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STUDY OF MATERIALS AND ADHESIVES FOR SUPERCONDUCTING CABLE FEEDTHROUGHS

A. Perin, R. Macias Jareño, and L.Metral

Abstract

Powering superconducting magnets requires the use of cryogenic feedthroughs for the superconducting cables capable of withstanding severe thermal, mechanical and electrical operating conditions. Such feedthrough shall provide the continuity of the superconducting circuit while ensuring a hydraulic separation at cryogenic temperature. A study about the adhesive and polymers required for the production of thermal shock resistant feedthroughs is presented. The strength of the busbar to adhesive joints was first investigated by compression/shear tests as well as pin and collar tests performed with four epoxy adhesives. After the selection of the most appropriate adhesive, pin and collar tests were performed with four different polymers. Based on the results, a superconducting cable feedthrough for 6 busbars of 6 kA and 12 busbars of 120 A was constructed and successfully tested.

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STUDY OF MATERIALS AND ADHESIVES FOR SUPERCONDUCTING CABLE FEEDTHROUGHS

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ABSTRACT

Powering superconducting magnets requires the use of cryogenic feedthroughs for the superconducting cables capable of withstanding severe thermal, mechanical and electrical operating conditions. Such feedthrough shall provide the continuity of the superconducting circuit while ensuring a hydraulic separation at cryogenic temperature. A study about the adhesive and polymers required for the production of thermal shock resistant feedthroughs is presented. The strength of the busbar to adhesive joints was first investigated by compression/shear tests as well as pin and collar tests performed with four epoxy adhesives. After the selection of the most appropriate adhesive, pin and collar tests were performed with four different polymers. Based on the results, a superconducting cable feedthrough for 6 busbars of 6 kA and 12 busbars of 120 A was constructed and successfully tested.

INTRODUCTION

The Large Hadron Collider, currently under construction at CERN will use high field superconducting magnets operating at liquid helium temperature. The electric current is transported to the magnets by copper stabilized superconducting cable busbars (SB). In several places of the machine it is necessary to ensure a hydraulic separation while maintaining the continuity of the superconducting busbars. This function will be performed by high current Superconducting Cables Feedthroughs (SCF) that must withstand the specific conditions found in LHC: low temperature operation, radiation environment and large temperature gradients. We investigated a possible configuration for such SCF by first determining the compression/shear strength of copper joints made with four different epoxy adhesives and then by simulating the operating condition of the SCF with pin and collar specimens reproducing the configuration of the SCF.

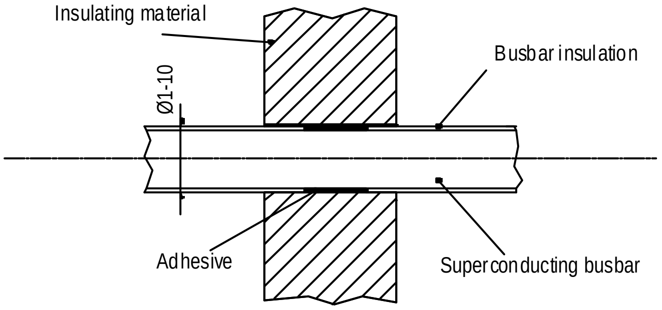

FIGURE 1. Configuration of a superconducting cable feedthrough

CONFIGURATION AND MATERIALS

A schematic view of the proposed SCF configuration is presented in figure 1. The main difficulty in this construction is to cope with the differential thermal expansion between the SB and the insulating material. In particular, during a quench of the SB, the copper temperature can reach 300 K within a few seconds while the rest of the SCF is still at very low temperature, thus creating a huge temperature gradient between two parts bonded together.

The thermally induced stress at the adhesive interfaces can be decomposed into an axial shear stress and radial compressive/tensile stress. For polymers, the insulator has a higher thermal contraction than the SB, the radial stress is thus compressive under most operating conditions. To investigate the properties of adhesives for such a SCF, the usual lap shear strength tests are therefore not appropriate and it is necessary to qualify the adhesive by using combined compressive/shear strength measurements.

Materials for the insulating plate

For the reliable operation of a SCF the thermally induced stresses shall be kept as low as possible. When the temperature of the assembly is homogeneous, the lowest stresses are achieved by matching the Coefficient of Thermal Expansion (CTE) of all materials, but when there is a big temperature difference between the SB and the insulating plate, as it happens during a resistive transition of the SB, the determinant parameter is given by the product of the thermal strains by the elastic moduli. Table 1 shows this factor at 77 K (strain relative to 293 K) for G11 which has a CTE similar to the one of copper but has a very big elastic modulus and for unfilled Polyimide that has a big CTE but a small elastic modulus. Table 1 shows that, in the presence of extreme thermal gradients, unfilled polymers are a potential solution to limit the thermally induced stresses. In order to investigate the potential of this solution we selected four polymers that are known to have good properties under LHC conditions [1], which are shown in table 2.

TABLE 1 . Thermal strain (relative to 293 K) α multiplied by the elastic constant E for G11 and unfilled Polyimide at 77 K [1]

Material	E(GPa)	a	a x E (MPa)
G11 parallel to fibers	45	0.25%	112
Unfilled polymer (Polyimide)	5.8	0.90 %	52

TABLE 2. List of tested polymers

Material	Commercial name	manufacturer
PI, Polyimide	Vespel SP1®	Dupont
PAI, Poly(amide-imide)	Torlon 4203L®	BP Amoco Polymers
PEI, Polyethetimide	ULTEM 1000®	GE Plastics
PEEK, Polyetheretherketone	PEEK ®	Victrex

Adhesives for SCF

Under the conditions found in LHC, in particular the cryogenic temperatures and radiation, epoxy adhesives have shown to have excellent performance. The improved resistance to thermal shocks and the enhanced thermal conductivity of filled adhesives with respect to unfilled ones led to select 4 filled epoxy adhesives for this study, which are shown in table 3. For ease of use, only two components adhesives requiring room temperature curing were included in the study.

EXPERIMENTAL PROCEDURES

Tests

Preliminary tests performed with various materials and adhesives showed that, in the configuration of figure 1, damage occurred preferentially by adhesion failure at the adhesive/copper interface. It was therefore decided to first investigate the compressive/shear strength of copper to copper joints made with the four epoxy adhesives at room temperature and at 77 K. The adhesives were then submitted to thermal shocks, in a "pin and collar" configuration simulating a SCF with polyimide polymer. The compatibility of the four insulating polymers with the best performing adhesive was then investigated and a prototype SCF with real SB was built and tested.

Compression/shear tests

The compression/shear specimens are shown on the left part of figure 2. In order to simulate a surface preparation compatible with superconducting cables, no chemical etching was applied on the surface of the copper blocks. The surface was only cleaned with 16 µm sandpaper until all visual traces of oxidation disappeared and the blocks were then cleaned with an ultrasound cleaner using isopropylic alcohol. Finally the blocks were dried using clean compressed air and then mounted on a special tool that ensured a very good relative alignment and provided a 0.5 mm gap for the adhesive layer. The traces left on the

TABLE 3. List of tested epoxy adhesives

Adhesive	Supplier
Araldite CW 1304 GB/ HY 1300 GB	CIBA
Eccobond 286 A/B	Emerson & Cuming
Stycast 2850FT / catalyst 9	Emerson & Cuming
Epo-Tek T7110	Epoxy Technology, Inc

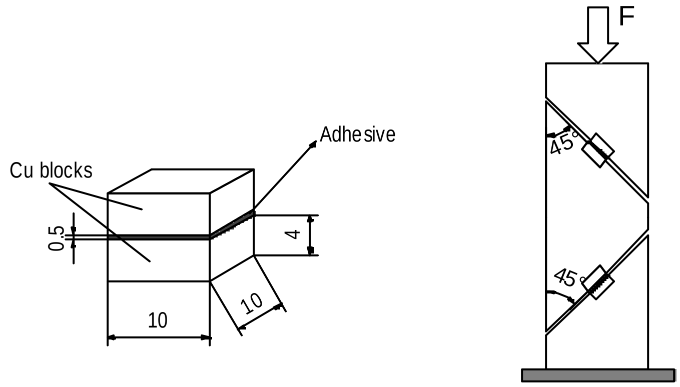

FIGURE 2 : Test specimen (left) and test configuration (right) for the compression/shear tests.

copper surface by the sandpaper were aligned in the same direction. Adhesive application was performed less than 15 minutes after the sandpaper treatment.

The adhesives were prepared following the instructions provided by the manufacturers. All were mixed at room temperature and degassed for 4 minutes in a vacuum chamber at a pressure of 10 mbar. The adhesives were applied in air at atmospheric pressure and room temperature. The test specimens were then cured in a controlled temperature environment at a temperature of 26 °C ± 2 °C for the duration recommended by the adhesive supplier. After complete curing of the adhesive the samples were removed from the alignment and spacing tool. The small amounts of adhesives on the lateral sides of the blocks were removed with sandpaper.

The compression/shear tests followed a technique described by Drexler at al. [2] as shown in the right side of figure 2. A specimen orientation angle of 45° was chosen, thus resulting in the same magnitude for the shear and compressive stresses. The traces left by the sandpaper treatment were aligned parallel to the shear stress. The compressive force F was applied with a screw driven test machine with a crosshead velocity of 0.5 mm/min.

Each test was performed with 2 virgin specimens. The force was increased until a failure occurred in one of the specimens. The shear strength was defined by dividing the shear component of the applied force by the area of the specimen.

The tests were performed at room temperature and at 77 K in a bath of liquid nitrogen. In order to avoid misalignments during the cooldown process, a preloading force of 1500 N – 2000 N was applied before cooldown and continuously kept in this range until the test system was submersed by LN$_2$.

Pin and collar tests

The configuration chosen for this tests, simulating the situation found in a real SCF, is shown in figure 3. The polymer was cleaned with ultrasounds in isopropylic alcohol and dried with dry compressed air. The surface of the copper pin was prepared with the procedure as used for compression/shear specimens. Sandpaper cleaning was performed along the longitudinal axis of the pin. The adhesives were prepared following the same procedure described for the compression/shear tests. The mixed and degassed adhesives were then injected with a syringe through the injection hole until complete filling of the gap between the pin and the collar. The specimens were then cured with the procedure described for the shear/compression tests. In order to eliminate the singularities of the injection hole and of the exhaust ring around the pin, the specimens were finally machined to reach the configuration shown on the right side of figure 4.

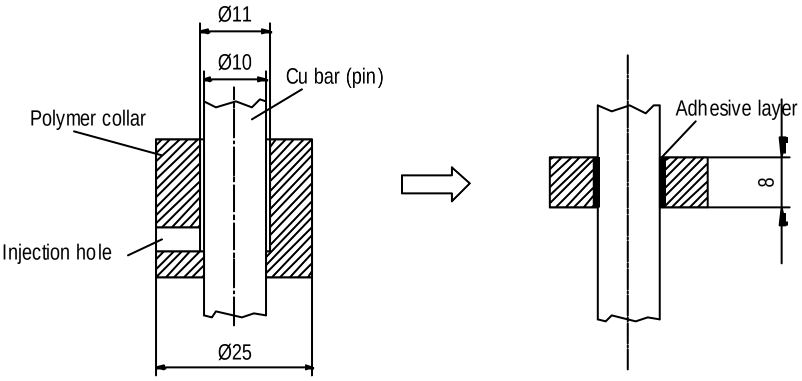

FIGURE 3. Specimens for pin and collar tests, adhesive injection configuration (left) and final shape (right)

The tests performed on the pin and collar specimens were essentially aimed at simulating the thermal conditions found during the operation of the LHC. As most mechanical properties do not evolve significantly below 77 K, thermal cycling was limited to LN_2 temperature. The tests consisted of thermal cycles followed by a room temperature leakage measurement performed with a helium leak detector. When the measured value was below the detection level of 10^{-9} mbar l / s, the result is reported to be "no leakage". Temperature sensors were placed on the surface of the pin and of the collar and the temperatures were continuously recorded by a computer system. The following sequences of cycles were performed on all samples:

1. "cable quench simulation"
   The specimens were cooled at 2 K/min to 77 K. They were then extracted and the copper pin was immersed in flowing water at 50 ° C while the polymer collar was held in an insulating envelope. During this process, the temperature of the copper bar increased at a rate greater than 25 K/s resulting in a temperature difference greater than 100 K between the copper and the polymer collar. This cycle was repeated 5 times and the leak tightness measured after each cycle.

2. "fast cooldown"
   The specimens were initially at room temperature. The copper pin was then immersed into LN_2 while the collar was held in an insulating envelope. This process resulted in temperature drop speed of 2 K/s on the copper bar. The specimens were then totally immersed into LN_2 and subsequently warmed in hot water. This cycle was repeated 5 times and the leak tightness measured after each cycle.

RESULTS AND DISCUSSION

Compression/shear tests with four adhesives

Compression/shear tests were performed at room temperature and at 77 K on specimens prepared with the four epoxy adhesives. The thickness of the adhesive layer was 0.43 mm ± 0.05 mm. In all tests, the rupture occurred by adhesion failure at the adhesive/copper interface.

The shear strength of all tested specimens is shown in FIGURE 4, average values and relative standard deviation are given in TABLE 4. At room temperature, three adhesives (Araldite, Eccobond and Epotek) have a very similar shear strength of about 35 MPa. It is to be noted that this value is significantly higher than the usually measured lap shear

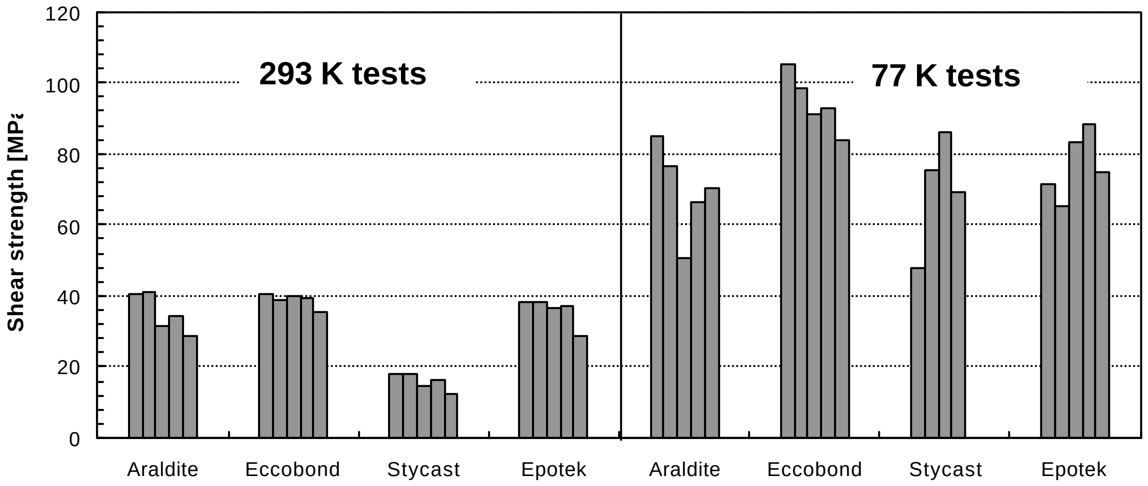

FIGURE 4. Results of the compression/shear tests at 293 K and 77K

strength which is about 15 MPa for these adhesives. Only the Stycast adhesive appears to have a significantly lower strength of 15.3 MPa. Concerning the spread of the measured values, the Eccobond specimens show a significantly smaller relative deviation of 5%, while the other adhesives have all relative deviations between 12 % and 16 %.

At 77 K the average shear strength increases considerably with all values ranging between 70 MPa and 95 MPa. While all adhesives show improved strength at 77 K, the Eccobond adhesive, with an average shear strength of 95 MPa produces joints with significantly higher strength than the other adhesives. The relative standard deviation of the measured shear strength at 77 K is the same as the ones obtained at room temperature. The value of 8 % obtained with the Eccobond adhesive is also significantly lower than the value measured with the other adhesives.

Pin and collar tests with four adhesives

Pin and collar specimens were prepared with the four adhesives using Vespel as the material for the collar. Vespel was chosen because, in preliminary tests realized with various epoxy adhesives, failure always occurred at the copper/adhesive interface and never at the Vespel/adhesive interface. Two specimens were produced and tested with the above described procedure for each adhesive.

All specimens were measured before thermal cycling and no leakage was detected. The samples were then submitted to the "quench simulation" sequence of cycles where specimens produced with Araldite, Eccobond and Epotek showed no leakage. For specimens produced with Stycast, a leakage of $2{\cdot}10^{-4}$ mbar/l s appeared after the first cycle

TABLE 4. Summary of compression/shear tests results at room temperature and 77 K

Adhesive	293 K tests		77 K tests	
	Average shear strength [MPa]	Rel. St. Dev.	Average shear strength [MPa]	Rel. Std. Dev.
Araldite	35.1	15 %	69.9	18 %
Eccobond	38.9	5 %	95.4	8 %
Stycast	15.3	16 %	69.7	23 %
Epotek	35.2	12 %	76.8	12 %

TABLE 5. Results of the pin and collar tests with four adhesives

Adhesive	**“quench simulation”** Leakage after 5 cycles [mbar l / s]	**“rapid cooldown”** Leakage after 5 cycles [mbar l / s]
Araldite	No leakage	No leakage
Eccobond	No leakage	No leakage
Stycast	10-3 (first cycle)	2 10-4
Epotek	No leak	No leakage

and did not evolve after subsequent cycles. The “fast cooldown” sequence was then performed on the specimens and no evolution was observed during this sequence.

The origin of the leakage on the Stycast specimens was found to be a complete adhesion failure at the copper/adhesive interface. It appears that, although Stycast has adhesive properties at low temperature that are similar to the other adhesives, its lower strength at intermediate and room temperature leads to a failure of the joint when it is submitted to severe thermal cycles.

Pin and collar tests with four polymers

Pin and collar specimens were then prepared for the four materials using Eccobond adhesive. Eccobond was chosen because the tests performed on the adhesives showed that it has the best properties among the four tested adhesives. Two specimens were produced and tested with the above described procedure for each material. The results are summarized in table 6.

No leakage was detected on the specimens before thermal cycling. A “quench simulation” sequence of cycles was then performed: specimens produced with Vespel, Torlon and Ultem showed no leakage while a leakage of $1{\cdot}10^{-3}$ mbar/l s was measured on the PEEK sample after the first cycle. No further degradation was observed during subsequent cycles and during the “fast cooldown” sequence. The cause of the leakage on the PEEK collar specimen was identified as an adhesion failure at the PEEK/adhesive interface.

Prototype feedthrough

A prototype SCF with a diameter of 40 mm was fabricated with 5 busbars for 6 kA (11 strands, total diameter 6 mm) and 11 busbars for 120 A (single strand, diameter 1.6 mm) using Vespel and Eccobond with the procedure described for the production of the pin and collar specimens. The resulting SCF is shown in figure 6.

The thermal cycles sequences described for the pin and collar specimens were

TABLE 6. Results of the pin and collar tests with four polymers

Collar material	**“quench simulation”** Leakage after 5 cycles [mbar l / s]	**“rapid cooldown”** Leakage after 5 cycles [mbar l / s]
Vespel	No leakage	No leakage
Torlon	No leakage	No leakage
Ultem	No leak	No leakage
PEEK	$2\ 10^{-4}$ (first cycle)	$2\ 10^{-4}$

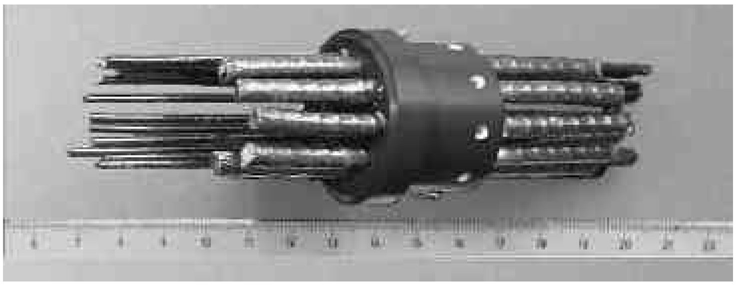

FIGURE 6. Prototype SCF with 5x6kA busbars and 12 x 120 A busbars

performed with the prototype SCF and no leakage was detected. The SCF was then thermally cycled 50 times with successive immersion into LN_2 and hot water and again no leakage was detected.

CONCLUSIONS

The compression/shear tests performed at room temperature showed that among the four tested epoxy adhesives, Araldite, Eccobond and Epotek produce joints to copper with similar properties while assemblies made with Stycast are much weaker. The results show also that the spread of strength between the specimens obtained with Eccobond is smaller than the one obtained with the other adhesives. At 77 K, the shear strength of all adhesives is higher than at room temperature. At this temperature Eccobond shows the highest strength and also the smallest spread. The behavior of the adhesives was confirmed by pin and collar tests where the specimens produced with all epoxy adhesives except the Stycast passed all thermal shock tests with no detectable leakage. Among the tested adhesives, Eccobond appears to have the best properties for the construction of cryogenic feedthrough based on the proposed technology.

As concerns the materials used for the insulating plate, Vespel, Torlon and Ultem gave excellent results, with no leakage detected after all thermal cycles while specimens with PEEK collars failed after the first thermal cycle.

We demonstrated that thermal shock resistant cryogenic feedthroughs can be fabricated with unfilled polymers and epoxy adhesives. Following the results obtained on adhesives and materials, a cryogenic feedthrough with 5 x 6 kA busbars and 12 x 120 A busbars was produced and successfully tested.

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