Developing a Vacuum Pressure Impregnation Procedure for CORC Wires

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***Abstract*—Superconducting magnets designed for high energy physics and nuclear fusion require mechanical and electrical integrity to perform at high currents and magnetic fields. Vacuum Pressure Impregnation (VPI), a process of curing epoxy in and around the superconducting wires, is often used to support and consolidate a magnet. However, the heat and mechanical stresses associated with the process can degrade the wires, significantly lowering their critical current. This study explores different methods of potting and curing CORC wire with the aim of reducing wire performance degradation to less than 3% measured at 77 K, self-field. The wires were 2.9 mm in diameter consisting of a total of six REBCO tapes (three layers of two tapes). Two bending radii (20 mm and 50 mm) were tested to mimic the winding shape of a magnet. Mix 61 epoxy was used in preliminary tests for potting. For each test, two wires were used, and their critical currents were measured simultaneously in liquid nitrogen at 77 K – in their straight form, then bent, followed by the heat treatment used for Mix 61 but without epoxy and finishing with the full epoxy impregnation test. Later tests were performed using CTD-528 to explore a room temperature cure, limiting possibility of degradation from thermal expansion and prolonged exposure of the REBCO tapes to elevated temperature. Here we report the experimental results with multiple CORC wires and different curing schedules. The results obtained are the first steps toward identifying the VPI process with minimum degradation in critical current to be implemented in high-field magnets using CORC wires.**

***Index Terms*—High energy physics, high temperature superconductors, nuclear fusion, rare earth barium copper oxide, vacuum pressure impregnation.**

I. Introduction

HIGH Temperature Superconductors (HTS) have become more prevalent in research relating to high energy physics and nuclear fusion. HTS accelerator magnets can enable a dipole field beyond 20 T for future circular particle colliders. In addition, HTS compact fusion magnets can provide magnetic fields of 20 T and higher that could create breakthroughs for clean energy helping to solve the world energy and environmental crisis. REBCO (Rare Earth Barium Copper Oxide) is a leading material for HTS applications because of its capability for strong magnetic fields over a broad temperature range. However, with strong magnetic fields come stronger Lorentz forces, leading to potential wire degradation from mechanical stresses. Therefore, supporting the wire is necessary to provide proper electro-mechanical stability. To achieve this, a process called Vacuum Pressure Impregnation (VPI) is often used where epoxy resin is cured around the wire, providing extra support. As earlier experiments show, when the epoxy is cured in direct contact with REBCO tapes, the higher thermal contraction of the epoxy could cause the REBCO tapes to delaminate [15], [19]. Several solutions have been successfully identified to address this issue by using a different impregnation medium such as paraffin wax which has a lower bonding and tensile strength and so it does not delaminate REBCO tapes [5]. Similarly, one can identify or engineer a medium whose coefficient of thermal expansion (CTE) matches that of REBCO [5]–[9]. Another effective approach is to isolate the REBCO tapes from the impregnation medium [10], [11]. The Conductor on Round Core (CORC) wire, made from winding REBCO tapes around a copper core, utilizes a heat shrink tube surrounding the wire to prevent epoxy from seeping into the wire [12], [13]. Despite the use of the isolation tubing, one needs to experimentally verify that a specific VPI procedure does not degrade the CORC wire as the heating cycles used to cure the epoxy could cause an increase in joint resistivity or oxygen deficiency within the tapes [14].

Other issues arise when bending the wires during magnet fabrication. It has been shown that CORC wire in particular starts to degrade significantly at a bending radius of about 20-25 mm [12], [13]. One question to address is whether the existing degradation from bending becomes worse causing further degradation after impregnation.

TABLE I
Experimental Procedure Parameters

Sample Label	Bending Radius (mm)	Epoxy	VPI Procedure
1a	50		
1b	20		
2a	50	Mix 61	60°C for 16 hours 100°C for 24 hours
2b	20		
3a	50		
3b	20	CTD-528	7 days at room temperature

This table shows the differences in test parameters for each CORC wire. The major differences between tests were the bending radius (either 20 or 50 mm) and the VPI procedure – both the epoxy and heating schedule were varied.

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TABLE II
I_C AND N-VALUES OF THE SAMPLES MEASURED AT 77 K, SELF-FIELD, AFTER EACH TEST

Sample	I_c Baseline (A)	I_c After Bending (A)	I_c After Heating (A)	I_c After VPI (A)	I_c After Thermal Cycle (A)	n-value Baseline	n-value After Bending	n-value After Heating	n-value After VPI	n-value After Thermal Cycle
1a	337	295	-	279	-	7.8	6.5	-	5.74	-
1b	279	259	-	224	-	6.1	5.7	-	4.7	-
2a	221	212	160	140	-	4.54	4.6	4.5	3.8	-
2b	265	231	220	224	-	7.0	5.2	5.0	5.44	-
3a	247	246	-	246	246	5.6	5.7	-	5.8	6.2
3b	251	243	-	244	244	5.8	5.3	-	6.1	6.34

This table shows the results of critical current and n-value measurement baseline and after going through the VPI procedures.

In this experiment, the bending radius and VPI procedures were explored together to mimic a typical magnet setup. A procedure was identified that shows no significant degradation for the two bending radii considered in this study.

The overlap length of the heat shrink tubing around the CORC conductor is also an issue to be considered (not discussed in this work) as currently the tubing is not long enough to cover the entire wire in one piece, so smaller strips must be overlapped for full coverage. Future work will include studies to address this issue and explore possible degradation caused by epoxy entering the locations where the two pieces of tubing overlap.

II. Experimental Setup and Methods

A. CORC Wires

The CORC wires used in this experiment all had six tapes and three layers, two tapes per layer. The tapes were 2 mm wide with a 30 μm substrate, commercially available from SuperPower Inc. The 30 μm thick heat shrink tubing covered the full wire diameter and length. The wire diameter was 2.9 mm including the heat shrink tube. The expected critical current of the wire at 77 K and self-field condition was around 260 A.

Three groups of wires are used in the experiments reported here. Each group consists of two wires (Table I). All six wires are nominally identical except that the wires for Group 3 were re-lubricated before the reported tests.

B. Experimental Setup

An aluminum (6061-T6) fixture, chosen for its easy manufacturability and common use for REBCO wires, was developed to bend the wires to two different radii – 20 and 50 mm. These two sizes were chosen based on the typical magnet bend. No degradation was expected for the 50 mm bend based on previous testing [7]. The smaller (20 mm) radius was expected to degrade the wire to identify any limitations associated with a sharper bending radius, especially in the VPI procedure. The pig tail connections (Fig. 1) were connected to power supply cables for current injection. The hole in the middle of the fixture was used to screw in mandrels to facilitate bending reproducibility. The groove diameter was 4.5 mm for the measurements of sample Groups 1 and 2. The groove diameter was increased to 10 mm for Group 3.

Inside the electrical termination, the tapes of the CORC wires were tapered depending on the layer number to help the current migrate uniformly into all tapes. To complete the fabrication of the termination, a copper tube 6.35 mm in diameter was slid over the wire. Next, the termination was clamped in a vertical position

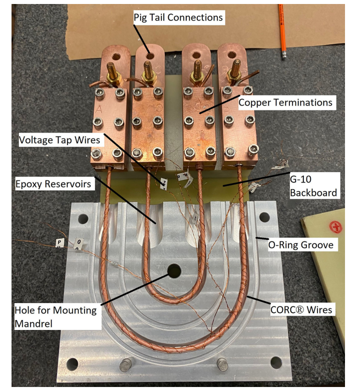

Fig. 1. CORC wires 1a and 1b soldered to the terminations and bent to respective radii 20 and 50 mm. Another mirroring aluminum plate was placed on top of the plate shown to seal the reservoirs and compact O-rings.

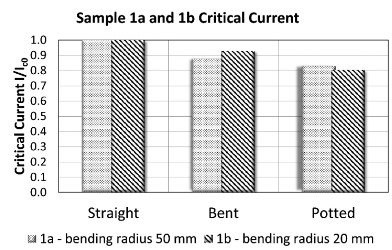

Fig. 2. Normalized critical current of wires 1a and 1b in the straight configuration, bent to the two radii (20 and 50 mm) and potted and cured with Mix 61 epoxy. The measurements error ranged from 4% to 9%.

and corrosive active Alpha 260HF flux was applied and wiped before 99.99% pure indium solder was melted into place to fill the empty volume within the tube. Voltage taps were added at distances 5, 40 and 75 mm up from the bottom of the copper terminations. The soldering process fixed all the voltage tap wires in place and electrically connected the ends of the cut tapes to the copper tube for proper current injection. To follow is a more detailed description of all the samples and the tests performed.

C. Measurement Procedure for Samples 1a and 1b

The first two wires used were tested in three different conditions, all at 77 K, self-field. First, both wires were tested in a straight configuration to get the baseline I_c measurement. Second, after warming up to room temperature, the wires were bent into the aluminum fixture within the grooves of 20 and 50 mm radii (Fig. 1). Winding mandrels of both sizes were screwed into the middle hole of the aluminum plate and the wires were bent around them. The aluminum fixture was then placed into the cryostat for the second round of I_c measurements. This step was performed to study the I_c degradation due to bending.

The last step was the epoxy impregnation procedure. NHMFL-Mix 61 epoxy was used due to its viscosity, fracture toughness and thermal shock resistance that make it a good choice for superconductor impregnation [16]–[18]. The maximum curing temperature of 100 °C was not considered an issue. After being mixed and degassed at room temperature, the epoxy was injected into the epoxy holder grooves in the aluminum fixture and was degassed again in a vacuum chamber. It took about 30 minutes for the epoxy to fully cover the wire and the air was slowly let back in the chamber after no visible bubbles appeared in the epoxy holder grooves. The whole fixture was then placed in an oven to start the curing schedule as shown in Table I.

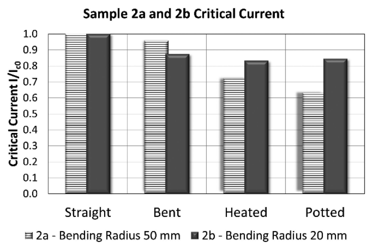

Fig. 3. Normalized critical current of wires 2a and 2b in the straight configuration, bent to the two radii, heated following the curing schedule and potted and cured with Mix 61 epoxy. The measurements error ranged from 3% to 8%.

D. Measurement Procedure for Samples 2a and 2b

Samples 2a and 2b followed the same procedure as Samples 1a and 1b except for one additional test of the curing heating step. In this additional test, the wires went through the curing procedure (without epoxy) and I_c measurements were recorded. After taking the I_c measurements following the curing procedure (heated configuration), the wires were impregnated with NHMFL-Mix 61 epoxy and the I_c was measured again.

E. Measurement Procedure for Samples 3a and 3b

Samples 3a and 3b followed the same measurement steps as Samples 1a and 1b the following some changes. A room-temperature cured epoxy, CTD-528 [19], was used instead to impregnate both samples. The room-temperature cure would allow identification of possible issues caused by the heat treatment used during the curing process of NHMFL-Mix 61.

To confirm the results achieved from the final round of testing, another thermal cycle was performed where the wires were warmed up to room temperature and then were cooled down again to 77 K for the I_c measurement.

The I_c and n value for all the measurements were determined according to (1).

$$\boldsymbol{V} = \boldsymbol{V_c}\left(\frac{\boldsymbol{I}}{\boldsymbol{I_c}}\right)^{\boldsymbol{n}} + \boldsymbol{I}*\boldsymbol{R} \tag{1}$$

where V_c is the voltage criterion of 20 μV and R is the slope of the measured resistive foot of the *V-I* curve. The summary of the measured critical current and *n*-values are shown in Table II and will be discussed below.

III. Results and Discussion

A. Samples 1a and 1b

The data plotted in the following sections are all from the voltage taps that were the closest inwards and therefore the most presentative for the transport performance of the wires.

The results obtained from the first round of tests were different than expected (Fig. 2). The I_c degradation was 12% in Sample 1a and 7% for Sample 1b after bending and before VPI, although Sample 1a had a larger bending radius of 50 mm. The fact that both wires show I_c degradation is possibly due to the tight clearance between the samples and the groove in the aluminum fixture. The thermal contraction during cooldown causes the aluminum fixture to shrink, placing a mechanical stress on both wires. We may also not rule out the potentially different handling of the two samples during bending that can lead to varied I_c degradation.

After epoxy impregnation and curing, the I_c of Sample 1a degraded ∼17% from the straight configuration and that

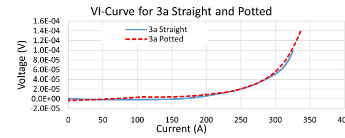

Fig. 4. The plot shows the V-I curves for the straight and potted configurations for Sample 3a.

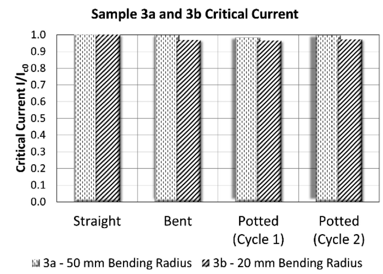

Fig. 5. Normalized critical current of wires 3a and 3b in the straight configuration, bent to the two radii and potted and cured with CTD-528. The measurements error ranged from 3% to 12%.

of 1b degraded ∼21%. This further degradation from the impregnation procedure suggested that either the particular epoxy or the curing schedule caused the degradation. The degradation due to bending also can become worse after impregnation with NHMFL Mix-61.

B. Samples 2a and 2b

Fig. 3 shows the results obtained for Samples 2a and 2b. The measurements indicate significant degradation from all the test conditions considered after the straight sample test. First, the bent test degraded both samples again which added to suspicion that the thermal contraction of the aluminum plates was the main cause of this initial drop in electrical performance. Contraction calculations were performed using (2).

$$\boldsymbol{dl} = \boldsymbol{L}_0\, \boldsymbol{\alpha}\, (\Delta \boldsymbol{T}) \tag{2}$$

Where *dl* is the change in length, L_0 is the initial length, α is the linear expansion coefficient (23 μm m^{-1} $^\circ$C^{-1} for aluminum) and ΔT is the change in temperature.

The predicted change of length based on the height of the fixture of 115 mm was about 0.5 mm which may not be enough to cause interference; however, a new fixture design was used in the third set of experiments (Sample 3a and 3b) to eliminate this possibility entirely. Interestingly, the main degradation in Sample 2a occurred between bending and heating (total 28% degradation from straight I_c measurement) suggesting that the heating procedure for curing had more of an impact than the epoxy impregnation process which further degraded the sample by about 9%. An overall degradation of ∼37% and ∼15% was recorded for Samples 2a and 2b respectively from the straight configuration to the fully potted one.

Recently, Lu *et al.* reported the oxygen out-diffusion in REBCO tapes and the resulting I_c degradation after being subject to heating [10]. Using the model developed in [14], to reach the 0.1 μm out-diffusion length that can result in the I_c degradation requires a significantly longer cure time than the 24 h of heating at 100 $^\circ$C as used in the experiments here. On the other hand, the tendency of the copper plating surrounding the REBCO tapes to act as an oxygen sink may significantly speed up the oxygen out diffusion from the tapes. The heating may also cause an increase in resistance within the terminations of the wires leading to inhomogeneous current injection, ultimately affecting the I_c fit. In future tests, the voltage taps may be placed further from the terminations to clarify where degradation is occurring.

For the third round, changes were made to address all possible sources of degradation. The first change made was the size of the aluminum fixture groove to hold the wires. Because of the suspected thermal contraction stress, the groove diameter was increased from 4.5 mm to 10 mm to eliminate any interference. The other change made was the epoxy used for impregnation. CTD-528, a room-temperature cured epoxy was chosen for the impregnation of 3a and 3b which has a 7-day room temperature cure. Another proposed issue was the lubricant on the wire can evaporate if it is sitting over time and does not work as well. For the last set, the samples were relubricated with a fresh Teflon based lubricant.

C. Samples 3a and 3b

The results of the last set of samples (3a and 3b) are shown in Fig. 4 and Fig. 5. Sample 3b (20 mm bending radius) degraded by 3.5% from the tighter bend while 3a (50 mm bending radius) degraded by <1% which aligned with expectation. No significant degradation was observed from bending especially in Sample 3a, which suggested that either enlarging the aluminum fixture groove diameter and/or the relubrication was successful in removing the additional stress. The epoxy (CTD-528) and the room-temperature cure did not cause additional degradation. I_c and n-values remained the same after the second thermal cycle.

IV. Conclusion

The results presented in this work suggested that Mix 61 with a maximum cure to 100 °C caused significant degradation on the CORC wires. The separate heat tests from Samples 2a/b led to the hypothesis that the heat treatment used in curing was the main reason for this degradation which is likely due to oxygen out-diffusion within the REBCO tapes or an increase in resistance between the terminations; however, many parameters were altered in the 3rd round of testing and therefore it cannot be confirmed with full certainty. CTD-528 epoxy cured at room temperature caused negligible I_c degradation (<1%). Overall, the CTD-528 epoxy using a 7-day cure did not degrade the wire and seems to be a good candidate for CORC wires with the heat-shrink tubing.

To confirm and improve this preliminary round of testing, more configurations of testing are needed. Future measurements will consider a bending radius tighter than 20 mm to test the limits on how tight a coil using CORC wires may be wound and its impact on the following epoxy impregnation. Other tests might explore a new epoxy or low heat curing schedule to speed up the 7-day process it takes for CTD-528 to achieve the full cure to reduce the overall time it takes to fabricate a magnet. Now that an impregnation procedure is established without degrading the CORC wires, we can perform tests with varied overlap length of heat shrink tubing on CORC wires to identify possible issues with epoxy seeping through and interacting with the tapes directly.

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