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# Development of persistent joints for superconducting Bi-2212 coils

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## Abstract

$\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+x}$  (Bi-2212) multifilamentary wire is the only high-temperature superconductor manufactured in the form of an isotropic round wire, and so offers a number of advantages for the designers of high field magnets. However, for high-field ( $>25$  T), high-stability magnet applications, ultra-low resistance superconducting joints ( $R < 10^{-12} \Omega$ ) will be needed to take advantage of the excellent properties of the Bi-2212 wire. This study focuses on the fabrication of compact melt processed joints in small coils of Bi-2212/Ag multifilamentary round wires and the testing of their superconducting performance by inductive resistance measurements. Microstructural analysis is carried out to correlate the microstructure to the superconducting performance of the joints. Our optimized technique led to a reliable process for the preparation of small coils with melt processed joints that occupy very small volumes but can still carry the highest persistent currents reported so far for Bi-2212.

Keywords: applied superconductivity, magnet design, microstructure

## 1. Introduction

The highly homogeneous static magnetic fields required for magnetic resonance spectroscopy (NMR) and imaging place stringent requirements on the temporal stability of the magnet. While power supplies continue to improve, persistent magnets are still the design of choice for magnetic resonance applications, particularly at high field. Persistent magnets require persistent grade joints (PGJs) that carry substantial currents in

fields of several tesla with a resistance of less than  $10^{-12} \Omega$  [1]. For low-temperature superconducting (LTS) materials, this joint performance is achieved as an industry standard, but for high-temperature superconducting (HTS) materials it has been difficult to achieve similar values in either wires or tapes [2]. Some success has been recently reported in ReBCO tape joints [3, 4], but there are fewer reports of PGJs using Bi-2212 wires [5]. This is disappointing because Bi-2212/Ag multifilamentary conductor is the only HTS material manufactured in the form of a round wire [6], and so offers several advantages for the designers of high field magnets [7]. This wire is manufactured using a powder-in-tube method [6, 8, 9], with the wires undergoing a complex heat treatment following the drawing process to achieve high  $I_c$  performance. The heat treatment involves the controlled peritectic decomposition of the 2212 phase, and then a lengthy slow cooling in

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which the superconducting phase is reformed with the correct microstructure, and the oxygen content is adjusted [10, 11]. Since the wire is very brittle after this heat treatment [12, 13], it is normally wound into coils prior to the treatment (the wind-and-react method). This presents challenges for the fabrication of PGJs, since it is difficult to make a joint between vulnerable, brittle wires following the heat treatment without damaging the superconducting path. One strategy to overcome this problem is to prepare a joint before heat treatment so that the coil and the joint can be processed in a single heat treatment, which reduces the need to manipulate wires after melting with the risk of damaging them. Chen *et al* [5] have demonstrated that use of a termination joint formed by a pot of 2212 external to the coil can successfully create persistent performance, with a critical current of around 70 A and a residual resistance of approximately  $5 \times 10^{-12} \Omega$ . This process successfully follows the strategy used for LTS magnets, where joints made using solder are very reliable, but due to the relatively poor superconducting properties of the solder [14] must be situated in the lower field regions outside the main magnet windings (less than 1 T) [15]. This approach may not be so effective for Bi-2212 wires that are not manufactured in the very long lengths in which LTS materials are commonly available [6, 16].

This paper focuses on the development and testing of prototype PGJs for Bi-2212/Ag round wire that are sufficiently small to be considered for an in-winding joint strategy. The aim is to offer magnets designers the opportunity to leverage the advantages of Bi-2212/Ag round wire in the design of the larger magnets needed for resonance systems operating at very high fields ( $>20$  T).

## 2. Experimental methods

Standard Bi-2212 multi-filamentary round wire, manufactured by Bruker-OST, was used to wind small Bi-2212 superconducting coils. The cross section of the unreacted Bi-2212 round wire is shown in figure 1(a). The unreacted wire, with a diameter of 1.3 mm, is composed of  $18 \times 121$  Bi-2212 filaments embedded in an Ag stabilizer with an outer sheath of Ag-0.2 wt.% Mg alloy. While the general form of the heat treatment process is well known [17], careful calibration, including microstructural characterization and superconducting property measurements, was carried out to optimize the annealing process in our furnace. The optimized heat treatment profile was achieved using a  $T_{\max}$  of 895 °C for 0.5 h and final annealing at 835 °C for 60 h in an oxygen flow of  $600 \text{ cc min}^{-1}$ . Figures 1(b) and (c) show the microstructure of the Bi-2212 filaments before and after this optimized process. The isolated filaments have been melted and reformed as a network of interconnected superconducting filaments within the Ag matrix.

Several closed coils with joints have been fabricated, heat treated and characterized. Figure 2 illustrates the steps to assemble Bi-2212 solenoids. First, as-drawn Bi-2212 round wire was wound around an Inconel 600 alloy former. The coil consisted of 2 layers, each with 14 turns, with a total conductor length of 1.2 m. The inner diameter, outer diameter and

height of the coil are 11 mm, 13 mm and 20 mm, respectively. The filaments at the wire ends must then be exposed by either polishing or etching. These techniques are critical to allow a superconducting path to be generated through the joint and have been discussed in detail in previous work [18]. In figure 2 the wires are shown bent through the top of the former. Then, using the most reliable process arising from [18], the free ends are polished into a scarf geometry by mechanical abrasion and the exposed Bi-2212 filaments of the two wires placed in close contact and secured with Ag wire. An Ag tube with internal diameter of 6 mm was prepared as the outer sheath of the joint, and after inserting the joint into the tube, one end was sealed. Then the tube was filled as tightly as possible by Bi-2212 precursor powder (the same powder as is used in original wire and supplied by Engi-Mat) from the open end and compressed and sealed. The total length of the joints made by this technique was about 2 cm. Finally, both wires with the joint were folded through 90 degrees and secured by the top supporting plate as shown in step 4 (figure 2 (4)). The entire assembly was heat treated through our optimized annealing process. The final product after annealing is illustrated in figure 2 (5), where the Inconel former has changed color as a result of oxidation.

The persistent operation of these Bi-2212 solenoids with integrated Bi-2212 superconducting joints was tested by the inductive resistance measurement (IRM), sometimes referred to as a field decay method.

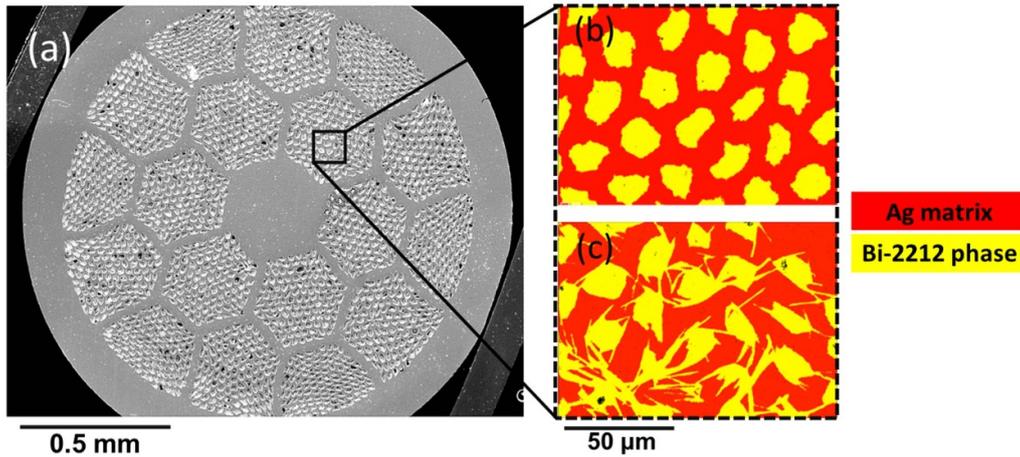
Figure 3 outlines the basic layout of the measurement using an Attocube magnetic force microscope system. The sensor employed for temperature monitoring was located as near as possible to the test coil, at the very bottom of the experimental probe. The Lakeshore HGT-2101 Hall probe was positioned in the middle of the test coil.

An IRM measurement is the tool of choice to characterize joint performance [14], and the basic procedure involves the induction of a current into the coil via a change in the background field [19, 20]. In figure 3, the external magnet (b) can be used as both an energization tool to induce current in the test coil and to generate a background field in which to test the joint performance.

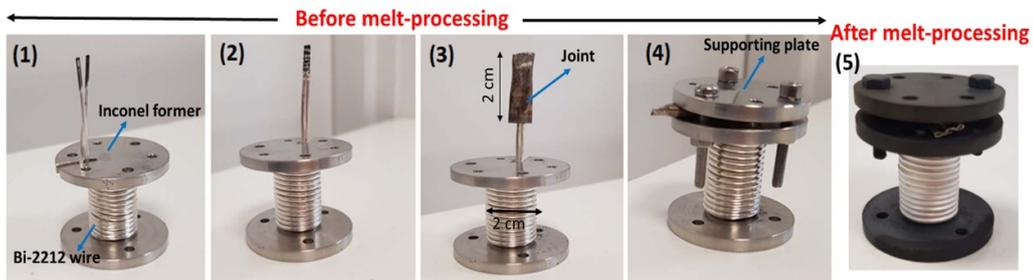
Field is first applied to a level of  $B_{\max}$ , the energizing field, before being lowered back down to zero, or to a desired background field. The resultant  $\Delta B$  drop in field results in a current being induced in the superconducting coil, in effect ‘trapping’ the applied field within the coil, assuming the joint is performing to specification. The critical current of this joint can then be extracted from the following relation between circulating current and axial field, derived from the Biot–Savart law, given the geometry and dimensions of our coils:

$$I = \frac{B\sqrt{4r^2 + l^2}}{\mu_0 N}$$

where  $I$  is the circulating current,  $B_{\text{center}}$  is the field at the center of the coil (where the Hall Probe is carefully positioned),  $l$  is the coil length,  $r$  is the inner radius of the coil,  $\mu_0$  is the magnetic permeability of vacuum and  $N$  is the number of turns in the coil.



**Figure 1.** (a) Cross-sectional SEM image of the unreacted Bi-2212 round wire. Phase maps of the Bi-2212 wire (b) before and (c) after the annealing process, generated from EDX elemental maps. The red and yellow regions identify the silver matrix and Bi-2212 phase respectively.



**Figure 2.** The steps in manufacturing Bi-2212 superconducting coils.

The decay of the trapped field is characterized by the equation,

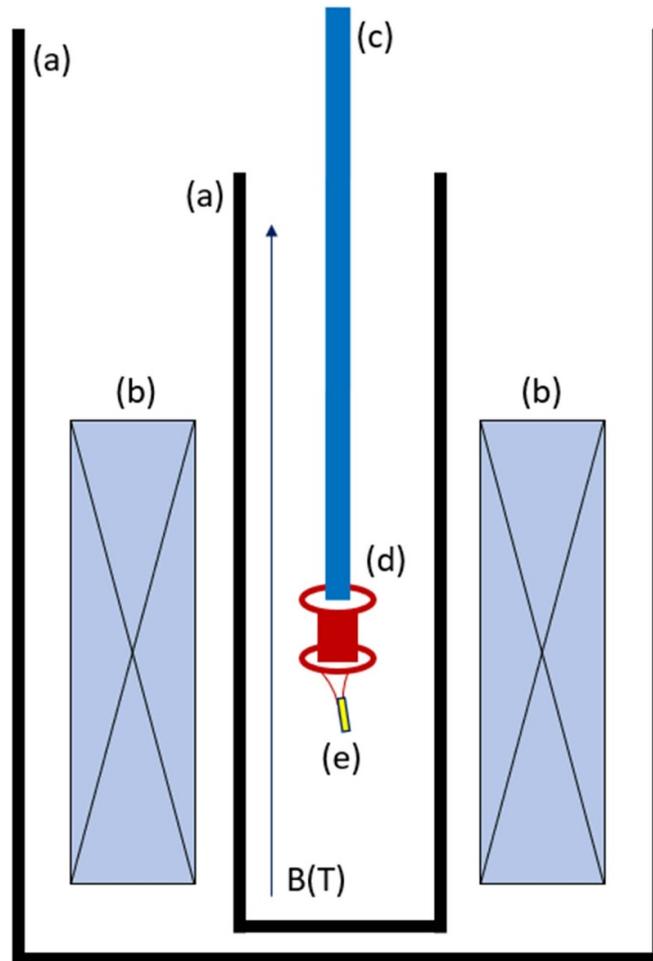
$$B_t = B_0 e^{\left(\frac{-R}{L}\right)t}$$

where  $B_t$  is the induced magnetic field at time  $t$ ,  $B_0$  is the initially induced magnetic field,  $R$  is the joint resistance and  $L$  is the coil inductance. The coils were calculated to have an inductance of around  $33 \mu\text{H}$ , based on the long solenoid approximation. These kinds of measurements can reliably measure resistances down to values of  $\approx 10^{-14} \Omega$  [21].

After decay field measurements, some joints were cut and polished for microstructural characterization. To prepare the joint section for the microstructural characterization, the joint was mounted in epoxy at an appropriate orientation to expose the desired section of the joint, including both wires and the remelted powder matrix forming the continuous superconducting path. The morphology and elemental/phase maps of the joints were studied using scanning electron microscopy (SEM) and energy-dispersive x-ray (EDX) analysis, using a Zeiss Merlin SEM operating at 20 kV accelerating voltage with an Oxford instruments (OI) 150 mm<sup>2</sup> XMax EDX detector. The EDX data was analyzed by the OI Aztec software.

### 3. Results

Figure 4 shows the connectivity after melt-processing between the Bi-2212 formed from the precursor powder in the joint region and the Bi-2212 filaments in the wire. In this region the local microstructure of the Bi-2212 phase can be seen, as well as sections of the Ag wires used to wrap two Bi-2212 wires together. It is clear in figure 4(a) that the outer silver layer of the two wires has been successfully scarfed away, so that the filaments have been exposed to the filler powder. The Bi-2212 precursor powder in the joint region has melted, and good wetting between this melt and the silver has resulted after peritectic re-solidification in the formation of obvious continuous pathways of 2212 phase between the two wires inside the external silver tube. Higher magnification images of the microstructure at the interface between the wires and the filler material in figures 4(b) and (c) also show that the melt-process has resulted in the desired plate-shaped microstructure, leading to the formation of 2212 grains which penetrate and make direct contact with the Bi-2212 grains in the filaments of the wire. In the regions where the wetting of the silver has been successful, there are no pores or gaps at the interface between the matrix and the Bi-2212 filaments in the wires.



**Figure 3.** A schematic of the essential components of the IRM experimental set up. (a) Inner and outer cryostat. (b) External magnet (c) the experimental probe, which contains a magnetic field sensor (usually a Hall probe) at the tip. This tip is inserted in the middle of the test coil (d), which is closed by joint (e).

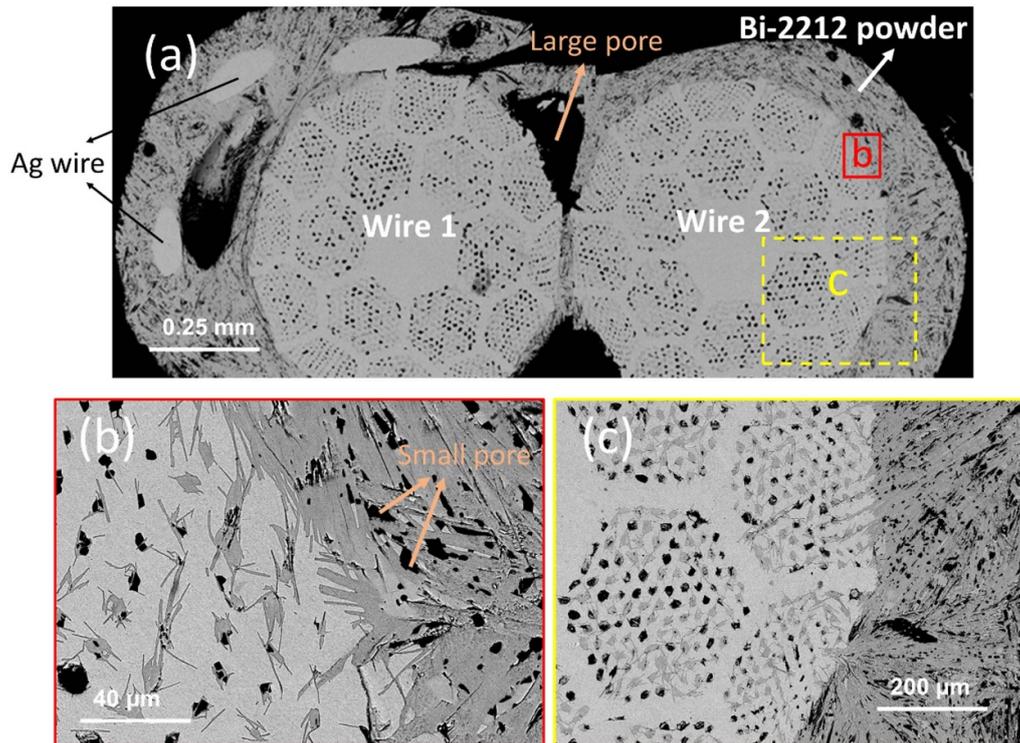
However, figure 4 also shows the presence of numerous pores in the joint region, with a wide range of sizes from few microns (visible in figure 4(b)) to  $500\ \mu\text{m}$  (shown in figure 4(a)). The large pores are an expected result of the substantial reduction in volume of the precursor Bi-2212 powder on melting and re-solidification, by as much as 30%, which can create large gaps in the supercurrent paths in the joint. A number of techniques widely used to manipulate powders in the ceramics processing industry [22] were examined to increase the density of the powder inside the silver tube and to prevent the formation of these large pores. A longitudinal section of the silver tube, Bi-2212 matrix and wire after an optimized packing process and melt-processing is presented in figure 5. The substantial reduction in porosity resulting from the high filling fraction of initial precursor powder in the joint is crucial in creating a continuous superconducting pathway between the two Bi-2212 wires capable of carrying large persistent currents through the joint.

In addition to examining the connectivity and density of pores in the joint region, we have investigated the chemical homogeneity and any possible impurity phase formation during the melt process. It has been reported, for instance, that

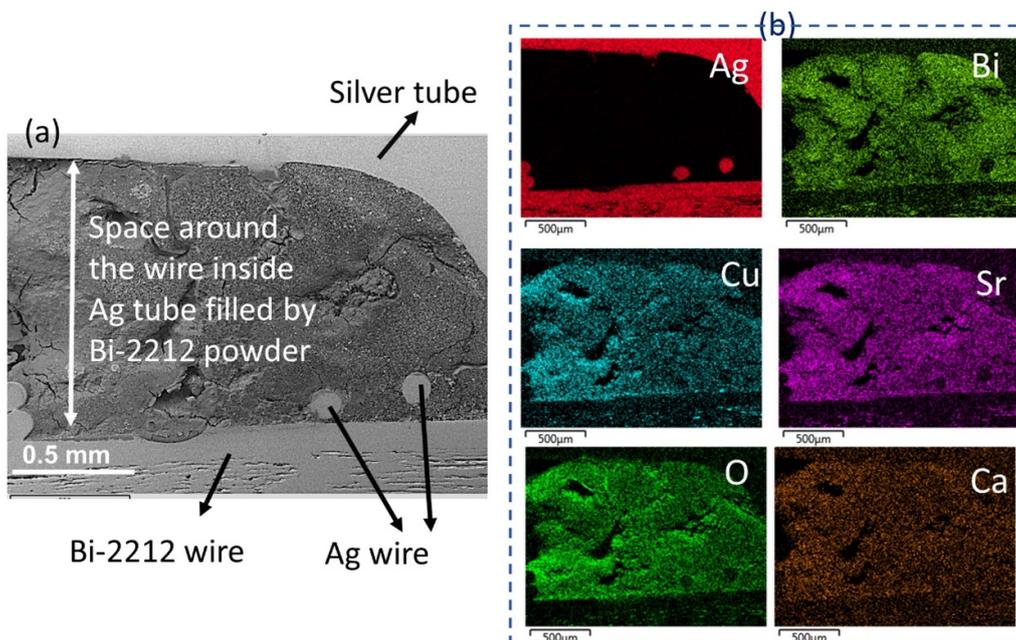
undesirable phases like Bi-2201 and Bi-2223 can be formed during the peritectic solidification of the precursor Bi-2212 powder if the processing parameters are not optimized [17, 23]. Figure 6 shows a high magnification SEM image of a longitudinal section of a joint, along with elemental maps from EDX analysis. In the layered EDX image (figure 6(b)) only two phases can be seen; Bi-2212 (blue) and silver (red), and there is no sign of any undesired phases, at least at the resolution offered by this kind of analysis. This phase pure microstructure is essential for a high-performance superconducting joint. There may be isolated unit cells containing 2201 or 2223 stacking [24, 25] but these are not expected to substantially affect the macroscopic current carrying capacity of the joint.

#### 4. Field decay measurements

The results of IRM field decay measurements at 4.5 K and in self-field on 8 short-circuited coils are given in table 1. These results demonstrate persistent currents of at least 83 A at residual resistance values below  $10^{-12}\ \Omega$  in 7 of the 8 coils tested; a reasonable degree of reliability for a programme



**Figure 4.** (a) Cross-sectional SEM images of two Bi-2212 wires in a typical joint region after melt-processing, (b) and (c) are high magnification images of the selected regions in (a).

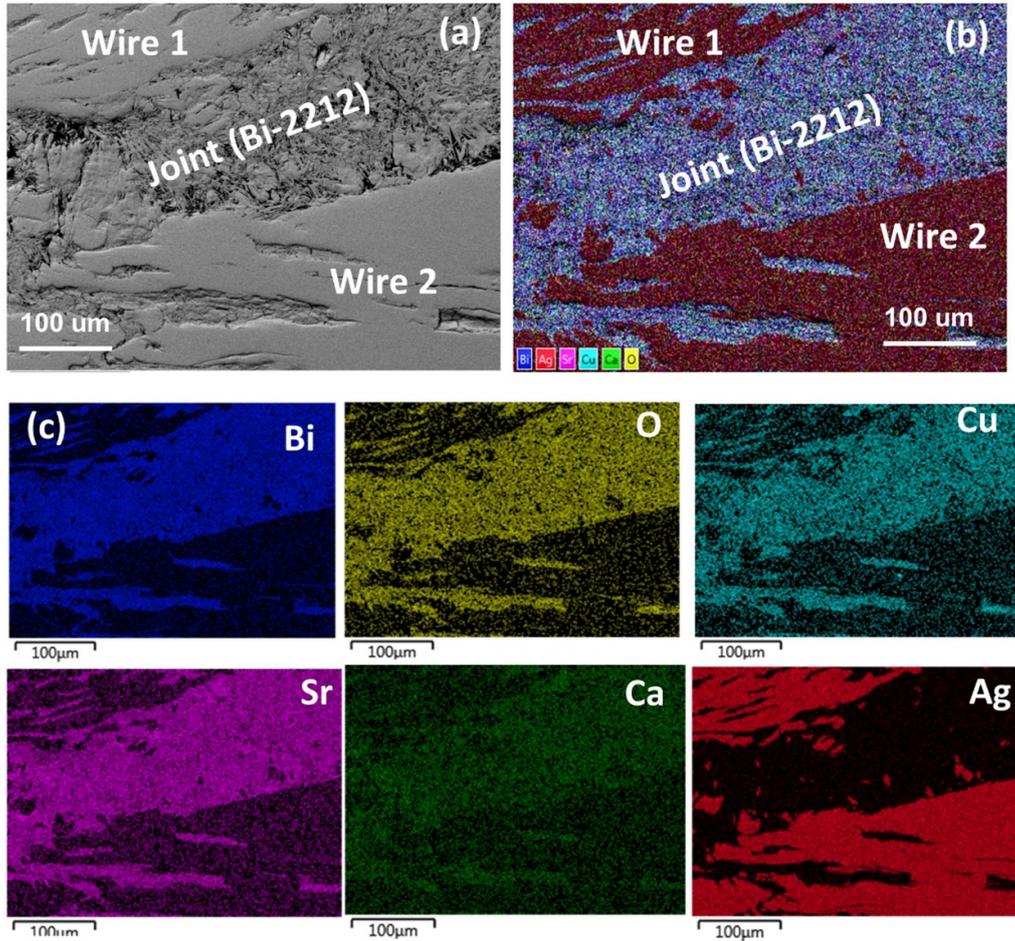


**Figure 5.** (a) Longitudinal section of an optimized joint showing good filling of the space inside the silver tube around two Bi-2212 wires.

developing the joint manufacturing processes. The only significant difference between these 8 samples is the length of the joint, which seems to have little effect on the performance. Figure 7 shows a typical plot of measured and calculated properties for Coil 2, and when this coil was tested in a background

field of 1 T the persistent current of 105 A only decreased by 11% to approximately 93 A.

We have previously shown that the  $I_c$  values of these Bi-2212 wires from transport measurements (with a standard voltage criterion of  $100 \mu\text{V m}^{-1}$ ) to be  $>500 \text{ A}$  in 14 T [18] but



**Figure 6.** (a) Longitudinal SEM image of the joint region between two Bi-2212 wires, (b) EDX layered image and (c) elemental maps of the joint region.

**Table 1.** Table summarizing the primary properties of the tested joints obtained from the IRM field decay measurement. All measurements were taken in self-field and at 4.5 K.

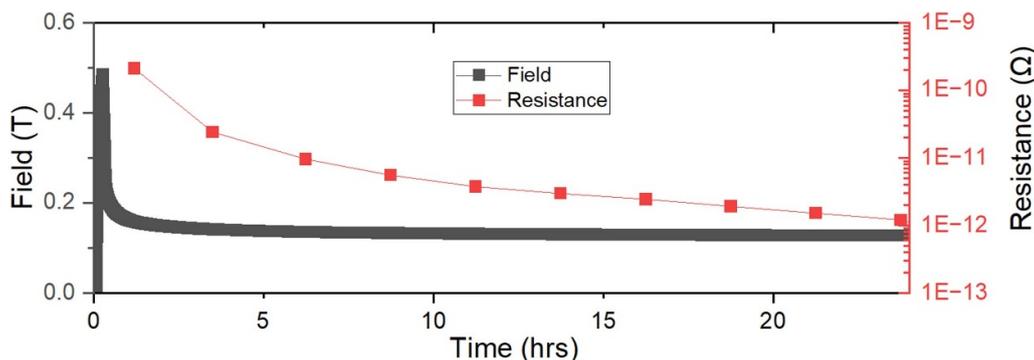
Coil	Critical current (A)	Resistance ( $\Omega$ )	Settling time (h)
1	83	$<10^{-12}$	22
2	105	$<10^{-12}$	25
3	123	$<10^{-12}$	30
4	101	$<10^{-12}$	34
5	105	$<10^{-12}$	32
6	0	$\approx 10^{-9}$	N/A
7	99	$<10^{-12}$	33
8	97	$<10^{-12}$	60

note that this voltage criterion corresponds to a residual sample resistance of approximately 10 n $\Omega$ , a value at least three orders of magnitude higher than we have imposed to define persistence. We have no comparative values of wire  $I_c$  in background field or at 1 T, so direct calculation of the critical current ratio for these joints is not possible, but it is clear that there is considerable scope to improve their performance.

The settling time values given in table 1 represent how long it took for the resistance of the joint to settle to below  $10^{-12}$   $\Omega$ , which is useful parameter for the magnet designers. The critical currents quoted in table 1 are extracted after the

coil has settled to persistence. It is notable that in comparison to solder joints between Nb–Ti wires, there is a prolonged time of at least 1 d during which the performance of these Bi-2212 coils continues to settle. This is not the case for Nb–Ti coils of similar sizes, which we have observed to settle within 1 h at equivalent currents and background fields.

After storage in the laboratory for 6 months, with no protective measures, Coil 2 was re-tested in self-field and 4.5 K, and demonstrated identical  $I_c$  and resistance characteristics, showing that this process can produce coils that are resilient to atmospheric oxidation or other environmental degradation,



**Figure 7.** A field decay curve showing the performance of coil 2 when energized with a 0.5 T pulse. 0.125 T of field was trapped by the coil, which is a trapped current of approximately 105 A. The coil took approximately 25 h to settle below the persistent-grade criterion of  $<10^{-12}\Omega$ .

and also show resilience with respect to thermal cycling, which is of considerable importance in practical magnet design.

Compared to the only previous report of the performance of a single melt processed Bi-2212 joint measured by IRM field decay under persistent conditions [5], we have achieved a 73% increase in persistent  $I_c$  in a much smaller joint with less thermal mass and potentially more suitable for on-coil jointing strategies, in addition to demonstrating some progress towards the reliability essential for an industrial manufacturing process.

## 5. Conclusion

We have demonstrated a reliable and repeatable process for creating small melt-processed persistent-grade joints between Bi-2212/Ag multifilamentary wires. These joints could carry up to 123 A in self-field and 93 A in 1 T background field, while settling after a few days to a resistance below  $10^{-12}\Omega$ . While there is still room for improvement in the persistent  $I_c$  values, these joints nonetheless approach the specifications for high field NMR magnets. We have also showed promising progress in demonstrating both the reproducibility of the manufacturing process and the resilience of the joints to storage and thermal stress.

The microstructures of the joints show obvious connectivity in the 2212 phase between the two wires, and do not show any traces of undesirable phases, supporting the existence of a superconducting pathway between wires. The porosity resulting from the volume reduction from powder to melt remains the primary factor limiting the joint  $I_c$  values achieved. Current work is focused on improving the matrix density by optimizing the powder packing technique and the geometry of the joint.

## Data availability statement

The data that support the findings of this study are openly available at the following URL/DOI: <https://github.com/Petr-Zagura/Development-of-persistent-joints-for-superconducting-Bi-2212-coils> as well as <https://ora.ox.ac.uk/objects/uuid:384ecd39-cf15-45df-bbcd-1eefdd3f7dc1>.

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