D. Impedance probe fabrication and characterization

This section summarizes the fabrication process of the MicroCard bioimpedance probes.

The characterization process is also described and the main electrical features are listed.

D.1. Probe fabrication

The fabrication of the bioimpedance probes is carried out at the Centro Nacional de Microelectrónica (CNM) clean room facilities. The technological process consists of two photolithographic steps starting from a thermal oxidation to grow a thick field layer (800 nm) on four-inches P-type <100> Si wafers with a nominal thickness of 525 µm. The first photoresist layer is applied and patterned on the wafer surface in order to pattern a double titanium-platinum layer (30 + 150 nm) by using the so-called lift-off technique. Then, two Low Pressure Chemical Vapor Deposited (LPCVD) layers of SiO_2 and Si_3N_4 (300 + 700 nm) acting as passivation layer are deposited and patterned using the second photolithographic level to open the electrodes and the bonding pads.

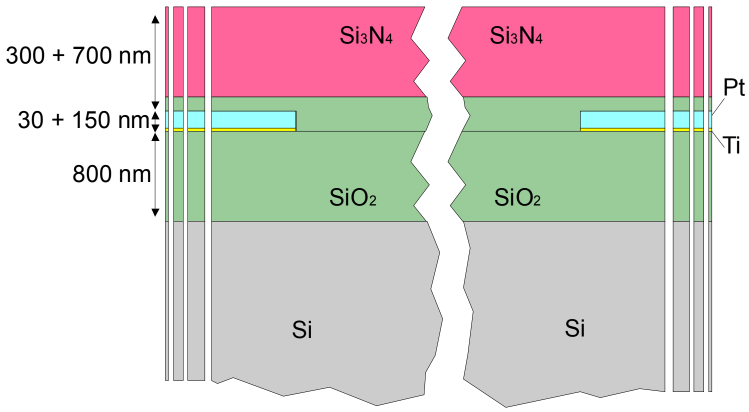

Figure D. 1. Silicon probe cross section showing all the materials used for its implementation.

After the clean room processes, the wafer is sawed by successive parallel and oblique cuts which result in a significant amount (>500) of needle shaped probes.

Each probe is fixed on a tiny Printed Circuit Board (PCB) with gold contacts and wires are connected to the electrodes through the PCB by wedge bonding. The packaging process ends with complete covering of the PCB with an epoxy resin (H77 from Epoxy Technology, Billerica,MA, USA)[1].

Afterwards, as described in chapter 2, an electrochemical deposition of platinum black on the electrodes is performed in order to decrease the electrode-electrolyte interface impedance.

[1] For certain applications, the PCB is covered with a biocompatible epoxy (OG603 from Epoxy Technology, Billerica,MA, USA).

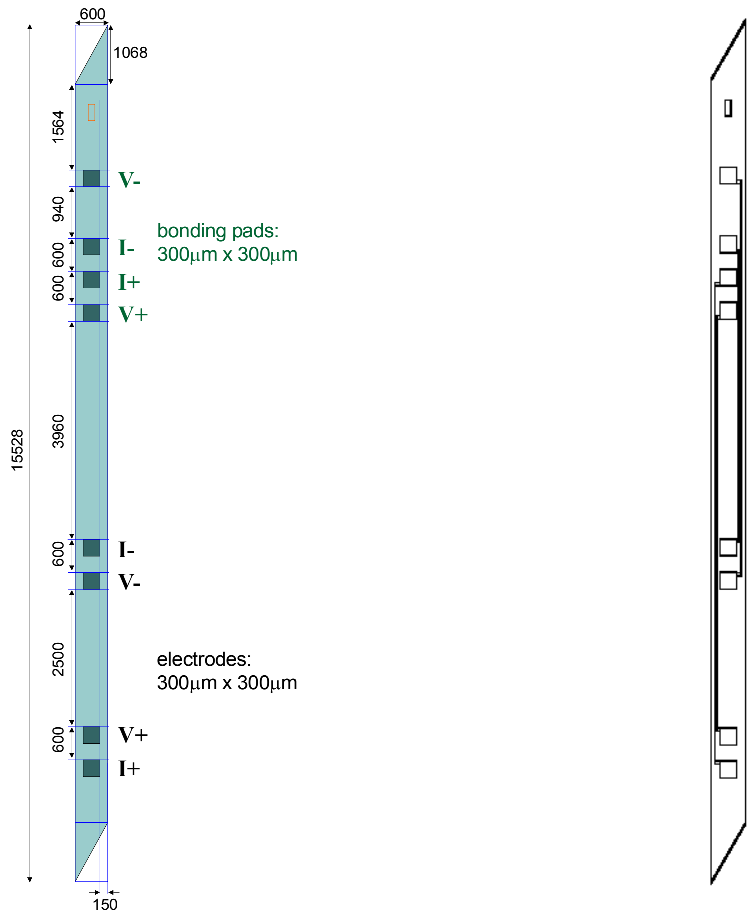

Figure D. 2. Probe dimensions (μm) and layout. The pad-electrode track width is 15 μm.

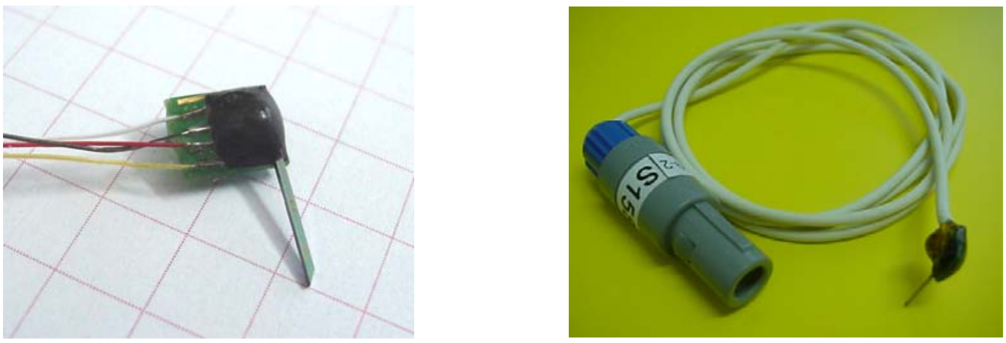

Figure D. 3. Details of probe packaging and wiring.

D.2. Electrical features

The characterization of the probes was performed by using a commercial impedance analysis system (SI 1260, Solartron Analytical from The Roxboro Group plc, Cambridge, UK) after the fabrication process has been completed. Dry inter-electrode and electrode-pad impedance measurements were performed to obtain parasite capacitances and parasite resistances.

In order to characterize the electrode-electrolyte interface impedance, the probe was immersed in physiological saline solution (0.9% NaCl, resistivity at 298 K = 71.3 Ω.cm) and impedance spectroscopy was obtained. For each electrode pair, a frequency scan from 10 Hz to 1 MHz was performed at a constant voltage amplitude of 100 mV (Figure D. 4).

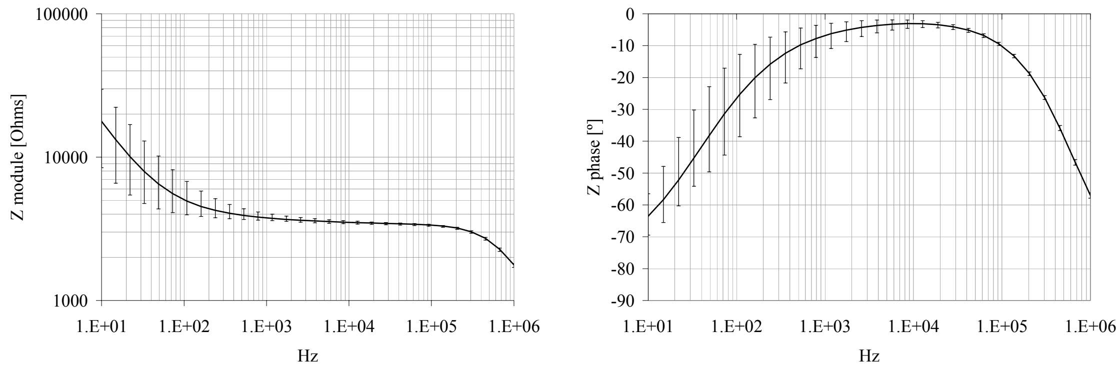

Figure D. 4. Inter-electrode impedance modulus and phase measured in NaCl 0.9% (mean ± standard deviation)

With the aid of a front-end to enhance the input properties of the SI 1260 (see the Annex C), four-electrode measurements in physiological saline solution from 10 Hz to 1 MHz were also performed in order to assess the useful frequency band.

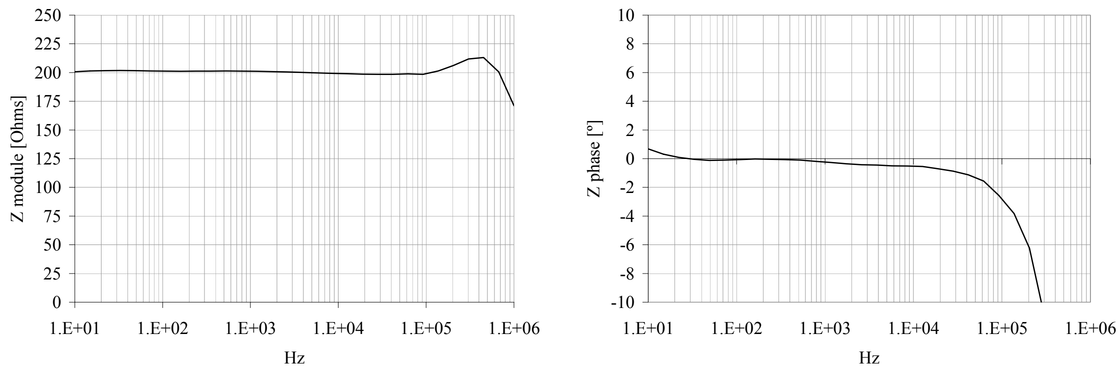

Figure D. 5. Measured four-electrode impedance magnitude and phase angle of a NaCl 0.9% solution.

The electrode-electrolyte interface impedance (Figure D. 4) becomes very high at frequencies below 100 Hz and that can involve important tissue impedance measurement errors, especially in heterogeneous tissue where each electrode can have completely different interface impedances. On the other hand, at frequencies beyond 100 kHz, the capacitive coupling of the wires is strongly manifested (Figure D. 5). Thus, it can be considered that the useful frequency band goes from 100 Hz to 100 kHz.

The results from the impedance probe characterization after the manufacturing are summarized in Table D. 1.

Table D. 1. Summarized results from the probes characterization.

parameter	conditions	minimum	typical	maximum
electrode-pad resistance:				
I+	T_A = 298 K	1050 Ω	1200 Ω	1300 Ω
V+		900 Ω	1000 Ω	1100 Ω
V-		850 Ω	1000 Ω	1050 Ω
I-		600 Ω	700 Ω	800 Ω
inter-electrode capacitance	T_A = 298 K		5 pF	6 pF
inter-electrode impedance magnitude in saline solution	T_A = 298 K 0.9 % NaCl V_{OSC}= 100 mV_P			
	10 Hz	5 kΩ	8 kΩ	25 kΩ
	100 Hz	3.5 kΩ	5 kΩ	7 kΩ
	1 kHz	3.6 kΩ	3.8 kΩ	4 kΩ
	10 kHz	3.4 kΩ	3.5 kΩ	3.6 kΩ
	100 kHz	3.2 kΩ	3.3 kΩ	3.4 kΩ
cell constant (k=ρ/R) R= measured resistance ρ= resistivity	T_A = 298 K 0.9 % NaCl V_{OSC}= 100 mV_P		0.35 cm	
spatial resolution	error < 1 %		4 mm	