How do the Dislocations Multiply at the Onset of Plastic Deformation in Silicon ?

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In situ x-ray topography of initially defect-free silicon crystals submitted to tensile stress at elevated temperature has provided evidence that dislocation arrays become non-planar with time, that cross-slip events are frequent even in the bulk and that sources of primary slip dislocations can be formed with the help of secondary slip systems.

Plastic deformation, the part that remains after stress has been removed (as opposed to elastic deformation), is tremendously important because it is used to shape many of the objects around us - car bodies for example. It is now well known that plastic deformation is related, at the microscopic level, to the behavior of line-shaped crystal defects, known as dislocations. However, a lot remains to be learned about the physics of this process. The present investigation deals with silicon, a fascinating material in many ways. On the one hand, silicon is grown industrially, for the microelectronics industry, in the form of single crystals with unprecedented purity, or with small, perfectly controlled, amounts of impurities (this process of doping is essential in electronics). This is convenient for the investigation of dislocation movement because impurities interact with dislocation lines. On the other hand, plastic deformation of silicon at room temperature does not occur - another unusual property of silicon, making it valuable in micro-mechanical applications such as ink-jet nozzles. Furthermore, silicon crystals are usually dislocation-free in the as-grown state, and achieving plastic deformation (at high temperatures) implies that dislocations have to be created and have to multiply. Finding out how this occurs, through direct observation, is the purpose of the present work.

How do dislocations multiply ?

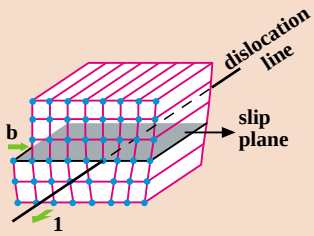
In crystals, plastic deformation generally proceeds by a glide of dislocations (see insert). Therefore, initially dislocation-free materials, like silicon, raise a simple question: how do dislocations multiply during the first stages of plastic deformation until they reach a typical density of about 10^8 cm.cm^{-3} at the macroscopic yield point?

When a dislocation-free silicon sample is submitted to an applied stress at a temperature high enough for dislocations to be mobile, the first dislocations form at residual defects acting as stress concentrators, preferentially surface defects resulting from machining (scratches, micro-cracks...), more rarely internal micro-defects inherited from crystal growth. However, such defects are too few to account for the observed filling of the whole sample with dislocations. Therefore, new sources must be continuously generated during plastic deformation, and most probably these new sources are formed by the existing dislocations themselves.

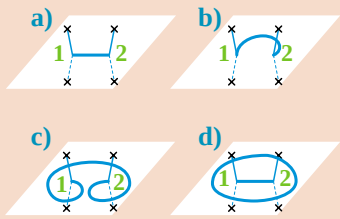
The basic configuration for dislocation multiplication is the Frank-Read source (see text in the frame) consisting of a dislocation segment which bows out between two pinning points, generating a set of concentric

GLOSSARY

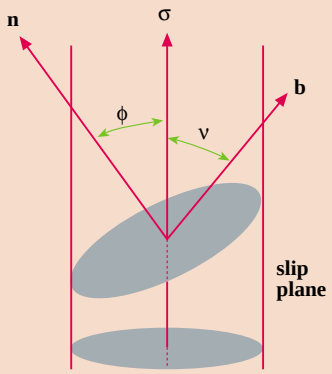
Dislocation: It is a line defect characterized by its direction **l**, which changes if the dislocation is not straight, and its **Burgers vector b**, which is constant along the whole dislocation. **b** defines the shear displacement produced in the crystal when the dislocation crosses it.



Frank-Read source: A dislocation segment, pinned at its extremities, (a), bows out in its slip plane under the shear stress (b, c). The two opposite dislocation segments annihilate, generating a closed loop and restoring the initial configuration (c, d).



Schmid factor: The stress which does work when a dislocation glides is the shear stress parallel to the slip plane, in the direction of the Burgers vector. The relationship between the resolved shear stress τ, on a given slip system, and the applied tensile stress σ, is $\tau = s.\sigma$ where s is the Schmid factor $s = \cos\varphi \cos\nu$ ($0 \leq s \leq 0.5$).



loops. The multiplying segment must have first escaped from its original slip plane and have been transferred onto a parallel plane, where it is free to bow out around pinning points. In fcc crystals, this is usually assumed to proceed by double cross-slip, since dislocations with 1/2<110> Burgers vector can glide in two dense {111} planes. However, cross-slip is not a simple process and conditions for its occurrence are still poorly known, especially in the bulk. The purpose of the present experiment was to shed some light on this mechanism.

The understanding of the rather complex behavior of dislocations has largely relied on their observation, and continues to do so. Because they entail long-range distortion of the lattice, dislocations can locally affect Bragg diffraction, hence be seen through transmission electron microscopy (TEM) in thin foils, and, in the bulk, by x-ray Bragg-diffraction imaging, traditionally called x-ray topography. An important feature is that both characteristics of single dislocations can be assessed, in particular through the disappearance of the image ("out-of-contrast" situation) for some Bragg reflections.

WHY TOPOGRAPHY ?

Dislocation sources were observed by *in situ* TEM but, in thin foils, surface effects are predominant. X-ray topography offers a unique possibility to observe large volumes of crystal, under well-defined stress and temperature conditions. Its main drawback is a spatial resolution which limits the acceptable dislocation density to ~ 10^4 cm.cm^3, far below its value at the macroscopic yield point. The present study was based on synchrotron radiation Bragg-diffraction imaging, which is very well suited to *in situ* investigations, at the ESRF ID19 beamline. It was complemented by the laboratory technique for x-ray topography, Lang's method.

In silicon, dislocation velocity is thermally activated and is roughly proportional to the resolved shear stress τ, which, on a particular slip system is equal to $s \cdot \sigma$, where s is the Schmid factor (see insert) and σ the applied stress. The kinetics that are compatible with *in situ* observations at the ESRF prevail at temperatures of ~ 1000 K under nominal stresses σ of about 25 to 50 MPa, which are convenient for tensile testing.

Experiments were performed with a mechanical testing system custom designed and constructed at Nancy for the "horizontal" diffractometer of the ID19 beamline. Tensile samples (~ 15 x 4 x 0.7 mm^3 in size) were used. The stress axis was parallel to [1, $\bar{1}$, 4], which corresponds to a double slip orientation, with two primary slip systems of s ~ 0.45. Corresponding cross-slip systems had s ~ 0.23. The samples were polished with great care, in order to remove machining defects. Vickers micro-indentations were made on the large faces of the samples in order to nucleate the first dislocations.

Dislocation configurations were followed under creep conditions (constant stress and temperature), in a neutral gas to avoid oxidation. Topographs were recorded on x-ray films, using a –2,2,0 Bragg reflection with a monochromatized beam (λ ~ 0.7 Å) at chosen time intervals. The FRELON camera was used for real-time observations of the evolving configurations. The experiments were stopped before the dislocation density was too high, so that a complementary stereo-pair and Burgers vector analysis could be performed later by conventional Lang topography. Since plastic deformation always created elastic strain gradients in the sample, it was necessary to scan over ~ 0.02 - 0.1 degrees around the Bragg angle to obtain a full image, so that the exposure time could not be less than 30 seconds.

ANALYSIS OF THE TOPOGRAPHS

Figure 1 shows two topographs from a sequence recorded at T = 1070 K, σ = 22 MPa and a Lang topograph of the final configuration. A dislocation source, labeled S, can clearly been seen. Such a source was seldom observed and it results from a complex sequence. What follows is a description of some of the elementary mechanisms involved:

(i) cross-slip. Its presence is apparent from the hexagonal half- or full-loops (marked by arrows on Figure 1c) coming out of dense slip bands. These bands consist of 1/2 [$\bar{1}$,0,1] dislocations lying in (1,$\bar{1}$,1) planes, which are seen nearly end-on here. Analysis confirmed that out-going loops have the same Burgers vector and develop in (111), i.e. the corresponding cross-slip plane. Further, cross-slip started in the bulk and not at a free surface.

(ii) non-planarity of dislocation arrays. Another striking feature appears on the dislocation array running from right to left above the source. Only

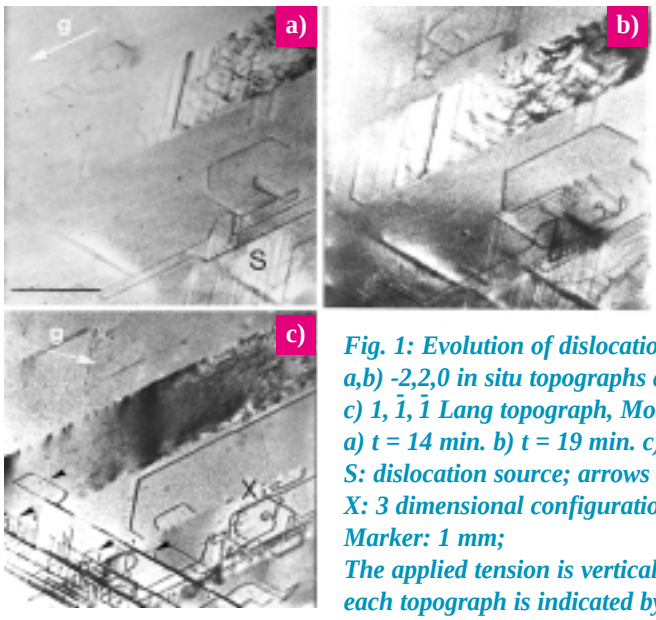


Fig. 1: Evolution of dislocation configurations in silicon:
a,b) -2,2,0 in situ topographs at T = 1070 K, σ = 22 MPa.
c) 1, $\bar{1}$, $\bar{1}$ Lang topograph, Mo Kα radiation.
a) t = 14 min. b) t = 19 min. c) after cooling down at t = 27 min, no stress applied.
S: dislocation source; arrows in (c): cross-slipped loops.
X: 3 dimensional configuration formed on two intersecting {111} planes (see text).
Marker: 1 mm;
The applied tension is vertical. The direction of the diffraction vector used for each topograph is indicated by its projection on the image plane.

one slip system is active in this band, which is out-of-contrast in Figure 1c, topograph: 1/2 [0,$\bar{1}$,1](111) (s ~ 0.454). As usual in silicon, the leading dislocations (seen on the left) are straight and parallel to [0,$\bar{1}$,1]. Dislocations become more and more wavy as their distance from the leading ones increases. They then consist of segments developing in several parallel planes connected by jogs. Such non planar configurations may result from cross-slip, but also from climb due to absorption or emission of point defects. Jogs may provide pinning points for a Frank-Read source.

(iii) reactions with secondary slip systems. Unexpected dislocations with low s were repeatedly observed. They were formed at micro-indentations or as the result of reactions between dislocations of other systems. A typical configuration is shown in Figure 1c, where primary dislocations are out-of-contrast. Some dislocations have 1/2 [$\bar{1}$,1,0] Burgers vector and develop simultaneously on (1,1,$\bar{1}$) and (111), both planes having the same resolved shear stress (s ~ 0.18). Such three-dimensional configurations seem to be important for the development of new sources in formerly inactive slip planes.

CONCLUSION

The synchrotron radiation delivered by the ESRF enabled us to perform unique *in situ* observations of evolving dislocations within the whole volume of thick and large crystals under stress. They have revealed several microscopic mechanisms involved in the filling of the samples with dislocations starting from a few initial sources. The temperature and stress dependencies of these mechanisms remain to be determined. This will imply a significant decrease in the exposure times, which should be feasible with the existing equipment through further optimization.

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