## Superconducting properties of aligned crystalline grains of  $Y_1Ba_2Cu_3O_7 - \delta$

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A new method has been used to produce uniaxially aligned small single crystals of  $Y_1Ba_2Cu_3O_{7-\delta}$ . The superconducting critical current deduced from magnetization data is strongly field dependent and highly anisotropic. In the basal plane at  $T = 4.2$  K we estimate that the critical current at zero field is in excess of  $10^7$  A/cm<sup>2</sup>.

Despite the fact that anisotropy is widely regarded as being a key feature of the 90-K ceramic superconductors, the first single-crystal magnetic data on such materials were reported only very recently, by Dinger, Worthington, Gallagher, and Sandstrom.<sup>1</sup> They used specially processed crystals of Y<sub>1</sub>Ba<sub>2</sub>Cu<sub>3</sub>O<sub>7</sub>-<sub>8</sub> that had a volume of  $\sim$  10<sup>-5</sup> cm<sup>3</sup> and a transition temperature of  $\sim$ 85 K. Critical-current densities were strongly anisotropic with the largest value reported being  $\sim 3 \times 10^6$  A/cm<sup>2</sup> for current flow in the basal plane. It is very desirable to obtain this type of information for crystals produced by the standard processing route that have transition temperatures in excess of 90 K and are much smaller  $(-10^{-11}$  $cm<sup>3</sup>$ ). Since the magnetization of a single crystal of this size is too small to measure, we have devised a method to permanently align quantities of them in a nonmagnetic matrix.

If a crystal possessing anisotropy in its normal-state paramagnetic susceptibility is placed in a magnetic field, it will tend to align so that the direction having the greatest magnetic susceptibility lies along the field. The torques involved are small, but a detailed analysis<sup>2</sup> indicates that alignment of Y<sub>1</sub>Ba<sub>2</sub>Cu<sub>3</sub>O<sub>7</sub> -  $_{\delta}$  can be rapidly established with available magnetic fields and a suitably chosen medium. (Torques due to shape anisotropy also exist, but can be shown<sup>2</sup> to be negligible for the case of  $Y_1Ba_2Cu_3O_{7-\delta}$ . Our small crystals were produced by grinding an essentially single-phase sample whose lowfield magnetic properties and grain size were reported previously.<sup> $3$ </sup> The roughly spherical single-crystal grains of the resulting powder were  $2-4 \mu m$  in diameter. The original (resistive)  $T_c$  was 92.3 K with a 10%-90% breadth of 1 K and the (magnetic)  $T_c$  of our final aligned sample was identical to this within the experimental resistive measurement uncertainty of 0.5 K. A permanently aligned sample was produced by vigorously mixing the powder with Duro TM-51, a commercial epoxy which is weakly diamagnetic,  $2$  then curing for 15 min in a magnetic field of 9.4 T at room temperature. We expected that uniaxial ordering would be produced with the  $c$  axis lying along the field

direction and with the  $a$  and  $b$  axes having random orientations in the plane perpendicular to the field. An x-ray powder pattern obtained with the incident beam oriented perpendicular to the suspected  $c$  axis is shown in Fig. 1(b) and compared with that from an epoxy-embedded sample prepared in the same manner but without applying the magnetic field [Fig. 1(a)]. All the  $(hkl)$  reflections with



FIG. 1. (a) X-ray powder diffraction pattern from an epoxyembedded sample of  $Y_1Ba_2Cu_3O_{7-\delta}$  powder. The most conspicuous  $(hkl)$  planes with nonzero values of  $l$  are indexed. (b) Powder pattern obtained from an aligned sample, as described n the text. The (110) peak has been truncated to  $\frac{1}{3}$  of its actual height.

nonzero I values are absent in the ordered sample, confirming the anticipated  $c$ -axis orientation. [Note that only the most conspicuous reflections are indexed in Fig. 1(a).] The Laue diffraction pattern obtained with the incident beam close to the  $c$  axis consisted of a single sharp spot, confirming the high degree of  $c$ -axis alignment. The sample of this material that was used for our magnetization studies had dimensions of  $3 \times 2.5 \times 2$  mm<sup>3</sup> and contained  $4.0 \times 10^{-3}$  cm<sup>3</sup> of Y<sub>1</sub>Ba<sub>2</sub>Cu<sub>3</sub>O<sub>7-8</sub>. Magnetization data were obtained with a commercial superconducting quantum-interference device (SQUID) susceptometer. Sample orientation with respect to the magnetic field was estimated to have an angular uncertainty of  $\pm 3^{\circ}$ .

Figure 2(a) shows the magnetic moment measured at 4.2 K with the magnetic field perpendicular  $(m<sub>1</sub>)$  and parallel  $(m_{\parallel})$  to the basal plane. The dotted line labeled  $m_0$  represents the moment that would be observed if all the ceramic material was in the form of perfectly diamagnetic spheres. The actual moments are smaller in both field directions. This is because the small crystal size results in a significant degree of field penetration. Although the penetration depth is clearly anisotropic the possible presence of crystal-shape anisotropy complicates the further analysis of this weak-field data. These geometrical complications are not present at higher fields, and Fig. 2(b) displays the first magnetic data obtained for any 90- K superconductor that permits an unambiguous determination of an upper critical field, in this case  $H_{c2, \perp}$ . In



FIG. 2. (a) Sample magnetic moment as a function of field at T=4.2 K for *H* perpendicular and parallel to the basal plane  $(m_{\parallel}$  and  $m_{\perp}$ , respectively).  $m_0$  represents the moment that would be observed for spheres of perfectly diamagnetic material. (b) Sample magnetic moment  $m<sub>+</sub>$  as a function of field at  $T = 89.5$  K.

polycrystalline material, anisotropy destroys the linear magnetization behavior near the upper critical field and makes an accurate assignment of  $H_{c2}$  impossible.<sup>5</sup> By contrast, the classic<sup>6</sup> type-II linear behavior below  $H_{c2,1}$ is clearly apparent in Fig. 2(b). (The slope above  $H_{c2, \perp}$  is due to the strong paramagnetic susceptibility of the  $Y_1Ba_2Cu_3O_{7-\delta}$  which is only partially canceled by the diamagnetic contribution from the epoxy.) From measurements close to  $T_c$  we obtained

$$
dH_{c2,\perp}/dT|_{T=T_c} = -2.3 \times 10^3 \,\text{Oe/K} \, ,
$$

substantially smaller than the rough estimate made for polycrystalline samples.<sup>7</sup> We have also found that the field dependence of  $m<sub>⊥</sub>$  exhibits (small) changes of slope at fields significantly larger than  $H_{c2,\perp}$  and these are beng investigated further.  $H_{c2,II}$  was not studied in the present work.

Finally, Fig. 3 shows the large hysteresis loop obtained for  $m_{\perp}$  at 4.2 K and half of the much smaller one obtained for  $m_{\parallel}$  at the same temperature. We note in passing that evidence was obtained for flux creep in certain sections of these loops, as in Ref. 1. In our work, data were taken  $\sim$ 10 min after latching the field and the points plotted are believed to be close to the equilibrium values. The overall shape of the  $m_{\parallel}$  loop is very similar to that for  $m_{\perp}$ . However, the magnitudes of the observed moments are less by a factor of 10, the same anisotropy ratio as reported in Ref. 1. We can also evaluate a critical current from the critical-state model applied to a spherical grain,<sup>8</sup> viz.  $J_c = (17\Delta M/R)$  A/cm<sup>2</sup>, where R is the crystal radius in cm  $(-1.5 \times 10^{-4})$  and  $\Delta M$  is the magnetization hysteresis at some particular field in emu/cm<sup>3</sup>. At  $T = 4.2$ <br>A and  $H = 0$  we obtain  $J_c^2 \sim 2 \times 10^7$  A/cm<sup>2</sup> and  $J_c^{\parallel}$   $\sim$  2  $\times$  10<sup>6</sup> A/cm<sup>2</sup>. The  $J_c^{\perp}$  value is not far below the ultimate depairing limit for 90-K materials, namely,  $\sim 10^8$ A/cm<sup>2</sup>. Both  $J_c^{\perp}$  and  $J_c^{\parallel}$  are also significantly higher than reported by Dinger et al.,<sup>1</sup> who used crystals that were



FIG. 3. Magnetic moment hysteresis loops at  $T = 4.2$  K. The complete loop for  $m_{\perp}$  is shown (data points, open circles). Half of the much smaller loop for  $m_{\parallel}$  is indicated by the open triangles.

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essentially free of defects. It is therefore tempting to speculate that the higher values reported here may be the result of flux-pinning sites introduced into the crystals during grinding: Although we have not yet made a systematic study, we note that both critical currents fell off' rapidly with increased field and temperature. At 4.2 K they appear to have the same (exponential) dependence on field, both dropping about an order magnitude on increasing the field from 0 to 15 KOe.

In conclusion, we have discovered a simple technique for producing uniaxial alignment in high- $T_c$  materials and

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have applied it to small single crystals of  $Y_1Ba_2Cu_3O_7 - \delta$ . Our method should be of value for many fundamental investigations of high- $T_c$  superconductors, and its further development might facilitate the eventual production of aligned bulk ceramics with useful critical currents.