Simultaneous observation of Abrikosov vortices and defects in thin $Bi_2Sr_2Ca_1Cu_2O_x$ single crystals

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Crystal structural defects and decoration patterns of the vortex structure in a high-T_c superconductor have been observed simultaneously by transmission electron microscopy. Features seen in the vortex structure stem from defects which are oriented along the growth axis of the single crystals.

A regular triangular vortex lattice should be observed in ideal (i.e., isotropic and defect-free) type-II superconductors.[1] In real superconducting materials, on the other hand, it is extremely rare to see a regular vortex lattice, since the pinning caused by the defects or irregularities, which are always present in some number, disrupts the strict order in the lattice.

The visualization of Abrikosov vortices by decoration with dispersed ferromagnetic particles is a useful method for studying the morphology of vortex structures and pinning mechanisms. For such studies, the most informative method is that which combines (on the one hand) a visualization of the vortex structure on the surface of the superconductor by decoration with ferromagnetic particles with (on the other) transmission electron microscopy, which reveals defects distributed in the volume of the crystal. The main difficulty confronting such studies is the need to prepare thin foils for the transmission microscopy. This method was implemented in Ref. 2 for the case of deformed single crystals of Nb; a vortex structure and a dislocation structure were observed simultaneously there for the first time. Unfortunately, the methods of which we are aware for thinning single crystals of high-T_c superconductors substantially degrade the surface, to the point that it becomes impossible to visualize vortices by the decoration method. In the present study we used ribbon-shaped BSCCO single crystals of the 2212 composition, transparent in transmission electron microscopy, without additional thinning. As a result, we have succeeded in simultaneously observing a vortex lattice and structural defects of the crystal.

Thin single crystals were grown from a bismuth-enriched stock material by quenching from 1100 to 300 °C and by then holding the material at 840 °C in flowing oxygen for a long time (3 to 5 days).[3] The thin single crystals, which grew to cover the entire surface of the quenched seed, had lengths up to 15 mm, a thickness of a fraction of a micron, and widths from a few microns to several tens of microns. According to electron microprobe analysis and electron microdiffraction, the composition of the resulting single crystals corresponds to the $Bi_2Sr_2Ca_1Cu_2O_x$ phase. The superconducting transition temperature is $T_c \simeq 80$ K. Significantly, the single crystals were not subjected to further treatment in the course of the surface study. Because of the small

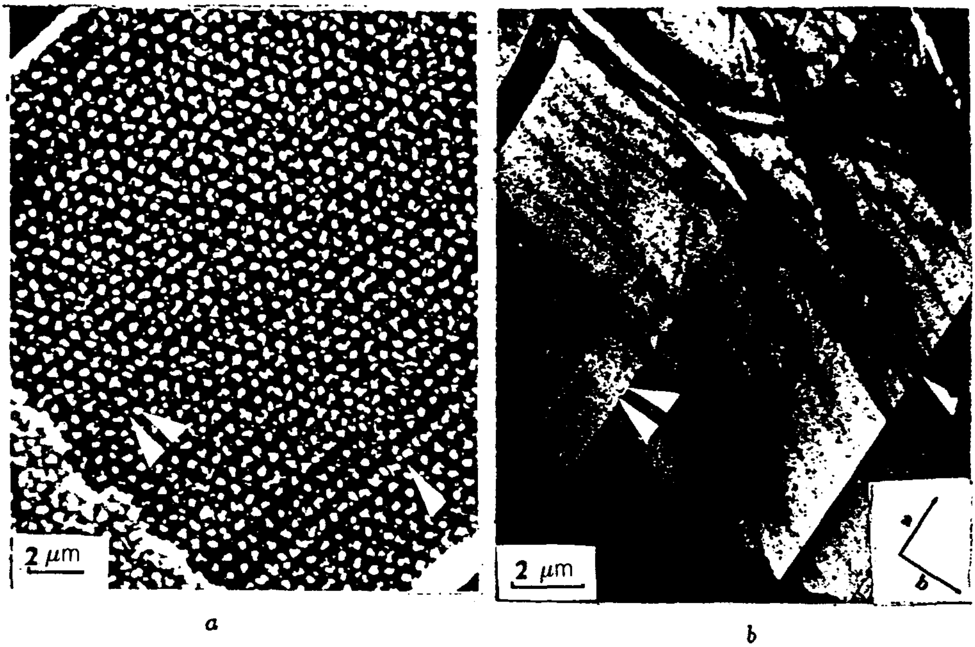

FIG. 1. Photomicrographs of the same region on a $Bi_2Sr_2Ca_1Cu_2O_x$ single crystal taken in the scanning mode (a) and in the transmission mode (b) after decoration. The sample was decorated in a magnetic field $H=52$ Oe.

thicknesses (<0.2 μm) of the test samples, they could be studied in either the (reflection) scanning mode or the transmission mode of a JEOL 100CX microscope at an accelerating voltage of 100 keV. The vortex structure was visualized by decoration with dispersed ferromagnetic particles at 4.2 K after the samples had been cooled in a magnetic field ($H\sim 20$–100 Oe) from a temperature $T>T_c$.

Figure 1a is a photomicrograph of the vortex structure in a $Bi_2Sr_2Ca_1Cu_2O_x$ single crystal taken in the scanning mode after decoration in a field $H=52$ Oe. The bright points on the surface are contrast resulting from the accumulation of the dispersed ferromagnetic particles; these points correspond to the positions of Abrikosov vortices. Figure 1b is photomicrograph of the same region of the $Bi_2Sr_2Ca_1Cu_2O_x$ single crystal, in this case taken in the transmission mode. An opaque part of the copper grid on which the sample was mounted before decoration can be seen in the right-hand corner of the photograph. In this mode, the accumulations of the ferromagnetic particles decorating the Abrikosov vortices are seen as dark spots against a bright background, because of the absorption contrast, which is considerably weaker than the diffraction contrast. In addition to the defects which can be visualized in the transmission mode (Fig. 1b), we can clearly see a vortex lattice on both micrographs. The distortions in the vortex lattice along the **a** axis are smaller than those in the two other close-packed rows of vortices. It can be seen from Fig. 1 that one of the close-packed rows of the lattice is parallel to the long axis of the single crystal, which coincides with the

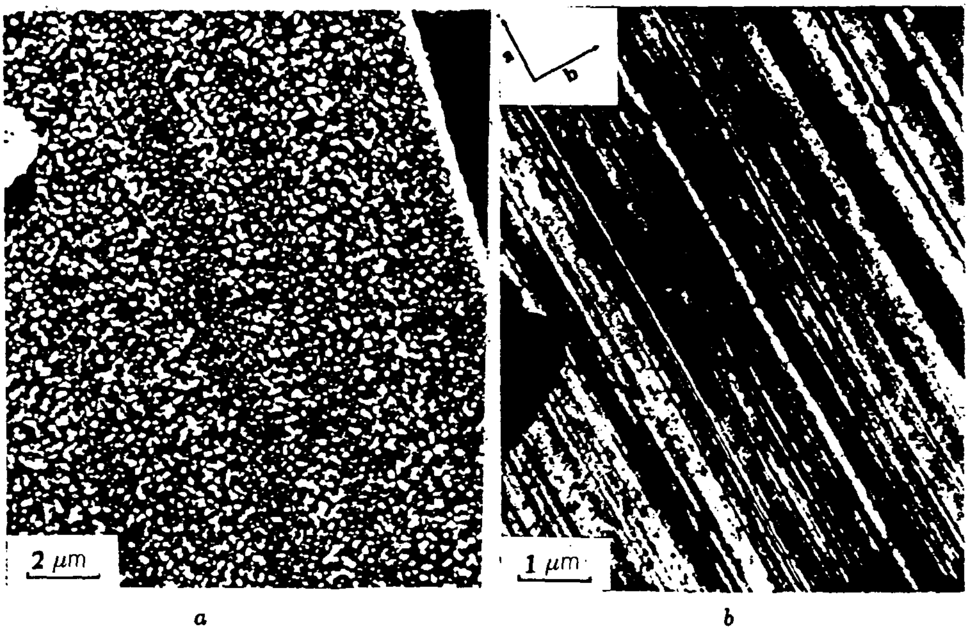

FIG. 2. Vortex structure of a $Bi_2Sr_2Ca_1Cu_2O_x$ single crystal with a high defect density. *a*—Scanning mode; *b*—transmission mode. The sample was decorated in a magnetic field $H=23$ Oe.

crystallographic **a** axis, as follows from the corresponding microdiffraction pattern [the **a** and **b** crystallographic directions can easily be distinguished on the microdiffraction patterns by virtue of the superstructural reflections along the **b** axis: $b' \simeq 4.7b$ (Ref. 3)]. Strictly oriented in the same direction are those defects in the single crystal which are visualized in the transmission mode of the electron microscopy by virtue of the diffraction contrast.[4] We frequently see a row of vortices "sitting" on such a defect, along which the contrast changes sharply (Fig. 1b). These defects differ in the extent to which they affect the vortex lattice. For example, the defect marked by the single arrow in Fig. 1b causes an increase in the density of the vortex row (Fig. 1a), while there are no obvious features at the similar defect marked by two arrows. Similar patterns are observed in the other single crystals of relatively high quality which we studied.

Figure 2 shows a vortex structure in a single crystal which differs radically from that in Fig. 1. We see that the vortex lattice is completely disrupted, but a "texture" of the magnetic flux along the **a** axis persists. In Fig. 2b we see, in addition to the vortices, a high density of defects, which are also oriented strictly along the **a** direction. Among the thin crystals studied there are thus both relatively high-quality crystals, with a relatively low defect density, in which a vortex lattice is resolved, and low-quality crystals, with a high defect density, in which a vortex lattice is not observed.

At this point the nature of the defects observed here has not been precisely established. We can only assert that these defects do not constitute a twin structure similar to that observed[5] in $YBa_2Cu_3O_7$, since the microdiffraction patterns of the defective $Bi_2Sr_2Ca_1Cu_2O_x$ single crystals do not exhibit an alternation of **a** and **b** in terms of direction. Nevertheless, the observed defects are also strong pinning centers. In terms of the nature of the changes in the diffraction contrast when the sample is tilted in the electron microscope, these defects are similar to plane boundaries between regions of a crystal which are slightly disoriented. However, a final conclusion regarding the nature of these defects will require a more detailed electron-microscopy study. We believe that a combined study of the vortex structure and the defects in a crystal will eventually make it possible to evaluate the efficacy of defects (dislocations, interfacial boundaries, intercrystal boundaries, etc.) as pinning centers.

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