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Fabrication and Texture Characterization of Bulk $(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$ and $Bi_2Sr_2CaCu_2O_x$ Superconductors*

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FABRICATION AND TEXTURE CHARACTERIZATION OF BULK $(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$ and $Bi_2Sr_2CaCu_2O_x$ SUPERCONDUCTORS*

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ABSTRACT

Bulk $(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$ (Bi–2223) and $Bi_2Sr_2CaCu_2O_x$ (Bi–2212) superconductors were fabricated by sinter forging. Bi–2223 (≈90% Bi–2223, 10% $(Bi,Pb)_2Sr_2CaCu_2O_x$ + other phases) and Bi–2212 (nearly phase pure) powders were first synthesized and then cold pressed into bars that were ≈50% dense. These bars were surrounded by Ag foil, heated in air to ≈845°C, and compressed for 3–6 h. The resultant bars were dense and highly textured. At 77 K, the Bi–2223 exhibited transport critical current density (J_c) values of 2000–8000 A/cm^2; the Bi–2212 exhibited very low J_c. Extent of texture was evaluated by three X–ray diffraction methods: 2θ scans, rocking curves, and orientation distribution functions. It was found that J_c correlated best with the orientation distribution functions.

INTRODUCTION

It has been shown that J_c of bulk high–temperature superconductors is strongly related to texture. Because of the anisotropic nature of each of the high–temperature superconductors,[1] c–axis alignment is required for high J_c,[2-6] and there are indications that alignment within a-b planes may also be required.[6-9]

The texture of a bulk superconductor can be described in many ways. Standard 2θ X–ray diffraction scans yield qualitative information.[10] Quantitative information on grain orientation can be obtained from diffrac-

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tion information by rocking curves,[4,10] pole figures,[10-12] or orientation distribution functions (ODFs).[10-14] These techniques all sample large numbers of grains at once. Texture data can also be obtained on a grain–by–grain basis by, for example, transmission electron microscopy[9] or electron backscatter methods in scanning electron microscopy (SEM).[6]

In previous work on sinter forging of Bi–based high–temperature superconductors, it was confirmed that strong c–axis alignments were produced in dense specimens of good phase purity.[15-17] The work reported here summarizes correlations made between J_c and texture, as measured by 2θ X–ray diffraction scans, rocking curves, and ODFs.

EXPERIMENTAL PROCEDURES

Specimen preparation

$Bi_2Sr_{1.7}CaCu_2O_x$ (Bi–2212) and $(Bi,Pb)_{2.2}Sr_2Ca_2Cu_3O_x$ (Bi–2223) powders were synthesized from mixtures of oxides and carbonates.[16,18] X–ray diffraction and differential thermal analysis revealed that the Bi–2212 was virtually phase pure and the Bi–2223 ≈90% phase pure. The powders were cold pressed in dies 44.5 x 7.6 mm. The resultant bars were ≈50% dense and moderately textured.

Ag foil was placed on top and bottom of the bars. Each bar was then heated in air to 840–850°C, and compressed for 3–6 h. Compression rates were 0.001–0.01 mm/min, with the fastest rates being used at the beginning of the compression cycle. Immediately after compression was completed, each bar was cooled to room temperature at ≈3°C/min. The Bi–2212 and Bi–2223 bars were > 95% dense and exhibited obvious c–axis texture in SEM (Fig. 1).[16-18]

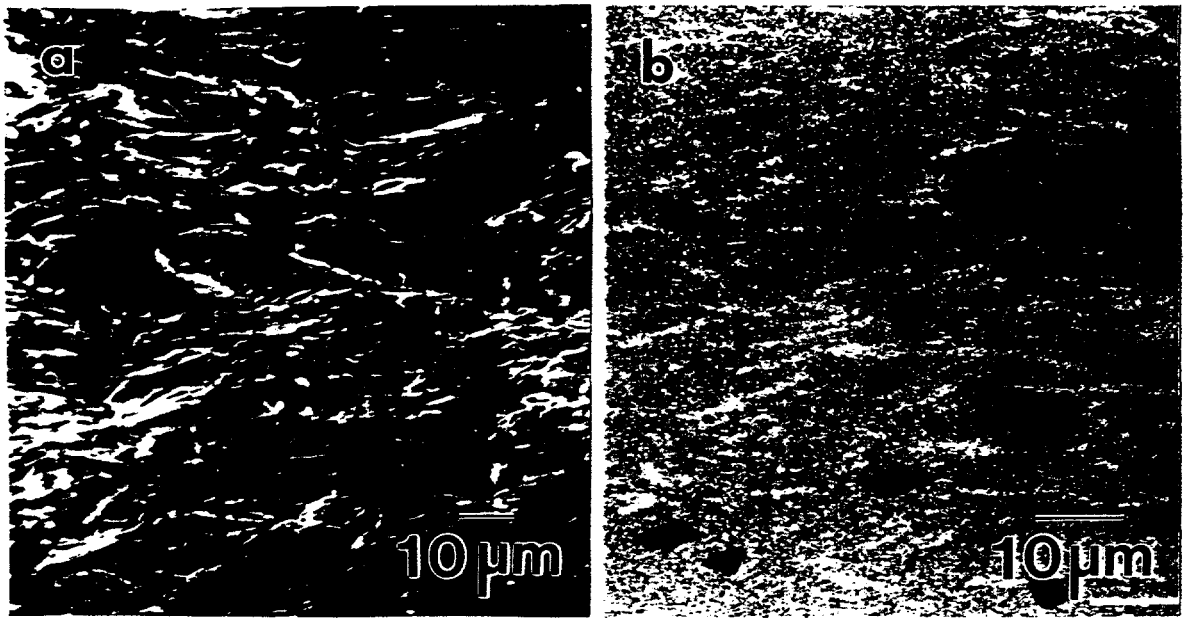

Figure 1. SEM photomicrographs of (a) fracture surface of typical Bi–2212 bar and (b) polished cross section of Bi–2223 bar (light–gray streaks are Bi–2212 phase and dark regions are alkaline–earth cuprates).

J_c and texture measurements

J_c was measured at 4.2 K for the Bi–2212 and 77 K for the Bi–2223. The criterion was 1 μV/cm. Because of the high currents needed for the Bi–2223, a pulsed DC current source was used.

For each bar examined, the Ag was removed and a compression face was polished. X–ray 2θ scans and rocking curves were obtained with a Phillips rotating anode diffractometer. The 0 0 10 peak at 24.0° was selected for the rocking–curve analysis. At least two measurements were made for each selected bar. Full width at half maximum (FWHM) was determined for each rocking curve.

Pole figures needed to generate the ODFs were obtained with a Siemens three–circle goniometer unit. The methods used have been described.[15,16,19]

RESULTS AND DISCUSSION

Despite the excellent texture, density, and phase purity of the Bi–2212 bars, J_c values were very low. Even at 4.2 K, J_c did not exceed 1000 A/cm^2. It has been observed previously that the presence of a liquid phase during processing appears to be necessary to produce high transport J_c values in polycrystalline superconductors.[16,20] The Bi–2212 bars were virtually phase pure, and thus very little or no liquid was present during sinter forging. The resultant transport properties were dominated by weak links. It is of interest that, although the J_c values of the Bi–2212 bars were very poor, the elastic properties were equal to those of single crystals.[20] Therefore, the weak links cannot be attributed to obvious defects such as cracks or second phases.

Because the Bi–2212 was weak linked, the Bi–2223 was used for relating texture to J_c. Although the Bi–2223 was less phase pure than the Bi–2212, reactions to form Bi–2223 occurred during the forging process, and liquid was present during the reactions.[16] Transport J_c values at 77 K in self–field were found to be 2000–8000 A/cm^2.

Bi–2223 bars with J_c values of 3000 and 7600 A/cm^2 were selected for detailed texture analysis. Each had a microstructure similar to that shown in Fig. 1b. As shown in Fig. 2, both exhibited strong c–axis texture. There was little obvious difference between the two diffraction patterns.

Rocking curves of the 0 0 10 peak of the two bars were also similar. All FWHM values were 8.6–9.1° (Fig. 3). No clear trend was observed for J_c vs. FWHM.

Rocking–curve analysis has proved to be quite successful in comparing the quality of single crystals and thin films.[22] For such materials, the c–axis texture is not merely strong, it is nearly perfect. It does not appear that rocking curves correlate equally well with properties of a bulk, polycrystalline superconductor.

Pole figures for the two Bi–2223 bars are shown in Fig. 4. These pole figures were used to generate ODFs for each bar (Fig. 5). It is clear that the higher J_c bar exhibited superior c–axis texture.

An ODF is a complete statistical description of the orientation of grains within a volume. Thus, in addition to information on texture normal to the plane, information on in–plane texture is incorporated. ODFs of high–

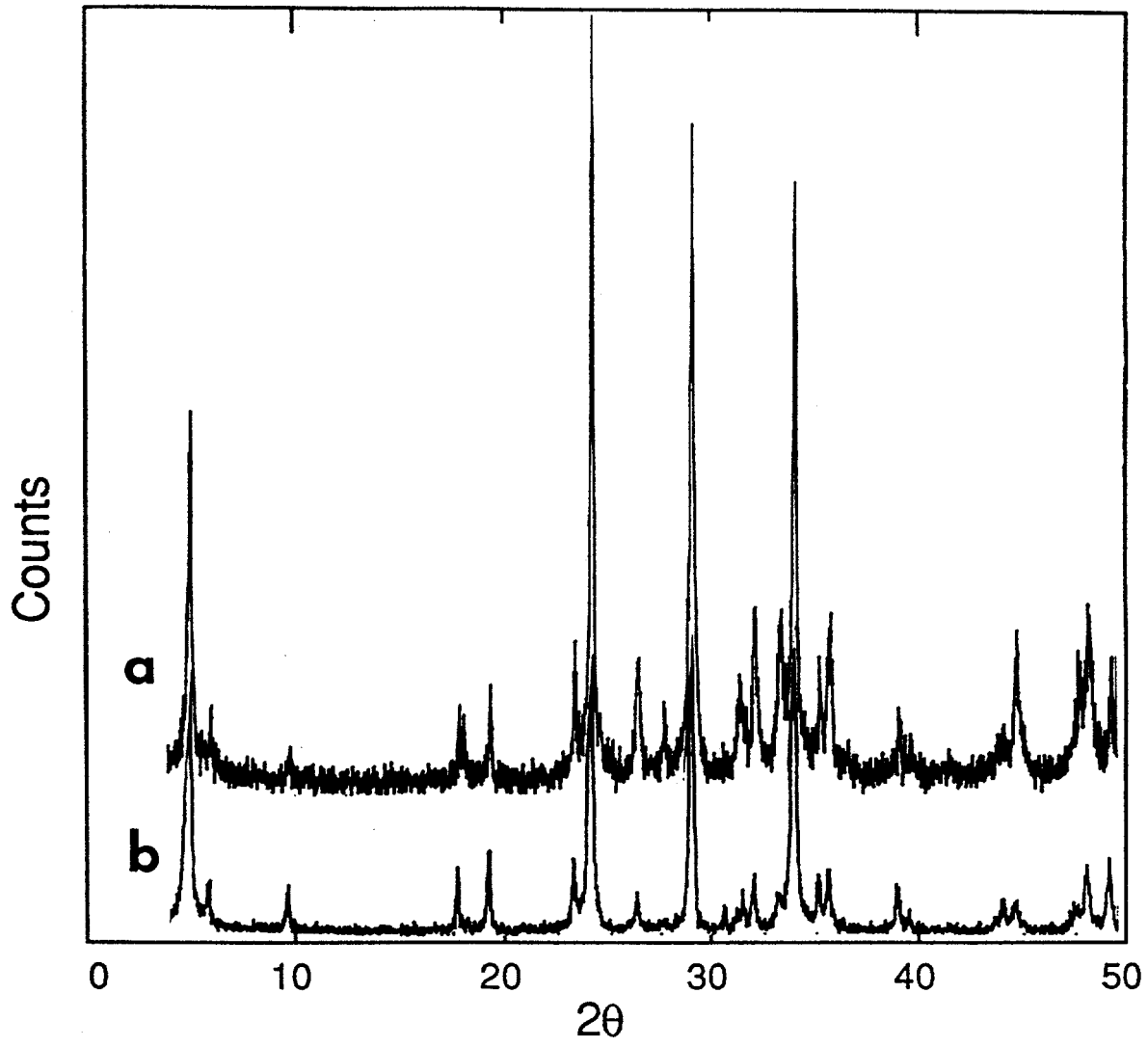

Figure 2. 2θ X–ray diffraction scans of Bi–2223 bar: (a) J_c = 3000 A/cm^2, (b) J_c = 7600 A/cm^2.

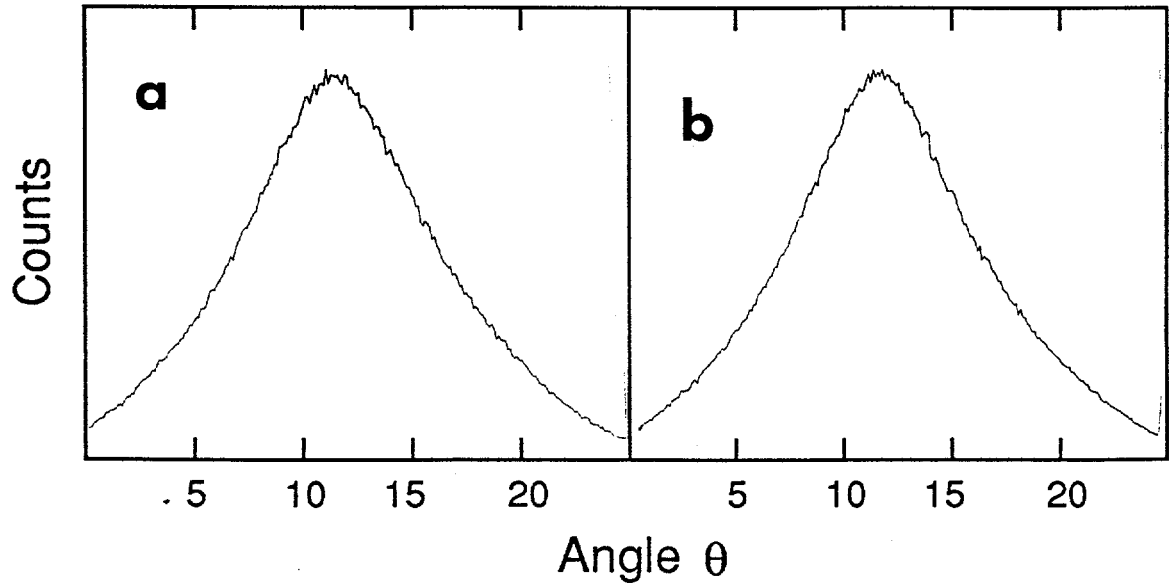

Figure 3. Rocking curves of 0 0 10 peak of Bi–2223 bars: (a) J_c = 3000 A/cm^2, (b) J_c = 7600 A/cm^2.

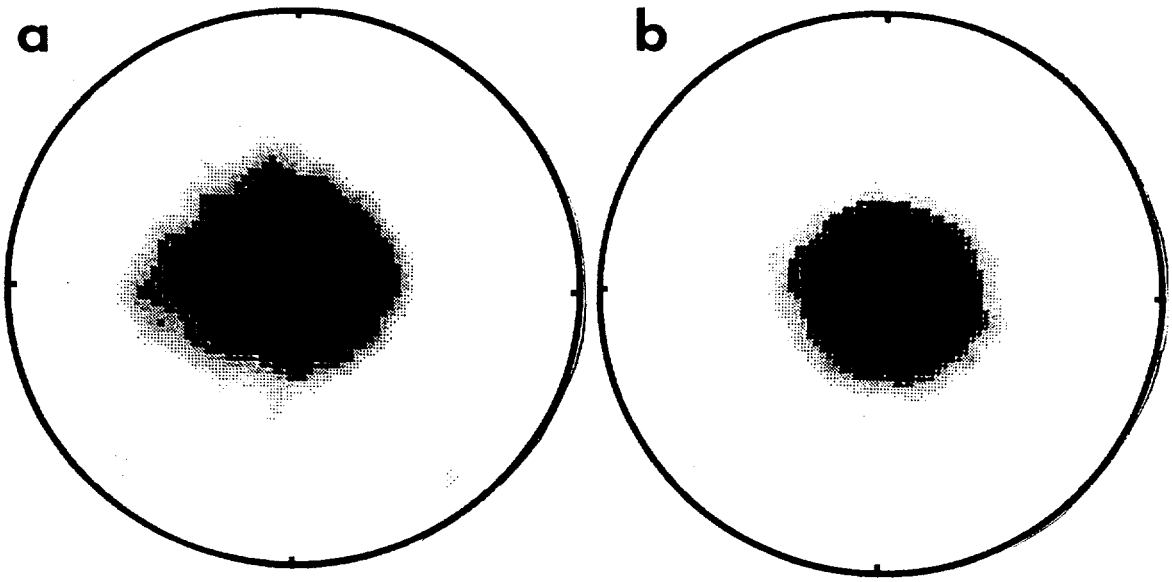

Figure 4. X–ray (0 0 1) pole figures of Bi–2223 bars: (a) J_c = 3000 A/cm^2, (b) J_c = 7600 A/cm^2.

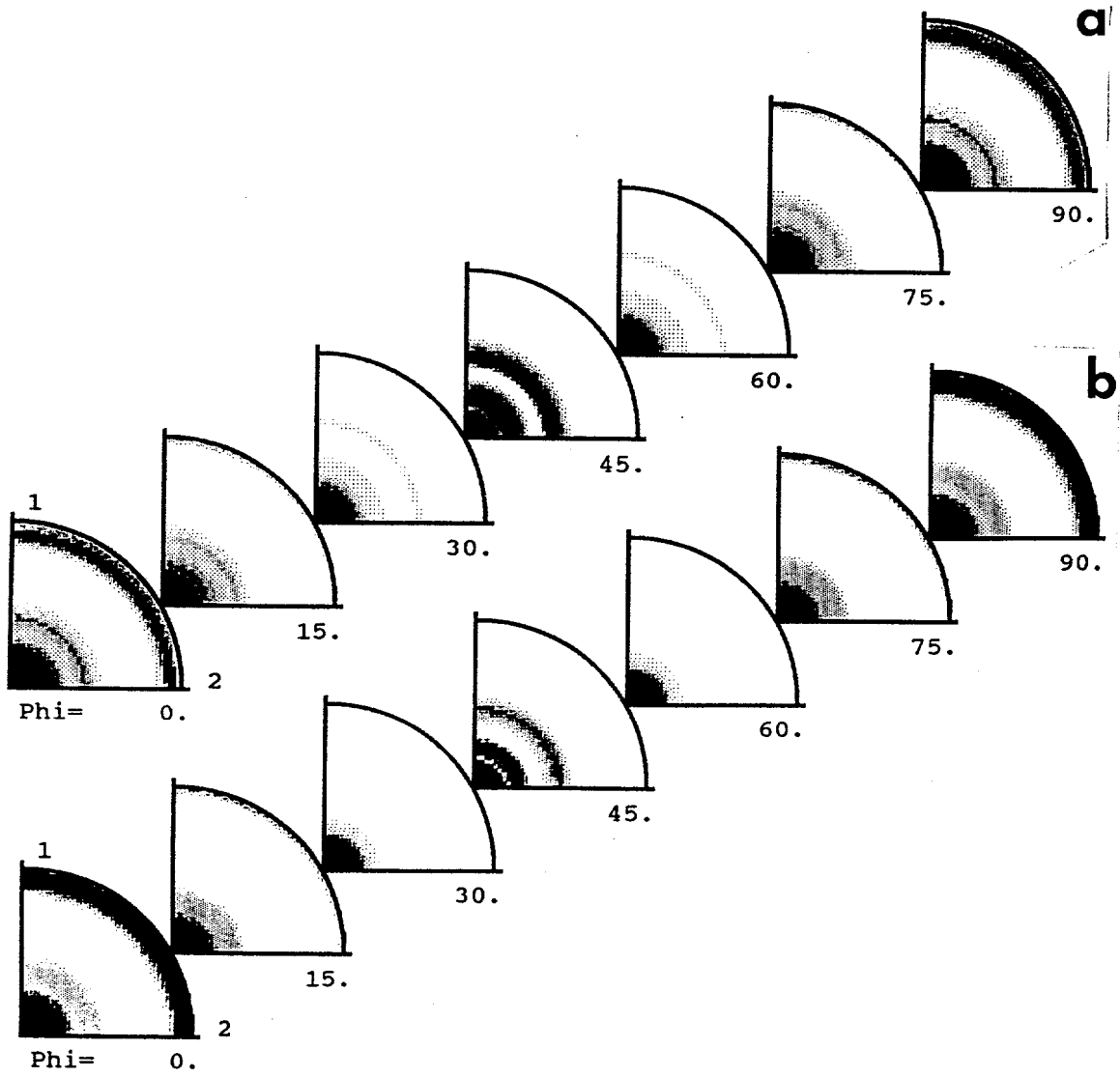

Figure 5. ODFs of Bi–2223 bars: (a) J_c = 3000 A/cm^2, (b) J_c = 7600 A/cm^2.

temperature superconductors have been obtained with X–rays[12,15,19] and neutrons.[19,23] In either case, largenumbers of grains are sampled. Coupled with microstructural observations by electron microscopy, ODFs can be an effective tool in determining the quality of a bulk high–temperature superconductor. Transport J_c has been shown to correlate well with texture for Ag–clad tapes[14,19] and, as shown here, monolithic Bi–2223 superconductors.

CONCLUSIONS

Bi–2212 and Bi–2223 superconductors were fabricated by sinter forging. The Bi–2212 bars were weak linked and exhibited low transport J_c. The Bi–2223 bars exhibited J_c values at 77 K of 2000–8000 A/cm^2. J_c for the Bi–2223 correlated strongly with texture as quantified by orientation distribution functions.

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