also present, all of them being unretouched.

The main reason to apply traceological analysis to this raw material here is to find out whether its introduction into the site as large tools has a specific significance. Moreover, through the comparison of traceological studies on quartz and flint material, we want to determine whether there was a differentiation in terms of tasks performed with different raw materials or not.

7.2 Previous functional studies on the Payre assemblage

The investigation of the function of stone tools from the Payre site has been an important research concern in the last few years. Both use-wear and residue analyses were applied on selected samples of the lithic assemblage. Different raw materials were subjected to use-wear analysis.

Only a limited number of quartz and quartzite samples were microscopically analysed in two recent Master theses (Borel, 2007; Borel et al., 2008; Martin, 2012). Due to the limited number of artefacts analysed (13 in Borel, 2007; 2 in Martin, 2012), it is difficult to propose a functional hypothesis to reconstruct the human occupations of the site. However, these works demonstrated the suitability of microwear analysis on both quartz and quartzite.

Regarding flint, the most abundant material at the site, flint convergent points and side-scrapers were systematically considered for functional analysis, combining morpho-metrical data and macro-trace analysis (Moncel and Chacón, 2008; Moncel et al., 2009; Chacón et al., 2016). A variety of the kinematics was observed: longitudinal, transversal as well as rotational movements were identified. Morpho-technical attributes were also considered in these studies. Regarding flint side-scrapers and quartz and quartzite tools, no clear relation between the morphology of the blanks and the activities performed was observed (Borel et al., 2008; Moncel and Chacón, 2008). On the contrary, flint convergent points showed a clear selection pattern based on the morpho-technical characters of the artefacts (Moncel et al., 2009). Triangular artefacts were used to pierce, and use-wear was generally found on the pointed end, while evidence of transversal and longitudinal actions was also found on their lateral edges. A clear relationship between retouch types and uses was also observed. In pointed artefacts used for piercing, retouch is usually marginal, while on edges used for scraping activities retouch can be limited to a part of the edge or can be all along the edge portion (when the frontal view is either concave or convex).

Residue analysis was also applied on flint material, where 182 artefacts were microscopically observed. A remarkable high percentage (68,7%) of this sample showed some kind of preserved residue (Hardy and Moncel, 2011). Evidence of starch grains, vegetal and animal fibres, animal hair and fish scales were observed. The identification of such a variety of residues on the same collection is a rare event, mainly because of preservation issues. The authors suggested that the presence of these kinds of residues could be connected to the dietary system of the Neanderthal groups inhabiting the site. The consumption of small preys (birds and rabbits), of starch plants and fish would prove that the dietary habits of Neanderthals were based on a differential spectrum of environmental resources. However, up to now, no consistent proofs of processing of avian carcasses have been observed in the faunal record (Rufá et al., 2017). The presence of fish residues is also difficult to interpret, as no fish osteological remains were found at the site.

7.3 Publication 7:

Pedergnana, A., Ollé, A., Borel, A., Moncel, M.H., 2016. **Microwear study of quartzite artefacts: Preliminary results from the Middle Pleistocene site of Payre (South-eastern France).** *Journal of Anthropological and Archaeological Sciences* (in press)., DOI: 10.1007/s12520-016-0368-2

In this paper, the results of a preliminary use-wear analysis of the quartzite assemblage of Payre are presented. A selection of a sample which includes different sized-artefacts was done in order to check the preservation conditions of the assemblage as well as to test the methodology designed within this thesis.

The results are promising and although more work is necessary to better assess the role of quartzite throughout the various occupational phases, they allowed us to propose preliminary hypotheses.

Figure 7.2 shows the metric characters of the samples analysed in comparison to the whole quartzite assemblage. The artefacts selected for analysis are representative of the general composition of the assemblage. Large tools as well as small unretouched flakes were considered for use-wear analysis.

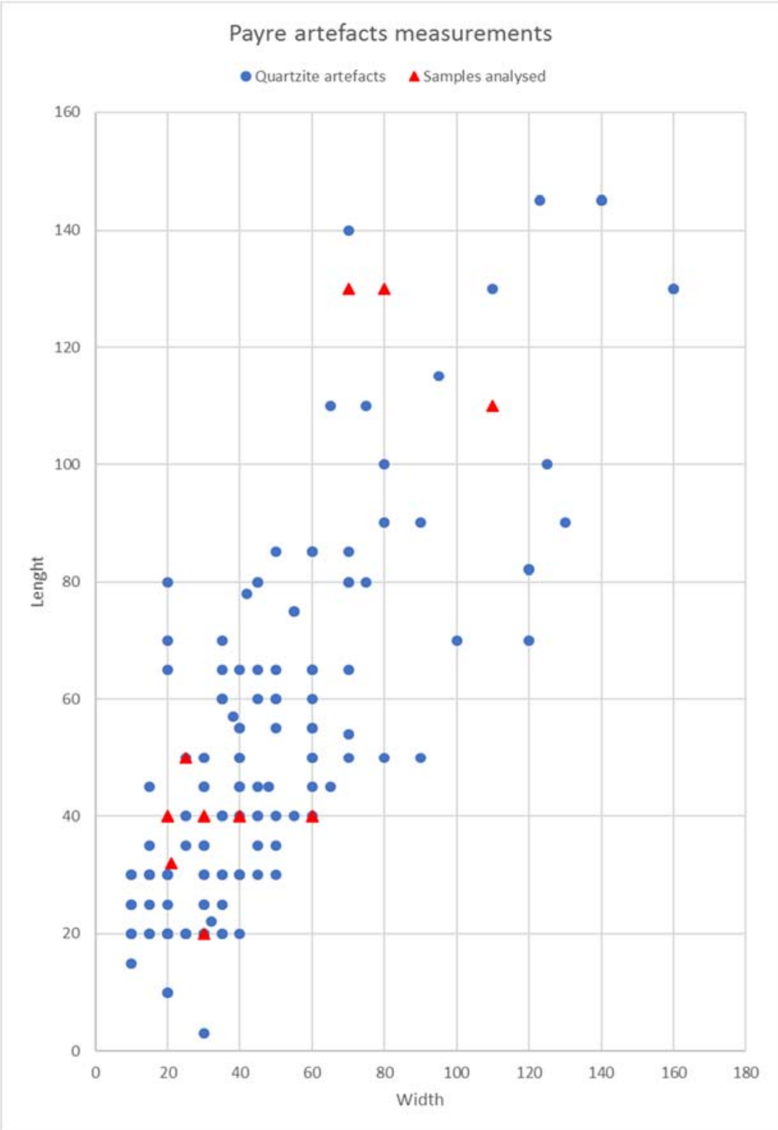

Fig. 7.2: *Graph showing artefact measurements of the sample analysed in relation to the entire quartzite assemblage of Payre. The sample submitted to microwear analysis is representative of the entire assemblage.*

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ORIGINAL PAPER

Microwear study of quartzite artefacts: preliminary results from the Middle Pleistocene site of Payre (South-eastern France)

Antonella Pedergnana [1,2,3] · Andreu Ollé [1,2] · Antony Borel [3] · Marie-Hélène Moncel [3]

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Abstract Preliminary functional results obtained from the quartzite assemblage of the Early Middle Palaeolithic site of Payre (South-eastern France) are presented. In an area rich in flint, hominins at Payre also collected quartzite in their local environment, specifically along the Rhône River banks. Although the Payre lithic assemblage is largely composed of flint, quartzite was introduced in the site mainly as large cutting tools knapped outside. This fact pointed out an apparently highly differential treatment of the raw material types available in the region. A major concern is to understand the reason why. Is there any functional reason for the introduction of those artefacts, perhaps to perform specific activities related to the toughness of quartzite? Or is there any functional differentiation among the various raw materials? Use-wear analysis is a useful tool for better understanding human technological choices and strategies of lithic raw material management. Before attempting to extensively apply use-wear analysis on the quartzite assemblage, we analysed a limited sample to evaluate the general surface preservation. A specific experimental programme with the same local quartzite was carried out in order to provide a reliable comparative reference for interpreting use-wear evidence on archaeological implements. Methodological difficulties related to use-wear analysis applied to quartzite artefacts are also discussed. Both Optical light microscopy (OLM) and Scanning Electron Microscopy (SEM) were employed in this study; however, interpretations were elaborated considering principally SEM micro-graphs.

The analysis of the archaeological material showed a good state of preservation of the surfaces with a low incidence of post-depositional alterations. The documented use-wear allowed us to identify the active edges, the kinematics and, more rarely, the worked material. Chopping activities were documented on two large artefacts suggesting a specific utility of those tools.

Keywords Lithic use-wear analysis · Quartzite · France · Payre · Early Middle Palaeolithic

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Introduction

As several authors have pointed out (Cristiani et al. 2009; Gibaja et al. 2002; Gibaja and Carvalho 2005; Igreja 2008; Mansur 1999), non-flint raw materials have not been thoroughly studied in the domain of use-wear analysis. Usually, they have been analysed employing the methodology designed for flint (or chert) artefacts (among others, Grace 1989; Keeley 1980; Van Gijn 1990; Vaughan 1985). The adaptation of the attributes observed on flint implements to the analysis of other raw materials may be misleading because this might introduce some errors and decrease interpretation accuracy.

The main reason why we detect distinct patterns of use-wear development and distribution on the various lithic raw materials employed in knapping activities is that they have extremely varied chemical and physical properties. Even among different flint varieties (therefore, the same raw material), a high use-wear intra-variability has sometimes been

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documented (Beyries 1982; Levi-Sala 1996; Lerner 2014; Vaughan 1985). Hence, it might be irrelevant to infer the function of non-flint implements by analysing the presence/absence and morphology/extension of use-wear features normally recognised on experimental tools made of flint. Consequently, the a priori definition of analogies among different lithic raw materials might force the interpretation of the functional evidence based on a simplistic deductive process which lacks strong analytical methods. So far, significant efforts have been carried out to develop particular methodologies for some non-flint raw materials such as quartz, basalt or obsidian (among others, Hurcombe 1992; Knutsson 1988; Richards 1988; Sussman 1988; see also Asryan et al. 2014; Knutsson et al. 2015; Kononenko 2011; de la Peña et al. 2013 for examples of recent applications), although in many cases without a clear continuity.

Regarding quartzite, the lack of specific methods and precise terminology is more evident, as the research applied to this rock has been quite intermittent. In regions where quartzite plays a significant role within lithic assemblages, scholars made significant improvements basically adapting the methodology defined for flint (Alonso and Mansur, 1990; Aubry and Igreja 2009; Cristiani et al., 2009; Gibaja et al. 2009; Hroníková et al. 2008; Igreja et al. 2007; Igreja 2008; Lemorini et al. 2014; Pereira 1993, 1996; Plisson 1986), and only in few cases the intrinsic peculiarities of this rock have been investigated (Beyries 1982; Clemente-Conte and Gibaja-Bao 2009; Gibaja et al. 2002; Leipus and Mansur 2007). In our opinion, a thorough characterisation of use-wear on quartzite is required to improve the knowledge of its mechanical behaviour under stress by means of specifically designed, carefully monitored and well-imaged experimental programmes (Pedergnana and Ollé 2014, 2016; Pedergnana et al. 2016).

Furthermore, the employment of the same terms to identify different use-wear traits might generate misunderstandings concerning both specific terminology and interpretation. Even if one is able to deal with this terminological confusion, communication among scholars is not straightforward and leads to an increasing difficulty to compare data from different archaeological contexts. Therefore, it is essential to reconsider the definitions and characterisations of use-wear on quartzite and provide a standardised terminology (Gibaja et al. 2002).

However, the underlined problems are not related only to terminology, but also to methodology. Generally, both low-power and high-power approaches are employed to investigate use-wear on lithic tools (for a recent revision, see for example Marreiros et al. (2014); Stemp et al. 2015). The suitability of the low-power approach has been intensively debated, due to the fact that scars can be the result of use, trampling and even retouching (e.g. Tringham et al. 1974; Levi-Sala 1996; Burroni et al. 2002). Additionally, if macro-scars are clearly visible on flint (and other fine-grained raw materials), they are quite difficult to observe on quartzite.

Specifically for quartzite, the high-power approach was thought to be unable to provide reliable results on use-wear (Grace 1989, 1990). This happens because the light reflection of a conventional optical microscope on extremely irregular surfaces (such as quartzite) prevents the formation of clear images. A consequent phenomenon is frequently observed in the form of a very intense bright diffraction halo, which sometimes makes the focusing of edges impossible. Although the employment of the DIC (Differential Interference Contrast) or NIC (Nomarski Interference Contrast) microscopy (Heath 2005) can be very useful to avoid light reflection of flat reflective samples (Igreja 2008, 2009; Knutsson et al. 2015), did not prove to be successful in producing similar results when observing quartzite (Pedergnana and Ollé, 2016).

In this article, we face the methodological problems described above, through the study of a set of quartzite tools from the early Middle Palaeolithic site of Payre (South-eastern France). At Payre, as in many other sites located in areas rich in flint, quartzite is often encountered in lower proportions, but may not be only a "secondary" raw material. In fact, large tools knapped outside and obtained from cobbles collected on fluvial terraces connected to the Rhône River were introduced into the site. Additionally, small quartzite flakes, possibly resulting from the rejuvenation of the large cutting tools, are also present. The question is: why were the large tools imported? Perhaps for a specific use related to the toughness of quartzite? Or perhaps it is not possible to find a clear functional differentiation among the various raw materials present at the site (flint, basalt, quartz, and quartzite). If so, quartzite would have been collected in an opportunistic way only in accordance to paleo-environmental constraints. It seems that it would have been easier for hominins to collect flint from the various outcrops situated on the southern plateau (5–30 km from the site) than to knap tough quartzite cobbles, even if they were available in large quantities less than 1 km from the site. Generally speaking, technological and morphometric analyses have pointed out a probable differential use of the various lithic raw materials at Payre during the different occupational phases (Moncel, 2008; Moncel et al. 2008). Considering the extensive availability of flint in the surroundings of the site (Fernandes et al. 2008), it is convenient to assume that there might have been clear reasons to employ a more tenacious lithic raw material such as quartzite for performing specific tasks.

To set up the bases for verifying this hypothesis, we analysed a sample of quartzite artefacts from different layers (dated from the beginning of MIS 7 to the end of MIS 6). Firstly, we aimed at verifying the feasibility of a future use-wear study on the whole quartzite assemblage by assessing the preservation state of the material. Secondly, we discuss until which point the preliminary archaeological results can be used to better understand the functional role of quartzite within the Payre lithic assemblage.

Materials and method

The Payre site corpus

Payre is an early Middle Palaeolithic site located in the Rhône valley, South-eastern France (Fig. 1). The site was initially a cave which later collapsed and became a rock shelter. The stratigraphic sequence is composed of eight main levels which are dated from the end of the MIS 8 to the beginning of the MIS 7 (levels Gb to Fa) and from the end of MIS 6 to the beginning of MIS 5 (levels E and D) (Valladas et al. 2008).

The quartzite assemblage represents less than 5 % (155 pieces) of the entire lithic collection. It is found throughout the archaeological sequence, even if it slightly decreases through time (Table 1) (Moncel et al. 2008). Large tools made of limestone, quartz and basalt were also documented all along the sequence. In each layer, large flakes and tools made of quartzite appear to have been knapped on cobbles away from the site, by means of unipolar and, more rarely, centripetal flaking methods. Cobbles were probably collected on the Rhône River banks, approximately 1 km from the site (Moncel et al. 2008). In addition to the large tools made from large flakes or cobbles, we can observe some small flakes considered either as evidence of rejuvenation of large tools or as an importation from knapping events that occurred off-site. All of them are unretouched flakes. Some large flint flakes were also introduced in the site from southern outcrops and used as large cutting tools, unretouched flakes or support for the knapping. However, most of the flint waste products are small in size (10 to 50 mm), obtained from flaked-cores or

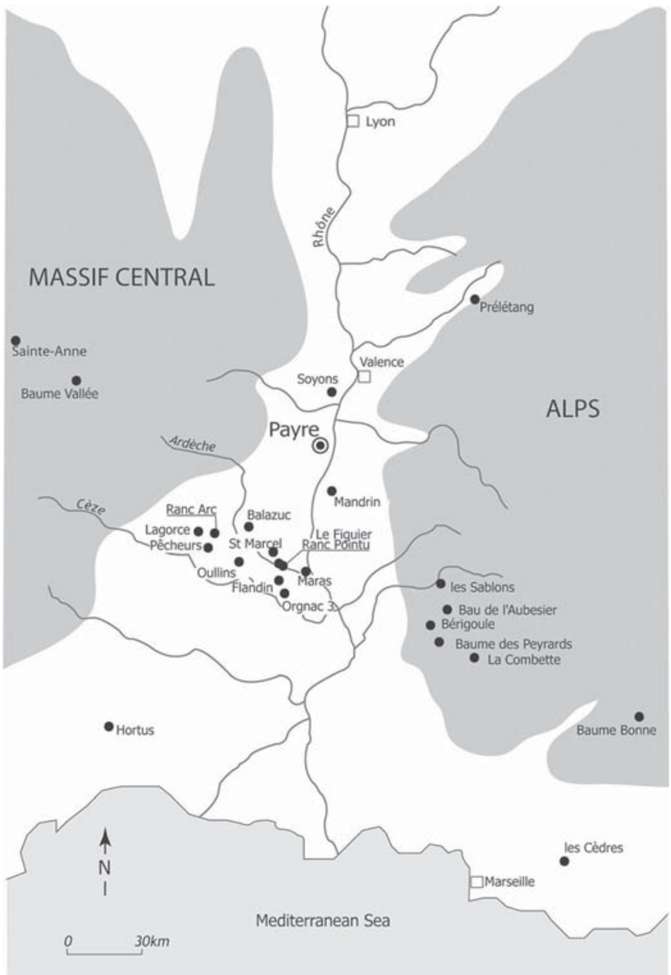

Fig. 1 Geographical location of the Payre site in the Rhône Valley (South-eastern France)

small nodules, by discoid methods, among others (Baena et al. in press). Few artefacts (10–15 % of the assemblage) are retouched into scrapers or points. For this study, a sample of 11 quartzite artefacts (simple flakes and large shaped tools) coming from levels D, F and G of the Payre site were selected. This preliminary sample was chosen taking into account the tool category, the macroscopic surface preservation and the presence/absence of cortex. Flakes with complete cortical surfaces were initially rejected, as it is more difficult to distinguish between natural and use-wear traits on cortical surfaces.

Functional study

Experiments

Experimentation has always been essential prior to any kind of functional analysis in order to carefully monitor and document use-wear and its distribution with regard to specific actions, kinematics and worked materials (e.g. Hayden 1979; Semenov 1964; Tringham et al. 1974; Vaughan 1985). As each lithic raw material has its own characteristic micro-topography, structure, hardness, toughness, etc., the use-wear appearance might be very different with respect to the rock type used. For this reason, it is very important to create specific use-wear repository for each raw material type and to carefully describe the resulting use-wear traits.

The available reference collection for use-wear on quartzites comes from previous experiments, including different varieties of quartzite coming from Northern Spain (characterised by different granulometry, grain disposition, grain compaction, inclusions, metamorphism degree) (Ollé 2003; Pedergnana and Ollé 2014, 2016; Pedergnana et al., 2016). This wide and ongoing experimental programme was sequentially documented. Experimental pieces were normally employed for various stages of use, keeping some variables fixed, and were then observed by means of different microscopic techniques. The controlled variables were the used edge angle, the performed movement, the contact angle, the contact material and the number of strokes. Changes due to use were detected and later described by using resin replicas of the original (un-used) edges. Through the comparison of the same points before (on the replicas) and after (directly on the tools) use, the attestation of the observed wear due to use was more secure (Ollé and Vergès 2008, 2014) (Fig. 2a, b).

We added a series of experiments with a variety of quartzite from the surroundings of the Payre site (Table 2) to the set of experimental data previously obtained (Pedergnana and Ollé, 2016). Our aim was to identify characteristic traits related to this local rock type and to evaluate the degree to which some observations carried out on the archaeological material may be conditioned by them. In an effort to provide a reliable reference collection, we reproduced a limited number of activities, divided into transverse and longitudinal movements. The worked materials were fresh hide, bone (*Cervus elaphus*) and wood *(Quercus ilex*). The elapsed time was sufficiently long in order to document well-developed wear connected to the different contact materials. Specifically, flakes were used for a defined number of strokes in order to compare the extension of the wear.

The experimental flakes were knapped from a single quartzite cobble collected near the Payre site. This variety of quartzite exhibits brownish-yellowish colouring, a macroscopically medium-grained structure, and a good ability to fracture conchoidally, despite the presence of some internal joint planes. Microscopically, the Payre quartzite used for our experiments has a general heterogeneous structure typical of polycrystalline materials (Fig. 3a). The semi-angular grains of crystalline quartz vary significantly in size (10–200 μm) and sometimes display a consistent porosity. The presence of neoblasts (small quartz grains neo-formed during the process of metamorphism) (Pettijohn et al. 1987) strongly affects the micro-topography (Fig. 3c, d). Planes dislocations, slopes, holes, and other discontinuities are frequent and contribute to the general surface irregularity. Compaction of quartz grains is quite high due to a medium-high degree of metamorphism (Fig. 3b).

Table 1 The Payre quartzite assemblage divided into technological categories and level of provenience (modified from Moncel, 2008)

Levels	Gb	Ga	Fd	Fc	Fb	Fa	E	D	Tot.
Entire cobbles								1	1
Broken cobbles		1		1?			1	3	6
Pebble-tools		3				6		1	10
Flakes <15–20 mm					3				3
Flakes >20 mm	3	44	1	1	4	20	12	38	123
		15 flake-tools							
Tools on large flake	2		1?			6		2	11
Cores and broken cores						1?		–	1
Total	**5**	**48**	**2**	**2**	**7**	**33**	**13**	**45**	**155**
% in level	*0.8*	*1.4*	*0.3*	*0.4*	*0.9*	*1.1*	*3.9*	*1.7*	

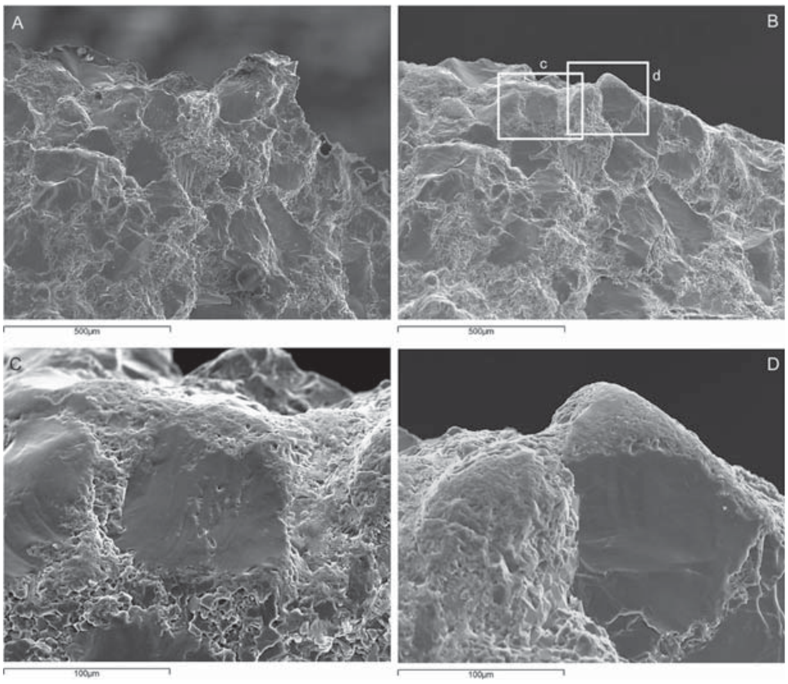

Fig. 2 An example of polish formation after ca. 20 min of wood scraping (500 strokes). From the comparison with the edge prior to use and the same point after use (**a**, **b**, original mag.: ×100, scale bar: 500 µm), it is possible to see that only the highest parts of the micro-topography are affected by wear. The depressions between the largest quartz grains are unchanged after use. The most affected points are highlighted by squares and displayed at higher magnifications in **c** (original mag.: ×500, scale bar: 100 µm; the upper border of the quartz grain is polished and tiny striations perpendicular to the edge are visible) and **d** (original mag.: ×500, scale bar: 100 µm; well-developed polish spots on the more prominent asperities)

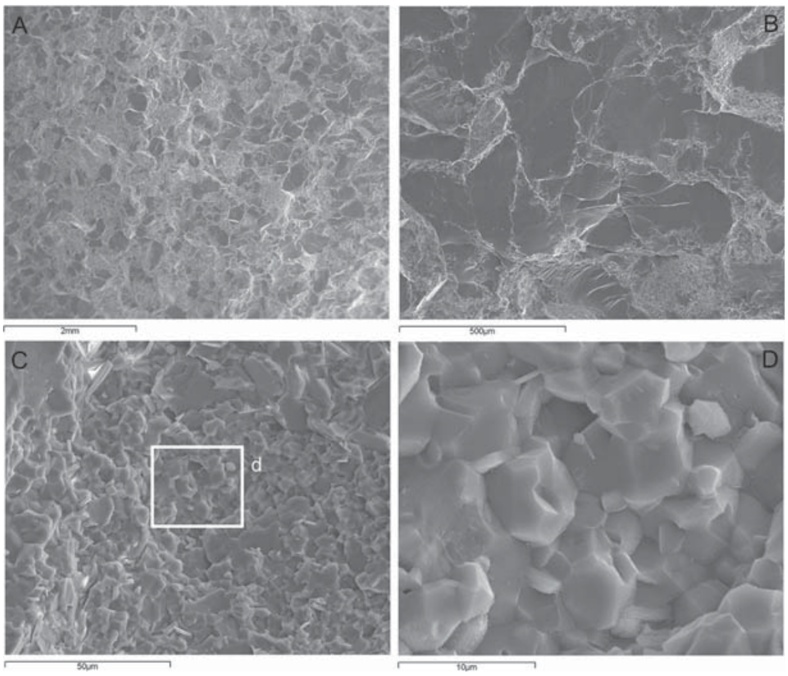

Fig. 3 SEM micro-graphs showing the texture of the Payre quartzite variety employed in our experimentation. The texture of metamorphic rocks might be indicative of the metamorphic processes which they have been subjected to. General view (**a**- = original mag.: ×20, scale bar: 2 mm) and details of bigger quartz grains (**b** = original mag.: ×100, scale bar: 500 µm) and neoblasts (**c** = original mag.: ×1000, scale bar: 50 µm; **d** = original mag.: ×4000, scale bar: 10 µm). **d** An enlarged view of the portion contained in the *white square* in **c**. Usually, neoblasts are not affected by use-wear. The general uneven surface of heterogeneous rocks strongly influences the distribution of use-wear and also the way it forms. The analysis of the natural rock topography helps to understand the distribution of wear and therefore to better interpret use-wear, when present

Table 2 Experimental tools and main controlled variables using a quartzite variety collected near the archaeological site of Payre. Edges with similar angles were selected for use and were not subsequently retouched

Reference N.	Edge angle	Activity	Movement	Contact angle	Contact material	Species	N. of strokes
EXPAY1-01	45°	Cutting	Unidirectional	90°	Fresh wood	*Quercus ilex*	1000
EXPAY1-02	45°	Sawing	Bidirectional	90°	Fresh Wood	*Quercus ilex*	1000
EXPAY1-03	45°	Scraping	Unidirectional	60–70°	Fresh Wood	*Quercus ilex*	500
EXPAY1-04	50°	Planing	Unidirectional	30–50°	Fresh Wood	*Quercus ilex*	500
EXPAY1-05	45°	Scraping	Unidirectional	60–70°	Fresh hide	*Cervus elaphus*	4000
EXPAY1-06	45°	Scraping	Unidirectional	60–70°	Fresh bone	*Cervus elaphus*	500

Use-wear analysis

For observations, we employed an optical microscope Zeiss Axio Scope A1 with a set of four objectives (5×/0.13 HD, EC epiplan 10×/0.2. HD, LD epiplan 20×/0.4 HD DIC, LD epiplan 50×/0.5 HD DIC) allowing direct observation from 50 to 500×. Images were taken with a 5MP DeltaPix digital camera (Invenio 5SII model). Although the DIC (Differential Interference Contrast) system was always used at 200× and 500×, the quality of the pictures did not always significantly improve.

Detailed analyses were performed using two SEM microscopes: a JEOL JSM-6400 and a FEI Quanta 600, both equipped with an INCA system from Oxford Instruments for digital image acquisition and microanalysis by electron probe (EDS). SEM-high vacuum observations require to cover the sample with thin layers of conductive materials (gold or carbon), performed through sputtering systems. We usually deposited a 30A layer of gold on each sample. Although most of the observations were done at high vacuum mode, we employed the FEI Quanta 600 low vacuum mode when we faced problems with the gold coating. When extremely uneven surfaces present deep holes or irregularities, it is very difficult to obtain a continuous layer of gold, which implies that conductivity is interrupted. If the sample is not totally conductive, the resulted image is not of good quality because the SEM detectors cannot read any signal. In all the cases, SEM images were obtained from detecting secondary electrons (with an ETD-Everhart-Thornley detector).

The combined use of optical and electron microscopes in functional studies on flint has proved to be very useful (Borel et al. 2014; Monnier et al. 2012). From our point of view, the use of SEM is essential to investigate use-wear formation processes because it allows reaching higher magnifications comparing to optical devices, and therefore detecting even slight modifications of lithic surfaces (Pedergnana and Ollé 2014, 2016). Another reason which makes the use of SEM an advantageous choice for analysing quartzite implements is that polish on quartzose materials is hard to document with optical devices (Beyries 1982; Grace 1990; Igreja 2009). In fact, the incident light of a regular optical microscope disturbs the detection of surface modifications when analysing highly reflective rocks such as quartz and quartzite.

Results

Experimental results

When observing quartzite with regular optical microscopes, we faced many technical problems. For instance, areas requiring very high depth of field are not always successfully imaged (even through extended focus systems) (Figs. 4b and 5f). Sometimes, it is not even possible to focus the edge outline (Fig. 4b, d). The DIC was sometimes used and proved to be helpful in removing the halo from quartz crystals (Cristiani et al. 2009; Igreja 2008, 2009). According to our observations, this system can be very useful to analyse relatively uniform reflective surfaces (milky quartz and rock crystal) (Fig. 13), but not very practical when it comes to irregular coarse materials (Pedergnana and Ollé, 2016, Fig. 22: b). In fact, the prisms mounted in the DIC system are designed to enhance contrast when observing transparent samples (Murphy 2001). The adjustment of the light caused by the Nomarski prisms does not provide the expected results when observing colourful and coarse samples (Fig. 5b, d). Big, flat and whitish crystals are the most affected parts when the two prisms are rotated. Consequently, those crystals are more easily observable and the DIC successfully removes the halo (Fig. 4c). Even so, wear is extremely hard to locate and difficult to image (the crystal must be extremely flat, hence bearing a uniform reflection of light which can be differently orientated through the use of DIC). Consequently, wear must be present on those "visible" crystals (visible and imaged with OLM). Crystals being oriented in a way that renders their OLM imaging impossible are not observable. Therefore, wear possibly present on those crystals is lost and cannot be integrated in the final interpretation.

SEM overcomes those technical problems by providing extremely high quality in-focus images. As images are not formed through light, light reflection is not a problem. Then, the actual shape of crystals and wear is easily distinguishable (Figs. 4c, d and 5a, c, d). Striations are also more difficult to

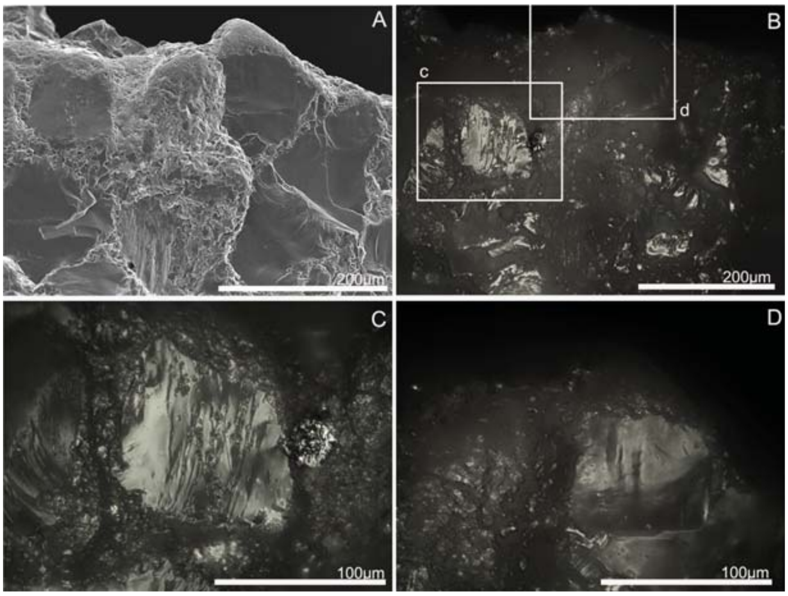

Fig. 4 Comparison of the same experimental wood polish imaged through SEM (**a**) and OLM (**b**). Close-ups of the main points of interests are taken (**c**, **d**). The comparable SEM images are found in Fig. 3 (**c**, **d**). Original mag. and scale bar: **a**, **b** ×200, 200 μm; **c**, **d** ×500, 100 μm. Extended focus images were obtained through the stitching of 30 pictures

image with OLM, where only those with an adequate orientation are visible by rotating the DIC prisms (Fig. 5h). Attempts to obtain extended focus images of the striations shown in image 5 were performed, but no satisfactory results were obtained. The shortest striations were not visible during OLM observations, while SEM succeeded in imaging them (Fig. 5g). Furthermore, greater details are not possible to obtain with regular optical microscope, while with the higher SEM magnifications the morphology of striations is better evaluated (Fig. 6c).

A similar visibility problem is documented in relation to polish (Knutsson, 1988; Grace, 1990). Since it is not brilliant (as on flint), it is not detected from OLM images (Fig. 5b, d, f), while from SEM micro-graphs it is evident (Fig. 5a, c, e). Sometimes, differences in structure can be deduced (Fig. 4c, d), but only after a direct comparison with SEM pictures (Fig. 3c, d). Based on those observations, SEM appears more suitable than OLM for observing wear on quartzite. Hence, interpretations in this work were mainly based on SEM micrographs, which were undoubtedly clearer.

We are aware that mineral composition, internal structure (fabric) and texture (granulometry, grains cohesion, orientation and porosity) have a great influence in the development of wear, because surface modifications on quartzite, as on other rocks, always start to occur on the highest parts of the topography. As a result, a structural description of the rock prior to any functional analysis is an unavoidable step and petrographic analyses, if possible, is also recommended (Pedergnana et al., 2016).

Knowing that the use-wear features can largely differ depending on the variety of quartzose materials, we must describe how the variety chosen at Payre is generally affected by use. Essentially, the typical brittle behaviour characterising quartzose rocks was observed (Clemente-Conte and Gibaja-Bao 2009; Igreja 2008; Knutsson 1988). It was also observed that use-wear initially forms on the highest parts of the topography (Fig. 3b) (Gibaja et al. 2002; Pedergnana and Ollé 2014, 2016), propagating to larger areas as soon as the duration of the action increases. Compared to other varieties previously studied (Pedergnana and Ollé 2016; Pedergnana et al. 2016), minor differences of wear appearance were noticed. Probably because of the lower isotropy of this variety, use-wear develops with more difficulty than in other varieties previously investigated (i.e. well-developed meta-quartzites, which are completely sorted rocks, characterised by granoblastic texture, strong grain cohesion and low or no porosity between grains). In fact, in this intermediate phase the anisotropic organisation of crystals and the sparse presence of neoblasts (between large quartz crystals) seem to slacken the development of use-wear.

Nevertheless, we were able to observe the presence of furrow striations (for terminological issues see Ollé et al. 2016; Pedergnana and Ollé 2016) within the grains surfaces (Figs. 5g, h and 6b, C) and some polished surfaces in the form of rare spots on the rim (Fig. 6d), which sometimes covered the interstitial spaces between grains (Fig. 6a, e). This kind of striations was considered to be typical of wood working (Knutsson 1988), but they are associated also with other materials. Then, considering all the worked materials included in

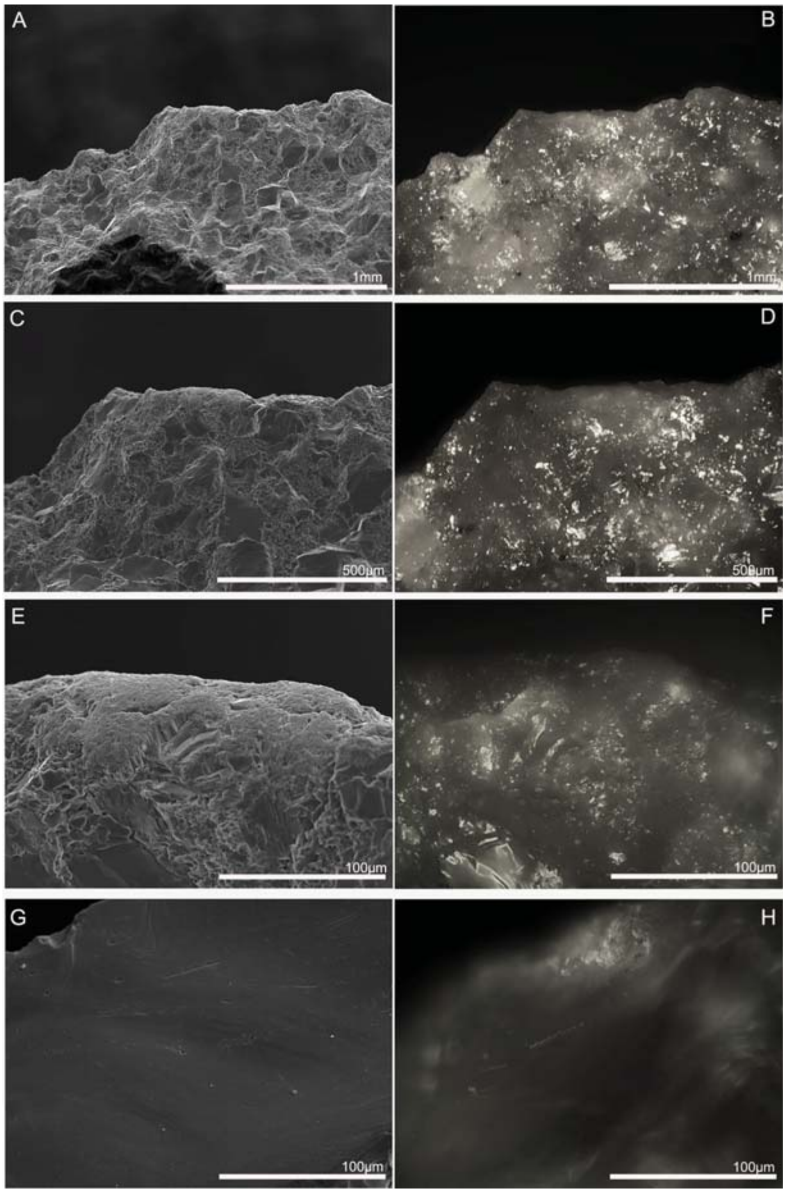

Fig. 5 Comparison of two points of interest seen at different magnifications imaged through SEM (*left column*) and OLM (*right column*). The rounded edge on an experimental tool used to plan a wood stick is barely visible in OLM pictures (**b**, **d**), while it is much clearer in the SEM micrographs at equivalent magnification (**a**, **b**). Details of the polish texture are easily imaged through SEM (E), while the same spot does not seem to be polished under the optical microscope. Furrows striations are clearly distinguishable in the SEM picture (**g**), while in the OLM one only two linear features are visible (**h**). Original mag. and scale bar: **a**, **b** ×50, 1 mm; **c**, **d** ×100, 500 µm; **e**–**h** ×500, 100 µm. Extended focus images were obtained through the stitching of: 50 (**b**), 25 (**d**), 20 (**f**) pictures

the experiments, we recognised a slight modification of the quartz grains' borders as the dominant use-wear feature. It is displayed both in the form of slight polish (Fig. 6b) and of edge crushing (Fig. 6f).

Moreover, polish can be distinguished on the basis of its visual appearance when observed with SEM. Polish type has been regarded as a good optical indicator to distinguish among different worked materials, at least concerning flint (Keeley and Newcomer, 1977; Keeley 1980; Vaughan 1985). Bearing in mind that we do not dispose of all the parameters normally used to differentiate among different polish types while analysing quartzite (i.e. brightness and linkage degree of the

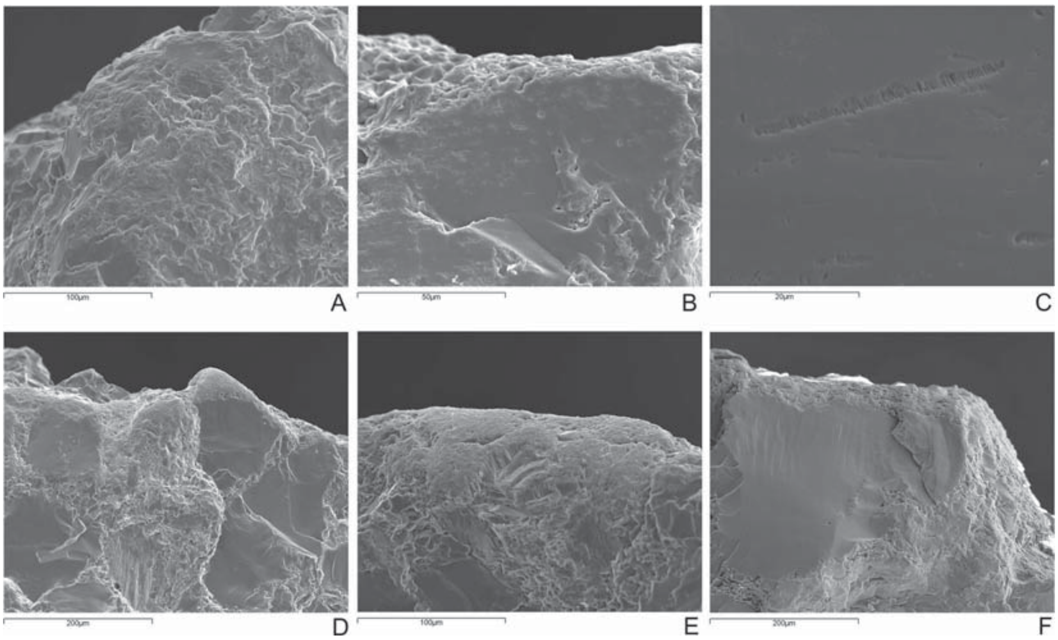

Fig. 6 Experimental use-wear features related to wood working. **a**, **b** Cutting, unidirectional movement. **c** Sawing, bidirectional movement. **d**, **e** Scraping, unidirectional movement. **f** Planing (low working angle), unidirectional movement. Striations occur mainly in the form of furrows (**c**), on the flat surfaces of the grains (**b**) and they are more abundant on implements used for longitudinal activities. Edges used for transversal activities show more or less developed polished spots (**d**–**f**). Pictures were taken with a SEM (high vacuum mode). Original mag. and scale bar: **a**, **e** ×500, 100 μm; **b** ×1000, 50 μm; **c** ×2500, 20 μm; **d**, **f** ×250, 200 μm

polished surfaces), we should rely more on the specificities of the polish topography and try to correlate them with the intervened materials. Brightness is not a usable criterion in this study because light reflection on quartzite surfaces (while using OLM) is extremely high and prevents the detection of polished spots. Curiously, polished areas on quartzite are dull, whereas some quartz crystals are very brilliant due to their variable orientation. This fact is one of the main obstacles for the detection of use-wear on this type of rock through OLM (Grace 1989, 1990). We also discarded the linkage degree of the polished areas, frequently used in use-wear studies, (González Urquijo and Ibáñez Estévez, 1994; Juel Jensen 1994; Rots 2010) due to the slow development of polish and the low occurrence of very extensive polish areas on quartzite.

The formation of polish on quartzite, independently of the intervened material, is thought to be an essentially abrasive phenomena (Pedergnana and Ollé 2016). However, some plastic deformation can be observed on the smoothest areas. Differences on polished surfaces in these experiments are not as clear as those documented for other quartzite varieties: in this variety, definite topographical traits generally associated with a specific worked material sometimes overlap features obtained after the processing of different worked materials. In fact, the smooth character of wood and bone polishes is found here with no consistent differences (Fig. 7). From previous observations on other quartzite varieties (Pedergnana and Ollé 2014, 2016) we described the polish texture derived from bone working as having characteristic undulations on the very smoothest parts. This characteristic seems not to be present on bone polish found on the Payre variety. Bone polish (Fig. 7d) appears to be smoother than wood polish (Figs. 6a, b and 7b). Wood polish can also be rough in some cases (Fig. 6e), resembling the one found on edges used to scrape hide on other quartzite varieties (Pedergnana and Ollé, 2016, Fig. 11). This is very likely due to the fact that wood polish is more widespread on the general topography of the rock, affecting also the smaller granular fraction, while bone polish seems to be limited to the larger quartz grain surfaces. It appears that textural variability of polish on this quartzite variety is not enough to discriminate among different worked materials. Therefore, under those circumstances, wear distribution assumes a predominant role. In fact, even if the observed polished spots are not generally linked together in an extensive plot, the distribution of the spots varied with respect to the activity performed and, to a much lesser degree, the worked material.

On implements used for performing longitudinal activities, polish distribution is quite patchy, sometimes asymmetrical

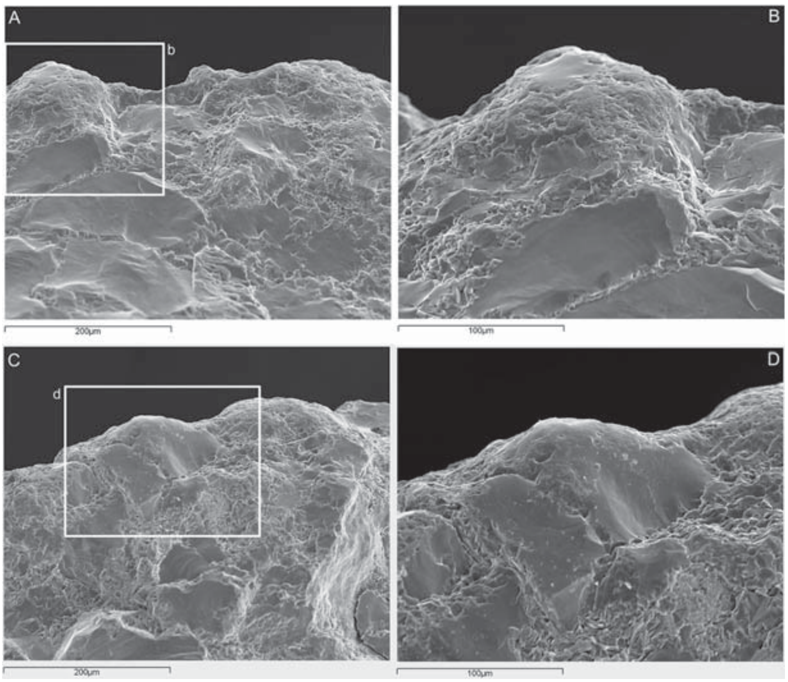

Fig. 7 Different visual appearance of polish surfaces on quartzite, deriving from scraping fresh wood (**a** = original mag.: ×250, scale bar: 200 μm) and bone (**c** = original mag. 250, scale bar: 200 μm). Details of the polish textures at higher magnifications in **b**, **d** (original mag.: ×500, scale bar: 100 μm) (close-up of the areas in the *white squares*). The textural aspect of polish alone might be misleading for a correct correlation with the intervened material. High-magnification details of the micro-topography of the polish spots and their disposition are necessary and complementary information

regarding the two faces. Since sawing activities (bidirectional) implied an active edge held at ca. 90 degrees, polish tends to form on both sides of the edge (ventral and dorsal); but for cutting (unidirectional) actions the angle is usually more acute (60°–80°), as every stroke implies a readjustment of the edge on the worked material. For this reason, polish areas are mostly distributed on the face bearing the major contact with the worked material (Fig. 6a). Sawing actions produce clear micro-scars on the borders of the large quartz grains and deeper striations (furrow type) than in cutting actions; these furrow striations appear to be wider and more abundant. When it comes to transverse actions, this asymmetric distribution of polish is more evident being quite curiously more invasive on implements used for scraping (higher contact angles, Fig. 6e) than for planing (lower angles, Fig. 6f).

Archaeological results

Despite the restricted sample of quartzite artefacts analysed ($n = 11$), coming from the three main levels of the Payre site (Table 3), we could compare our experimental data with the archaeological material. The assessment of the preservation conditions was important to understand whether use-wear analysis on the entire assemblage was feasible or not. We provide a confidence scale (from 1 to 5), in order to numerically assess the reliability of our interpretations. It is essential to provide a quantitative method with which to associate a reliability level to each tool/interpretation. This value is based on several factors, such as the degree of post-depositional modifications, the development degree of the use-wear or the presence/absence/combination of clear traits. It remains a subjective and relative value which does not allow to compare with other assemblages but which does gives a better idea of the state of each artefact and about the weight of their interpretation (e.g. Borel 2012: 209–210; Borel et al., 2016; Rots et al. 2016).

The implements analysed are well preserved, although in some cases post-depositional surface modifications (PSDM) hampered the detection of use-wear ($n = 1$). PSDM are produced from the same phenomena than use-wear (e.g. abrasion, surface polishing, scratching, which are induced by the contact with sediment and water) and therefore, the visual appearance can be equifinal (Pedergnana and Rosina, 2015). Generally, they are identified considering their location on the tools' surfaces (aleatory striations, polished areas in the centre of the tool or in the totality of the edges, etc.) as well as the intensity of wear (deep striations, incredibly well-developed polish, pits, intense fracturing, etc.).

Concerning the remaining part of the analysed sample, 8 artefacts exhibited some type of functional evidence, whereas two did not show any use-wear, suggesting that they were not used.

Table 3 Functional results: on pieces whose surfaces are slightly or non-affected by post-depositional modifications, used edges were always identified. In the majority of the cases the relative motion and the worked material hardness were deduced. In few cases, use-wear was clear enough to permit the identification of the worked material with a considerable high reliability degree. PSDM stands for post-depositional surface modifications. The reliability degree is subjectively measured on an increasing scale from 1 to 5

Reference N.	Layer	MIS	Technological category	Edge angle	Measures mm	Activity	Movement	Worked material hardness	Worked material type	PSDM	Reliability level
PAY L9 D 257	D	6/5	Unretouched flake	40°	37-57-12	Cutting	Longitudinal	Medium	–	–	3
PAY J11 D3 70 D	D	6/5	Unretouched semi-cortical flake	55°	36-20-9	Sawing	Longitudinal	Medium-hard	Wood or bone	X	2
PAY M4 F1–1	F	8/7	Bifacial tool	55/65°	127-64-46	Chopping	Percussive	–	–	–	3
PAY L6 F1 201	F	8/7	Large retouched semi-cortical flake	60°	116-76-45	–	Longitudinal/ (percussive?)	–	–	–	3
PAY L5 F2 238	F	8/7	Large tool	55°	102-106-38	Chopping	Percussive	Medium-hard	Wood	–	3
PAY L8 F5 630	F	8/7	Unretouched flake	70°	38-38-13	Scraping	Transverse	Medium-hard	Wood	–	4
PAY N7 G1 456	G	8/7	Unretouched flake	–	49-21-10	No use-wear traces	X	4			
PAY M6 G1 207	G	8/7	Retouched flake	45°	49-68-9	–	–	Soft	–	–	3
PAY M4 G2 439 Ga	G	8/7	Unretouched flake	45°	32-21-5	Scraping	Transverse	Medium-hard	–	–	4
PAY N3 G3 628	G	8/7	Broken flake	40°	28-23-8	Sawing	Longitudinal	Hard	–	X	3
PAY N7 G5 Ga	G	8/7	Unretouched flake	–	36-35-18	No use-wear traces	–	4			

The implements exhibiting use-wear traces are retouched and unretouched flakes, ranging from 28 to 127 mm in length. Use-wear was also found on two large tools, one bifacially shaped, and the other exhibiting removals all around its circular perimeter.

Movements

Transverse actions were registered on two artefacts, longitudinal ones were present on four pieces and possible percussive activities are proposed for the two large tools. We were able to infer the general hardness categories of the worked material, ranging from soft materials (1) to medium (1), medium-hard (4) and hard ones (1). Specific worked material types were recognised in three cases (wood/bone).

Location and angles of the used edges

Angles of the used edges are comprised between 40 and 70°. When the identified action was connected to scraping activity (n = 2), use-wear is concentrated on the distal edge, whereas for cutting actions (n = 4) the used edge was always lateral.

Polish appearance and activities

The clearest documented evidence concern three artefacts showing well-developed use-wear patterns connected with scraping medium-hard materials and sawing hard materials (wood or bone) (Fig. 6). Basically, the primary adopted criterion for the identification of the worked material is the "polish" appearance on SEM micro-graphs (rough or smooth). Rough polish is indicative of wood or hide working, while extremely smooth polish could be connected with bone or vegetal processing.

Besides the appearance of polish, we carefully considered distribution and minor topographical details of the polished areas. Thus, we were able to identify wood polish (Fig. 8b) from the one related to bone. Sometimes, polish texture and distribution were not clear enough to identify the worked material type and a general label, such as medium-hard worked materials, was proposed (Fig. 8d).

Specifically regarding one semi-cortical flake (Fig. 8: 3), we identified polish evidence related to both wood and bone. Most of the polish areas found on one of the edges are easily associable with our experimental observations on implements used for processing bone. Nevertheless, some polished topographical traits are remarkably similar to wood polish of our experimental samples (Fig. 8f). Consequently, a possible double function may be proposed for this artefact. Based on the experimental observations and pointing out the ambiguous nature of polish topography on this specific quartzite

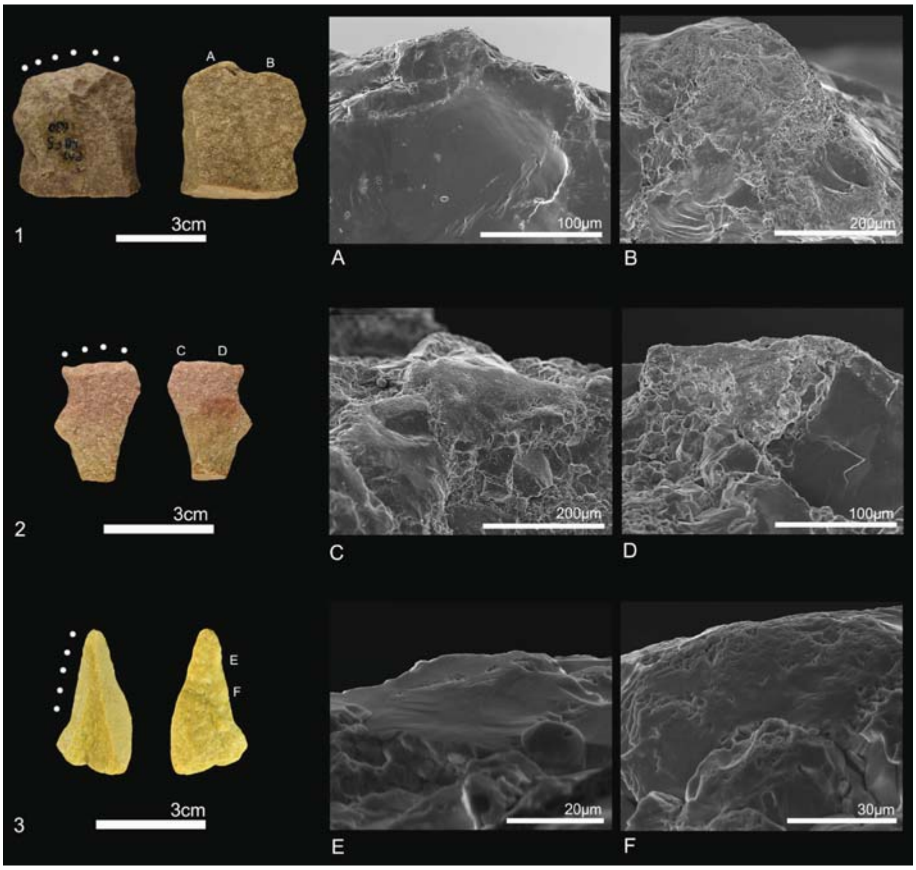

Fig. 8 Three analysed archaeological tools. *1* PAY L8 F5 630, use-wear connected with wood working is distributed all over the transversal edge, in some cases affecting just the very crystals borders (**a** = original mag.: ×500, scale bar: 100 μm) or expanding inward due to a transversal motion (**b** = original mag.: ×250, scale bar: 200 μm). *2* PAY M4 G2 439 Ga, use-wear on the distal part of the implement, whilst the lateral edges are free from any microscopic modification. Several rough polished spots have been identified (**c** = original mag.: ×250, 200 μm; **d** = original mag.: ×500, scale bar: 100 μm). *3* PAY J11 D3 70 D, in this case the used edge corresponds to the lateral one (*left*), where smooth polish, normally found on implements used to process bone has been observed (**e** = original mag.: ×2000, scale bar: 20 μm; **f** = original mag.: 1500, scale bar: 30 μm)

variety, we chose to be more cautious and we only propose a generic label for the worked material (medium-hard).

A small broken flake shows continuous crushing scars of the right edge (Fig. 9: 1), accompanied by rare parallel and short striations, which allow to propose a longitudinal action on hard materials. Polish was present on very tiny spots and was not well-developed. A longitudinal action was also identified on the right edge of a semi-cortical flake (Fig. 9: 2), while the opposite edge, as well as the interior surface, are free from post-depositional modifications.

Another singular artefact exhibiting a clear use-wear distribution is a bifacially shaped tool (Fig. 10). Use-wear is concentrated on the apex, and descends to some extent along the left edge. Use-wear appears to marginally affect the original micro-topography of the rock through minor abrasion phenomena. For this reason, we think that the worked material might pertain to the soft category (meat, vegetal matter, etc.) or that the contact with a possible medium or even hard material might have been limited due to short chopping/adzing actions. To respond to this question, further experimental activity focused on the utilisation of large cutting tools is necessary.

A large semi-cortical retouched flake (Fig. 11) exhibits three macro-scars on the cortical right edge, pointing out to a possible forceful action. On the distal part of the same edge and on the proximal portion of the opposite edge, polish and

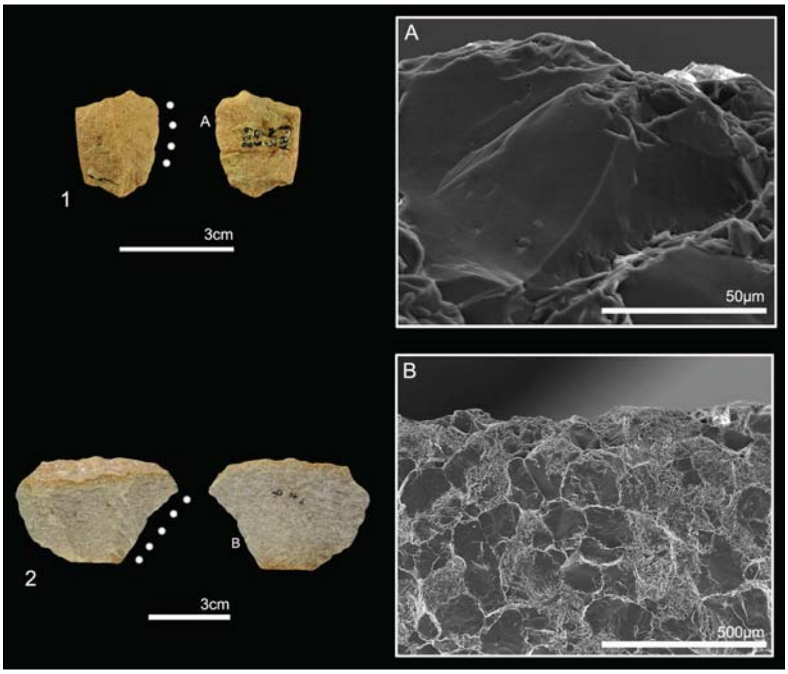

Fig. 9 Two analysed archaeological tools: *1* PAY N3 G3 628, where use-wear revealed one used edge to perform a longitudinal action on hard materials. *2* PAY L9 D 257, where a cutting action on medium hardness material was identified. Original mag. and scale bar: **a** ×1000, 50 μm; **b** ×100, 500 μm

very narrow linear features allowed the identification of longitudinal motions (Fig. 11a, b). Images of the interior of the surface (Fig. 11c) showed absolute fresh crystals, which demonstrates the absence of PDSM, reinforcing the adscription of the described wear to use.

Macro-detachments are also visible in several portions all around a large circular tool (Fig. 12), for which a percussive motion has been also proposed. In this case, besides a continuous crushing (Fig. 12a), a specific portion on the proximal left edge shows intensively polished areas, displaying a texture and other diagnostic features connected to wood polish (Fig. 12b). As in previous cases, the interior parts showed fresh surfaces.

Discussion

The presence of coarse-grained lithic materials within prehistoric collections has been generally perceived only as the result of paleoecological constraints, simply based on the raw material availability. This belief, together with a real lower suitability of quartzite for knapping compared to chert, has contributed to the idea that quartzite was just a secondary flakable material for a rapid knapping and for makeshift activities. Based on this paradigm, from the appearance of traceology (Semenov 1964), functional studies focused on the characterisation of the lithology commonly referred to as chert. From that time onwards, methodological improvements were obtained from the study of chert collections (Keeley 1980; Levi-Sala 1996; Vaughan 1985). However, based on experimental observations, it can be stated that the methods and use-wear descriptions developed for fine-grained rocks can no longer be employed to analyse coarse-grained lithic materials, being extremely heterogeneous. Hence, it is essential to deeply investigate each raw material type and define clear and distinctive patterns related to use (Lerner et al. 2007; Lerner 2014; Pedergnana et al., 2016).

Regarding quartzite, so far only very few use-wear descriptions are available. The accessible research pointed out the inadequacy of the low-power approach to investigate use-wear traits on quartzite, particularly edge scarring, one of the main parameters used to infer both the movement and the worked material hardness (Mansur 1999; Gibaja et al. 2002; Gibaja and Carvalho 2005; Leipus and Mansur 2007; Igreja 2008; Clemente-Conte and Gibaja-Bao 2009). Exceptionally, edge micro-scarring appears to be a good indicator of the performed action when dealing with quartzite cortical flakes (Cristiani et al. 2009).

Moreover, the formation of quartzite polish is slower than on chert, giving raise to completely different distributional patterns (Clemente-Conte and Gibaja-Bao 2009; Stemp et al. 2013). In fact, clear polish on quartzite forms exclusively

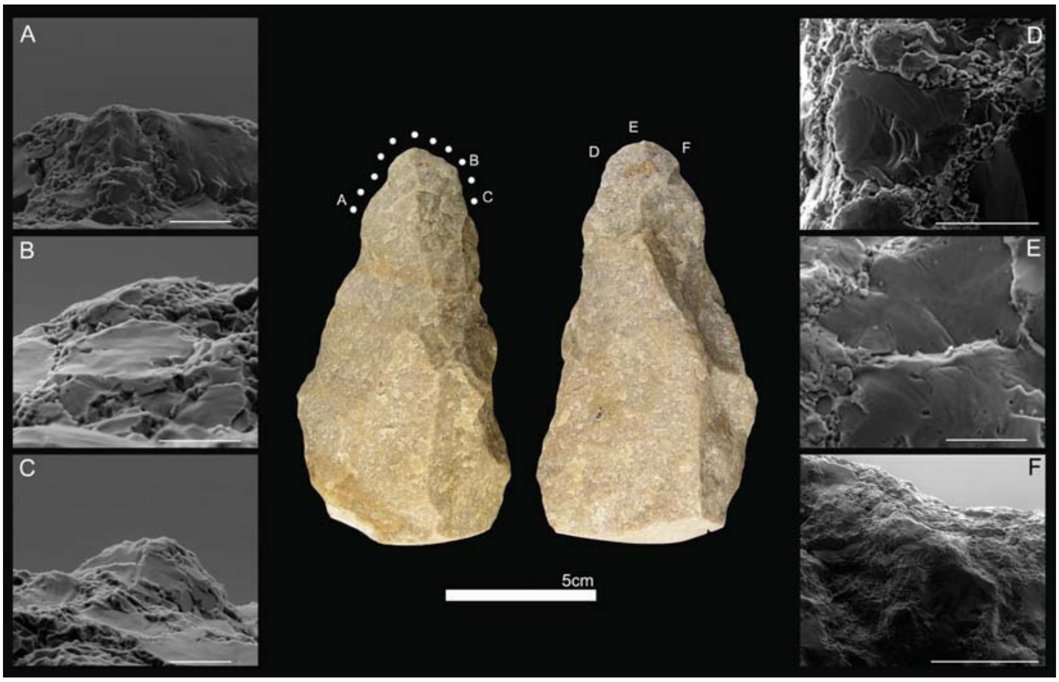

Fig. 10 Use-wear evidences on a quartzite bifacial piece (PAY M4 F1-1). Use-wear is concentrated on the apical part, both on the dorsal and ventral faces and it consists in a very slightly abraded rim (**a**, **b**, **c**, **f**) and of a not well developed abrasion of the quartz grains borders (**e**, **f**). Original mag. and scale bar: **a**, **c** ×1500, 50 μm; **b**, **e** ×2000, 50 μm; **d** ×1250, 100 μm; **f** ×260, 500 μm

under particular conditions: long duration activities in connection with very abrasive worked materials (Mansur 1999; Gibaja et al. 2002; Clemente-Conte and Gibaja-Bao 2009; Cristiani et al. 2009; Pedergnana and Ollé 2014, 2016).

At times it may be difficult to properly image use-wear on quartzite with an optical device. This can happen not only because of the light reflection of the quartz grains but also because of the very irregular topography of quartzite. DIC microscopy can be useful for flat samples as rock crystal (Fig. 13b). Milky quartz also has relatively flat and large crystals with similar orientation, which explains the quality improvement of DIC images (Fig. 13c, d). Unfortunately for quartzite studies, results are not as satisfactory as for the other two raw materials (Fig. 13e, f). The smaller crystals (50–100 μm) of quartzite have multiple faces which provokes a differential reflection of light. For this reason, the DIC system is not always a valid solution. Although it always provides better images, some areas still remain invisible under the OLM.

Conversely, a regular SEM is very useful for analysing quartzite because it avoids light reflection of the quartz crystals, which is the main obstacle in optical light microscopy. Moreover, it is possible to document even the tiniest polished spot thanks to extremely high magnifications achieved by SEM (Borel et al. 2014; Pedergnana and Ollé 2014, 2016). Another advantage of SEM is that it has a high depth of field compared to classical optical devices. However, although this specific obstacle of optical microscopes is being progressively solved with different focus variation systems on optical and digital microscopes, only confocal microscopy seems to be able to satisfactorily image micro-topographical details of quartzite surfaces (Stemp et al. 2013). Quartzite tools can be easily observed with a SEM, instantly going from low to very high magnifications, without the need of changing the orientation of the observed zone.

Conversely, under optical microscopes tools surfaces require to be in a certain orientation to the light source to be visible. In fact, the samples have to be constantly moved to find the proper axis, and optical parameters (light intensity, DIC rotation, etc.) have to be continuously adjusted, which can be very time-consuming. Despite the application of these technical stratagems, very frequently there are edge portions which simply cannot be observed and imaged. This implies that if such as “optically invisible or unobservable” portions bore use-wear traces; the functional information would be lost. For all of those reasons, SEM provides more reliable results than OLM when observing quartzite tools. Thus, only after having formulated proper and specific methods for the studied

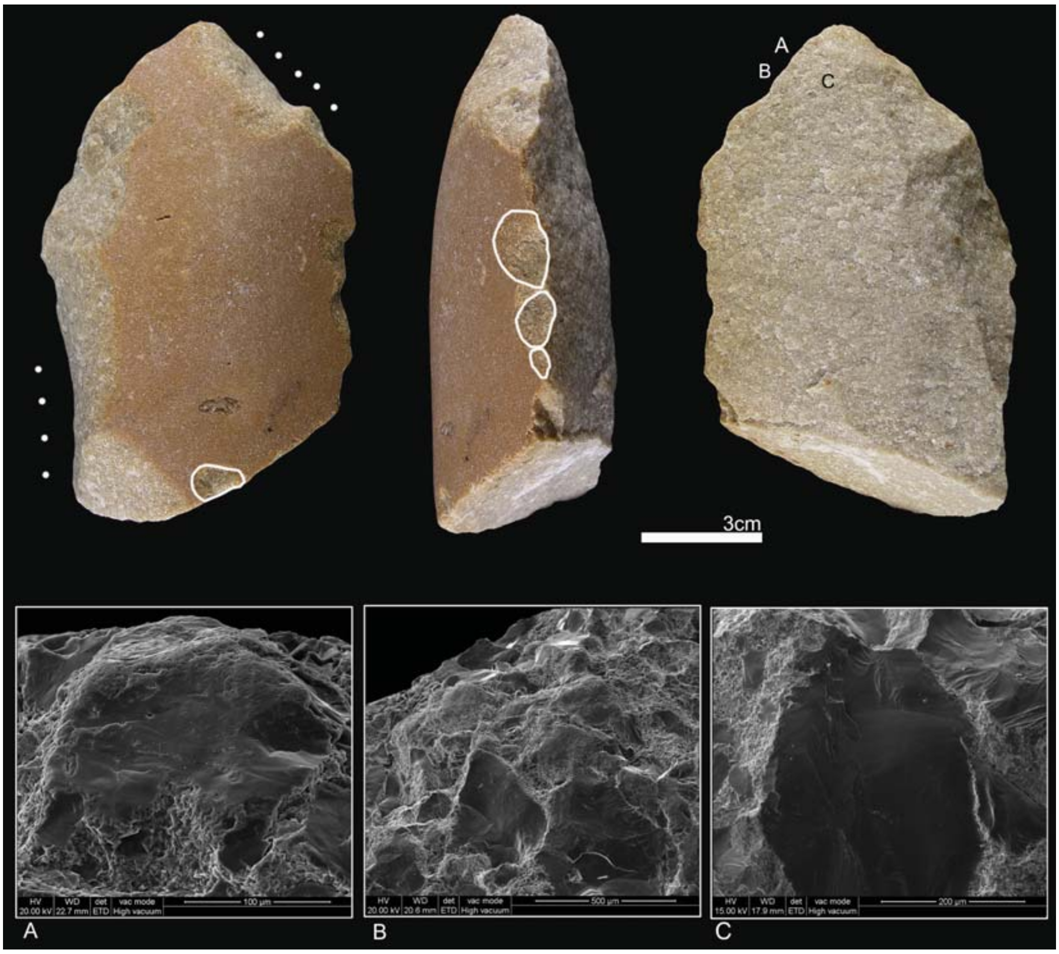

Fig. 11 A large retouched semi-cortical flake (PAY L6 F1 201) showing macro-detachments on its cortical edge. On the retouched portion of the same edge use-wear indicates a longitudinal action (**a**–**c**). Use-wear was poorly developed and the worked material was not identified. Original mag. and scale bar: **a** ×130, 100 μm; **b** ×250, 500 μm; **c** ×640, 200 μm

raw material, one should attempt to analyse archaeological collections to try to deduce behavioural aspects of prehistoric groups through functional data.

Despite the sparse attention given to quartzite in the past, we should remember that it is found throughout the entire human evolutionary pathway within very different geographic and chronological contexts. Therefore, by overcoming the idea that quartzite was a second grade material, we propose to take into consideration the opposite view, which sees in the composition of a lithic assemblage as a direct consequence of a human predetermined selection process. In fact, knowing that quartzite is one of the more tenacious rocks knapped throughout prehistory, the reduction of quartzite blocks might have required more energy in terms of physical as well as cognitive approaches. Physical, considering the requirement of a stronger force (F = ma), almost certainly leading to a selection of hammers with a larger mass (m). Cognitive, as it is more difficult to control quartzite than chert fracture, the obtaining of predetermined products might have implied a major psychomotor effort. Those assumptions are particularly intriguing in the specific case of Payre, considering the richness of flint, both on the surrounding plateaux and on the Rhône River banks. Besides, we face a very profitable geographic position where flint outcrops are very abundant and easily available. This abundance is indeed reflected in the composition of the Payre lithic assemblage. The reduced

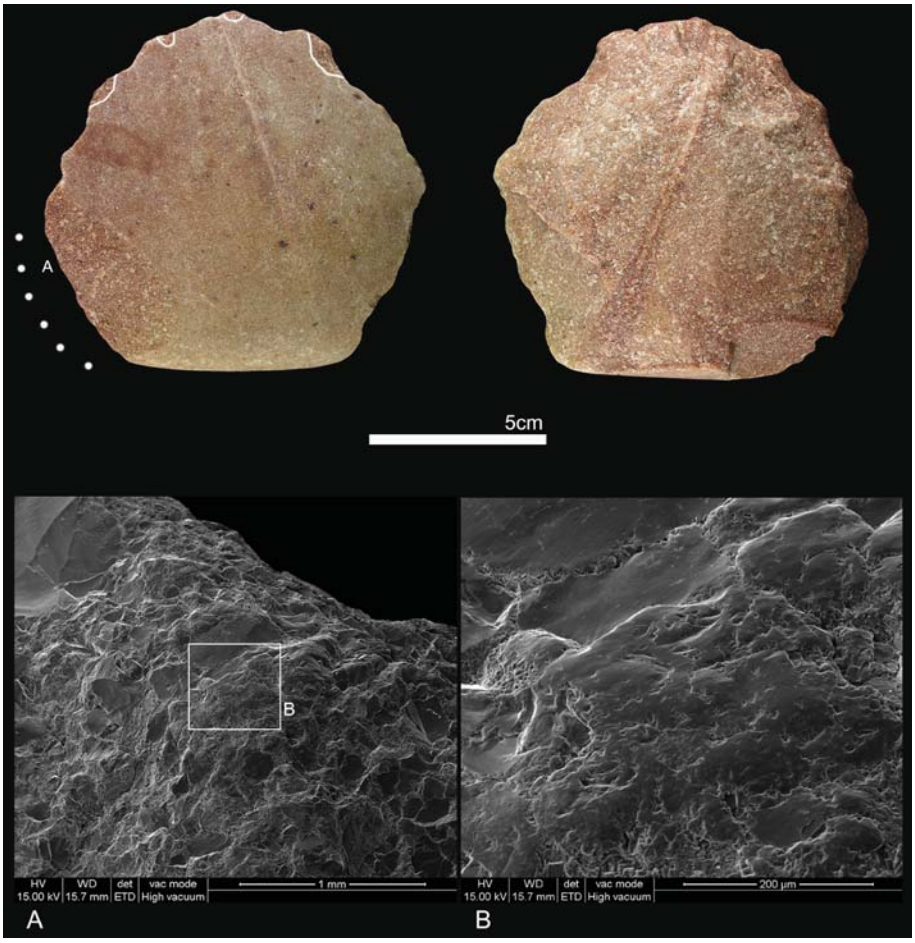

Fig. 12 A large tool (PAY L5 F2 238) was found with evidences pointing out to percussive actions on wood. Macro-scars are visible on the cortical edges. In the interior parts, no post-depositional modifications were found. Original mag. and scale bar: **a** ×125, 1 mm; **b** ×640, 200 μm

presence of quartzite (characterised by different *chaînes opératoires* compared to those related to flint) and the occurrence of basalt (with a technological treatment similar to that documented for quartzite) led us to hypothesise that non-chert raw materials played a specific role at Payre site that we have to understand, being part of the Neanderthal behaviour. Both quartzite and basalt large flakes and tools seem to have been knapped outside and subsequently imported. Consequently, we are facing two facts: (1) the selection of local raw materials in addition to good quality flint; (2) outside debitage with those local materials, while the short distance from the site could have allowed a direct production at the site. Some small quartzite pebbles were also introduced in the site (perhaps used as hammers) as well as a large number of variable size basalt cobbles which were not knapped later.

Preliminary data from micro-wear studies suggest several activities performed with quartzite tools, similar to those observed on flint (Moncel and Chacón-Navarro 2008; Hardy and Moncel 2011). Apparently, quartzite flakes were not employed for specific activities or to work particular materials, except from the large cutting tools. Anyhow, particular residential strategies perhaps demanded the production of big tools employing raw materials other than flint, more tenacious and resistant. It might have been the need to perform specific tasks, possibly percussive ones as those documented for two large tools, for which the use of flint was not suitable enough. We have to keep in mind that the toughness of quartzite could have helped to exploit large herbivore carcasses at the site (Moncel, 2008) (breakage of thick bones or dismembering of carcasses, etc.). It is also possible that the use of various

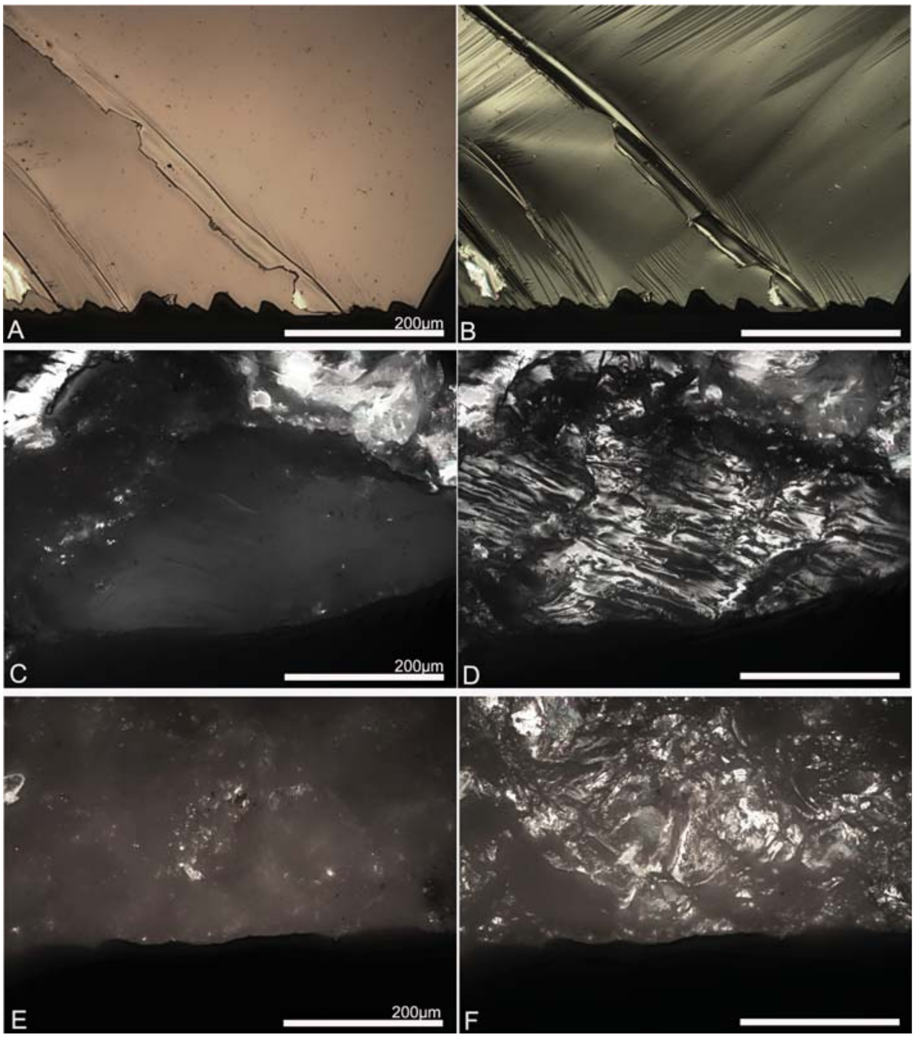

Fig. 13 OLM observation of different quartzose raw materials with (*right column*) and without (*left column*) the use of DIC (Differential Interference Contrast Microscopy). The exact points on rock crystal (**a**, **b**), milky quartz (**c**, **d**) and quartzite (**e**, **f**) are imaged. Although the use of DIC incredibly improves the quality of the pictures in all the cases, best images are obtained for flatter materials (**b**, rock crystal). Details of flat quartz crystal on milky quartz become visible when DIC is used (**d**). Quartz grains in quartzite are comparatively smaller and more irregular. They bear numerous and distinctly oriented faces, which render the use of DIC less effective. The edge itself is also more difficult to focus, due to the residual bright halo (**e**). All the micro-graphs are taken at ×200 with a 200-µm scale bar. Also, the coarser the specimen, the higher number of pictures is necessary to obtain satisfactory in-focus images (**a**, **b** = 10; **c**, **d** = 30; **e**, **f** = 40)

raw materials to perform the same activities without clear functional reasons was a traditional behaviour of the human groups occupying Payre. In other terms, the use of quartzite could be a wider manifestation of the already discussed complex behaviour characterising Neanderthal groups since the MIS 8, displaying varied and complex exploitation strategies of several environmental resources (Hardy and Moncel 2011).

Obviously, more work is needed to be able to reach our main archaeological objective, which is the understanding of the differential use of lithic raw materials by Neanderthals at Payre over time. Larger samples of the quartzite assemblage should be analysed and results will be compared with functional data available for other materials as flint (Moncel et al. 2008, 2009; Hardy and Moncel 2011) and quartz (Martin 2012).

Conclusion

A main concern of this contribution was the improvement of the use-wear analysis accuracy on quartzite. A new step in the creation of a specific method for the study of quartzite from a functional point of view was provided. What emerged is that at least a restricted experimental activity employing the same raw material composing the lithic assemblage of the studied site is always desirable, with the main purpose of identifying specific use-wear patterns related to it. Advantages and disadvantages of two microscopic techniques (OLM, SEM) when quartzite tools are analysed were discussed and SEM proved to far better fulfil the needs of use-wear analysis.

As a case study, we analysed a selected quartzite sample coming from the Payre site (France). After having observed a general good state of preservation of the archaeological material, we consider the analysis of the whole assemblage to be very promising. Preliminary functional results were presented, underlining the utilisation of several quartzite artefacts to work different materials (wood and bone). Wear was not always sufficiently developed to permit its ascription to a specific worked material. However, the identification of continuous modifications on specific portions of the tool edges allowed the identification of the active edges in all the cases. The interpretation of the active edges was specifically reinforced when the internal surfaces and the prominent ridges of the tool surfaces were free from post-depositional modifications. In some cases, particular features as the wear distribution or striations provided clues on the tool kinematics.

At this point of our research, we do not dispose of statistically valid data to clearly interpret the role of quartzite within the Payre lithic assemblage. This will be the main objective of our future research. In fact, functional analysis performed on the quartzite assemblage will allow us to understand the differential use of lithic raw materials throughout the entire sequence of the site. Furthermore, by comparing functional data of the various raw materials present at the site (flint, quartz, quartzite and basalt), we will provide a more accurate reconstruction of the lithic raw material exploitation by the Neanderthal groups residing at the Payre site. In this framework, technical behaviours related to the raw material selection will be better understood.

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Chapter 8: Synthesis of results and Discussion

8.*1 Summary of results*

The main aim of this research was to build up a reference collection of use-wear on quartzite surfaces. To respond to this need, sequential experiments were carried out and the ways in which use-wear forms and develops were observed and later described. A secondary objective was to initiate a reference collection of micro-residues of the worked materials by analysing the surfaces of the experimental tools before cleaning them.

Both use-wear and residues were analysed by means of multiple microscopic techniques to underline advantages and disadvantages of each of them. Besides, we wanted to assess the most suitable combination of microscopic techniques which provides the best results and guarantees trustworthy interpretations.

Optical Light Microscopy and Scanning Electron Microscopy were systematically used to document use-wear and residues on experimental artefacts. Confocal Microscopy was also occasionally used to provide quantitative data of polished surfaces.

The issue of contamination in microscopic studies on lithic artefacts was also addressed. Some common contaminants were analysed with the same microscopic techniques (OLM, SEM) and an effort to provide a thorough description of them was done. A deep characterisation of contaminants is thought to help traceologists to recognise them during their own analyses and to discard them from functional interpretations of the archaeological record.

Another important issue was to discuss the vicinity of technological and traceological studies, which have been usually maintained separated until now.

8.1.1 Use-wear and residue reference collections

This use-wear reference collection for quartzite includes experimental tools used on a variety of materials (animal and vegetal matter). It is composed of 46 tools, the majority of which were not retouched. In fact, only 4 artefacts were subjected to secondary retouched before being used. 81 sequential experiments were carried out and the systematic observation of the same micro-surfaces allowed us to monitor the development of wear throughout the various stages of use. 4 tumbling experiments were also carried out in order to address the appearance of traces related to soil movements and hydraulic transport.

All observations were performed after having acquired deep knowledge of the nature of the lithology studied. Quartzite is a metamorphic rock, mainly originating from quartz-rich sandstones (quartz-arenites). After being exposed to high temperatures and pressures, the resulting rock is characterised by an equigranular (consisting of minerals or clasts of approximately the same size) structure (Tucker, 2001). Pure quartzite is white; the variety of colours displayed by quartzite is a consequence of minor amounts of impurities being incorporated with the quartz during metamorphism. Although a quartz-rich sandstone can look similar to quartzite, a fresh broken surface of quartzite will show breakage across quartz grains, whereas the sandstone will break around quartz grains. Sometimes, a silica cement might deposit around the quartz grains, which could resemble a sort of 'matrix'. In the description of sedimentary rocks, the term matrix is used to define the finer-grained sedimentary material, in which larger clasts are embedded. As quartzites comprise only metamorphosed rocks, whose structure has completely lost all sedimentary relicts and has rearranged in an equigranular mosaic, no matrix can be present. If matrix is observed on a geological specimen, therefore it cannot be quartzite. Macroscopically, sandstones resemble quartzite and microscopically, they may present both matrix and larger grains or clasts. Moreover, silica or calcite cement may redeposit around the quartz grains of sandstones. The only case where two size populations are observed among quartz grains on quartzites, is when there is neoblasts (newly formed grains during metamorphism) growing. Neoblasts are quartz grains significantly smaller than the large (often visible to the naked eye) grains of quartzite. However, differences in the main measurements of regular quartz grains and neoblasts do not have the same proportions of the clasts and matrix in clastic rocks.

Almost all available functional studies on quartzite describe use-wear found on crystals and on matrix differently. Specific features (*e.g.* rough, domed polish, or flat) are said to be found only either on quartz crystals or on the matrix found between them (*e.g.* Alonso and Mansur, 1990; Mansur, 1999; Hroniková et al al., 2008; Clemente-Conte and Gibaja-Bao, 2009; Lemorini et al., 2014; Berruti et al., 2016). Based on geological definitions, matrix cannot be found in quartzites, therefore no use-wear can form on it. If the smallest fraction of quartz grains on quartzite is interpreted to be the matrix, our results can be compared to those published elsewhere.

Our observations did not corroborate the clear difference of traces described on large quartz grains and on the smaller fraction. No significant differences were observed, with the only exception that sees less developed traces on the smaller fraction than on large crystals. Hence, no differences in polish texture are found on the two different grain-size populations, as described elsewhere (*e.g.* Alonso and Mansur, 1990; Clemente-Conte and Gibaja-Bao, 2009; Lemorini et al., 2014). Polish develops in the same way on big and small crystals, and texture depends mainly on the contact materials. The observed lower rates of polish on the smaller fraction (neoblasts) is thought to depend on the general micro-topography of quartzite. Neoblasts are found between the quartz grains and generally they are located on lower heights than the upper surface of large crystals. Because polish forms firstly always on the highest parts of the micro-surface, large grains are the most affected by polish. When polish is present on sufficiently large areas, it then extends to lower areas, affecting also the small-grained fraction (if it is present). Basically, what matters is the extreme irregularity of quartzite (and other coarse-grain rocks). If the elapsed time of the action is not sufficiently long for polish to be form on large areas, it will then be present only on reduced areas coinciding with the highest parts of the edge portion which was in contact with the worked material. The irregular micro-surface of quartzites is characterised by "valleys" and "hills" and polish is usually encountered only on the "hills". This is why polish takes more time to form and expand to large areas than on fine-grained rocks (Clemente-Conte and Gibaja-Bao, 2009:97).

Linear features (striations) are only present on large and flat crystals, a fact which has been previously observed (*e.g.* Gibaja et al., 2002; Clemente-Conte and Gibaja-Bao, 2009). The reason why striations can only form on relatively large (60-100 μm) and flat surfaces is easily understandable. When a rock particle is dragged across the surface during the performance of a task, it is capable of incising it only if the exerted pressure is high enough. Therefore, the said particle would maintain enough contact with the surface by exerting a constant amount of pressure only on flat surfaces. Conversely, on angular surfaces, rock particles are easily ejected from the interstitial medium composed of the worked material and the rock particles embedded in it.

Different descriptive parameters were defined for each action performed (kinematics and worked material) after the analysis of all experimental tools. Linear features are the only suitable parameter to define kinematics, although the disposition of other traces, mainly scars might add some additional information.

Polish was then defined based on its visual appearance and two broad categories were described. Smooth and rough polishes form through contact with different materials. Sometimes, a combination of the two textures may be found on the same artefact (*e.g*. dry skin, wood).

The 'corrosion' of crystals, claimed to form after contact with abrasive materials, was not observed (Clemente-Conte and Gibaja-Bao, 2009; Lemorini et al., 2014; Berruti et al., 2016). We did observe extremely rough textures of the polished areas resulting from the processing of abrasive materials (*e.g.* skins), but no signs of corrosion were observed. As corrosion is defined as the gradual degradation of materials (usually metals) through chemical reactions, it is difficult to understand how it can be measured with a microscope. Hence, the meaning of this term given by the authors who employ it to describe worn surfaces on quartzite is not straightforward in our opinion. Therefore, it is difficult to make comparisons of the published data with our own results.

Furthermore, traces related to soft animal matter have been described as "barely recognisable" (Berruti et al., 2016:117), underlining the presence of only under-developed polish and few striations (Gibaja et al., 2002:80). In other studies, use-wear related to the butchering activity has only been observed at magnifications higher than 400x and better imaged with an ESEM (Cristiani et al., 2009:15). Our observations confirm the under-development of polish and the scarcity of linear features on experimental artefacts used in butchering activities. The softness of the worked material is certainly the main cause for the few traces observed.

Furrows are the most frequent type of striations on quartzite. They mostly originate from contact with hard materials, but they are also present on tools which worked soft materials. The highest concentrations of furrows were observed on artefacts which performed longitudinal actions on wood. In such cases, entire flat quartz grains were found completely covered with relatively long furrows, parallel to the used edges. On tools used to modify soft animal matter, furrows were extremely short. This is particularly true for transversal actions. It seems that edges performing transversal movements have more reduced contact with the worked material than those involved in longitudinal actions.

In general, excluding the processing of wood, furrows (as other linear features) are very rare on quartzite. They are present only on very flat surfaces, generally large crystals, and on prominent topographical zones. Striations are more likely to form where some drastic changes in the topography, such as ridges or depressions, occur.

Other kinds of linear features, such as sleeks, scratches and grooves, were observed, but less frequently than furrows. Moreover, some sleeks are the result of the obliteration of previously existing furrows (Ollé et al., 2016). This observation was only possible thanks to the sequential monitoring of the same portions of surfaces (Ollé and Vergès, 2014).

Partial Hertzian-cones were never observed on experimental samples; they only appeared on archaeological tools. They are interpreted as originating from strong post-depositional soil movements. Interestingly enough, we did not observe the same signs on the artefacts which were submitted to tumbling experiments. This might be the result of the short time of the experiments (20 and 30 hours).

Another challenge in the use-wear analysis of quartzite implements is related to the brittle behaviour of this rock (Knutsson 1988a). In fact, edges break continuously during use and micro-chips are detached from them. As a consequence, some portions of the edge bearing use-wear are lost (Clemente-Conte and Gibaja-Bao, 2009:97). This fact might complicate the obtaining of functional interpretation of the archaeological record. We should consider that some artefacts displaying no apparent use-wear might actually have been used but their use-wear might have been lost due to micro-fracturing.

8.1.2 Residue reference collection

A reference collection of micro-residues of the worked materials worked with stone tools was initiated through the analysis of 23 experimental artefacts before cleaning them. 21 of them were manufactured from quartzite within the scope of this research, while 2 of them were made of flint and took part of an experiment designed to respond to questions other than those presented in this work. Nevertheless, results connected to the observation of some kinds of residues (avian tissue and feathers) were of extreme interest and fitted precisely the objective of the construction of a residue referential set of data.

8 different materials were described: meat, skin, feathers, hair, bone, antler, wood and cane. Residues were analysed by means of optical and electron microscopes. For each type of residue, elemental data was also provided.

The results obtained were of much interest, because they allowed us to improve the identification rates of residues of some worked materials frequently used in Prehistory. The extensive photographic material, including both OLM and SEM micro-graphs, added complementary insights to knowledge acquired from recent publications on this topic (*e.g.* Borel et al., 2014; Monnier et al., 2012; Xhauflair et al., 2017). Difficulties in differentiating various materials based on morphological characteristics of fibres have been strongly emphasised. Therefore, alternative sources of data are particularly needed to improve the method.

The acquisition of elemental spectra for each residue is thought to be the first step towards a deeper and more consistent interpretation of micro-residues. We are aware that the investigation of the residues' chemical composition would add solid data to our reference collection and would allow us to reach a high degree of reliability of the interpretations provided. To name some of the analytical techniques recently incorporated into the protocols involved in the analysis of residues:

- FT-IR, Infrared reflectance spectroscopy (Prinsloo et al., 2014; Monnier et al., 2013; 2017a, 2017a);
- GC-MS, Gas chromatography-mass-spectrometry (*e.g.* Eerkens, 2005; Evershed, 2008; Cârciumaru et al., 2012):
- TD-GC-MS and Py-GC-MS, sequential thermal desorption-gas chromatography and pyrolysis-gas chromatography-mass spectrometry (Hardy et al., 2012; Buckley et al., 2014).
- GCxGC-TOFMS, comprehensive two-dimensional gas chromatography-time-of-flight mass spectrometry (Perrault et al., 2016).

Direct molecular evidences significantly enrich the final interpretations of micro-residues. We believe that the recent explorations aimed at adding biochemical data to the traditional descriptions of residues have been a turning point within the method.

Additionally, vegetal residues were not intensively studied in this work. Only a woody species was considered (*Quercus ilex*). Knowing the different features that diverse wood types can display (Monnier et al., 2012), it would be good to include more taxa in our future experiments. Starch grains and phytoliths were also not considered, as the analysis of such residues required standard procedures for their extraction and particular sample treatment (*e.g.* Keahlofer et al., 1999; Hardy et al., 2009). Considering the implications of this kind of residues for assessing past human diet (*e.g.* Mercader, 2009; Hardy et al., 2012; Weyrich et al., 2017), it would be important to integrate the analytical methods required for their analysis in our background in the near future.

Other residues which were not considered in our work and possibly present in the archaeological record were shells, ochre, ichthyofaunal remains, ivory, etc. (Christensen, 1995; Pawlik, 1995; Wadley, 2005; Lombard, 2007; Hardy and Moncel, 2011). An integration of them in future experimentations is sought as well.

8.1.3 Combination of microscopic techniques

Knowing the obstacles previously faced by analysts when observing reflective coarse materials (e.g. Kamminga, 1982; Knuttson, 1988a; Grace, 1990), we thought to combine several microscopic techniques since the initial phases of our research. There are different stratagems that traceologists adopted to cope with the brightness of the surfaces. One of these involves the observation of positive (Plisson, 1983) or negative (Lemorini et al., 2014; Berruti et al., 2016) replicas of the edges. While the use of positive replicas might be a valid alterative to avoid the halo of quartz crystals, the use of moulds can be questionable. This method has not proved to be valid yet, as no published detailed account is available.

When regular optical light microscopes are coupled with the Differential Inference Contrast (DIC), it is claimed that the reflected light significantly diminishes (*e.g.* Igreja, 2009). As a consequence, wear is more easily observable. This might be true in some cases, depending on the textural characteristics of the rock observed. During our observations, we realised that the employment of the DIC not always assures better results than regular optical microscopes. In some occasions, it helped us to obtain better images of use-wear which were visible also without employing the DIC. Nevertheless, when some traces (previously observed with the SEM) were invisible under the OLM, the use of DIC did not render them visible. In fact, there are technical constraints in optical microscopy which impede the observation of particular traits on very irregular surfaces. First, out of focus light from points outside the focal plane reduces image clarity and this is particularly inconvenient when observing highly reflective materials. Second, quartz grains are formed by several faces having a different orientation. This is one reason for the high reflectance of quartzite surfaces. Therefore, sometimes it is impossible to obtain an in-focus image of all the faces of a crystal. Moreover, sometimes some faces are so reflective that they cannot be observed even with the aid of the DIC. In such cases, wear possibly present on these surfaces is not detectable.

In our study, we systematically employed OLM and SEM to analyse use-wear on quartzite surfaces as well as the micro-residues of the worked materials. We decided to do so in order to evaluate which one was provided the best results (during direct observations and resolution of the final images). Hence, we usually compared the same portions of surfaces (with use-wear or residues) imaged using these two microscopes. The fact that exactly the same portions were imaged (usually with the same angle of observation) (as in Borel et al., 2014), allowed us to evaluate the advantages and disadvantages of both microscopes.

Confocal Microscopy was also occasionally used to quantify polished zones on quartzite. As the interpretation of such data is still in progress, the real contribution of this technique to our study has not been assessed yet.

In evaluating the combination OLM-SEM for the analysis of quartzite surfaces, major advantages of SEM over OLM have emerged. First, the higher depth of field of SEM allows a better visualisation of both use-wear and the contextual background (rock texture and topography). Second, a particularly high resolution of SEM micro-graphs provides better appreciation of the use-wear traits. Third, the higher magnifications reached by this equipment allow the detection of tiny modifications of the surface, which would be impossible to be imaged with a regular optical microscope. Finally, it completely avoids the problem of reflection. As image formation of SEM does not rely on visible light, the obstacle given by the high reflectance index of quartz and quartzite is entirely overcome.

Additionally, all of these qualities made SEM a particularly interesting tool to investigate the formation of use-wear and to assess the variability of linear features (*e.g.* Hayden, 1979 Ed. and references therein; Mansur-Franchomme, 1983; Yamada, 1993; Ollé and Vergès, 2008).

OLM is a good tool for an initial observation of quartzite assemblages; it might help in the selection process of pieces for further analysis by eliminating those presenting post-depositional traces for example. While OLM can be decisive in the analysis of use-wear on fine-grained rocks, its unique employment in the analysis of coarse-grained materials is questionable. Based on our observations, quartz represents a particular case within coarse-grained materials. Some varieties of milky quartz, presenting large crystals and relatively regular surfaces, can be satisfactorily scanned with regular optical microscopes (Ollé et al., 2016). Strictly regarding quartzite elements, we believe that further analysis needs to be done at higher magnifications, therefore with a SEM.

While it has been suggested that the analysis of macro-traces on quartzite might provide better results than the analysis of micro-traces by means of optical devices (Grace, 1990), we strongly discourage providing functional interpretations only based on the distribution of macro-scars. The reasons for this are extensively discussed in Chapter 3. At present, when macro-traces are contemplated in functional studies, they are generally presented along with the evidence of micro-traces (low-power and high power approaches). Nonetheless, there are rare studies which still propose interpretations based only on macro-traces data (Chen et al., 2014, 2017).

Generally speaking, combining multiple microscopic techniques always contributes to the obtaining of in-depth results. The selection of which types of microscope to be used should be based on the specificities of each single case study.

8.1.4 Contamination

The issue of contamination in functional studies has been addressed with increasing interest during the last decade (*e.g.* Langejans, 2011; Crowhter et al., 2014; Rots et al., 2016; Xhauflair et al., 2017).

Our study contributes to this debate by addressing some potential common contaminants present on the surface of the lithics (such as skin flakes, moulding clay, graphite signs, clothing fibres, etc.). Through the systematic comparison of images of the same micro-residues but taken with different microscopes (OLM and SEM), we provided an extensive set of comparable data available to the scientific community. Through comparisons with the published materials, analysts will have an additional tool to interpret microscopic evidences and to discard possible contaminants from the functional interpretations of both experimental and archaeological materials.

A standard protocol has been proposed to avoid contamination of the lithics prior to functional analyses, already suggested in other studies (Lombard, 2008). The most feasible way to treat stone tools before microscopically analysing them in order to maintain the risk of contamination to a minimum would be the careful extraction from the sediment and the direct storage in plastic zipped-bags. Contact with hands should be avoided during the entire process and the artefacts would then be removed from the bags only directly before analysis. Therefore, no other analyses should be carried out (such as technological analysis, refitting analysis, raw material determination, etc.) before functional analyses. No marking should be allowed either. All of these measures would be taken with the main aim to avoid any modern substances to deposit onto the surface of stone tools and be mistaken for ancient residues during residue analysis. Besides, if it can be assured that no hand contact has been made with the artefacts, a possible discovering of human skin flakes on their surfaces would be clearly interpretable as ancient. Surely, discoveries like this are near impossible at present not only because of their clear uniqueness, but also due to the way the lithic material is normally handled after excavation. We are perfectly aware that the proposed handling protocol is not applicable to the entire archaeological record recovered from a site, due to obvious organisational issues (first of all, the study and labelling of the material). Our proposal in fact envisages a proper way to treat the archaeological material in order to provide trustworthy results. Therefore, it should be applied to a number of selected artefacts from which residue and use-wear analyses are expected to donate interesting insights. Such selection would be made directly in the field and should be dictated by the archaeological questions to be addressed, specific to each site. Having said so, we acknowledge the fact that there are much more sophisticated methodologies to recover stone tools which are capable of virtually avoiding the inclusion of any external contaminant. Such methodologies are based on forensic sciences and aim to obtain uncontaminated information during the excavations of sites (mobile clean-room; Kuchimba project, University of Calgary). By adopting such methodologies, one can expect to later analyse uncontaminated lithic surfaces at the laboratory and therefore, to obtain reliable results.

8.1.5 Integration of technological and functional studies

The idea that technological studies and use-wear/residue analyses are deeply connected has been sustained throughout this thesis. Although they respond to different questions, the ultimate purpose is the understanding of the essence of stone tools and beyond that, of human behaviour.

Technological studies are more concerned with the production phases of artefacts, from raw material procurement to the application of specific methods and techniques to produce predetermined objects. Techno-functional analysis focuses on the products of the operational sequences and, by analysing the general structure of the artefacts and the combination of different techno-functional units, establishes techno-functional groups. The artefacts pertaining to each group share the same functioning modalities. This represents a step forward toward a thorough understanding of tools and of technical intentions. In fact, it allows to interpret single steps involved into the production of tools and to get closer to the significance of the secondary modification of edges. Knowing that lithic production always depends on several constraints, technological, historical, cultural and functional, the techno-functional approach allows to incorporate all of these aspects in the definition of an assemblage. Regarding the functional aspect, the functional potential of tools is considered. When use-wear analysis is subsequently applied, use-wear traces can confirm or not the interpretations given by the techno-functional analysis (Bonilauri, 2010).

When use-wear analysis alone is applied to an assemblage, technological data are rarely considered. Often, retouched pieces or specific typological types are preferred. To correct for this potential bias, the incorporation of technological data may be crucial. As the operational sequences were conceived to produce stone tools to be used, the functions performed with such tools are their ultimate aims. Therefore, their knowledge is central to reach a complete understanding of the lithic production at a site.

In the same way, once some activities have been identified on a number of tools, it is important to go back to the production phase to understand which place of the operational sequence is occupied by the used tools. In other words, are particular functions always performed with the predetermined products of the lithic production or are several functions performed indiscriminately with all objects? Is retouch applied on artefacts presenting similar structures or is it used to make objects with multiple structures to converge into a desired, standardised structure? Is it applied only to slightly modify the original edge characters to obtain transformative techno-functional units apt to incise matter? By responding to such questions, a deep understanding of the archaeological lithic record can be provided.

It is important to underline that the combination of techno-functional and use-wear study may succeed in giving a broad vision of the used tools of an assemblage; broader than if the two analyses are separately applied. The traditional typological series (Bordes, 1988; Laplace, 1972) are concerned with the grouping of retouched implements into pre-established categories. These implements may or may not have been used. The sole presence of retouch is not a prove of the utilisation of artefacts, but it is indeed an evidence of the will of obtaining specific objects (characterised by standardised structures and techno-functional units, highlighted by techno-functional analysis). By means of the techno-functional analysis, one can explore the universe of tools by identifying their functioning modalities. Besides, it may include used tools which display use-wear, used tools with no use wear and tools which have not been used. In fact, there may be tools whose utilisation did not substantially modify their surfaces (for example short actions on soft materials), therefore they would not be identified through regular use-wear analysis. If modifications are also too sparse and do not present clear patterns, they are not normally conceived as use-related by traceologists.

We assume that use-wear analysis is a valid tool to identify used edges, however there are cases which can be problematic. This is why, a combined application of techno-functional and use-wear analyses may help in giving a comprehensive idea of lithic assemblages. Moreover, the advantage of use-wear analysis is that it can document used artefacts made on different technological categories and it is not restricted to retouched artefacts. However, there is a number of used tools which probably would remain undiscovered despite the application of both approaches. As said before, there are artefacts which may have been used, but they cannot be identified by either techno-functional and use-wear analyses. Therefore, we must apply caution when we provide functional interpretations and always consider the limits of the analyses themselves.

In this thesis, we applied techno-functional analysis on the retouched and unretouched flake sample, as a trial to test our theoretical assumptions. Detailed descriptions of the operational sequences are not provided, as this would fall beyond the scope of this research. However, the ascription of the artefacts to techno-types helped to identify the potential used edges and therefore, helped in the process of sample selection for subsequent use-wear analysis. It was also useful to localise the prehensile parts on artefacts and so, to understand objects as wholes. However, as the analysis was seen as a complementary aid to use-wear analysis, the techno-types identified were very broad categories. We are aware that a more detailed

techno-functional analysis would probably document multiple sub-types, based on technical characters here overlooked.
Anyhow, the results obtained are very positive, a high matching score between data coming from both analyses has been highlighted. For this reason, we think that the combination of these two approaches is very promising and should be additionally explored in the future.

8.2 Archaeological results

The ultimate aim of every experimental activity in Archaeology is to reach a better comprehension of the archaeological record. The application of the knowledge and expertise acquired from experiments to the analysis of archaeological collections is a powerful tool to shed some light on the significance of the past objects unearthed at excavations.
In Traceology, experimental activity is a fundamental step in the formation of all researchers because it allows them to construct a solid background. In fact, it is fundamental to rely upon sound experimental data before even attempting to interpret microscopic traces found on archaeological artefacts.
This is why, most of the efforts of this thesis were put into the development of a wide experimental reference for quartzite. Nonetheless, we applied the methodology developed through extensive experimental activity to two archaeological assemblages in order to test it. The analysis of the two assemblages responded to different archaeological questions, based on the respective contexts of the sites.

8.2.1 Payre site

The first site studied was Payre, located in southern France. The lithic assemblage is mainly composed of flint, which is very abundant in the surroundings of the site. Other raw materials, such as quartz, quartzite, basalt and limestone, are present in lower percentages. Quartzite is present throughout all the chronological sequence of the site, which spans from MIS 7-8 until MIS 5-6. It was important to understand the role of this raw material, especially the introduction into the site of large tools knapped at outside locations. Because of this, the selection of the sample to be analysed comprised artefacts from all the levels of the site. Positive results were obtained: surfaces were well preserved and microwear was successfully described and connected to specific functions. Woodworking as well as the processing of bone were identified. Specifically, percussive activities were identified only on large tools, which could help to explain why these tools were produced at outside locations and then used at the site. To better assess this proposition, more artefacts should be analysed and specific experiments involving large tools used to perform percussive activities should be carried out.

Additionally, the limited number of artefacts analysed did not allow to reach a thorough understanding of the role of quartzite at the site, which was the main question of this study. Moreover, as functional data on other coarse raw materials (*e.g.* quartz and basalt) is not available yet, comparisons have not been possible to make.

One of the future steps will be to enlarge the sample analysed, possibly including other raw materials, to gain a deeper understanding of the tasks performed at the site during the various occupations identified.

8.2.2 Gran Dolina site

Out of all the 3,608 artefacts forming the quartzite assemblage of GD-TD10.1, 14,4% is composed of entire products (unretouched and retouched flakes) larger than 20mm. All these products were technologically analysed and after this, techno-functional analysis was applied (Annex 6). 25% (n=129) of the artefacts analysed were ascribed to techno-functional types. Three main groups were identified and several sub-types were carefully described.

The analysis of the sub-types underlined the recurrence of specific transformative and prehensile techno-functional units (t-TFUs, p-TFUs). Very frequently, the p-TFUs are found on the edge opposed to the t-TFUs, which may be retouched or not. Retouch is not particularly frequent and it seems to have been employed to slightly modify the edges, depending on the action performed. Clear example of this are related to the obtaining of concave frontal delineations, where a notch was created by a single stroke (*encoche*). On one artefact presenting this kind of delineation, use-wear related to transversal actions on hard materials were individuated. Retouch was also employed to obtain the emphasise the convergence of some pointed artefacts. On one of these artefacts, use-wear related to rotational movements was found on the pointed extremity obtained through retouch. In a few cases (n=3), retouch was used to prepare the p-TFUs. In two cases, the modified p-TFUs are thought to have been hafted.

Additional insights are related to the selection of blanks. A high quantity of objects display a lateral backed edge, often cortical, which has been identified as the p-TFU. Therefore, cortical and semi-cortical secant products, coming from the reduction of the convexities of the pebbles, were systematically selected for their natural combination of TFUs apt to be manually held. t-TFUs may be retouched or unretouched.

When several unretouched regular edges are present on the same object, the description of t-TFUs is difficult. In fact, the analysis of poorly retouched assemblages may be very challenging (Koehler, 2009; Rocca, 2013).

Overall, the data coming from the techno-functional analysis have been further corroborated by the analysis of use-wear. When use-wear was found on artefacts previously ascribed to techno-functional groups, it was always located on the respective t-TFUs. A single exception was encountered, where use-wear was found on the pointed apical part of a convergent tool, while the t-TFUs of that artefacts have been interpreted to be the two linear lateral edges.

The data obtained from the techno-functional analysis were considered during the selection of artefacts to be microscopically analysed. Use-wear analysis was further performed on 51 implements (30 unretouched and 21 retouched artefacts), which corresponds to 1,4 % of the entire assemblage, 6,3% of all the entire products (n= 814), and 9,8% of the products larger than 20mm (n=519).

Use-wear as well as residues (even if they were not correlated to the function performed) were observed.

As the analysed sample is not representative of the entire lithic assemblage of TD10.1 unit (composed of more than 21,000 implements), inferences about the function of the site are difficult to be obtained. However, one of the reasons why we selected to analyse the quartzite assemblage was related to the poor preservation conditions of chert (Font Rosselló, 2009; Font et al., 2010). Therefore, considering the percentages related to the quartzite assemblage only, the degree of representability of our results is higher.

Despite the limited number of the artefacts analysed, the mosaic character of the functions identified might be representative of the activities which were actually carried out at the site. Several actions and worked materials were identified, pointing to a diversification of tasks at the site. Butchering activities were found on fewer artefacts than expected. The high presence of cut-marks on processed bones of this level (Rodríguez-Hidalgo, 2015; Rodríguez-Hidalgo et al., 2015) made us think that the predominant function at the site could have been related to the processing of animal carcasses. The high number of animal bones with several anthropogenic modifications, the low incidence of carnivores in the formation of the bone record and the clear human selection of the hunted animals (Rodríguez-Hidalgo, 2015, 2016; Rodríguez-Hidalgo et al., 2015) make it clear that TD10.1 is a palimpsest of several long-term occupations where the butchering of animals was a central subsistence activity. Moreover, it has been suggested that data deduced from the faunal assemblage could have important implications for the understanding of human evolution, mainly on the division of labour and on food sharing among different members of a group (Rodríguez-Hidalgo, 2016:38). Based on the evidence and also considering the large number of lithic artefacts recovered, TD10.1 level has been interpreted as a residential site (Ollé et al., 2013; Rodríguez-Hidalgo, 2015, 2016).

Our data can contribute to this interpretation in multiple ways. The low number of artefacts used in the butchering activity may simply depend on the limited number of artefacts analysed. It may also have other explanations. For instance, chert might have been preferred for this kind of task (remembering than the chert artefacts yielded at TD10.1 are *ca.* 12,880, of which *ca.* 5,400 are non-retouched and retouch flakes). Also, as highlighted elsewhere (Gibaja et al., 2002; Berruti et al., 2016), wear on quartzite originated from contact with soft animal matter may be under-developed. Hence, its detection may be sometimes problematic.

If we consider traces connected to soft animal matter (meat, skin), 5 artefacts are connected to skinning or de-fleshing activities. Hide scraping was identified on two artefacts, demonstrating that animal carcasses were also exploited for purposes other than protein intake. Hides (or skins) were then worked, probably to remove meat and prolong then their preservations. Hides might then have been used for garment or shelter purposes.

Artefacts connected to bone working can also be related to butchering activities. Those presenting traces of longitudinal actions may have been used to disarticulate or dismember deer, bison or horse carcasses.

Other artefacts used to modify bone testimony the intention of obtaining marrow as a diet complement. 5 artefacts presented traces of transversal actions on bone and greasy material, which suggest that they were used during the removal of periosteum or muscle tissue from long bones. A single artefact displayed traces related to percussion activities on bone; therefore, this is the only artefact we can directly relate to a chopping action aimed at breaking bones for extracting marrow.

All of these actions (skinning, de-fleshing, periosteum removal, bone breakage through percussion) have been identified after the taphonomic analysis of the faunal record of TD10.1-bone bed (Rodríguez-Hidalgo, 2015, 2016; Rodríguez-Hidalgo et al., 2015). Therefore, our results benefit from a well-established contextual panorama, which strengthens the functional hypotheses proposed. In fact, scraping marks (Fisher, 1995) have been observed on 55 bones pertaining to medium and big size animals (deer, bison and horse) (Fig. 8.1). Normally they are located on long bones (53 cases), but they have been observed also on a lower jaw (1) and a calcaneus (1). In eight cases these marks have been related to the removal of the periosteum before fracturing the bones, all pertaining to deer or to medium-size animals (2 humerus, 2 femurs and 3 long bones). In the other cases, scraping marks have been related to defleshing and skinning activities (Rodríguez-Hidalgo, 2015).

Furthermore, 9 quartzite implements were found with evidence of woodworking; 2 of them had traces of longitudinal actions and 6 of them of transversal actions (scraping and whittling). Woodworking may be related to a number of tasks, but all of them imply the interaction of complex conceptions and gestures to modify matter to obtain a tool. Whatever the tools, the applications of different *chaînes opératoires*, from the collection of wood, the selection of the branches, the production of the lithic artefacts with which to modify the wood and the action itself, probably involving several different gestures, are all evidence of complex cognitive capacities. The final products may have been wood poles, handles or spears. Rare evidence of preserved wooden spears yielded from Lower Palaeolithic horizons (Thieme, 1999; others) demonstrate that this kind of technology was mastered since at least MIS9.

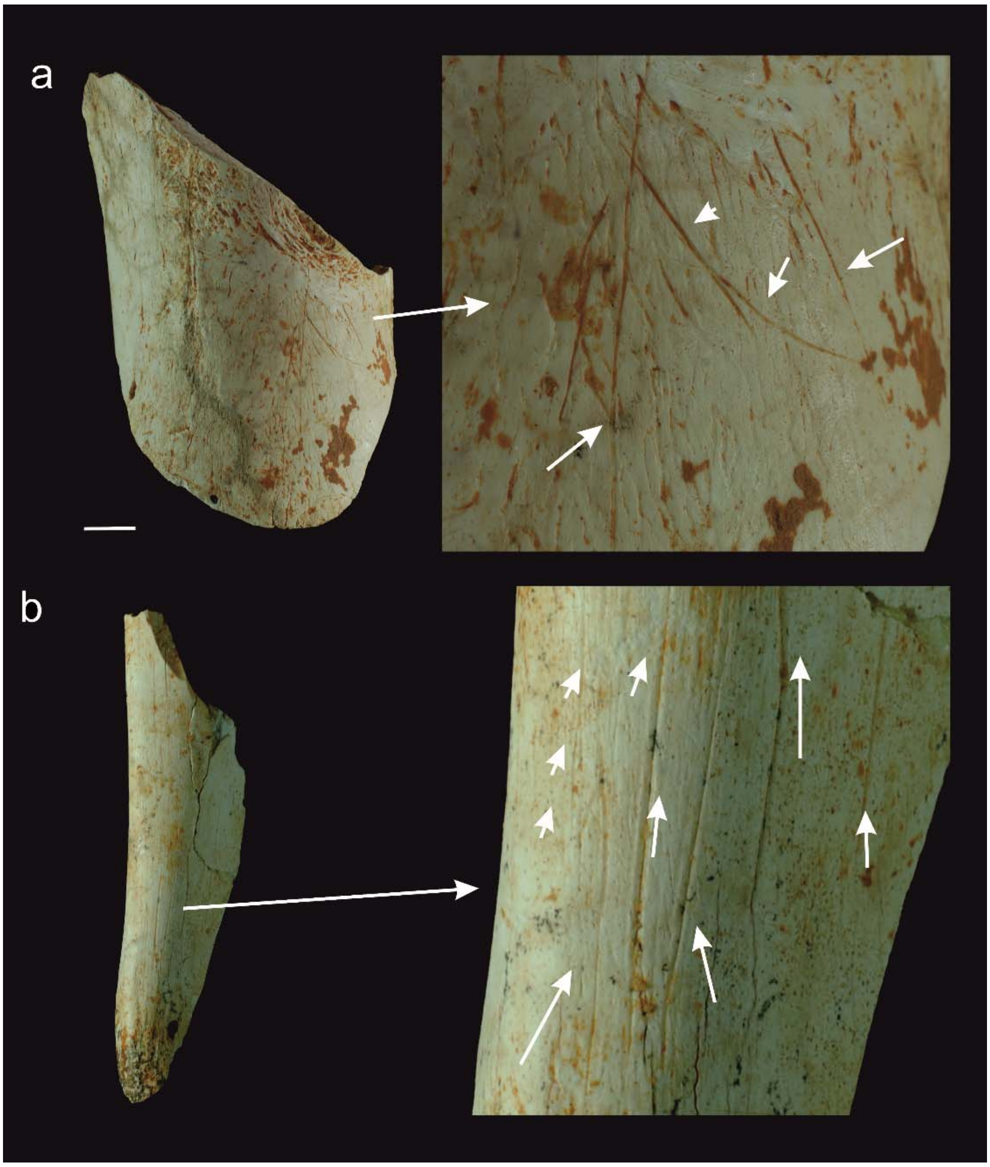

Fig. 8.1: *Examples of cut-marks observed on archaeological bones from TD10.a level. a) incisions on a fragment of a rhinoceros humerus; b) scraping marks on a fragment of a deer radius. Scale bar: 1cm (Rodríguez-Hidalgo, 2015: 154, Fig. 6.8).*

Recent analyses of 9 spears and other wooden artefacts from Schöningen (Germany) described long and complex operational sequences involved in their production (Schoch et al., 2015). Two different species of wood were selected as the raw material (*Picea sp.* and *Pinus sylvestris*) and small trunks were used to produce the spear. After the removal of the bark, wood was worked in order to manufacture the ending points and to eliminate branches or knots. Remarkable traces of polishing the surface and cutting off the branches of the spears are the most ancient direct evidences of woodworking in the World (Schoch et al., 2015: 222, 223). It results that the employment of wood into everyday activities during the Lower Palaeolithic could have been much more frequent than previously thought. Because the preservation of organic matter at Prehistoric sites requires extraordinary conditions, use-wear on stone tools may be a key factor to better assess wood exploitation in Prehistory.

Moreover, two artefacts bear traces connected to rotational movements (*e.g.* piercing, drilling) on hard materials (bone or wood). This kind of activities, as well as woodworking, are indicative of residential occupations. Experimental data connected to rotational movements are not available from our reference collection, therefore it is difficult to provide confident interpretations. However, one may hypothesise that these pointed artefacts were used in the manufacturing process of possible handles or other wooden tools.

Finally, possible evidence of hafting has been found on three artefacts. Use-wear were also found on all of them. Two of them were related to longitudinal actions on hard material, while the third one displayed traces of hide scraping. Details of the haft (material, hafting mode, etc.) are not proposed, as no comparable experimental data has been previously generated.

Thus, use-wear evidence at GD-TD10.1 sustains the hypothesis which sees the function of the cave as a residential camp, where different activities took place. The exploitation of soft animal materials (hides) and woodworking indicate the performance of activities other than those strictly connected to subsistence (butchering of animals).

Other evidences which support the interpretation of TD10.1 level as a residential camp site comes from the refits analysis. Refit studies allow to distinguish knapping areas and events of transport movement. The refit process provides direct connections (refits and conjoins) and also associations or clusters of several elements capable of being knapped from the same pebble (indirect connections) (López-Ortega et al., 2011, 2017).

Use-wear analysis was applied in conjunction with the ongoing refit study of the lithic assemblage of TD10.1 unit (López-Ortega et al., in preparation).

All the implements composing one refit were microscopically analysed in this thesis and two artefacts showed traces of transversal actions. The core, the distal part of a large cortical flake and other smaller flakes did not present any surface modification. All of the artefacts composing the refit were found on a defined area of the surface of the site, while the core and the proximal part of the largest cortical flake showed transportation from what is thought to be the knapping area (Fig. 8.2). While the movement of the retouched semi-cortical flake is easier to explain, as this is precisely one of the artefacts bearing traces of a transversal action, the transportation of the core is more cryptic. More work is needed to relate the results of use-wear and refit analyses, which may allow us to localise possible functional areas on the surface of the site. Certainly, the combination of refit and use-wear data is very important to reconstruct human activities which took place at the cave.

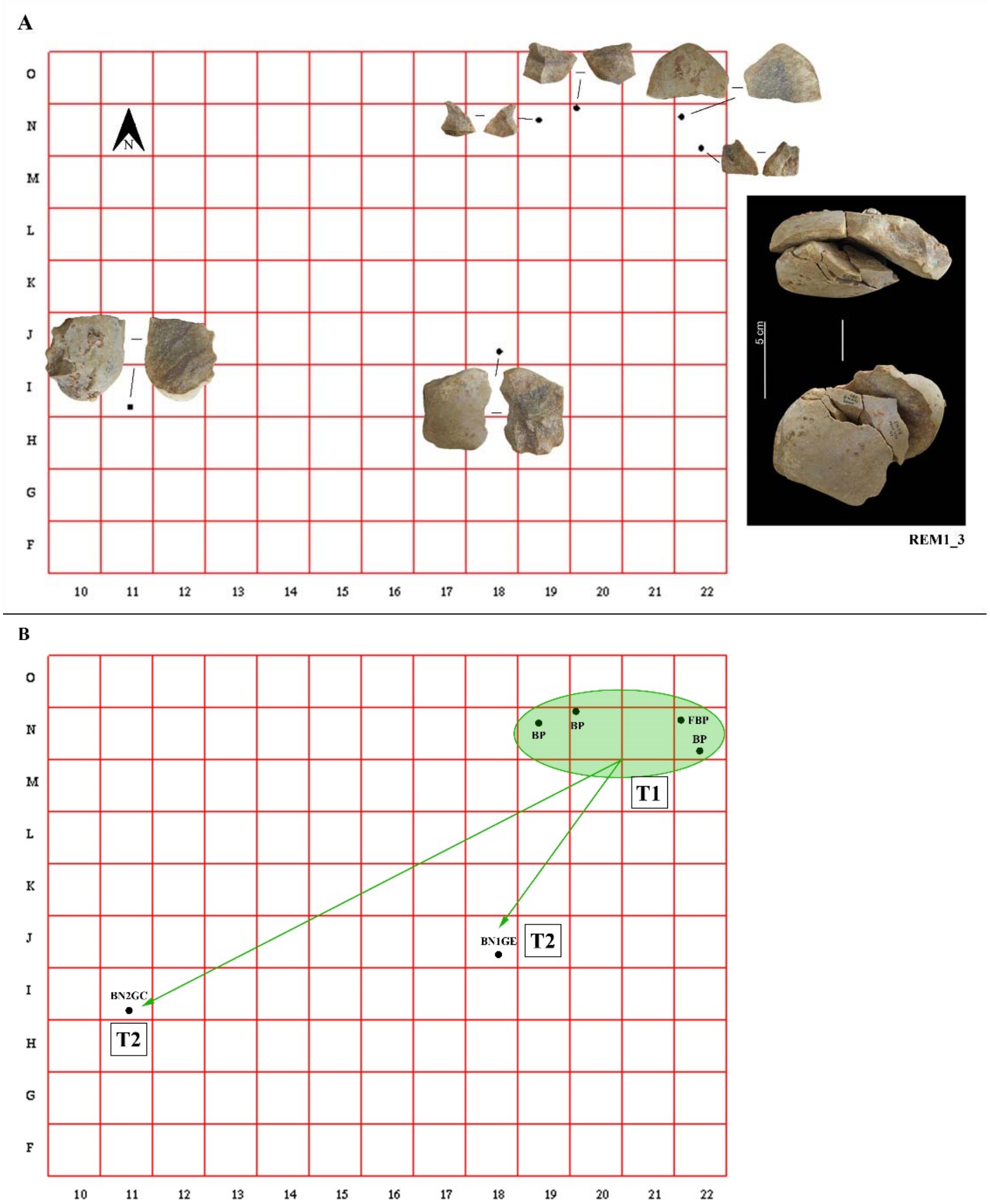

Fig. 8.2: *a) artefacts composing the REM1_3 refit and their original locations within the excavation grid; b) Structural categories (SLA) of the pieces composing the REM1_3 refit: green colour represents the knapping area (T1) and the subsequent movement of the core and the retouched flake towards the south-western part of the cave (T2). (López-Ortega et al., in preparation).*

Conclusion

This work provided a solid reference collection usable to interpret use-wear on quartzite assemblages. An extensive collection of graphic material has been made available (Annexes 3-4).

The potential of OLM and SEM to analyse quartzite surfaces was evaluated and it emerged that SEM is more efficient. However, as the access to this equipment is not always possible and it is expensive in time and resources, the best solution is to combine the two approaches. In fact, in an integrated approach the disadvantages of one technique are compensated by the advantages of the other one.

Moreover, a reference collection of micro-residues of the worked materials was initiated. Morphological and elemental data have been collected and used to describe the experimental residues.

Afterwards, use-wear and residue analyses were applied to artefacts selected from the quartzite assemblages of Payre (southern France) and GD-TD.1 (northern Spain) sites.

The main results of our study were made available in the form of published papers. Thus, 7 peer-reviewed papers formed the backbone of this thesis.

Future perspectives

There are many future perspectives raised by the results of this thesis.

First, the experimental reference collection should be enlarged by adding new experiments including different variables. More data about macro and micro-traces formed on quartzite retouched edges are needed. Activities and worked materials not included in previous experiments, such as chopping activities, rotational movements (*e.g.* piercing and grooving) and hafting practices would significantly enrich our experimental collection. The generation of hafting traces would be particular important in order to corroborate the functional interpretations of three archaeological pieces given in this work. In the same way, the inclusion of large tools to perform percussive activities would serve to better understand the brittle behaviour of quartzite.

Furthermore, it would be interesting to test the use of abrasives in the processing of hides and evaluate their impact on the development of use-wear on quartzite. The working of more vegetal species (ligneous and non-ligneous plants) would be interesting as well.

Additional work using Confocal Microscopy, following the results of the trial accomplished in this thesis, is necessary to gain more insights on the quantification of polish on quartzite.

Regarding the analysis of residues, many paths might be opened by this research. A deeper characterisation of experimental residues could be sought through the employment of analytical techniques. The addition of biochemical data to our experimental data is thought to be essential to be able to provide more reliable results in the near future.

Moreover, the possibility of incorporating more vegetal residues into our experiments could be explored. The detection of vegetal residues on the surfaces of stone tools is directly connected to the assessment of past diet. Moreover, these kinds of residues are incredibly informative as they provide indirect evidence of the human knowledge of the surrounding environment. The role of plants in the diet of our ancestors may have been traditionally overlooked. In fact, recent publications are pointing out that human diet in pre-agrarian populations may have relied on vegetal species more than previously thought (*e.g.* Hardy et al., 2015a, 2015b).

Furthermore, burial experiments are crucial to monitor the decay of residues and understand the changes that post-depositional movements may cause to both the visual aspect and chemical structures of residues (Langejans, 2010). It would be important to carry out monitored experiments to gain a better knowledge about this topic.

Regarding the archaeological material, it would be desirable to enlarge the samples of both the assemblages analysed in order to provide a better comprehension of the human occupations of the sites. In the case of Payre, the analysis of more artefacts would help in the understanding of the role of quartzite throughout the different chronological levels of the site. For Gran Dolina-TD10, the extension of the sample analysed would provide more data on the subsistence activities carried out at the site. If the selection comprised only artefacts coming from the TD10.1 bone bed layer, the spatial distribution of the different activities inferred would allow us to define functional areas on the surface of the site. This would be particularly feasible for this specific layer because it has been interpreted as a palimpsest, therefore it would be reasonable to analyse spatial data.

Annexes

Annex 1: A first trial to quantify polish on quartzite by using Laser Scanning Confocal Microscopy

This annex unifies the information of a first trial to quantify polished surface on quartzite through Laser Scanning Confocal Microscopy (LSCM). Although the selected variety is different from those treated in this thesis, the obtained results are of great interest even in the frame of this research. In fact, because of the very extensive nature of the polished areas analysed, the results of this study are comparable in a more general way to other varieties of quartzite.

Although several microscopic techniques were used to quantify use-wear on flint (Evans and Donahue, 2008; Lerner et al., 2007; Stemp and Chung, 2011; MacDonald, 2014), quantification studies are still in their infancy. Especially regarding quartzite, only one case study is currently found in the literature (Stemp et al., 2013).

In our study, the LSCM was used to obtain quantitative data of polished surfaces formed after the contact with different materials. The main objective was to understand if metrological analysis could be a viable option to ascribe the analysed polish to specific worked materials. If so, the interpretation of the worked material from the analysis of polished surfaces would be based on objective data and would acquire a more significant meaning.

At the same time, a secondary, but not less important, objective was to compare the images taken with SEM and LSCM to better appreciate the visual characters of the polished areas. In fact, we noticed that both the optical and laser images of the LSCM are incredibly explicative regarding the micro-topography of the analysed surfaces.

Method

The experimental flakes were obtained from the same cobble of meta-quartzite (VHS4) in order to limit the intra-variability of the raw material (Fig. A1). It was important that the analysed worn areas were produced on original surfaces with similar characteristics (roughness, granulometry, grain disposition, compaction, etc.). The cobble was collected at Villasur de Herreros, a village near the Sierra de Atapuerca (Northern Spain). Five flakes were selected and then used in the experiments. Two unused flakes were also kept to obtain measurements of the original, unworn surfaces.

Five different materials, commonly associated with early prehistoric tasks – wood, bone, antler, fresh hide, dry skin, and cane, were worked for an hour. These have been generally worked in a fresh state, except the dry skin. Antler was soaked in H_2O before the experiment for 48 hours. The selected species of wood was a type of softwood, Aleppo pine (*Pinus halaepensis*). A long bovid bone (*Bos tauros*), a red deer antler (*Cervus elaphus*) and stems of giant cane (*Arundo donax*) were also used.

The activity type was limited to whittling/scraping in order to control variables that may impact on polish development. All the experiments were also performed by the same person (A.P.), aiming at maintaining all the variables as constant as possible (such as the amount of exerted pressure, velocity, number of strokes per min). The length of the experiments was prolonged (60 min) to assure the formation of large well-developed polished areas knowing that polishing takes longer to form on coarse materials than on smooth ones (Leipus and Mansur, 2007; Clemente-Conte and Gibaja-Bao, 2009; Stemp et al., 2013).

Afterwards, each tool was first studied by means of Scanning Electron Microscopy (FEI quanta 600 SEM and a JEOL JSM-6400) to identify the areas with more widespread surface polishing. The same locations were then studied with an Olympus LEXT 4000 using the 50x (0.95NA) objective at 1x zoom (Fig. A2). Measurements performed on the LSCM images and subsequent analysis of the data have been conducted using DigitalSurf MountainsMap. A set of 20-25 measurements per image was provided and the sampled areas measured 10μm^2. Afterwards, confocal image data were converted into quantitative data always using DigitalSurf MountainsMap. The variable selected for data comparison was surface roughness (Rq). Both used and unused flakes were analysed through the same procedures.

Before SEM analysis, the tools were cleaned using a very robust method to remove residues. They were firstly soaked in water and then subjected to ultrasonic baths in hydrogen peroxide (10%) for 15min, in a neutral soap solution (Derquim) for 15min and in acetone for 5min. Only previously LSCM and due to the very sensitive character of the analysis, the tools were additionally soaked in 10% NaOH for 10min, and in water for 10min. Then, they were rinsed with chromatography grade ethanol and dried immediately before analysis.

Fig. A 1: *The five experimental quartzite flakes (VHS4) used in the whittling/scraping experiments. 1) Used on cane stems; 2) used on bone; 3) used on antler; 4) used on softwood; 5) used on fresh skin.*

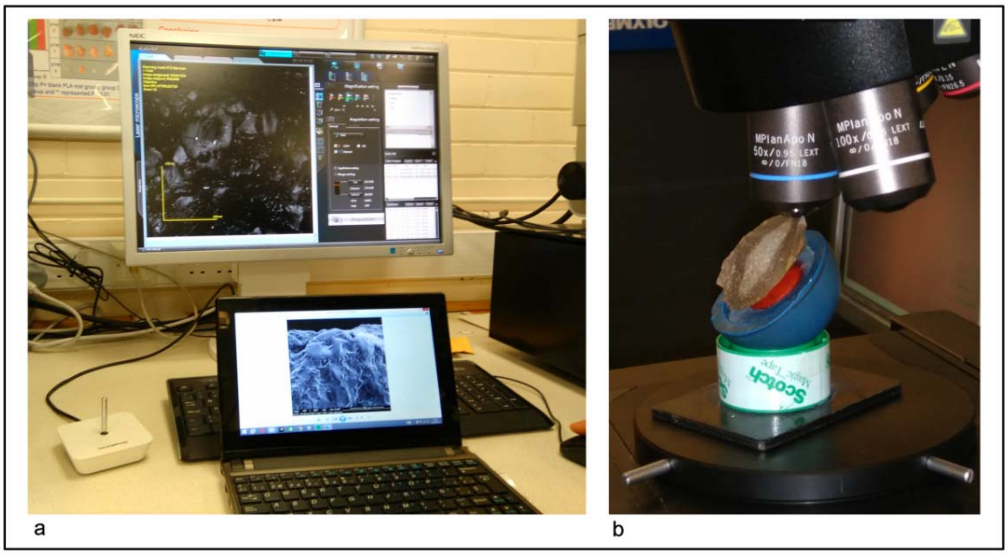

Fig. A 2: *a) LEXT software employed during LSCM analyses and in the front, a SEM picture used to localise exactly each polished point with the LSCM; b) an experimental sample placed under the LSCM.*

Results

Results of this first trial performed on a limited number of experimental replicas concern two distinct aspects involved in the description of use-wear. First, LSCM grey-scale pictures add topographic information and, used in conjunction with SEM images, contribute to a more thorough description of the visual aspect of the worn areas (Fig. A3, A4).

Second, when quantitative data are extracted from LSCM images, it is possible to associate numerical indices to differences of the micro-surfaces' roughness.

Some mineral inclusions, such as that shown in Fig. A5, are extremely visible in both LEXT optical and laser images (Fig. A5: 1). Normally, differences in grey-scale colour of different minerals were imagining using a regular SEM-backscattered electron detector (Fig. A5:3). It is interesting to note that LSCM also offers this advantageous possibility.

After the collection of metrical data through the analysis of the LSCM images, statistical analysis has been applied.

A diverse disposition of the LSCM measurements of the polished areas originated from the contact of different material is evident when quantitative data are plotted (Fig. A6).

The clear divergent position of polishes originated from cane and hide working relies on the practically opposite nature, the former being extremely smooth, the latter being typically rough. This only confirms what is frequently observed using conventional optical microscopy.

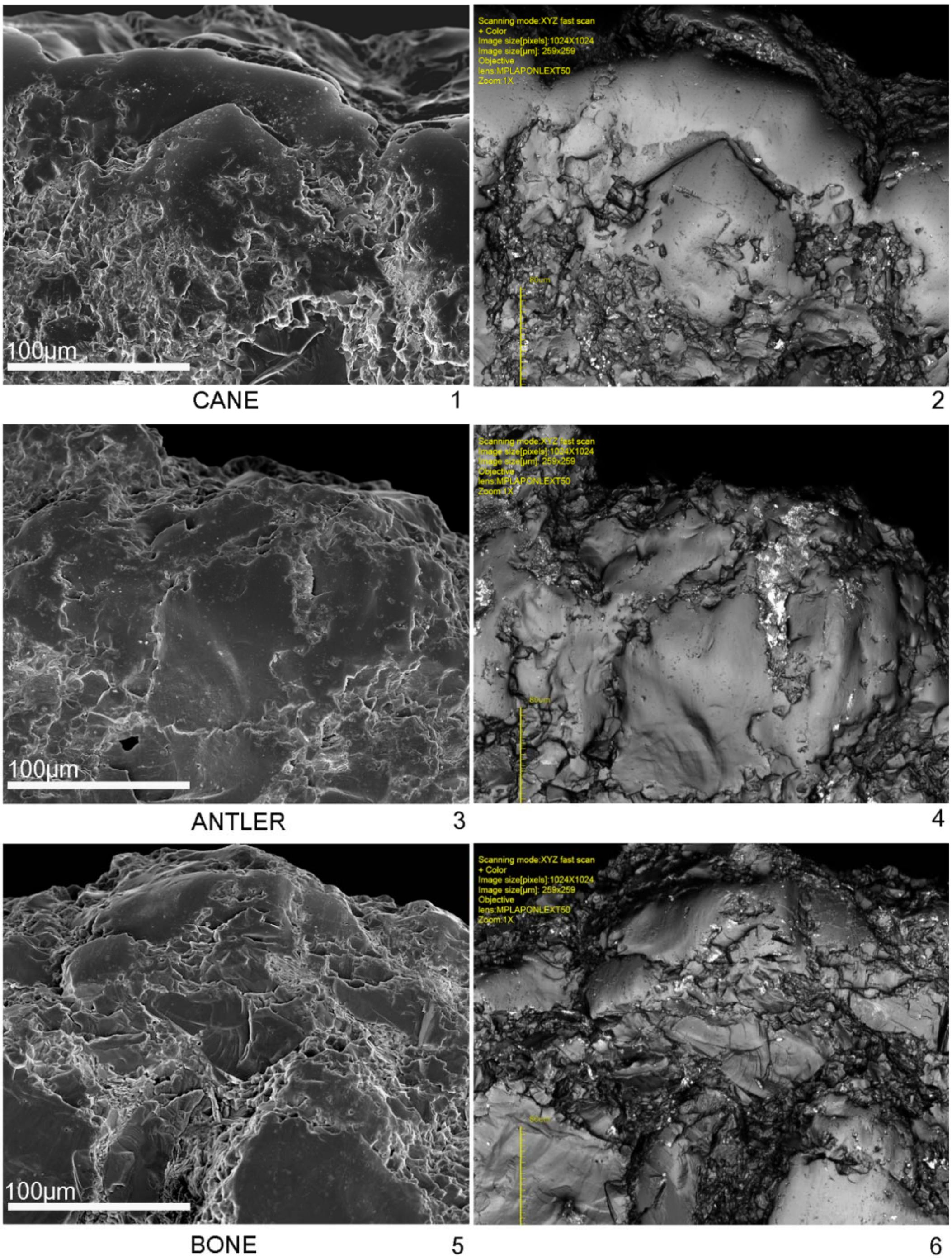

Fig. A 3: *Visual comparison of the same polished areas with SEM (on the left column) and LSCM (on the right column). The LSCM graphs were then used to perform the roughness measurements. 1, 2) Cane experiments; 3, 4) Antler experiments; 5, 6) Bone experiments. Original magnification: 450x (SEM graphs). The LSCM graphs are obtained using the 50x objective at 1x zoom.*

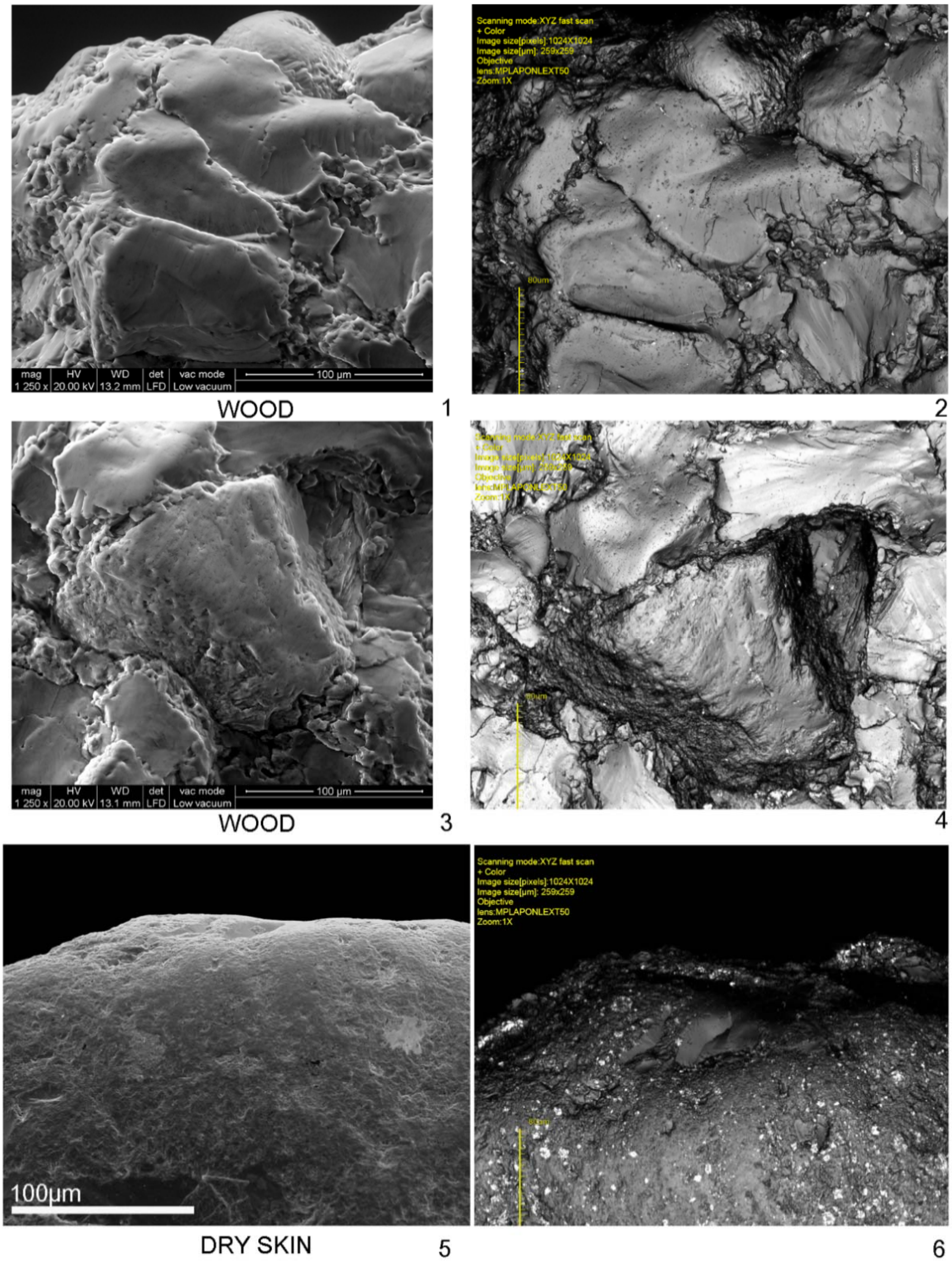

Fig. A 4: *Visual comparison of the same polished areas with SEM (on the left column) and LSCM (on the right column). The LSCM graphs were then used to perform the roughness measurements. 1-4) Wood experiments; 5, 6) Dry hide experiments. Original magnification: 450x (SEM graphs). The LSCM graphs are obtained using the 50x objective at 1x zoom.*

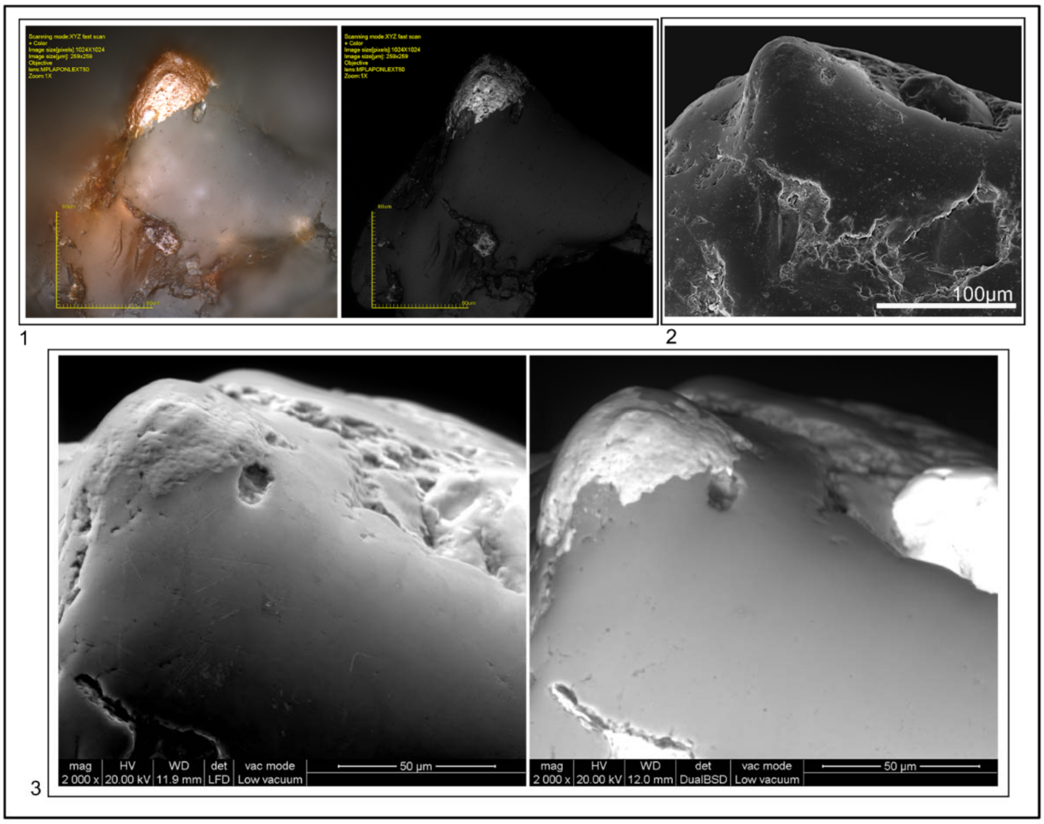

Fig. A 5: *Comparison of the same polished area obtained after whittling a cane stem observed under a LSCM (1), high-vacuum SEM (2) and low-vacuum SEM (3, secondary electron and back-scattered electron detectors). An accessory mineral mainly composed by Ti is visible under the LSCM and under the SEM-back-scattered elector detector, where it appears lighter than the rest of the surface. (3, right picture). EDAX analysis provided the elemental composition of the mineral. Polish seems to affect differently the areas mainly composed by quartz and those having a different composition. Original magnifications and lens: 1) 50x, at 1x zoom, 2) 450x; 3) 2000x.*

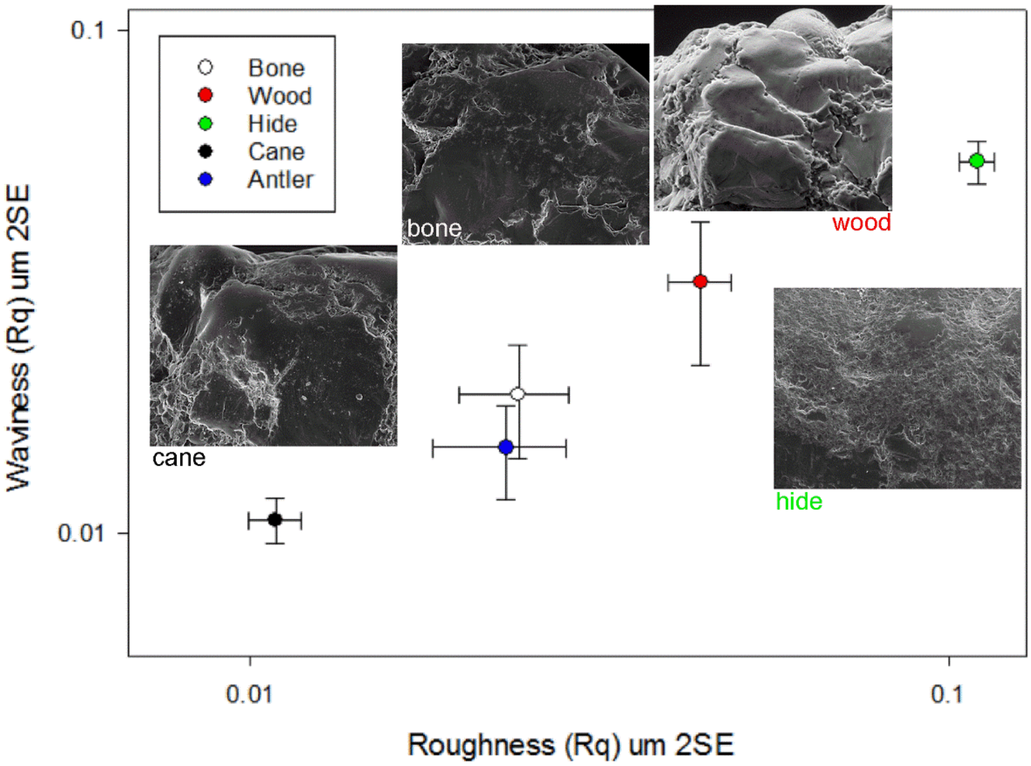

Fig. A 6: *Graphs showing the roughness measurement of the polished areas resulting from the five different worked materials. The SEM pictures at the interior of the graphs remind the visual aspect of the polished surfaces connected to each material.*

Final remarks

The use of LSCM in use-wear analysis is relatively new and therefore, still unexplored. This microscope has several advantages with respect to conventional microscopy.

It is manageable and more precise than regular optical microscopes. It provides good high-resolution optical pictures, allowing for a rapid comparison with laser images and therefore to situate where the measurements are taken with exact precision.

The true advantage of this microscope is the possibility to get quantitative data of the micro-surface of the sample. Features like smoothness, roughness, and waviness can be measured and ordered in numerical categories. In that way, the correlation of the same numerical values of those features would be objective and comparable between different studies.

Results of the measurements performed on a quartzite sample used in this preliminary study are very promising. The variable of surface roughness proved to give results consistent with the observations previously made using SEM. The different polished surfaces have been separated, indicating that in the future these experimental data might be helpful in the attempt to discriminate archaeological polishes originating from the contact of different materials.

Annex 2: Publication 8

Pedergnana, A., Blasco, R., 2016. **Characterising the exploitation of avian resources: An experimental combination of lithic use-wear, residue and taphonomic analyses.** *Quaternary International* 421, 255-269.

This publication has been separated from the corpus of the thesis for two different reasons. First, the experimental tools presented here are made of flint. Second, it involves a discipline different from those presented in this thesis. In fact, this work is an experimental attempt to cross data coming from a set of three different analyses, use-wear and residue analyses applied on stone tools and taphonomic analysis of bones.

Nevertheless, it is appropriate to insert it in the manuscript, as it provided outstanding results regarding residue identification. Particularly, a type of residues frequently connected with the consumption of avian species in Prehistory is characterised. Fragments of feathers, although rarely identified in the archaeological record, were recognised on tools with old chronologies and presented in several publications.

From our study, we understand that a correct identification of feathers with only reflected light microscopes of this kind of residue is improbable. Moreover, the probabilities that feathers survive in the archaeological record seem to be very low. However, the combined use of OLM and SEM with the addition of EDX, significantly improves the reliability of identifications of this kind of residue, at least to an experimental level.

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Characterising the exploitation of avian resources: An experimental combination of lithic use-wear, residue and taphonomic analyses

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ABSTRACT

Pilot experiments involving the butchering of bird carcasses and the use of non-retouched flint flakes were performed. The executed actions comprised skinning and defeathering various avifaunal species (*Circaetus gallicus* and *Gyps fulvus*). The main aim of this experimental programme was to document the use-wear on flint implements employed in the treatment of the avifaunal carcasses in order to help researchers identify this activity in the archaeological record. An additional focus of this study concerned the experimental organic residues (soft tissue and feathers) associated with the bird species used in the experiments. For each residue type, a detailed chemical elemental analysis and morphological characterisation were performed, with the aim of creating an experimental database for comparison with the micro-residues that will potentially be found on archaeological stone tools. For microscopic observations, we employed both Scanning Electron Microscopy (SEM) and Optical Light Microscopy (OLM). A detailed description of the use-wear features and residue types was achieved through a systematic comparison of micrographs taken with both techniques. In addition, EDS (energy-dispersive x-ray spectroscopy) was applied to determine the elemental composition of the residues. Taphonomic analysis of the bones of the carcasses used in the experimental programme was performed with the principal aim of comparing the distribution of cut marks on bones with the use-wear pattern on the lithic implements employed. Future developments of our research will improve the methodology by expanding the experimental programme and by applying it to archaeological collections (at sites where the processing of these kinds of animals has already been identified).

1. Introduction

Feathers form a highly complex integumentary appendage found in the avian class, and they are characterised by an organised branched structure that grows according to a unique mechanism (Chuong and Widelitz, 1998). Feathers have always been admired by various cultures around the world and incorporated into both composite tools and garments. In the Americas, for example, indigenous cultures from Alaska to Patagonia employed feathers displaying an astonishing variety of shapes, sizes and colours to create items of both social and ritual significance. Colour plays a particularly important role in the selection of plumage for ornamental purposes. Efforts intended to change or emphasise tonalities have been documented in some native populations (Azevedo Luíndia, 2004) and the predetermined combination of feathers with different colours during the manufacture of artefacts is sometimes connected with deep symbolism (Lívero Sampaio and Pobikrowska Tardivo, 2010). In ecosystems rich in avian species, feathers have repeatedly been used as ornaments in diadems, headdresses, wristbands, earrings, cloaks, capes and sceptres (Levine, 1991; Lívero Sampaio and Pobikrowska Tardivo, 2010). Further, in geographical areas where the same colourful specimens were not available, feathers appear as a constant within the material culture (Levine, 1991; Pearlstein et al., 2012). Feathers have also been technologically important, featuring in complex *chaînes opératoires* like the fletching of arrows (Bartram, 1997; González-Ruibal et al., 2001) and the making of artificial flies for fishing. Moreover, on the basis of rock art paintings, the worldwide use of plumage throughout prehistory has been extensively documented,

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from its utilisation in ornaments and rituals to its employment in the fletching activity (Obermaier and Wernert, 1919; Jordá Cerdá, 1971; Pessis, 2003; Borgens do Lago, 2008; Martin, 2008; Viñas, 2014). Sometimes pigments with different and lighter tonalities from the substrate of the paintings were used to highlight ornamental feathers (Viñas and Morote, 2013).

Although the ethnographic literature has established that birds have been hunted for both their meat and plumage, the importance of avian resources during prehistory has only recently become a topic of investigation. The exploitation of bird carcasses is usually inferred from taphonomic studies (Fiore et al., 2004; Blasco and Fernández Peris, 2009, 2012; Peresani et al., 2011; Finlayson et al., 2012; Morin and Laroulandie, 2012; Blasco et al., 2014; Romandini et al., 2014; Radovčić et al., 2015), whereas direct evidence of bird residues, such as feather fragments, is much more difficult to document because of preservation constraints. When they are documented, feathers usually appear as micro-fragment remains on the surfaces of stone tools (Robertson, 2002; Dove et al., 2005; Hardy and Moncel, 2011). Sometimes, the identification of birds from archaeological residues has been performed to the level of either species (Dove and Peurach, 2002; Dove et al., 2005) or order (Hardy et al., 2001, 2013; Robertson, 2002).

Because this kind of evidence is rarely encountered, a refined methodology for its documentation and description has yet to be formulated. This is why we have focused on the creation of a suitable methodology for identifying the exploitation of avifaunal resources during prehistoric times. Based on the assumption that lithic tools were employed to butcher the animals and, subsequently, to work the plumage (if employing it for ornamental or technological purposes), we considered the two types of evidence most likely preserved in the archaeological record: use-wear and micro-residues of organic matter. Therefore, evidence related to experimental avian bones and experimental flint tools was documented in an attempt to understand whether or not cut marks on the bones were correlated with the identified use-wear on stone tools. The proposed methodology combines lithic and faunal examinations, with special attention to the microscopic characterisation of feather fragments adhered to the lithic surfaces. Especially regarding residue identification, a strong methodological foundation is needed for the analysis of the archaeological material. Researchers have yet to establish an experimental reference for comparison with the archaeological evidence. Before we attempt to identify ancient feather fragments on lithic tools, we need to clearly define their diagnostic attributes that are discernible under the microscope and to select the microscopic techniques that will provide the best results.

1.1. Feather structure

Feathers are highly ordered, branched structures that are intricately formed in order to endure the aerodynamic forces involved in flight. They also play a fundamental role in the thermo-regulation of birds. A combination of stiffness and

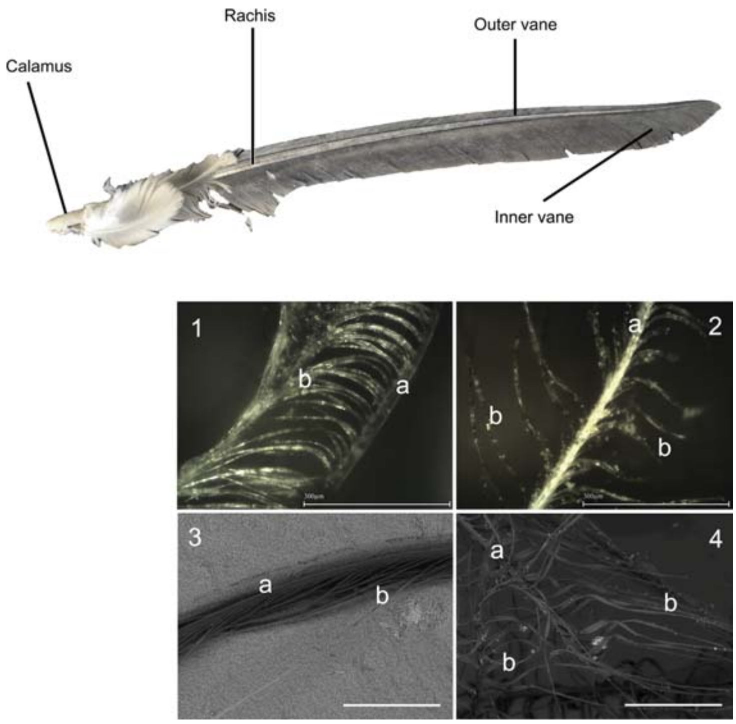

Fig. 1. A vulture (*Gyps fulvus*) contour feather composed of a longitudinal central shaft called rachis, to which the vanes are attached. Vanes are divided into downy (plumulaceous) barbs, located in the proximal part, and the pennaceous barbs. Barbs are magnified in the micrographs, illustrating the barbs (b) attached to the ramus (a). In the OLM pictures the birefringence character of barbules is visible, whereas SEM images enable us to better appreciate the micro-structure of barbs and barbules. (OLM pictures, 1–2: mag. = 200× scale bar = 300 μm; SEM pictures 3–4: mag. = 510× scale bar = 200 μm).

lightness is necessary to confer the ability to fly, converging in a singular structure. Feathers are composed of two main portions, a central shaft and the vanes extending from it (inner and outer vanes) (Fig. 1). The shaft must be resistant to damage and consists of two segments, the calamus and the rachis. The calamus is a relatively short tubular structure with a slight elliptical cross-section, which is attached to the skin of the bird. The remainder of the shaft is termed the rachis and bears the vanes of the feathers.

There are different types of feathers. The major ones are contour feathers. They have the same basic structure exhibited by all feather types, with barbs branching out from the rachis, as well as barbules, which in turn branch from the barbs. The barbs resemble microscopic feathers in appearance, meaning that they are composed, as feathers are in general, of a central shaft named the ramus (or rachilla) to which the barbules (the smallest division of feathers) are attached (Fig. 1: 1–4). Two barb/barbule types are recognised: downy (or plumulaceous) ones located in the proximal portion of the vanes (at the proximal part of the feather) near the calamus, and pennaceous ones located in the central and distal portions of the vanes. Barbules consist of a base and a pennulum (Fig. 7: F). The base is located near the ramus, whereas the pennulum displays different microscopic features depending on the barbule type. Pennaceous barbules are interlocked by tiny structures called hooklets, whereas downy ones bear diagnostic features used for the identification of bird orders and even species. In fact, the morphology and distribution of the nodes, which are the junctions of the cells composing plumulaceous (downy) pennulum barbules, vary depending on bird species (Dove, 2000; Dove and Koch, 2010).

The main constituent of the compact parts (rachis and rami) of avian feathers is keratin, a polypeptide common in the structural components of the body tissues of other vertebrates, such as mammalian fur, hoofs, horns, beaks and claws. Keratin (from the Greek *keras*, meaning horn) refers to a family of fibrous proteins composed mainly of 20 different amino acids (among them glycine, alanine and cysteine). Cysteine deserves special mention for being rich in sulphur and playing an important role in the stability and cohesion of keratins (McKittrick et al., 2012). There are two primary groups of keratins, α-keratins and β-keratins, which are distinguished from each other in terms of their structure, composition and properties. The *α-keratins* are present mainly in mammalian hair, horns and claws, whereas the tougher *β-keratins* are the main components of reptile scales, beaks and feathers (Huggins, 1980).

2. Materials and methods

2.1. Experimental series

A series of pilot experiments involving two bird species, griffon vulture (*Gyps fulvus*) and short-toed snake eagle (*Circaetus gallicus*), was conducted. Two carcasses, one for each species, were butchered using five non-retouched flint flakes. All the flint flakes were

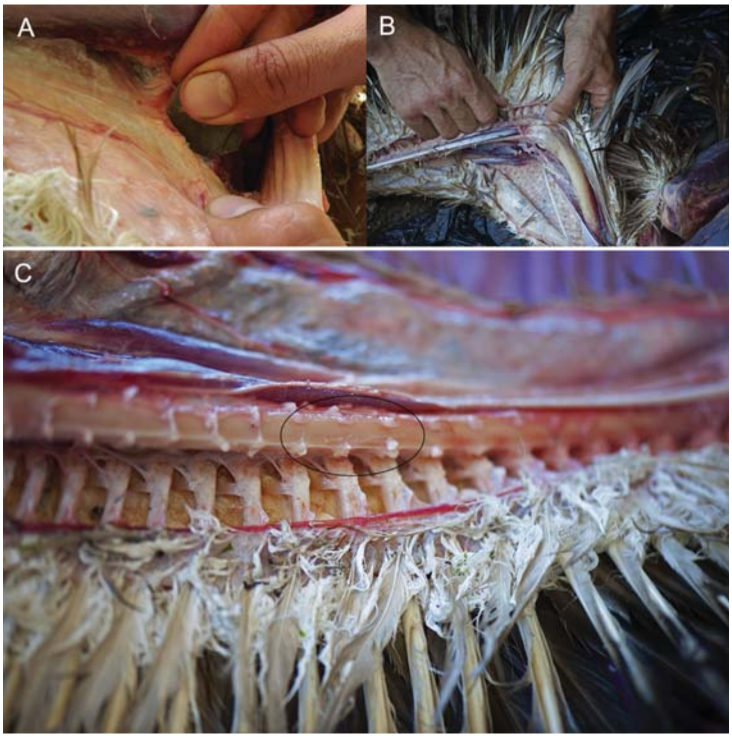

Fig. 2. Experiments documentation. A) Detail of a non-retouched flint flake used to uni-directionally cut vulture meat; B) Illustration of the plumage extraction from a vulture wing; C) Close-up of two cut marks on an ulna shaft of *Gyps fulvus*.

obtained from a unique chert nodule probably originated in calcareous lithofacies/formation. The chert nodule was collected on the floodplains of the Francolí River (Tarragona, Spain).

The experiments were designed with the principal aim of providing experimental evidence regarding the exploitation of avian carcasses in order to facilitate the future evaluation of its degree of visibility in the archaeological record. Experimental data included use-wear on lithic tools as well as organic residues and damage on bird bones. The main controlled variables in our experiments are summarised in Table 1.

Table 1
The main controlled variables of the experimental activity involving two bird species, the main parameters of the used lithic tools and the elapsed time of each experiment. The position of the used edge is recorded considering conventional dorsal view of the lithics. Length only refers to the used portion of the used edge.

Experimental reference	Avian species	Movement	Used edge	Length mm	Working angle	Elapsed time
FLINT 1	*Gyps fulvus*	Unidirect-Bidirect.	left	36	80°–90°	50′
FLINT 2	*Gyps fulvus*	Unidirect-Bidirect.	right	37	70°–80°	48′ 39″
FLINT 3	*Gyps fulvus*	Unidirect.	left	40	80°–90°	40′ 50″
FLINT 4	*Circaetus gallicus*	Unidirect.	left	31	40°–80°	40′
FLINT 5	*Circaetus gallicus*	Unidirect.	right	34	40°–90°	25′

The lithic tools were hand-held during all the experiments (Fig. 2: A). They were mainly utilised with unidirectional movements, with very few exceptions where the experimenters performed some bidirectional strokes. Such exceptions happened in concomitance with situations judged to be tricky by the experimenters, such as the extraction of particularly resistant sinews. Any momentary changes in the fixed experimental variables were carefully noted on experimental forms. Photographic and videotape documentation accompanied all the experiments. Plumage extraction was carefully monitored (Fig. 2: B) and the distribution of macroscopically visible cut marks was recorded (Fig. 2: C).

2.2. Use-wear and residue analyses

For lithic use-wear analysis, we employed an optical light microscope (Zeiss Axio Scope A1) with magnifications ranging from 50× to 500×. The use-wear traits were recorded by plotting their precise locations on sketches of the lithic objects.

For residue characterisation, we used optical light microscopy (OLM) as well as scanning electron microscopy (SEM) (SEM-FEI Quanta 600). Extended focus pictures (using Helicon Focus software) were sometimes obtained in order to contrast differential depths of field that occurred when residues were imaged with OLM. Micrographs of the residues were systematically obtained by means of the two techniques and were then compared in order to obtain a better description of the various residue types encountered on lithic surfaces.

All SEM observations were performed at low vacuum mode, which does not require any sample preparation. We also investigated the elemental composition of the residues using the SEM microanalysis system (energy-dispersive x-ray spectroscopy, EDX or EDS), because this kind of analysis is crucial to the recognition of the correct residue type (Jahren et al., 1997; Pawlick, 2004; Cristiani et al., 2009; Dinnis et al., 2009; Monnier et al., 2013).

Residue distribution was also recorded in order to better understand the distributional patterns on lithic surfaces subsequent to avian butchering. Residue analyses were always performed before observing the use-wear and hence prior to any cleaning processes (Pedergnana and Ollé, 2014). Ultrasonic baths were particularly avoided, since it is known that this cleaning procedure can modify the natural structure of feathers (Laybourne et al., 1992). After analysing the organic residues and before performing use-wear analyses, the experimental tools were subjected to a cleaning procedure aimed at eliminating the residues. The procedure consisted of ultrasonic baths with hydrogen peroxide, a neutral phosphate-free detergent (Derquim®) and acetone.

2.3. Bone-damage analysis

Bones resulting from the experiment were analysed in order to detect superficial and structural damage associated with skinning and defeathering. Different types of human alterations were observed, such as cut marks and, to a lesser extent, bone breakage.

Cut marks are understood to be accidents that occurred when the edge of the lithic tool came into contact with the surface of the bone during extraction of the external resources (in our case, skin and feathers). Therefore, these modifications are unintentional and are therefore attributed to physiological determinants of the animal's anatomy. However, it should be noted that the technology employed and/or specific cultural guidelines may lead to a certain margin of variability.

Cut marks are defined as elongated, often linear striations of variable length, width and depth. These striations have a V-shaped section and display internal micro-striae arranged lengthwise and parallel to the axis (Binford, 1981; Potts and Shipman, 1981; Shipman and Rose, 1983; Shipman et al., 1984). Shipman and Rose (1983) reported the presence of barbs or small striae that diverge at the beginning and/or end of the main groove during a short cut. Barbs are often produced by small hand movements at the beginning or end of a cut. Apart from these secondary striae, there are others that Shipman and Rose (1983) refer to as the "shoulder effect". They are intermittent and run parallel to the main groove, and they occur when a protrusion near the edge of the device comes into contact with the cortical bone as a result of different hand movements. Other features can provide directionality criteria, such as narrowing at the ends of the main striation, the micro-steps at the base and the presence of Hertzian cones (Bromage and Boyde, 1984). These cones are small, raised, triangular sections located on the sides of the main ridge. They are produced by the different pressures exerted on the bone's surface during cutting and by the resistance this offers in contact with the tool. Despite these criteria, the general morphology may vary depending on the type of tool used (material, shape, dimensions, degree of wear and presence or absence of retouching), the conditions of the agent responsible for its emergence (strength, degree of lifting of the hand with respect to the bone and intention) and the age, size and state of the carcass itself.

We have recorded three main types of cut marks (Binford, 1981; Potts and Shipman, 1981; Shipman and Rose, 1983; Shipman et al., 1984) in our experimental series by using a stereo light microscope with magnification of up to 120×:

- Incisions: These are thin grooves with variable depth, width and length. They occur when the edge of the tool comes into contact

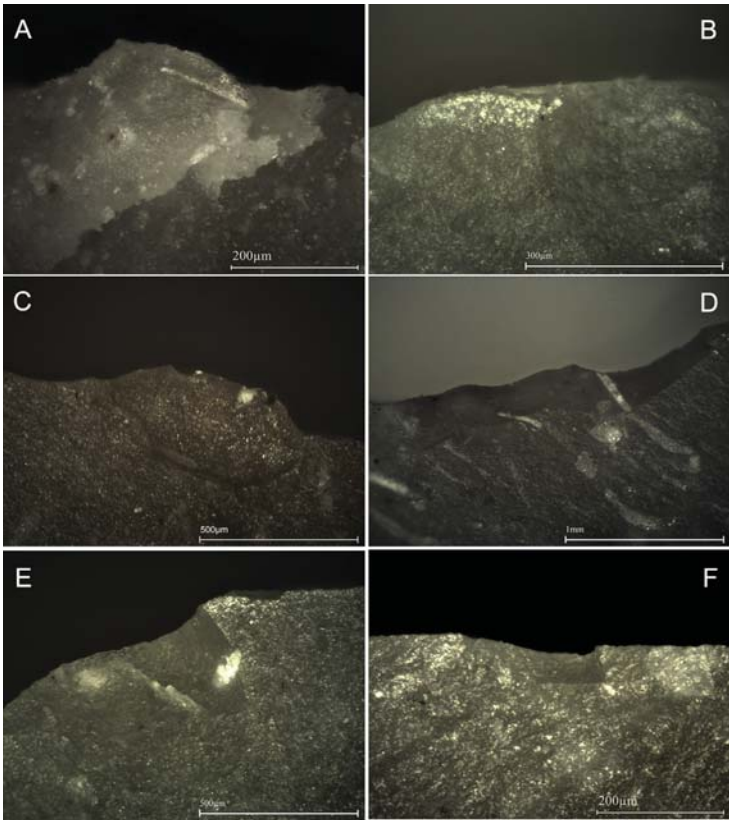

Fig. 3. Experimental use-wear connected to avian butchery activity. Polished spots, sometimes displaying linear outlines (A, B) were imaged as well as scarring (C, D). An interesting combination of both scars and polish departing from one edge of the scar was noted (E, F). (A: mag. = 200× scale bar = 200 μm; B: mag. = 200× scale bar = 300 μm; C: mag. = 100× scale bar = 500 μm; D: mag. = 50× scale bar = 1 mm; E: mag. = 100× scale bar = 500 μm; F: mag. = 200× scale bar = 200 μm).

with the bone's surface following an identical movement. The tool is oriented in the same direction as the cut. The incisions may appear isolated or may form groups, and their orientation may be longitudinal, oblique or transverse to the axis of the bone, with a straight, curved or sinuous localisation.

- Sawing marks: These are short, deep incisions that are concentrated in zigzags. Although several overlapping incisions were observed, the sawing marks correspond to a repeated movement during which the edge of the lithic tool remains in continuous contact with the surface of the bone. Normally, they are transverse or oblique to the longitudinal axis of the bone.
- Scrape marks: These are shallow and wide striae that run lengthwise down the bone. They occur when the edge of the tool comes into contact with the transverse bone surface.

From a taphonomic point of view, not all the cut marks respond to the same process. Depending on their state, layout, orientation and location on the bones, we can identify, along with other elements, the activity within the animal processing sequence to which they correspond. With this aim, we developed an experimental series that deals solely with characterising the location and types of marks produced during skinning and defeathering. For this reason, during the analysis of alterations, variables such as the type of mark, its location on the skeletal element (surface and anatomic region), orientation, delineation and measurements were recorded.

In addition to cut marks, some bone fractures also occurred during the experimental process. Fresh bone fractures tend to be associated with the disarticulation or extraction of marrow, fat and/or cartilage. For example, Laroulandie et al. (2008) described, both experimentally and archaeologically, the bone damage produced when dismembering a forelimb using overextension of the elbow on several bird species. This activity (overextension) leads to a breakdown of the olecranon fossa of the humerus, with a medial wrench of the distal part and a fracture of the proximal joints of the radius and ulna. This process can also produce other types of associated damage, such as peeling. Peeling is defined as a roughened surface with parallel grooves and a fibrous texture and is characterised by superficial flaking on the bone (White, 1992; Pickering et al., 2013). This modification typically appears on the flat bones of larger-sized animals, but it has also been documented in various bird species (Laroulandie, 2000, 2004, 2005; Blasco et al., 2014). Laroulandie (2000) experimentally reproduced such traces on the ulna of both goshawk (*Accipiter gentilis*) and buzzard

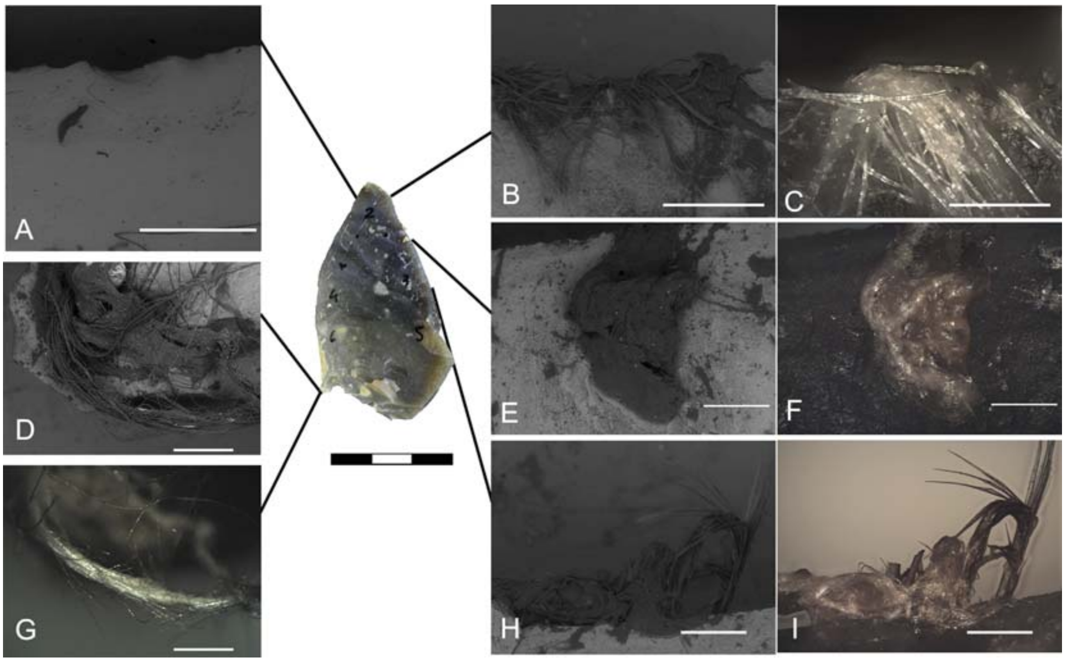

Fig. 4. An experimental flint flake (FLINT 1) showing sparse distribution of residues after butchering a griffon vulture specimen. Barbules are sometimes arranged into tangled bundles with a diameter of 100 μm (D, G) or are found embedded in macroscopically visible tissue residues (E, F). When barbules are found alone their birefringent character is visible with light microscopes (C). (A: mag. = 60× scale bar = 2 mm; B: mag. = 260× scale bar = 400 μm; C: mag. = 100×, scale bar: 400 μm; D, E, H: mag. = 135× scale bar = 500 μm; F, G, I: mag. = 50× scale bar = 500 μm. All the SEM images were provided with through a back-scattered electron detector).

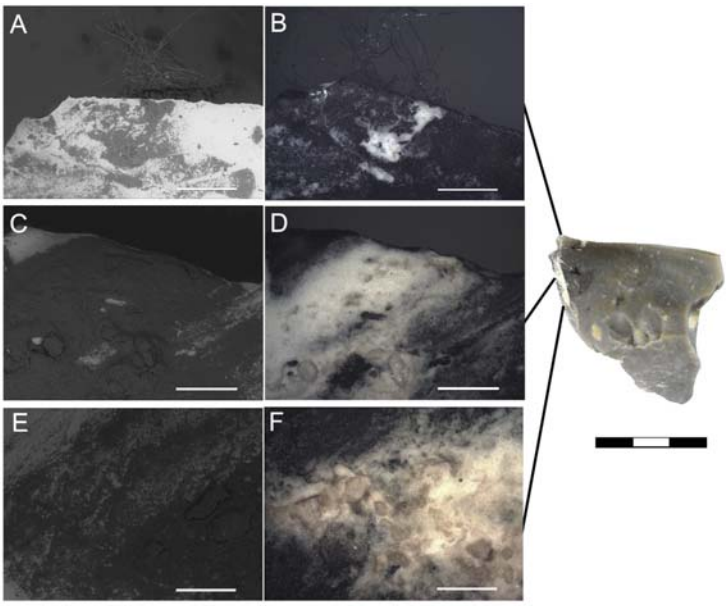

Fig. 5. An experimental flint flake (FLINT 5) exhibiting a tissue distribution all along the used edge after butchering a short-toed snake eagle specimen. The same fragment of a feather barb is shown with the aim of two microscopic techniques (A: SEM and B; OLM). The granular structure of the bird tissue is better visible in SEM images (C, E), while the distinct colour is only visible in OLM micrographs (D, F). (A, C, E: mag. = 135×, scale bar = 500 μm; B, D, F: mag. = 50×, scale bar = 500 μm. All the SEM images were provided through a back-scattered electron detector).

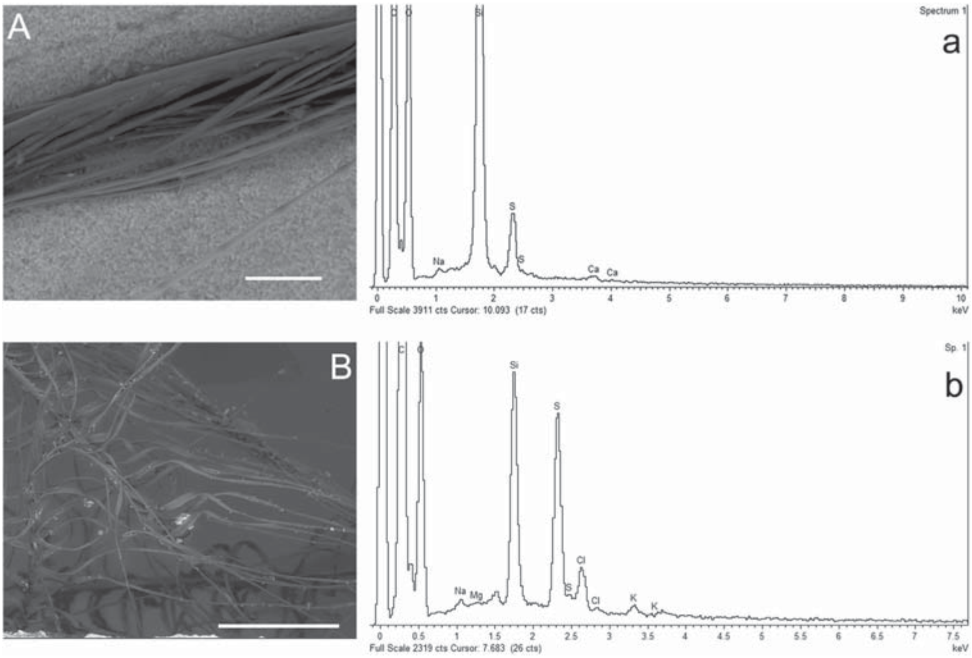

Fig. 6. SEM-BSE micro-graphs of vulture (A) and eagle (B) feathers fragments and related EDX spectra (a–b). Sulphur (S) appears in both spectra, confirming the presence of keratin. Other elements are present, though are not abundant: Sodium (Na), chlorine (Cl), and potassium (K). (A: mag. = 1250× scale bar = 50 μm; B: mag. = 510× scale bar = 200 μm).

(*Buteo buteo*) subsequent to the dismembering of the elbow by overextension. This author detected the same type of damage on archaeological material, specifically on the zones adjacent to the area of breakage of the articular ends. However, in the case presented here, bone breakage and peeling were not produced by overextension and dismembering, but were related to the extraction of primary flight feathers—primaries are connected to the carpometacarpus and phalanges and are the longest and narrowest of the regimes (particularly those attached to the phalanges).

3. Results

3.1. Use-wear patterns

Use-wear was poorly developed on the experimental flakes we analysed, even if, as in some cases, they were used for a considerably long time. The butchering of large mammals does not usually result in major changes to the lithic micro-surfaces, although differences in wear development on experimental and ethnological artefacts have been described (Beyries, 1993). A lower degree of

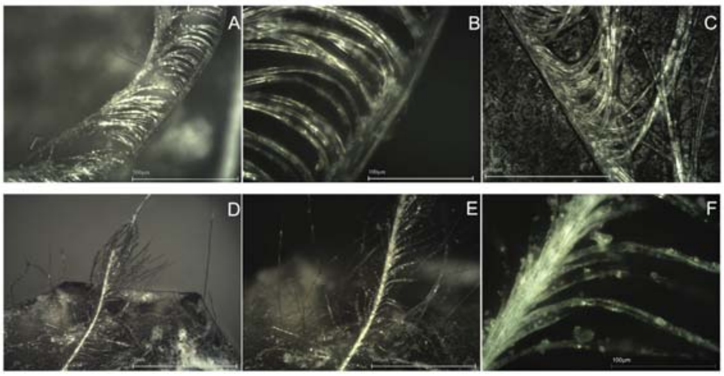

Fig. 7. Morphological structure of contour feather barbs of a short-toed snake eagle depicted through optical light microscopy (OLM). Barbules and rami are distinguishable. The employed magnifications are not succeeding in showing nodes (which are considered as the most diagnostic characters for identifying bird order and species). Nevertheless, the main parts of the barbules, the base and the pennulum, are visible (F). E and F are magnified pictures of the barb showed in the D image. (A, E: mag. = 100× scale bar = 500 μm; B, F: mag. = 500× scale bar = 100 μm; C: mag. = 200× scale bar = 300 μm; D: mag. = 50× scale bar = 1 mm).

use-wear development should be expected subsequent to butchering small game animals, considering the shorter elapsed time involved.

The edges of the flakes were poorly modified, and edge scarring was the major documented evidence. Polish, which can occur as linear features located very near the edge rim, was very rare (Fig. 3: A, B); scars were much more evident, even at low magnifications (Fig. 3: C, D). Various scar morphologies were registered (scalar, rectangular, half-moon), but none of them seemed to be prominent. No specific use-wear pattern distribution was noticed, although some recurrences can be underlined. For instance, a clear association between tiny polished areas and edge scarring was noticed (Fig. 3: E, F).

Most of the documented use-wear features were found on the face of the flake which had the major contact with the worked material (the contact face) (Table 2), whereas the opposite face retained only scar evidence. The use-wear distribution was uniform with regard to the used portion of the edge. No particular correlation between use-wear type and position on the used portion of the edge (proximal or distal) emerged. Regarding the polished linear features (which appeared sometimes as clear striations and sometimes as wider polished areas, but maintained a clear linear trend despite this difference), lengths from 50 to 250 μm were measured. The angle (upper angle) formed by them with the used edge was measured as well (always orienting the edge in the same way), with the principal objective of detecting any correlation with the performed movement of the lithic object. There was a recurrence of broad angles (>140°), with only one exception (30°). This may be related to the mainly unidirectional movements executed. To evaluate this data, more experiments involving more lithic specimens are needed.

3.2. Experimental avian residues

Griffon vulture residues were sparse and did not follow a logical distribution with regard to the used edge. Concentrations of soft tissue were found on both faces (ventral and dorsal) and were randomly distributed across the entire surface. Tissue residues were organised in compact masses of substance displaying light reddish to dark brownish tones, though they were very often accompanied by whitish tonalities, possibly due to the presence of collagen (Fig. 4: F, I). In general, colour is very important for characterising residues, and it seems to be also a fundamental aspect for distinguishing among avian residues.

Feathers occurred as single fibres identified as barbules, either found embedded within the meat remains or dispersed on the lithic surface (Fig. 10: A, B), and as fibre concentrations. Only barbules, separated from tissue, appeared as white, birefringent fibres. Nodes seemed to be somehow distinguishable, even if secure attestation was not corroborated using higher magnifications (Fig. 4: C).

In contrast to griffon vulture residues, short-toed snake eagle residues exhibited an ordered disposition spread along the entire used edge (Fig. 5). Probably due to the highly greasy nature of eagle tissue, residues accumulated on the zones involving long-term contact with the carcasses. Short-toed snake eagle residues were essentially white and exhibited a singular granular structure (Fig. 5: E, F). Colour was observable with the aid of optical devices, which showed that the white aspect of eagle tissue is sometimes complemented by pinkish tonalities (Fig. 5: F). Even if the colour is missed when the sample is scanned with SEM, the typical granular structure is better imaged though electron microscopy (Fig. 5: C, E).

Table 2
Use-wear recorded on experimental flint specimens. Use-wear types are described one by one and their mean measurements, position with respect to the flake surface (dorsal or ventral) and to the utilised edge portion (proximal, distal) are summed up.

Reference	Face	Use-wear type	Upper angle	Length μm (striae)	Width μm (scars)	Position on the used portion	Observations
FLINT 1 Left edge	Ventral	Scar associated to a linear polish	140	50	100	Proximal	Polish line is found above the scar
		General scarring	–	–	–	Proximal	–
FLINT 2 Right edge	Ventral	Striation	30	100	–	Proximal	–
		Striation	150	250	–	Distal	–
		Striation	150	150	–	Distal	
		Polish	–	–	–	Distal	Parallel to the edge
		Scar associated to a linear polish	140	100	100	Distal	Polish line is found below the scar
FLINT 3 Left edge	Ventral	Polish line	–	100	–	Proximal	–
		Scar associated to a linear polish	0	100	400	Proximal	Polish line is found above the scar and parallel to the edge
FLINT 4 Left edge	Ventral	Polish line	140	100	–	Proximal	–
	Dorsal	Half-moon scar	–	–	400	Proximal	–
		Half-moon scar	–	–	300	Distal	–
FLINT 5 Right edge	Ventral	Half-moon scar	–	–	400	Proximal	–
		Half-moon scar	–	–	300	Proximal	–
		Half-moon scar	–	–	600	Proximal	Two scars
		Half-moon scar	–	–	400	Distal	–
	Dorsal	Scar associated to a linear polish	0	50	400	Distal	Polish line is found above the scar and parallel to the edge

The scars' width was registered as well (from 100 to 600 μm), and the scars' associations with polish were described. Polish lines were found with the same frequency above and below the related scar, sometimes at the same broad angle recorded for striations, sometimes parallel to the used edge.

Because use-wear was not highly developed, we think it would be useful to document it at higher magnifications using SEM. However, as this was not the main aim of the paper, we did not resort to this technique for illustrating modifications due to use.

It is worth noting that despite the frequent contact of stone tools with the plumage of the birds and the frequent detachment of downy feathers during the experiments, we did not find many occurrences of feather fragments. When they did appear, the distinctive feather structure was observable through both microscopic techniques, though the diagnostic features usually employed for feather identification were not documented (Fig. 5: A, B; Fig. 6: A, B). The barb shown in Fig. 5 can be ascribed to a fragmented pennaceous barb (not displaying nodes or inter-nodes). No downy

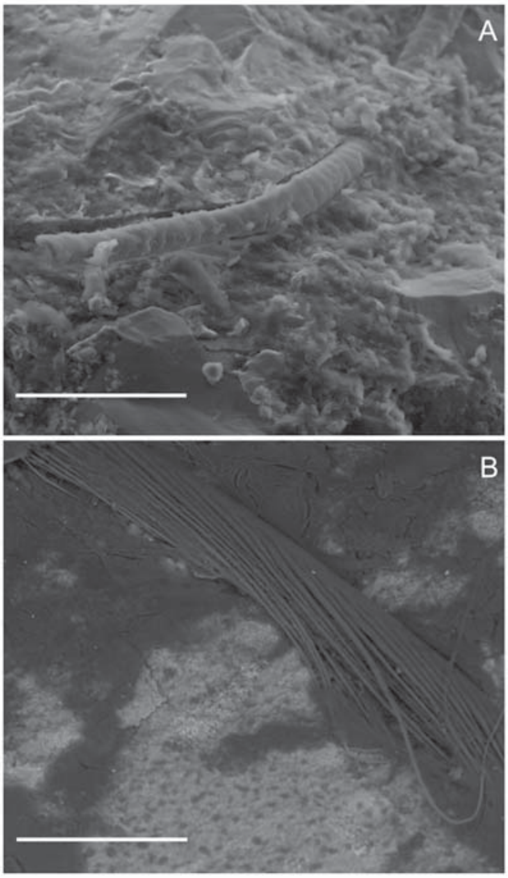

Fig. 8. Detailed micro-graphs of a modern fragment of deer hair (*Cervus elaphus*) (A) and of some griffon vulture barbules entangled all over the ramus. Even if single barbules might be misunderstood for hair fragments when scanned through OLM, the employment of SEM allows analysts to distinguish between the two residue types. The scaly cuticles of the outer layer of mammal hair are clearly visible in the LFD-SEM micrograph. (A: LFD-SEM detector, mag.: 1000×, scale bar: 100 μm; B: BSD-SEM detector, mag. 510×; scale bar: 200 μm).

feather fragments were recorded, meaning that we would not be able to provide identification at the order level, even on experimental specimens.

3.2.1. Feather characterisation

Microscopic feathers on stone tools appear as bundles of fibres, with fragmented barbs embedded in tissue patches or isolated barbules dispersed on the lithic surface (Fig. 10: A, B). The feathers' optical microscopic characteristics have been extensively described within the domain of forensic science (Dove, 2000; Dove and Koch, 2010). Such studies were able to individualise divergences among bird orders and species. Although those underscored characteristics appear to have extremely high diagnostic value for analysing relatively large feather samples, they may not be valuable for identifying archaeological feather residues. In fact, we can notice that the methodology designed for the identification of large plumage portions (Dove, 2000) is not applicable to archaeological feathers for the simple reason that the available micro-residues on stone tools frequently consist of tiny organic fragments. More importantly, their colour and structural appearance might have a less than optimal resemblance to modern residue collections due to post-depositional modifications, a fact that could make any direct comparison difficult (Monnier et al., 2012). Hence, we should adapt the knowledge offered by forensic science to specific cases in our discipline, complementing it with additional empirical techniques.

The first thing to define is how the structure of archaeological feathers is displayed at a microscopic level. Bearing in mind that archaeological residues are generally directly observed on the surfaces of stone tools, hence they are not usually properly mounted on microscope slides or stubs, many of the so-called diagnostic features (node morphology, node pigmentation and node distribution) might not be visible. Also, it should be remembered that if the observed feather fragment pertains to the pennaceous part (the most extensive part of contour feathers), it does not display any nodes and therefore cannot be identified on the basis of that feature. We observed that feathers have a birefringent appearance under an optical microscope (Fig. 7) and that SEM pictures allow us to better appreciate the barb and barbule structures. Entire barbs were easily recognisable, but single barbules could be mistaken for a hair fragment or vegetal fibres when scanned with an optical microscope. All experimental feathers displayed some microfibrils, recognised as barbules, which can be twisted together around the length of the quill (ramus) (Fig. 8: B, vulture feather), whereas in other cases single barbules were found embedded in tissue residues (Fig. 4: D, E, H).

Feather structure is better observable with an electron microscope (SEM), because it provides considerably more detail than other methods as a result of its higher magnifications and improves our capacity to describe feathers pertaining to different bird species (Lei et al., 2002; Yildiz et al., 2009). Even when nodes are not present (pennaceous barbs or the base of plumulaceous barbs), topographic traits are distinguishable through this technique (Fig. 6: A, B). Moreover, through SEM micrographs, feather structure can be more effectively distinguished from other residues, such as hair fragments and vegetal fibres. For instance, the scaly cuticle composing the outer layer of hair can be clearly differentiated from feather barbules that instead display a regular surface (Fig. 8). Furthermore, SEM offers a very useful technique by which to investigate the elemental composition of the sample, thereby improving the feasibility of residue identification (Anderson, 1980; Jahren et al., 1997; Pawlik, 2004; Byrne et al., 2006; Cristiani et al., 2009, 2014; Dinnis et al., 2009; Pawlik and Thissen, 2011). Energy-dispersive x-ray spectroscopy (EDX) provides instantaneous spectra of selected points, giving some indication of the most abundant chemical elements. The spectra directly associated with feathers (Fig. 6: a, b) show sulphur as the main peak and a lower presence of sodium and calcium. Chlorine and potassium were also detected from an eagle-feather fragment. The sulphur peak testifies to the presence of keratin, the main component of both hair and feathers (Leon, 1972; McKittrick et al., 2012).

At any rate, through both morphological and chemical elemental analyses, it is possible to obtain a satisfactory feather characterisation. Therefore, in addition to keratin detection (which points to the possible identification of the residue as either hair or a feather fragment), structure depiction plays a crucial role in the process.

3.3. Human-induced bone damage

Cut marks were the most common bone damage observed subsequent to the experimental process. These alterations were

Table 3
Cut-marked bones resulting from experimental series. Cmc = carpometacarpus; Tmt = Tarsometatarsus; Cm = Cut-marks; Inc = incisions; Saw = sawing marks; Scr = Scrape marks; Obl: oblique; Long: longitudinal; Tr: transverse; Str = straight; Curv = curved.

Taxa	Skeletal element	No. Cm	No.striations by group	Cm type	Location (region and side)	Orient.	Delin.	Measurements (mm)
G. fulvus	Furcula	3	2-1	inc	prox area, anterior side	tr	str	2.58–2.64
		2	3	inc	middle area, anterior side	obl	str	2.87–5.02
		1	1	inc	interclavicle, anterior side	tr	str	2.94
	Humerus, right	8	2-6	inc-saw	mid-shaft, medial side	trans(obl)	str-curv	2.31–6.18
		5	1-1-3	inc-saw	distal end, medial side	obl	str	3.38–5.49
	Humerus, left	7	3-4	inc-saw	distal shaft, medial side	trans	str	1.97–6.35
		4	2-2	inc	distal shaft, anterior side	trans(obl)	str	2.79–5.81
	Ulna, right	2	1-1	inc	mid-shaft, posterior side	obl(long)	str	6.43–5.48
		2	2	inc	distal shaft, medial side	obl(long)	str	13.11–13.85
		4	1-2-1	inc	distal shaft, posterior side	obl	str	3.52–4.44
	Ulna, left	8	8	inc	prox shaft, posterior (lat) side	tr-obl	str	5.79
		9	9	scr	mid-shaft, posterior (lat) side	long	str	18.39–47.13
		3	1-2	inc	distal shaft, medial side	obl	curv	7.98–9.27
		1	1	inc	distal shaft, medial (ant) side	obl	str(curv)	18.04
	Radius, right	1	1	inc	prox shaft, anterior side	obl	str	8.41
	Radius, left	3	3	inc	prox shaft, anterior (med)side	obl	str(curv)	3.91–15.37
	Cmc, right	4	1-3	inc	extensor process, ventral side	long	str	2.99–3.43
		1	1	inc	prox minor mc, ventral side	long	str	6.71
		3	3	inc	prox shaft, ventral side	obl	str	4.88–10.37
		5	5	inc	prox shaft, dorsal side	obl	str	3.44–12.65
		7	2-4-1	inc	mid-shaft, dorsal side	obl	str-curv	2.42–18.91
	Cmc, left	6	1-5	inc-saw	humeral trochlea	obl	str	2.1–7.05
		9	1-5-3	inc-saw	infratrocheal fossa, ventral side	long	str	2.17-4.52
		8	1-5-3	inc	prox insertion with minor mc, ventral side	long	str	3.45–10.41
		3	3	inc	prox shaft, ventral side	obl	str	3.34–6.89
		8	8	inc	distal end & insertion with minor mc, ventral side	long	str	2.67–7.14
		2	2	inc	prox shaft, dorsal side	obl	str	5.27–5.51
		1	1	inc	mid- shaft, dorsal side	obl	str	6.79
	First digit, right	5	5	saw	prox shaft, ventral side	obl	str	1.58–2.46
	Radiale, right	3	3	inc	dorsal side	obl	str	6.39–10.31
C. gallicus	Humerus, right	3	2-1	inc	mid-shaft, medial side	obl	str-curv	1.32–3.16
		2	2	inc	distal shaft, medial side	obl	str	2.34–3.21
	Humerus, left	8	5-3	saw-inc	distal shaft, medial side	trans	str	1.57–3.66
		2	2	inc	distal shaft, anterior side	obl	str	1.99–2.85
	Ulna, right	3	2-1	inc	mid-shaft, posterior side	long	str	2.47–6.06
		1	1	inc	distal shaft, medial side	obl(long)	str	3.57–6.58
		5	3-2	inc	distal shaft, posterior side	obl	str	1.89–4.31
	Ulna, left	2	2	inc	prox shaft, posterior side	obl	str(curv)	1.47–2.87
		4	3-1	inc	distal shaft, medial side	obl-long	str-curv	2.81–5.78
		2	2	inc	distal shaft, ant side	obl	curv	4.29
	Radius, right	4	2-2	inc	prox shaft, anterior side	obl	str(curv)	2.36–3.01
	Radius, left	5	3-2	inc	mid-shaft, anterior side	obl	str	1.69–3.05
	Cmc, right	8	2-6	inc-saw	humeral trochlea	obl-tr	str	1.23–3.31
	Cmc, left	5	2-3	inc-saw	humeral trochlea	obl-tr	str	1.47–3.06
	Radiale, right	2	2	inc	dorsal side	obl	str	0.97–1.38
	Radiale, left	1	1	inc	dorsal side	obl	str	1.25
	Tmt, right	2	2	inc	distal end (trochlea), anterior side	tr	str	0.85–1.13
	Tmt, left	1	1	inc	distal end (trochlea), anterior side	tr	str	1.27

documented mainly in the forelimb, including the humerus, ulna, radius, carpometacarpus, first digit and radial, and in the anterior face of the furcula (fused clavicle or collarbone). To a lesser extent, cut marks were also observed in both tarsometatarsus joints in the experimental series involving the short-toed snake eagle (Table 3).

Despite the difference in size between the two species—male griffon vultures weigh 6.2–10.5 kg and females weigh 6.5–11.3 kg, whereas short-toed snake eagles weigh 1.2–2.3 kg (Del Hoyo, 1994; Ferguson-Lees and Christie, 2001)—the distribution patterns of the cut marks tend to appear in the same anatomical areas. More specifically, the humerus has a clear tendency to exhibit marks on its distal shaft—of 15 groups of incisions observed on the humeri, 11 were on the distal zone. Conversely, the proximal end and shaft remained unchanged in both avian species. In soaring birds, this area is covered by strong muscular attachments (e.g., biceps brachii and pectoralis muscles) that protect the bone and prevent the tool from coming into contact with it during skinning/defeathering. However, the distal part has very little muscle covering and is therefore more susceptible to modification. The ulna and the carpometacarpus were the most altered skeletal elements. The ulna showed 20 groups of cut marks (11 in the vulture and 9 in the short-toed eagle). They appeared mainly on the distal shaft ($n = 13$) and mid-shaft ($n = 5$) (Fig. 9: A). The marks located on the mid-shaft of the ulna were mainly longitudinal and, to a lesser extent, oblique (but never transverse) orientations, and they were also the longest marks (up to 47.13 mm in a griffon vulture). This type of mark and its orientation seem to be closely related to the anatomic anchorage structure of the secondary remiges to the ulna. The ligaments that bind these remiges to the bone connect to small, rounded projections, known as quill knobs, on the ulna. During defeathering, the intuitive movement consists of placing the tool longitudinally to the bone to cut and unpin the feathers, occasionally generating longitudinal incisions over the quill knobs (Fig. 9, B). In contrast, the radius shows very little alteration ($n = 6$), and this is located almost exclusively on the proximal shaft (Fig. 9: C). Only two groups of incisions were detected on the anterior face of the mid-shaft in the case of the short-toed snake eagle. The carpometacarpus is the

Fig. 9. Some examples of damage associated to skinning and de-feathering on several skeletal elements of *Gyps fulvus* from our experiment series: A) humerus; B) ulnae; C) radius; D, E) carpometacarpi.

skeletal element that exhibited marks with the greatest frequency, with 24 groups of incisions (20 in the vulture and 4 in the short-toed eagle) (Fig. 9: D, E). In the case of the griffon vulture, the incisions are isolated and grouped along the entire carpometacarpus, with a predominance towards the proximal part ($n = 13$). By contrast, the short-toed eagle bore incisions only on the humeral trochlea of the carpometacarpus. However, this area received a different treatment compared with the vulture, because in this case the wrist joint was dismantled and the carpometacarpus was separated from the carcass and extracted with the skin. The dismembering of this area or the skinning itself also resulted in the alteration of the radial bone, which exhibited two groups of incisions in the griffon vulture and one in the short-toed eagle.

The differences observed seem to lie mainly in the decisions the butcher makes regarding the area set as the limit for removing the skin. In the case of the griffon vulture, the butcher established the carpometacarpus of both wings and the distal end of both tibiotarsi as the limit. The carpometacarpal was highly modified in both wings, although the hindlimbs showed no damage. This is probably linked to the high proportion of tendons and ligaments covering the tibiotarsal joint and the tarsometatarsus, which prevent the tool from coming into contact with the bone. However, the limits of use of the skin/feathers in the short-toed snake eagle were different. In this case, the butcher stopped the cut on the wrist joint formed by the distal end of the ulna-radius and the humeral trochlea of the carpometacarpus, leaving the latter with the skin. For this reason, the carpometacarpus showed hardly any modification compared with the griffon vulture, and alterations were concentrated exclusively on the humeral trochlea. In the hindlimbs, the limit was established at the distal end of the tarsometatarsus, leaving the phalanges adhering to the skin. This led to the tool coming into contact with the bone on two occasions, generating cuts on the trochlea of both tarsometatarsi. However, the tarsometatarsus of the griffon vulture remained intact and attached to the carcass.

Variations in processing resulted in differences regarding the location of marks on the skeleton and the presence/absence of fracturing as result of bending certain bones. Thus, only in the case of the griffon vulture was fracturing of the right carpometacarpus observed mesiodistally. This fracture has the typical characteristics that define peeling by its lateral surface with a roughened surface with parallel grooves and fibrous texture (Fig. 9: E2), and it is related to the removal of the primary feathers. From this perspective, the differences in the location of the cut marks seem to be related not only to the anatomy and physiology of the birds, but also to the technique and type of processing carried out by the butcher. The experience probably also involves a change in the frequency of the appearance of cut marks on the bones. The researchers who conducted the experiment had no experience of plucking and skinning poultry, even though they had reproduced the use of ungulate carcass usage sequences with stone tools. Thus, the data presented here as trends may vary if the sample is broad and the variables increase.

4. Discussion

4.1. Residues on archaeological record

Usually, when we analyse micro-residues, we are dealing with a set of methodological problems. First, many residues display overlapping morphologies. Particularly when we consider single fibres, we frequently find very similar morphologies and colourings, no matter which residue types we are looking at. Difficulties in discerning residue types as a result of using optical light

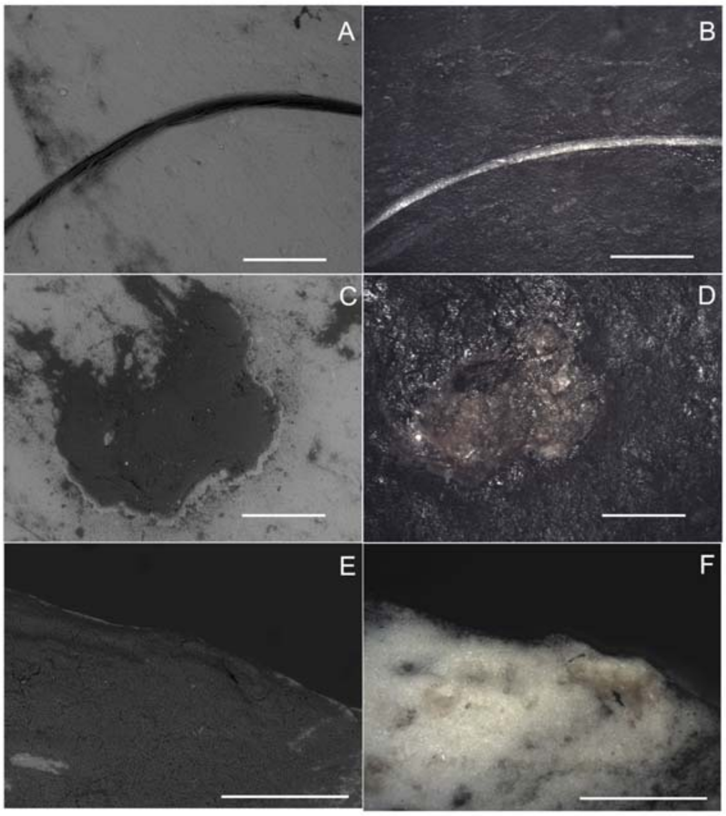

Fig. 10. Comparison of the same points that illustrate experimental avian residues obtained with different microscopic techniques, showing great complementarity. A barb of a griffon vulture specimen: the optical micrograph (B) succeeds in exhibiting the typical birefringence of feathers, which is partly responsible to hamper the detection of single barbules, instead visible in the SEM image (A). A spot of griffon vulture tissue showing characteristic reddish tonalities (D). The same point imaged through SEM (C) shows a better definition of the residue outline and the presence of secondary smaller spots being invisible with OLM (probably due to the similar colour of residue and the rock background). A relatively extended area covered with short-toed snake eagle organic tissue. Characteristic structure and colour are visible respectively in SEM (E) and OLM (F) pictures. (A, C: mag.: 135×, scale bar: 500 μm; B, D) mag.: 50×, scale bar: 500 μm; E: mag.: 260×, scale bar: 400 μm; F: mag.: 100×, scale bar: 400 μm). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

microscopy (OLM) alone have been reported elsewhere (Lombard and Wadley, 2007; Monnier et al., 2012, 2013).

The experimental data showed that fibrous material is sometimes very difficult to assign to a specific material. Animal and vegetal fibres sometimes share morphological and textural attributes, which makes their identification even more problematic. Based on our experimental observations, we noted that feather fragments also overlap morphologically with other residue categories. For this reason, it is dangerous to base the identification on optical scanning alone. Therefore, a methodology that combines different microscopic techniques is always desirable (Borel et al., 2014). In fact, the major advantage of OLM is colour observation, which helps researchers locate the presence of residues on the surfaces of stone tools (Fig. 10: B, D, F). On the other hand, the major advantages of SEM are a wider depth of field together with a high image resolution (Fig. 10, A, C, E). Due to these advantages, in addition to the possibility of reaching much higher magnifications, residue micro-topography is better observed with SEM. A large field detector (LFD) proved useful in documenting residue morphology, whereas a backscattered electron detector (Dual BSD) enabled us to more easily locate the residues thanks to phase-contrast observation. In fact, when using this detector type, different atomic numbers result in differences in greyscale tonalities. The heavier the element, the brighter its resultant image recorded by backscattered electron detector, which means that the rock surface always appears to be brighter within the darker organic material (Fig. 10: A, C, E). Systematically comparing micrographs of the same residue portion imaged by means of two microscopic techniques results in a method that maximises the potential of residue microscopic analysis (Monnier et al., 2012; Borel et al., 2014). Single barbules are detectable through OLM thanks to their birefringence (Fig. 10: B), but their structure is visible only through SEM (Fig. 10: A). Tissue colour is very useful when it contrasts with the rock substrate, helping in the rapid detection of residues (Fig. 10: F), therefore OLM is the most suitable technique to be adopted in this case. Conversely, when the residue colour is quite similar to the rock substrate, SEM is able to record even the tiniest organic spot thanks to the element inferential contrast of the back-scattered electron detector (Fig. 10: C).

A second problem, having been all but ignored within archaeological residue studies, but recently underlined by Langejans (2010), is the taphonomic effects influencing the preservation of the different types of residue. Knowing the preservation index of each residue type, direct analogies of the documented residues interpreted as the worked material are no longer possible.

We also assume that intra-site post-depositional processes modify the visual appearance of the organic residues to a large extent. It is reasonable to speculate that unavoidable changes in the residues' colour and structure occurred following burial processes. This is an additional reason why the use of optical microscopes alone is not sufficient for accurate residue identification.

4.2. Feather identification on archaeological lithic tools

There have been few attempts to identify feather residues by analysing both ethnographical (Harwood, 2011) and archaeological artefacts (Hardy et al., 2001, 2013; Hardy and Moncel, 2011; Dove and Peurach, 2002; Robertson, 2002; Dove et al., 2005).

Before going into the details of each case, some methodological considerations are in order. Although in some cases a precise procedure taken from forensic science (Dove, 2000) was used to identify ancient feather fragments (Dove and Peurach, 2002; Dove et al., 2005; Harwood, 2011), in other cases residue interpretation was based only on visual approximation from optical micrographs (Hardy et al., 2001, 2013; Hardy and Moncel, 2011; Robertson, 2002). A strict methodology for bird order/species identification requires careful specimen preparation, including the mounting of the residue on a microscope slide, microscopic observation and its morphological comparison with a wide modern feather reference collection. Thanks to these procedures, it is possible for some special characteristics, such as node morphology and feather pigmentation, to be observed. Furthermore, a large feather sample size is necessary for the evaluation of node distribution, and, when the number of barbules (of the same sample) is limited, an ornithologist's opinion should be sought (Dove and Peurach, 2002).

Unfortunately, in archaeology we do not frequently deal with feathers that show a high degree of preservation thanks to atypical environmental conditions (Dove and Peurach, 2002; Dove et al., 2005). In the majority of cases, archaeological circumstances provide us with the presence of very tiny fibres or tissue spots on the surfaces of the lithic objects employed to perform certain tasks.

Regarding the diagnostic features required for feather identification (Dove, 2000), we maintain that it is very difficult to trace them in the archaeological record. As stated above, this is the case because only downy barbules show the characteristic nodes, which permit the visual microscopic identification of feathers. Further, the ascription of feather fragments to a bird order requires the use of long barbules to evaluate node distribution or the presence of different kinds of nodes (triangular, prong, ring, crocus and spine) (Dove and Koch, 2010). Very often, however, we have nothing more than a single, fragmented portion of one barbule, which is probably insufficient to support a confident attestation of any order or family of birds.

Even if some publications' images seem to be clear enough to discern the presence of nodes (Hardy and Moncel, 2011), in others the evidence of feather residues is questionable. For instance, in Hardy et al. (2013), it was suggested that one artefact bore evidence of Accipitriformes feathers. In the image provided by the authors, nodes are not easily appreciable, and the description of the presence of one prong on the fragment would contradict this order identification, because nodes in Accipitriformes are spined, not pronged (Dove and Koch, 2010). On another artefact, a feather fragment would point to another bird order, Anseriformes. The proposed micrograph is not detailed enough to be comparable with microscopic pictures taken from microscopy manuals. Further, because downy barbules of Anseriformes exhibit very specific characteristics (the presence of both triangular-shaped nodes and prong nodes) differently distributed on their lengths, long barbules (almost complete) should be necessary (Dove and Koch, 2010). If any triangular nodes can be said to be present on this specimen, then the identification of this fragment as pertaining to Anseriformes overlooks some diagnostic criteria.

When we attempt to interpret our experimental results in light of the underlined methodological problems, we note the very rare occurrence of feather residues on flint tools after the butchering of complete avian carcasses (skinning and defeathering). If a very limited incidence of feather residues is documented on experimentally used tools, we are forced to conclude that their presence will be even less frequent on archaeological materials due to the various taphonomic processes that might have altered their primary structure until eventually making them disappear.

Moreover, in the fortunate circumstance where some archaeological feather fragments survived, we should determine their provenance through node documentation only if they are distinctive parts of a downy feather. The identification of plumage based only on light microscopy investigation has been shown to be problematic for the simple reason that not all feather fragments possess the diagnostic criteria necessary for accurate identification. Experimental data demonstrated the low frequency of downy feathers on stone tools and the high ambiguity of pennaceous barbs recognition. In fact, they do not exhibit the kind of specific morphological traits (detectable with a conventional microscope) or colour characteristics that allow us to correctly identify those fibres.

However, when observing feather fragments on archaeological tools, interpretation should always be accompanied by a chemical residue characterisation. Having recognised the presence of sulphur (which points to keratin), the investigation of its structure through SEM scanning would enable us to discard the attribution of the residue to the category of mammalian hair (Fig. 8).

Thus, with respect to the possibility of identifying feather residues on stone tools, we should remember that identification using an optical microscope may be possible when the regular feather structure is discernible, which is not always the case in very small samples or when the residues have undergone intense post-depositional processes. For this reason, we suggest always supporting optical observations with SEM analyses. We demonstrated that SEM observation and EDX, when used in conjunction, greatly improve the accuracy of residue identification (Vergès and Ollé, 2011; Monnier et al., 2012; Borel et al., 2014). SEM employed in low vacuum mode proved very useful for residue analysis, because it is neither invasive nor destructive. Its versatility is also highlighted by the fact that it does not require any special preparation of the sample and allows the direct observation of humid specimens exhibiting the exact point where EDX analysis is applied.

Moreover, the analyses applied to the lithic implements should be complemented by those from the taphonomy field, because this discipline will provide direct evidence of bird processing in the fossil bone record.

5. Conclusions

By combining data from different disciplines, we presented preliminary experimental data intended to suggest an innovative method for better determining the degree of exploitation of avifaunal resources by prehistoric human groups. A major effort was made to establish the base for a solid experimental method, in order to be further able to interpret the avifaunal impact in the diet of prehistoric human groups. After an initial phase comprising the performance of pilot experiments, we plan to expand the experimental activity to provide more data in the near future. New avian species might be included in further experimentation in order to provide a broader experimental residue collection. For a more precise chemical characterisation of residues, one potential further step is the application of more powerful techniques, such as Raman spectroscopy and Gas chromatography. Through experimentation, we demonstrated that the microscopic technique employed for residue identification plays a fundamental role in and might even influence the final interpretation. The final phase of our research will be the application of the method developed in this study to some archaeological lithic assemblages where the exploitation of avian species has already been documented through taphonomic analysis. In this way, we will be able to evaluate the methodology proposed here as well as to collect important archaeological data. In fact, since the presence of feathers in the archaeological record may point to cultural manifestations, we recognised the need to improve the microscopic method in order to investigate the direct evidence embodied by organic residues. This data can contribute useful information to the indirect evidence of avian butchering (cut marks) traditionally observed in the archaeological record.

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