JOURNAL OF MATERIALS SCIENCE 31 (1996) 2373–2378

Observation of slip transmission through a grain boundary in ice

K. JIA, I. BAKER, F. LIU
Thayer School of Engineering, Dartmouth College, Hanover, NH 03755, USA
M. DUDLEY
Department of Materials Science and Engineering, State University of New York at Stony Brook, Stony Brook, NY 11794, USA

In-situ X-ray topography has been used to provide the first clear demonstration of slip transmission through a grain boundary in hexagonal ice. Although the ice was deformed under creep conditions, the relative orientation of the grains and the orientation of the grain boundary with respect to the loading direction discouraged grain boundary sliding and, thus, suppressed dislocation nucleation at the grain boundary. This particular geometrical arrangement is rarely encountered, suggesting that slip transmission through grain boundaries in ice is a rare event.

1. Introduction

Since Ahmad *et al.* [1] first demonstrated the utility of synchrotron X-ray topography for dynamic studies of dislocation motion in single crystals of bubble-free, high-purity hexagonal ice (I_h), there have been a number of papers presenting dynamic observations of dislocation/grain boundary (GB) interactions in polycrystalline ice [2–4]. In these latter studies, dislocations have been observed to pile up at GBs and dislocation nucleation has been observed at GBs. However, dislocation pile-ups have not been observed to lead to either dislocation nucleation or slip transmission in the adjacent grain. Instead, dislocations were found to nucleate at GB facets at which stress concentrations arose, probably from GB sliding.

Liu *et al.* [4] suggested that whether slip is transmitted through a GB in ice will depend more on the sense of the local stress concentrations than on having a favourably oriented external stress, and that dislocation transmission through a GB is more likely to occur when the misorientation between the two grains is selected such that basal slip plane in each grain would fulfill the plastic compatibility conditions at the GB, i.e. the (0001) planes are nearly parallel. Since this is a very special geometrical condition, a general GB in ice will act as a strong obstacle to dislocation motion through plastic incompatibility. Liu *et al.* [4] further suggested that the rôle of the misorientation between two grains is to enhance dislocation generation at GBs through elastic incompatibility.

This paper presents X-ray topographic observations of ice deformed under creep conditions in which dislocation pile-ups at a GB in one grain led to slip transmission into the adjacent grain.

2. Experimental details

The growth of columnar-grained ice with a low initial dislocation density and the subsequent specimen preparation for X-ray topographic observation in the Ice Research Laboratory, Dartmouth College have been described in detail elsewhere [2–4]. The specimen used in the present study contained four grains, see Fig. 1. Only the two grains labelled G_I and G_{II}, were studied using X-ray topography. The orientations of G_I, G_{II}, the GB normal, $\mathbf{N}_{GB}$, and the loading direction, **F** are shown on a stereogram, see Fig. 2. The basal (slip) plane normals of G_I and G_{II} made angles of 25° and 56° with **F** whilst $\mathbf{N}_{GB}$ was almost perpendicular (88°) to **F**. The latter meant that there was little reason for GB sliding to occur between these two grains.

In-situ deformation studies were performed using a specially-built compression jig assembled with a cryostat [5] at the National Synchrotron Light Source at Brookhaven National Laboratory, Long Island, NY. The desired loads (up to 25 N, corresponding to a stress of 3.5 MPa) and load durations were chosen based on the dislocation mobility data of Shearwood and Whitworth [6]. The temperatures used were in the range -12 to -40°C. Topographs (Laue patterns) were produced by allowing a highly collimated beam of white X-rays to fall on the specimen; details are given elsewhere [7]. Several diffraction spots were recorded simultaneously on a single X-ray film with exposure times of ~ 2–8 s.

3. Results

Fig. 3a shows the central portion of the GB at -40 °C after cooling from -10 °C at a rate of $10\,°\mathrm{C\,h^{-1}}$. This cooling rate was used in order to determine if point defect condensation would occur. As expected, no

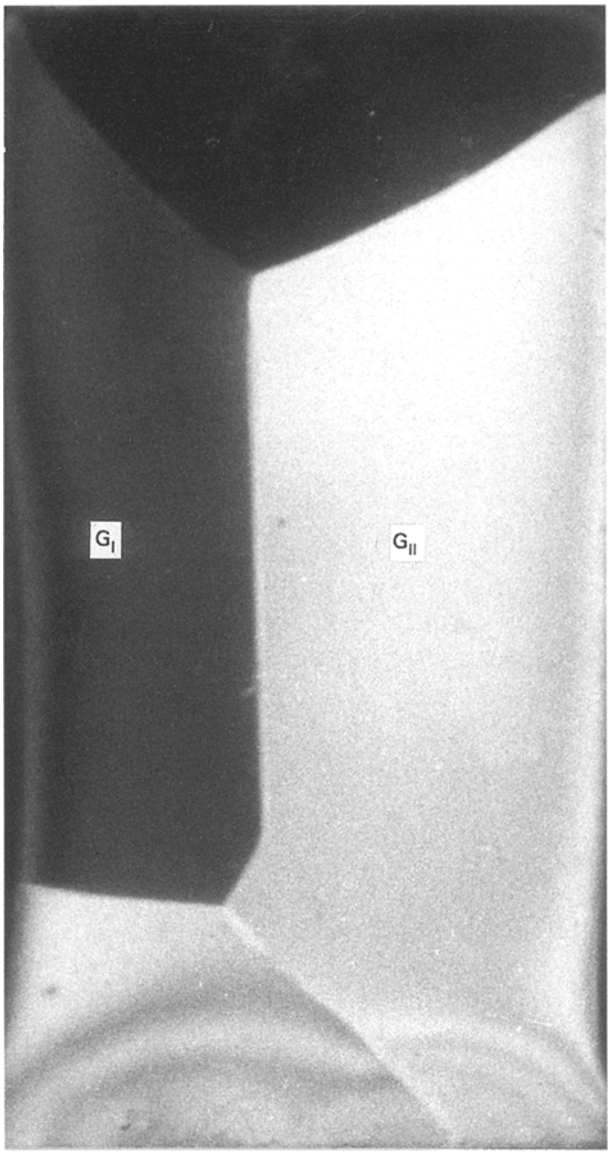

Figure 1 An optical micrograph of the ice specimen viewed through cross polarizers. G_I and G_{II} are the two grains on which X-ray topographic observations are based.

point defect condensation was observed, consistent with the observations of Oguro and Hondoh [8]. The two diffraction vectors, $(10\bar{1}0)_I$ and $(11\bar{2}0)_{II}$, used and their relationships to G_I, G_{II}, $\mathbf{N}_{GB}$ and **F** are indicated on the stereogram, Fig. 2. Note that for the diffraction conditions used, the basal planes in G_{II} are viewed close to edge-on so that the basal dislocations appear as nearly straight lines parallel to the trace of the basal plane. Several features of this initial structure are worth noting. First, there are only a few dislocations at the GB prior to straining. Second, most of the dislocations appear to be lying on the basal plane. Third, at the resolution available ($\sim 10\,\mu$m) there are no facets on the GB. Fourth, the fine Pendellösung fringes at the GB indicate the high degree of perfection of both the nearby lattice and the GB. Fifth, there are a number of non-basal dislocations (arrowed).

The specimen was then loaded for 70 min at 0.21 MPa and for a further 52 min at 0.4 MPa, Fig. 3b. This produced darkening along the GB due to the internal stresses which arise because of the difference in elastic modulus along particular directions in the two grains. During loading, the dislocations in G_{II} moved very slowly and no dislocations were generated from the GB. Since dislocation mobilities are very low at $-40\,°$C, further experiments were performed at higher temperature.

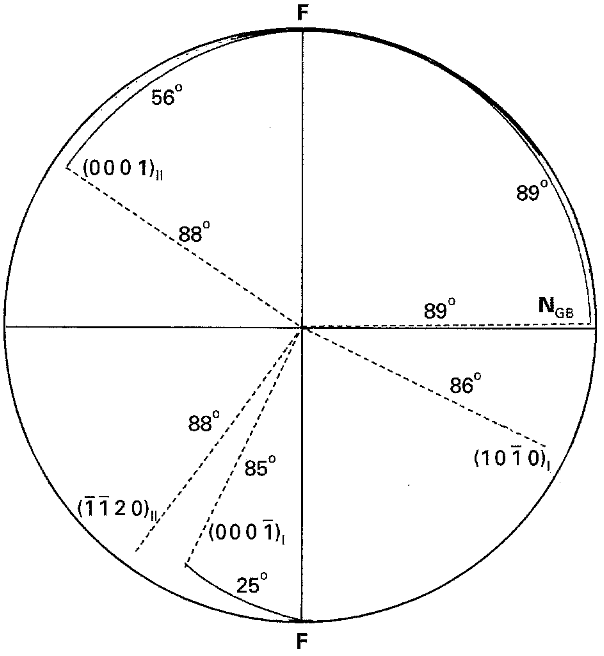

Figure 2 Stereogram showing the orientations of the grains, G_I and G_{II}, the GB normal, N_{GB}, and the loading direction, **F**. The two diffraction vectors, $(10\bar{1}0)_I$ and $(11\bar{2}0)_{II}$, used are also indicated.

The specimen was allowed to warm up slowly to $-12\,°$C where it was maintained for 10 h after which the stresses at the GB had been relaxed, as is evident by the disappearance of the darkening along the GB, see Fig. 4a. The subsequent topographs, Fig. 4b–f, were taken at increasingly higher loads applied to the specimen. Even the lowest applied load (0.16 MPa) caused the darkening at the GBs, indicative of internal stresses, to reappear, Fig. 4b. As the load was increased the extent of the darkened area increased, indicating the stress building up there. During initial loading there was some movement of dislocations in G_I, Fig. 4b. However, in G_{II} there was much more significant movement of the dislocations towards the GB, and they can be clearly seen to intersect the GB. (The straight basal dislocations, which appear as dark lines parallel to the trace of the basal plane, move relatively slowly whereas the non-basal dislocations move much more rapidly.) With further increases in load, the darkening along the GB increased indicating greater internal stress build-ups, Fig. 4c. At higher loads, some dislocations in G_I can be observed gliding towards the GB, Fig. 4d. Eventually, at even higher loads new dislocations were nucleated at the GB and moved into G_I, Fig. 4e and f (note that no image of G_{II} is available for Fig. 4e). The velocities of the straight and the curved dislocation segments in G_I were determined from the topographs and the resolved shear stress calculated (from the far-field stress) for slip both on the basal plane and on a non-basal plane. Comparing these results with the mobility determinations of

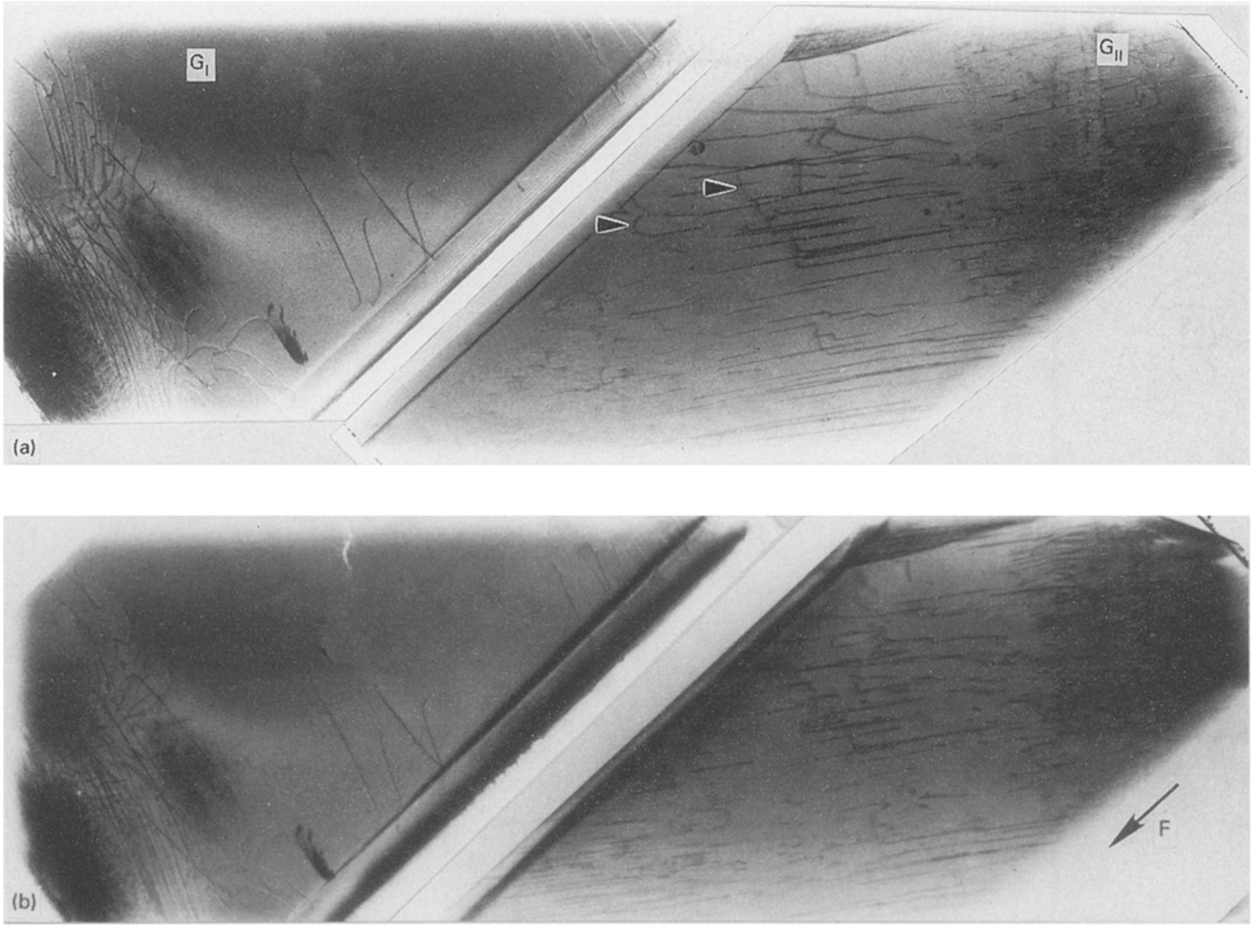

Figure 3 Topographs at $-40\,^{\circ}\mathrm{C}$ showing $(10\bar{1}0)_{\mathrm{I}}$ images of $\mathrm{G_I}$ ($\lambda_{\mathrm{I}} = 0.076$ nm) and $(11\bar{2}0)_{\mathrm{II}}$ images of $\mathrm{G_{II}}$ ($\lambda_{\mathrm{I}} = 0.076$ nm): (a) with no applied load after cooling from $-20\,^{\circ}\mathrm{C}$ at $10\,^{\circ}\mathrm{C\,h^{-1}}$; and (b) after loading at 0.21 MPa for 70 min and 0.4 MPa for 52 min.

Shearwood and Whitworth [6] indicated that the straight segments were basal dislocations and the curved segments were non-basal dislocations, as indicated earlier.

Fig. 5 shows the specimen after unloading and annealing for 1 h at $-12\,^{\circ}\mathrm{C}$. Two features are evident. First, further dislocation movement has occurred, and second, this has led to the internal stresses on the GB being reduced as indicated by the reduction in darkening at the GB.

4. Discussion

There have been numerous dynamic transmission electron microscope (TEM) observations of slip transmission in metals. The very thinness of TEM specimens raises questions about the applicability of some of these observations for bulk materials. The technique of *in-situ* straining X-ray topography has the advantage that much thicker (2 mm in the present case) specimens can be used. Thus, the behaviour observed here, using X-ray topography, is probably typical of bulk material, [7, 9].

The present results clearly showed that slip transmission occurred through a GB in ice, i.e. dislocation impingement on one side of a GB led to dislocations being emitted from the other side of the GB. Due to the low resolution of X-ray topography, it was not possible to distinguish between dislocation transmission through the GB and nucleation of dislocations at the GB, arising from the stress concentrating effect of the impinging dislocations. The particular GB studied was rather special for several reasons. First, the external loading favoured basal slip in both grains. Second, the GB orientation was such that GB sliding would not be expected. Third, there were no observable facets on the GB. And, fourth, the angle between $(0001)_{\mathrm{I}}$ and $(0001)_{\mathrm{II}}$ on the GB was small (11°). In other words, the specimen was set up so that whilst basal slip was favoured in both grains, dislocation nucleation at GB facets due to stresses built up from GB sliding, as previously observed [2–4], was eliminated. In addition, the small angle between the basal slip planes would favour slip transmission from one grain to the next. Thus, slip transmission occurred in a rather special situation. That this is a special situation is corroborated by the observations made by the authors that in experiments on over 25 boundaries in the same temperature and stress ranges a slip transmission event was only observed once.

Finally, it is worth noting that the only previous X-ray topographic observations of GB slip transmission have been in the cubic semiconductors Si and Ge [10–12].

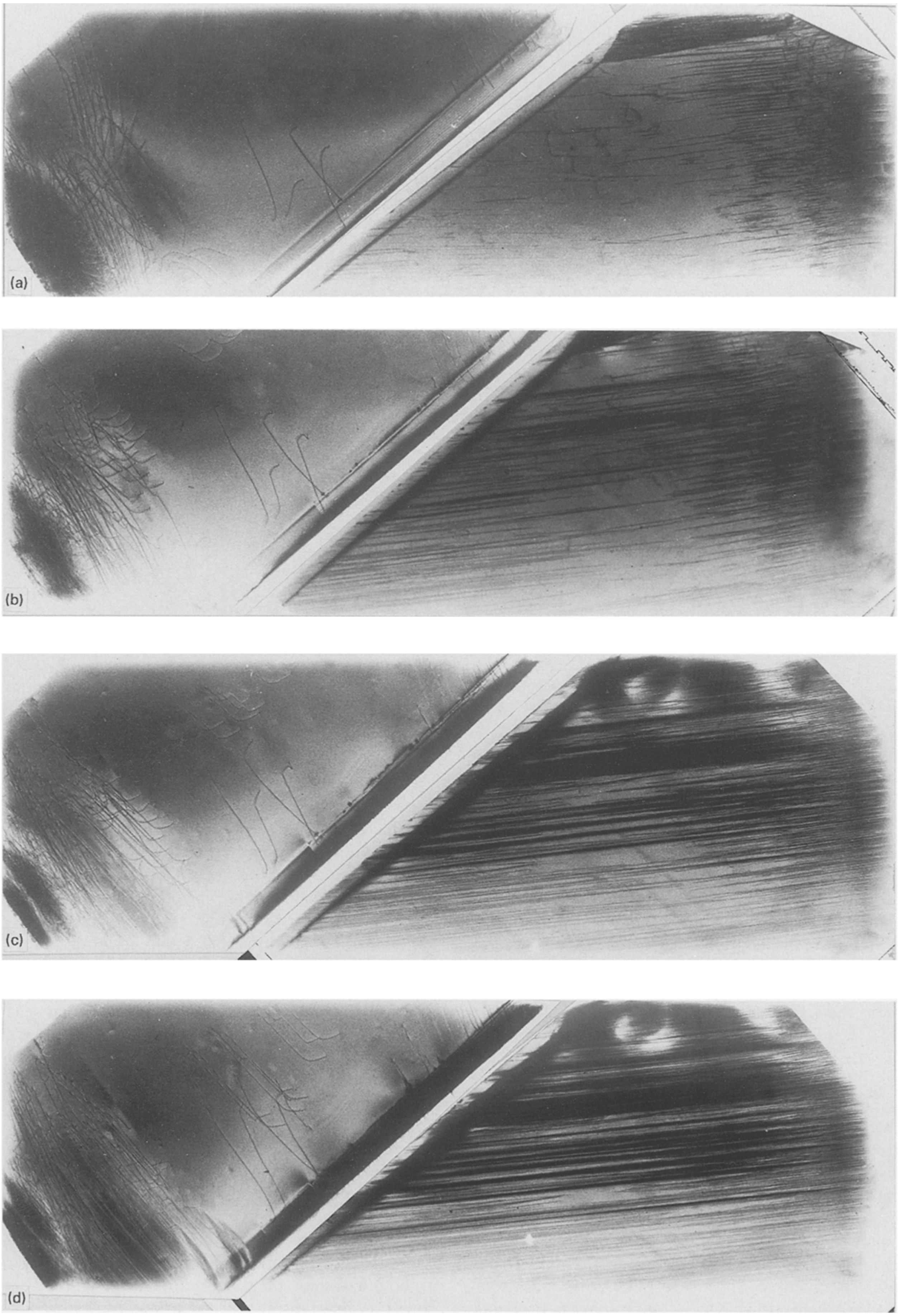

Figure 4 Topographs at $-12\,^{\circ}$C showing $(10\bar{1}0)_{\rm I}$ images of $G_{\rm I}$ ($\lambda_{\rm I} = 0.076$ nm) and $(11\bar{2}0)_{\rm II}$ images of $G_{\rm II}$ ($\lambda_{\rm I} = 0.076$ nm): (a) with no applied load after holding for 10 h; after loading at (b) 0.16 MPa for 38 min, (c) 0.23 MPa for 10 min and 0.31 MPa for 5 min, (d) 0.35 MPa for 10 min, (e) 0.29 MPa for 30 min, and (f) 0.33 MPa for 30 min.

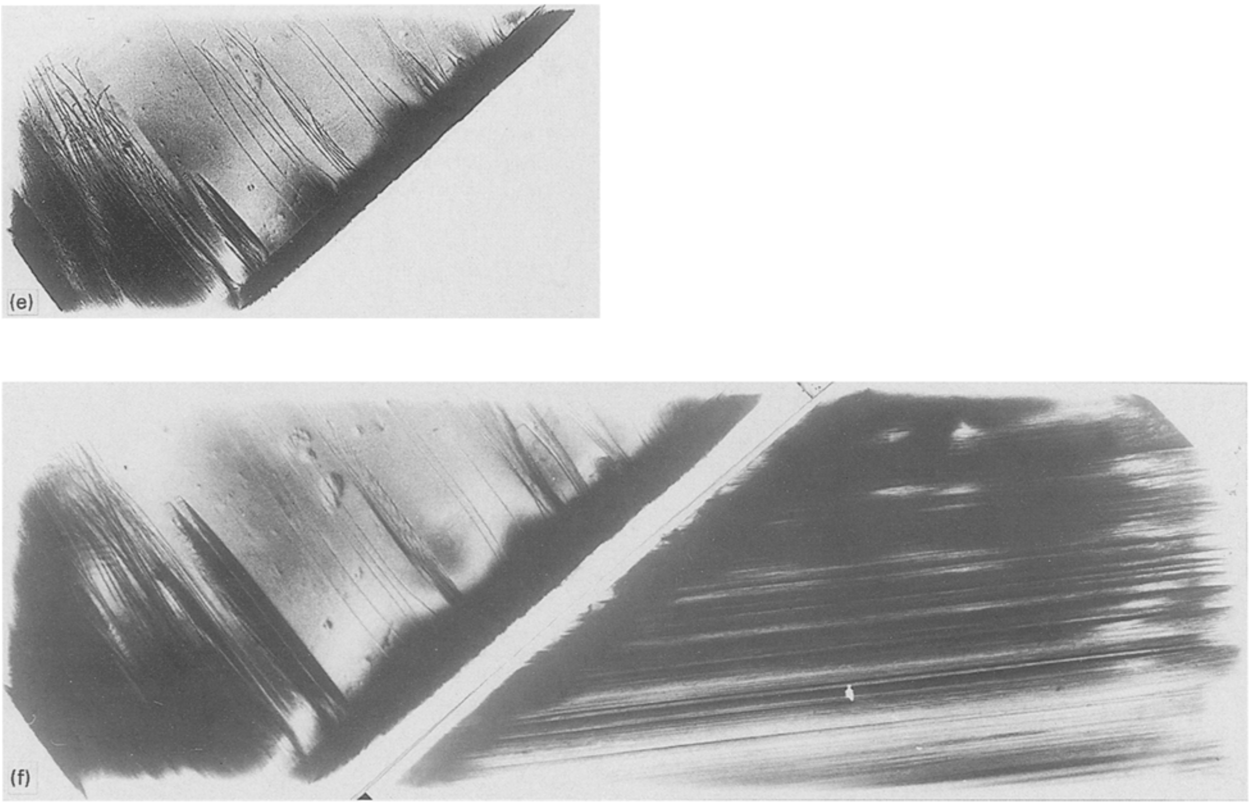

Figure 4 Continued

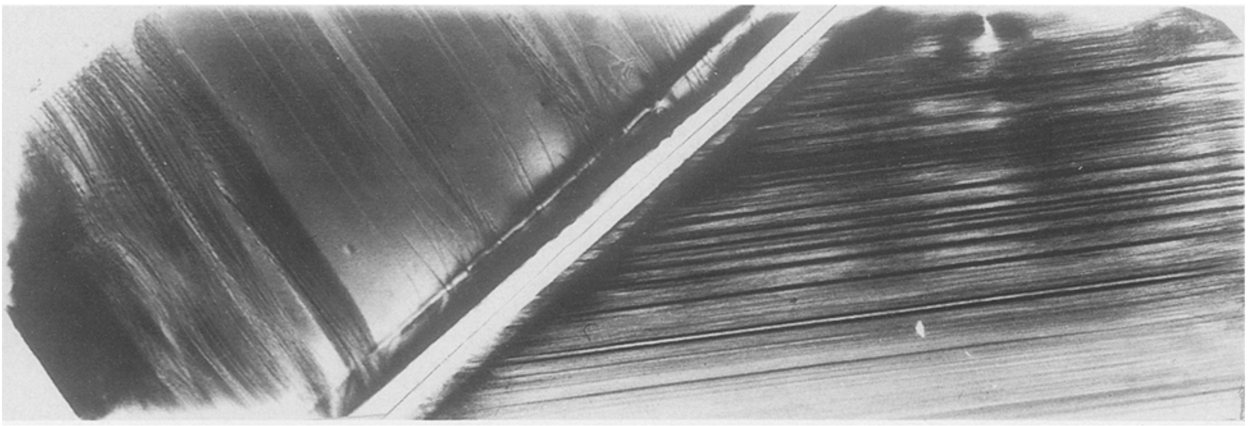

Figure 5 Topograph at $-12°C$ showing a $(10\bar{1}0)_I$ image of G_I ($\lambda_I = 0.076$ nm) and a $(11\bar{2}0)_{II}$ image of G_{II} ($\lambda_I = 0.076$ nm) after holding at $= 12\,°C$ for 1 h.

5. Summary

Dynamic loading *in-situ* X-ray topography under creep conditions has been used to demonstrate, for the first time, the occurrence of slip transmission through a GB in hexagonal ice. The unusual geometrical arrangement used was unfavourable for grain boundary sliding so that dislocation nucleation at grain boundary facets was not favoured. The particular geometrical arrangement used would rarely be encountered, which means that slip transmission through GBs in ice is unlikely to be a common event.

Acknowledgements

This work was supported by the Army Research Office through grant DAAH04-93-G-0061, by the National Science Foundation, through grant DPP-9218336, and in part by the donors of the Petroleum Research Fund, which is administered by the American Chemical Society (MD). Professor E. M. Schulson is acknowledged for the use of the Ice Research Laboratory at Dartmouth College. Synchrotron X-ray topography was carried out at the Stony Brook Topography Station (beamline X-19C) at the NSLS which is supported by DOE through grant DE-F902-84ER-45098.

References

1. S. AHMAD, M. OHTOMO and R. W. WHITWORTH, *Nature* **319** (1986) 659.
2. F. LIU, I. BAKER, G. YAO and M. DUDLEY, *Phil. Mag. Lett.* **65** (1992) 279.

3. F. LIU, I. BAKER and M. DUDLEY, *Phil. Mag. A* **67** (1993) 1261.
4. *Idem, Phil. Mag.* **71** (1995) 15.
5. F. LIU and I. BAKER, *Meas. Sci. Technol.* **4** (1993) 416.
6. C. SHEARWOOD and R. W. WHITWORTH, *Phil. Mag. A* **64** (1991) 289.
7. F. LIU and I. BAKER, *Microsc. Soc. Amer. Bull.* **24** (1994) 351.
8. M. OGURO and T. HONDOH, in "Lattice Defects in Ice Crystals", edited by A. Higashi (Hokkaido University Press, Sapporo, Japan, 1988) p. 54.
9. I. BAKER and F. LIU, in Proceedings of the Materials Research Society, 1994, Vol. **319**, (Materials Research Society) p. 203.
10. X. BAILLIN, J. PELISSIER, J. J. BACMANN, A. JACQUES and A. GEORGE, *Phil. Mag. A* **55** (1987) 143.
11. A. JACQUES, H. A. BENHOMA, A. GEORGE and X. BAILLIN, in Proceedings of the 6th International Conference on Intergranular and Interphase Boundaries, Material Science Forum, 1993, Vol. 126–128, p. 333.
12. H. M. MICHAUD, X. BAILLIN, A. JACQUES, H. A. BENHOMA and A. GEORGE, in Proceedings of the 6th International Conference on Intergranular and Interphase Boundaries, Material Science Forum, 1993, Vol. 126–128, p. 491.

Received 28 March
and accepted 17 October 1995