

Magnetic properties of $Y_1Ba_2Cu_3O_7$ single crystals and ceramics at $T/T_c > 0.85$

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The penetration depth of the magnetic field, $\delta_0 \approx 1.03 \times 10^{-5}$ cm and the critical current density, $j_c \approx 2.3 \times 10^5$ A/cm² (at $T/T_c \approx 0.98$) have been determined from the results of a magnetometric study of superconducting single crystals at $T \gg 77$ K, using a high- T_c SQUID.

Extensive studies of the magnetic properties of high- T_c oxide superconductors showed that the variation of the magnetization of single crystals and ceramics as a function of time, of the magnetic field strength, and of the temperature is similar¹ at least when the field penetrates the sample ($H > H_{c1}$). This result is a consequence of the fact that high- T_c superconducting crystals are inhomogeneous systems which are similar (like the ceramics) to the spin glass.² It was expected that a comparison of the behavior of the magnetic properties of these systems near T_c (the region which is most sensitive to the weak links) would identify the difference between these systems. In the present study we have detected a qualitative difference in the magnetization-vs-temperature curves of the superconducting ceramics and crystals at $T_c > T > 0.85$ in fields $H_c = 10^{-3}$ – 10^0 G.

The measurements were carried out using a device which consisted of an asymmetric two-hole rf SQUID,³ used as a magnetometer, and a magnetic screen⁴ made from $Y_1Ba_2Cu_3O_7$ ceramic. The intrinsic noise of the SQUID was $\sim 2 \times 10^{-4}$ Φ_0 /Hz^{1/2} over a range 1– 10^2 Hz; the field penetration into the screen (at 77 K), which was appreciable at the sensitivity level of the SQUID, was detected at $H_c > 7$ G. The experimental setup is shown schematically in the inset in Fig. 1. The temperature of the sample was varied between ~ 77 K and ~ 150 K, while the SQUID was held at liquid-nitrogen temperature. A turn with a current, situated at the site of the sample, was used to calibrate the device.

We studied samples of high- T_c superconductors of solid ceramic with dimensions $\sim 1 \times 1 \times 0.3$ mm and six single crystals with typical dimensions $\sim 0.8 \times 0.8 \times 0.03$ – 0.05 mm, synthesized by the method described in Ref. 5. The study was carried out in the Institute of Physics Problems, the N. S. Kurnakov Institute of General and Inorganic Chemistry, and the Institute of Chemistry of the Academy of Sciences of the USSR, Moscow. The temperature T_c of the onset of the superconducting transition (which was measured for these samples at $H_c \parallel c$) was in the range 89–93 K and the width of the superconducting transition was 0.5–2 K (we note that the resistive measurements generally gave a considerably smaller width). In this geometry the fraction of the magnetic moment M_{FC} , which appears when the sample is cooled in an external static field H_c , amounted, at $H_c < 10^{-2}$ G, to 10–50% of the ZFC moment which was

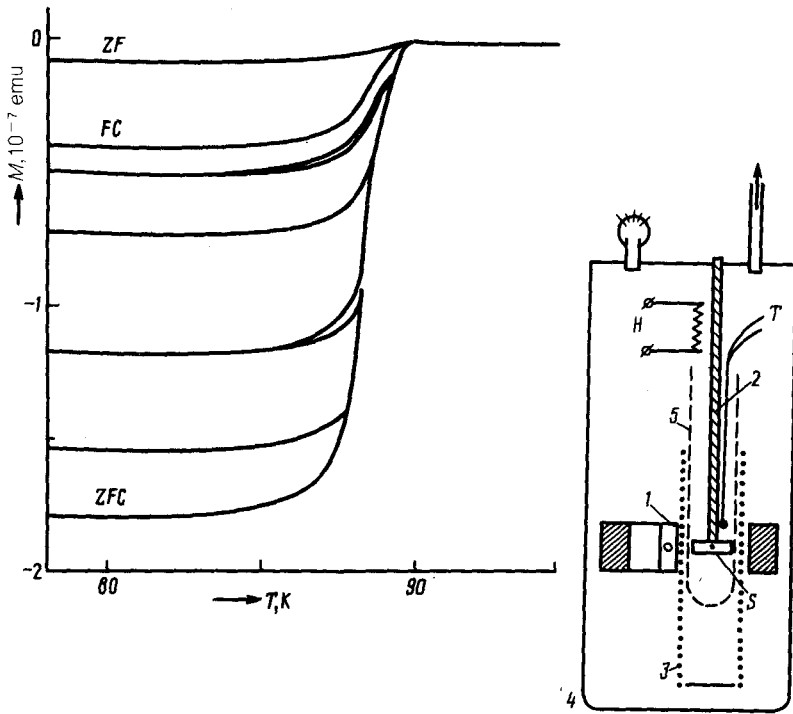


FIG. 1. Trace of the variation of the magnetic moment of a single crystal as a result of temperature cycling between 77 K and T (as T gradually increases to T_c). ZF—Magnetic moment produced upon cooling the sample in a residual field of the device ($\sim 10^{-3}$ G); FC—the same magnetic moment in a field ($H_c \approx 2 \times 10^{-2}$ G); ZFC—the moment which the sample acquires upon cooling as in ZF by applying the same field at $T < T_c$. Inset—Experimental setup. 1—Two-hole high- T_c SQUID; 2—heat sink-sample mount S; T—thermocouple; H—heater; 3—solenoid H_c ; 4—vacuum container; 5—radiation shield between the sample and the SQUID.

produced as a result of the application of a field at $T < T_c$ after the sample was cooled in the absence of a field (Fig. 2). Upon increasing the field above 0.1 G, this fraction of the magnetic moment decreased appreciably (to several percent at 2 G), while the ratio ZFC/H_c remained constant. This behavior is probably attributable to the particular features of the Meissner effect in the samples with a large demagnetizing factor ($n \sim 0.95$ in our case), such as the flux trapping by surface edge currents. No time-dependent effects were detected under our experimental conditions; if the first 0.5-1 min after the field has changed are subtracted, the upper limit of dM/dt will be $< 10^{-4} M_0/h$, a value at least two orders of magnitude smaller than the values obtained in Ref. 6. We assume that the absence of relaxation stems from the low fields which we used ($H < H_{c1}$). The absence of the time-dependent effects has made it possible to determine (by means of thermal cycling, as shown in the inset in Fig. 2) the dependence $TRM = ZFC - FC = f(T)$. This dependence for several samples is shown in Fig. 2 for $H_c \approx 10^{-2}$ G. While the ceramic samples exhibited a continuous (nonlinear) decrease of the moment beginning at 77 K, the single crystals showed no appreciable

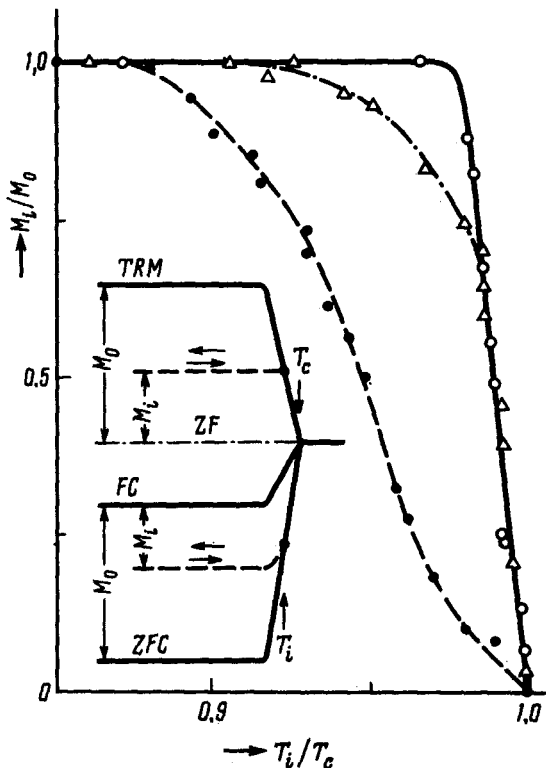


FIG. 2. A comparison of the variation with temperature of the intermediate warming T_i of the magnetic moment (which is associated with the peripheral screening currents TRM and ZFC-FC) for single crystals and ceramics. Circles and triangles—the results for two single crystals; points—the results for the ceramic (the relation TRM = FC - ZFC applied over the entire measurement range). The inset shows the method used to construct the curves in Fig. 2.

variation of this quantity at $T < 85$ K. At higher temperatures, however, the moment decreased rapidly, generally in a stepwise manner at the beginning of the transition to the normal state. The magnetic moment which we have studied apparently depends on the magnitude of the peripheral superconducting currents which are excited in the sample, and it decreases when the screening current reaches the value of the critical current I_c . The critical current in a ceramic is determined by the properties of the weak contacts; in crystals this current is much higher. The current which screens the field therefore reaches the value $I_c(T)$ only near the transition to the normal state. The steps on the M_i/M_0 curves show that the superconductivity is disrupted in a nonuniform manner because the current is concentrated at the corners of the sample. Estimates of the critical current density based on the relation

$$j_c = H_e / [(I - n)\delta(T)] \quad (1)$$

and those based on the results of a calibration experiment give the same value, $j_c \approx 2.3 \times 10^5$ A/cm² at $I - T/T_c \approx 0.02$, which is close to the Ginzburg-Landau depairing current.

In the case of the orientation $H_c \parallel ab$, the difference between FC and ZFC moments is typically much smaller. In samples of higher quality we have FC > 90% (ZFC), and in some crystals this value decreases to 50%. In the case of all test

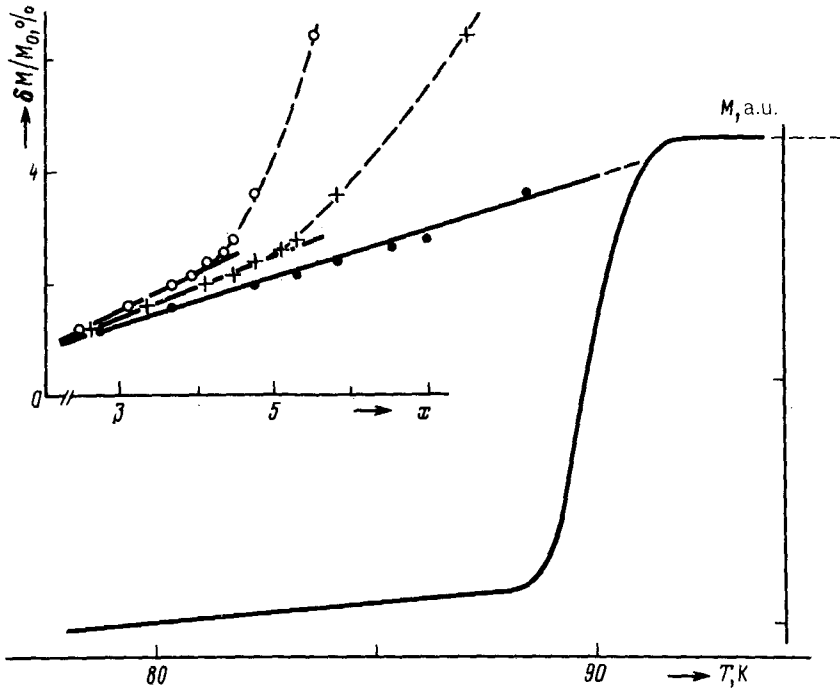


FIG. 3. Typical temperature dependence of the Meissner moment (FC) of a single crystal at $H_c \parallel ab$. The temperature variation rate was no greater than 0.2 K/min. The inset is a comparison of the temperature dependence of $\delta M/M_0$, where $\delta M = M(T) - M(80 \text{ K})$, and M_0 is the total variation of the magnetic moment between $T = 77 \text{ K}$ and $T > T_c$, where x varies as $[1 - (T/T_c^*)^4]^{-1/2}$, see Eq. (2). The results of the analysis of the data are represented by the circles, the crosses, and the points for three parameter values, $T_c^* = 89.4, 89.1,$ and 88.8 K . The values of δ_0 , determined in the region $[(\delta M/M_0) \propto x]$ for these parameter values are $\approx (16, 14,$ and $10.3) \times 10^{-6} \text{ cm}$, respectively.

samples, the temperature T_c at which the magnetic moment begins to change sharply at $H_c \parallel c$ exceeded the corresponding value at $H_c \perp c$. This difference δT_c was in the range 0.5–2 K, depending on the quality of the sample. The samples with the highest values of δT_c exhibited a noticeable hysteresis on the FC curves near the superconducting transition. The hysteresis on the FC(T) curves, the finite value of δT_c , and the discrepancy between the values of ZFC and FC, which were observed in some crystals, show that these crystals have nonuniform properties which (as was determined) are seen most clearly when measurements are carried out in $H_c \perp c$. A similar conclusion was reached previously on the basis of the results of an experimental study of crystals in a microwave field.⁷

At $T < T_c$ the temperature dependence of the magnetic moment even in highest-quality crystals (see Fig. 3) would be reasonable to attribute to the dependence of the magnetic-flux penetration depth $\delta_{ab}(T) = \delta(T)$ on the temperature, since at $\delta \ll a$ we have

$$M(T)/M_0 \approx 1 - 2\delta(T)/a, \text{ where } \delta(T) = \delta_0 x, \quad x = [1 - (T/T_c^*)^4]^{-1/2}, \quad (2)$$

and a is the thickness of the sample. In the analysis of the experimental dependences the parameter value T_c^* was chosen in such a way that $\delta M(T)/M_0$ would coincide with relation (2) over the broadest range of the values of x . Such an analysis is shown in the inset in Fig. 3. A change in the value of T_c^* from 89.4 K to 88.8 K causes the region x , in which $\delta M(T)/M_0 \propto x$, to increase by an order of magnitude, from ~ 2 to ~ 20 . The value of δ_0 for $T_c^* = 88.8$ K is 10.3×10^{-6} cm. The value $T_c^* = 88.8$ K determined in this manner is equal to the temperature at which M_i/M_0 begins to decrease significantly in $H_c \parallel c$ (Fig. 2). In the case of a crystal characterized by $FC \approx 50\%$ (ZFC) the estimated value of δ_0 is twice as large.

The value of δ_0 which we obtained is in agreement with the values determined in a similar way in Ref. 8 but is much smaller than the value obtained for a polycrystalline sample⁹ in a field higher than that used by us by a factor of 1.5. Taking into account the imperfection of the samples we studied, the value of δ_0 can be interpreted only as an approximate depth to which the field penetrates a $Y_1Ba_2Cu_3O_7$ crystal.

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