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Dynamic observation of dislocation sources at grain boundaries in ice

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ABSTRACT

This Letter presents the first clear demonstration of a grain-boundary dislocation generation mechanism in ice. The dislocations generated as semihexagonal loops were observed to glide on the basal plane.

§ 1. INTRODUCTION

In single-crystal ice, the dislocation mechanisms which operate in the early stages of plastic deformation have been fairly well established (Higashi 1988, Ahmad and Whitworth 1988). However, the situation for polycrystalline ice is much less satisfactory, even though ice naturally occurs as polycrystals. An examination of the role of grain boundaries during deformation has been attempted previously using conventional X-ray topography (Hondoh and Higashi 1983), but the true dynamic behaviour was not revealed because of the long exposure times required (10 min–2 h). In this letter, we present topographs, which were obtained using white radiation from a synchrotron source, of dislocation generation at a grain boundary in ice.

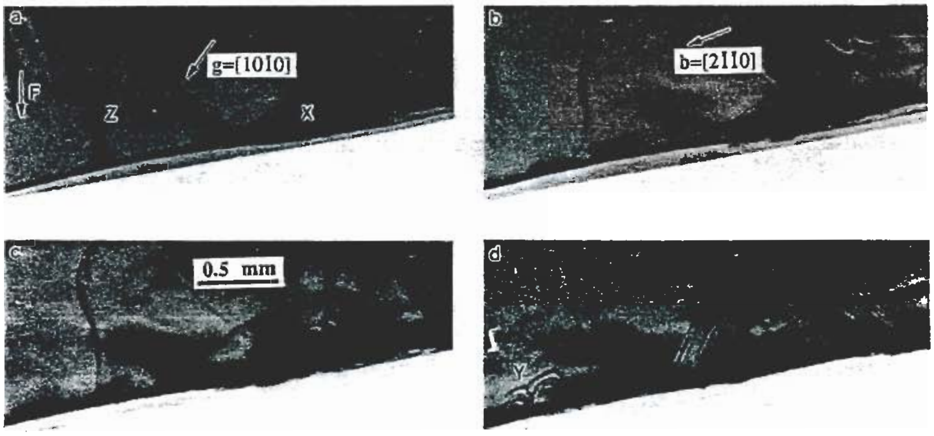
§ 2. EXPERIMENTAL

Columnar-grained ice with a very low initial dislocation density was grown using a seedless Czochralski method. Topographs were obtained by allowing a highly collimated beam (6 mm × 15 mm) of white X-rays to fall on a large-angle grain boundary in a large-grained polycrystalline ice sample 2 mm thick. Several diffraction spots were recorded simultaneously in a single exposure which typically took 8 s. Each Laue diffraction spot is an image of the grain in which a grain boundary and dislocations can be observed (provided $\mathbf{g} \cdot \mathbf{b} \neq 0$, where $\mathbf{g}$ is the diffraction vector and $\mathbf{b}$ the Burgers' vector). The orientation of the grain examined is such that the basal planes on which dislocations glide in ice were projected with a little geometrical distortion onto the plane of the film. *In situ* straining experiments were performed using a specially built compression jig assembled within a cryostat. During dynamic loading, the sample was maintained at − 12°C, and a sequence of compressive stresses, ranging from 0·4 to 2 MPa, were applied for periods of about 5 min between exposures.

§ 3. Results and discussion

The figure shows enlarged portions of four topographs, taken between intermittent loadings, in which three dislocation sources were observed in the same area of a grain. The photographs shown are of a $[10\bar{1}0]$ spot at a Bragg angle of 7·9°, corresponding to a wavelength of 1·07 Å. The loading direction, shown as F in the figure, makes an angle of about 80° with the grain boundary. The shear stresses exerted on the grain boundary are of the order of 0·1 MPa. After pre-stressing this sample, the grain boundary was subjected to a shear stress, and a stress field is built up around the facetted grain boundary, marked X in (*a*). Note that there were only a few dislocations pre-existing in the grain prior to deformation. The extent of the strained area indicates the strain energy stored in this area. Upon further loading, semi hexagonal dislocations with $[2\bar{1}\bar{1}0]$ Burgers' vectors were pushed out of the stress concentration centre on the grain boundary, shown in (*b*). In the topograph in (*c*), the main dislocation segment visible in (*b*) has glided about 0·8 mm on the basal plane into the interior of the grain, while the dislocation generation process continues. At the same time, several stress fields are generated and they cover a large portion of the grain boundary. The further that the dislocations glide away from the dislocation source, the larger are the dislocation spacings. Near the dislocation source, individual dislocations cannot be discerned because the spacing between the adjacent dislocations is below the resolution limit of X-ray topography (about 10 μm). These $[2\bar{1}\bar{1}0]$ dislocations glide on basal planes continuously as the compressive strain increases. That the strain field is relaxed by dislocation generation is indicated by the smaller scale of the stress field near the source in (*d*) compared with that in (*c*). The spacing between the first and the second dislocations, shown in (*d*), is 28 μm, while the source is still the centre of these $[2\bar{1}\bar{1}0]$ dislocations. Further stressing activates another two similar sources, marked Y in (*d*). The closer spacing of the screw than the 60° segments is fully in accord with dislocation velocity measurements (Shearwood and Whitworth 1991).

On a more microscopic scale, this dislocation generation mechanism at the large-angle grain boundary is not wholly clear. A possible explanation of the mechanism is as follows. Most grain boundaries in columnar ice are not straight. The curved grain boundary is composed of low-energy planes and steps formed by intrinsic grain-



Four X-ray topographs showing operation of the dislocation sources at a large-angle grain boundary. The slow glide of the basal dislocation, marked Z in (*a*), is driven by a small resolved shear stress on the basal plane, which is purely determined by the external stress.

boundary dislocations. When a compressive stress is applied to the ice sample, grain-boundary sliding occurs, driven by the shear stress on the grain boundary. Then stress fields are generated and extend from the intersections of the grain-boundary facets. In this case, the accumulation of extrinsic grain-boundary dislocations gliding to the intersections may also contribute to the large stress concentration. Once the stored elastic energy reaches a critical value, lattice dislocations are generated and then glide away from the facet intersection to release the stored energy. However, there was no evidence from the topographs to show how intrinsic grain boundaries form the facetted grain boundary and whether extrinsic grain-boundary accumulation contributes to stress concentration at the dislocation source. No dislocation absorption at the grain boundary was observed either. According to our explanation, dislocation generation and multiplication should occur when the grain boundary is under a shear stress and a resolved stress on the basal plane is provided. Since these are general geometrical conditions under which polycrystalline ice is deformed, and since basal glide has been proven to be the main plastic deformation mechanism (Hobbs 1974), this dislocation generation mechanism should be dominant in the early stages of plastic deformation of polycrystalline ice.

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