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Tape Development: Microstructure and Critical Current Densities

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ABSTRACT

The formation of the textured Bi,Pb(2223) phase in mono- and multifilamentary tapes is described as a result of several reactions, involving an intermediate phase, Bi,Pb(2212), which is formed as a solid solution of the initial phase Bi(2212), the latter having a monoclinic superstructure.

For Bi,Pb(2223) tapes, a direct correlation between the degree of texturing of the initial Bi(2212) grains after rolling (before reaction) and of the current carrying Bi,Pb(2223) platelets (after reaction) is shown. The difference between the critical current densities of pressed and rolled tapes is discussed by comparing the maximum applicable uniaxial pressure with the local pressure exerted by the rolls. The optimization of the complex preparation process leading to long, rolled Bi,Pb(2223) tapes is discussed. The lateral distribution of the transport critical current densities in monofilamentary Bi,Pb(2223) tapes with $j_c(77K, 0T) = 28'000 \text{ A/cm}^2$ shows a marked minimum at the centre and a maximum at the sides, the highest value reaching $54'000 \text{ A/cm}^2$. At 4.2K and 14T, the same tape reached the value $j_c = 75'000 \text{ A/cm}^2$.

For long (>1m), rolled multifilamentary Bi,Pb(2223) tapes with 7 and 19 cores, critical current densities up to $23'000 \text{ A/cm}^2$ have been obtained. The field dependence of j_c is essentially the same as for monofilamentary tapes.

The variation of the critical current density in Bi,Pb(2223) tapes as a function of B and T is presented and discussed within the « railway switch » model.

KEYWORDS: High T_c superconductors, critical current density, current distribution, crystal structure

INTRODUCTION

In spite of the extraordinary metallurgical complexity of the multinary system Bi-Pb-Sr-Ca-Cu-O in the region of the so-called Bi,Pb(2223) phase, a substantial progress has been achieved in the last years in producing long tapes with high critical current densities. It is remarkable that the highest critical current values for long tapes were obtained by industrial manufacturers, *e.g.* Sumitomo (Japan) or American Superconductors or IGC (USA): this shows the importance of an extremely precise control of several fabrication parameters, *e.g.* powder precursors, calcination processes, deformation procedures and reaction conditions. It appears that close control is only possible if a certain number of sophisticated techniques are available. Currently, the fabrication by the « Powder-In-Tube » technique has reached a relatively high level: high critical current density values have been reported for rolled tapes:

$j_c(77K, 0T) = 40'000 \text{ A/cm}^2$ for short lengths[1], and values close to $30'000 \text{ A/cm}^2$ for lengths reaching close to 100 m [2,3]. Recently, the value of $30'000 \text{ A/cm}^2$ at 77K, 0T was obtained in our laboratories for monofilamentary Bi,Pb(2223) tapes of > 0.5 m length [4,5].

We are still far from a complete understanding of these processes, but simple correlations have been established which contribute to an enhanced knowledge of the complex mechanism. Some of these correlations have been described in a recent review article [5], other correlations are more recent [6,7]. In addition, such fundamental elements as the crystal structure are still matter of interest.

CRYSTAL STRUCTURE OF Bi(2212) and Bi,Pb(2212)

There are several ways to start the reaction leading to Bi,Pb(2223): either with a precursor containing Bi(2212) or Bi,Pb(2212) [8,9]. However, we have found that this difference is of little importance for the reaction process: in the case of Bi,Pb(2212), the Pb diffuses out at temperatures $>400^{\circ}\text{C}$, so that the situation at $>800^{\circ}\text{C}$ is very similar in both cases [10]. The crystal structure of both phases has been recently refined by single crystal X-Ray diffractometry [11]: it follows from Fig. 1 that Bi,Pb(2212) can be considered as a solid solution of Bi(2212) because of the similar structures.

The « average » crystal structure of Bi(2212) is known to be orthorhombic. It has been found recently that this compound has a monoclinic, commensurable « superstructure » [11]. The enhanced precision in determining the local neighbors of each atom of the elementary cell with 135 atomic sites may be of importance for theoretical models describing the occurrence of superconductivity in these materials.

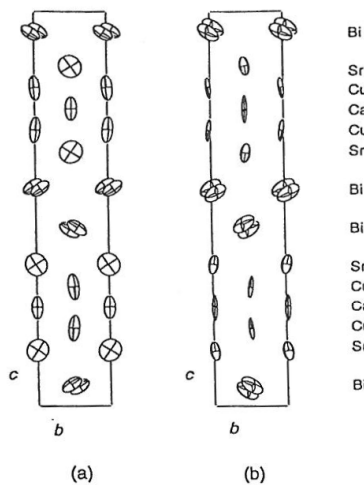


Fig. 1. Sections of the average structures a) Bi(2212) and b) Bi,Pb(2212) in projections along [1 0 0]. Ref. [11].

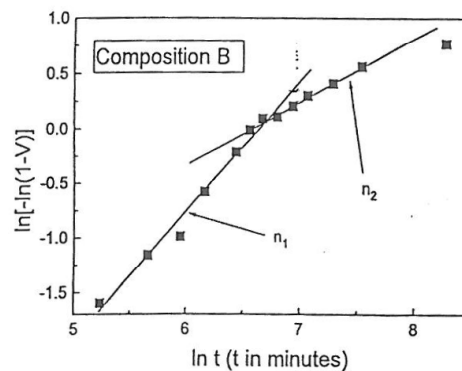


Fig. 2. Variation of $\ln[-\ln(1-V)]$ vs. $\ln(t)$ for pellets reacted at 857°C , where V is the volume of Bi,Pb(2223) phase [12].

THE REACTION KINETICS FROM Bi(2212) to Bi,Pb(2223)

The reaction from Bi(2212) to Bi,Pb(2223) can be described by a two-step reaction: it starts with a relatively rapid grain growth of Bi,Pb(2212)[10], which is completed within <30 hours. During this time, Pb atoms partially substitute the Bi atoms, while the ratio of the elements with respect to each other is thus of a different stoichiometry. The formation of Bi,Pb(2212) starts immediately after reaching the reaction temperature ($830 - 840^{\circ}\text{C}$ inside a Ag sheath), as can be deduced from the change

of the lattice parameters measured on samples heated a few minutes only. During the first 10 hours of reaction, a growth of the Bi,Pb(2212) grains from ~ 1 to $> 30 \mu\text{m}$ is observed. As a consequence of the Bi,Pb(2212) grain growth, the amount of liquid is continuously decreasing, which was confirmed by performing DTA measurements of the tapes after various reaction times at 840°C [12].

The first traces of Bi,Pb(2223), observed by X-Ray diffraction, are only observed after a few hours. This period corresponds to the time needed for the Bi,Pb(2212) phase to reach a certain critical composition of the solid solution, above which it becomes unstable.

The further transformation to Bi,Pb(2223) still contains some unresolved points, and several models have been advanced how to get from a two-layer to a three-layer configuration, but none of them gives a satisfactory picture. A detailed TEM analysis is needed in order to solve this problem.

We have recently studied the kinetics of the Bi,Pb(2223) phase formation by plotting the variation of the exponent n of the Avrami equation for isothermal transformations as a function of time. For a series of different compositions, a kink in the curve $\ln[-\ln(1-V)]$ vs. $\ln(t)$ was found (Fig. 2) after approximately 12 hours of reaction [12] (V is the volume fraction of the Bi,Pb(2223) phase as determined from X-Ray diffraction). The value $n_1 = 1.5$ in Fig. 2 for times < 12 hours is interpreted as the formation and beginning of growth of Bi,Pb(2223) crystallites from the germ nuclei formed at the beginning of the process. The value $n_2 = 0.85$ after $t > 12$ hours reflects the Bi,Pb(2223) grain growth (a value of $n = 1$ would correspond to a pure 2D growth). The formation of grain boundaries - the decisive mechanism for the critical current densities - occurs well after 12 hours.

CRITICAL CURRENT DENSITIES

The variation of j_c vs. B at 77K for Bi,Pb(2223) tapes is shown in fig 3 for a monofilamentary tape of 0.5 m length [4].

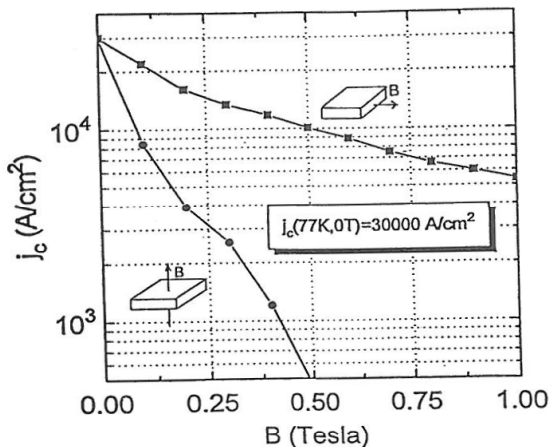


Fig.3. j_c vs. B at 77K for a rolled Bi,Pb(2223) tape.

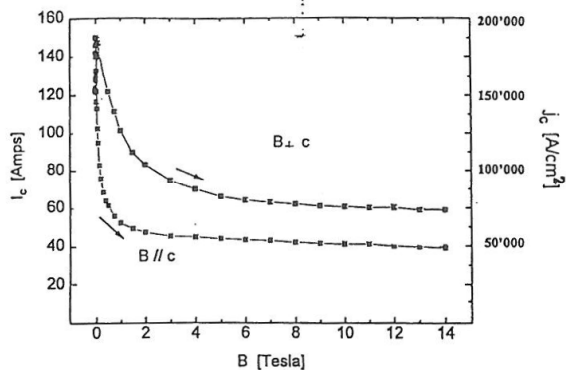


Fig.4. j_c vs. B at 4.2K for the same tape as in Fig.3.

The anisotropy as well as the field dependence are not essentially different from those of ref.1, with $j_c(77\text{K}, 0\text{T}) = 40'000 \text{ A/cm}^2$. This indicates that the essential difference between these tapes is mainly due to a larger number of strongly coupled grain boundaries in the tape with higher j_c , the current carrying mechanism being unchanged. The corresponding variation of j_c vs. B at 4.2K for our tape with $j_c(77\text{K}, 0\text{T}) = 30'000 \text{ A/cm}^2$ is shown in Fig.4, from which a value of $j_c(4.2\text{K}, 25\text{T}) \approx 70'000 \text{ A/cm}^2$ can be extrapolated. A comparison with a large number of tapes confirms that it is possible to estimate the value of j_c at 4.2K and very high fields (e.g. 25T) for B//a,b once the value $j_c(77\text{K}, 0\text{T})$ is known.

Recently, a correlation was found between the degree of texturing of the Bi(2212) grains in a tape after deformation (before heat treatment) and the properties of the Bi,Pb(2223) tape after the reaction heat treatment [4], as shown by Figs.5 and 6. The texture of Bi(2212) grains is determined by X-ray diffraction analysis, and is defined by the parameter

$$f = \frac{I(115)}{I(115) + I(008)}$$

From Fig.5, it can be seen that f is smallest at the end of deformation, which means highest degree of texturing. After reaction, texturing in the region close to the oxide/Ag interface increases due to the internal pressure exerted by the growing grains, and another way of defining the degree of texturing has to be used. In Fig. 6, the effective transport misalignment angle Φ_e as defined by angle dependent j_c measurements [13] has been plotted as a function of tape thickness. A comparison between Figs.5 and 6 allows to establish a correlation between the texturing of Bi(2212) grains and the texturing of the Bi,Pb(2223) which carry the current.

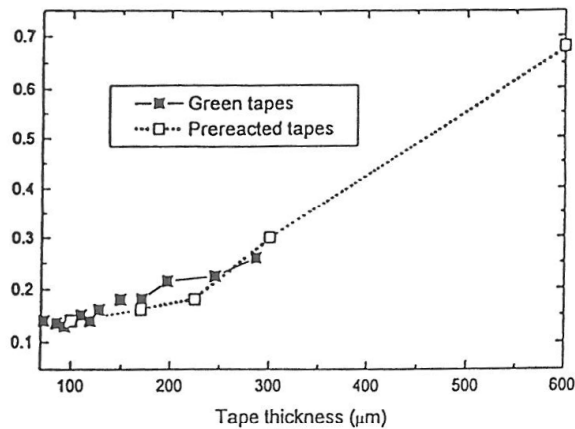


Fig.5. Variation with tape thickness of the degree of texturing of the unreacted Bi(2212) after deformation.

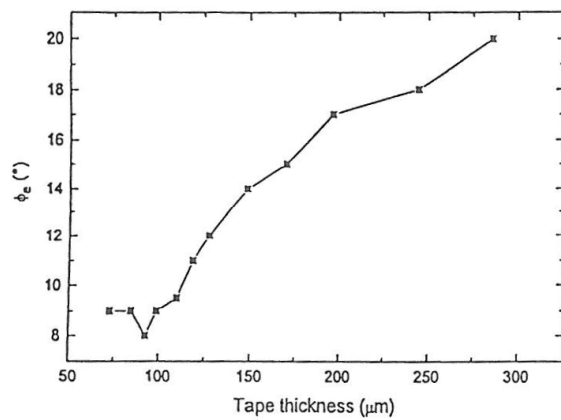


Fig.6. Correlation between tape thickness and effective transport misalignment angle, Φ_e [4].

Another question related to the deformation process is why j_c of Bi,Pb(2223) tapes produced by pressing is generally higher than for rolled tapes. An answer has been given recently [7], comparing the forces acting on the tape during deformation. As shown in Fig.7, j_c increases monotonically for pressures up to >2GPa, while for rolled tapes, a sharp decrease is observed above 0.6GPa [7]. This can be explained by the formation of cracks in transversal direction because of the higher elongation during the rolling process.

CURRENT DISTRIBUTION

The lateral transport current distribution in monofilamentary Bi,Pb(2223) tapes was determined by the strip-cutting technique [14] and is shown in Fig.8 for two tapes prepared by the rolling procedure with $j_c(77K,0T) = 22'000$ and $28'000$ A/cm², respectively.

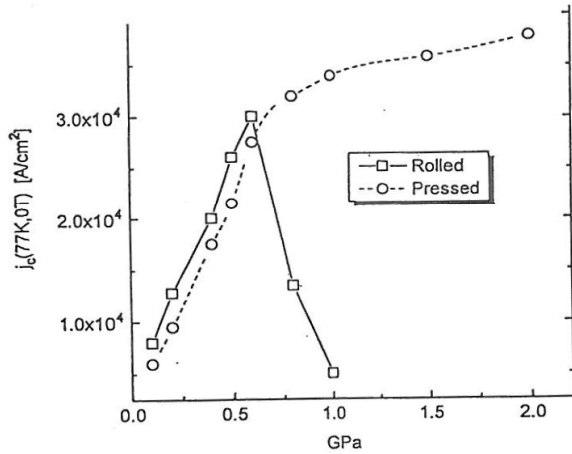


Fig. 7. j_c of pressed and rolled Bi,Pb(2223) tapes as a function of the effective pressure during deformation [7].

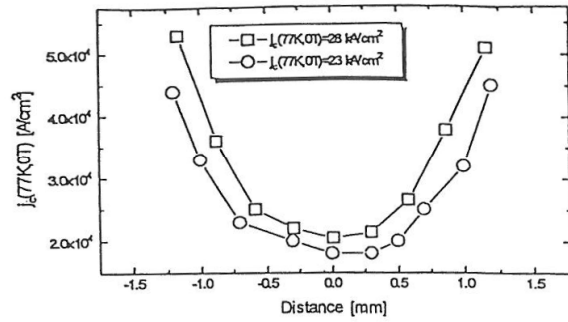


Fig. 8. Lateral j_c distribution for two Bi,Pb(2223) tapes with different j_c values [14].

It has been found that the critical current density at the sides is considerably higher than at the centre, the maximum transport j_c at the sides being $54'000 \text{ A/cm}^2$ [14]. This distribution was confirmed on the same samples by non-invasive Hall probe measurements [15] and by magneto-optical imaging [16]. Several reasons lead to the observed "parabolic" behavior of j_c : a) a higher compression of the oxide layer is observed at the sides, b) a larger amount of foreign phases is observed at the centre of the tape, and c) the degree of texturing at the inner centre of the tape is lower than at the sides [14].

MULTIFILAMENTARY TAPES

The cross-section of a 19 core Bi,Pb(2223) tape is shown in Fig.9, at the end of the drawing process (diameter 1.25mm) and after rolling to 0.18mm thickness. The superconductor ratio was 0.18. The critical current density at 77K, 0T was $23'000 \text{ A/cm}^2$, which corresponds to an overall j_c of $4'140 \text{ A/cm}^2$. The variation of j_c vs. B measured at 70 K is shown in Fig. 10. The decrease between $B = 0$ and 1T is somewhat smaller than for the monofilamentary tapes (Fig. 3), but this is due to the lower temperature, $T = 70 \text{ K}$.

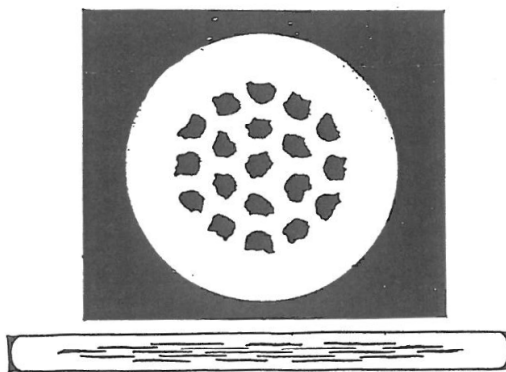


Fig.9 Cross-section of a 19 core Bi,Pb(2223) tape (wire diameter 1.25mm and tape thickness 0.18m).

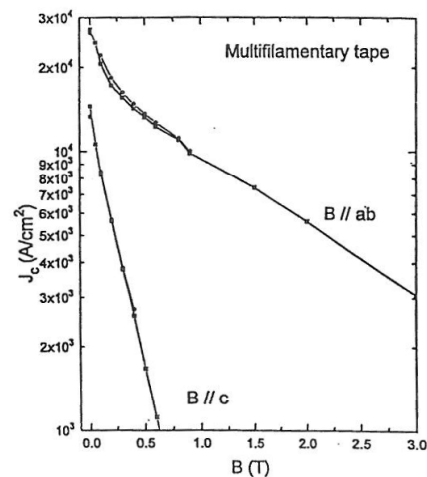


Fig. 10. j_c vs. B of a 19 core Bi,Pb(2223) tape at 70K.

After correcting the temperature difference, there is no noticeable difference between the variation $j_c(B)$ of our mono- and multifilamentary tapes, in agreement with Ref.1. The smaller j_c value, 23'000 with respect to 30'000 A/cm² for monofilamentary tapes is attributed to fluctuations of the individual filament cross-sections along the tape. It is to expect that an improvement of the regularity of the filament cross-section will lead to very similar j_c values as for monofilamentary tapes.

CURRENT TRANSFER IN Bi,Pb(2223) TAPES

The current transfer from one Bi,Pb(2223) grain to another has been recognized as occurring through small-angle c-axis tilt grain boundaries [17]. Due to the anisotropic nature of the Bi,Pb(2223) structure and to the textured microstructure inside a tape, a detailed consideration of the current transport requires a careful definition of the various components. A model for the current transfer between Bi,Pb(2223) platelets requires necessarily the occurrence of current densities j_c° along the c-axis. These currents are required to distribute the net a-b plane current also in the bulk of the grain. From a direct measurement of the resulting critical current density j_c^n perpendicular to the tape surface as a function of the applied field, it was concluded that the value of the intragrain critical current density j_c° is the main limiting factor [17]. Another important factor influencing the overall value of j_c is the total number of strongly coupled connections ("railway switches") transferring the current inside the tape.

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