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ABSTRACT

Etching and replicating in conjunction with a microtoming technique and scanning electron micrography has been used to provide evidence in polycrystalline ice of climbing of basal dislocations on planes parallel to the c-axis.

RÉSUMÉ

On s'est servi de l'attaque à l'acide et de la réplication, en association avec une technique de microcoupe et la photomicrographie éelctronique à balayage, pour prouver l'existence, dans la glace polycristalline, du phénomène de montée des fractures basales sur des plans parallèles à l'axe c.

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CHAPMAN AND HALL

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Dislocation climb in ice observed by etching and replicating

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Ice belongs to the family of hexagonal crystals usually existing in nature at temperatures greater than $0.8\,T_m$, where T_m is the melting point in Kelvin. At these high temperatures a single crystal of ice deforms, when loated, primarily by slip on the basal plane because non-basal slip is significantly more difficult. In a polycrystalline state, however, both types of slip have been observed [1]. Polycrystalline ice creep also involves dislocation climb, and because of the high temperatures involved, diffusion-controlled climb of dislocations on planes normal to the basal plane may even be the rate-controlling process during steady-state creep [2]. Etching and replicating in conjunction with a microtoming technique developed earlier [1] has been used to provide evidence in polycrystalline ice of climbing of basal dislocations on planes parallel to the c-axis.

The process of etching and replicating ice surfaces has been described in detail [1]. Briefly, it consists of preparing the required surface of ice to a mirror finish by carefully removing the disturbed surface layers by means of a microtome with a freshly prepared blade. The surface quality is examined visually using both reflected light from a distant source and an optical microscope. The surface is then coated with a dilute solution (1 to 10%) of polyvinyl formal (Formvar) in ethylene dichloride and allowed to dry under controlled conditions. As the solution dries it etches the specimen to different degrees depending on concentration and drying condition. Thus the process allows both etching and replicating to be conducted at the same time. When dry, the plastic replica is peeled off or the ice is removed by sublimation. Replicas up to 80 mm × 120 mm have been made in this way, providing large areas for examination. The replica is mounted on a large glass plate with the replicated surface facing upwards so that it can be observed with an optical microscope. If required, selected areas are prepared for examination with a scanning electron microscope (SEM) by vacuum deposition of a layer of carbon followed by plating with gold.

A basal dislocation that lies parallel to the glide plane can be detected readily by the formation of a centrally depressed, elongated etch pit at the point where it intersects other planes. If the intersecting surface is normal to the basal plane (0 0 0 1), then the etch pit is symmetrical with respect to the central depression and its long axis is parallel to the c-axis or $\langle 0001 \rangle$ axis of the crystal. Examples of tiny elongated-dislocation etch pits on the $\{11\bar{2}0\}$ surface may be seen in Fig. 1, where large evaporation pits [1], with their major plane parallel to the basal plane, clearly show the direction of the crystallographic axis of symmetry. The central depression corresponds to the core of the dislocation.

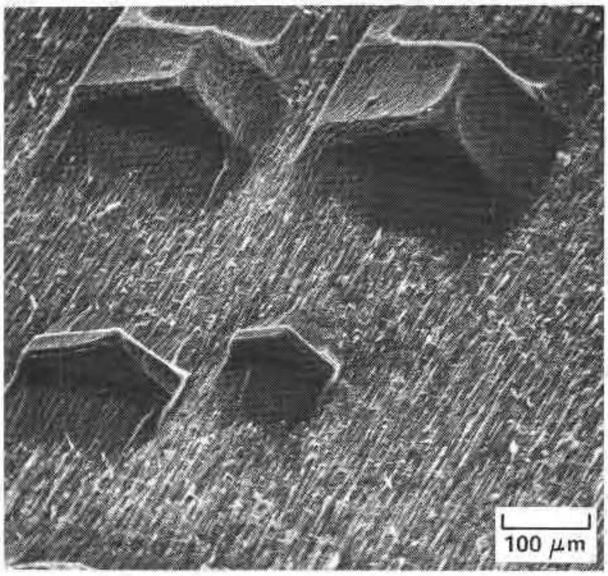

Figure 1 Scanning electron micrograph of a replica of large-evaporation and minute-dislocation etch pits on $\{11\bar{2}0\}$ surface. Dislocation etch pits are elongated parallel to $\langle 0001 \rangle$ axis.

The depth of the central depression can be controlled by suitable choice of etchant concentration, thickness of etching layer, and vapour pressure (evaporation time). Etch pits with depressions as long as 200 μm along the core may be produced in this way, corresponding to a stationary dislocation [3]. When

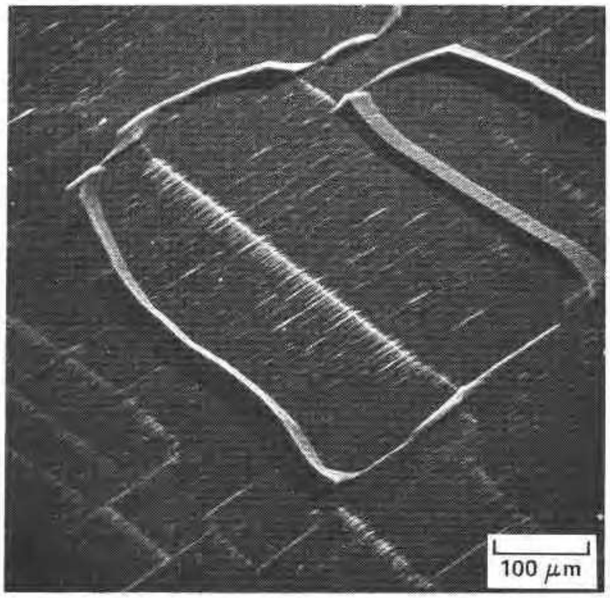

Figure 2 Scanning electron micrograph of etch features of basal dislocations inside a previously deformed grain.

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observed by SEM, these etch pits give the appearance of "whiskers" in the replica and have, in fact, been used by the author to remove the ambiguity sometimes associated with the correspondence between etch pits and dislocations [1]. For most studies it is sufficient to develop only the centrally depressed, elongated etch pits by choice of the etching conditions because whiskers often obstruct the view.

Movements of dislocations can be detected by etching and replicating the required surface while the material is under load. Only the slowly moving dislocations can be detected by the formation of etch tracks along the paths traversed by the line defects. Mobility of non-basal [1] and basal [3] dislocations has been demonstrated. If the dislocations are blocked then pile-up may occur, as shown in Fig. 2 for basal dislocations in a previously deformed ice specimen. Note that the pile-up direction is normal to the direction of the long axis of the etch pits.

Climb of basal dislocations out of their slip plane should produce etch tracks parallel to the long axis of the etch pits or the $\langle 0001 \rangle$ axis while etching prismatic surfaces under a load. Both slip and climb usually occur, producing etch tracks at right angles to each other so that the replica resembles a fabric. Occasionally the author has noticed only etch tracks parallel to the $\langle 0001 \rangle$ axis, corresponding to the motion at right angles to the direction of slip. An optical micrograph of such a replica is shown in Fig. 3. In this case the ice was transversely isotropic, with the $\langle 0001 \rangle$ axis of the grains randomly oriented in the plane of isotropy and the replicated surface.

Most of the etch tracks in Fig. 3 extend from one grain boundary to the other, indicating that the grain boundaries act as sources and sinks for the dislocations, and that dislocations generated at the boundaries travel to other boundaries through the matrix. Non-uniformity of the stress field is also evident in this micrograph in the form of patches of high-density etch tracks. These high-density patches are closely associated with triple points (for example, top right corner of the centrally located small grain, Fig. 3) or turning points in the grain boundaries. Some of the etch tracks have one end at a grain boundary and the other end terminating abruptly at a point inside the grain (particularly in the central area of the micrograph). Scanning electron micrography (Fig. 4) shows that such etch tracks seem to start at the boundary and end inside the grain. The end points of the climbing dislocation are revealed by the formation of whiskers, indicating that for some reason dislocation came to a stop while the etching process continued along the core of the dislocation. In the replica it appears as a whisker. It may also be seen here that some of the etch tracks have ends without whiskers.

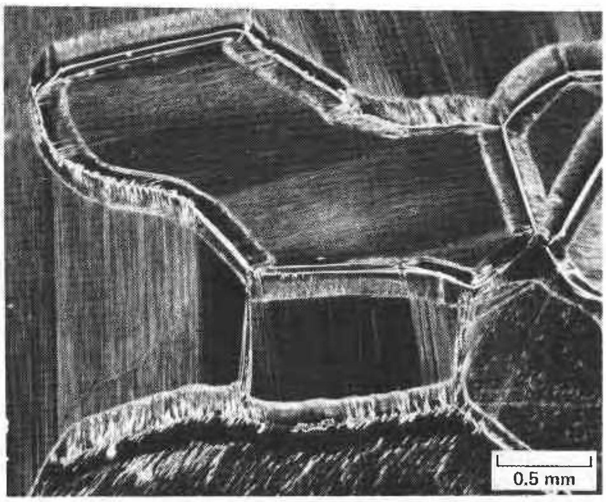

Figure 3 Optical micrograph exhibiting a few grains and etch tracks corresponding to moving dislocation.

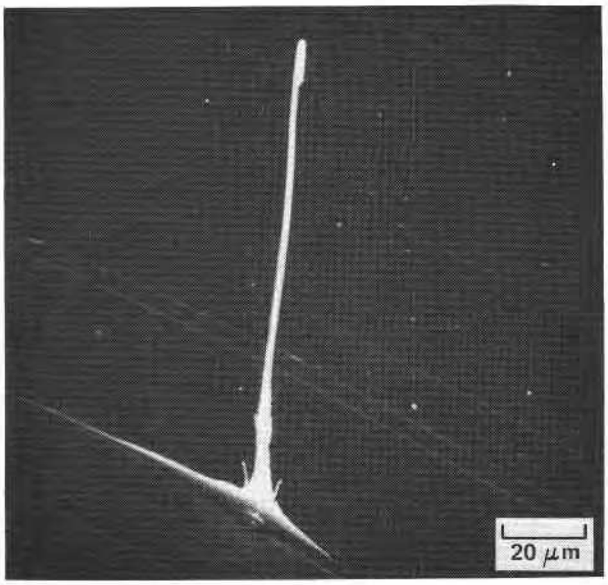

Figure 4 Etch tracks and a "whisker" corresponding to the core of a dislocation.

Fig. 5 shows a bent whisker at the end of a track and a series of etch tracks with different widths corresponding to different velocities. As the whiskers are very delicate, they are often not stiff enough to stand on their own. Instead, they fall back on the surface, as may be seen in Fig. 6, which also shows many elongated etch pits that indicate the direction of the

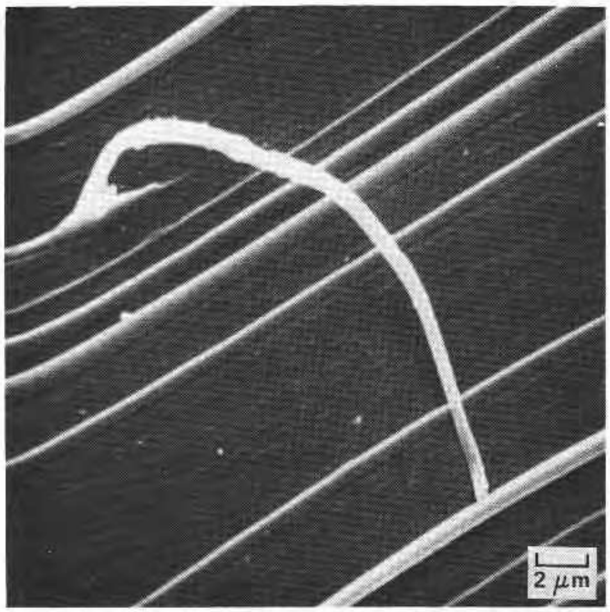

Figure 5 Bent whisker at the end of an etch track, and tracks of different widths showing different dislocation velocities.

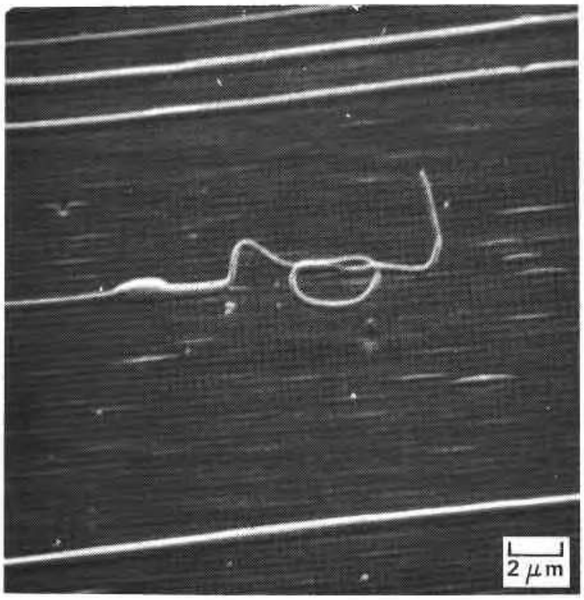

Figure 6 Etch pits, whisker and etch tracks.

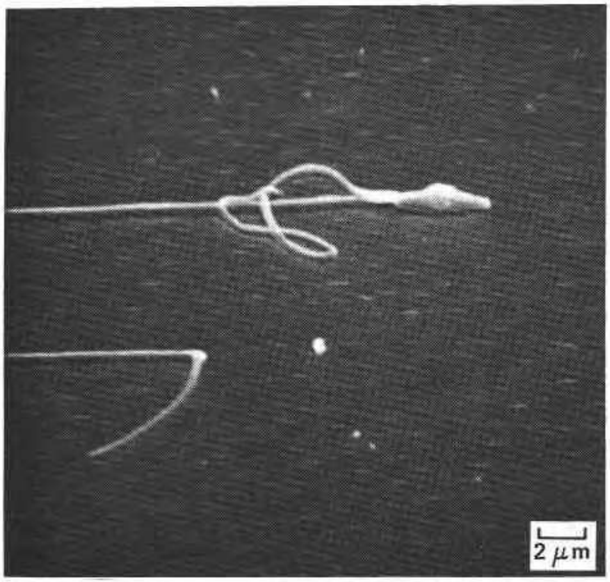

Figure 7 Different arrival times indicated by whisker lengths.

c-axis. Fig. 7 shows two whiskers of different length, signifying that one came to a stop earlier than the other. Fig. 7 also provides an opportunity to estimate the density of the basal dislocations in the ice from the number of etch pits, which are rather uniformly distributed. The density was found to be about $6 \times 10^5\ m^{-2}$.

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