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TEXTURED-POWDER BI-2212/AG WIRE TECHNOLOGY DEVELOPMENT*

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Abstract

Progress is reported in developing of textured-powder Bi-2212 cores as a new approach to Bi-2212/Ag wire technology. The process builds upon earlier work in which Bi-2212 fine powder can be highly textured in its a-b plane orientation and fabricated into square-cross-section bars. The current work concerns an Enhanced Textured Powder (ETP) process, in which silver nanopowder is homogeneously mixed with the Bi-2212 powder. We report studies of the effect of the addition on the phase dynamics near melt temperature. ETP cores are being prepared for compounding into a billet to fabricate multi-filament wire.

STATE-OF-ART: OPIT BI-2212 WIRE

Bi-2212 is the only one of the high-temperature superconductors that has been successfully fabricated into multi-filament round wire. The state-of-art fabrication route is oxide-powder-in-tube (OPIT), in which fine-powder ~phase-pure Bi-2212 is loaded into Ag or Ag alloy tubes, the tubes are drawn, stacked, and re-drawn to make multi-filament wire [1]. The wire is then cabled and wound into final-form windings, and the windings are subjected to a 'partial melt' (PM) heat treatment at ~887 C during which the Bi-2212 solid particles are fully melted and then gradually re-crystallized. Several properties of this PM heat treatment lead to difficulties and until recently limited engineering current density to je~200 A/mm^2:

- The void space among particles in the powder becomes bubbles in the liquid (porosity), and capillary forces coalesce the bubbles to form macro-voids that can block current transport.
- The liquid is chemically aggressive, and etches along grain boundaries in the silver matrix. This creates dendritic bridging among filaments (good and bad).
- Connectivity among grains remains a limit to performance.

A dramatic improvement in performance to je~800 A/mm^2 was obtained last year using overpressure processing at ~5-10 MPa during the formation heat treatment [2,3]. However, overpressure processing remains a challenging proposition for long dipoles of a future hadron collider.

TEXTURED POWDER BI-2212

We began several years ago to develop an alternative method for fabricating the Bi-2212 cores for wire, in which fine-powder Bi-2212 was uniaxially pressed to form square-cross-section rods [4]. The pressing aligns the orientation of the a-b planes in the micaceous powder to a remarkable degree. The motivation of the development is to achieve connectivity among textured grains of Bi-2212 within a core using solid-phase diffusion at a temperature slightly below melt. Conductivity is 100x greater in the a-b plane than in the c-axis, In a non-melt heat treatment the transition to liquid is never made and the favorable texture is preserved.

The procedure is summarized in the photos of Fig. 1: fine-powder Bi-2212 is loaded into the 4x150 mm^2 aperture of a female forming die. The male die is inserted into the aperture above the powder, the die assembly is loaded into a hydraulic press, and the powder is compressed by 200 MPa. Bi-2212 is micaceous, and therefore the powder particles are flat flakes, and they naturally align when compressed to yield ~80 % texture as measured using X-ray diffraction [4].

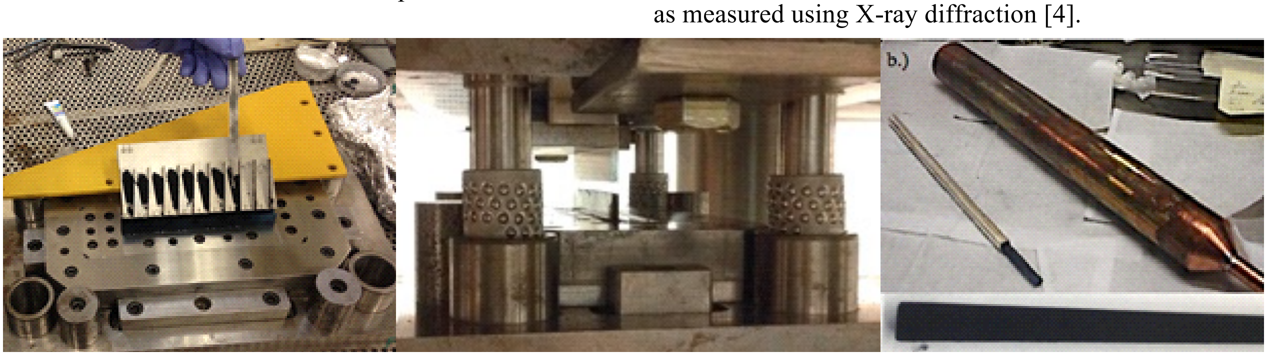

Figure 1: Fabrication of square-cross-section textured-powder Bi-2212 core a) load fine-powder Bi-2212 into rectangular die in oxygen atmosphere; b) compress to 200 MPa; insert bar into square hole in silver tube.

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The Ag-sheathed monocores were extruded and drawn to fine wire. The textured-powder (TP) cores drew extremely well – no sausaging or breaks – down to ~0.4 mm wire diameter. The drawing properties are interpreted to result from the ability of textured micaceous powder to slip easily in drawing. A green-state density ~80 % was attained in the sub-element cores. A heat treatment development was undertaken to find a domain that would produce connectivity. Remarkable growth of the a-b planes of all grains was observed, as shown in Fig. 2. But there was little evidence of grains growing into one another (by twinning and twisting), so that the weak links among grains persisted. Transport measurements were made on monocore wire samples, and no significant superconducting transport was observed [4]. Even though the TP process produced aligned grains and excellent density, connectivity was not achieved.

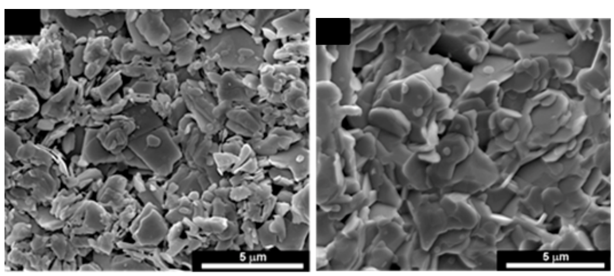

Figure 2: SEM micrographs of TP cores before and after non-melt heat treatment at 879 C for 2 hours.

NANO-AG-ENHANCED TP CORES

It has long been realized that the interface between silver and Bi-2212 strongly affects the properties of the Bi-2212 at or near melt temperature [5]. The melt temperature is lowered at the interface, when the temperature is lowered from melt recrystallization tends to align along the Ag interface, and in magnetic imaging studies in conventionally processed OPIT wire the preponderance of current flows near the Ag interface.

We conjectured that perhaps this interface effect could be enhanced if the silver were dispersed as a nanopowder homogeneously throughout the Bi-2212 fine powder of a core. In this way the Ag/Bi-2212 interface is everywhere in the core, and its benefits might be realized throughout. A process has been developed to disperse nano-Ag in fine-powder Bi-2212 and use the mixture to form enhanced textured-powder (ETP) cores. We report studies of the microstructure in green-state cores and in cores that have been heat-treated at temperatures above and below melt temperature.

To disperse a nanopowder in a 1 μm-grain-size powder is not easy. Electrostatic charging causes a propensity for aggregation, both of the Bi-2212 powder and of the Ag nanopowder. Homogeneous dispersion was achieved using high-energy acoustic mixing. Initial studies of microstructure and heat treat studies were made using a pellet press to compress 2 cm diameter round pellets of the powder mixture.

The ETP pellets were heat treated with a temperature profile similar to Ref. [6]. Samples were heat treated with several values of maximum temperature T_{max} and dwell time at T_{max} with and without a post-treatment anneal at 845 C for 48 h, as summarized in Table 1. The microstructure of samples was studied using scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS). At a maximum temperature in the range 876 C and 879 C (just below melt temperature) interconnectivity was good enough that distinguishing between individual grains was difficult. At 880 C the sample melted and alkaline earth cuprates (AEC) and copper free (CuF) regions were visible, (Fig. 3). Eliminating the annealing of 845 C for 48 hours was also tested. It was found that when annealing was eliminated parasitic phases became common, see Fig. 4a. However, no parasitic phases have been seen when annealing is included as part of the heat treatment.

Table 1: Overview of Figures Shown

Fig	T_{max} (C)	Duration (hrs)	Annealed	Parasitic phases	AEC, CF
3a	878	48	Yes	No	No
3b	881	0.5	No	No	Yes
4a	876	6	No	Yes	No
4b	876	6	Yes	No	No

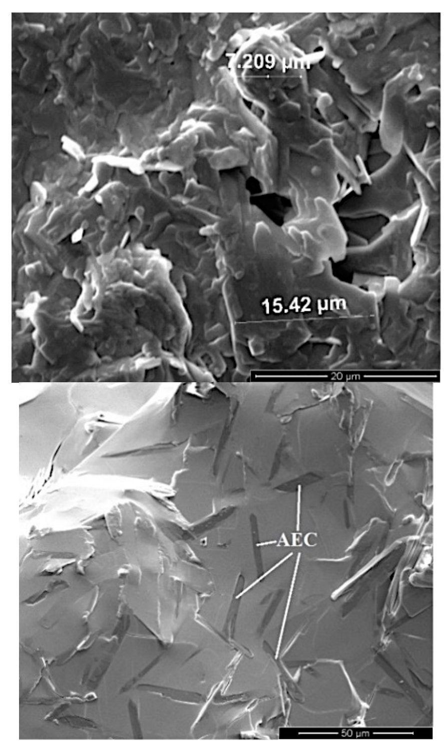

Figure 3: a) T_{max} = 878 C, not melted; b) T_{max} = 881 C, melted.

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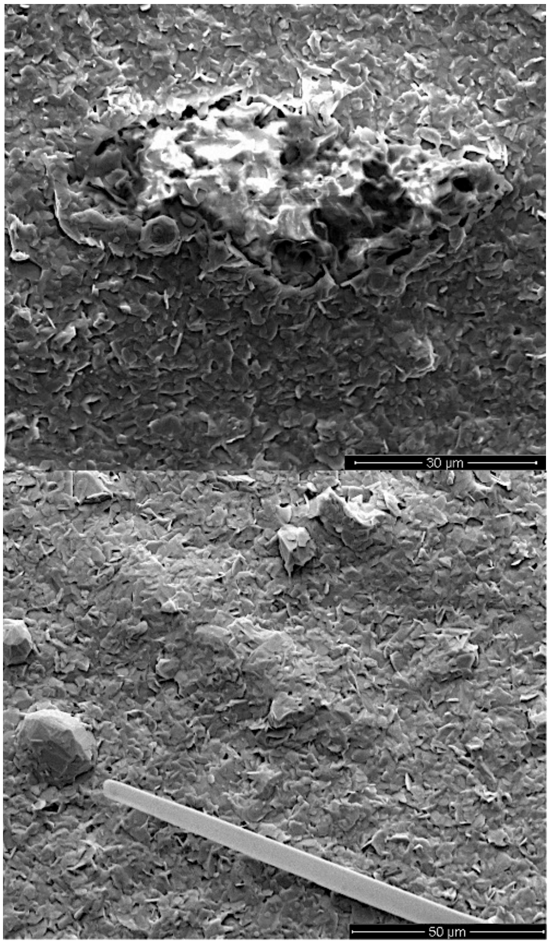

Figure 4: ETP cores heat treated at T_{max} =876 C: a) no annealing, parasitic phase visible; b) annealed, phase-pure Bi-2212.

Indeed the spire in Fig. 3b is a perfect single crystal of Bi-2212 that grew spontaneously on the surface of the sample. The globules visible on the surface are pure Ag, which appears to diffuse from the bulk and deposit on the surface during heat treatment.

A resistance measurement on an ETP pellet was done in a Physical Property Measurement System (PPMS) (see Fig. 5). A four point silver tab configuration was pressed into a green-state ETP pellet. The pellet and tabs were heat treated with a maximum temperature of 878 C. Probes were connected to the silver tabs and resistance was measured as a function of temperature at several values of magnetic field. Macroscopic supercurrent transport was achieved over ~1 cm scale. The pellet that was tested was not annealed and therefore includes parasitic phases.

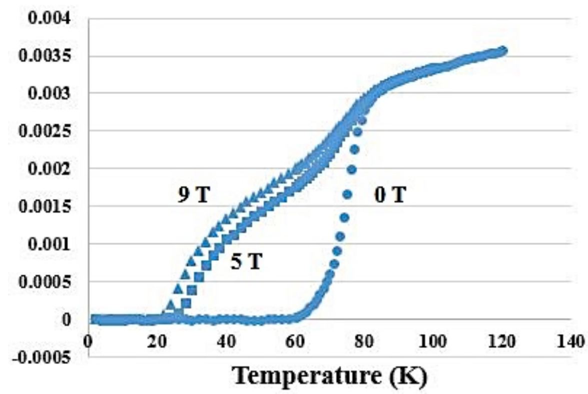

Figure 5: Resistance measurement of ETP pellet at 0 T, 5 T, and 9 T.

FUTURE WORK

ETP cores are being prepared similar to the TP cores described above. Four ETP cores will be made using two sizes and two weight concentrations of silver nanopowder, see Table 2. The cores will be loaded end to end into a silver alloy tube and drawn down to a monofilament wire < 1mm in diameter. The wires will be segmented, wound on spools, and heat treated according the results found in studying the pellet microstructure. The current transport properties of each spooled wire will be tested. Finally select wires will be dissected and analysed using SEM and EDS similar to the pellet analysis.

Table 2: Bi-2212/Ag Mixture Compositions

Mixture	Ag / Weight %	Ag particle size
Large 3%	3%	80-100 nm
Large 5%	5%	80-100 nm
Small 3%	3%	20 nm
Small 5%	5%	20 nm

A 2 x 2 configuration of ETP cores will be prepared in a similar way described above for the monofilament wire. However, this configuration will be drawn and restacked to produce a multifilament wire to be heat treated and tested as the monofilament wire will be.

CONCLUSION

Samples in which silver nanopowder is homogeneously mixed with Bi-2212 fine powder exhibit excellent grain growth and interconnectivity during a non-melt heat treatment, and a pellet specimen supports macroscopic supercurrent transport without melting the Bi-2212. No parasitic phases have been found in samples that were annealed.

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