Kinetics of the changes in the twin structure in $YBa_2Cu_3O_{7-x}$ single crystals

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The temperature dependence of the saturation time of $YBa_2Cu_3O_{7-x}$ single crystals in the field of a concentrated load has been studied. The activation energy for the motion of twinning boundaries has been determined ($Q = 0.59 \pm 0.05$ eV). This motion is limited by a reorientation of CuO chains due to a migration of oxygen.

Crystals of the high-temperature superconductor $YBa_2Cu_3O_{7-x}$ (YBaCuO) are usually divided into twins, which differ in the orientation of the CuO chains in the planes between barium ions (the b axis).[1] Their boundaries act as strong pinning centers for Abrikosov vortices[2] and may lead to a significant change in the supercon-

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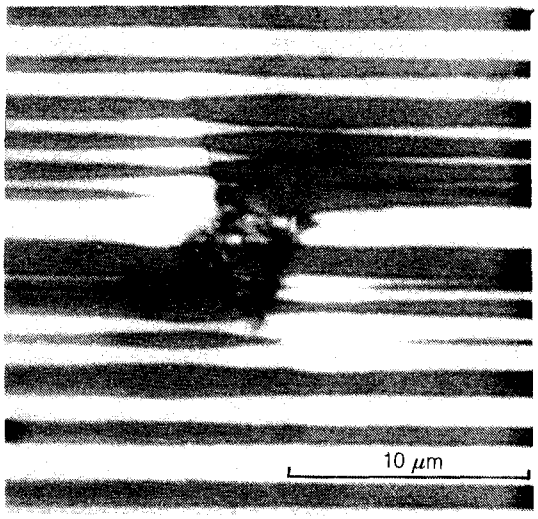
ducting transition temperature.[3] Certain structural features on the temperature dependence of the electrical properties of YBaCuO are often attributed to changes in the twin structure. So far, however, there has been no systematic study of the motion of twinning boundaries.



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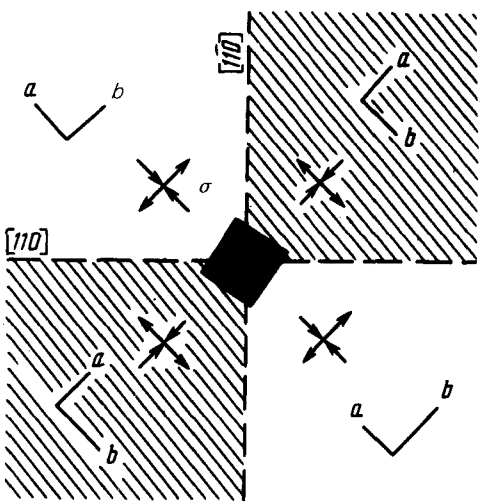

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FIG. 1. a: Change in the twin structure in the basal plane of a $YBa_2Cu_3O_{7-x}$ single crystal in the residual stress field set up by the indentation made by an indenter. The polarized-optics contrast between domains was deliberately created by introducing an additional birefringence in the microscope. b: Stress (σ) distribution and directions of the a and b axes in the predominant domains.

In this letter we are reporting a study of the change in the twin structure in the residual stress field left by the indentation of an indenter on the (001) basal surface of YBaCuO single crystals. The twin structure was monitored with a polarized microscope in reflected light.[4]

The YBaCuO single crystals, grown by the standard technique, were annealed beforehand in oxygen (1.0 atm) at 450 °C for 10 h. The superconducting transition temperature found after this annealing, from measurements of the magnetic susceptibility, was ~90 K. The crystal was cemented to a sapphire plate. An mhp-100 microhardness-measurement attachment to a Neophot microscope, equipped with polarizers for creating a contrast between the twins, then indented (punctured) the crystal at the selected spot. The load applied to the indenter was 10 g. The plate with the sample was then transferred to a heated stage of a polarizing microscope. The temperature was monitored throughout the measurements with a Chromel-Alumel thermocouple which was cemented to this plate.

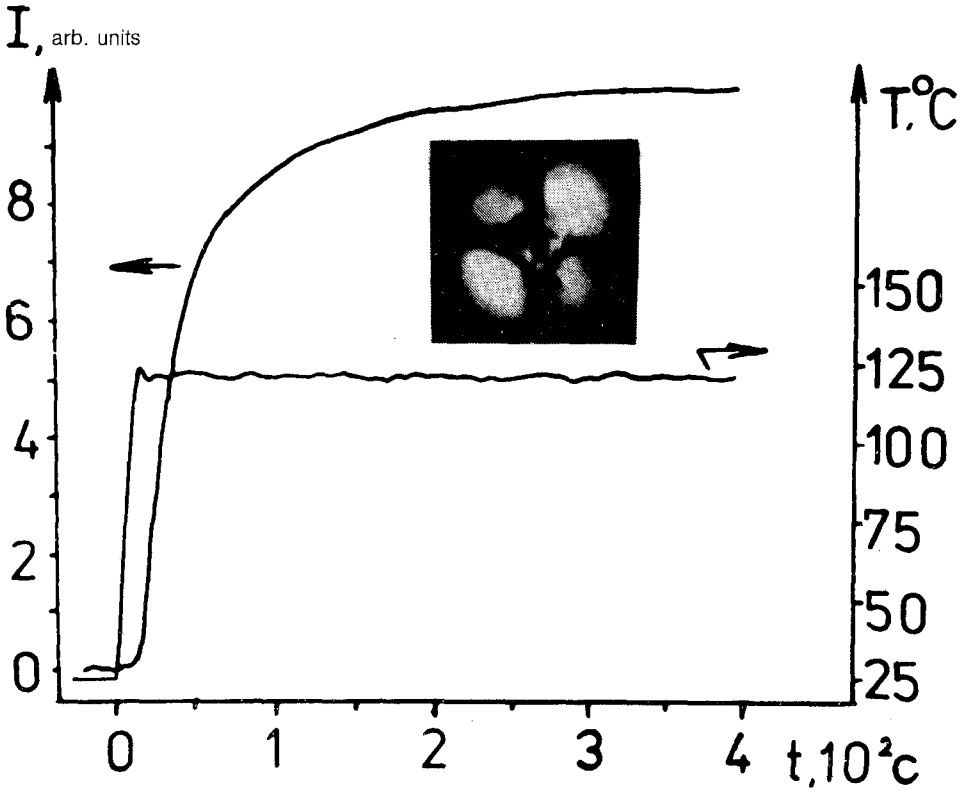


FIG. 2. The light intensity I from a part of the crystal with optically unresolved twins, containing a group of indentations, in crossed polarizers after a rapid increase in the temperature T to 124 °C. The inset shows the brightening rosette which formed at one of the indentations after heating (no contrast between domains was created).

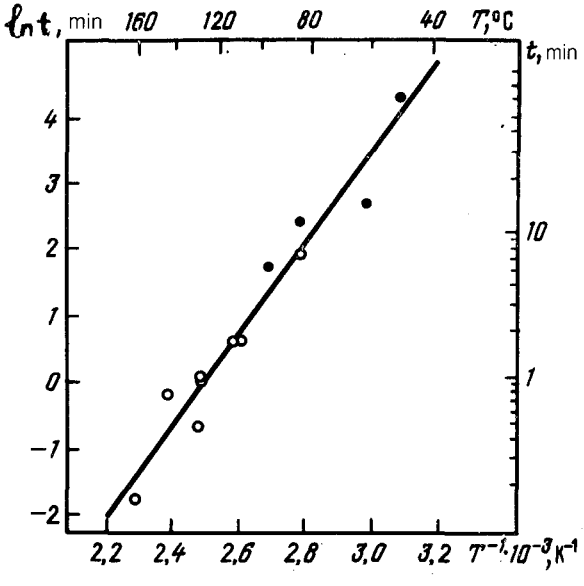
It was found that after the crystal was heated some of the twins near the indentation gradually contracted and were displaced by neighboring twins (Fig. 1a). The residual stress distribution determined the formation of four sectors with $<110>$ boundaries, within which the shrinking and expanding domains traded places, in accordance with the orientation of the orthorhombic a and b axes in them (Fig. 1b). The direction of the b axis was determined with the help of a Berek compensator, as the polarization direction of the light which underwent an additional phase delay upon reflection.[4] Note that similar changes in the twin structure had been observed previously[5] in crystals of rock salt subjected to a concentrated load. A distinguishing feature of the process studied in the present experiments is its clearly expressed relaxation nature (just after the indentation is made, there was no displacement of the twinning boundaries; such a displacement arose only after a hold at room temperature or an annealing at an elevated temperature). The time required for the complete displacement of a shrinking twin increased with the width of this twin; domains with a width on the order of 1 μm or more never did break up in the range of compressional stresses studied.

This saturation process occurred in a stabler fashion when the width of the twins was less than the optical resolution. In this case a brightening rosette ~ 10 μm in diameter gradually appeared at the indentation in crossed polarizers against a uniform dark background of the optically isotropic crystal with narrow twins (see the inset in Fig. 2). The shape of this rosette corresponded approximately to the contour curves of the tangential stress acting in the twinning plane. The intensity of the brightening was proportional to the square of the difference between the volume fractions of the domains with the different directions of the a and b axes. From the growth rate of the brightness rosette near an indentation we were able to draw conclusions regarding the velocity of the twinning boundaries, under the assumption that the saturation process occurs more rapidly as the density of boundaries increases. In order to maximize the reproducibility of the measurements, we carried out the indentation in regions with a uniform and dense twin structure far from the edges of the sample and also far from any visible defects.

The time-dependent changes in the twin structure were detected in two ways. At temperatures below 100 °C, where the formation of the rosettes at the indentations took several hours, the crystal was photographed at intervals from 2 min to 1 h. On the series of negatives which was obtained as a result, we recorded profiles of the blackening density along the diagonals of the rosettes, using a microdensitometer. The amplitude of these blackening profiles increased with time, while the shape of the profiles remained essentially the same. These results are evidence that the detwinning time is only slightly dependent on the stress. We measured the difference between the background blackening and the maximum blackening, and we plotted the increase in the rosette brightness, I, against the time t.

The other method was used at high temperatures, where I reached saturation over a time ranging tens of seconds to a minute. In this case we found a curve of $I(t)$ directly from the output signal from a photomultiplier, to which we directed a stopped-down light beam from the rosettes on a compact group of fresh indentations (Fig. 2).

As a measure of the rate of the process we selected the time over which the rosette image brightness reached 2/3 of its maximum value. This time corresponded roughly to the end of the steep section of the $I(t)$ curve. The measurement time interval was quite long in this case, and the structural features which were seen on the curve of I as it approached saturation, which were associated with the relatively rare wide domains,



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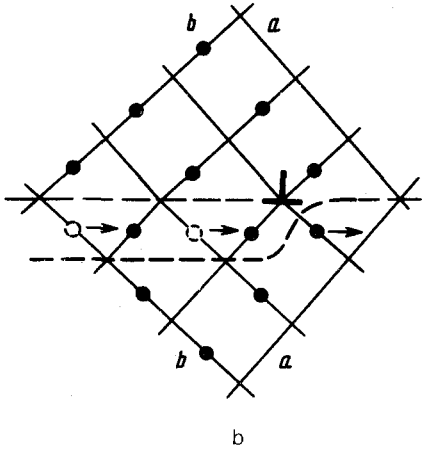

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FIG. 3. a—Temperature dependence of the time required to reach 2/3 of the maximum brightness of the detwinned regions which appeared around indentations left by the indenter as the crystal was heated; b—simplified diagram of the motion of a twinning boundary in $YBa_2Cu_3O_{7-x}$, accompanied by a migration of oxygen atoms in the crystal (the circles) into planes with CuO chains.

and which differed from realization to realization, were eliminated from consideration. Furthermore, to keep the average width of the domains from varying substantially in the different measurements, we made the indentations as close to each other as possible. Values of the logarithm of the time required to reach a brightness level of 2/3 versus the reciprocal measurement temperature found for one crystal are shown in Fig 3a. The filled circles correspond to measurements by the photographic method, while the unfilled circles show the direct measurements with the photomultiplier. The slope of the least-squares straight line drawn through the experimental points determines the activation energy for the motion of the twinning boundaries: $Q = 0.59 \pm 0.05$ eV.

Twinning boundaries move as a result of the motion of kinks on these boundaries (twinning dislocations), as has been well established.[6] A distinctive feature of the crystal structure of YBaCuO is that there is a sequential displacement of oxygen atoms in planes with Cu–O chains in the process (Fig. 3b). A similar elementary mechanism underlies the self-diffusion of oxygen in these crystals, as is confirmed by the fairly good agreement between the value which we found for Q and the activation energy for the migration of oxygen vacancies in YBaCuO (Refs. 7 and 8). It thus becomes possible to work from the change in the twin structure to study the kinetics of the redistribution of oxygen during low-temperature annealing of YBaCuO and related compounds. This method has the advantage that the measurements can be taken at a fixed oxygen concentration.

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