

# Chapter 3

## Thermoelastic test results

The module components are glued together using a radiation-hard epoxy. The thermoelastic properties of the adhesive and its layer thickness are key parameters in the thermoelastic design of cryogenic modules. The experience of the RD39 Collaboration in operation of cryogenic sensors has shown that silicon sensors glued on glass-epoxy printed circuit board can break when cooling down. This is due to the fact that the thermal dilatation of standard epoxies is one order of magnitude higher than that of silicon. Therefore, when cooling down silicon-epoxy-silicon joints, a large stress is induced, which may result in the cracking of either the joint or the brittle substrate. Given the lack of available data in literature, an effort was made to understand and measure the thermoelastic properties of radiation hard epoxies as a function of temperature.

Several models describing epoxy matrix composite properties (Section 3.4) predict a lower thermal dilatation coefficient and a higher Young modulus for epoxies that are filled with low thermal dilatation particles. The mechanical problems arising from the large difference in thermal dilatation between epoxies and silicon suggested to study the thermoelastic properties of filled epoxies.

In this chapter we present a series of tests that have been conducted at CERN to measure the integral thermal dilatation (Section 3.1 and Section 3.2) and mechanical properties (Section 3.3) as a function of temperature of radiation-hard epoxies filled with 2  $\mu\text{m}$  fused quartz powder. The studies of radiation hardness carried out by F. Guarino *et al.* [31][32] pointed out Araldite® 2011 (Ciba), Stycast® 1266 (E&C), Rutapox L20/Rutadur SL (postcured at 60 °C) and Epoxy Type L (R&G) as suitable epoxies for high energy physics applications. In their work, the mechanical properties of samples exposed to different gamma radiation doses between 0 MGy and 4 MGy were measured. They pointed out that the mechanical properties of cyanoacrylates and silicone adhesives were seriously degraded by radiation. The epoxies chosen for our study were the suggested Araldite® 2011, Stycast® 1266 and Type L. Part of these results have been published in [33]. Section 3.5 presents a study on the glue layer thickness carried out using the measured properties and finite element simulations.

### 3.1 Measurements of the thermal dilatation of filled epoxies

#### Sample preparation

The samples were prepared by mixing weighted batches of the resin, hardener and quartz powder (Figure 3.1). After centrifugation at 2500 rpm to remove air, the samples were cured in an oven at 23 °C for 24 h, to avoid temperature fluctuations. Then they were machined to cylindrical shape with conical

ends required by the sample holder contact surfaces. All the samples were prepared and machined at CERN. A total of twenty samples were studied.

Microbubbles remaining trapped in the matrix have been reported to have a negligible effect on thermal dilatation by Hamilton *et al.* [34]. They measured the effect of these bubbles on the thermal dilatation by comparing two samples, one containing several thousand bubbles per cubic centimetre (average size of 0.1 mm) and the other free of bubbles. The total differential expansion of the samples from 300 K to 2 K was  $2.7 \cdot 10^{-4}$ . The effect of the bubbles in integral thermal dilatation is less than 10 %, with the bubbled sample expanding the most. The glass transition temperature of the samples was measured in order to control the degree of polymerization.

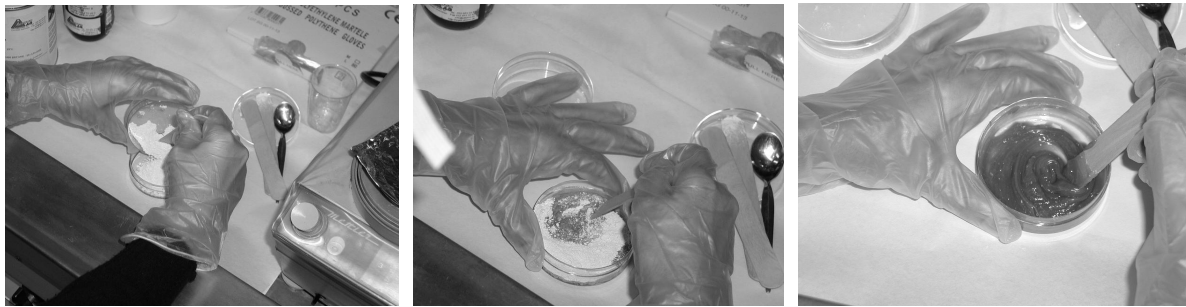


Figure 3.1 Preparation of fused quartz filled epoxy samples at CERN by mixing Stycast® 1266 and fused quartz powder.

### Experimental setup

The samples of 50 mm length were mounted in a holder sitting at the end of a tube. A rod sliding into the tube rested on the upper extremity of the sample, which was thus held in place just by the weight of the rod. An inductive displacement transducer was used to measure the position of the end rod relative to the tube. The whole apparatus was made of quartz, chosen for its low thermal contraction and conduction.

The sample holder was mounted into a stainless steel tube, which was inserted into a helium cryostat, and whose vertical position could be adjusted (Figure 3.2). The rough control of the sample temperature, measured with carbon and platinum resistor thermometers, was obtained by positioning the sample-holder with respect to the helium level, and was fine-tuned by feedback to the heater.

In order to calibrate the apparatus, a Cu sample of certified thermal contraction (SRM 736L1) was measured between 4 K and 300 K (Figure 3.3). The maximum relative error of the integral thermal dilatation was less than 3 %. The temperature was allowed to stabilize for 30 minutes before recording each data point, to ensure thorough thermalization of the sample.



Figure 3.2 Experimental setup to measure the thermal dilatation of fused quartz filled epoxies, sitting at the Low Temperature Laboratory at CERN.

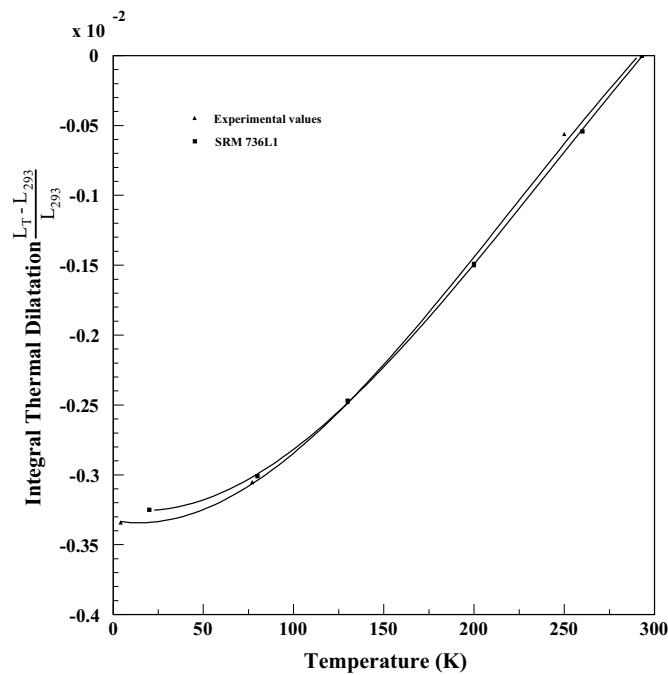


Figure 3.3 Comparison between standard (squares) and experimental values obtained with our setup (triangles) of the integral thermal dilatation of a copper sample of certified thermal contraction (SRM 736L1).

## 3.2 Results on integral thermal dilatation of filled epoxies between 4.2 K and 300 K

### Integral thermal dilatation of pure epoxies as a function of temperature

We have measured the thermal linear expansion of the three unfilled epoxies from 4.2 K to 300 K. The results are summarised in Figure 3.4. The results for Stycast® 1266 are in good agreement with those published by Oorsprong and Gijsman in 1978 [35], which are also shown in the figure. Araldite® 2011 shows the largest integral thermal dilatation, while that of Stycast® 1266 and R&G Type L is lower and of equal magnitude.

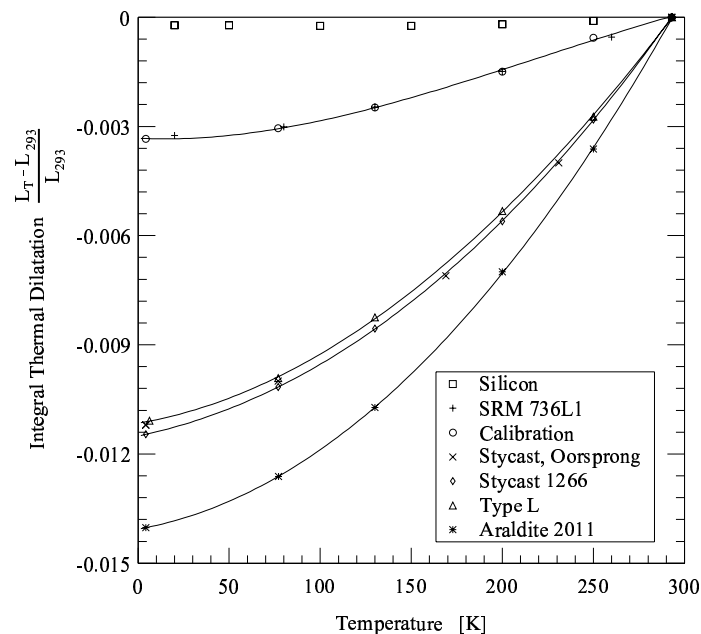


Figure 3.4 Integral thermal dilatation of silicon, copper (SRM 736L1) and unfilled Araldite® 2011, Stycast® 1266 and R&G Type L epoxies. The results for Stycast® 1266 are in good agreement with those measured by Oorsprong. The lines are fits of the data.

### Integral thermal dilatation of fused quartz filled epoxies as a function of temperature

The comparison between the integral thermal dilatation of pure and filled epoxy with different filling factors (up to 40 % in the case of Sycast® 1266, 30 % for Epoxy Type L and 10 % for Araldite® 2011) is shown in Figure 3.5. The Stycast® sample filled to 2.6 % is filled with colloidal silica<sup>1</sup>, while the others are filled with fused quartz powder.

<sup>1</sup> The colloidal silica used to prepare the samples was provided by West System® and is silicon dioxide with a particle size of 0.012 μm.

The results show how one can significantly reduce the thermal dilatation of an epoxy by filling. The thermal dilatation of Stycast® 1266 at 4.2 K is reduced by a factor of 1.5 when it is filled to 40 %. The same ratio is achieved by R&G Epoxy Type L when it is filled only to 30 %.

An interpolation function (Equation 3.1) of the variables filling factor  $\phi$  and temperature  $T$ , was fitted with the data for Stycast® 1266, Epoxy Type L and Araldite® 2011.

$$\frac{\Delta L}{L} = -A + BT + CT^2 - DT^3 + E\phi - F\phi T - G\frac{\phi}{T} \quad (3.1)$$

The results of the fit are shown in Figure 3.5, which correspond to the constants shown in Table 3.1:

Table 3.1 Coefficients of interpolation function 3.1 for Stycast® 1266, Type L and Araldite® 2011.

	A	B	C	D	E	F	G
<b>Stycast® 1266</b>	$1.15 \cdot 10^{-2}$	$8.00 \cdot 10^{-6}$	$1.26 \cdot 10^{-7}$	$7.33 \cdot 10^{-11}$	$1.38 \cdot 10^{-4}$	$4.46 \cdot 10^{-7}$	$1.00 \cdot 10^{-4}$
<b>Type L epoxy</b>	$1.12 \cdot 10^{-2}$	$7.68 \cdot 10^{-6}$	$1.29 \cdot 10^{-7}$	$9.11 \cdot 10^{-11}$	$1.47 \cdot 10^{-4}$	$4.75 \cdot 10^{-7}$	$1.17 \cdot 10^{-4}$
<b>Araldite® 2011</b>	$1.40 \cdot 10^{-2}$	$7.45 \cdot 10^{-6}$	$1.47 \cdot 10^{-7}$	$3.30 \cdot 10^{-11}$	$1.98 \cdot 10^{-4}$	$6.30 \cdot 10^{-7}$	$1.40 \cdot 10^{-4}$

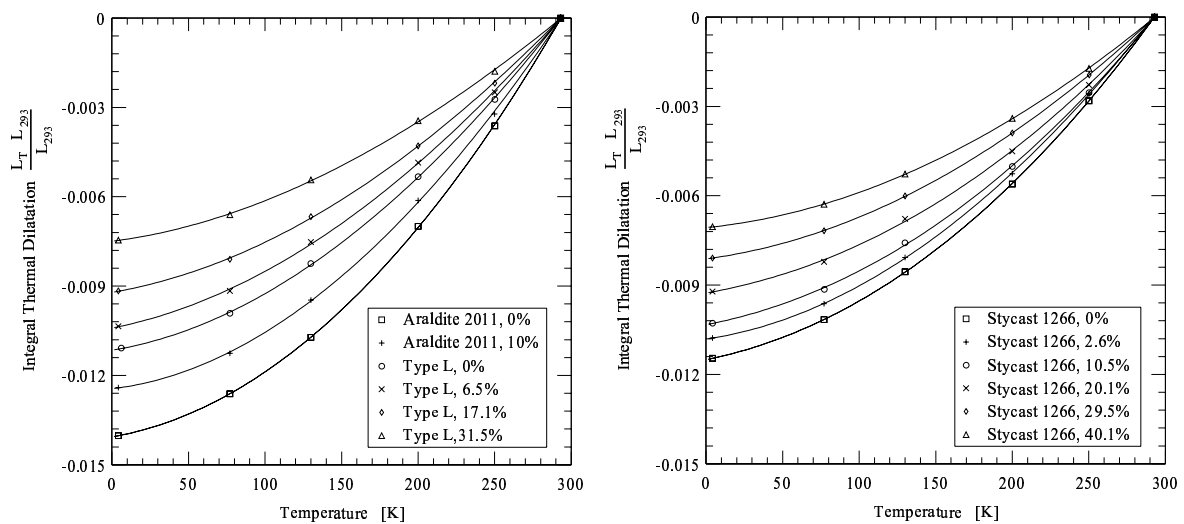


Figure 3.5 Measured (points) and fitted (lines) integral thermal dilatation of fused quartz filled Araldite® 2011 (maximum filling factor 10 %), R&G Type L (maximum filling factor 31.5 %) and Stycast® 1266 (maximum filling factor 40.1 %) as a function of temperature. The curve of the pure epoxy exhibits the larger thermal dilatation, and is progressively reduced as it is filled.

### Geometrical correction

The epoxy sample is a 50 mm long cylinder with two conical ends, where the sample is fixed through two quartz balls which are assumed to have zero thermal dilatation (see Figure 3.6). When the epoxy sample contracts, the quartz ball slides on the conical end of the sample. The length of the sample that is measured by the sensor is larger than the effective length ( $L$ ), leading to a too conservative value of the integral thermal dilatation. In order to correct this effect, a geometrical correction to the coefficient of thermal dilatation was applied.

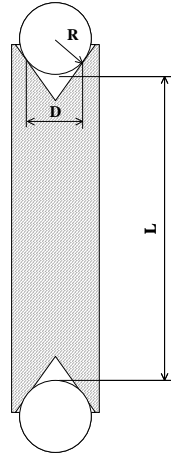


Figure 3.6 The epoxy sample is a cylinder of 50 mm with two conical ends of 90 degrees where the sensor quartz balls are inserted.

The effective contraction of the sample ( $\delta L$ ) is the measured one ( $\delta L_m$ ) to which a correction ( $\delta L_c$ ) is added:

$$\delta L = \delta L_m + \delta L_c. \quad (3.2)$$

By definition, the first term in Equation 3.2 is:

$$\delta L = \gamma \cdot L \cdot \Delta T, \quad (3.3)$$

where  $\gamma$  is the coefficient of thermal dilatation,  $L$  the effective length of the sample and  $\Delta T$  the temperature difference between two measuring points. Since the cone is 90 degree (before and after contraction), the vertical distance that the ball slides (accounting for both ends) is:

$$\delta L_c = D - (D - \gamma D \Delta T) = \gamma D \Delta T, \quad (3.4)$$

where  $D$  is related to the quartz ball radius by

$$D = \sqrt{2} R. \quad (3.5)$$

Therefore, the total correction is:

$$\delta L_c = 2 \cdot \frac{D}{2} \cdot \gamma \cdot \Delta T = \sqrt{2} \cdot R \cdot \gamma \cdot \Delta T. \quad (3.6)$$

The coefficient of thermal dilatation is then:

$$\gamma = \frac{\delta L_m}{(L - \sqrt{2} R) \Delta T}, \quad (3.7)$$

where  $\delta L_m$  is the measured value,  $L$  is the initial length,  $R$  is the radius of the quartz sensor ball. This correction has been applied to all the results.

## Error analysis

The accuracy of the integral thermal dilatation measurements is limited by several factors. A systematic error is due to the system not reaching complete thermal equilibrium during the measurement. In order to minimize this error and ensure thorough thermalization, the sample was kept oscillating around the measuring temperature (max.  $\pm 0.5$  K) during 30 minutes. Figure 3.7 shows the length of the sample measured by the transducer as a function of temperature when measuring the integral thermal dilatation at 130 K. The maximum error in the measurement can be observed in the zoom shown by the figure on the right. The length difference for two different measurements at 130 K is of the order of  $0.5 \mu\text{m}$ , which entails an error of less than 0.2 % in the integral thermal dilatation.

The temperature was measured with a calibrated carbon composite resistance thermometer in the liquid helium temperature range, and with a Pt-100 resistance thermometer for the upper temperature range. The maximum error in the temperature measurement is 1.6 %.

The inductive displacement transducer, which measures the position of the end rod relative to the tube, is sensitive down to the nanometric scale (20 V correspond to 2 mm). Therefore, the error associated to the apparatus is negligible.

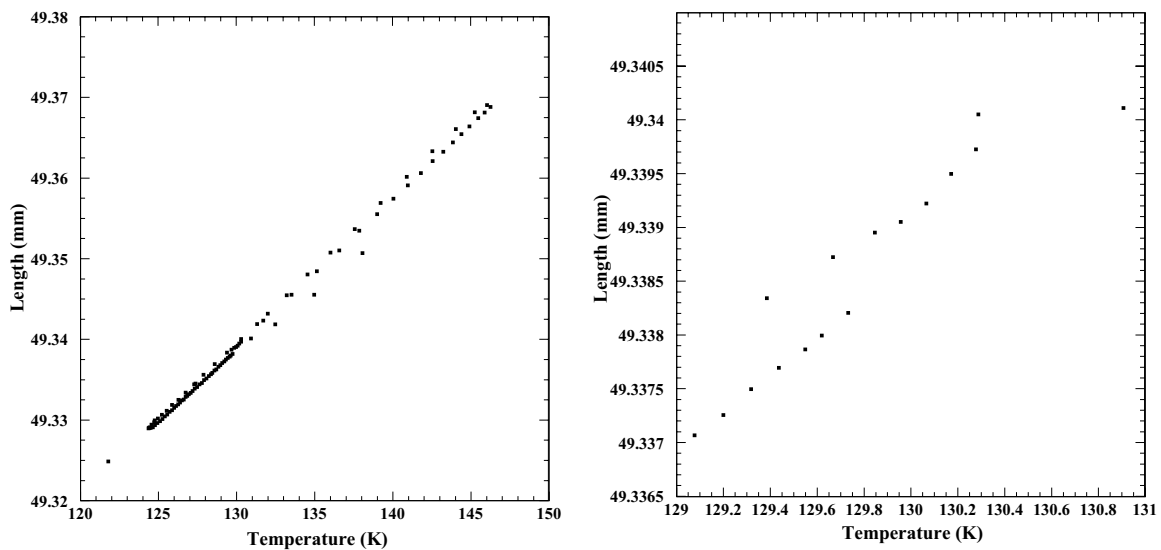


Figure 3.7 Length of the sample measured by the transducer as a function of temperature when measuring the integral thermal dilatation at 130 K (left) and magnification around the setpoint (right). There is a systematic error in the measurement of the integral thermal dilatation due to the fact that the sample does not reach thermal equilibrium. The figure shows that the maximum error in the sample length measurement is of the order of  $0.5 \mu\text{m}$  due to this lack of thermal equilibrium.

At least three different samples for each epoxy and filling factor were measured. The difference between the three measurements is less than 0.2 %. The points plotted in Figure 3.4 and Figure 3.5 are averaged values for three samples.

Therefore, both systematic and statistical errors are very small. The experimental data is presented in the figures without error bars since they are smaller than the symbols representing the measured points.

### 3.3 Measurements of the mechanical properties of filled epoxies

Young modulus, tensile strength, yield strength and elongation at break at 77 K and 300 K were measured for unfilled and filled samples of Araldite® 2011, Stycast® 1266 and Type L epoxies specially prepared and machined at CERN for this purpose. The tensile tests were conducted at a loading speed of 0.5 mm/min.

#### Mechanical properties of pure epoxies at 77 K and 300 K

The results are summarized in Table 3.2. At 300 K, Araldite® 2011 shows the lowest Young modulus (2 GPa) and the largest elongation at break (21 %). The Young modulus is 7 times higher at 77 K, and there is a clear loss of the elongation at break, which decreases below 1 %. The tensile strength is 33 MPa at room temperature and doubles its value at low temperature. The yield strength cannot be measured at low temperature since the material becomes brittle, breaking without entering into the plastic region.

Table 3.2 Pure Araldite® 2011, Stycast® 1266 and Type L mechanical properties.

	Tensile strength (MPa)	Yield strength 0.2% (MPa)	Young modulus (GPa)	Elongation at break (%)
<b>Araldite® 2011</b>				
<b>300 K</b>	33.2 ± 5.2	24.9 ± 0.4	1.8 ± 0.4	21.1 ± 10.3
<b>77 K</b>	67.8 ± 11.7	-	14.3 ± 5.3	0.7 ± 0.2
<b>Stycast® 1266</b>				
<b>300 K</b>	58.9 ± 2.8	50.9 ± 3.1	3.2 ± 0.3	1.2 ± 0.4
<b>77 K</b>	120.0 ± 11.6	-	11.0 ± 3.0	0.8 ± 0.5
<b>R&amp;G Type L</b>				
<b>300 K</b>	65.0 ± 2.6	54.0 ± 4.0	2.6 ± 0.9	1.6 ± 0.5
<b>77 K</b>	106.3 ± 17.1	-	15.5 ± 4.9	0.4 ± 0.2

At room temperature, Stycast® 1266 presents the highest Young modulus, a rather low elongation at break (~ 1% ) and a tensile strength of 59 MPa. R&G Type L has similar properties as Stycast® 1266. A loss of ductility is also appreciated at low temperature.

A typical strain-stress curve of these tests is shown in Figure 3.8. The sample is subjected to a certain strain, then released and charged again for a good measurement of the Young modulus (slope of the curve in the elastic region). However, hysteresis, and therefore a plastic behaviour is already observed for low loads, which is a common behaviour in polymers.



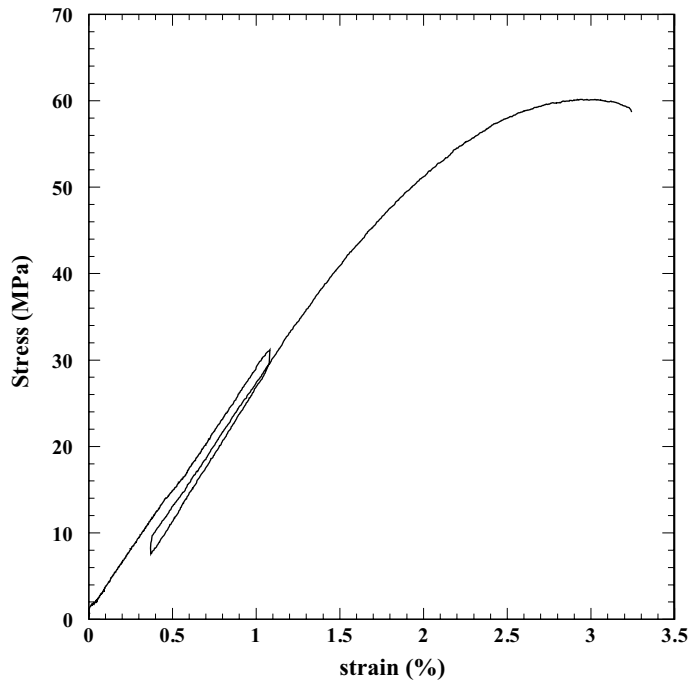


Figure 3.8 Strain-stress curve for unfilled Stycast® 1266, from which we yield the following mechanical parameters: Young modulus 2.91 GPa, elongation at break 3.24 %, yield strength 49.6 MPa and tensile strength 60.2 MPa.

### Mechanical properties of fused quartz filled epoxies at 77 K and 300 K

The same mechanical properties were measured for filled Stycast® 1266 and Type L at 77 K and 300 K. The results are summarized in Table 3.3 and Table 3.4. A large quantity of bulk material was needed in order to machine the samples. When the resins are heavily filled, it is more difficult to de-air the samples. Therefore, it was not possible to prepare many samples with high filling fractions. Stycast® could be filled up to 40 % but Epoxy Type L only to 17 %.

Table 3.3 Filled Stycast® 1266 mechanical properties at 77 K and 300 K.

filling	Tensile strength (MPa)	Yield strength 0.2% (MPa)	Young modulus (GPa)	Elongation at break (%)
<b>300 K</b>				
0 %	58.9 ± 2.8	50.9 ± 3.1	3.2 ± 0.3	1.2 ± 0.4
20 %	45.0 ± 2.8	41.5 ± 2.1	4.4 ± 0.4	0.7 ± 0.1
40 %	59	23.3	9.8	0.4
<b>77 K</b>				
0 %	120.0 ± 11.6	-	11.0 ± 3.0	0.8 ± 0.5
20 %	76.0 ± 30	-	19.0 ± 6.8	0.5 ± 0.3

In the case of Stycast® 1266, the Young modulus increases with the filling factor (triples when filling up to 40 %), while the elongation at break decreases to 0.4 % when the epoxy is filled to 40 %. The tensile strength seems to decrease with the filling factor both at room and at low temperature. The measured value of the sample filled to 40 % is not very reliable, since only one measurement could be done.

The results for Type L are shown in Table 3.4 and show the same tendencies as for Stycast®.

Table 3.4 Filled R&G Type L mechanical properties at 77 K and 300 K.

filling	Tensile strength (MPa)	Yield strength 0.2% (MPa)	Young modulus (GPa)	Elongation at break (%)
<b>300 K</b>				
<b>0 %</b>	65.0 ± 2.6	54.0 ± 4.0	2.6 ± 0.9	1.6 ± 0.5
<b>17 %</b>	52.0 ± 1.4	48.5 ± 2.1	5.0 ± 0.4	0.4 ± 0.1
<b>77 K</b>				
<b>0 %</b>	106.3 ± 17.1	-	15.5 ± 4.9	0.4 ± 0.2
<b>17 %</b>	74.5 ± 2.1	-	14.4 ± 4.1	0.5 ± 0.1

### 3.4 Comparison of the measured thermoelastic properties with theoretical models

There are several models that predict the thermoelastic properties of filled composites. The model of Hamilton, Greene and Davidson [34] treats the filler as a non-reactive non-expanding diluent. Therefore, the thermal dilatation of the composite depends linearly on the volume percentage of the filler (see Figure 3.9 and Figure 3.10).

The models of Kerner [36] and Hartwig [37][38] have a different approach, and yield a thermal dilatation and Young modulus deviating from the linear dependence on the filling factor. Both models take into account the thermal stress distribution in the filler and in the epoxy matrix. An elastic linear behaviour of the components and a complete adhesion between filler and matrix is assumed. The Young modulus of the filler is much larger than that of the resin in order to ensure that the filler can only be deformed by the temperature change.

In the Kerner model [36], the bulk modulus ( $k_0$ ) and the coefficient of thermal dilatation ( $\gamma_0$ ) of the composite are derived through tensor analysis of a large matrix, which is subjected to hydrostatic compression. The bulk modulus is:

$$k_0 = \frac{\frac{k_1(1-\phi)}{3 \cdot k_1 + 4 \cdot \mu_1} + \frac{k_2\phi}{3 \cdot k_2 + 4 \cdot \mu_1}}{\frac{(1-\phi)}{3 \cdot k_1 + 4 \cdot \mu_1} + \frac{\phi}{3 \cdot k_2 + 4 \cdot \mu_1}}, \quad (3.8)$$

where the subscript 0 refers to the composite, 1 to the resin and 2 to the filler,  $\phi$  is the filling factor and  $\mu$  is the shear modulus. The shear modulus of the composite can be calculated by:

$$\mu_0 = \frac{\frac{\mu_2 \cdot \phi}{(7 - (5 \cdot \nu_1)) \cdot \mu_1 + (8 - (10 \cdot \nu_1)) \cdot \mu_2} + \frac{(1 - \phi)}{15(1 - \nu_1)}}{\frac{\mu_1 \cdot \phi}{(7 - (5 \cdot \nu_1)) \cdot \mu_1 + (8 - (10 \cdot \nu_1)) \cdot \mu_2} + \frac{(1 - \phi)}{15(1 - \nu_1)}}, \quad (3.9)$$

where  $\nu$  stands for the Poisson's ratio<sup>1</sup>.

The expansion coefficient of the composite is

$$\gamma_0 = \gamma_1(1 - \phi) + \gamma_2\phi + 4 \cdot \frac{\mu_1}{k_0} \cdot \frac{k_0 - k_2}{4\mu_1 + 3k_2} (\gamma_1 - \gamma_2)\phi. \quad (3.10)$$

In the limit when  $\phi = 100\%$  we find that the expansion coefficient of the packed aggregate still depends apparently on the properties of the resin, but in this limit we should assume that the shear modulus of the matrix and the fluid are the same and the expansion coefficient of the resin is to be 0. Therefore for the packed aggregate the expansion coefficient is the following:

$$\gamma_0 = \gamma_2 \cdot \phi + 4 \cdot \frac{\mu_0}{k_0} \cdot \frac{k_2 - k_0}{4 \cdot \mu_0 + 3 \cdot k_2} (\gamma_2 \cdot \phi). \quad (3.11)$$

However, this is a theoretical case since the resin charged to a high percentage of filler, is no longer able to cope with its function of adhesive.

Inferring a Poisson's ratio for the pure resin of  $\nu_1 = 0.36$ , as suggested by Hartwig, and assuming a linear dependence of the Poisson's ratio with the filling factor, we can draw the Young modulus ( $E$ ) of the composite from the bulk modulus  $k$  (Equation 3.8) with the following expression:

$$k = \frac{E}{3(1 - 2\nu)}. \quad (3.12)$$

The Hartwig model [37][38] is developed for spherical and cubic particles. In the former case, the maximum allowed filling factor is 0.74. The Young modulus is:

$$E_0 \geq \frac{E_1 E_2}{E_1 \phi + E_2 (1 - \phi)}, \quad (3.13)$$

where  $E_i$  are the Young moduli. The thermal dilatation of the composite is:

$$\gamma_0 = \gamma_1 - \frac{3(1 - \nu_1)(\gamma_1 - \gamma_2)\phi^+}{2(1 - 2\nu_1)\phi^+ + 1 + \nu_1 + \frac{E_1}{E_2}(2 - 4\nu_2)(1 - \phi^+)}, \quad (3.14)$$

where  $\phi = 0.74\phi^+$ .

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<sup>1</sup> Poisson's ratio  $\nu$  is the ratio of transverse contraction strain to longitudinal extension strain in the direction of stretching force.

The comparison between the theoretical models and experimental results for the composite Young modulus favours the Kerner model, as shown by Figure 3.9.

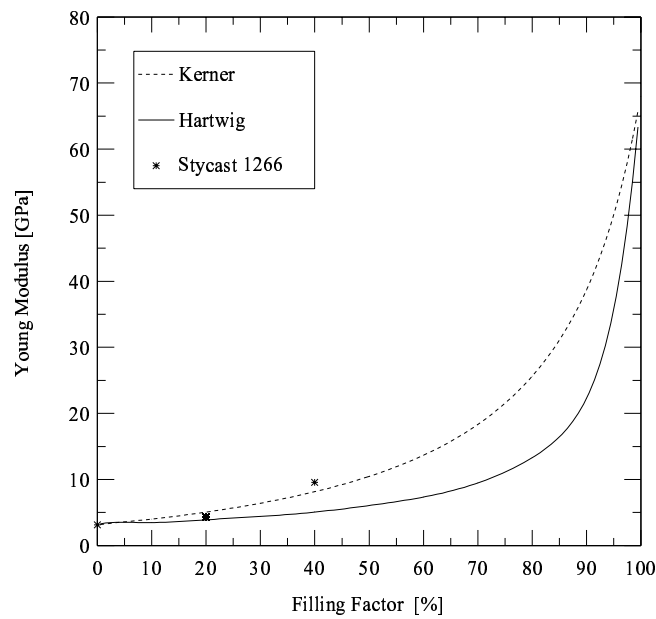


Figure 3.9 Comparison between the theoretical models which predict the Young modulus of filled composites (lines) and the experimental data (points). The Kerner model is in good agreement with the measurements of Stycast® 1266 at room temperature for filling factors of 20 % and 40 %.

Comparing the experimental thermal dilatation results with the theoretical models (Figure 3.10), the experimental points fall between the Hamilton and Kerner models. We note that the colloidal silica filled sample presents a particularly low  $\gamma$ . However, one can only get low filling factors with this nanometric powder.

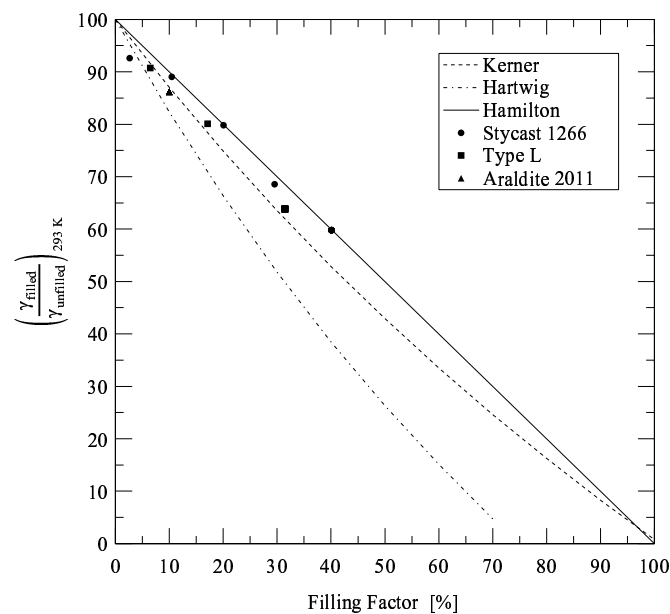


Figure 3.10 Comparison between the theoretical models (lines) which predict the thermal dilatation coefficient of filled composites and the experimental data (points). The thermal dilatation coefficient has been calculated at 293 K.

## 3.5 Epoxy layer thickness

A key parameter when gluing silicon to silicon is the thickness of the glue layer. As the thickness increases, the thermal stress induced in the silicon substrate becomes larger, and may exceed the rupture limit. A series of  $3 \times 3 \text{ cm}^2$  silicon-epoxy-silicon joints were prepared with the three candidate epoxies and with different thicknesses of glue layer. After full polymerization of the epoxy, the samples were thermal cycled from 300 K into an open bath of liquid  $\text{N}_2$  at 77 K. Samples with glue layers between  $20 \mu\text{m}$  and  $100 \mu\text{m}$  supported ten thermal cycles without the silicon or the adhesive joint breaking. Since the critical thickness of the glue layer was the parameter to be determined, thicker glue joints with Araldite® 2011 were prepared and thermally cycled. Samples with glue layers between  $500 \mu\text{m}$  and  $600 \mu\text{m}$  broke at the first thermal cool down (Figure 3.11).

Silicon to silicon joints with Araldite® 2011 layer thickness ranging from  $100 \mu\text{m}$  to  $700 \mu\text{m}$  have been simulated. The mechanical properties measured for this epoxy at low temperature (Section 3.3) were used for the simulation. However, the Young modulus at low temperature has a large experimental error, and therefore the results which are presented in Table 3.5 must be taken as orientative.

Furthermore, there is not much agreement in literature about the silicon failure strength value. While some authors [39] point out values around 7000 MPa, others quote only 300 MPa. The latter value is in fair agreement with our experimental results, since the simulations point out that stresses of that order of magnitude are developed in the silicon when thermally cycling joints with glue layers between  $500 \mu\text{m}$  and  $700 \mu\text{m}$ .

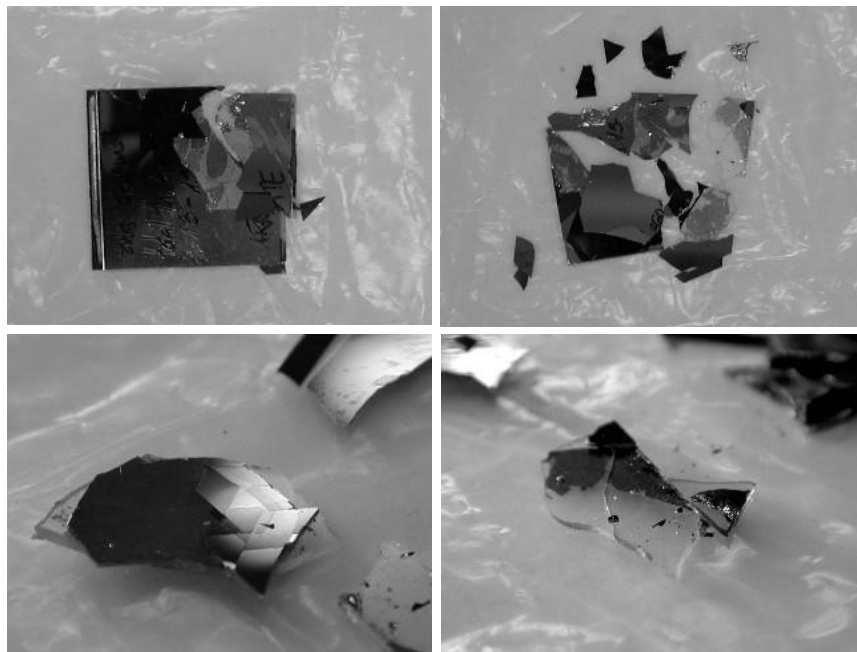


Figure 3.11 Broken silicon-epoxy-silicon joints after thermal cycling into an open bath of liquid  $\text{N}_2$ . The thickness of the Araldite® 2011 layers were ranging from  $500 \mu\text{m}$  to  $600 \mu\text{m}$ .

Table 3.5 Stress induced in a silicon-epoxy-silicon when thermal cycled to liquid N<sub>2</sub>, as obtained in the finite element simulation.

Glue layer thickness	$\sigma_{eq}$ Silicon (max)	$\sigma_{eq}$ Adhesive (max)
100 $\mu\text{m}$	80 MPa	270 MPa
300 $\mu\text{m}$	170 MPa	281 MPa
500 $\mu\text{m}$	245 MPa	289 MPa
700 $\mu\text{m}$	298 MPa	323 MPa

### 3.6 Summary of the thermoelastic properties measurements

Due to the lack of data on thermoelastic properties in literature, and being a key factor in the cryogenic module design, a series of measurements were conducted at CERN. The thermoelastic properties of Araldite® 2011 (Ciba), Stycast® 1266 (E&C) and Type L (R&G) epoxies filled with fused quartz powder were measured as a function of temperature. The choice of the epoxy and filling factor depends on the geometry and materials to glue.

The measurements show that filling these epoxies with fused quartz powder considerably reduces their thermal dilatation, nearly matching that of metals. This is very convenient in our application, since less stress would be induced when cooling down. At equal filling factors, Type L shows a larger reduction of the thermal dilatation than the other epoxies.

However, filling increases the Young modulus of the composite, which is not convenient in our application. The Kerner model fits best for predicting the dependence of the Young modulus of the composite with the filling factor. The tensile tests confirm that pure epoxies become brittle at low temperatures, when their elastic modulus increases and the elongation at break is reduced. The Young modulus at 77 K is between 4 and 8 times higher than that measured at 300 K. The tests have shown that the increase of the Young modulus at low temperature for the pure material is larger than the increase due to the change of temperature when the resin is filled. Similar embrittlement effects were found when filling the epoxy.

These measurements were used for numeric modeling (ANSYS) and comparison of silicon-epoxy-silicon joints with the three different epoxies and layer thickness. According to the simulations, the critical adhesive layer, which would lead to the silicon substrate cracking in a thermal cycle to 77 K, is of the order of 600  $\mu\text{m}$ . As thin layers as possible should be used to induce the minimum stress on the silicon substrate.

Based on these results, unfilled Araldite® 2011 was chosen for the prototype module assembly. While this epoxy is very similar to Stycast® 1266 in terms of mechanical properties (low Young modulus and large elongation at break), Araldite® 2011 is very convenient from a practical point of view for its low viscosity and packaging with a mixing nozzle. The latter prevents air bubbles being incorporated when mixing the resin and hardener (and therefore the centrifugation step can be avoided), and guarantees good proportions of both components.