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GRAIN BOUNDARY SLIDING IN ICE

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Résumé - Il est bien connu que les mécanismes actifs aux joints de grains - glissement intercrystallin, migration du joint - , contribuent à la déformation à hautes températures des matériaux polycristallins, en particulier pour les métaux. Récemment, ces mécanismes ont été corrélés à la structure des joints. Notre but est l'étude du glissement et de la migration des joints dans la glace, en fonction de la température, de la contrainte appliquée et des paramètres cristallins caractérisant les joints. Nous présentons ici nos premiers résultats, obtenus sur des bicristaux ayant des joints de grand angle.

Abstract - It is well known that grain boundary processes - intercrystalline sliding and grain boundary migration - make an important contribution to high temperature deformation of polycrystalline materials, particularly for metals. More recently, these processes have been correlated with the atomic structure of the boundaries. Our goal is to study sliding and migration of ice boundaries, with respect to temperature, stress, and the crystallographic parameters which characterize the boundaries. We here present our first results, obtained on bicrystals with high angle boundaries.

1. Introduction

The importance of grain boundaries in the high temperature deformation of polycrystalline materials is now well established. In metals, grain boundary sliding has been measured to make an important contribution to high temperature creep. Recent studies have been able to correlate the structure of grain boundaries to their migration and sliding during deformation.

Our purpose is to study the kinetics of grain boundary sliding and migration in ice as a function of stress, temperature, and the internal parameters which characterize the atomic structure at the boundary. This is done by submitting bicrystals of different orientations to creep shear stresses. In this paper we present our first results on randomly oriented bicrystals submitted to a constant shear stress parallel to the grain boundary macroscopic plane.

2. Experimental Procedure

The bicrystals used for these experiments were grown by a modified Czochralski solidification technique, used by Landauer [1] and by Homer and Glen [2]. A Peltier cell, which acts as a heat sink, is placed in contact with a plate, onto which are frozen two preoriented single crystal seeds, or a bicrystal seed. The Peltier cell and plate are placed on top of a beaker of water so that the seeds just touches the water surface. The water in the beaker is kept at constant temperature. A temperature gradient between the water and the ice seed is then established, driving the seed's growth into the liquid. The growth rate is controlled by slowly removing water from the beaker through an outlet.

The crystallographic orientations of the bicrystals were determined by indexing the optical back reflections obtained from hoar frost crystals grown on the surface of a transverse cut at the end of the bicrystal. Etch pits were also grown in order to confirm the measurements. The actual specimens for the shearing experiments were obtained by hot die extrusion, which produces a long ingot with a rectangular cross section, divided in half by the grain boundary. This method has been used by Itagaki [3], and produces a low dislocation density near the surface. Samples were then cut from the ingot by

perpendicular cuts, followed by a slight melting of the cut surfaces. (Figure 1) Their dimensions are 15 mm height, and a 24 mm x 12 mm section. The grain boundary plane has a mean surface of about 15 x 12 mm. Scratches were made on the sample surface so as to reveal the relative displacement between the crystals.

The creep device used in our experiments has grips which allow the application of a stress state in which the shear stress is concentrated on the grain boundary plane. (Figure 2) The average shear stress on the boundary is specified by the load which must be transmitted across the boundary from one grip to the other. Tension and compression stresses may be present parallel or perpendicular to the boundary, particularly if there is deformation within the grains. Parasitic torsion and bending stresses are minimized by the symmetrical arrangement of the sample in the grips. The displacement was measured by an LVDT and recorded continuously. The bicrystals were placed between crossed polarizing sheets, and backlighting allowed observations with crystalline contrast during the test. Photographs were taken at regular intervals during the tests. The entire experimental setup was placed inside an insulated box in a cold room. Near the sample the temperature oscillations were less than 1° C.

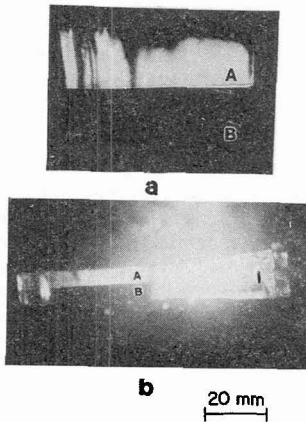


Figure 1. (a) Bicrystalline grown ingot. (b). Parallelepiped hot extruded ingot. A and B are the crystals; I corresponds to a support of ice for the extrusion.

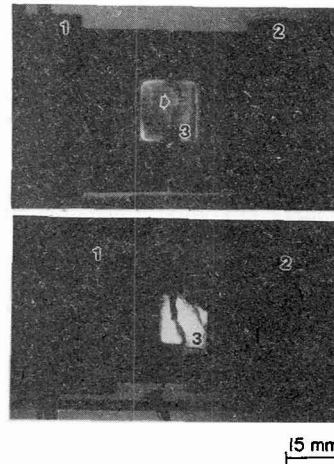


Figure 2. Two view of the shearing device 1-Fixed Grip, 2-Mobil Grip, 3-Bicrystal sample. White arrow shows GB migration during a preliminary test. Lower bicrystal shows cracking that occurred when the load was removed from Bicrystal 2.

3. Experimental Results.

The experimental set up described above was used for a series of shearing creep experiments. We will describe the results for three separate bicrystals (identified as 1, 2, and 3). All three experiments were done at the same temperature: -3.5 ± 0.5 °C ($0.986 T_m$). The nominal applied shear stresses were 0.1 MPa or 0.4 MPa, applied alternatively during the tests. Creep curves for each sample are shown in Figure 3, 4, and 5. The evolution of the bicrystals during the creep tests was followed by sequential macrographs, as shown on the creep curves.

Figures 3 - 5 also show the orientations of the crystals and the position of the boundary in stereographic plots. The direction of the shear must be properly specified with respect to the orientations of the crystals. The point marked C_A is the c-axis of the crystal on the right side, which has the stress forcing it down. Samples 1 and 2 were cut from the hot die extrusion so that the shear stress acted perpendicular to the growing direction. For sample 3 the shear stress acted parallel to the growing direction.

The three bicrystals showed remarkably different behavior. Bicrystal 1 showed clear evidence of grain boundary sliding at 0.1 MPa. The boundary of Bicrystal 2 showed no sliding at 0.4 MPa, but became wavy and was eventually consumed by the growth of newly nucleated grains. Bicrystal 3

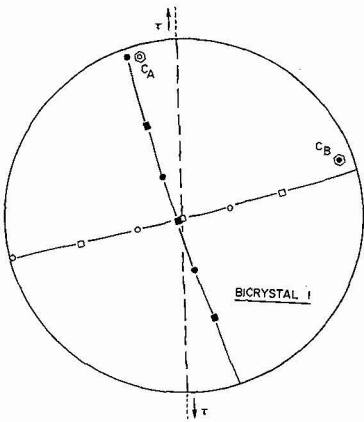


Figure 3. Bicrystal 1. Left: stereographic projection of the orientations of the crystals and the plane of the boundary. Right: creep curve with macrographs at different times. Black arrows show GB sliding; white arrows show GB migration.

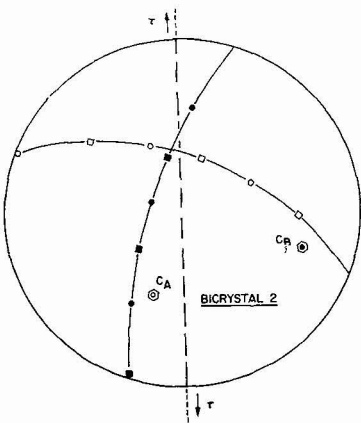
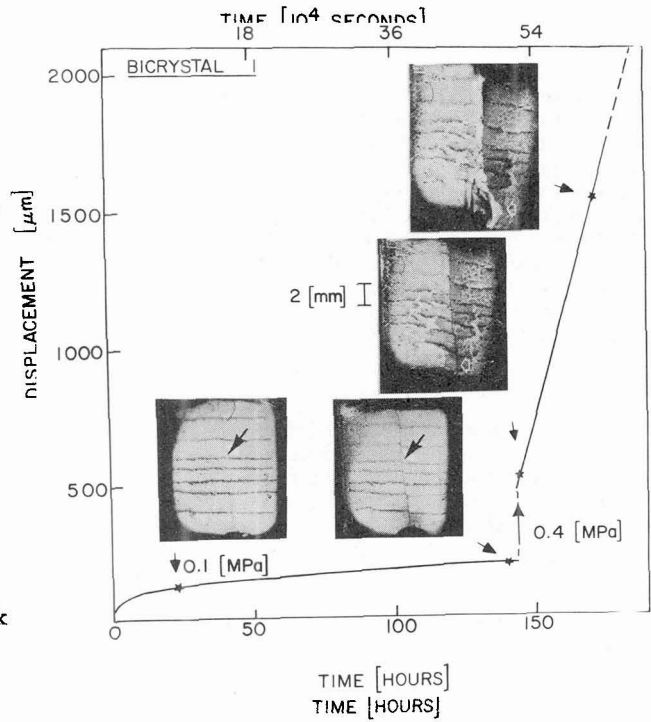
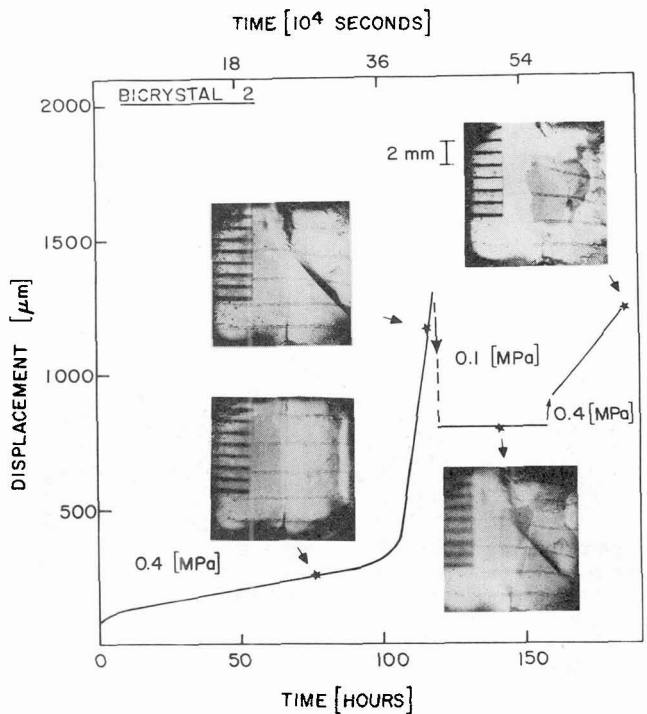


Figure 4. Bicrystal 2. Left: stereographic projection of the orientations of the crystals and the plane of the boundary. Right: creep curve with macrographs at different times.



developed a prominent intracrystalline slip band, which was eventually consumed by migration of the boundary.

i.) Bicrystal 1.

In Bicrystal 1 (Figure 3), the grain B has the basal planes slightly rotated with respect to the GB plane. In grain A, one family of prismatic planes are perpendicular to the GB plane, their intersection being the direction of the shear stress. At the initial stress of 0.1 MPa, the creep curve showed a standard primary stage, followed by a constant displacement rate of 0.5 $\mu\text{m/hr}$. The macrograph at 145 hours shows that a displacement of $200 \pm 30 \mu\text{m}$ was concentrated at the grain boundary. This displacement of the scratches agreed with the measured displacement of the grips. After 145 hours, the stress was increased to 0.4 MPa, and during the next 39 hours the displacement rate was 45 $\mu\text{m/hr}$. At this stress, the scratches became curved, as evidence of intracrystalline deformation near the boundary. The relative displacement by sliding at the GB did not appear to increase at the higher stress. But 24 hours later, evidence appeared in the lower part of the sample of grain boundary migration. The experiment was stopped at a total displacement of 2.1 mm.

ii) Bicrystal 2.

The orientation of Bicrystal 2 (Figure 4) is such neither crystal presents any particular position with respect to the GB plane, or the direction of applied shear stress. At the initial shear stress of 0.4 MPa, the creep curve showed a normal primary stage, followed by a constant displacement rate of 17.5 $\mu\text{m/hr}$. Initially, the macrographs showed no relative displacement between the crystals, even after 50 hours, and no noticeable intracrystalline deformation. After about 70 hours, steps began to form at the boundary. After a few more hours, the steps at the boundary became prominent, and slip lines appeared, particularly in one crystal. The continuity of the scratch lines showed that no GB sliding was active. After 90 hours of creep, the displacement rate increased, reaching 67.3 $\mu\text{m/hr}$. When the displacement reached 1.3 mm, the shear stress was lowered to 0.1 MPa. After some anelastic relaxation, no deformation was recorded during the next 38 hours. The slip lines gradually disappeared, and some new grains appeared in the boundary region. Finally the stress was increased again to 0.4 MPa. This produced a constant displacement rate of 13 $\mu\text{m/hr}$, and continued growth of the new grains. The experiment was stopped when the displacement once again reached 1.3 mm.

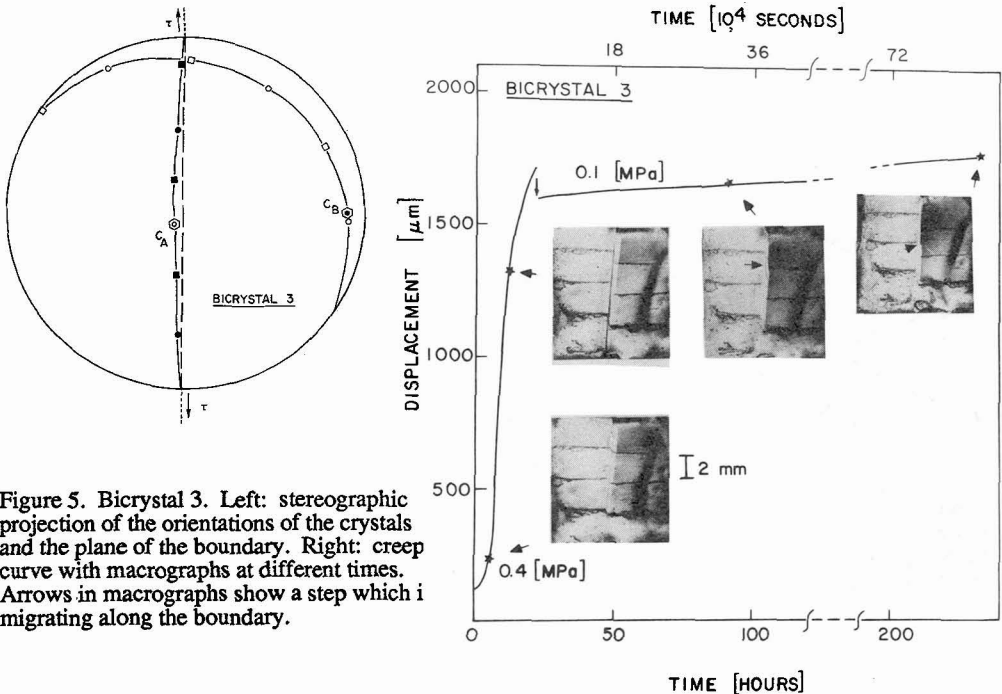


Figure 5. Bicrystal 3. Left: stereographic projection of the orientations of the crystals and the plane of the boundary. Right: creep curve with macrographs at different times. Arrows in macrographs show a step which is migrating along the boundary.

iii.) Bicrystal 3

In Bicrystal 3 (Figure 5), basal planes of grain B are very close to the GB plane, and the direction of shear stress is almost parallel to the common prismatic direction of both grains. Basal planes of grain A are then perpendicular to the GB plane.

The initial shear stress was 0.4 MPa. Contrary to the other samples, the creep curve presented a sigmoidal shape with an inverted primary and an intermediate period of constant rate of 150 $\mu\text{m/hr}$. The macrographs show that this high displacement rate resulted from a shear band in grain B, parallel to the GB. It was therefore difficult to observe whether there was any relative displacement of the scratches across the grain boundary. At a displacement of 1.7 mm, the stress was lowered to 0.1 MPa. The displacement rate decreased dramatically and remained constant at 0.5 $\mu\text{m/hr}$ during the following 300 hours. Evidence of GB migration appeared as a ledge which moved down from the top of the boundary. As the GB moved into the highly deformed region, the relative displacement between scratches continued to increase. It is possible, but not conclusively demonstrated, that this displacement included some GB sliding during the migration. The experiment was stopped at a total displacement of 1.7 mm.

4. Discussion

Grain boundary sliding has been assumed to operate as an important deformation process in polycrystalline ice. For example, Sinha [4] incorporated the process into his mathematical model for creep deformation of ice. There has, however, been very little direct observation of boundary sliding in ice. Our purpose here is to measure sliding quantitatively, in a controlled geometry that allows a correlation of the sliding behavior and the crystallography of the boundary. Our major preliminary result is that grain boundary sliding is sometimes observable, but that it does not always occur. This implies that the sliding depends on the relative orientations of the crystals.

Much of the difficulty in studying grain boundary sliding is that it may be masked by intracrystalline deformation. Deformation of ice single crystals is known to occur primarily by dislocation glide on the basal planes. This means that for those bicrystals with the basal planes of one crystal parallel to the boundary plane, an applied shear stress on the boundary plane will also be a resolved shear stress on the crystal slip system. For our experiments at the stress level 0.4 MPa, the basal glide is significant. For bicrystal 3, which has the most favorably oriented basal planes, the shear band next to the boundary completely overwhelms any possible observation of grain boundary sliding. For Bicrystal 1, with a basal plane at 18° to the boundary plane, basal glide does not overwhelm the observation of sliding, at least at the applied stress of 0.1 MPa. We conclude that sliding can be observed for a reasonably wide enough range of crystal orientations, without being masked by intracrystalline slip.

Our preliminary conclusion that sliding depends on crystallography depends on the fact that we did not observe measurable sliding in Bicrystal 2, which is oriented such that basal glide should not be strongly activated in either crystal. We might generally expect that the sliding rate would increase with increasing stress, but Bicrystal 2 at 0.4 MPa slides less than Bicrystal 1 at 0.1 MPa. It is, of course, possible that the difference between Bicrystals 1 and 2 results from the difference in the initial stress levels. This explanation requires a locking mechanism to suppress the sliding, which might be provided by intracrystalline dislocation activity. Such locking definitely occurs at a later stage, after the displacement rate accelerates: after the development of intracrystalline slip bands, the boundary becomes jagged or faceted where the slip bands intersect the boundary. (Figure 6) This would greatly reduce the sliding rate. [5] Similar behavior was demonstrated by Lim and Raj [6] in certain Ni bicrystals after cyclic deformation, when there was an incompatibility between the slip systems of the two

crystals. This mechanism, however, does not appear to operate during the first 70 hours, when the displacement rate was low, and the boundary remained flat on the scale of our observations.

Although we appear to find an effect of crystallographic orientation on sliding, we cannot yet say whether this results from differences in the intrinsic atomic structure at the boundary, or from differences

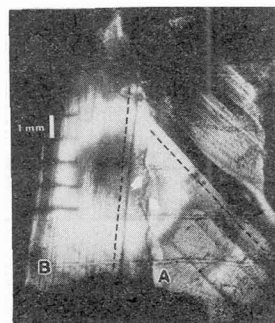


Figure 6. Slip bands in Bicrystal 2 at 120 hours, photographed with oblique lighting. Arrows show jagged zones at the boundary.

in the interactions of the boundary with the intracrystalline deformation. In metals there have been several studies which relate the sliding and migration of boundaries to the atomic structure. These studies rely on the coincidence site lattice (CSL) model for describing boundary structure. [7] For example, in hexagonal metals, Bell et al. [8] and Watanabe et al. [9] and found that GB sliding is more difficult to activate in boundaries with exact or nearly exact coincidence orientations. On the other hand, Aust and Rutter [10] found that migration is much faster for coincidence orientations. The coupling of both mechanisms has recently been observed for coincidence boundaries in Zn [11], Al [12], and NaCl [13] in a manner predicted by CSL theory. We expect similar behavior in ice.

In ice, Hondoh and Higashi [14] studied G. B. migration for large angle $\langle 1010 \rangle$ symmetric tilt boundaries. They found a strong anisotropy of migration depending on the orientation of the boundary plane with respect to the tilt axis. Facets corresponding to high density CSL planes were observed, which impeded the migration in a direction perpendicular to them. Recently Hondoh [15,16] has proposed a mechanism for GB sliding in these tilt boundaries, based on GB dislocations which move on the facets parallel to the high density planes.

The boundaries used in this study were not high coincidence boundaries, but were chosen at random. A detailed analysis in terms of the CSL framework is therefore beyond the scope of this paper, particularly for Bicrystal 2. Future work will use the present testing techniques on bicrystals grown with specified orientations, chosen to allow simple modelling.

Acknowledgements

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COMMENTS

L.LLIBOUTRY

Your bicrystal 2 showed an important reverse strain on partial unloading, and the others not. In my opinion, this reversible strain is linked with the very local stresses and bundles of dislocations due to the incompatibility of the strains for two neighbouring grains.

(In a recent paper published in Tectonophysics, the adjustment of incompatible strains by boundary migration is theoretically demonstrated).
Have you comments on this reversible strain ?

Answer :

The reverse anelastic strain was observed in bicrystal 2 (and to a smaller extent in bicrystal 3) when the stress was lowered from 0.4 to 0.1 MPa. We expect that this results from the relaxation of dislocation pile-ups at the boundary.

T. HONDOH

Grain boundary sliding is impeded by its non-planar shape, which critically depends on the misorientation between two crystals. Did not you find out a correlation between GB shapes and the sliding rates ?

Answer :

Our experiments clearly showed that the mutual orientation is an important factor for grain boundary sliding. We expect that the shape of the boundary (that is, the degree of deviation from planarity) is important, but we cannot yet report on the correlation.