Journal of Glaciology, Vol. 9, No. 57, 1970

INSTRUMENTS AND METHODS

A TECHNIQUE FOR PRODUCING STRAIN-FREE FLAT SURFACES ON SINGLE CRYSTALS OF ICE

By T. M. Tobin *and* K. Itagaki

(U.S. Army Cold Regions Research and Engineering Laboratory, Hanover, New Hampshire 03755, U.S.A.)

Abstract. The top surface of an accurately aligned ice crystal is melted by an aluminum surface and then frozen to a warm "Lucite" plate and tapped free. Etch-pit development shows that the dislocation density on the resulting surface is similar to the bulk dislocation density determined by X-ray topographic methods.

Résumé. *Une technique pour préparer des surfaces planes libres de tensions sur un monocristal de glace.* La surface supérieure d'un cristal de glace soigneusement aplanie est fondue par une surface d'aluminium puis fixée par regel à une assiette chauffable "Lucite" et libérée par tapotements. Le développement superficiel de figures gravées montre que la fréquence des dislocations du réseau cristallin sur la surface résultant de ce traitement est semblable à la fréquence des dislocations dans la masse que l'on peut déterminer par une étude de localisation aux rayons X.

Zusammenfassung. *Eine Technik zur Herstellung spannungsfreier, ebener Oberflächen auf Einkristallen von Eis.* Die Deckfläche eines genau ausgerichteten Eiskristalls wird mit einer Aluminiumfläche angeschmolzen, dann auf eine warme "Lucite"-Platte aufgefroren und freigeklopft. Die Entwicklung von Ätz-Löchern zeigt, dass die Dichte der Versetzungen auf der entstandenen Oberfläche ähnlich der Gesamtdichte ist, die sich mit Röntgentopographie bestimmen lässt.

Introduction

During the course of surface self-diffusion studies of ice by scratch-decay techniques, results of which will be published later, it was found necessary to develop a method of producing strain-free flat surfaces. The strain-free condition must be satisfied, otherwise additional mass transfer due to straining was expected. (Itagaki (1967) observed some peculiar phenomena such as mass transfer from positive curvature surfaces to flat surfaces which was prohibited if one assumes that the mass transfer is driven by the vapor pressure difference as depicted by Thompson's formula. The probable explanation of this phenomenon was the vapor pressure difference caused by the high dislocation density on the surface, since the ice samples were prepared by microtoming and sanding.) Flat and smooth surfaces are another essential requirement to produce finely spaced sinusoidal grooves with symmetric profiles (Maiya and Blakely, 1965) on the sample surface.

The "freeze-tap method", described in this paper, was originally developed and studied to some extent by one of the present authors (K.I.) in 1967. The freeze-tap method was found to be suitable for surface self-diffusion studies of ice by laser interference microscopy. With slight modification this method is applicable in a wide range of ice surface studies.

Experimental procedure

The freeze-tap method involves four separate steps, which are applied after the ice samples (about 5 cm × 3 cm × 1 cm) have been cut with a band saw to the desired crystallographic orientation and frozen to a microscope slide.

First, the ice sample G is oriented parallel to a "Lucite" (polymethylmethacrylate) plate, D (see Fig. 1). This may be done either by a level or an optical method. In either case the ice sample is placed on the movable platform A of the jack, B. Next, the "Lucite" plate (20 cm × 5 cm × 1.3 cm) is placed on the fixed supports C. If a level is used, then one merely adjusts the ice sample by means of the rotatable head E, which is attached to the movable platform, until the bubble readings on the level match those of the "Lucite" plate. If instead the optical method is used, then a microscope slide H must be placed on top of the ice sample. Two reflections are observed through the illuminated telescope eyepiece F, one from the "Lucite" plate and the other from the microscope slide. By adjusting the movable platform so that the two reflections coincide, the ice sample is correctly oriented parallel to the "Lucite" plate.

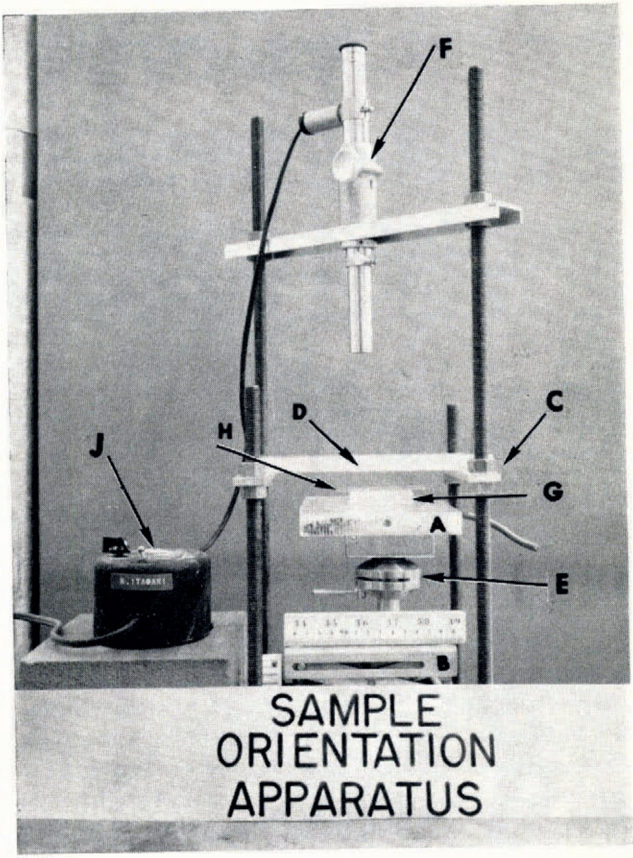

Fig. 1. Step one: ice sample orientation. A—moveable platform. B—laboratory jack. C—fixed supports. D—"Lucite" plate. E—rotatable head. F—telescope. G—ice sample. H—microscope slide. J—transformer.

Second, the top layer of the ice sample is melted by replacing the "Lucite" plate by a warm aluminum heat slab, I (temperature $\approx 30°C$), on the fixed supports (see Fig. 2). The jack is then raised until the ice sample comes in contact with the warm aluminum heat slab. This slab melts the top layer (≈ 1 mm) of the ice sample in a few seconds. The ice sample is immediately lowered so that the slab and the sample are no longer in contact.

Third, the aluminum heat slab is immediately replaced by a warm "Lucite" plate. The ice sample is raised until it makes contact with the "Lucite" plate (see Fig. 3). The sample orientation is then checked as in step one. To ensure that no air is trapped between the ice sample and the "Lucite" plate, the plate is moved slightly until any air bubbles that are trapped are displaced from the melted surface layer of the sample. The ice sample is then allowed to freeze to the "Lucite" plate. The freezing process normally takes 90 to 120 min (at $-10°C$).

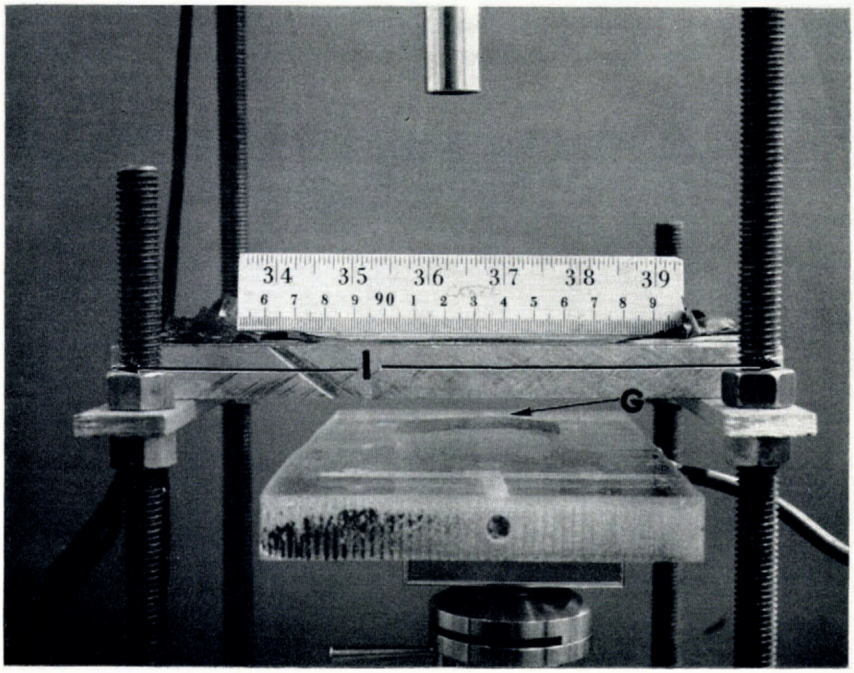

Fig. 2. Step two: melting top layer of the ice sample G with aluminum heat slab I.

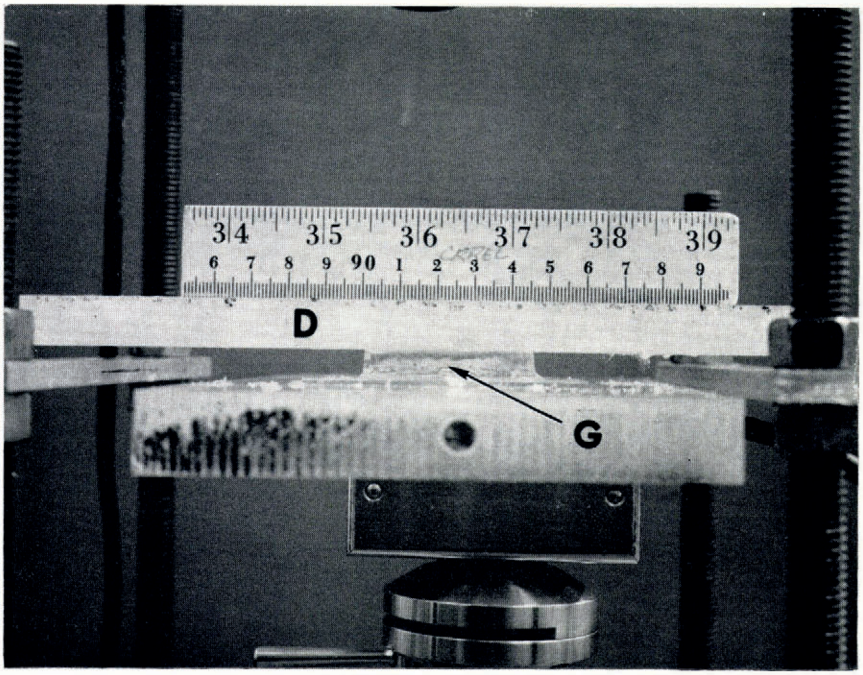

Fig. 3. Step three: freezing the ice sample G to the "Lucite" plate D.

https://doi.org/10.3189/S0022143000022905 Published online by Cambridge University Press

The fourth and last step (see Fig. 4) involves tapping the ice sample free from the "Lucite". This is accomplished by a sharp blow on the back of the "Lucite" plate over the center of the ice sample. (The rubber-covered handle of a medium-sized screw driver worked very well.)

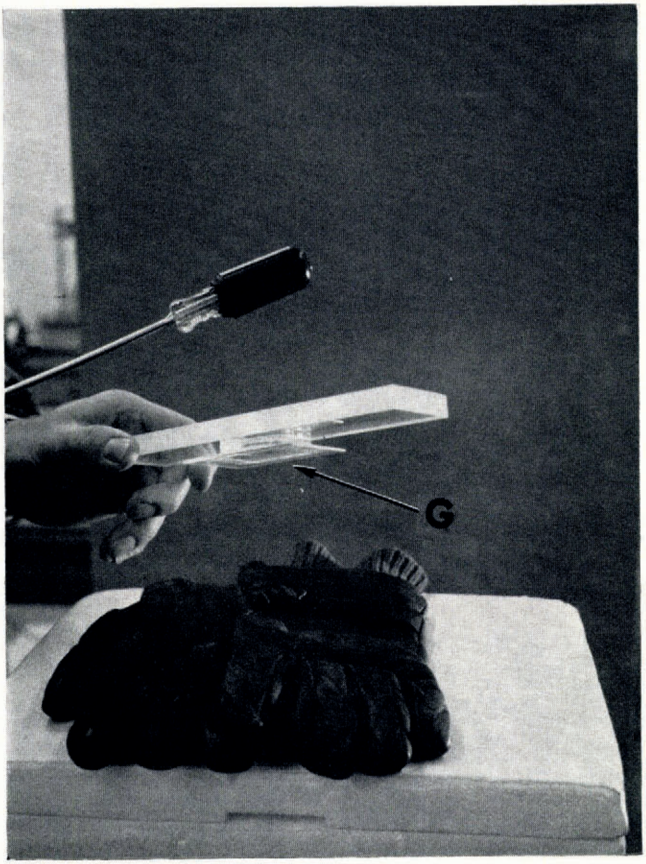

Fig. 4. Step four: tapping the ice sample G free from the "Lucite" plate.

Results and discussion

Immediately after successfully tapping the ice sample from the "Lucite" plate a microscopic interferogram (Ingelstam, 1960) was taken of the ice surface by a Zeiss interference microscope (see Fig. 5). From this interferogram it can be seen that the interference bands are even, straight, and parallel, which indicates a flat, smooth surface (Barakat, 1966; Torge, 1966). Grooves, scratches and etch pits on the surface of the ice sample would be observed by deflections of the interference bands produced by the interference microscope (Feuersanger, 1963). The etch-pit density, immediately after tapping, was about 10^3 etch pits/cm^2 which was lower than the bulk dislocation density obtained by X-ray topographic methods. The etch-pit density of the interferogram two hours after separation from the "Lucite" plate was about 5×10^4 etch pits/cm^2. This is apparently due to the insufficient development of the etch pits in the first photograph as the etch-pit density after two hours agrees reasonably with the bulk dislocation density. Since the bulk dislocation and surface etch-pit densities are about

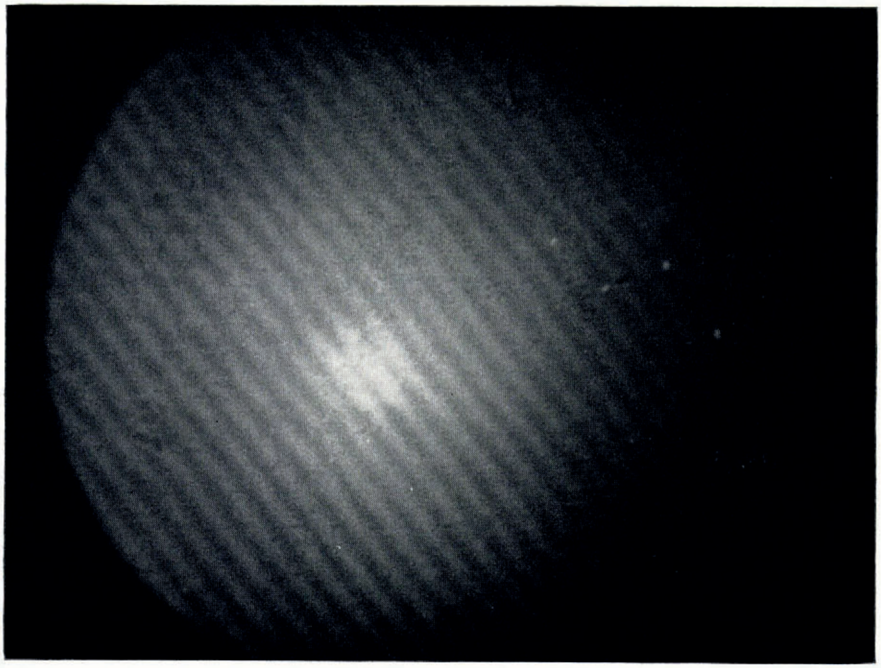

Fig. 5. Interferogram of ice surface immediately after tapping.

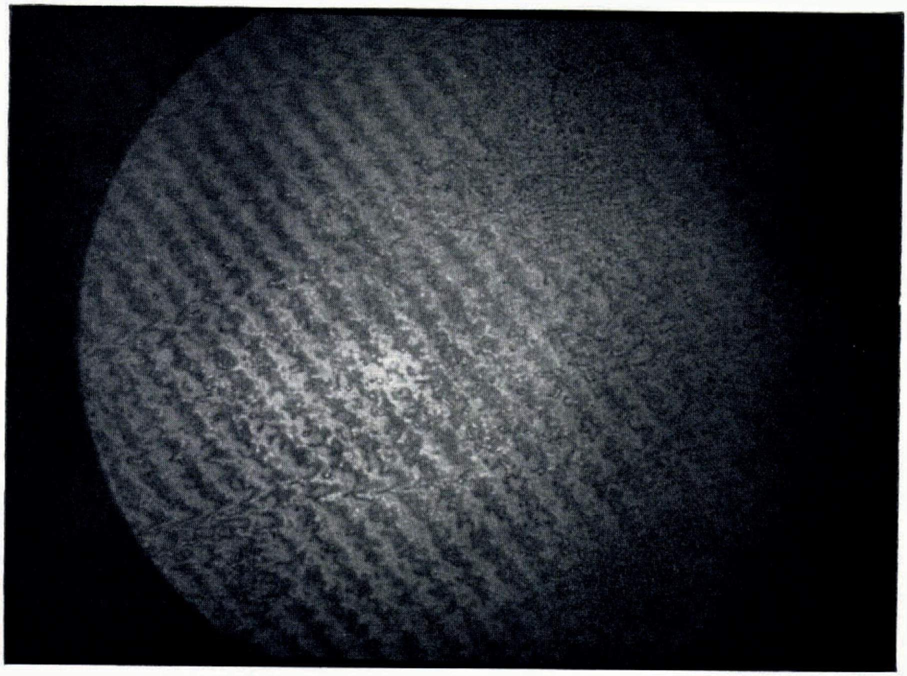

Fig. 6. Interferogram of ice surface two hours after tapping.

the same it can be inferred that no extra strains are introduced by the freeze-tap method and the resulting surface is strain-free. In Figure 6 taken two hours after the sample was tapped free the effect of preferential evaporation leading to the formation of etch pits and grooves along grain boundaries can be seen.

Obviously the most dangerous step of the method is the tapping. The chances of shattering the sample on tapping are considerably decreased by limiting the freezing process to the 90 to 120 min range (around −10°C) previously stated, and by using only one tap. The probability for successful tapping is about 75% provided the freezing process is between 90 and 120 min. For periods greater than this the probability decreases to about 50 to 25%. Presumably there is an increase in the adhesion of ice to this substrate with time. This ageing effect may occur because of molecular rearrangement or some other mechanism and should be taken into account in basic studies on ice adhesion.

ACKNOWLEDGEMENTS

The authors wish to express sincere thanks to Dr Charles Keeler, F. J. Sanger and S. F. Ackley for critically reviewing this paper.

MS. received 13 January 1970

REFERENCES

Barakat, N. 1961. Interferometric microscopical studies and measurements of growth features on crystal surfaces. *Zeiss-Mitteilungen*, Bd 2, p. 235–39.
Feuersanger, A. E. 1963. Interference microscopy of thin films and semiconductor structures. *Solid State Design*, Vol. 4, No. 10, p. 29–32.
Ingelstam, E. 1960. Problems related to the accurate interpretation of microinterferograms. (*In Interferometry: a symposium held at the National Physical Laboratory on 9th, 10th, 11th June 1959*. London, H.M.S.O., p. 137–63.)
Itagaki, K. 1967. Some surface phenomena of ice. *Journal of Colloid and Interface Science*, Vol. 25, No. 2, p. 218–27.
Maiya, P. S., *and* Blakely, J. M. 1965. A technique for study of mass transport mechanisms at crystal surfaces. *Applied Physics Letters*, Vol. 7, No. 3, p. 60–62.
Torge, R. 1966. The interference microscope. *Zeiss Information*, Vol. 15, No. 61, p. 100–03.