

Thermal and mechanical properties of advanced impregnation materials for HTS cables and coils

N Bagrets, S Otten, K.–P. Weiss, A. Kario, W. Goldacker

Institute for Technical Physics, Karlsruhe Institute of Technology, Germany
E-mail: nadezda.bagrets@kit.edu

Abstract. In the growing field of high-temperature superconducting (HTS) applications, finding an appropriate impregnation material for cables and coils remains a challenging task. In HTS cables and coils, tapes have to be able to withstand mechanical loads during operation. Impregnation is playing a role as mechanical stabilization. However, material properties usually change significantly when going to low temperatures which can decrease performance of superconducting devices. For example, a large mismatch in thermal expansion between a conductor and impregnation material at low temperatures can lead to delamination and to degradation of the critical current. Impregnation materials can insulate tapes thermally which can lead to damage of the superconducting device in case of quench. Thus, thermal conductivity is an important property which is responsible for the temperature distribution in a superconducting cable or in a coil. Due to Lorentz forces acting on structural materials in a superconducting device, the mechanical properties of these materials should be investigated at operating temperatures of this device. Therefore, it is important to identify an advanced impregnation material meeting all specific requirements. In this paper, thermal and mechanical properties of impregnation material candidates with added fillers are presented in a temperature range from 300 K to 4 K.

1. Introduction

Impregnation of HTS devices is applied for mechanical reinforcement against Lorentz forces or to facilitate heat conduction in conduction-cooled coils. Common impregnation materials for superconducting as well as resistive coils are epoxy resins. Early attempts to impregnate HTS devices with such resins, however, showed that the thermal expansion mismatch can lead to damage of the conductor [1]. Therefore, alternative impregnation materials including epoxy resins with filler particles [2], paraffin [3], or introducing barrier between conductor and epoxy resin [4] are considered and applied. Filler particles decrease the thermal expansion mismatch with the conductor; beeswax, paraffin, or epoxy with shrink tube as barrier do not bond to the conductor and, in this way, prevent transmission of thermal stresses. The main aim of this work is characterisation and analysis of several commercially available resins for Roebel cable impregnation. Recently, it was shown that transverse mechanical loads lead to degradation of critical current of the Roebel cable due to its complex architecture [5]. However, such transverse stresses are unavoidable in superconducting magnets, therefore appropriate impregnation of the cable is needed.

In this work, the low-temperature properties of several commercially available filled epoxy resins are investigated. The thermal conductivity (TC) and thermal expansion (TE) are determined as a function of temperature in the range from 4 K up to 300 K. The tensile stress-strain behaviour is measured at room temperature, in liquid nitrogen ($T = 77$ K) and in liquid helium ($T = 4.2$ K).



2. Samples and measurement procedure

2.1 Materials description

Two groups of epoxies – candidates for impregnation of cables – were investigated: electrically conductive and insulating. Silver and graphite-filled epoxies (Duralco 125/127) were purchased from Polytec. Insulating resins were offered for testing by Huntsman Corporation. These are Araldite CY5538 with hardener HY5571, and Araldite CW5730N with hardener Aradur HY5731 and with $\text{Al}(\text{OH})_3$ as a filler. Araldite CY5538 with hardener HY5571 is supplied unfilled. As filler in different mixing ratios (50 and 60 %), fused silica flour Silbond FW600 EST with a median grain size of $4\mu\text{m}$ was used. Araldite CW5730N is pre-filled with 56 wt% aluminium hydroxide. An overview of the materials tested is shown in table 1.

The samples for the thermal expansion measurements were prepared in a Teflon mould according to the instructions of the manufacturer. The resulting samples had a size of 60 mm x 10 mm x 5 mm. For TC measurements, cylindrical samples of 6mm diameter and 2–3 mm length were used. These were cut from the larger TE samples.

The samples for mechanical test were shaped according to the DIN EN ISO 527–2 norm. Those samples were machined from a 30 mm x 50 mm x 90 mm block. In the case of silica-filled resin, preparing such a large sample could result in agglomeration of particles leading to an inhomogeneous sample. To avoid this, the mould was rotated continuously around its horizontal axis during the curing process.

Table 1. Overview of tested materials.

	Viscosity [Pa.s]/ Processing temperature [°C]	Filler	Filling ratio [wt%]	Product name
Electrically conductive fillers				
epoxy+silver	20/20	Silver	60-80	Duralco 125
epoxy+graphite	50/20	Graphite	50-60	Duralco 127
Electrically insulating fillers				
Epoxy	<0.2/40			Araldite MY750/ Aradur HY5922
epoxy+silica 60	<4.5/80	Silbond FW600 EST	60	Araldite CY5538/ Aradur HY5571-1
epoxy+silica 50	<5/80	Silbond FW600 EST	50	Araldite CY5537/ Aradur HY5571s-1
epoxy+ $\text{Al}(\text{OH})_3$	0.7/60	$\text{Al}(\text{OH})_3$	56	Araldite CW5730N/ Aradur HY5731

2.2 Measurement methods

The thermal conductivity is measured with the axial heat flow method within the Physical Property Measurement System of Quantum Design using a Thermal Transport Option in the steady-state measurement mode [6]. The thermal conductivity k is defined as

$$k = \frac{P\Delta x}{A\Delta T} \quad (1)$$

where P is the heat power, A is the cross-sectional area, Δx is the distance between the two measurement points with the temperature gradient ΔT .

Thermal expansion measurements were performed in the temperature range from 4.5 K up to 290 K in a glass-fibre reinforced plastic (GFRP) ^4He -bath cryostat. For sample deformation measurements clip-on extensometers are used [7]. The samples with extensometers mounted on it is then placed in a ^4He -bath cryostat and cooled down in a flow of He-gas. Temperature sensor was placed directly on the sample. At the end, a moderate amount of liquid He is filled. The measurement is performed when all liquid ^4He is evaporated and the temperature in the cryostat slowly rises due to the non-ideal insulating vacuum. The warm-up takes approximately 12 hours for the whole temperature range from 4.5 K to 290 K at an average rate of < 0.5 K/min.

For load-displacement measurements, a 100 kN tensile testing machine was used. Tests were performed according to DIN EN ISO 527-2. For measurements of the elongation, a system of extensometers was used, which was mounted directly on the sample. The start length is defined as the distance between the tips of extensometers. Further, the elongation of the sample results in a deformation of the extensometer frames and change in output voltage signal, which provides the real elongation via a calibration factor. The strain is then evaluated as

$$\varepsilon = \Delta L/L_0, \quad (2)$$

where ε is a strain, ΔL is an elongation, and L_0 is a start length.

3. Results

As it was mentioned above, thermal expansion is an important property for impregnation of cables and coils since it determines the level of thermal stresses between epoxy and REBCO tapes. It differs by up to two times for different epoxies and fillers. TE in temperature range from RT down to 4.5 K is shown in Table 2. TE of REBCO tape, epoxy with alumina (Stycast® 2850FT blue) [8] are shown for comparison.

Table 2. TE of different resins in temperature range from 4.5 K up to RT.

Material	TE RT-77 K %	TE RT-4.5 K %
epoxy+silver	-0.96	-1.04
epoxy+graphite	-0.53	-0.58
Epoxy	-1.04	-1.15
epoxy+silica 60	-0.51	-0.60
epoxy+silica 50	-0.61	-0.67
epoxy+Al(OH) ₃	-1.02	-1.11
Stycast® 2850FT	-0.43	-0.49
REBCO tape	-0.25	-0.26

The TE of unfilled epoxy is several times larger than that of REBCO tape. Pure epoxy has a thermal expansion of -1.15 % going from RT to 4.5 K. Since filler materials themselves have lower TE, all fillers decrease the thermal expansion as it was expected. Epoxy with silver and epoxy with Al(OH)₃ filler shows the thermal expansion of about -1% going from RT to 4.5 K. Effect of adding fillers is much more pronounced for epoxies with silica and graphite – their TE is about -0.5 – -0.7 %, which is much closer to TE of a REBCO tape.

TC measurements results are shown in Fig. 1. Thermal conductivity of epoxies with good conductive fillers (aluminium, graphite, silver) is much higher than that of pure epoxy resin. However, effect of fillers is much more pronounced for RT and 77 K than for 4 K. At 4 K thermal conductivity

of all resins is quite low – around 0.05 W/Km (except of epoxy+silver, which TC is approx. three times higher) – and the effect of fillers doesn't show any clear trend.

Pure epoxy has lowest thermal conductivity. Adding non-metallic fillers increases TC. Adding silica fillers doesn't show that large impact on TC, whereas graphite filler increases TC appreciably. Epoxy with silver shows highest TC values, due to high thermal conductivity of silver.

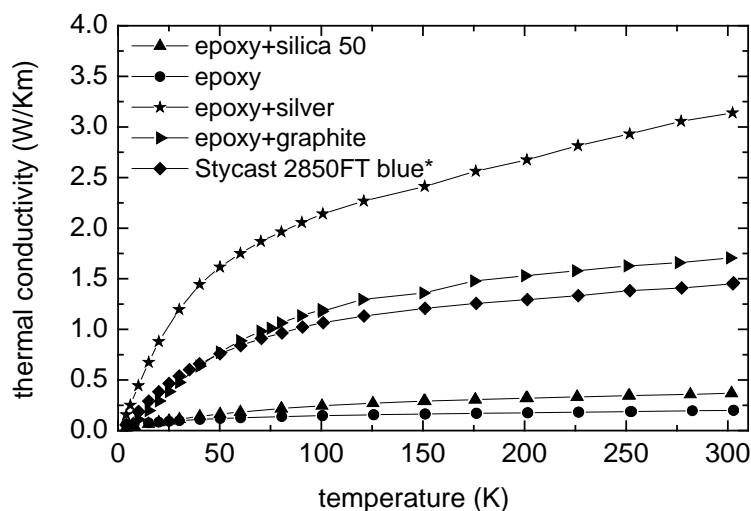


Fig.1 Thermal conductivity as a function of temperature for different epoxies with fillers TC of *Stycast® 2850TF blue [8] is shown as well for comparison.

For impregnation of Roebel cables and coils epoxies with fillers with possibly lower TE und higher TC are desired. However not all materials described above can be recommended for this application. One reason is high viscosity of electrically conductive resins (see table 1) that impedes epoxy flow into open spaces within the cable. Conductive resins are not needed for impregnation of coils, however they can be considered for other HTS applications where electrical connection is desired as moderate coupling of strands in cables.

Taking into account TE, TC and viscosity two epoxy's were chosen for mechanical tests pure epoxy (Araldite MY750/HY5922) and epoxy+silica 50 (Araldite CY5537/HY5571-1 with Silbond FW600 EST).

In Table 3 the main mechanical properties – Young's modulus, proof strength $R_{p0.1}$, and ultimate strength – are summarized for all samples at RT, 77 K, and 4.2 K. The proof strength was evaluated using the offset method with 0.1 % of strain.

Table 3. Mechanical properties of epoxy and epoxy with filler at different temperatures.

Material	Temperature K	Young's modulus GPa	$R_{p0.1}$ MPa	Ultimate strength MPa
Epoxy	RT	3.06	38	57.6
Epoxy	77	6.67	125	177
Epoxy	4.2	7.28	156	158
epoxy+silica 50	RT	6.91	48	66.4

epoxy+silica 50	77	13.92	-	149
epoxy+silica 50	4.2	15.33	-	101

Adding silica filler to epoxy increases Young's modulus approx. twice. The data obtained for epoxies correlate well with data given in literature [9]. Young's moduli for unfilled epoxy are very similar to that reported in [9], whereas for filled epoxy we got higher values as reported in [9] for other commercially available filled resins.

Ultimate strength is higher for unfilled resin than that filled for at 77 K, and 4 K. At room temperature ultimate strength is slightly higher for filled resin. Ultimate strength shows the same trend and similar values for filled and unfilled resin, as it was reported in [9]. Ultimate strength is higher at 77 K than that at RT, and it is lower for 4 K than that for 77 K.

4. Conclusions

The thermal expansion, thermal conductivity and mechanical behaviour under tensile stress were analysed for several filled epoxy resins in the temperature range 4.5–293 K. It was found that all tested fillers (silver, graphite, fused silica and aluminium hydroxide) lead to a significant reduction of the thermal expansion, which will decrease thermal stresses when cooling down in impregnated devices. Filler particles, especially silver, increase thermal conductivity. However, this increase is not pronounced at liquid helium temperature. Fused silica filled resin showed an increased Young's modulus and decreased ultimate strength in the entire temperature ranges. The first impregnation result of Roebel cable with fused silica filled resin was recently shown [10]. Sufficient improvement in stability of cable against transversal stress was achieved, however still some degradation of critical current was reported. Trials on cable impregnation avoiding current reduction are ongoing.

The resins investigated were qualified for cryogenic usage; the needs of the specific HTS applications have to refer to the individual enhanced properties, where desired strength, heat conduction and thermal expansion play a role.

Acknowledgments

We would like to thank Huntsman Corporation for providing epoxy resins and Quarzwerke GmbH for providing the silica flour.

- [1] Takematzu T, Hu R, Takao T, Yanagisawa Y, Nakagome H, Ugliette D, Kiyoshi T, Takahashi M, Maeda H 2010 *Physica C*. **470.1718** 674
- [2] Barth C, Bagrets N, Weiss K, Bayer C, Bast T 2013 *Superconductor Science and Technology*. **26.5** 055007
- [3] Matsumoto S, Kiyoshi T, Otsuka A, Hamada M, Maeda H, Yanagisawa Y, Nakagome Y, suematsu H 2012 *Superconductor Science and Technology*. **25.2** 025017
- [4] Trociewitz U P, Dalban-Canassy M, Hannion M, Hilton D K, Jaroszynski J, Noyes P, Viouchkov Y, Weijers H W and Larbalestier D C 2011 *Appl. Phys. Lett.* **99** 202506
- [5] Uglietti D, Wesche R and Bruzzone P 2013 *Superconductor Science and Technology*. **26** 074002
- [6] PPMS - Thermal Transport Application Note [Online]. Available: www.lot.orient.com/uk/en/home/ppms, 2011.
- [7] Nyilas A. *Conf. Proc. 824 Advances in Cryogenic Engineering: Transactions of the International Cryogenic Materials Conference-ICMC*. **52**, 27.
- [8] Bagrets N, Goldacker W, Schlachter S I, Barth C, Weiss K. *Cryogenics*. **61** 8
- [9] Reed R P, Walsh R P, *Conf. Proc. Advances in Cryogenic Engineering: Transactions of the International Cryogenic Materials Conference-ICMC*. **40**, pp. 1129-1136.
- [10] Otten S, Dhallé M, Gao P, Wessel W, Kario A, Kling A, Golgacker W 2015, *Superconductor Science and Technology*. **28** 065014