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Anomalous magnetization peak effect in spiral-grown $Bi_2Sr_2CaCu_2O_y$ crystals

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Magnetic hysteresis loops were measured on spiral-grown $Bi_2Sr_2CaCu_2O_y$ (Bi-2212) crystals. An anomalous peak effect at a magnetic field of 1000–2000 Oe was observed both in high-T_c (86 K) and oxygen underdoped (T_c=76 K) spiral-grown crystals between 20 and 40 K. The peak effect was observed to be stronger than that induced by oxygen vacancies, defect dislocation networks reported in Bi-2212 crystals. Further, the anomalous peak almost completely disappeared after removing growth spiral patterns from the crystal surface. Edge barriers associated with the growth spirals are suggested to be responsible for the strong peak effect for the spiral-grown Bi-2212 crystals and not oxygen vacancies or screw dislocations [S0163-1829(97)52206-5]

Flux pinning in the high-T_c superconductors is very important, both from a theoretical and practical point of view. Critical current density in Y-Ba-Cu-O and Bi-Sr-Ca-Cu-O can be improved by introducing pinning centers. Low-angle grain boundaries,[1] stacking faults,[2] twin boundaries,[3] impurity phase,[4] growth dislocations,[5,6] and oxygen vacancies[7] are some of the potential pinning centers. Among them, screw dislocations were found to be possible effective pinning centers in the YBa_2Cu_3O (Y-123) thin films.[5] Spiral growth induced by the screw dislocations has been widely observed in the Y-123 thin and thick films, and in melt-grown Y-123 single crystals.[5,6,8,9] The spiral growth was suggested as the intrinsic growth mechanism in the Y-123 compound. Among investigations of flux pinning, the peak effect, an anomalous magnetization peak in the magnetic hysteresis loop (M-H) for $0.2 \leqslant T/T_c \leqslant 0.4$, has been observed for $Bi_2Sr_2CaCu_2O_y$ (Bi-2212) single crystals.[10–13] Oxygen vacancies[7] and defect dislocation networks[14] were reported to be responsible for the origin of the peak effect. The spatial variation of oxygen ordering may well account for the peak effect in Y-123. Recently, it was reported that edge barriers can produce the peak effect and that they are important only when bulk pinning is weak.[13,15–17] No studies on the relation between the growth spirals of Bi-2212 and flux pinning have been carried out to date, because of the commonly accepted idea that spiral growth does not occur on these materials. The structure of Bi-Sr-Ca-Cu-O is found to be more two dimensional in the ab plane compared to Y-123. The formation of spiral dislocations in the Bi-Sr-Ca-Cu-O is extremely difficult, because the bonding between adjacent Bi atoms in the double Bi-O layer is very weak. Therefore, the growth mechanism of the Bi superconductors is probably two-dimensional nucleation and layer-by-layer growth, as widely observed for the Bi-Sr-Ca-Cu-O in all forms of thin and thick films, bulk ceramics, and single crystals produced by various techniques. Very recently, we have found that the crystal growth of Bi-2212 single crystals is mainly governed by a spiral-growth mechanism when the crystal is grown on the flux surface of KCl.[18] In this paper, we report a strong peak effect at 20–40 K for the spiral-grown $Bi_2Sr_2CaCu_2O_y$ crystals with slight oxygen underdoping and high underdoping. We show that edge barriers due to the growth spirals instead of screw dislocations and oxygen vacancies are responsible for the very strong peak effects for the spiral-grown Bi-2212 crystals.

The spiral-grown Bi-2212 crystals described in this paper were grown using KCl flux as reported previously.[18] A small amount of Bi-Sr-Ca-Cu-O material, in a proportion of 5 wt. % Bi-2212 to 95 wt. % KCl flux was used for the crystal growth. The crystals were grown on the KCl flux surface and the growth was observed to be mainly controlled by a spiral-growth mechanism. Single, double, and multiple spirals with different shapes and the same and opposite signs were observed on the crystal surface. The densities of the growth spirals varied with growth conditions. Although some crystals with dimensions of 1×1 mm^2 only contained 3–5 spirals, there were several hundred turns for each of the spirals. Three crystals were chosen for the measurements. Sample 1 with four hexagonal spirals on its surface visible by optical and atomic force microscopy. The same dimensions were $1.2\times0.6\times0.02$ mm^3 and it was slightly oxygen underdoped with a T_c of 86 K. Sample 2, a typical spiral-grown crystal with dimensions of about $1\times0.7\times0.035$ mm^3, was oxygen underdoped (T_c=76 K), with 5 hexagonal spirals visible on the surface. Sample 3, growth by a two-dimensional layer-by-layer growth mechanism using the self-flux method, was also oxygen underdoped, with the same T_c of 76 K as sample 2. These three samples were determined to be single 2212 phase by x-ray diffraction. Measurements of the hysteresis loops were performed by a Quantum Design Physical Property Measurement System, using the extraction method. The sample was translated with a constant speed (1 m/sec) right through two sets of coils, inducing a signal in them according to Faraday's law. The coils were arranged in a first-order gradiometer configuration. The magnetic moment was calculated from the resulting voltage-time response. It takes about one second to perform a measurement. The sweep rate of the field for all the hysteresis loops was 2.1 Oe/second. T_c was determined from ac susceptibility measurements using the same system in ac measurements mode. The amplitude and frequency of the alternating field were 0.1 Oe and 117 Hz, respectively.

The M-H loops for sample 1 and sample 2 were measured at 5, 15, 20, 30, 40, and 50 K. An anomalous magnetization peak appears clearly between 20 and 40 K as shown in Figs.

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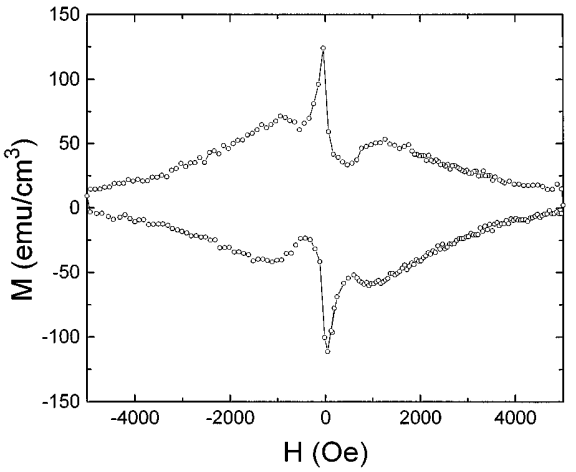

FIG. 1. M-H loop for spiral-grown Bi-2212 crystal (sample 1) with T_c of 86 K, at 30 K.

1 and 2. The field of the anomalous magnetization peak, H_{peak}, was about 1000 Oe for both samples between 30 and 40 K and increased to 2000 Oe at 20 K, as shown in Figs. 2(a) and 2(b). It should be noted that, although sample 2 has a quite low T_c (76 K) compared to sample 1 (T_c= 86 K), an anomalous magnetization peak at H_{peak}=1000 Oe is still observed even at the temperature of 40 K.

The observation of the peak effect in such a low T_c Bi-2212 crystal has not been reported so far. It was reported that the peak effect only appears for $0.2 \leqslant T/T_c \leqslant 0.4$ for Bi-2212 crystals with $T_c \sim 87$–89 K.[10–14] It should be noted that the peak effect which appeared in our spiral-grown crystals is much stronger than that induced by oxygen vacancies, defect dislocation networks and edge barriers as reported for Bi-2212 crystals, in which the H_{peak} was located between 600–900 Oe at 30–40 K.[13,14] In our experiments, almost the same H_{peak} was observed for both for samples 1 and 2 between 30 and 40 K. This indicates that the dominant flux pinning mechanism for the spiral-grown crystals is independent of T_c and is very strong even at these relatively high temperatures (T/T_c=0.5). This is indicative of the peak effect feature of Bi-2212 crystals which is relatively independent of temperature.[13,14]

Oxygen vacancies have been regarded as one of the origins of the peak effect in Y-123 and Bi-2212 crystals. Sample 2 is oxygen underdoped with a very low T_c of 76 K, corresponding to a large amount of oxygen vacancies in the sample. In order to determine whether the peak effect observed for the spiral-grown crystals is related to the oxygen vacancies or to growth spirals visible on the crystal surface, two types of experiments were performed. First, to check the possible effect of surface growth spirals on the peak effect, the visible growth spiral patterns were removed from the surface of one of the spiral-grown crystals (sample 2) by cleaving using adhesive tape. Using this method of removal, no surface damage occurs on the new crystal surface and no growth spiral patterns were visible on the new surface either by optical microscopy or atomic force microscopy (AFM). After removing the tape, 80% of the visible growth spiral patterns on the surface had disappeared and T_c did not change as measured by ac susceptibility. Approximately

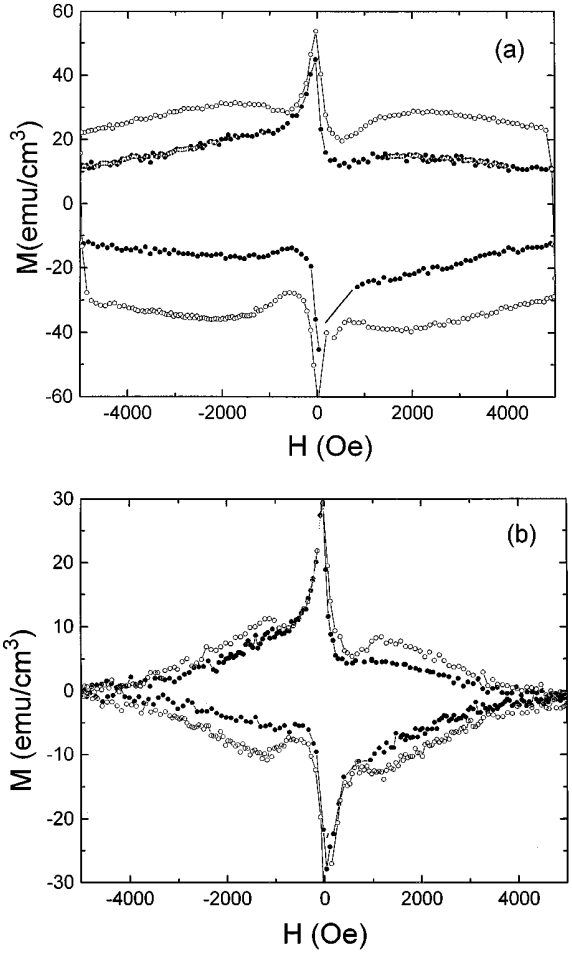

FIG. 2. M-H loops for spiral-grown Bi-2212 crystal (sample 2) with oxygen underdoped (T_c=76 K) at 20 K (a) and 30 K (b) before (open circles) and after (closed circles) removing the growth spiral patterns from the surface.

30% of the sample volume was estimated to be lost due to the surface removal process. The M-H loops were then measured again (closed circles) as shown in Fig. 2. It is clearly seen that the peak effect (anomalous peaks at 1000 Oe at 30 K and 2000 Oe at 20 K) almost completely disappeared after removing the surface growth spiral patterns. The residual spiral patterns are believed to contribute to a very small peak in the M-H loops (closed circle). This strongly indicates that for the surface with visible growth spirals, other than oxygen vacancies are responsible for the peak effect in spiral-grown Bi-2212 crystals.

In the second type of experiment, an M-H loop of a Bi-2212 crystal which was grown by the two-dimensional layer-by-layer growth mechanism with the same oxygen underdoping as sample 2 (T_c=76 K) was also measured (Fig. 3) Its M-H loop is typical of high-quality layer-by-layer grown Bi-2212 crystals at 30 K. No anomalous peak can be seen. This further confirms the previous conclusion that the growth spirals are responsible for the peak effect.

The microstructures of the growth spirals for samples 1 and 2 were examined by optical and atomic force microscopy (AFM). Figure 4 shows an AFM image of growth spiral patterns for sample 2. Sample 1 was also observed to have

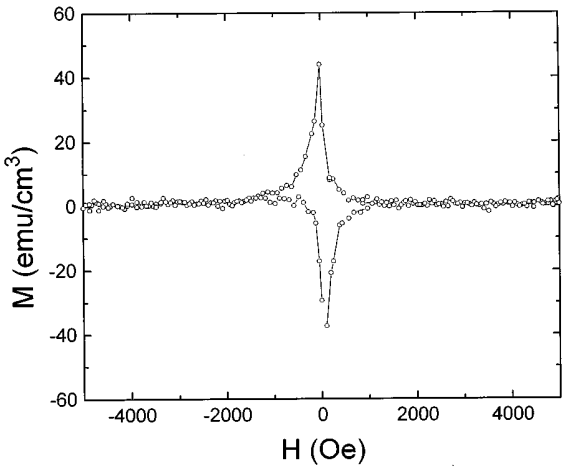

FIG. 3. M-H loop for a Bi-2212 crystal (sample 3) with oxygen doping (T_c=76 K) grown by two-dimensional layer-by-layer mechanism at 30 K.

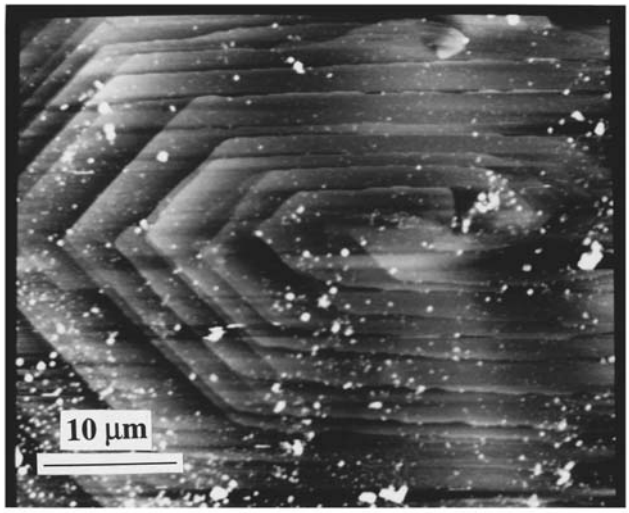

FIG. 4. AFM image of growth spiral pattern of sample 2.

the same growth spiral pattern. The widths and heights of spiral steps are determined to be 1–5 μm and 3–5 unit cells (about 9–16 nm), respectively. These two samples contain only 3–6 large hexagonal spirals. A large number of effective pinning centers ascribed to screw dislocations is not likely to be due to such a small number of spirals, even though it was reported that screw dislocations can pin vortices in Y-123 thin film.[5] However, although there are only several screw dislocations in the samples, there are several hundred spiral turns for each of the spirals. The kinks between steps can be expected to be pinning centers when magnetic field H is parallel to the c axis. The number of vortices which can be pinned by the kinks along the spirals was estimated to be about $10^8/\mathrm{cm}^2$ when $H \| c$ at H=0.2 T. This value is only two orders of magnitude lower than the density of screw dislocations in Y-123 thin films with J_c of 10^7 A/cm^2 at H=1 T and 4.2 K.[5] Further, edge barriers (surface barriers and geometry barriers) were recently found to be responsible for the peak effect at elevated temperatures for Bi-2212 and Y-123 crystals.[13,15–17] Large numbers of steps with heights of 9–16 nm on the spiral-grown Bi-2212 crystal surface can also be regarded as a large density of edge barriers. Enhanced flux pinning and a large peak effect can be expected for such crystals with many spiral surface patterns. This was indeed observed for our spiral-grown Bi-2212 crystals (Figs. 1 and 2), giving strong support to edge barriers as the effective pinning centers.

Measurements of the hysteresis loops measured for sample 1 with different sweep rates of the field show that the dynamic flux creep rate is different for fields entering and exiting the sample (not shown here). This is further evidence for the edge barrier as the origin of the peak effect.[13]

For the low T_c (76 K) spiral-grown Bi-2212 crystal, at a temperature as high as 0.5 T_c, the peak still exists. This is in good agreement with previous findings that surface barriers are the source of the peak effect when the bulk pinning is weak, i.e., at these elevated temperatures.[13,17] Furthermore, the value of H_{peak} is above 1000 Oe at 30 and 40 K, and larger than 2000 Oe at 20 K. This value is much higher than that reported for oxygen vacancies, defect dislocation network, or geometry barrier of crystals, indicating a much higher flux pinning associated with the growth spirals in the spiral-grown Bi-2212 crystals compared to the crystals grown according to a layer-by-layer growth mechanism. It should also be noted that after removing the spirals from the surface, the irreversibility field H_{irr} is still higher than 5000 Oe, (H_{irr} is only about 2000 Oe for the layer-by-layer grown crystal as shown in Fig. 3) indicating that strong pinning still exists in the remaining part of the spiral-grown crystals, even though the peak effect has almost completely disappeared. This may be due to the defects along the spiral steps inside crystals, and it still requires further study.

In summary, a strong peak effect was observed both in high-T_c (86 K) and oxygen underdoped (with a very low T_c of 76 K) spiral-grown crystals between 20 and 40 K. Edge barriers due to growth spirals are suggested to be responsible for the strong peak effect for the spiral-growth $Bi_2Sr_2CaCu_2O_y$ (Bi-2212) crystals, and not screw dislocations or oxygen vacancies.

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