Temperature Dependence of Dislocations in Notched Ice Crystals

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A study of the effect of temperature on the dislocation structures in notched ice crystals in constant strain rate tests was performed in order to investigate the fundamental processes involved in the brittle-ductile transition in ice. It has been shown that there is far more dislocation activity around notches at high temperature (−7.8 °C) than at lower temperatures (−40 and −60 °C).

Introduction

Deformation studies of single-crystal silicon have shown that dislocation emission in front of a crack tip is associated with the transition from brittle to the ductile behavior.[1−5]

In contrast, the brittle−ductile transition in ice single crystals has not been studied on either the macroscopic or the microscopic level. Macroscopically, it is difficult to carry out such experiments on ice single crystals oriented for easy glide since they are still ductile at temperatures as low as −70 °C,[6a] which is lower than the temperatures normally experienced terrestrially (0 to −50 °C) by ice Ih. For single crystals oriented for nonbasal glide, deformation tests have only been carried out in the ductile regime. Microscopically, what is happening in front of cracks at the dislocation level is poorly documented. The first *direct* observation of dislocations in front of a notch in ice was made by Hu, Baker, and Dudley[7] using synchrotron X-ray topography. They showed that dislocation emission from a loaded notch depends strongly upon the crystallographic orientation of the single crystal. In a favorable orientation for dislocation glide, the dislocations near the notch move easily and multiply, thereby contributing to the crack tip ductility.

In order to examine the brittle fracture behavior of ice single crystals under our current experimental conditions, we needed to use the following approaches: (1) use ice crystals which are as brittle as possible by choosing an appropriate orientation (described in more detail in the Experimental Section); (2) set the testing temperature as low as possible; (3) set the strain rate as high as possible.

The aim of the experiments described in this paper is to understand the dislocation behavior around loaded notches at different temperatures by direct observation of their arrangements using X-ray topography. Similar to the observations in silicon, we expected to see that for ice specimens deformed at high temperature, numerous dislocations would be generated at the stationary notch, while for ice deformed at low temperature, fewer dislocations would be emitted. These experiments provide the basis for studying the brittle−ductile transition in ice in the near future.

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Experimental Section

The growth of ice crystals and the general preparation of specimens have been described in detail elsewhere.[8−10] Notched sheet tensile specimens with (0001) perpendicular to the loading direction were produced as described elsewhere.[7] In this orientation, the shear stresses on all slip planes from the far stress field are zero. Such crystals are expected to be more brittle than those with any other crystallographic orientation.[7]

Specimens were strained at a constant strain rate of 1×10^{-8} s^{-1} at either −7.8, −40, or −60 °C in a loading jig equipped with an environmental cryostat controlled to ±0.1 °C. Strain was determined by counting the number of pulses sent to the stepper motor which was mounted on the end of the jig. For each temperature, a series of X-ray topographs were taken to record the real-time dislocation activity in front of notches during *in situ* deformation.

Results and Discussion

Dislocation configurations in the notch-tip regions for the three specimens are shown in Figures 1 and 2. Darkened areas appeared in front of the notch tips on loading and disappeared upon stress removal, indicating that the darkened areas were caused by elastic stress fields. The symmetry of the elastic field in front of the notch was expected based on the configurations of the notch and the crystal. It is interesting to notice that the shape of the darkened areas is very similar to that of the contour of the shear stress on the basal/notch plane (Figure 3). These darkened areas are mainly caused by the distortion of the lattice and the distortion of the lattice is mainly due to the shear stress.

The crystals were so oriented that the basal planes were seen edge-on and dislocations on these planes appeared as straight lines.

Figure 1 shows the deformation at −7.8 °C. Existing dislocations (Figure 1a) moved quickly to the notch tip (Figure 1b,c), interacted with the tip, and then the notch tip began to evolve into an efficient dislocation source (Figure 1d). As can be seen in topograph Figure 1d, on the upper side of the notch, dislocations separated into two segments and moved in opposite directions. Thus, a dislocation-depleted area marked DD appeared. On the other side of the notch, a zigzag line nucleated from the notch tip and propagated away from the cracktip.

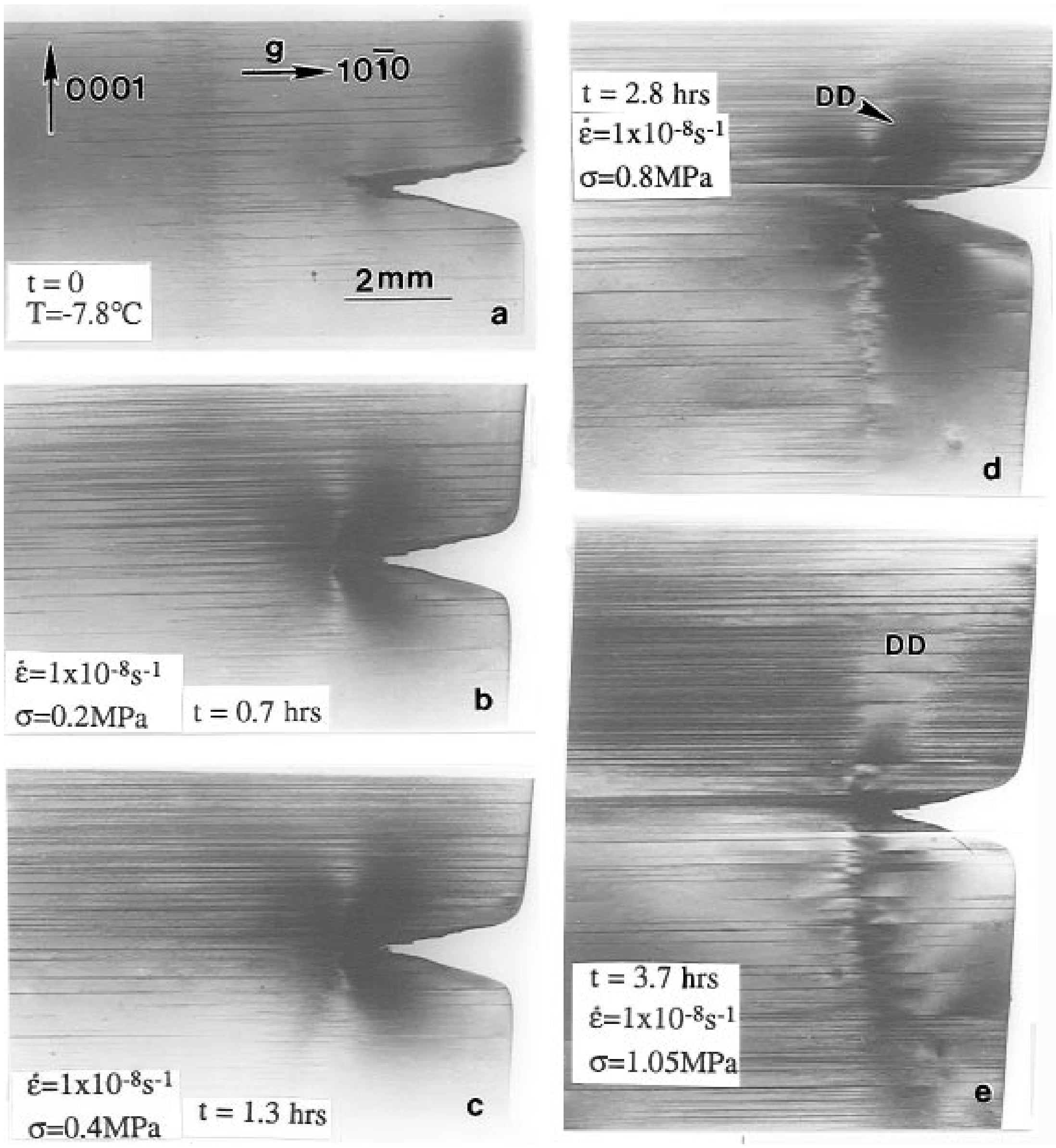

Figure 1. Sequence of topographs at −7.8 °C showing the dislocation configurations around the notch in ice single crystal during *in situ* deformation at constant strain rate of 1×10^{-8} s^{-1}: (a) before loading; (b) after deformation 1 h; (c) after deformation 1.3 h; (d) after deformation 2.8 h; (e) after deformation 3.70 h.

Meanwhile, numerous dislocations pile up on this line. All these features are clearer in Figure 1e where the load has been removed. The shear stress on the basal plane is symmetric with respect to the notch plane as indicated in Figure 3, but the dislocation motion on both sides of the notch is not symmetric. This may be because the moving dislocations on one side of the notch have opposite Burgers vector to that of dislocations on the other side of the notch and they move in the opposite direction under the same shear stress.

Figure 2 is a sequence of topographs showing deformation at −40 and −60 °C. Topograph 2a was taken before loading, while topograph 2b was taken at almost the same time as that for topograph 1d. Although some dislocations did increase in length after long straining times (topograph 2b), they did not increase as fast as the dislocations in Figure 1. In other words, the dislocation mobility is less at this temperature.[11] In addition, there is no evidence that the notch acted as an efficient dislocation source at this point.

Topographs 2c and 2d were taken before loading and after loading for 2.9 h (a very similar time to that for topograph 2b), respectively. As can be seen in topograph 2d, there is even less dislocation movement at this temperature. Hardly any new dislocations have been generated during deformation. The dislocations present were introduced during ice growth and by the notch-making technique. The elastic modulus along the *c*-axis increased ∼10% at this temperature compared to that at −7.8 °C.[6b] The shape of the darkened area is also different from those in Figures 1 and 2b, but this is probably due to the lesser acuity of the notch rather than a temperature effect in this case. Small spotty images are characteristics when the specimen is below −50 °C. They are associated with condensation of excess point defects. The dark features marked L are frost formed during cooling. They attached loosely to the specimen surface and did not affect the deformation of the crystal.

It is not possible to determine if dislocation behavior at −60 °C represents the brittle deformation behavior of ice single crystals for the orientation used, since the complete stress−strain curve is not available. Mechanical tests to obtain it are under the way.

The above observations that dislocations are more active in the region near the notch tip at higher temperature than at lower

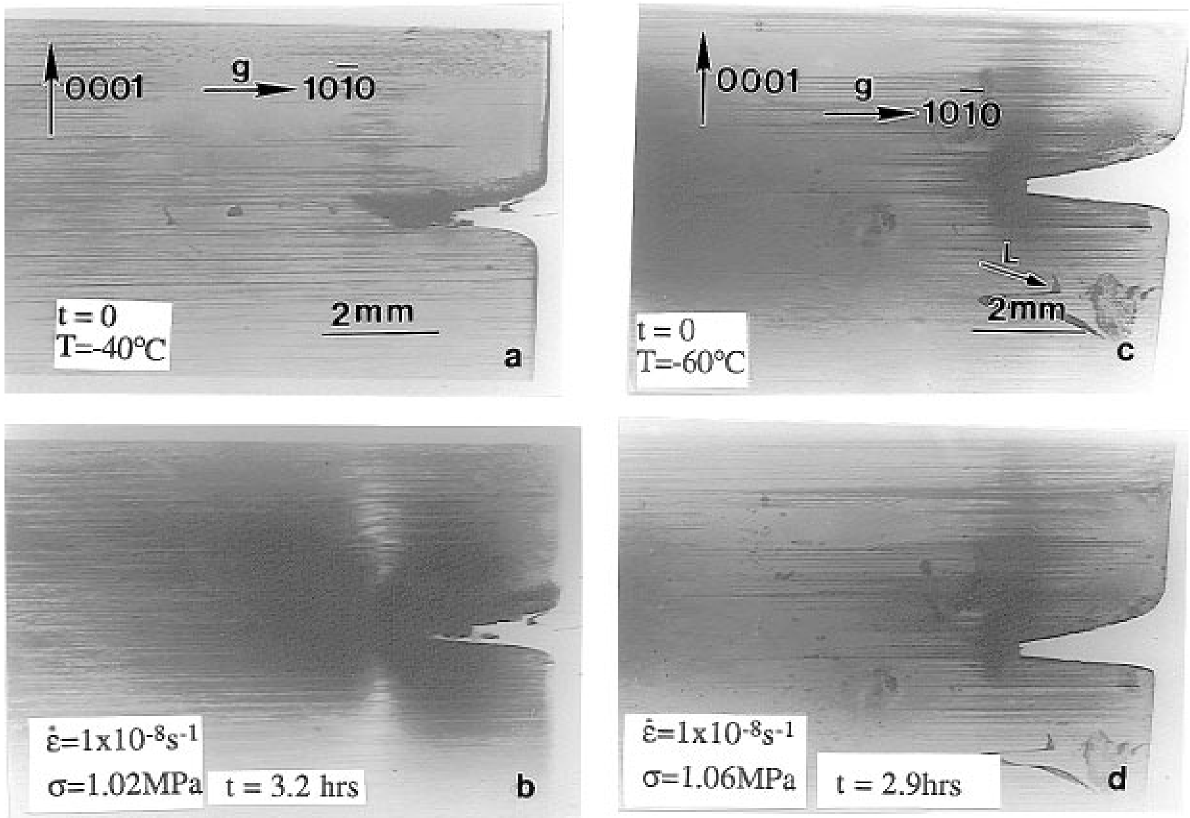

Figure 2. Sequence of topographs showing the dislocation configurations around the notch in ice single crystal during *in situ* deformation at constant strain rate of 1×10^{-8} s^{-1}: (a) at −40 °C, before loading; (b) at −40 °C, after deformation 3.2 h; (c) at −60 °C, before loading; (d) at −60 °C, after deformation 2.9 h.

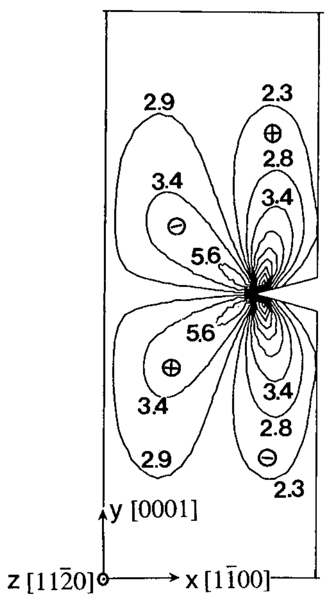

Figure 3. A schematic of the contour of the shear stress σ_{xy} around the notch under current loading condition, shown on the section normal to the notch plane. The value of the normalized stress is indicated on each contour. This calculation is based on the solution to the plane stress field around a crack subject to a uniaxial loading, assuming that the ice is isotropic. The magnitudes and signs of the shear stresses on the four regions indicate that the shear stress around the notch is symmetric with respect to the notch plane.

temperature indicate that the dislocation behavior in front of a notch may contribute significantly to the notch ductility.

Summary

This study of the temperature dependence of dislocation behavior around the notch tip of ice single crystals forms the basis for a study of the brittle−ductile transition in ice. It shows that the notch affects dislocation activity more significantly at higher temperature (−7.8 °C) than at lower temperatures (−40 and −60 °C).

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