



Bulk textured Bi–Pb–Sr–Ca–Cu–O (2223) ceramics by solidification in a magnetic field

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Received 6 February 1996

Abstract

Bulk textured Bi–Pb–Sr–Ca–Cu–O (2223) ceramics have been prepared by solidification in a magnetic field. We have determined the optimal temperature required in the melt in order to maximize the effect of the magnetic field. X-ray pole figures and scanning electron microscopy have been used to characterize the nature and degree of texture. Magnetic and electrical transport measurements in the superconducting state are also reported. Transport critical current densities as high as 1450 A/cm² at 77 K and 0 tesla have been reached. This value is significantly greater than that, 900 A/cm², obtained following the same heat treatment in the absence of an applied magnetic field.

PACS: 7470V; 7430C; 8120E; 8140R

1. Introduction

It is well known that the capacity of ceramic superconductors, in particular bulk Bi–Pb–Sr–Ca–Cu–O, to transport current is dependent on both the platelet alignment and the density of the material. Several techniques of texturation based on melting in a thermal gradient [1–3], or in a magnetic field [4–6] have been successfully used on YBaCuO and Bi–Pb–Sr–Ca–Cu–O ceramics. High pressure techniques such as isostatic pressing [7,8], hot forging or uniaxial pressing [9–12] have proved to be efficient in texturing Bi–Pb–Sr–Ca–Cu–O.

In this paper we first report on the feasibility of using a magnetic field of 8 T to induce orientation of Bi-2223 crystallites embedded in epoxy. Secondly, we describe the experimental conditions used during the magnetic melt texturation process (MMT). The optimal temperature of the melt attained during the heating–cooling cycle was determined using several methods of characterization: X-ray pole figures, microscopy, electrical transport and magnetization.

2. Magnetic orientation of Bi:2223 powder in epoxy

We have tested the possibility of using a magnetic field to induce the orientation of powdered Bi-2223 superconductor suspended in epoxy (slow hardening

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standard Araldite). The starting powder was Hoechst precursors (grade 1) $\text{Bi}_{1.8}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2.2}\text{Cu}_3\text{O}_{10.3+x}$ with a 2–4 μm granulometry. The suspension, contained in a teflon mold, was placed in a magnetic field of 8 T for 20 h at room temperature. We expected that the individual crystallites would orient with their c -axes directed along the field direction due to the anisotropy in the paramagnetic susceptibility associated with the CuO planes [13–15].

A high degree of orientation with the c -axis parallel to the field was indeed deduced from the magnetization anisotropy factor $\Delta M(H//H_a)/\Delta M(H \perp H_a) \approx 6$ at 4.2 K (Fig. 1) where ΔM is a magnetic hysteresis, H magnetic field measurement and H_a a magnetic texturing field applied. X-ray pole figure analyses confirm the relatively high degree of crystallite orientation. Fig. 2 represents the {002} experimental pole figure. The central pole of this figure denotes a high degree of preferred orientation with the crystallite c -axes parallel to the texturing direction; the dispersion is at most 8° from this direction at 10% of the maximum intensity. From this information we calculate the maximum distribution density to be approximately 23.5 m.r.d. (multiple of random distribution).

3. Solidification in a magnetic field

3.1. Experimental procedure

Powder samples $\text{Bi}_{1.8}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2.2}\text{Cu}_3\text{O}_{10.3+x}$ are pressed into pellets of 20 mm diameter and 10 mm thickness by application of an uniaxial pressure of 1 GPa. The pellets were annealed at 840°C for 90 h in air in order to induce superconductivity.

The heating cycle took place in a vertical furnace, placed in the room temperature bore of superconducting magnet. A maximum field of 8 T can be applied parallel to the axis of the furnace. The sample in air was contained in zirconium crucibles and placed in the maximum field. The temperature was controlled using a Pt/Pt–Rh thermocouple which also served as a sample support. The temperatures at which melting began and ended, reported precisely [16], served as a starting points for this study. An example of a typical thermomagnetic treatment is shown schematically in Fig. 3. The temperature was maintained at a maximum T_m for 1 h, then decreased slowly at a rate of $2^\circ\text{C}/\text{h}$. By physically characterising processed samples we have deter-

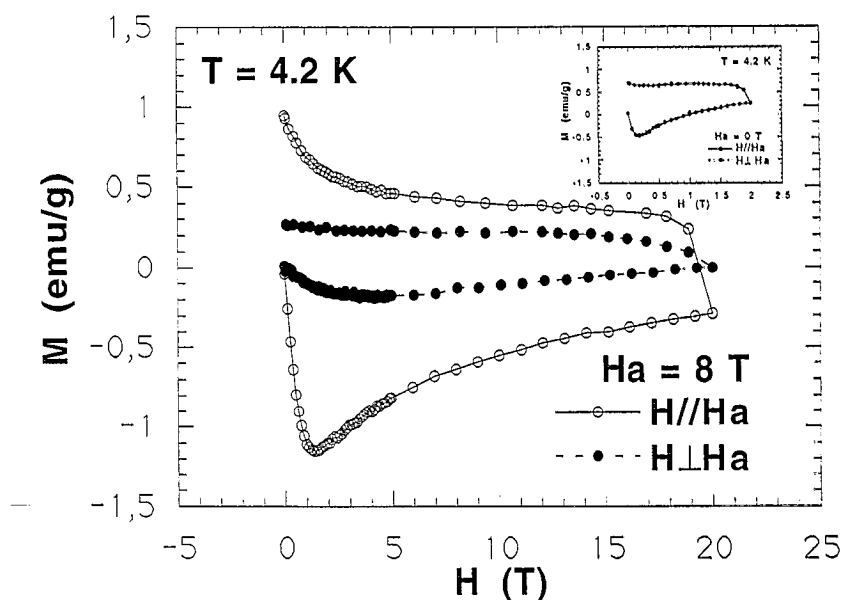


Fig. 1. Magnetization hysteresis at 4.2 K, with the measuring field H parallel and perpendicular to the texturing field, $H_a = 8$ T. Inset $H_a = 0$ T.

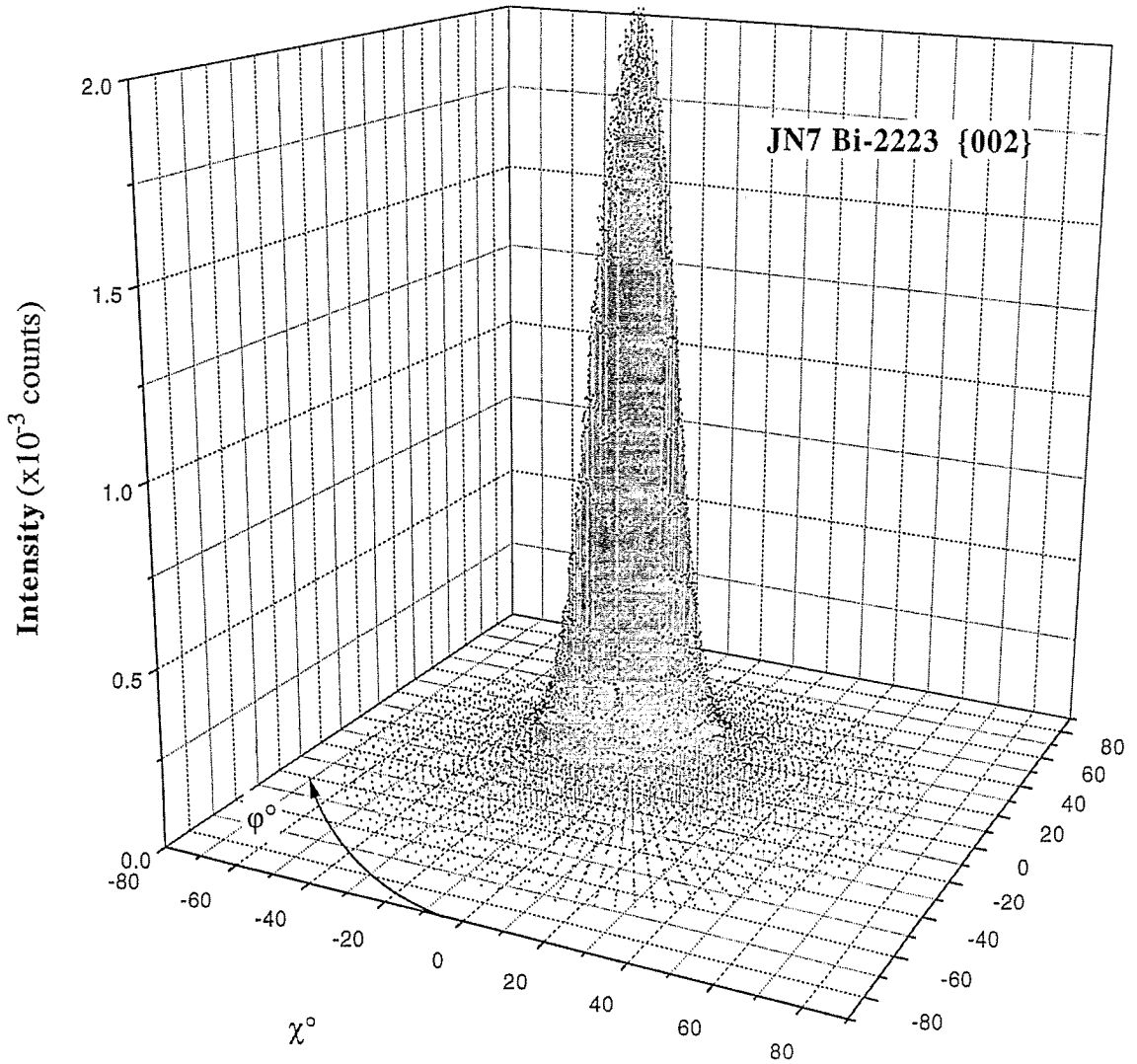


Fig. 2. {002} X-ray pole figure of embedded sample revealing the high degree of the *c*-axis orientation.

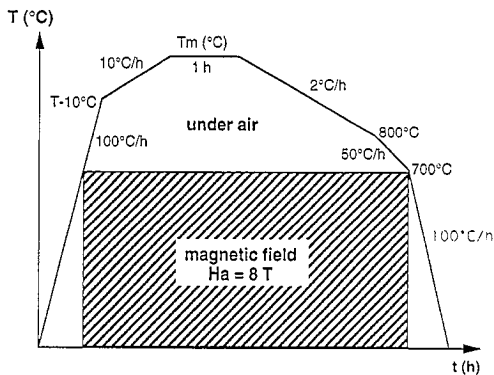


Fig. 3. Typical thermomagnetic treatment.

mined that the optimum value of T_m lies within the range $855\text{--}900^\circ\text{C}$.

3.2. Experimental results

3.2.1. X-ray diffraction

X-ray diffraction patterns of the face oriented perpendicular to the applied field are shown in Fig. 4. (001) peaks are by far the most intense which indicates a strong alignment of the *c*-axis, along the magnetic field direction. Traces of (*hkl*) lines indi-

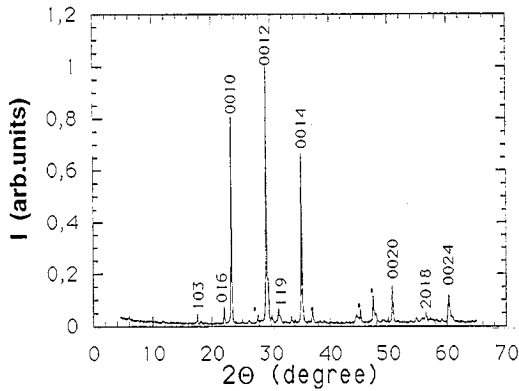


Fig. 4. X-ray diffraction spectrum for sample face perpendicular to the direction of applied field, (■) traces of Bi:2212 phase.

cate that the concentration of disoriented grains is low. Weak peaks of the Bi:2212 phase are also present.

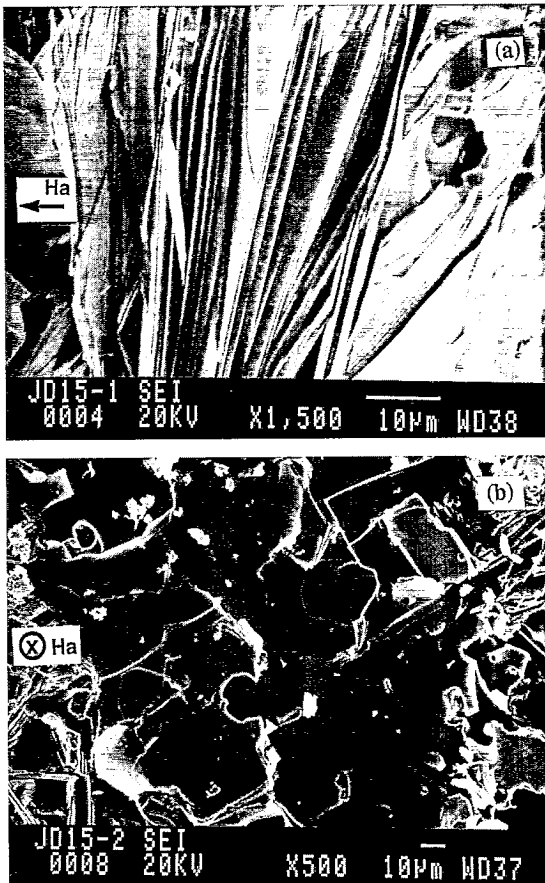


Fig. 5. SEM micrographs of sample faces (a) parallel and (b) perpendicular to the direction of the applied field.

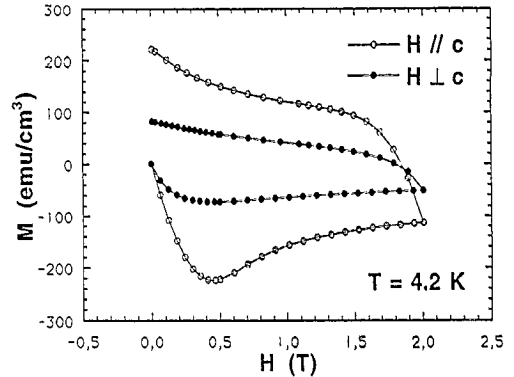


Fig. 6. Magnetization versus field at 4.2 K for field applied parallel (○) and perpendicular (●) to the *c*-axis of the sample.

3.2.2. Scanning electron microscopy

Scanning electron microscopy (SEM) measurements were made on faces broken parallel and perpendicular to the applied field (H_a) direction. When the face is parallel (Fig. 5a), a preferential orientation of the platelets perpendicularly to the magnetic texturing field is observed with i) an increase in platelet size ($> 50 \mu\text{m}$), ii) a decrease in the voids between grains lying in the plane of the face. The face lying perpendicular to the field direction consists of disordered large flat grains (Fig. 5b). These observations demonstrate alignment of the *c*-axis along the magnetic field direction.

3.2.3. Hysteresis cycles

The magnetic hysteresis cycles were measured in the superconducting state at 4.2 K. In Fig. 6, we show the curves of magnetization versus field obtained from $2 \times 2 \times 2 \text{ mm}^3$ cubes cut from pellets. The excitation field H is applied both parallel and perpendicular to the direction of the magnetic texturing field H_a . The hysteresis in the magnetization is greater when the direction of the measuring field is parallel to H_a , that is to say: $\Delta M_{H//H_a} / H_a > \Delta M_{H \perp H_a} / H_a$. This confirms the platelet alignment. At 0.4 T we determined the magnetization anisotropy ratio to be 2.7.

3.2.4. Transport critical current density

The optimal temperature T_m of the melt in a magnetic field has been determined to lie between 855 and 900°C using values of transport critical current densities J_c . We measured the transport criti-

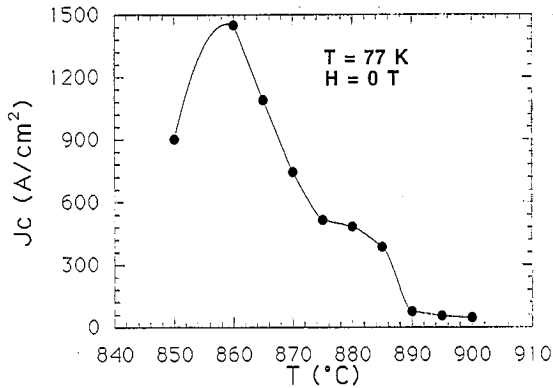


Fig. 7. Transport current density versus temperature T of melting in a magnetic field at 77 K and 0 tesla.

cal current using the four points method, with a 10 $\mu\text{V}/\text{cm}$ criterion. The measurements were performed at 77 K in zero field with the current parallel to the ab -planes. The bars were typically 10 mm long and $\approx 1 \text{ mm}^2$ in cross section. Contacts are painted onto the surface of the bars using silver loaded paint and annealed at 840°C for 30 min. The contact resistance obtained in this way was usually between 1–5 $\mu\Omega/\text{cm}^2$ at 77 K. A maximum J_c value of 1450 A/cm^2 was obtained for $T_m = 860^\circ\text{C}$ (Fig. 7) which corresponds to the optimum processing temperature. This value should be compared to 882 A/cm^2 obtained with classical sintering [17]. Above 860°C the decrease in the critical current density is associated with the presence of the sec-

ondary phases $(\text{Ca}/\text{Sr})_{14}\text{Cu}_{24}\text{O}_y$, CuO and $(\text{Ca}/\text{Sr})_2\text{CuO}_3$ observed by semiquantitative SEM analysis as discussed in Ref. [18]. Below 860°C, a decrease in the temperature T_m correlates with a decrease in J_c . In these conditions the proportion of the liquid phase is probably too small to allow rotation and growth of the platelets under the influence of the magnetic field.

Fig. 8 shows the angular dependence of the transport critical current density at 77 K under magnetic field ($H = 20 \text{ mT}$). This field is applied perpendicular to the direction of current flow and the sample can be rotated around this axis. Θ is the angle between the magnetic field and (a, b) planes. A peak is observed when the field is parallel to the (a, b) planes at $\Theta = 0^\circ$. This phenomenon is due to the intrinsic pinning by the layered structure of the bulk materials [19,20].

4. Conclusion

Bulk textured $\text{Bi}_{1.8}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2.2}\text{Cu}_3\text{O}_{10.3+x}$ ceramics have been prepared by solidification in a magnetic field of 8 T. We obtained highly textured ceramics exhibiting critical current densities of 1450 A/cm^2 compared to 900 A/cm^2 or 882 A/cm^2 obtained respectively without an applied magnetic field or by classical sintering.

Magnetic field allows one to induce and control the direction of crystallite oriented. The encouraging results of this paper demonstrate that magnetic texturing of Bi:2223 is feasible. Further improvement in the performance is possible by the optimization of other parameters such as the rate of solidification, or the duration of the high temperature dwell time.

Acknowledgements

J.G.N. acknowledges his fellowship from ADEME and Alcatel Alsthom. This work is supported by CPR on superconductivity of PIRMAT. We are grateful to P. Amiot for her help in carrying out the SEM studies.

The authors thank J. Torbet for careful reading of the manuscript.

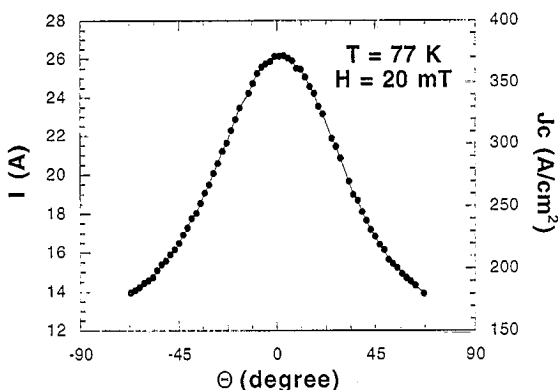


Fig. 8. Angular dependence of the transport critical current at 77 K under magnetic field ($H = 20 \text{ mT}$).

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