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Deformation-induced texture in cold-rolled Ag sheathed Bi(2223) tapes

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Abstract

We have found that the Bi(2212) grains in unreacted Ag/Bi(2223) tapes at the end of the cold-rolling process show already a high degree of texture. X-ray diffraction analysis and current transport measurements at 77 K in magnetic fields up to 0.5 T have been performed before and after reaction on tapes with thicknesses in the range from 75 to 286 μm (filament thicknesses between 30 and 115 μm). The critical current density j_c increases for smaller tape thicknesses, a maximum being found for a tape thickness of about 90 μm, below which sausaging effects are observed. The mean misalignment angles of Bi(2223) grains in these tapes have been determined by angle-dependent transport critical current data at 77 K. We have found that the main reason for the enhancement of j_c for thin tapes is due to the higher average texture of the Bi(2223) grains when compared to thick tapes. This is a direct consequence of the high degree of texture of Bi(2212) grains after the cold-rolling process, which has been observed here on 90 μm thick tapes with j_c(77 K, 0 T) reaching between 25 and 30 kA/cm^2.

1. Introduction

Ag sheathed Bi(2223) tapes with high transport critical current densities are commonly prepared by the powder in tube method (PIT method) [1]. Reacted powders of various nominal compositions (mainly composed by the Bi(2212) phase) [2–5] are filled inside pure or reinforced Ag tubes [6], which are then cold deformed in order to obtain a tape-like shape. These tapes are finally heat treated with some intermediate densification steps in order to form a textured Bi(2223) filament.

Typically, in optimized tapes Bi(2223) grains show mean misalignment angles of about 7–10° with respect to the tape plane, as can be observed by the SEM investigation of the filament microstructure, and by anisotropy measurements of the critical current density in an applied magnetic field [7].

Mono- and multifilamentary tapes with j_c values at the liquid-nitrogen temperature of ≥ 30 kA/cm^2 have been prepared by the PIT method with an industrially scalable process [8–10]. Local j_c measurements on high-quality cold-rolled tapes have revealed that the transport properties of long samples can be further improved, a maximum local j_c(77 K, 0 T) value of about 46 kA/cm^2 being measured at the tape sides [11].

It is well known that the cold deformation process

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during the tape fabrication is crucial in order to obtain a smooth Ag–oxide interface with reduced sausaging [12]. Moreover, the high powder density inside the Ag sheath induced by the cold deformation has been recently correlated with the transport properties of the tapes [13,14].

In this work, we have analyzed the effect of cold deformation on the texture of the Bi(2212) grains in the unreacted (or green) tapes before the formation of Bi(2223). By direct X-ray diffraction measurements, we have found that cold deformation induces a relevant texture of the Bi(2212) grains in the tape plane, helping the subsequent formation of well textured Bi(2223) grains. The texture of the Bi(2223) grains in reacted tapes of various thicknesses has been analyzed by both X-ray diffraction analysis and transport critical current density measurements. In particular, by X-ray diffraction it has been found that the Bi(2223) grains at the Ag–filament interface are well aligned regardless to the tape thickness (in accordance with Refs. [15] and [16]), while by transport measurements it has been found that the average texture of the grains which really contribute to the current transport is strongly thickness dependent, the anisotropy being lower in thicker tapes.

2. Experimental details

Ag sheathed Bi(2223) tapes have been prepared by the PIT method. Coprecipitated precursor powders with nominal cation ratio Bi : Pb : Sr : Ca : Cu =

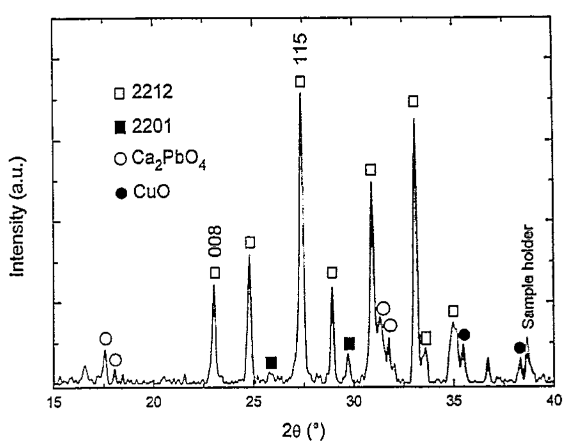

Fig. 1. X-ray diffraction pattern of the prereacted powders; Bi(2212) is the main phase.

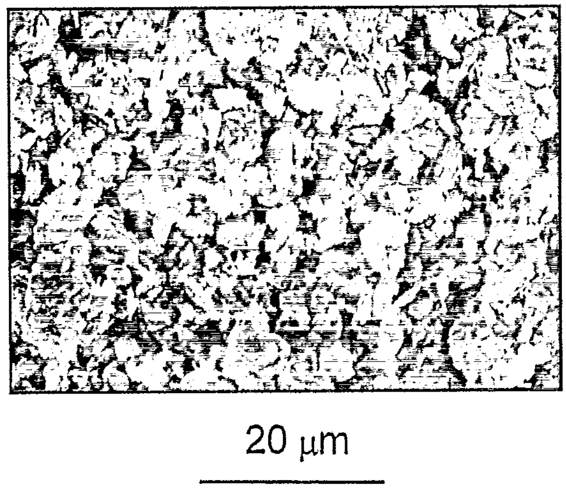

Fig. 2. SEM observation of the prereacted powders for the determination of the grain size.

1.72 : 0.34 : 1.83 : 1.97 : 3.13 have been used. After the short treatment at moderate temperature (< 500°C), needed to take off the organic impurities and water, the powders have been calcined at a temperature of 820°C in air for about 50 h with several intermediate grindings. As shown by the X-ray diffraction pattern (Fig. 1), the calcined powder is mainly composed of Pb free Bi(2212), Ca_2PbO_4, CuO and Bi(2201). The grain size has been evaluated by SEM observation (Fig. 2), and is found to be typically 1 to 5 μm. This powder is filled inside pure Ag tubes and densified by pressing with a piston to reach an effective powder density of about 70% of the theoretical density ($\rho_{th} = 6.6$ g/cm^3). The Ag tubes are cold deformed by swaging and drawing to an external wire diameter of about 1–2 mm. Finally these wires are rolled in several steps with cylinders of 105 mm in diameter to obtain the tape-like shape. The tape thickness has been reduced by about 10% for each step. The tapes are finally heat treated at a temperature of 830–840°C in air for about 200 h, with up to three intermediate rolling densification steps which are needed to improve the connections between the grains.

The orientation of the Bi(2212) and Bi(2223) grains in the filament of unreacted and reacted tapes have been studied by X-ray diffraction analysis, using Cu Kα radiation with a Ni filter. The j_c values have been measured by the standard four-probe technique using the criterion of 1 μV/cm. To avoid the sample heating, the measurements have been performed by directly immersing the sample in liquid nitrogen. The field dependence and anisotropy of j_c have been measured in a field of up to 0.5 T.

3. Results

3.1. Texture of Bi(2212) in unreacted tapes

The texture of Bi(2212) grains has been investigated by direct X-ray diffraction analysis of the filament surface of green tapes, for which the silver sheath has been mechanically removed after cutting the tape sides. Due to the large amount of cold working, the grains in the green tapes are very small in size and it is sometimes impossible to distinguish the peaks of the Bi(2212) phase from the background [1]. However (as for example in Ref. [17]), it may be possible to detect the peaks of the Bi(2212) phase, as shown in Fig. 3 for a green tape with a filament thickness of about 100 μm (overall thickness of 245 μm). The intensity of the (115) peak, when compared to the calcined powders (Fig. 1), is strongly reduced. In order to have quantitative information about the texture of the Bi(2212) phase, we have defined a texture parameter t as

$$t = \frac{L(115)}{L(115) + L(008)},$$

where $L(115)$ and $L(008)$ are the integrals of the (115) and (008) peaks of the Bi(2212), respectively.

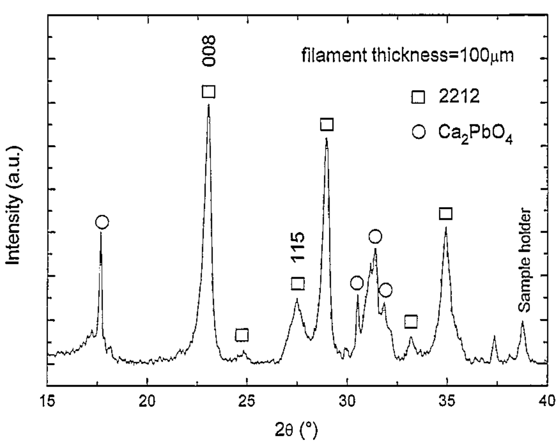

Fig. 3. X-ray diffraction pattern of the unreacted tape surface after mechanical remotion of the Ag sheath.

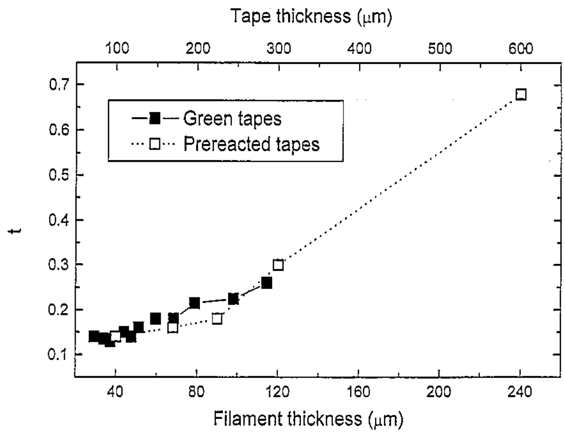

Fig. 4. Bi(2212) texture parameter t of unreacted and pre-reacted tapes as a function of the tape thickness.

For the powders, the t value lies typically between 2 to 4, while for the green tape of Fig. 3 we have found $t = 0.23$.

We have analyzed green tapes of different filament thicknesses between 30 μm and 115 μm (overall thicknesses between 75 and 286 μm). The filament thickness is about 40% of the overall thickness of the tape. For each tape the t parameter has been evaluated and the complete set of data is presented in Fig. 4. As a result one finds that the t value decreases with the thickness of the filament, from $t = 0.23$ at 100 μm to $t = 0.14$ at 50 μm, remaining nearly constant for lower thicknesses.

We tried to extend this technique to thicker tapes in order to study the evolution of the texture during the global cold-rolling process. However, thicker tapes are generally narrower, and it is no more possible to cut the sides, take off the silver sheath, and measure the texture of the narrow central slice, also because the filament crumbles very easily.

To overcome these technical problems, we have decided to heat treat the tapes for a short time (≅ 1 h) at a temperature of about 780°C before the X-ray diffraction measurements. A remarkable effect of this heat treatment on the tapes is the growth of Bi(2212) grains, which gives better-defined peaks in the X-ray patterns, as already reported by Grivel et al. [18]. A second effect of this heat treatment is the enhancement of the filament strength. It was thus possible to completely etch away the silver sheath, so that it was no longer necessary to cut the tape sides and the measurements can be extended to

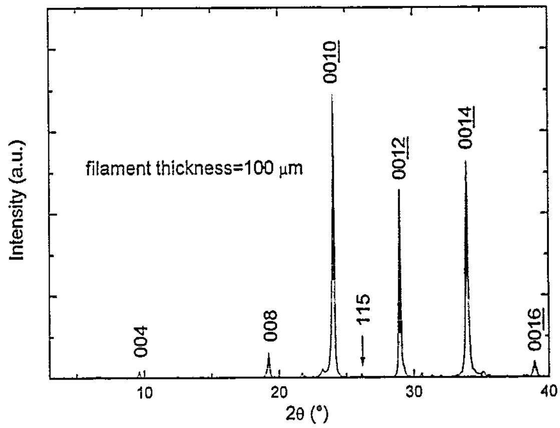

Fig. 5. X-ray diffraction pattern of the reacted Bi(2223) tape surface.

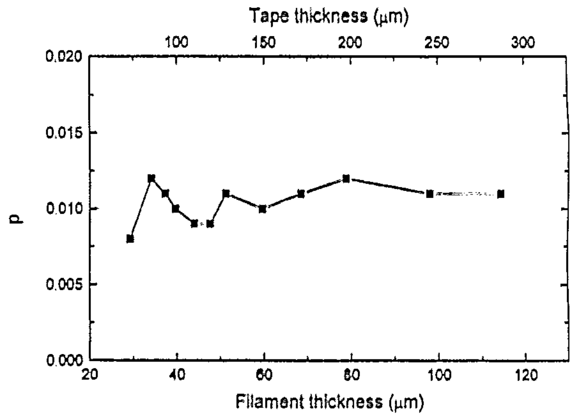

Fig. 6. Bi(2223) texture parameter p as a function of the tape thickness.

higher thicknesses. The Ag etching was performed with a proper mixture of ammonia water and potassium cyanide (see Ref. [19] for more details).

The results of the measurements after prereaction are shown in Fig. 4, for tapes which have been cold rolled using a thickness reduction of about 20% for each step. A strong texturing effect of the Bi(2212) phase has been found during cold rolling, the data for low thicknesses being in quantitative accordance with those measured on green tapes.

3.2. Texture of Bi(2223) in heat treated tapes

We have heat treated tapes with various thicknesses at a temperature of 830–840°C in order to form the Bi(2223) phase. After 200 h of treatment with several intermediate cold-rolling steps, we have reached the maximum j_c values, and we measured the X-ray diffraction patterns of the tape surfaces after mechanically taking off the silver sheath. In Fig. 5, the pattern is reported for a 100 μm thick filament. As already shown elsewhere [20], the Bi(2223) grains are very well textured, in particular at the interface between Ag and the filament. Again, we have defined a Bi(2223) texture parameter p as

$$p=\frac{H(115)}{H(115)+H(00\underline{10})},$$

where $H(115)$ and $H(00\underline{10})$ are the integrals of the (115) and $(00\underline{10})$ peaks of the Bi(2223) phase, respectively. The degree of texture of the Bi(2223) phase as a result is found to be much higher than the texture of the Bi(2212) phase in the green tapes. Moreover, no clear evidence of a thickness dependence of the texture parameter p has been detected (Fig. 6), suggesting that a very well textured Bi(2223) layer grows at the Ag–filament interface regardless of the texture of the precursor Bi(2212) grains.

3.3. Transport properties

We have measured the transport critical current density at liquid-nitrogen temperature of cold-rolled tapes of various filament thicknesses between 30 and 115 μm. The results are shown in Fig. 7. A strong thickness dependence has been observed, a maximum being found for a thickness of about 40 μm.

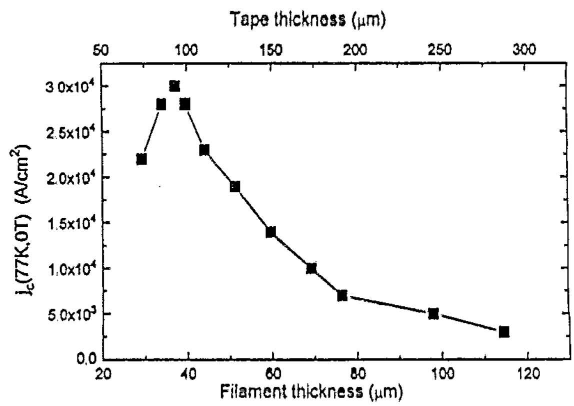

Fig. 7. Transport critical current density at 77 K as a function of the tape thickness.

The drop at low thicknesses has already been attributed to an increase of sausaging of the filament section [21]. The origin of the drop of j_c for thick tapes seems to be more complex, the lower powder density [13,14] being one suggested explanation.

We have performed magnetic-field dependence and anisotropy measurements of j_c at liquid-nitrogen temperature in order to evaluate the mean misalignment angle ϕ_e of the Bi(2223) grains which contribute to the current transport as a function of the filament thickness. The normalized field dependence of j_c has been plotted in Fig. 8 for three filaments of 50, 70 and 100 μm thickness (128, 173 and 245 μm thick tapes). It is clear from Fig. 8 that a thicker filament shows a stronger field dependence for the $B \perp c$ orientation, while for the $B \parallel c$ orientation the difference is less evident. Moreover, the j_c anisotropy is clearly lower in the 100 μm thick filament. From complete anisotropy measurements of j_c, we have determined the effective transport mean misalignment angles ϕ_e with the technique described by Hensel et al. [20] in the case of Bi(2223) tapes. According to these authors, a very efficient way to analyze the anisotropy consists of the representation

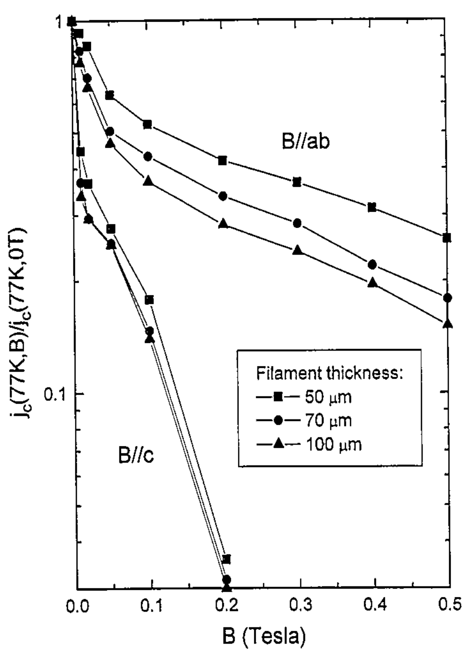

Fig. 8. Normalized magnetic-field dependence of transport j_c at 77 K for 50, 70 and 100 μm thick filaments and for both $B \parallel c$ and $B \perp c$ orientation.

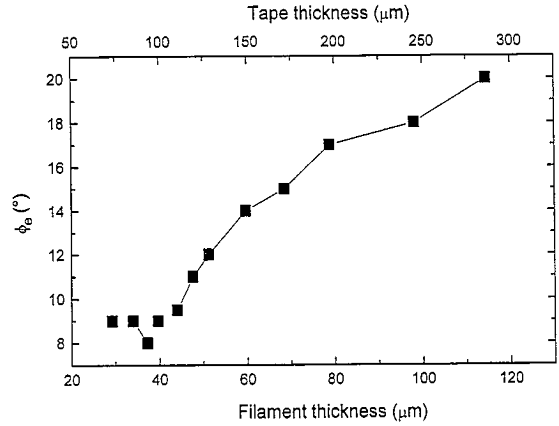

Fig. 9. Tape-thickness dependence of the effective transport mean misalignment angle ϕ_e.

of j_c as a function of $B_c = B\cos(\Theta)$, where Θ is the angle between the orientation of B and I. A plot of $j_c(B, \Theta)$ results in a master curve corresponding to $j_c(B, \Theta = 0)$ [20,22–23]. If $\Theta \cong 90°$, a plateau-like behavior is observed, due to the wide distribution of Bi(2223) grain orientations. The effective misalignment angle ϕ_e of the grains can be defined by $\cos(90° - \phi_e) = \cos(\phi_e) = B_c^*/B$, where B_c^* is given by the intersection of the plateau value with the exponential decay curve [20,22–23]. The effective transport misalignment angles are presented in Fig. 9, where ϕ_e has been plotted as a function of the tape thickness. We have found that ϕ_e is higher for thick tapes, in accordance with the $j_c(B)$ measurements shown in Fig. 8. For thin filaments (below 45 μm thick), the ϕ_e value of about 8–9° is in agreement with the data we have already published for high-j_c tapes [22].

4. Discussion

From X-ray diffraction analysis of the unreacted tape filaments it has been found that a relevant texture of the precursor Bi(2212) grains is induced as a consequence of the tape-fabrication process, at least at the filament surface. In particular, the cold-rolling process greatly enhances this Bi(2212) texture in the tape plane when the filament thickness is reduced down to about 40 μm. For filaments which are thinner than 40 μm, the texture of the Bi(2212) grains is almost constant, probably due to the increasing sausaging of the filament section.

The texture of the Bi(2223) grains in the reacted tapes has been analyzed in tapes of varying thicknesses, in order to determine if it is possible to establish a correlation with the texture of the precursor Bi(2212) grains. For this purpose, two common techniques for the determination of the texture have been used: direct X-ray diffraction analysis of the filament surface and calculations of the effective mean misalignment angles from anisotropy measurements of j_c in applied magnetic field. The results are presented in Figs. 6 and 9, and they are in apparent contradiction, in particular because the X-ray diffraction patterns are almost thickness independent. However, it is clear that with the two techniques we measured the texture of different Bi(2223) grains of the filament. In fact, due to the limited penetration length of the X-rays in the filament (the theoretical X-rays penetration length in ideal Bi(2223) crystals is of the order of 10 μm for a θ–2θ scan and for low angles ($\theta < 20°$)), mainly information from a relatively thin layer near the surface is obtained, especially at low diffraction angles. The X-ray diffraction results on reacted tapes demonstrate that at least a thin Bi(2223) layer at the Ag interface grows very well oriented regardless of the filament thickness and of the texture of the precursor Bi(2212) grains. On the other hand, the effective transport misalignment angle measurements of tapes with different thicknesses show that the average texture of the Bi(2223) grains which are on the current paths is generally lower than the texture of the Bi(2223) grains at the Ag interface. Indeed, as shown by Hensel et al. [24], who measured the critical current perpendicular to the tape surface for Bi(2223) tapes with $j_c > 25\,000$ A/cm^2, the contribution of the central part of the oxide layer to I_c is far from being negligible.

The apparent contradiction between the behavior of Figs. 6 and 9 can be explained by a kinetic growth model, by Flükiger et al. [25]. According to this model, the pressure exerted by the growing grains on their neighbors leads to a higher degree of texturing for the grains being situated at the Ag interface, while the degree of texturing decreases towards the center of the oxide layer.

5. Conclusions

Ag sheathed Bi(2223) tapes have been prepared by the PIT method with various filament thicknesses between 30 and 115 μm. X-ray diffraction analysis, microstructure investigation and transport-property measurements have been performed on both reacted and unreacted tapes in order to evaluate the grain texture. The results can be summarized as follows:

(1) the cold deformation of the Ag tapes during the tape fabrication induces a relevant Bi(2212) texture in the tape plane, at the oxide/Ag interface. In particular, the Bi(2212) texture greatly improves when the tape thickness is reduced;

(2) after prolonged reaction heat treatments (200 h at 830–840°C), we have found that, at the tape surface, the Bi(2223) grains are much more textured than the precursor Bi(2212) grains. Moreover, by X-ray diffraction, almost no influence of the filament thickness has been found on the Bi(2223) texture at the oxide/Ag interface;

(3) the transport j_c of these cold-rolled tapes has been measured. A magnetic field of up to 0.5 T has been applied at various orientations. A strong j_c dependence on the filament thickness has been found for thicknesses down to 40 μm (below 40 μm, sausaging effects lead to a decrease of j_c, but this is not relevant to the present considerations). The enhancement of j_c with lower filament thicknesses has been imputed to the higher degree of texture of the Bi(2223) grains which are on the current paths: the mean misalignment angle ϕ_c deduced by our transport measurements increases as the tape thickness increases.

The present data reflect a gradual variation of the degree of texturing, of the Bi(2223) grains, which is highest at the Ag interface and lowest at the center. Since the degree of texturing of the external Bi(2223) grains does not depend on the tape thickness, and the highest j_c values are measured for the smallest rocking angles (from transport measurements) of the order of $\phi_c \cong 8$ to 9°, it follows that the optimization of j_c requires a maximum degree of texturing of the grains at the center. In the present work, we have shown that this is in turn correlated to a maximum degree of preorientation of the Bi(2212) grains as a consequence of the carefully chosen rolling parameters.

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