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The final version of record is available at http://dx.doi.org/10.1109/TASC.2021.3064512

Superconducting Joint Structures for Bi-2212 wires using a Powder-in-Tube Technique

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Abstract— **Bi-2212/Ag superconducting wires are being considered as key conductor candidates for the development of high-field magnets. This work focuses on the design and testing of various joint structures for multifilamentary Bi-2212 wires using powder-in-tube techniques. The joints are made between the unreacted wires and subsequently heat treated with the wires. The microstructure and transport properties of different joint architectures including step joints, scarf joints and etched joints have been explored.**

The performance of etched-joints are found to be very sensitive to the etching parameters including etching time, making it difficult to develop a reliable jointing process. Scarf joints and step joints can carry higher currents (320 A and 475 A in self-field at 4 K) than the etched joints (225 A in self-field at 4 K) as a result of connecting a larger area of the exposed filaments without damaging the filaments.

Index Terms—**Superconducting joints, Bi-2212 wires, Powder-in-tube technique.**

I. Introduction

Bi2212/Ag multifilamentary round wire is the only high-temperature superconductor in a round wire form [1], and offers a number advantages for solenoid magnet design and fabrication including the ease of applying conductorinsulation and coil winding, as well as the ease of cabling for high field accelerator magnets [2,3]. The Bi-2212 wires are fabricated by a powder-in-tube technique [4,5] with the as-drawn wires being subsequently melt-processed using a complex heat treatment to form high critical current superconducting wire [6]. A key enabling requirement for using these promising high-temperature superconducting wires in high field magnets is development of low resistance joints between the Bi-2212 wires [7-9]. For MRI/NMR applications these joints need to have resistance values $<10^{-12}\ \Omega$ to allow operation in persistent mode [10,11]. Several joint techniques have been explored in the literature for fabricating persistent mode joints in these wires including soldering [12] using both PbBi and Pb-free superconducting solders [13-16] and powder-in-tube melt processing [17, 18]. Among these techniques, powder-in-tube is a promising approach with a potential of fabricating joints with resistivity as low as $5\times10^{-12}\ \Omega$ [17,18]. In this technique, the Bi-2212 filaments of unreacted wires are exposed and joints are made between them by wrapping the exposed wires together and placing the joint into a silver tube filled by Bi-2212 powder. The entire assembly then undergoes melt processing [17]. This technique can be optimized by exploring different joint structures and methods to expose the filaments. Here we have studied the powder-in-tube technique for different joint architectures to find out the best joint assembly for the highest critical currents. We have also examined the microstructure of these joints along with evaluating their transport properties to evaluate the overall performance of various joint assemblies and determine the most promising joint technique for these wires.

II. Joint Assembly and heat treatment

Bi-2212/Ag multifilamentary wires used in this work were provided by Bruker. The architecture and the typical microstructure of the wire can be found in [12]. Superconducting joints were fabricated between straight lengths of these wires using a powder-in-tube technique. The first step of this is exposing the superconducting filaments from the two wires. Fig. 1 shows schematically the different methods used to expose the filaments. To fabricate scarf joints, the wires are polished at an angle of about 5º using a Dremel tool to provide large cross-section surfaces from both wires. For step joints, a specific length of the wires is longitudinally polished away down to the middle of the wire resulting in exposure of long lengths of filaments.

For etched joints the outer silver layer of the Bi-2212 wire is removed using an etchant solution. Various ratios of NH_4OH (30%): H_2O_2 (30%) solution were used as the etchant [19], and it was found that a mixture with a ratio of NH_4OH 3:2 H_2O_2 is the most effective at dissolving the silver layer. Once the filaments are exposed by any of these methods, the wires are wrapped together with extra Bi-2212 powder in between such that the exposed filaments from the two wires are in contact and held together firmly using silver wire as shown schematically in Fig. 1a. This assembly is then placed in a silver tube which is filled with Bi-2212 powder and mechanically sealed from both ends. The entire assembly including joint and the wire is melt-processed at a constant oxygen flow of 600 cc/min. The final joint is relatively small with a length and width of about 2 cm and 0.5 cm respectively as shown in Fig. 1a. In all of these joints, the same Bi-2212 powder, provided by nGimat, and the same wire, provided by Bruker OST were used.

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The transport properties of these joints were measured at 4 K using 4-terminal transport measurements on short straight (~2 cm) sections of wire containing a single lap-style joint in applied magnetic fields of 0 to 14 T in a direction parallel to the wire axis. The distance between voltage taps was 2 cm. The measured transport properties of the Bi-2212 wire after melt-processing show that they can carry at 4.2 K more than 500 A at 14 T, the maximum values in our test facility, with no transition to the normal state.
The microstructure of each joint was examined by Scanning Electron Microscopy (SEM) using a Zeiss Merlin SEM with an Oxford Instruments (OI) 150 mm^2 XMax EDX detector.

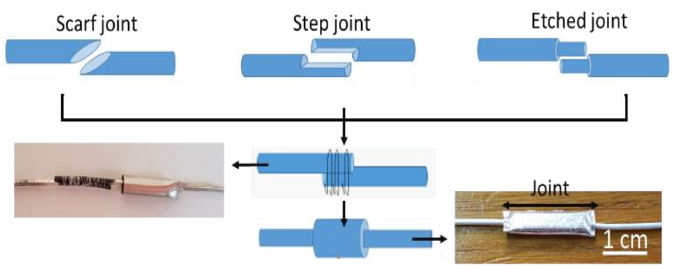

Fig. 1. Schematic images showing the procedure of joint development between Bi-2212 wires using powder in tube technique. Different joint architectures including scarf, step and etched joints are shown schematically.

III. Results

A. Etched joints

Fig. 2 shows cross-sections of the wires after exposing to the etchant solution at various lengths of time along with a plot showing the length of dissolved section as a function of etching time. The etching process seems slow at the beginning (etching rate: 1.5 µm/min) with only 40µm and 70µm of the silver layer dissolving after 30 mins and 45 mins respectively. At 50 mins, the silver layer seems to be entirely removed with no major damage to the superconducting Bi-2212 filaments, as seen in Fig. 2a. However, the etching rate becomes very fast (etching rate: 15.3 µm/min) after the entire silver layer is removed, and the superconducting filaments star dissolved in the etchant solution with a rate ten times faster than the etching rate for the silver layer as can be seen from the increase in slope of the plot presented in Fig. 2b. This is likely to be due to the loosely compacted powder of the unreacted Bi-2212 filaments within the silver matrix.
The plot in Fig. 2 also shows the percentage of filament damage as a function of etching time. Up to 45 mins the etching process is dissolving only the outer silver layer and consequently the damage to the filaments is zero. However, 15 mins after the silver has been removed, more than 80% of the filaments have been removed meaning that even if a successful joint is made for these wires in the following stages, the transport properties of the joint will be <20% of the original wire. Therefore choosing the correct etching parameters will play an important role in the final performance of the joint. It is a highly delicate stage which has to be controlled carefully in order to completely remove the silver layer with minimal damage to the filaments.

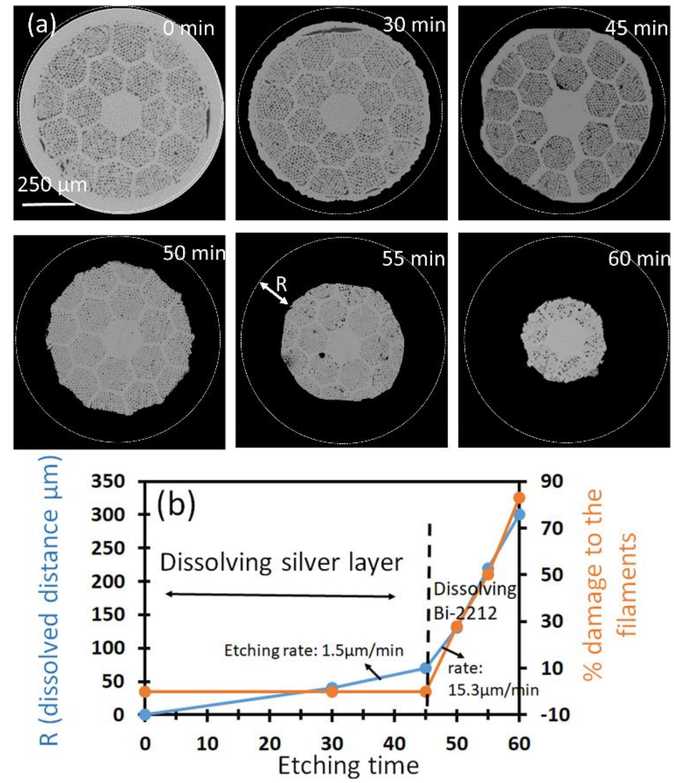

Fig. 2. (a) Cross-sectional SEM images of the Bi-2212 round wire after etching at different times, (b) the dissolved length (R) of the wire as indicated in the cross-section and percentage filament damage as a function of etching time.

Fig.3 shows a typical cross-section of an etched joint along with the transport properties of the joint. The Bi-2212 powder has wetted the wires well, and after melt-processing almost all of the powder has solidified around the two wires. High magnification images of the interface between the powder and the wires (Fig.3 b and c) show that excellent connections have been developed between the filaments of the wires and the Bi-2212 powder in the joint area. This suggests that the supercurrent can pass from the superconducting filaments of the wire 1 to the superconducting joint region formed by the Bi-2212 powder and finally to the filaments of the wire 2.
The transport properties of the etched joint is presented in Fig. 3d. At self-field, the joint shows has a critical current (I_c) of 250 A (determined using a 1 µV criterion), and at 14 T I_c > 80 A. Despite the observed superconducting paths between the filaments and the joint region, these currents are much lower than the Bi-2212 wire (>500 A at 14T). The first reason for the poor transport properties of this joint is the considerable degree of porosity in the joint region that can be seen in Fig 3a. This has formed during melt-processing as a result of volume reduction of the powder during the melting process. Secondly, the damage to the filaments through the etching process, as discussed above, may also contribute to deterioration of the superconducting performance of the joint.

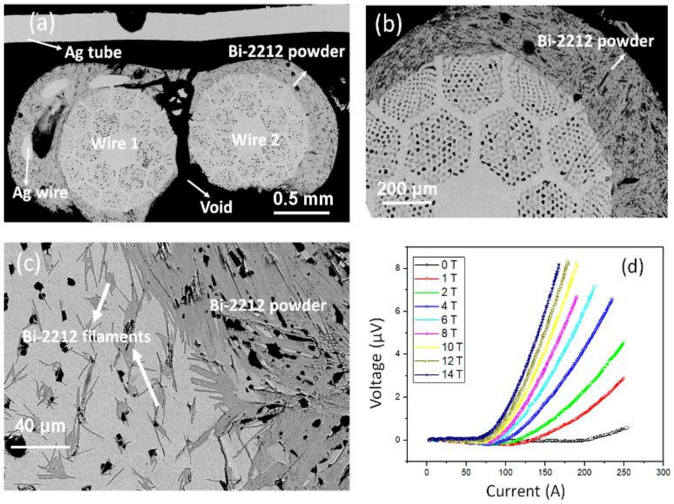

Fig. 3. (a) SEM image of a typical cross-section of an etched-joint. (b) and (c) high magnification images of the interface between the original Bi-2212 powder and the wire showing apparently good connections between the filaments of the wire and the powder in the joint, (d) transport measurements of the etched-joint at 4 K.

B. *Scarf joints*

Fig. 4a shows the longitudinal section of the scarf joint for the wires polished in an angle of about 5º. Depending on the polishing angle, the surface of the exposed filaments will be different. The smaller polishing angles provide a larger area of the exposed filaments and consequently a higher chance for connections between filaments and the joint region which finally leads to better joint performance. Using a simple calculation, the ratio of the filaments area after polishing to the filaments area before polishing is found to be proportional to $1/\sin(\alpha)$ where α is the polishing angle. Therefore, the smaller polishing angles considerably increase the area of the exposed filaments.

The transport properties of a typical scarf joint are presented in Fig. 4b. In self-field and 14 T, the joint I_c is 250 A and 120 A respectively. These critical currents are higher than achieved with etched joints, possibly owing to the fact that in the scarf joint architecture a larger area of the filaments can be exposed with no damage to the filaments. However, the critical current of the scarf joint is still low compared to the Bi-2212 wire itself because of the presence of a large amount of porosity in the joint region as discussed above.

C. *Step joints*

Using the step joint method, a large area of filaments can be exposed if the step is made right to the middle of the wire. It is also important that the step is the same length for both wires as this defines the joint length and also reduces the distance between the ends of the filaments from the two wires, ie higher chance of getting current through end-to-end of the wires. Fig. 5 shows the microstructure of the longitudinal section and transport properties of the step joint. As indicated by orange arrows, there are some connected areas between two wires. The high magnification image of one of these areas (Fig. 5 b) shows that the powder at the joint is well connected with the filaments of the two wires, creating a continuous path between the filaments. The corresponding EDX elemental map (Fig. 5c) confirms that the joint region contains Bi-2212 phase as the majority phase with some small regions (<30µm) of Bi-2223. These phases are formed during melt-processing and make good connections with filaments in both wires. This joint exhibits an I_c value of 475 A at self-field, and 150 A at 14 T.

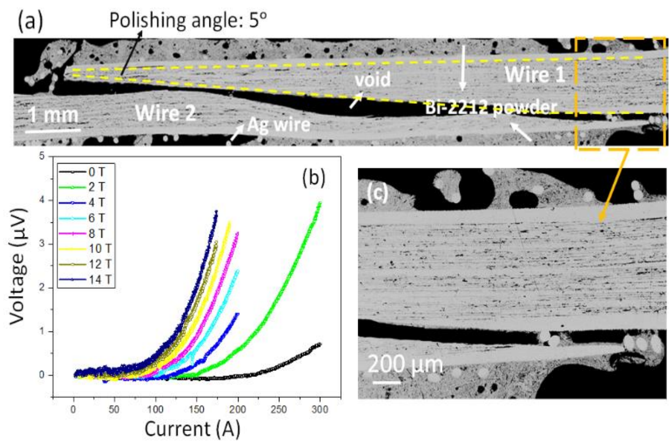

Fig. 4. (a) The microstructure of the longitudinal section of the scarf-joint between Bi-2212 round wires, (b) transport measurements of the scarf-joint at 4 K, (c) high magnification SEM image of the indicated area of the joint.

Table 1 summarizes the transport properties of these joints, using the standard voltage criterion of 1 µV. The n-value for each joint has been calculated through a power law assumption, $V=kI^n$ where V and I are voltage and current respectively. As a result of porosity at the joint region, the I_c values of these joints are lower than the I_c value for the Bi-2212 wire melt-processed at the same heat treatment conditions. The scarf joint and step joint exhibit superior transport properties compared to the etched joint as a result of effective exposure of Bi-2212 filaments in these joints. As a sensitive stage, the etching process needs a careful control and may damage the filaments and deteriorate the superconducting properties of the joint. On the other hand, by polishing the wire, a large contact surface can be created with undamaged exposed filaments.

IV. Discussion

The I_c values (4 K, 14 T) of the joints are much lower than that of the single wire. An extensive investigation of the microstructure of these joints suggests that the main reason for low I_c values of the joints compared to the wire is the presence of a large amount of porosity and even large gaps between the filaments of the two wires in the joint region. The presence of these pores at the joint region is a result of a large volume reduction of the Bi-2212 powder after melting. Estimated from a simple experiment, we found that after melt-processing, the volume of the powder used in our experiments reduces by a factor as high as eight once it has been melted and refrozen. This factor depends on the powder particles and the way that the tubes are filled by powder while assembling the joints. A refinement of the powder particle size (eg by milling) might improve the flow characteristics and the density of the powder inside the tube. Applying pressure on the tube after filled by

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powder can also reduce the porosity and empty spaces inside the tube. Another approach can be using high-pressure heat treatment which has been shown to efficiently suppress the porosity in the 2212 conductors and thus generate high engineering transport properties [20].

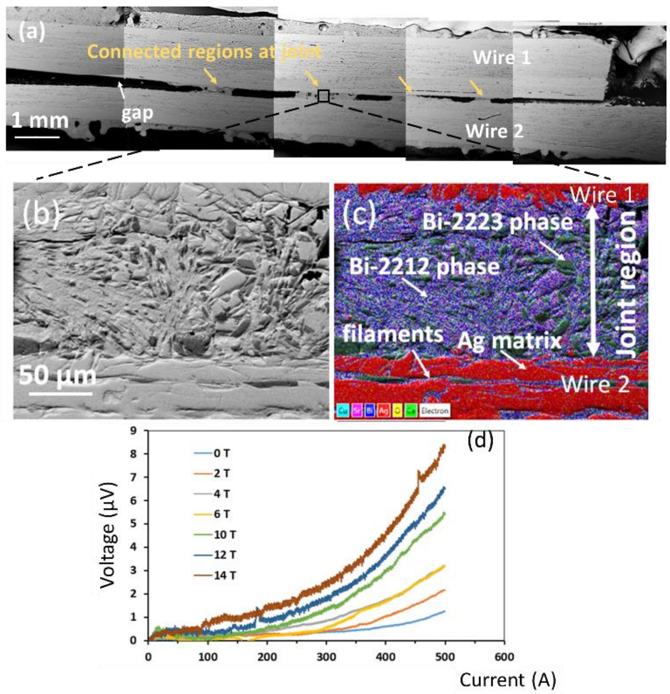

Fig. 5. (a) The microstructure of the longitudinal section of the step-joint between Bi-2212 round wires, (b) high magnification SEM image of one of the connected area at the joint region, (c) EDX elemental layered map of the indicated region showing a continuous superconducting path between two wires, (d) transport measurements of the step-joint at 4 K.

Table 1. Transport properties of the various joints made in this work. The I_c values are taken at a standard voltage criterion of 1 µV. The joint length (the distance between the voltage taps) is 2 cm for all the samples. The magnetic field of 14 T and the current of 500A are the maximum values in our measurement facilities.

	wire	etched joint	Scarf joint	Step joint
I_c (A) at 4 K, self-field	> 500	225	320	475
I_c (A) at 4 K, 14T	> 500	80	140	150
n value (self-field)	-	14	15	12
n value (14 T)	-	10	9	8

V. Conclusion

The powder-in-tube technique has been investigated for various joint architectures (etched, scarf and step joints) to assess their suitability as a persistent mode jointing technology for multifilamentary Bi-2212 round wires. The transport properties of the joints along with their microstructure shows that the wettability between the powder and the Bi-2212 filaments inside the wires is excellent. The powder forms well-connected paths of superconducting phase between the filaments from the wires. However, the volume reduction of the powder during the melt process is huge and creates large voids in the joint region which reduces the I_c values of the joints. Polishing the wires in either a scarf structure or step structure is an efficient way to expose large areas of the wire filaments to join without damaging the filaments. The etching process was found to be a highly sensitive procedure requiring a very careful optimization in terms of etching time and choice of etchant solution to avoid any damage to the filaments which can deteriorate the final performance of the joint.

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