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Report:

DISLOCATION MULTIPLICATION DURING THE VERY FIRST STAGES OF PLASTIC DEFORMATION IN SILICON OBSERVED BY X-RAY TOPOGRAPHY

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The first stages of plastic deformation of FZ silicon single crystals were investigated by *in situ* X-Ray topography at the ESRF, in creep conditions at temperatures between 975 K and 1075 K, and for applied stresses from 22 to 44 MPa. A special attention was given to dislocation multiplication, and several features relevant for this phenomenon were observed: cross slip at the surface or in the bulk of the specimen, instabilities in slip bands resulting from a non planar development of dislocations, creation of new sources by moving dislocations.

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Abstract:

The first stages of plastic deformation of FZ silicon single crystals were investigated by *in situ* X-Ray topography at the ESRF, in creep conditions at temperatures between 975 K and 1075 K, and for applied stresses from 22 to 44 MPa. A special attention was given to dislocation multiplication, and several features relevant for this phenomenon were observed: cross slip at the surface or in the bulk of the specimen, instabilities in slip bands resulting from a non planar development of dislocations, creation of new sources by moving dislocations.

I Introduction

Little is known about the mechanisms involved in dislocation multiplication, especially in materials which are initially dislocations free like silicon. The old problem: "How does a perfect crystal like silicon fill with dislocations from a few active slip planes ?" remains pending. This question has recently gained new interest, thanks to computer simulations of dislocation evolution in specimen of a nearly macroscopic size during a virtual mechanical test. In a simulation specially adapted for silicon (slip geometry and dislocations dynamics), Moulin et al. [1,2], were able to reproduce the most salient feature characteristics of silicon compressed at constant strain rate, namely the upper and lower yield stresses, using the simple assumption of an arbitrary distribution of fixed dislocation sources. However, Moulin showed that his simulation does not fit to the empiric multiplication law used by Alexander and Haasen [3] in their modeling of the first stages of plastic deformation by creep or constant applied strain rate:

$$d\rho/dt = K.\rho.v.\tau_{eff}$$

where ρ is the dislocation density, v the average dislocation velocity, and τ_{eff} the effective stress on dislocation segments. This relation was derived from etch pits counts in crept germanium crystals [4] but lacks of clear physical basis.

From the experimental point of view, a fixed distribution of pre-existing sources cannot be realistic in dislocation-free silicon and new sources must be created during plastic deformation by the moving dislocations themselves. For a source to be formed, some pole(s) must be created around which a dislocation arm might turn. Further, a mechanism must be found by which dislocations are not only multiplied in their initial slip plane but are transferred in parallel planes, which were formally inactive. Repeated cross slip is usually invoked but few data are available on such matter in silicon. More generally, it is believed that any event by which a dislocation can escape from its slip plane (jog formation, activation of secondary slip systems...) may also help.

High intensity, synchrotron X-Ray sources like ESRF made possible the design of new experiments, allowing the *in situ* observation by X-Ray Topography of a low density of dislocation segments moving in the whole gauge length of silicon samples, during creep experiments.

II Experimental

Tensile specimen, $15 \times 4 \times 0.7$ mm^3 in size were cut from FZ silicon wafers having a $(\bar{2}\,2\,1)$ or a $(1\,1\,0)$ surface (resp. "A" or "B" orientation, Figure 1.), and diamond and chemically polished in order to remove, as well as possible, surface defects which could act as dislocation sources. A few Vickers micro indents were done to promote original dislocation sources. A dual slip $[1\,\bar{1}\,4]$ tensile axis was chosen, in order to avoid a zero resolved shear stress on the cross slip plane, as would have been the case with a single slip [123] orientation. The two different orientations gave different views of the reactions, with primary or cross slip planes seen "flat" or nearly end on, and dislocation segments intersecting the surface being either screw or 60°.

The experiments were done at the ID 19 beamline of the ESRF. The specimen were observed in transmission using either a $\bar{g} = \bar{2}\,2\,0$ or $\bar{g} = \bar{2}\,\bar{2}\,0$ diffracting vector ("A" or "B" orientation) parallel to the specimen surface. Because of the small divergence of the beam, a low amplitude scan in θ angle (typically 0.08°) had to be applied to the specimen during the exposure in order to image the whole gauge length. The images were recorded simultaneously on X-Ray films and on the CCD FRELON camera (1000*1000 pixels, 10μm resolution) of the ID 19 beamline. Typical exposures lasted from 30 seconds to one minute, depending on the scan amplitude, and were repeated every few minutes, in order to get successive snapshots of the expanding dislocation loops.

The specimen were strained in creep conditions, at temperatures from 975 K to 1075 K, with applied loads from 22 to 44 MPa (i.e. resolved shear stresses on the primary slip systems from 10 to 20 MPa), in an *in situ* straining stage built on purpose. The experiments were done under a 5. 10^4 Pa Ar atmosphere, after a careful degassing of the furnace, in order to avoid surface oxidation. The specimen were cooled down under stress before the dislocation density became too high, and post mortem Burgers vector analysis was carried out by classical Lang topography.

III Results

Fig. 2 shows several snapshots taken of a specimen strained at 975 K, 44 MPa. A small number of slip bands are active. Their origin was always found at surface defects, either at the Vickers indents, or at some residual defects (on specimen sides) left after polishing. At these points, dislocations of different slip systems were generally emitted. They belong mainly to the slip systems with the highest Schmid factor, even if, during these experiments, all slip systems were present.

Several types of events can be observed: cross slip of individual dislocations, perturbations occurring in slip bands and formation of new dislocation sources.

1 Cross slip

An enlarged view of slip bands developing from an indent can be seen in Fig. 3. The image was taken post mortem on a "A" specimen with $\bar{g} = \bar{2}\,0\,\bar{2}$. There are inclined slip bands of the two primary slip systems (planes$\left(\bar{1}\,\bar{1}\,1\right)$ and (111)), and an "horizontal" band of a secondary slip is in residual contrast (i.e. g . b = 0) in plane $\left(1\,\bar{1}\,1\right)$. A great number of dislocation segments has cross slipped from the primary slip planes into the $\left(\bar{1}\,1\,1\right)$ slip plane. As all primary dislocation half loops develop from the same point, they all have the same sign. The dislocation segments which cross slip then all move to the same side of the primary slip plane: the right side for $[1\,0\,1](\bar{1}\,\bar{1}\,1)$ and the left side for $[0\,1\,\bar{1}](1\,1\,1)$ The dislocation lines moving in the cross slip plane intersect the surface at a point which moves along a [110] oriented line (the intersection of the cross slip plane and the surface). A dislocation like A is expected to have cross slipped at the surface, just after its creation, as the Vickers indent belongs to the intersection line. A dislocation which would have cross slipped later, when the screw segment was deeper in the specimen, will intersect the surface on a [110] line lower on the image, like B or C. In an expanding half loop of a primary slip system in a "A" specimen, the dislocation segments intersecting the surface are expected to have a 60° orientation, and the screw segments should move deeper, under the surface. The nucleation of a cross slipped segment on B and C dislocations is then thought to have occurred in the bulk.

2 Instabilities in slip bands

Fig. 4 shows a slip band of the $[0\,\bar{1}\,1]$ $(1\,1\,1)$ primary slip system observed in situ (22 MPa, 1075 K, "B" orientation). The original source was at a surface defect, on the right side of the specimen. The first emitted dislocations (A) have nearly crossed the specimen. They have a mainly screw orientation, and are well spaced. At point B, several dislocations have a more disturbed shape, and some of them seem to be interacting (see arrows). Even if the resolution of X-Ray topography does not allow to separate them, they obviously cannot glide on the same slip plane. In the right part of the image (C), the dislocation density is much higher. The dislocations loose their <110> orientation and move on several parallel slip planes. A careful analysis of this and other analogous configurations suggest that, at a given point, the density of jogs on moving dislocations increases with time.

3 Dislocation sources.

The original surface sources excepted, quite a few dislocation sources were observed during our experiments, at last at places where dislocation density was low enough to allow their analysis. Fig 5 shows an example, which concerns dislocations with the $\left[1\,\bar{1}\,0\right]$ Burgers vector. The first three topographs were taken in situ, and the last one post mortem with the $1\,\bar{1}\,\bar{1}$ g vector, which leaves primary dislocations out of contrast. After a first cross slip at point A (Fig. 5 a), the dislocation moves on both $(1\,1\,1)$ and $(\bar{1}\,\bar{1}\,1)$ planes with the same Schmid factor s = 0.18. The two segments react and move to the left, leaving a closed loop lying in the two slip planes (Fig 5.b). A further displacement of the dislocations leads to the destruction of this loop and the creation of a new one (Fig. 5 c), still lying on two planes. The segments of this loop further expanded in their own slip planes, leading to the creation of the segments seen Fig 5 d, which can turn around the deviation point (arrow). Such a source is unstable, as its pole is mobile.

IV Discussion and Conclusions.

These preliminary observations indicate that the first surface sources are later relayed by internal sources, which are formed by dislocation reactions involving cross slip. The activation energy of homogeneous nucleation of a cross slipped segment on a dissociated dislocation is clearly prohibitive [5]. It is thus necessary to assume that cross slip is initiated at special constriction points, perhaps linked to jogs. (Surface cross slip should be easier at a surface constriction due to image forces [6]). Jogs can be formed by interaction of the dislocation line with point defects (which can explain the perturbations observed in slip bands), or by reactions of dislocations from different slip systems.

A key point for dislocation multiplication is the stability of the poles. Often sources are unstable since poles are mobile. "Wandering" sources similar to ours were observed by Louchet [7] in metals. In order to form more efficient sources (to be reported elsewhere) a pole created by cross slipping must be stabilised by something else.

Acknowledgments:

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Figures

Fig. 1. Stereographic projection of the silicon specimen.

Fig. 2. Topographs taken in situ from a "B" specimen strained at 975 K, 44 MPa. a; 1 min 40 s. b; 3 min 40s. c; 10 min. Mark: 0.5 mm.

Fig. 3. Post mortem image taken on a "A" specimen strained at 825 K, 22 MPa, 51 min $\bar{g} = \bar{2}\,0\,\bar{2}$ Mark: 0.5 mm.

Fig. 4. Slip band moving in a "B" specimen at 1075 K, 22 MPa, taken in situ at 15 mn. Mark: 0.5 mm

Fig. 5. Unstable dislocation source observed in situ in a "B" specimen strained at 1075 K, 22 MPa, 15 min (a); 19 min 10 s (b); 26 min 40 s (c) and post mortem (28 min). Mark: 0.5 mm.

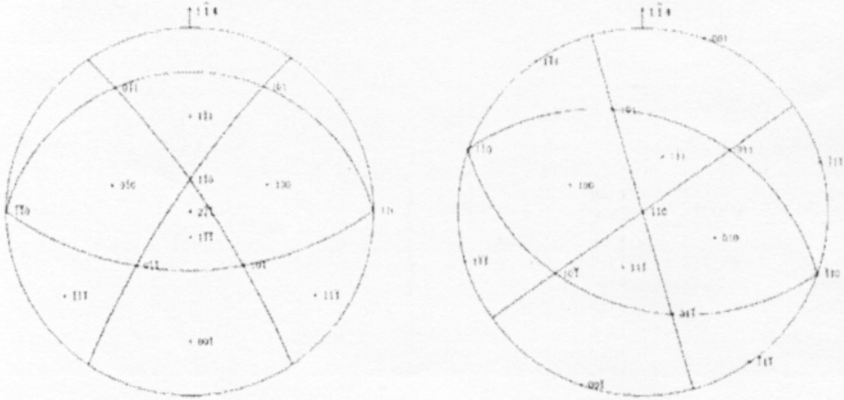
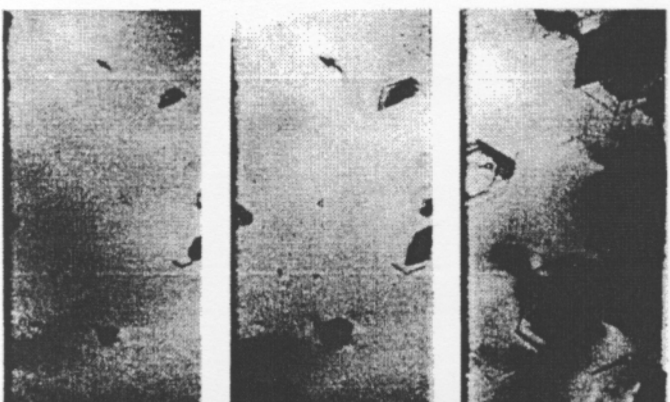


Fig. 1



a) b) c)

Fig. 2

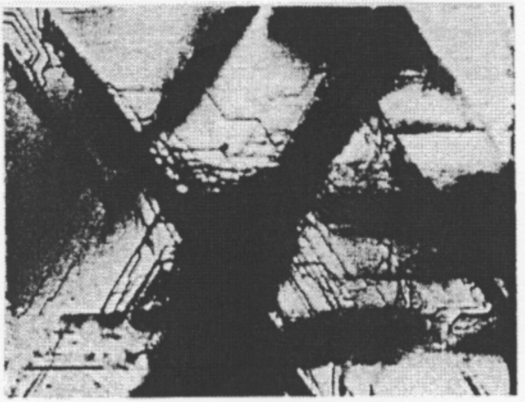


Fig. 3

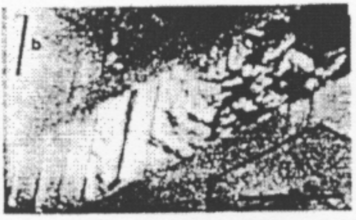


Fig. 4

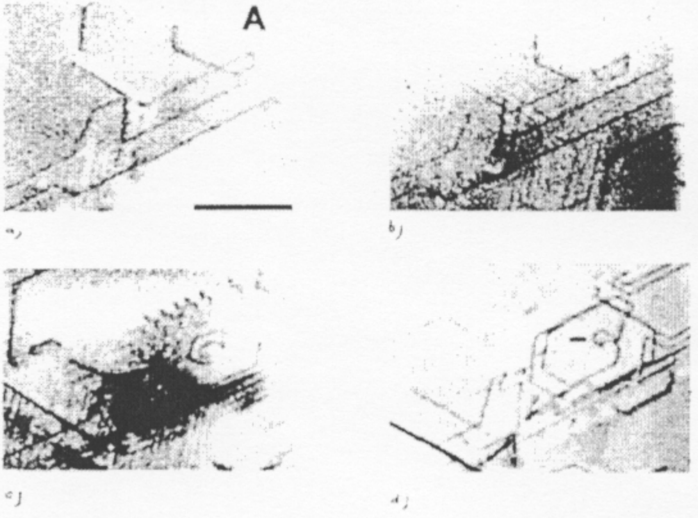


Fig. 5