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Plastic deformation of $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ and related structural defects

J. Rabier and M. F. Denanot

Laboratoire de Métallurgie Physique, URA 131 CNRS, Faculté des Sciences, 86022 Poitiers Cedex, France

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Résumé. — Des céramiques $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ ont été déformées en compression à la température ambiante, sous pression de confinement. Les dislocations résultant de cette déformation à forte contrainte ont été étudiées par microscopie électronique en transmission. Les vecteurs de Burgers des dislocations sont du type $\langle 010 \rangle$. Les boucles de dislocation glissiles appartiennent au plan (001) et une dissociation des dislocations a été mise en évidence. On discute les modes possibles de dissociation des dislocations.

Abstract. — $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ ceramics have been deformed in compression at room temperature under an hydrostatic pressure. Dislocations introduced by high stress mechanical deformation have been studied by transmission electron microscopy (TEM). Burgers vectors of dislocations are of $\langle 010 \rangle$ type. Glide dislocation loops have been found to lie in the (001) plane. Dislocation splitting has been evidenced. The possible dissociations of dislocations are discussed.

1. Introduction.

Technological applications of high temperature bulk superconductors require the evaluation of their mechanical properties in relation with their microstructures. Furthermore defects introduced by plastic deformation during the process are expected to influence the physical properties of these compounds. Indeed high T_C superconductors ceramics are characterized by very small correlation distances : the zero temperature coherence lengths ξ of $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ have been estimated to be 0.4 nm in the [001] direction and 3.1 nm in the perpendicular directions [1]. As a consequence, any defect which is

can be efficient in pinning the flux lines. These small coherence lengths and the strong coupling between elastic and super conducting behaviours could lead also to the existence of regions where T_C can be significantly altered [2]. This put forward the possible influence of lattice defects on superconducting properties in such materials.

Numerous defects have been found and studied in $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$: twins, ordering defects, stoichiometry defects (see for example [3-6]) which result from the synthesis process at high temperature. Defects

introduced at high temperature can be affected by point defect diffusion : in addition to strain effects, compositional changes at defect sites are also expected. Superconducting properties are very sensitive to a small composition change and variation in stoichiometry, so that it is of interest to separate the strain effects of defects from those compositional effects.

In this context plastic deformation at low temperatures can introduce defects, which are not related to point defects diffusion, on previously characterized samples. However, owing to the brittleness of these materials, plastic deformation in this range of temperature can only be achieved using specific

a confining pressure are the relevant techniques of deformation.

In this paper, we report preliminary results on the characterization of defects created by plastic deformation at room temperature in $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ under a confining pressure. Plastic deformation of ceramics at low temperature can be achieved by grain boundary sliding and/or plastic deformation of the grains. This study is concerned only with defects located within the grains and responsible for their plastic deformation.

2. Experimental.

Two types of sintered $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ samples were used in this study : type (A) with a small grain size and type (B) with a large grain size (of the order of 1 mm). These samples were cut with a diamond saw in the shape of parallelepipeds of $3 \times 3 \times 8 \text{ mm}^3$ and mechanically polished.

Room temperature deformation of $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ was achieved in a Griggs apparatus [7] set up in an Instron frame which allows to deform brittle solids. In order to suppress the nucleation and propagation of cracks, an hydrostatic pressure is superimposed to the applied stress. A constant strain rate of $\dot{\epsilon} = 2 \times 10^{-5} \text{ s}^{-1}$ and a confining pressure ranging between 0.4 and 0.7 GPa were applied to the samples. Deformation tests were conducted up to a permanent strain of $\epsilon = 0.1$, the engineering yield stress being about 700 MPa.

From these deformed specimens, disks 3 mm in diameter were cut perpendicularly to the compression axis and mechanically polished down to $80 \mu\text{m}$. Electron beam transparency was obtained by ion thinning. Thin foils were observed in a JEOL 200 CX electron microscope operating at 200 kV equipped with a goniometer stage allowing $\pm 45^\circ$ tilt in two orthogonal directions.

3. TEM observations.

Owing to the large unit cell, diffraction experiments are difficult to perform in satisfactory two beam dynamical conditions, so that it is necessary to multiply the observations in order to derive unambiguously the characteristics of defects.

Numerous defects resulting unambiguously from the plastic deformation are evidenced. Figure 1

Fig. 1. — Glide bands in plastically deformed YBaCuO (sample A).

shows a typical feature of the deformation substructure : glide bands built with a high density of dislocations which evidence that plastic deformation is achieved by dislocation glide. In this orientation the foil plane intersects the glide plane so that the deformation is seen heterogeneously distributed between glide bands. Reflection twins have been also evidenced. These twins are characteristic defects in orthorhombic $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ and it is difficult to know if they result from the deformation or from the high temperature tetragonal-orthorhombic phase transition. However an increase in twins number has been previously reported in shock-induced microstructures [8].

Dislocations were also observed in their glide plane. Dislocations of the configuration of figure 2 have been found to be out of contrast for $g=102$, 103 and 104 (see Fig. 2c) and contrasts are consistent with a $[010]$ Burgers vector. By tilting the foil plane, dislocation loops were found to lie in the (001)

Fig. 2. — Glide configuration of dislocations (sample A). Bright fields. a) $g = 010$, observation plane close to (001) ; b) $g = \bar{1}10$; c) $g = 104$: dislocations out of contrast.

plane. This configuration refers unambiguously to a glide one, showing evidence of a $[010](001)$ glide system.

Dislocations were also seen running through $\{110\}$ twin boundaries. Diffraction experiments show that, when they cross a twin boundary, these dislocation have a different Burgers vector. This is in agreement with the twin characteristics since $[100]$ and $[010]$ directions are exchanged between the parent and the twinned crystals. Dislocation parts labelled as B in figure 3 are out of contrast with a $g = 0\bar{2}0$ diffraction vector (which is consistent with a $[100]$ Burgers vector), whereas A parts are in contrast. Within the twinned crystal these dislocations are found widely splitted, differently of what is found in the parent crystal. Stacking faults are shown on figure 3b imaged with a 110 diffraction vector. The stacking faults contrasts are consistent with a displacement vector $R = a/2 [100]$. These dislocations are bowing out, under the applied stress, between the twin boundaries which seem to act as pinning points. Such a pinning process can promote the large splitting width evidenced on B dislocations.

a)

b)

Fig. 3. — Dislocations interacting with a mirror (110) twin (sample B). a) Bright field ($g = 0\bar{2}0$). B dislocation segments out of contrast, A segments in contrast. b) Bright field ($g = 110$): stacking fault contrasts.

Dislocations were also found arranged in sub-boundaries and widely splitted, as shown in figure 4. The stacking fault plane has been determined to be (001) . Partial dislocations are evidenced in figure 4a. It can be shown that dislocations are likely to be dissociated into two partials which seems also to be dissociated into two sub-partial. However their Burgers vectors were not determined.

a)

b)

Fig. 4. — Dislocation sub-boundaries (sample B). a) $g = 0\bar{2}0$ partial dislocations contrasts ; bright field. b) $g = \bar{1}10$ stacking fault contrasts ; weak beam dark field.

4. Possible dissociations of dislocations.

Burgers vectors of perfect dislocations can be of

unit translation vector of the lattice : $a[100]$, $b[010]$, $c[001]$. This yields to Burgers vectors length respectively of : 0.382 nm, 0.389 nm, 1.168 nm. On the basis of isotropic elasticity, the self energy E of dislocations can be written as $E \propto \mu b^2$ where μ is the shear modulus and b the Burgers vector length. This shows that dislocations with c Burgers vector have a larger strain energy and are unlikely to occur unless a dissociation effect can stabilize it. Strain energies of a and b dislocations should be of the same order of magnitude. However a strong elastic

anisotropy is expected from the cell structure, i.e. particularly between $[100]$ and $[010]$ directions from oxygen vacancy ordering [9], so that one of these dislocations can be energetically favoured. Another type of perfect dislocation can result from the combination of these two dislocations following the reaction :

In most crystal structures and oxides, the easy glide system satisfies the condition b/d_{hkl} minimum [10], since the distortion induced by a dislocation of Burgers vector b is spread out on a small distance (b) between two widely spaced dense glide planes (d_{hkl}). In $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ no dense planes exist and the layered structure suggests that the (001) glide plane could be the easy glide plane together with a stacking fault plane. This has been evidenced in the preceding section. However in the $[001]$ stacking the glide shear may occur between differently spaced planes. Dissociations can decrease the elastic energy of the dislocations and the ability of dissociation of dislocation is also related to the nature of the stacking fault which can be different depending on the location of the partial shear in this stacking sequence. One has then to look after the possible stacking faults resulting from a partial shear of the unit cell. A partial shear half of the cell parameter can be assumed from our observations.

Along the $[001]$ direction the stacking sequence can be described as follows :

The numbers 1, 2, 3 denote the (001) interfaces with different atomic and crystallographic structures. The structure is depicted in figure 5 with the preceding notations. Interfaces 1 and 2 are constituted with planes in which the $[100]$ and $[010]$ are equivalent, furthermore in the CuO_2 plane the sub-lattice of oxygen possesses a $a/2\langle 110 \rangle$ translation vector. Any shear of this type in this plane will produce a stacking fault in the Cu sub-lattice but no change in the next neighbours of oxygens. In the CuO layers

Fig. 5. — Unit cell of YBaCuO : possible shear planes are indicated.

(shear plane 3) the orthogonal $\langle 100 \rangle$ directions are not equivalent so that the partial shears $1/2[100]$ or $1/2[010]$ give rise to different kinds of faults. The change in first neighbours across a stacking fault induced by a shear $a/2[100]$ or $a/2[010]$ is indicated in figure 6, arrows indicate the neighbours violation. It can be seen that in the case of an interface 1, two neighbour violations occur by surface unit of (001) plane, whereas for the others stacking fault only one neighbour violation occurs. For interface 3, a $b/2$ shear yields to an oxygen-oxygen wrong bonding, whereas the $a/2$, yields to a Ba-O wrong bonding. These geometrical features show that the induced stacking faults are not equivalent.

From these possible stacking faults, dissociation reactions of dislocations can be written :

Reactions (1) and (2) yield to different stacking faults whose energy difference is inasmuch high as the shear occur between BaO and CuO layers. A sensible difference between these two defects can stabilize one dislocation rather than the other.

Fig. 6. — Next neighbours across a (001) stacking fault plane, wrong bondings are arrowed : a) shear $b/2$ between BaO and CuO_2 planes, b) shear $b/2$ between Y and CuO_2 planes, c) shear $b/2$ between BaO and CuO planes, d) shear $a/2$ between BaO and CuO_2 planes.

Reaction (3) is in principle unlikely since it gives rise to two orthogonal partial dislocations with no gain in elastic energy, but can be favoured if a strong elastic anisotropy exists. Reactions (3) and (4) should be related to interfaces 1 and 2. Reaction (5) with a (001) fault plane leads to a climb dissociation which has been evidenced in grain boundaries [11], with a change of composition which is not relevant to the present study. This reaction can be also assessed by a glide process in any crystallographic plane containing [001], but is not taken into account here.

5. Conclusion.

Dislocations have been introduced by plastic deformation at room temperature in $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$. A glide system [010](001) has been evidenced. Some dislocations have been found to be dissociated. The

interactions of glide dislocations with (110) twin boundary show that depending on the direction [100] or [010] of the Burgers vectors dislocation splitting can be very different. The analysis of the possible dissociation modes of [100] and [010] dislocations shows that different stacking faults are created in the (001) glide plane following the nature $a/2[100]$ or $a/2[010]$ of the displacement vectors and the atomic nature of the cut plane.

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