Structure of boundaries between twins and twinning complexes in $YBa_2Cu_3O_{7-\delta}$ single crystals

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X-ray-diffraction studies showed that the boundaries between the twins and the polytwin complexes in $YBa_2Cu_3O_{7-\delta}$ crystals are not monatomic planes but are instead transition regions, along which the parameter a continuously changes to the parameter b as it goes through the state of the tetragonal phase ($a = b$). The total volume occupied by these regions is $\sim 6\%$ of the crystal's volume.

We have previously reported that twin 1-2-3 crystals have well-developed boundaries between structural domains (twins).[1] In this letter we report the results of a detailed study of the structure of these boundaries, since the effect of a twinning structure on the superconducting characteristics has been established.[2] In formulating the experimental problem we took into account that in some twin crystals with a similar high-temperature superconducting tetragonrhombus phase transition the boundaries are not monatomic planes but are transition regions several microns in thickness, along which the rhombic angle changes continuously from one twinning orientation to another as it passes through the state of the tetragonal phase.[3,4]

We studied a crystal which was grown during a slow cooling of a molten mixture of oxides Y_2O_3, BaO, and CuO. The crystal had the shape of a rectangular black plate with mirror-finish faces. The dimensions of the crystal were $0.5\times1.0\times0.03$ mm. The lattice constants, measured by x-ray diffraction method, were $a = 3.86$ Å, $b = 3.92$ Å, and $c = 11.59$ Å.

The analysis was based on the use of a standard x-ray diffraction system in a transmission geometry. The distinguishing feature was the use of a point (5 μm) of radiation source in contrast with a conventional extended source. In this case it is possible in addition to the standard x-ray-diffraction studies to record the angular scanning topogram by placing a photographic plate in front of the radiation detector.[1]

Angular scanning topograms obtained as a result of reflection from {100} planes are shown in Fig. 1. Intense black spots A, $B(A',B')$ are reflections from the crystal fragments belonging to various polydomain complexes. A spot $A'(B')$ in the (020) reflection (b is a parameter) corresponds to each such spot $A(B)$ in the (200) reflection (a is a parameter). The topogram in Fig. 1b shows the reflection from a single polytwin complex. The vertical scale on these topograms corresponds to the disorientation angles of the fragments. The angles between the components A and A' and B and B' correspond to the twinning angle.

The faint diffuse lines which connect the images of the twin components represent the transition regions of the coupling of the twins and twin complexes with each other. Three types of connecting lines are discernible on the topogram. The lines AB' and BA' correspond to a continuous change of the interplanar distance from a to b, without changing the lattice orientation. The lines AB and $A'B'$ correspond to a continuous

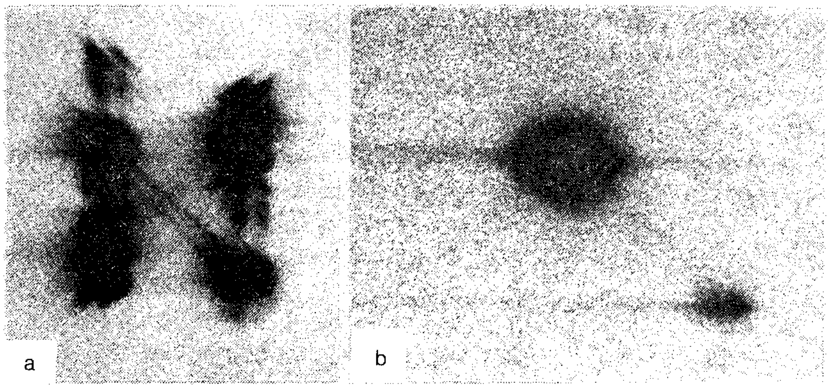

FIG. 1. Topograms of the angular scanning of the 1-2-3 crystals in the Laue geometry, with a {200} reflection. a—A crystal comprised of several polytwin complexes with mutually perpendicular twinning planes; b—a crystal comprised of a single polytwin complex.

change in the disorientation angle of the twin component with a specified value of the lattice constant (a or b). The lines AA' and BB' correspond to a continuous change of the lattice constant from a to b as the disorientation angle of the reflecting planes changes. The lines AA' and BB' run in the same direction as $\langle 110 \rangle$.

Let us consider more closely the "diffuse" lines of the type $AA'(BB')$, $AB'(BA')$, and $AB(A'B')$, which are interpreted as transition regions linking the twins and the twin complexes. We will consider lines of the type $AA'(BB')$. A simple interpretation of such a diffraction image could be based on the assumption that the diffuse lines are a Fourier transform of an array of thin twinning intercalations directed parallel to the (110) planes. In this case, however, the image must be symmetrical relative to the images of the macroscopic twinning regions $A(A')$ and $B(B')$. In the topograms (Fig. 1, a and b) the lines AA' and BB' actually do not extend beyond the limits of the macroregions $A(A')$ and $B(B')$. The image of a crystal in the (220) reflection also has no diffuse lines similar to the $AA'(BB')$ lines. It could also be assumed that the diffuse lines are due to the reflection from the thin twinning intercalations which run parallel to the $\{110\}$ planes and which have a parameter $(a+b)/2$ of the tetragonal phase. In this case, however, the intensity distributions would be expected to run along the AA' line, with a maximum situated at the midpoint between A and $A'(B$ and $B')$. A measurement of the integrated intensities between the (200) and (020) reflections shows, however, that the parameter $d(_{200}) + d(_{020})/2$ corresponds to a minimum, rather than a maximum, integrated intensity. We must assume, therefore, that the AA' and BB' lines represent the transition regions in the crystal, along which the lattice constant changes continuously from a for one twin orientation to lattice constant b for another twin orientation, accompanied by a simultaneous change in the orientation of the reflecting planes. Similar arguments apply to the lines AB, $A'B'$, AB', and BA'.

On the basis of our discussion we can propose several structures of the transition regions situated between the twins and the twinning complexes. These structures are illustrated in Fig. 2. We see in this figure a coherent twinning boundary (Fig. 2a), an incoherent boundary between twinning complexes which have mutually perpendicular twinning planes (Fig. 2, b and c), and a coherent boundary inside a single twin (Fig. 2, d and e).

The upper parts of the structures in Fig. 2, b and c, which are mirror images of the AA' line, give the outlines of the incoherent boundaries between the polydomain complexes with a single plane but opposite twinning directions. In the figures we clearly see the orientations of the reflecting planes when the boundary is crossed.

To estimate the size of the transition regions between the twins and the twinning complexes, we measured the integrated reflection intensities for various interplanar distances. The minimum peak intensity for interplanar distances, which lie between the orthorhombic cell parameters a and b and which correspond to the transition regions between the twins, amounts to $\sim 3\%$ of the peak intensity of the twinning components. Allowance for the half-width of the reflections shows that the transition regions in the crystal occupy at least 4% of the whole crystal. An estimate which takes into account an increase in the integrated intensity as one approaches the twinning components (a or b) yields a value of $\sim 6\%$.

The average width of the transition regions can be estimated from the values of

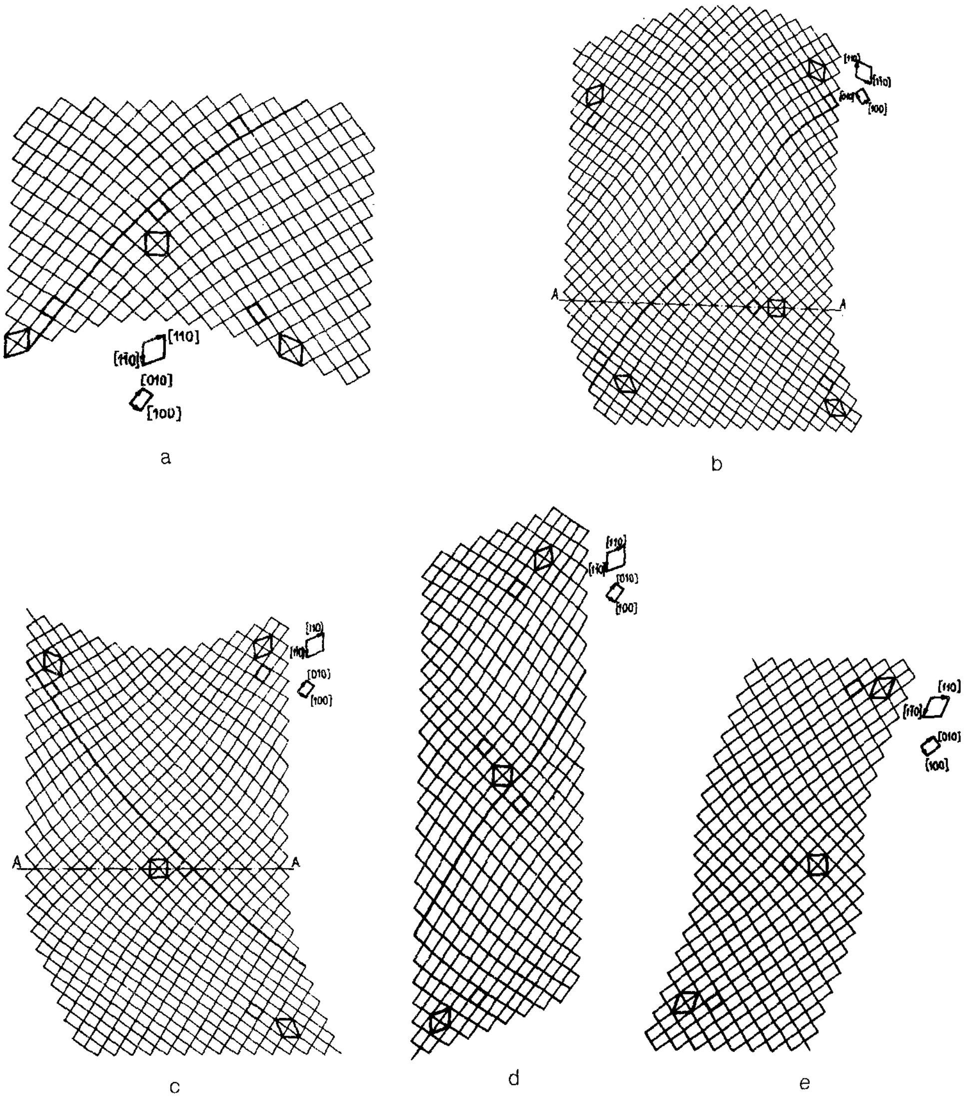

FIG. 2. Schematic diagrams of the conjugation boundaries of the twins and twin complexes.

the integrated intensity which we have obtained. Here the transition regions between the twins and twinning complexes should be singled out. The measurements of the integrated intensity carried out for a separate twinning complex showed that "coherent" twinning boundaries occupy a volume of no more than 0.5% of the volume of the twinning components. In the crystal under study the principal part of the integrated intensity recorded for the transition regions is thus attributable to the boundaries between the twinning complexes. The thickness of the "incoherent" twinning boundary, found from the average size of the twinning complexes (~ 100 μm) and from the average volume occupied by the transition regions (~4–6%) is therefore estimated to be ~2–3 μm. The size of the coherent twinning boundaries cannot, however, be determined from the data which we have obtained.

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(1) In the transmission geometry the photographic plate is stationary, in contrast with the Bragg geometry which involves a θ–2θ scanning.

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