

MANUSCRIPTPROPERTIES OF HOT ISOSTATICALLY PRESSED  $\text{Bi}_2\text{Sr}_{1.7}\text{CaCu}_2\text{O}_x^*$ *S. Nash*

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# PROPERTIES OF HOT ISOSTATICALLY PRESSED $\text{Bi}_2\text{Sr}_{1.7}\text{CaCu}_2\text{O}_x$

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## ABSTRACT

Dense  $\text{Bi}_2\text{Sr}_{1.7}\text{CaCu}_2\text{O}_x$  (2212) superconductor pellets were made by hot isostatic pressing in an inert atmosphere. The pellets exhibited a small amount of preferred orientation of the grains. The transport critical current density ( $J_c$ ) values were very low for all specimens. Dislocations, planar faults, and intergrowths of the  $\text{Bi}_2\text{Sr}_2\text{CuO}_x$  phase, which were produced during the pressing, caused the intragranular  $J_c$  to increase substantially.

## INTRODUCTION

The  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$  (2212) phase has highly anisotropic electrical and physical properties [1]. This phase and other Bi-based superconductors have been successfully densified by techniques such as hot isostatic pressing [2-6] or hot pressing [7,8]. This study is a continuation of previous work [3,6,9] on hot isostatic pressing (HIPing) of 2212. The previous work concentrated on mechanisms of consolidation [3,9], microstructures produced by various procedures [9], and effects of processing on  $T_c$  values [3,6]. In this paper, transport and magnetization  $J_c$  data are reported.

## EXPERIMENTAL METHODS

Powder of composition  $\text{Bi}_2\text{Sr}_{1.7}\text{CaCu}_2\text{O}_x$  was synthesized from a mixture of  $\text{Bi}_2\text{O}_3$ ,  $\text{SrCO}_3$ ,  $\text{CaCO}_3$ , and  $\text{CuO}$  powders [10]. The powders were ball-milled for 18-24 h in polyethylene jars containing methanol and  $\text{ZrO}_2$  grinding media. The milled powders were pan-dried, ground lightly with an agate mortar and pestle, and heated in air for 48-72 h at 815-840°C. The resulting powder was ground in a tungsten carbide rotary mill. The final average particle size was  $\approx 3 \mu\text{m}$ . No  $\text{Bi}_2\text{Sr}_2\text{CuO}_x$  (2201) phase was observed by X-ray diffraction or differential thermal analysis [10]. The 2212 powder was cold pressed into pellets which were then HIPed in an inert atmosphere. The pressure was 105 MPa, the temperature was 825°C, and the time at temperature and pressure was 15 or 120 min.

Geometric densities were obtained for all of the specimens. Many of the specimens were examined by X-ray diffraction, scanning electron microscopy (SEM), and transmission electron microscopy (TEM). In addition, the extents of crystallographic texture were quantified by X-ray diffraction through use of orientation distribution functions. For the texture measurements, a pellet was mounted face up in epoxy, ground, and polished. The 001 and 117 pole figures were obtained on these specimens with  $\text{Cu K}\alpha$  radiation [11-13]. From these pole figures, orientation distribution functions were calculated, and plotted with use of the Roe convention for assignment of angles [14]. The details of these measurements and calculations can be found in Refs. 12 and 13.

Small pieces were cut from the HIPed specimens with a slow-speed diamond saw. Transport  $J_c$  measurements were made on 1.5 cm long bars at 4.2 K with a voltage criterion of 1  $\mu\text{V}/\text{cm}$ . Ag electrodes were painted on the bars, which were then heated in air at  $\approx 750^\circ\text{C}$  to promote bonding. The magnetic-hysteresis measurements were performed on a commercial vibrating-specimen magnetometer that contained a superconducting magnet in a gas-flow-type cryostat. The field was ramped at a rate of 80 G/s. For  $T > 70$  K, a Pt resistance thermometer was used for temperature control. For  $T < 70$  K, a carbon glass thermometer was used.

## RESULTS AND DISCUSSION

All of the HIPed pellets were fully dense. The measured strengths, fracture toughnesses, and elastic moduli suggested that the grains were well connected [15]. As shown in Fig. 1, some preferred orientation of the grains was produced by the HIPing. The c-axis textures were far less pronounced, however, than has been observed for 2212 thick films [16] or powder-in-tube tapes [17,18]. The maximum textures in the orientation distribution functions were approximately twice random.

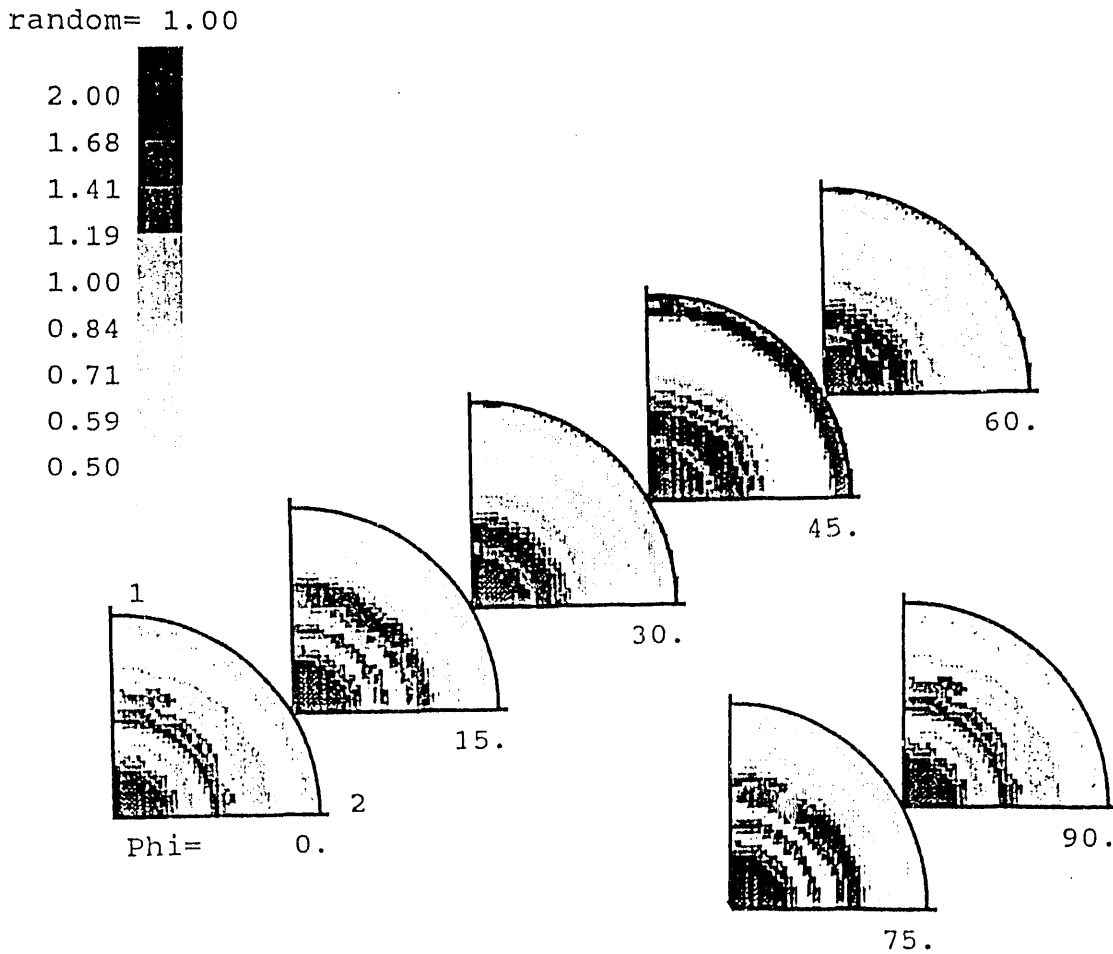


Fig. 1. Orientation distribution function of HIPed 2212 pellet plotted for one quadrant only.

In previous work on HIPed 2212 pellets, the transport  $J_c$  values at 77 K were very low. Since the  $T_c$  values were generally less than 84 K, this result was not surprising [6]. In this study, the  $J_c$  at 4.2 K was found to be low also: none of the specimens examined had a transport  $J_c$  greater than  $\approx 100$  A/cm<sup>2</sup>. These low values indicate that high density alone is not sufficient for good  $J_c$  in 2212.

One of the consequences of HIPing is that few of the grains are textured with respect to each other. The principal means of densification during HIPing is probably bending of grains [9]. The grains have little opportunity to form low-angle or low-energy grain boundaries. The grain boundaries that form do not appear to be able to conduct supercurrent well.

TEM of the HIPed pellets revealed high concentrations of dislocations and planar faults within the grains. In the 15-min samples, several types of complex dislocations, primarily in the a-b planes, planar faults, and possibly intergrowths of the  $\text{Bi}_2\text{Sr}_2\text{CuO}_x$  phase were observed (Fig. 2a). In the 120-min samples, the dislocations were largely present at subgrain boundaries and fewer faults were present (Fig. 2b). The concentrations of defects were substantially larger in the pellets that were HIPed for 15 min.

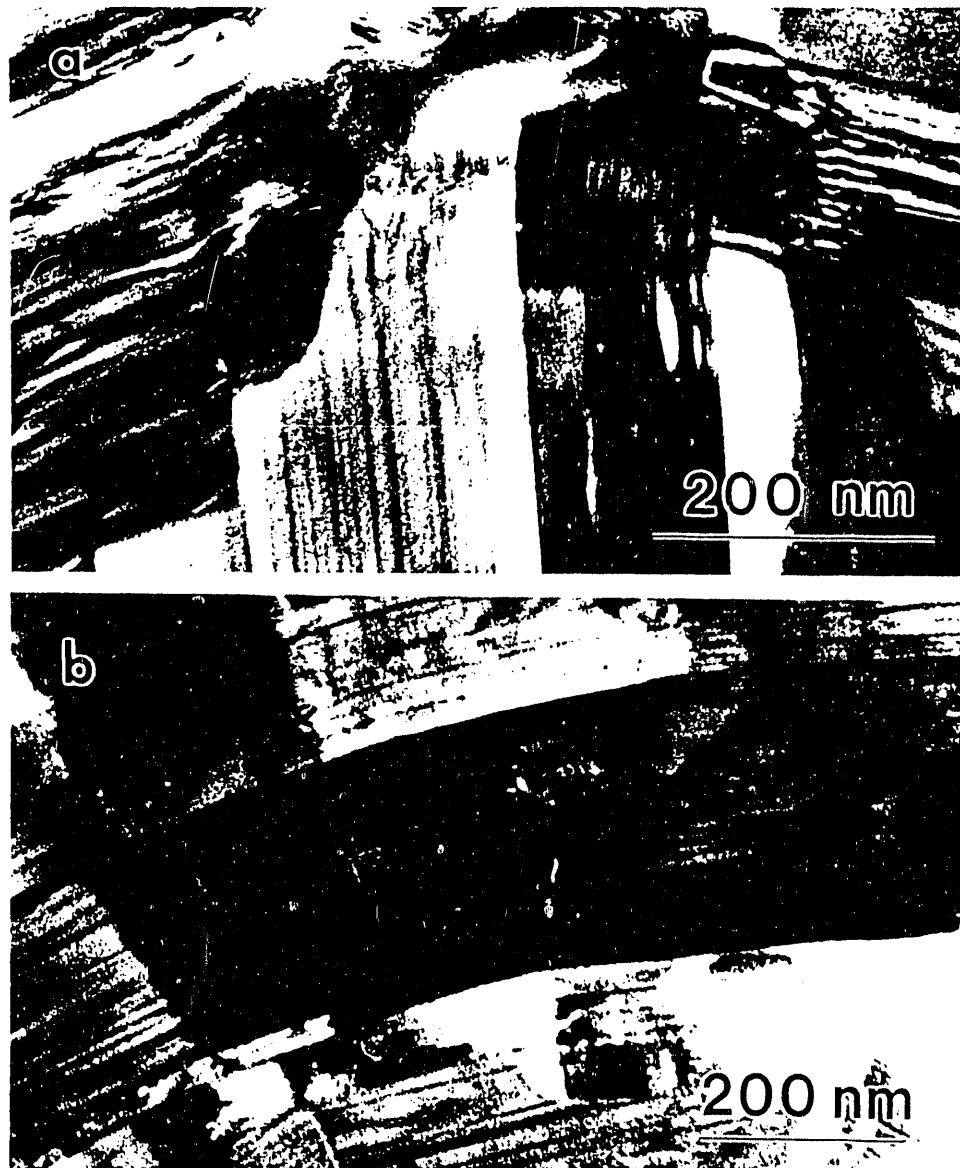


Fig. 2. TEM photomicrographs of 2212 HIPed for (a) 15 min and (b) 120 min.

Representative magnetic hysteresis measurements are shown in Fig. 3. The specimens that were HIPed for 15 min exhibited much larger loops than did the specimens that were HIPed for 120 min. At 35 K, however, the loops of both specimens were completely collapsed at fields less than  $\approx 10^3$  G.

The data indicate that dislocations and planar faults are effective flux pinning centers in 2212 at lower temperatures. It has been reported that high concentrations of dislocations and planar faults are effective in pinning flux in  $\text{YBa}_2\text{Cu}_3\text{O}_x$  [19-22]. There has also been some indication that pinning from dislocations may occur in  $(\text{Bi,Pb})_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_x$ ; however, no magnitude was given for that effect, and that conclusion was drawn from comparison of samples with vastly different densities and textures [5]. The flux creep behavior of high-temperature superconductors is highly dependent on orientation [23], and thus the effectiveness of dislocations in pinning flux effectively in Bi-Sr-Ca-Cu-O superconductors had not been convincingly demonstrated. The textures and grain sizes of the 2212 pellets HIPed in this study were virtually identical. Furthermore, the transport  $J_c$  measurements indicated that most of the grains must have been weakly linked. These specimens therefore allowed for direct comparisons of intragranular  $J_c$ .

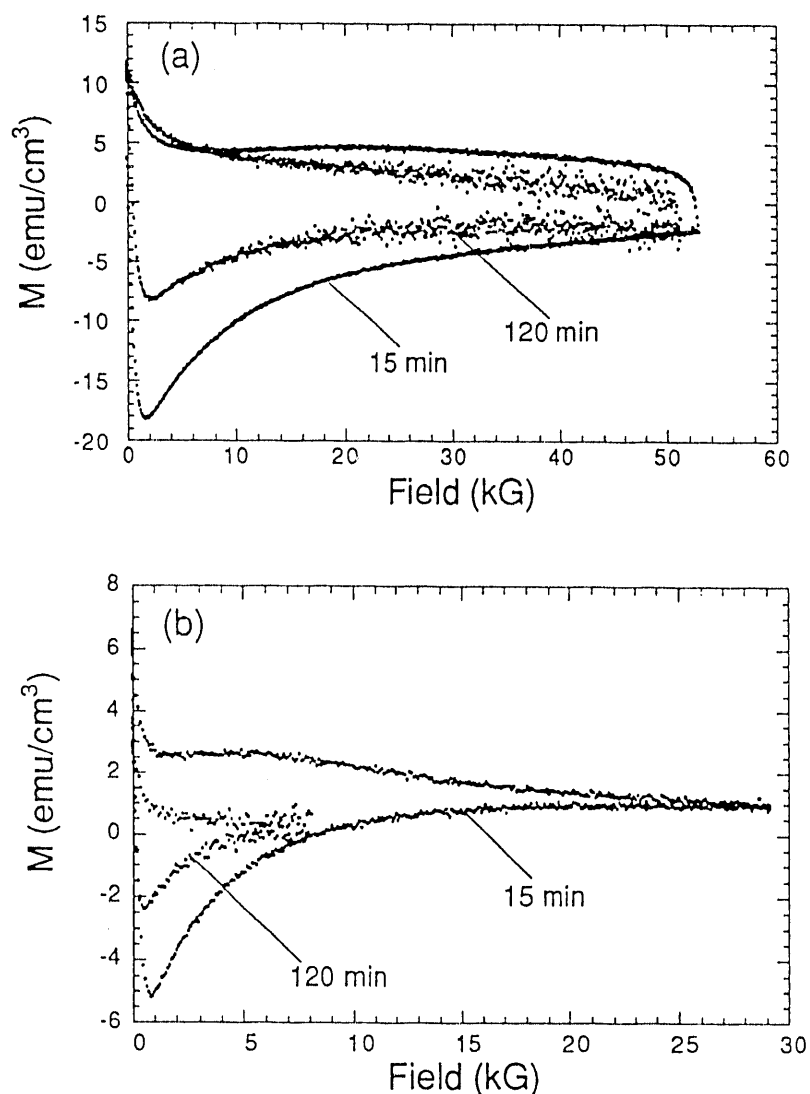


Fig. 3. Magnetic hysteresis data at (a) 5 K and (b) 25 K for 2212 HIPed 15 or 120 min.

None of the specimens exhibited appreciable flux pinning at temperatures greater than 30 K. This result is in agreement with the model of Kim et al. [24], who hypothesize that the irreversibility behavior of a high-temperature superconductor is dependent on the coupling between Cu-O layers. The 2212 superconductor exhibits poor coupling between Cu-O layers at temperatures greater than  $\approx 35$  K. As a consequence, pancake vortices can form and flux pinning centers lose their effectiveness.

It is not clear at this time whether a process such as HIPing can be used to improve the transport  $J_c$  of Bi-Sr-Ca-Cu-O superconductors through creation of pinning centers. High  $J_c$  values have been reported at 4.2 K for HIPed Ag-clad 2212 tapes [25]. However, the extent to which the HIPing improved flux pinning in these tapes was not investigated.

## CONCLUSIONS

Fully dense, modestly textured 2212 pellets were made by HIPing. The transport  $J_c$  values of these pellets were very low. Dislocations and planar faults introduced by the HIPing increased the intragranular  $J_c$  significantly at temperatures less than  $\approx 30$  K.

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