

Effect of powder pre-annealing on microstructural development inside Bi2223/Ag-sheathed tapes

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The fabrication of $(\text{Bi,Pb})_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$ (Bi2223)/Ag tapes with appropriately oriented microstructure and capable of carrying high currents at the boiling temperature of nitrogen is of great interest for the development of practical devices [1, 2]. Processing Bi2223/Ag superconducting composite tapes with cross sections of approximately $3 \times 0.2 \text{ mm}^2$ requires several steps including two or three heat treatments at 830–840 °C with intermediate mechanical densification. Of these steps, the first heat treatment plays a key role. The powder, which is initially packed into Ag-tubes of a few mm diameter, does not consist of the Bi2223 phase yet. Instead, a structurally related compound $(\text{Bi,Pb})_2\text{Sr}_2\text{Ca}_1\text{Cu}_2\text{O}_8$ (Bi2212) is the predominant phase. The Pb included in the nominal composition can be present in various forms depending on the details of the powder preparation process: (a) in the Ca_2PbO_4 phase, (b) in the $\text{Pb}_3\text{Sr}_{2.5}\text{Bi}_{0.5}\text{Ca}_2\text{CuO}_y$ (“3321”) phase, (c) incorporated in Bi2212. The powder preparation process determines the phase assembly, which in turn has an influence on the phase and microstructure development inside the tapes during further processing. Three tapes were prepared with powders corresponding to cases a, b, and c, as described above, in order to study the possible influence of the Pb location in the starting mixture. The properties studied include the microstructure, in particular the preferential orientation induced in the ceramic powder during deformation of the Ag sheath from a tube to a tape with high aspect ratio, as well as its development during the first heat treatment.

Besides being a non-destructive method in contrast to conventional X-ray diffraction that requires the removal of the Ag sheath, high energy ($\approx 100 \text{ keV}$) synchrotron radiation also provides information on the whole ceramic volume instead of probing a few micrometers of material only. Furthermore, *in-situ* studies of the phase transformation may be performed at elevated temperatures. The measurements were performed in transmission geometry on beamline BW5. High temperature investigations were performed between 500 and 830 °C in a specially designed furnace. Details about the experimental setup and data analysis can be found in [3].

Due to its strongly anisotropic crystal structure, the Bi2212 crystallites have a plate-like shape with high as-

pect ratio. The mechanical deformation leading to flat tapes naturally tends to result in a *c*-axis texture in these plates. The evolution of the Bi2212 phase texture during the first heat treatment is plotted in Fig. 1 for the three samples. It clearly appears that the deformation-induced texture (initial value) is significantly higher in the tapes containing Pb-poor Bi2212 (cases a and b) than in the one including heavily Pb-doped Bi2212 (case c). This feature can be understood by considering the structural differences induced by Pb substitution in the Bi2212 structure. The structure of all high-temperature superconducting Bi-based cuprates is characterized by the presence of two adjacent BiO 2-dimensional layers. Due to the presence of a lone pair of electrons on the Bi^{3+} ions, the bonding is very weak between the BiO layers and Bi2212 crystals can be easily cleaved [4]. As a consequence, during the mechanical deformation of tapes, the Bi2212 platelets are expected to be broken mainly at the BiO–BiO interface, thus keeping a high aspect ratio and sliding mostly along the deformation direction as long as the plates are not too thin to withstand the stresses acting perpendicularly to the atomic layers.

The situation is completely different in Pb-doped Bi2212 plates. Replacing part of the Bi^{3+} by Pb^{2+} results in a modification of the electronic configuration at the Bi(Pb)O–Bi(Pb)O interface and the Pb-doped Bi2212 crystals cannot be cleaved as easily as the Pb-free crystals. This is expected to result in an enhanced tendency to break the Bi2212 plates perpendicular to the Bi(Pb)O layers. The stresses acting perpendicular to the plates will thus play a more important role in breaking the plates during deformation. As a result, the average texture is actually expected to be worse in this case.

During the temperature ramp-up, a clear decrease of the full width at half maximum (FWHM) occurs from about 700 °C. This phenomenon is related to the extended 2-dimensional grain growth that was reported to start between 600 and 750 °C [5]. During the dwell at 830 °C, the texture of the Bi2212 phase slightly improves, while the amount of this phase progressively decreases as a result of the reactions leading to the formation of the Bi2223 phase. Nevertheless, after 10 hrs

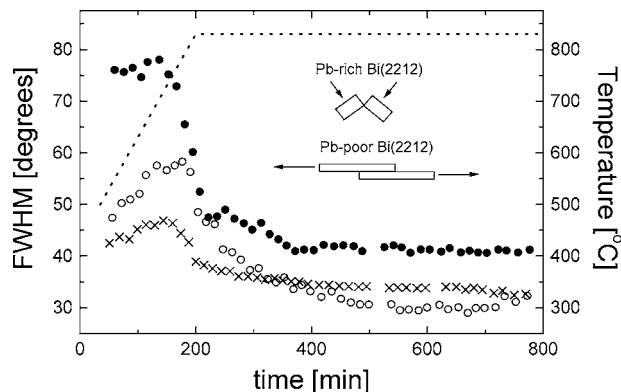


Figure 1 Evolution of the average texture of Bi2212 in the samples prepared with powders where Pb is present as: Ca_2PbO_4 (X), $\text{Pb}_3\text{Sr}_{2.5}\text{Bi}_{0.5}\text{Ca}_2\text{CuO}_y$ (O), incorporated in Bi2212 (●). The drawings illustrate the main breakage mechanism at play during initial mechanical processing as described in the text.

sintering, the texture of the initially Pb-doped Bi2212 phase is still far from the one achieved in the tapes prepared with Pb-poor Bi2212. Due to geometrical constraints, the Bi2223 phase, which also exists as plate-shaped grains with a high aspect ratio, is formed with a preferential orientation that is closely related to the one of the Bi2212 phase [6]. Since the texture of Bi2223 has a significant influence on the superconducting current transport capability of the tapes, it is essential to start the process with a well textured Bi2212 phase. The implication of the present finding is that starting

from a precursor powder including Pb-doped Bi2212 is likely to yield poorer performances unless the deformation process is modified in order to take into account its peculiar mechanical characteristics.

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