ICEC29-ICMC2024 IOP Publishing
IOP Conf. Series: Materials Science and Engineering **1327** (2025) 012208 doi:10.1088/1757-899X/1327/1/012208

Adhesion analysis of epoxy resin impregnation systems for Nb_3Sn superconducting magnets

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Abstract. The mechanical properties of Nb_3Sn coils are strongly influenced by the adhesion between the impregnation resin and the coil constituents, which may impact the magnets' performance. To improve the understanding of the parameters governing the adhesion in such superconducting magnets, a study was conducted on several impregnation systems with respect to different coil parts, notably copper, stainless steel, aluminium, and glass fibre. The study evaluated the adhesive strength in various test configurations, considering the influence of substrate surface conditions. The effect of cryogenic environment on the adhesion strength of the most commonly used epoxy resin was also studied. Additionally, the surface tension of the resins was measured, and an adhesion analysis was performed. The experimental adhesion results were found to be in accordance with the theoretical predictions of the adhesion analysis. The obtained results provide insights into potential modifications of the epoxy resin formulation and surface treatment methods to achieve specific wetting properties on the surfaces. This, in turn will impact the adhesion between the impregnation system and the coil constituents thereby potentially impacting the magnet's performance.

Keywords: Superconducting magnet, epoxy systems, adhesion, wetting analysis.

1. Introduction

At the status of the technology, accelerator magnets with a nominal field of 11 T or more rely exclusively on Nb_3Sn superconductors. The achievement of high fields comes at the cost of dealing with a brittle component such as a Nb_3Sn conductor after heat treatment reaction. Consequently, to minimize the degradation of the conductor, the Nb_3Sn coils are impregnated with an epoxy system whose function is to provide mechanical robustness and electrical insulation.

However, due to thermal contractions mismatches and high Lorentz forces, cracks, delamination, and pole-turn detachments have been reported on Nb_3Sn magnets following powering tests [1]. The epoxy cracking and bonding failures are two potential causes of Nb_3Sn magnets training and performance limitations [2][3].

Understanding the parameters controlling the interaction between material interfaces is critical for designing and constructing superconducting magnets. This study aims to evaluate and compare the adhesion strength of four epoxy systems for vacuum impregnation and two epoxy-based adhesives containing inorganic fillers, which are used in superconducting magnets, with respect to different substrates present in superconducting coils.

The study was divided into two parts to achieve the objective: a predictive assessment of the interaction between surfaces and epoxy, followed by experimental validation. First, wetting analysis was performed to understand the effect of surface modification on the substrates. Contact angles were measured, and surface energies were determined. The most suitable surface modification was selected for the preparation of all the samples used in the experimental phase. Surface tension of the resins was measured, and adhesion analysis was carried out to theoretically evaluate the behaviour of the epoxy with the surfaces. These predictions were then validated by conducting adhesion tests at room temperature using four different configurations: T-Peel, roller peel, double lap shear, and butt joint. The double lap shear joint was also performed for the resin CTD101K under cryogenic environment (77K). CTD101K was chosen because it is the epoxy system used to build the Nb_3Sn magnets of the High-Luminosity upgade of the LHC [4].

2. Materials and methods

2.1 Materials

The substrates considered in the study are Electrolytic tough-pitch copper (Cu ETP), austenitic stainless steel (SS 304L) and Aluminium EN AW 6082, in the following referred to as aluminium (Al).

Four epoxy systems of potential use for vacuum impregnation of Nb_3Sn coils were selected: CTD101K [5], MY750, Mix61[6], Araldite® F. The mechanical and visco-elastic properties, as well as the processing details of the above mentioned systems are widely discussed in references [7],[8],[9]. To provide a uniform adhesive thickness in the production of the adhesion test samples, S2-type fiberglass with sizing 493 was placed between the substrates, serving as a spacer.

The experimental part includes also the bonding strenght test of two other adhesive systems, namely Araldite® 2011 and Stycast 2850 FT (with CAT 24LV), which were selected since widely used in cryogenic application for encapsulation of electrical components and glueing of metals or plastics.

Table 1. Material properties of the selected vacuum impregnation systems from Gaarud et al. [7].

	CTD101K	***Araldite F***	***MY750***	***Mix 61***
Tensile modulus E (GPa)	3.53 ± 0.08	3.30 ± 0.06	3.05 ± 0.07	0.67 ± 0.07
Yield stress σ_y (MPa)	-	54	46	13
Ultimate stress σ_M (MPa)	46 ±3.2	59 ± 5.7	60 ± 0.4	20 ± 0.4
Strain at ultimate stress ε_B (%)	1.3 ± 0.1	2.0 ± 0.3	2.8 ± 0.1	19 ± 0.4

2.2 Surface treatment of substrates

To carry out the experimental tests, all the adherend surfaces were prepared by the following steps: (1) solvent cleaning, the surfaces were cleaned by first ethanol followed by acetone; (2) sandblasting; (3) atmospheric plasma cleaning with a power set to 200 W and treatment duration of approximately 1 minute at a distance from the target between 30-40 mm.

2.3 Wetting and adhesion analysis

The ability of the resin to spread on the surface was predicted by carrying out wetting analysis of the surfaces. Wetting analysis included the determination of the surface free energy (SFE) through contact angles measurement of the different substrates and the surface tension (ST) of liquids. For the measurement of SFE and contact angles, a Mobile Surface Analyzer (MSA) One-click

SFE(Kruss GmbH) was used. The machine was used in double sessile drop mode, with water and diiodomethane as test liquids. The drop volume was kept around 2 µL and the polar and dispersive components of the SFE of the solid surfaces were determined as per the Owens-Wendt-Rabel-Kaelble (OWRK) model [10]. For the measurement of ST of the resins, a Wilhelmy plate apparatus (KSV Nima) was used. The Wilhelmy plate used had the following dimensions: width of 19.62 mm, thickness of 0.1 mm, circumference of 39.44 mm and height of 38 mm. Adhesion analysis was then carried out based on the OWRK model combining the SFE of the soild surface, the contact angle and the ST of the liquids. Spreading coefficients (SC) were calculated for each pair of resin and substrate.

2.4 Bonding test configurations

The complex geometry of superconducting magnets can lead to various bonding failure modes. For this reason, four test configurations were carried out: T-Peel with specimens of 15 mm wide and bonded over a length of 241 mm according to ASTM D1876 [11], roller peel with specimens of 13 mm wide bonded over a length of 259 mm according to ASTM D3167 [12], double lap shear with specimens of 23.4 mm wide and overlap length of 12.7 mm according to ASTM D3528 [13] and butt joint with bonded circular surface according to ASTM D897 [14]. The double lap shear joint was also performed for the resin CTD101K under cryogenic environment (77K). CTD101K was chosen because it is the epoxy system used to build the Nb_3Sn magnets of the High-Luminosity upgade of the LHC. Tinius Olsen H5kT universal testing machine was used for conducting the tests. The fibre glass S2 493 was used to maintain the thickness of the adhesive as 0.175 mm. Each result was obtained by the test repetition with three samples.

3 Results and discussions

3.1 Substrate wettability and epoxy surface tension

The degree of wettability of the substrates was initially evaluated by analyzing the contact angle using the MSA. Figure 1 compares the SFE of substrates – untreated, sandblasted (SB), and sandblasted followed by plasma treatment. The total SFE value increased after the surface treatment. The observed rise in the polar component of the SFE demonstrates the increased ability of the substrates to interact with polar adhesives, thereby enhancing the overall bonding.

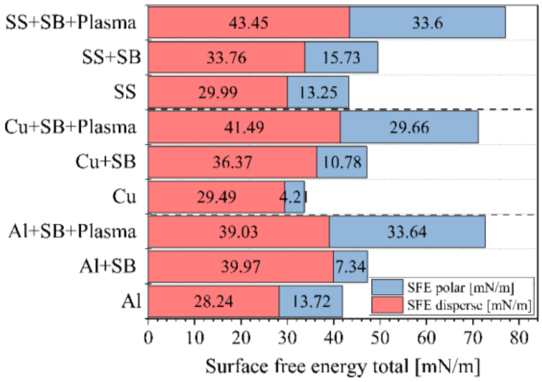

Figure 1. Surface free energy measurements after surface modifications for stainless steel 304L (SS), aluminium EN AW 6082 (Al) and copper ETP (Cu) substrates.

The ST values of liquids form the basis for conducting subsequent analysis of interfacial wetting. The ST of a liquid governs its ability to wet a functional surface. Therefore, the ST of the epoxies under consideration was determined by using Wilhelmy apparatus and reported in Table 2. The ST values for MY750 and Mix61 are lower, which means, that the cohesive forces within these epoxies are weaker and the liquid molecules have less attraction to each other at the surface.

Consequently, these resins tend to spread easily over solid surfaces and form a thinner and uniform film, thus leading to an enhanced wetting.

Table 2. Total surface tension of liquid epoxy impregnation systems.

	CTD101K	***Araldite F***	***MY750***	***Mix 61***
$\boldsymbol{Surface\ tension\ \sigma_L\ (mN/m)}$	41.22	40.61	35.04	34.02

The spreading coefficients and their corresponding isolines were determined to facilitate the prediction of wetting behavior for specific epoxy systems, substrate materials, and surface conditions. The spreading coefficient is defined as the difference between the substrate's surface free energy (SFE), the liquid phase's surface tension (ST), and the interfacial free energy at the liquid-solid interface. A positive spreading coefficient indicates a complete wetting condition. Therefore, wetting is favored by a high SFE of the substrate and low ST and interfacial free energy. For example, considering copper, the element constituing the outer surface of superconducting wires, complete wetting is anticipated for all epoxy systems on copper substrates treated with sandblasting and plasma, exhibiting an SFE of 71.65 mN/m. In contrast, an untreated copper surface with an SFE of 33.7 mN/m is unlikely to achieve complete wetting with epoxy systems such as CTD101K and Araldite F, which have ST values of 41.22 mN/m and 40.61 mN/m, respectively. This incomplete wetting can negatively impact overall adhesion performance.

Figure 2 represents the spreading coefficient isolines for copper, aluminum and stainless steel surfaces. These plots are useful to interpret how changing polar and dispersive components of the liquid ST will affect its wetting abilities. For epoxies, these plots can provide insights to guide potential resin system modifications, during the impregnation system development phase, by revealing the optimal balance between the polar and dispersive components of the ST. When the polar (σ^{s}) and dispersive component (σ^{D}) of a liquid are within the enclosed area of the generated envelope, complete wetting (i.e., contact angle = 0°) of the surface can be expected. The black dots indicate measured SFE values of epoxies as a function of polar and dispersive components.

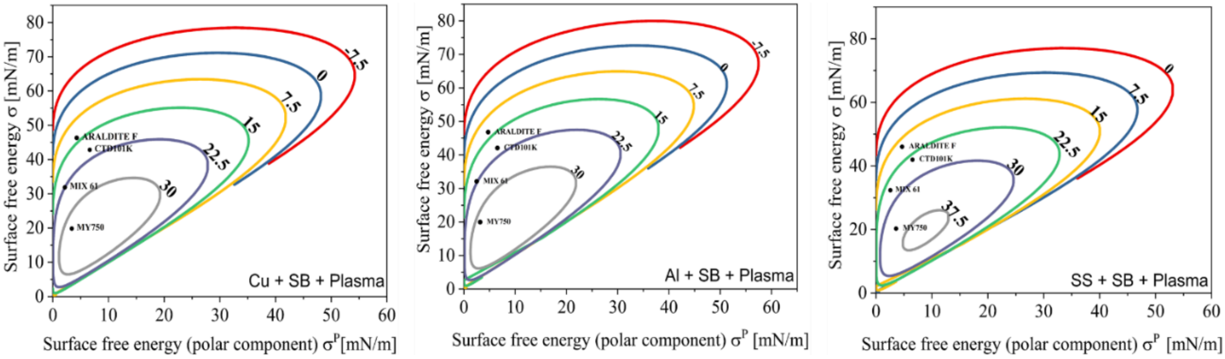

Figure 2. Spreading coefficient isolines [mN/m] for CTD101K, Araldite F, Mix 61 and MY750.

3.2 *Bonding strength of different resin/substrate combinations*

Experiments were conducted to investigate the bonding behavior of epoxies towards surface-treated substrates, involving four different test configurations. As shown in Figure 3, the bonding strengths of the epoxies MY750 and Mix61 were the highest among the four epoxy impregnation systems and two adhesive references across all test types. The high bonding strengths of epoxies MY750 and Mix61 can be attributed to their low surface tension, which reduces cohesive forces between surface molecules and enhances wetting. However, the superior performance of these systems cannot be explained solely by improved wetting. Paticulalry, the amine-cured systems, Mix61 and MY750, exhibit a lower Young's modulus and greater elongation at break (Table 1) compared to the anhydride-cured systems CTD101K and Araldite F [7], [9]. These mechanical properties may enhance the material's ability to accommodate mechanical stresses, thereby minimizing peak stress concentrations on the test specimens.

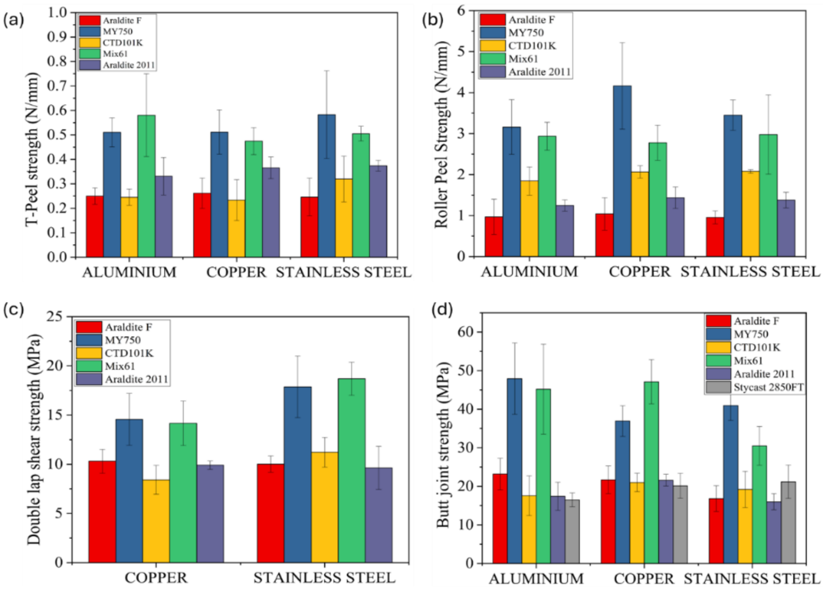

Figure 3. a) T-peel b) roller peel c) double lap shear joints and d) butt joint test results on SB+ plasma treated Aluminium EN AW 6082 (Al), Stainless steel 304L (SS), and Copper ETP (Cu).

Figure 4 shows the double lap shear strength of CTD101K on copper and stainless-steel surfaces. The shear strength increases under cryogenic conditions (77 K) compared to room temperature. However, for an impregnated Nb_3Sn coil—such as those used in the inner-triplet quadrupole MQXF for the LHC high-luminosity upgrade—the maximum calculated shear stress during magnet cooldown and operation is approximately 50 MPa [15]. Additionally, shear stresses exceeding 10 MPa are applied at room temperature during magnet assembly (i.e. loading). Consequently, it is reasonable to suppose that resin debonding occurs throughout the lifecycle of a high-field magnet, regardless of the epoxy resin system used, at least for the systems examined in this study. Furthermore, there is ongoing debate about whether an impregnation system should remain bonded to the superconducting cable or, conversely, be entirely detached to enahnce cable and magnet performance [16].

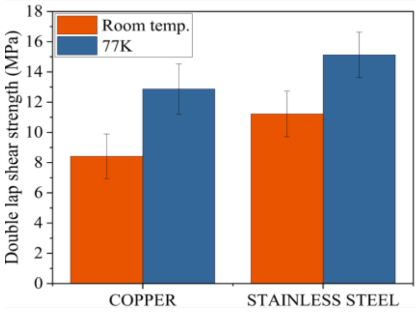

Figure 4. Double lap shear strengths for CTD101K with copper and stainless steel at room temperature and at 77K.

4 Conclusions

This research presents a method for evaluating the adhesion performance of impregnation resins to superconducting coil materials. The surface free energy (SFE) of substrates and surface tension (ST) of epoxies were measured, and adhesion analysis was conducted. The spreading coefficient, indicating adhesive-adherend compatibility, was also assessed. Adhesion behavior of various epoxy resins was examined through T-peel, roller peel, double lap shear, and butt joint tests with different adherends. Direct adhesion tests showed that MY750 and Mix61 epoxies, cured with amine hardeners, exhibited higher bonding strength. The experimental results aligned with theoretical predictions, offering valuable insights and test methods for selecting suitable impregnation systems for superconducting magnets.

Acknowledgements

The authors would like to acknowledge Prof. Theo van Voort and Dr. Pascal Studer, ETH Zurich for providing access to the tensiometer apparatus, which contributed to the completion of this study.

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