

## ICE PHYSICS

# A flexible and springy form of ice

Single-crystal ice microfibers recover their shape after bending to near their breaking limit

By **Erland M. Schulson**

**W**ater ice is ordinarily fragile and breaks if extended by just  $<0.1\%$  (1). On page 187 of this issue Xu *et al.* (2) show that fibers of cold ice a few micrometers or less in diameter can bend without breaking into a near-circular shape tens of micrometers in radius. Upon unloading, the fibers spring back to their original shape. Such strains are near the theoretical limit of  $\sim 15\%$  (3), so the deformation is completely elastic. The microfibers can transmit visible light as effectively as state-of-the-art on-chip light guides (4, 5). The authors also find that extreme bending creates a near-surface layer on the compressive side that transforms relatively quickly from ice  $I_h$  (hexagonal crystal structure) to ice II (rhombohedral crystal structure). This pronounced elasticity and transparency reflect the absence of defects within the material, and the structure change implies a low barrier for the ice  $I_h$ -to-II transformation.

Ice in nature usually contains pores, microcracks, grain boundaries, crystal dislocations, and other microstructural defects, as well as

surface irregularities. Such features originate through the growth and thermal-mechanical history of the material and act both to concentrate stress and to scatter visible light. However, the almost perfect microfibers studied by Xu *et al.* were produced with a method that used electric-field-enhanced growth (see the figure) (6). Examination with cryo-transmission electron microscopy revealed single crystals without defects and with very smooth surfaces (surface roughness  $<1$  nm).

Upon cooling to  $-150^\circ\text{C}$ , a fiber  $4.4\text{ }\mu\text{m}$  in diameter could be bent with a micromanipulator to a radius as small as  $20\text{ }\mu\text{m}$ . This process created an elastic strain of  $10.9\%$  within the near-surface region. Correspondingly, the outer-fiber stress reached  $\sim 1.4\text{ GPa}$  ( $1\text{ GPa}$  is the pressure on Earth at a depth of  $\sim 30\text{ km}$ ). After manipulator retraction, the fiber had no residual curvature, and multiple fibers exhibited similar mechanical behavior. Rarely have mechanical properties so near theoretical limits been attained in any material.

The optical quality of the microfibers is also attributed to the absence of both interior and exterior defects. The transmissibility was assessed by coupling visible light to one end of a fiber and then by measuring the position-dependent intensity of scattering. As Xu *et al.* suggest, defect-free microfibers of ice have

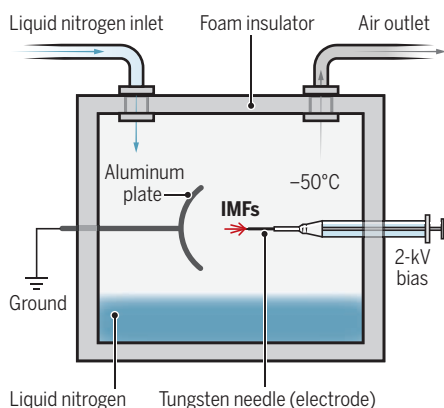
the potential to serve as low-loss, optical waveguides at low temperatures.

Under terrestrial conditions, ice adopts the  $I_h$  crystal structure, and its hexagonal symmetry is reflected in the shape of snowflakes. Under high pressure, however, structures of higher density are favored, of which a dozen or more exist (7). The transformation of  $I_h$  to ice II is marked by an  $\sim 25\%$  increase in density, from  $925$  to  $\sim 1150\text{ kg m}^{-3}$  (at  $-100^\circ\text{C}$ ), accompanied by a proportional decrease in volume. Within the surface region on the compressive side of a highly bent fiber, the stress reached  $\sim 0.4\text{ GPa}$ . Through the use of Raman spectroscopy, Xu *et al.* detected evidence of ice II in a cold fiber ( $-70^\circ\text{C}$ ). Under the combination of stress and temperature just noted, thermodynamics dictates that ice II is the more stable phase (8). The transformation to ice II within the fibers occurred within  $\sim 100\text{ s}$ , indicative of a relatively rapid process compared with sluggish kinetics within bulk ice (9). The small sample could contribute to faster kinetics as the fiber dimensions approach the critical nucleus size.

In showing that ice can reach a high level of mechanical integrity, and with high optical quality, Xu *et al.* revealed the potential for similar improvement through appropriate processing in the behavior of other brittle, crystalline materials. Intriguing issues still remain. Although the elastic strain and strength exhibited by the microfibers are extraordinarily high, they are still below the theoretical limit. Dislocations could have been generated at the free surface and nucleated microcracks that then propagated. The mechanism for scattering visible photons in these near-perfect ice fibers is also not apparent. The ice phase transition could occur within micrometer-sized asperities as they slide slowly past each other across opposing surfaces loaded in shear and compression, creating stick-slip friction. Bending microfibers could potentially achieve even greater pressure and detect transformations to other higher-density ices. ■

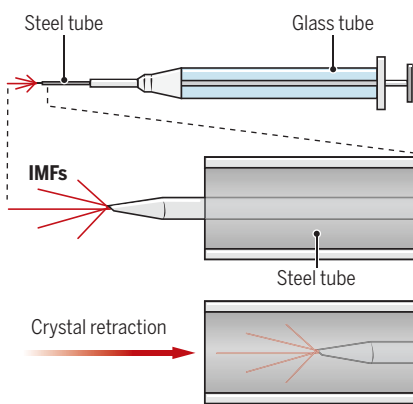
## Field-induced growth

Xu *et al.* grew ice microfibers (IMFs) using an applied electric bias to enhanced diffusion of water vapor toward the tip of a tungsten needle.



## Controlled growth

Schematic of the experimental chamber used to grow IMFs under temperature-controlled conditions is shown.



## Microfiber extraction

Retracting the tungsten needle into a cold coaxial steel tube (1-mm diameter) allows for sample transfer.

## REFERENCES AND NOTES

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