Enhanced-Textured-Powder Bi-2212/Ag Wire Development

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Abstract. A new method for preparing Bi-2212/Ag multifilament wire is being developed. Bi-2212 fine powder is homogenously mixed with nanoparticle Ag and uniaxially compressed into square-cross-section rods. The rods are assembled into a silver matrix and drawn into monofilament wire, restacked, and drawn into multifilament wire. Heat treatment studies have been conducted to optimize parameters for a non-melt heat treatment. Transport current at 77 K and microstructure measurements have been made to guide the process development.

1. OPIT Bi-2212 Wires

The current state of the art development techniques for Bi-2212 multifilament wire is the oxide-powder-in-tube (OPIT) process. For the OPIT process phase pure Bi-2212 fine powder is loaded into silver tubes, the filled tubes are then drawn, stacked, and re-drawn until the desired filament size and number is achieved [1]. The wire can then be cabled into various forms including Rutherford cable [2], and cable-in-conduit [3]. After the cabling is complete it can be wound onto its final form where it will undergo a partial melt (PM) heat treatment. The entire form is then cooled in a controlled manner to recrystallize and anneal the Bi-2212. However, the PM heat treatment leads to several difficulties:

- The small void spaces within the powder coalesce into large current occluding bubbles within the filaments when the powder is melted [4].
- The melt liquid is chemically aggressive and will etch away the silver matrix [5].
- The recrystallized Bi-2212 are not textured and have limited connectivity between individual crystal formations [6].

Overpressure processing at 5 – 10 MPa during the PM heat treatment has reduced the size of the coalesced void spaces that are formed, and therefore dramatically improved performance [7, 8]. We have pursued a process to create high density, textured Bi-2212 cores that undergo a non-melt heat treatment, and thereby avoided three major performance hindrances of the PM process. This work is the latest results in the pursuit of that process.

2. Enhanced Textured Powder Bi-2212 Cores

Phase pure Bi-2212 has been shown to become textured and densified when subjected to uniaxial pressure (~ 20 KSI) [9]. The powder can be enhanced by homogenously mixing fine Bi-2212 powder with nano scale silver (20-100 nm). This enhanced powder can then be uniaxially pressed into a long thin die to create an enhanced textured powder (ETP) core, shown in figure 1. If these cores undergo a non-melt heat treatment they have been shown to create large highly interconnected grains that retain

their texture [10]. Once several ETP cores have been prepared they can then be used to make a monofilament wire.

Figure 1. Square cross section ETP core

3. Wire Fabrication

The fabrication of the initial monofilament wire is the only step in the process of producing multifilament Bi-2212/Ag wires that differs from industry standard practices of OPIT wires. A silver tube is drawn over a square mandrel to produce a cylindrical tube with a square cross section hole. Once the tube has been thoroughly cleaned the ETP cores can be carefully inserted end to end so that no gap is left between each ETP core, shown in figure 2a. The ends are sealed, and the billet is precisely drawn so that the void space between the core and the tube is closed locking the ETP cores in place, without significantly altering their arrangement. The tube is then drawn, stacked, and redrawn according to normal practices to produce multifilament Bi-2212 wires, shown in figure 2b, and 2c. During the wire drawing process there were no indications of individual cores separating, or wire breakage.

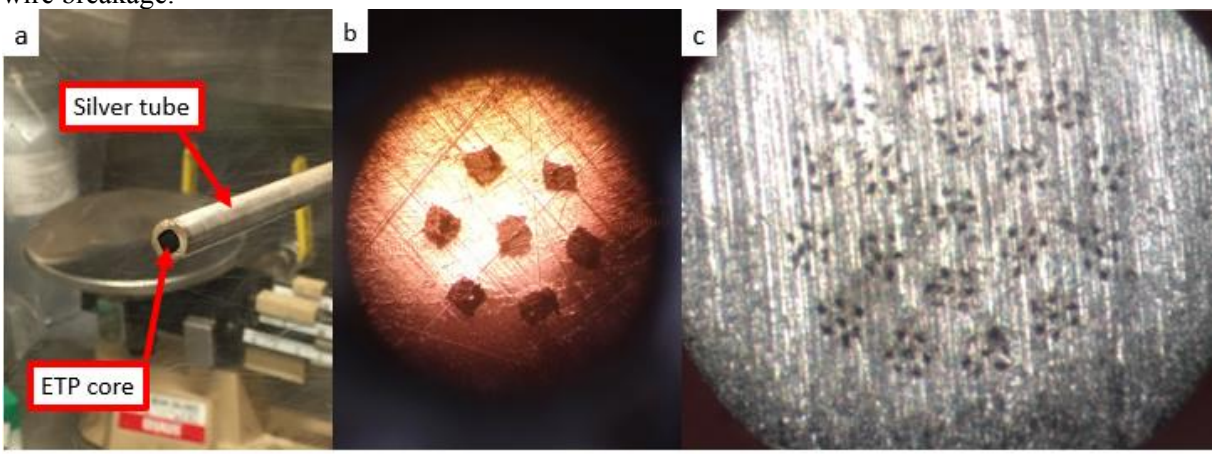

Figure 2. a) An ETP Bi-2212 core after insertion into a silver tube. Images of 6-on-1 (b) and 7 x 19 (c) multifilament wires made from ETP Bi-2212 cores.

4. Non-melt Heat Treatment

Pre-texturing the Bi-2212 would be irrelevant without a non-melt heat treatment as all texture is lost during a PM heat treatment. Initially a non-melt heat treatment was attempted for a textured powder core without silver nano particle enhancement but yielded little to no superconductivity [8]. The silver nano-particles locally lower the melt point of the Bi-2212 grains at the silver/Bi-2212 boundary allowing the area between textured grains to be melted and recrystallized, while never melting the bulk of the Bi-2212.

A typical Bi-2212 PM heat treatment profile, shown in figure 3, is used while altering the maximum temperature and increasing the dwell time at maximum to 24 hours. This ensures that the sample is allowed to completely and uniformly reach the maximum temperature. The internal furnace environment is 1 atm of pure oxygen, until the annealing phase when the furnace is purged with air.

Heat treatments for maximum temperatures between 875 °C and 879 °C were performed on short samples of 6-on-1 multifilament wires made from ETP cores, and ETP cores with embedded silver tabs, both shown in figure 4. The embedded silver tabs serve as solder points for current leads and voltage taps, without them the ETP bars were unable to be soldered.

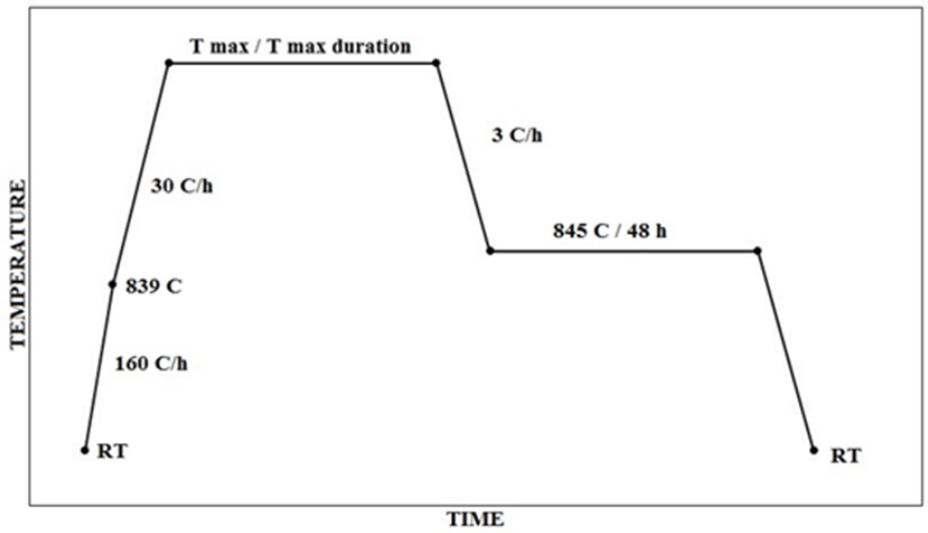

Figure 3. The non-melt heat treatment profile used during this study.

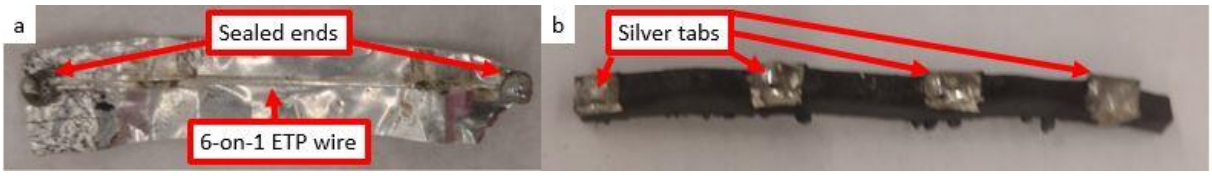

Figure 4. An ETP 6-on-1 multifilament wire (a) and ETP core showing silver tab solder points (b) after non-melt heat treatments

5. Liquid Nitrogen Testing

A liquid nitrogen test was used for the initial testing of both the ETP cores and ETP multifilament wire to limit costs while still providing valuable information of whether a superconducting state is reached or not. All samples of both the ETP cores and the ETP multifilament wires were soldered to the current leads of the LN test apparatus. The samples were precooled in LN vapor before being bathed in LN to minimize to chance of fracturing the fragile cores due to thermal shock. Once thoroughly cooled to 77 K, current was slowly increased and a voltage was measured between voltage taps. None of the multifilament wires showed evidence of superconductivity, but surprisingly several of the ETP cores that were heat treated simultaneously with multifilament wires showed considerable supercurrent transport, albeit with a higher than expected resistivity. Figure 5 shows current testing results of the ETP cores that were heat treated to a maximum temperature between 877.5 °C and 879 °C displaying clear evidence of superconductivity. The ETP cores heat treated below a maximum temperature of 877.5 °C show drastically higher resistance. The figure also shows an example of data from a multifilament ETP wire, labeled as silver wire. This data showing ETP cores superconducting would suggest that similarly heat treated ETP wires should also show evidence of superconductivity, but this was not the case.

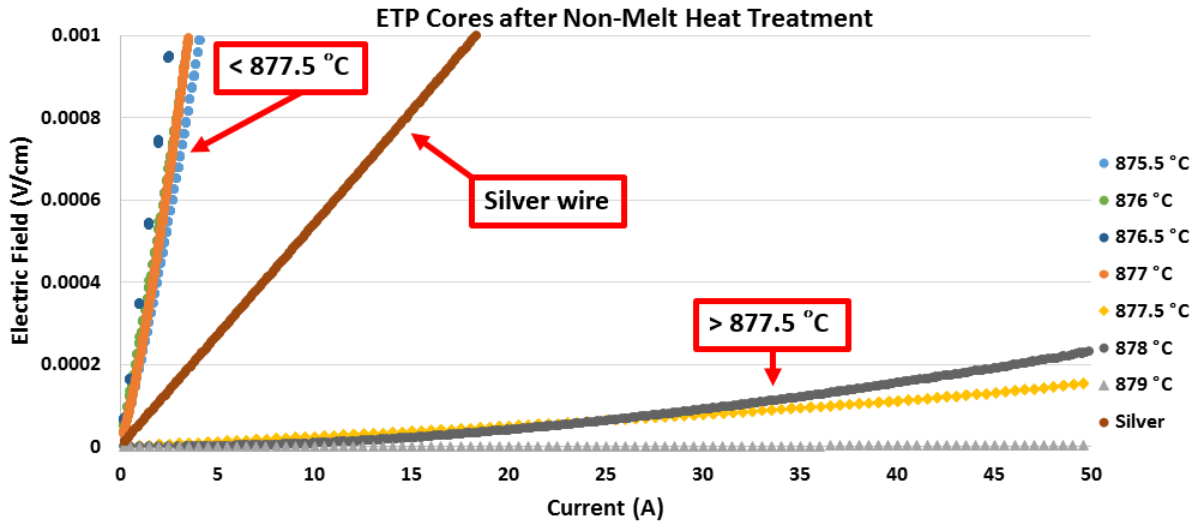

Figure 5. Results of LN testing of ETP cores after a non-melt heat treatment with different maximum temperatures. Note that those treated at 877.5 °C or above show signs of superconductivity while those treated below do not.

To explain the higher than expected resistivity a test of critical temperature was done using an ETP core that was heat treated to a maximum temperature of 879 °C. A thermocouple was mechanical attached to the sample and it was bathed in liquid nitrogen. Once cooled to LN temperature the sample was slowly allowed to warm in nitrogen vapor while a small current (~0.5 A) was passed through it. This was done to see how the voltage at a constant current changed as the sample changed temperature. The voltage was measured and recorded along with the temperature of the sample. Figure 6 shows that at LN temperature the sample is in the transition between the normal and superconducting states. This would explain the very gradual increase in voltage seen in figure 5, instead of the sharper transition that would be expected.

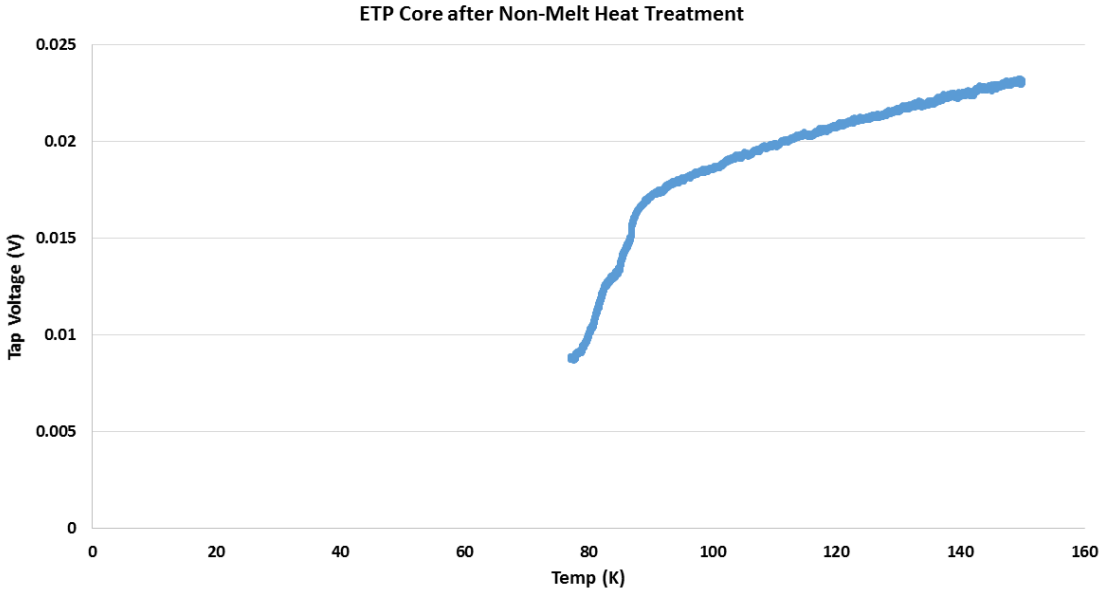

Figure 6. A figure showing the transition temperature of an ETP core after a non-melt heat treatment lower than expected. Note the ETP core is only partially in a superconducting state, thereby explaining the higher than expected resistivity seen in figure 5.

To verify beyond macroscopic visual inspection that the ETP cores that displayed superconducting properties did not melt, SEM images were taken. Figure 7 shows that the samples were not melted and when qualitatively compared to the images of textured samples achieved in [9], it highly suggests that texture was maintained. The images also clearly show interconnected grains of Bi-2212 both on the surface and internally.

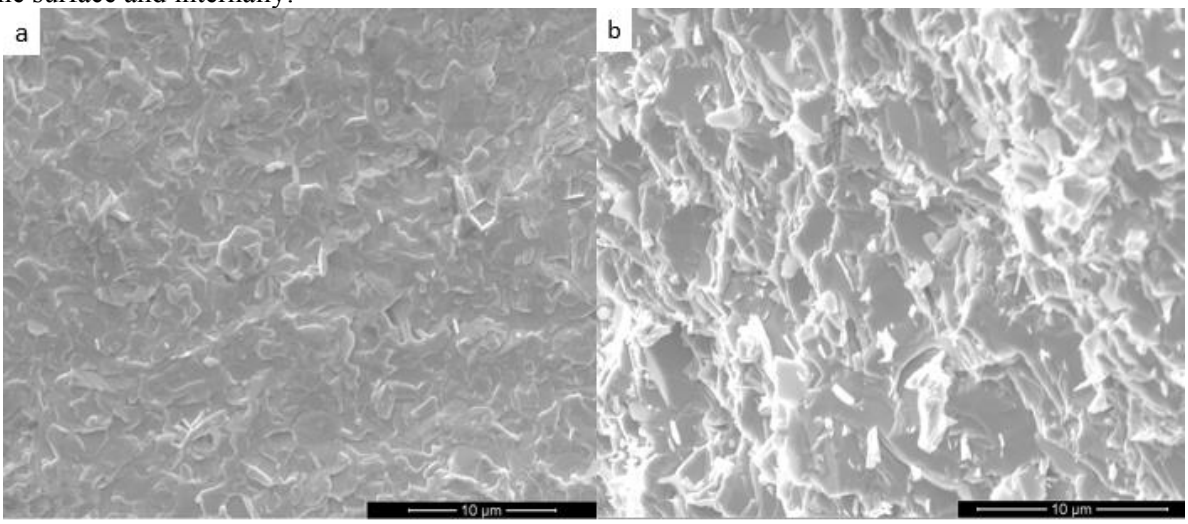

Figure 7. Micrograph images of an ETP core after a non-melt heat treatment at 879 C. a) The top surface of the core shows excellent interconnectivity of grains and suggests preservation of texture. b) Surface after breaking the ETP bar showing internal interconnectivity and suggests preservation of texture.

6. Conclusion

Supercurrent transport has been achieved in ETP cores at LN temperatures while maintaining texture, but still no evidence of superconductivity in ETP multifilament wires. The lower than expected critical temperature may explain the poor behavior of the samples that did carry supercurrent, it also shows that further study on the oxygen doping during the non-melt heat treatment may be required to produce a superconducting ETP multifilament wire.

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