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LETTER TO THE EDITOR

Growth and magnetic characterisation of $Bi_2Sr_2CaCu_2O_y$ crystals

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Abstract. In this Letter we describe the growth and magnetic characteristic of single crystals of $Bi_2Sr_2CaCu_2O_y$. Small (weight $\leqslant$ 1 mg) crystals were grown from a potassium chloride flux and their composition was determined by EPMA. The crystals were found to be superconducting (T_c = 87 K). H_{c1}(T) has been obtained from the deviation of linearity of the M–H curves. Results of ZFC, FC and remanent magnetisations show strong flux trapping and a behaviour for $T <$ 20 K indicative of a melting of the vortex lattice.

The $Bi_2Sr_2Ca_{n-1}Cu_nO_{4+2n}$ and $Tl_2Ba_2Ca_{n-1}Cu_nO_{4+2n}$ families of high-temperature superconductors show an unusual magnetic behaviour of the mixed state. Magnetic-hysteresis measurements reveal poor intra-grain current-carrying properties. In the mixed state strong relaxations of the screening currents are observed (giant flux creep). For the $Bi_2Sr_2CaCu_2O_y$ (Bi2212) superconductor the width of the hysteresis curves, extrapolated to zero-field sweep rates ($dB_a/dt = 0$), become essentially zero above about 30 K [1]. The motion of the vortices can be described well by a diffusion relation indicating weak pinning. Mechanical measurements by Gammel and co-workers [2] on Bi2212 single crystals indicate a drastic change in the shear modulus of the vortex lattice at 30 K. It has been suggested that this behaviour is caused by a melting of the vortex lattice (similar to the behaviour of thin superconducting films in perpendicular applied fields [3]), possibly with a Kosterlitz–Thouless type transition [4] or more likely a Lindemann-type melting.

We are engaged in a programme of magnetic studies of these effects. The superconducting properties of these high-T_c superconductors are highly anisotropic. Thus, for a detailed investigation we need to use single crystals. In this Letter we present our initial results of magnetic measurements on Bi2212 single crystals prepared by growing from the melt.

Single crystals of $Bi_2Sr_2CaCu_2O_y$ were grown from a potassium chloride flux. The starting composition consisted of the single-oxide components calculated to correspond to 20 wt% $BiSrCaCu_2O_x$ plus 80 wt% KCl. The mixture of oxides and KCl was heated in a 20 cm^3 platinum crucible covered with a lid, in a furnace with silicon carbide elements. The mixture was heated to 910 °C at a rate of 100 °C h^{-1} and held at this

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Table 1. EPMA results of $Bi_2Sr_2CaCu_2O_y$ crystals.

	at. %				
Crystal	Bi	Sr	Ca	Cu	O
A	18.5	14.5	7.8	21.7	37.7
B	19.5	14.5	8.5	19.9	37.7

temperature for 0.5 h. Subsequently the melt was cooled at 30 °C h^{-1} to 890 °C, then at 1 °C h^{-1} to 810 °C, followed by a rapid cooling to room temperature. The melt was soaked in water to dissolve the flux. The crystals were thin flakes with an area of about 1 mm^2 and thicknesses of up to 50 μm. The x-ray powder pattern of selected crystals corresponded to that of $Bi_2(Sr, Ca)_3Cu_2O_y$.

EPMA (electron-probe microanalysis) results were obtained of these crystals with a Jeol JXA-8600. The specimens were placed on a carbon-block specimen holder and small areas (10 μm × 10 μm) free from surface contamination were analysed. Results for two crystals from one batch are given in table 1. The data given for each crystal is an average of the EPMA results over five different areas of the crystal. The error in oxygen estimation can be very large. The measured compositions of our crystals agree well with results of other groups [5, 6]. The results for the different areas vary by less than 10% and we thus conclude that there are no strong compositional variations between different parts of the crystals. From our EPMA data we can not exclude composition variations on a length scale smaller than 10 μm.

Magnetic measurements were carried out using a home-built SQUID magnetometer over a temperature range $4\ K < T < 250$ K. The maximum applied field is 0.1 T. The magnetisation measurements reported in this Letter were performed on a single crystal with a mass of 1.01 mg, a thickness of 46 μm and an area of 3.25 mm^2. For most measurements presented in this Letter, the magnetic field was applied parallel to the plane of the crystal ($H_a \perp c$ axis), thus minimising the demagnetisation effects ($N_\perp$ = 0.034).

Figure 1 shows the zero-field-cooled (ZFC), field-cooled (FC) and remanent (rem) magnetisations as a function of temperature. The applied magnetic field was 1.0 mT. The superconducting transition temperature, T_C, determined from the onset of diamagnetism, was found to be 86 K (confirming that these crystals are Bi2212). The $M_{ZFC}(T)$ did not show saturation at low temperatures, unlike similar measurements on $YBa_2Cu_3O_{7-\delta}$ single crystals. At low temperatures (6 K) we obtained a volume susceptibility of −0.26 from M_{ZFC}, suggesting that this crystal is not a homogeneous superconductor. Magnetisation measurements on other crystals from the same batch gave similar results. The Meissner fraction was only 4%: M_{FC} in an order of magnitude smaller than M_{ZFC}, which has been usual in the Bi family of superconductors [7]. We also note that the relation $M_{FC} - M_{ZFC} = M_{rem}$ (as found for $YBa_2Cu_3O_{7-\delta}$ by Malozemoff and co-workers [8]) was found to be invalid even at the lowest temperatures (deviation about 10% at 6 K). In the derivation of this relation, Malozemoff and co-workers [8] assume a full exclusion of flux in a ZFC measurement. This is clearly not the case in our measurements on Bi2212. The rather high value for $M_{rem} \simeq M_{ZFC}$ clearly reveals strong flux trapping. This and the lack of full flux exclusion suggest that some type of inhomogeneity is present in these crystals. In contrast, our above-mentioned

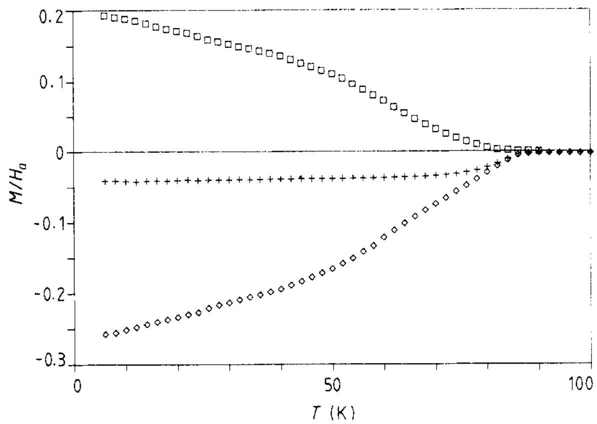

Figure 1. ZFC (◇), FC (+) and remanent (□) magnetisations for Bi2212 crystal with B_a = 1.0 mT ($B_a \perp c$ axis).

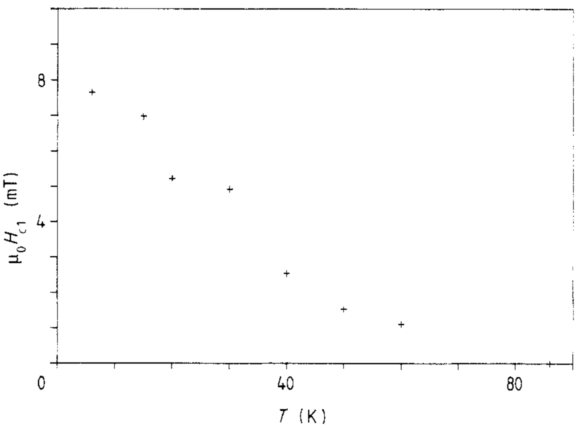

Figure 2. Lower critical field of Bi2212 for $B_a \perp c$ axis.

EPMA results give a homogeneous composition. We suggest that inhomogeneities in these materials are on a length scale smaller than that being probed in our EPMA studies (thus smaller than 10 μm).

The lower critical field, H_{c1}, was determined from the deviation from linearity of the M–H curve at a given temperature. This is only a rough estimate, as the M–H curves show a gradual deviation from linearity. We took a 10% deviation from a line determined by the initial slope as a criterion for H_{c1}. In figure 2 we have plotted $H_{c1}(T)$. These results have been corrected for shape effects (the correction of the magnetic field is about 1%). The extrapolated value for $\mu_0 H_{c1}$ at 0 K is 9 mT ($H_a \perp c$ axis). The unusual shape of the $H_{c1}(T)$ curve showing a roughly linear dependence is similar to that found by Batlogg and co-workers [9]. Results for applied fields along the orthorhombic axis ($H_a \parallel c$ axis) gave similar results ($\mu_0 H_{c1}(0) \simeq 12$ mT). The correction due to the demagnetising factor is large for $H_a \parallel c$ axis ($N_\parallel = 0.932$).

For applied magnetic fields larger than about 5 mT, $M_{rem}(T)$ showed an upturn for temperatures below 20 K (figure 3). This behaviour of the remanent magnetisation was

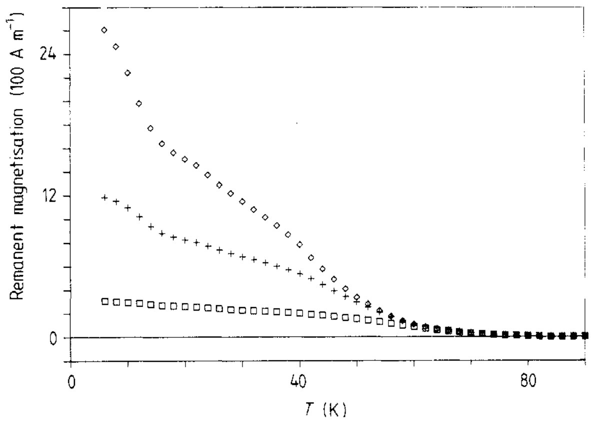

Figure 3. Remanence with increasing temperature after cooling down in fields of 2.21 mT (□), 9.73 mT (+) and 24.8 mT (◇) for $B_a \perp c$ axis.

found to become prominent as the magnetic field was increased above H_{c1}. The trapped magnetic flux, which gives rise to the remanence, is maintained by screening currents in the crystals. Our measurements reveal a strong decrease of these currents between the lowest temperatures and 20 K. A comparison of this data with our earlier magnetic hysteresis measurements on powders [1], reveals that this effect corresponds to a strong decrease of the critical current density in this temperature range. These observations reveal a strong decrease of the pinning force around 20 K, which is possibly due to a melting of the vortex lattice.

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