

**PRESSURE-INDUCED TRANSFORMATION PLASTICITY OF WATER ICE.** D. C. Dunand<sup>1</sup>, C. Schuh<sup>1</sup> and D.L. Goldsby<sup>2</sup>, <sup>1</sup>Department of Materials Science and Engineering, Northwestern University, Evanston, IL 60208, dunand@northwestern.edu, csc296@lulu.acns.nwu.edu, <sup>2</sup>Department of Geological Sciences, Brown University, Providence, RI 02912, David\_Goldsby@brown.edu.

Transformation plasticity (TP) is a low-viscosity deformation mechanism which occurs during phase transformation of a low-stressed material. Pressure-induced TP may be important for the dynamics of icy satellites and the earth's interior.

High-pressure polymorphs of ice exist in the interior of many icy satellites of the outer solar system [1, 2]. Because of its relevance to the tectonics of these moons, the rheology of ice polymorphs has been extensively studied [3]. While flow laws for dislocation creep and grain-boundary sliding (GBS) have been determined for ice polymorphs under isobaric and isothermal conditions [3,4], there has been to date no experimental demonstration of transformation-mismatch plasticity in ice produced by a change in isostatic pressure and/or temperature. Transformation-mismatch plasticity (referred to as *transformation plasticity*, or TP, in the present abstract) occurs when a material undergoes a phase transformation while being subjected to a small deviatoric stress [5, 6]. During transformation, the density mismatch between co-existing polymorphic phases produces internal stresses. If no external stress is applied to a transforming specimen, internal stresses give rise to plastic strains which are accumulated isotropically after the forward phase transformation (e.g., upon heating), and completely recovered after the reverse phase transformation (e.g., upon cooling). However, if an external deviatoric stress is applied to the transforming specimen, this stress biases the internal strains, resulting in a net strain increment in the direction of the applied stress after each phase transformation (e.g. both on heating and cooling). TP has been extensively studied in a variety of polymorphic metals, alloys and ceramics [5, 6] subjected to thermal cycling at atmospheric pressure. Strain increments produced after each thermal cycle can be accumulated, leading to very high elongations to fracture (tensile strains larger than 100% are commonly achieved in metals and alloys [5, 6]). TP is also acknowledged to be a relevant deformation mechanism in the earth's mantle through the pressure-induced transformation of olivine and spinel [7, 8]. However, TP produced by pressure variations has, to our knowledge, never been observed experimentally in any material.

Figure 1 shows the H<sub>2</sub>O phase diagram with equilibrium and metastable boundaries between fields for ice I, II, III and V [3]. The maximum pressure in the largest icy moons is expected to be well within the high-pressure polymorphic fields [1]. As for olivine and spinel in the earth's mantle, there will be critical depths in these

moons at which TP can be an active deformation mechanism. While the isostatic pressure in these moons is sufficient to trigger the ice polymorphic transformations, deviatoric stresses are provided by, e.g., convection, plumes, tidal and spin forces, or meteoritic impact. TP results in weak layers at phase boundaries which can influence the internal dynamics and tectonics of these moons.

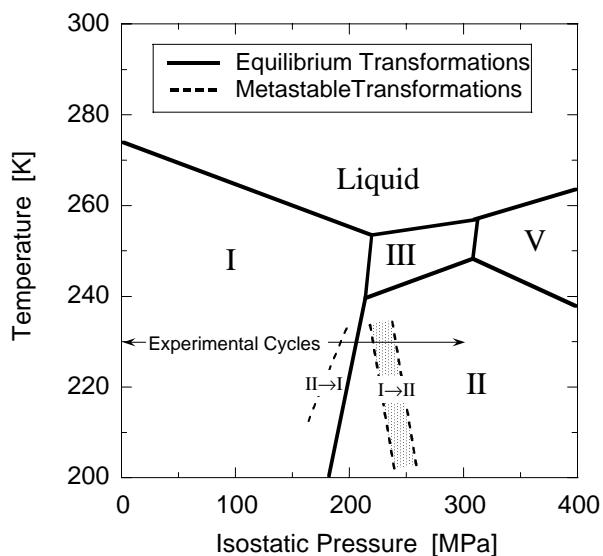


Fig. 1- Section of H<sub>2</sub>O phase diagram showing equilibrium and metastable boundaries between fields for ice I, II, III and V [3], as well as range of experimental pressure cycles.

**Experimental Method:** Samples were prepared by hot-pressing ice powders at 190 K under a uniaxial pressure of 100 MPa, yielding fully dense ice with a grain size of 25-40  $\mu\text{m}$  [4]. Specimens 12.7 mm in diameter and 24-27 mm in length were sealed in thin-walled (150  $\mu\text{m}$ ) indium jackets soldered at both ends to steel end-caps. The indium served as a plenum between the gas medium and the ice sample, accommodating the specimen deformation with negligible constraining force at the test temperature.

Using a cryogenic gas pressure vessel [9], specimens were first subjected to an isostatic pressure cycle up to  $p_{\text{iso}}=200$  MPa (within the field of ice I) at 230 K for 1 min., to eliminate porosity or small gaps in the sample assembly. The length of the sample assembly was measured to  $\pm 5$   $\mu\text{m}$ . The assembly was then spring-loaded in uniaxial compression using steel springs calibrated at the test temperature. The spring-loaded

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specimen was placed in the pressure vessel and thermally equilibrated for 10 min. at 230 K, pressurized at an average rate of 1 MPa/s up to  $p_{\text{iso}}=300$  MPa (within the metastable field of ice II, Fig. 1), held at this pressure for 60 s, then depressurized at a rate of 1 MPa/s. The length of the specimen assembly was then remeasured. In some cases, a specimen was subjected to a second pressure cycle, and in one case ( $\sigma=1.38$  MPa) the test temperature was lowered to 220 K. Finally, two control experiments were conducted under compressive stresses of  $\sigma=0$  and 2.1 MPa at 230 K by cycling to  $p_{\text{iso}}=200$  MPa (within the field of ice I) for the same total time as the transformation experiments.

**Results:** Control specimens cycled within the ice I field showed no measurable strain for  $\sigma=0$  MPa and a small strain of 0.4% for  $\sigma=2.1$  MPa. Since ice II is stronger than ice I [3], these control experiments show that deformation via creep outside the transformation range can be neglected for all I-II cycles.

The ice I-II transformation at 230 K was recorded as a rapid ( $< 1$  s) gas pressure drop at  $p_{\text{iso}}=257\pm 5$  MPa on pressurization and an equally rapid pressure rise at  $p_{\text{iso}}=195\pm 3$  MPa on depressurization. These transformation pressures are in good agreement with the metastable values given in Ref. [3] (see Figure 1).

Figure 2 shows the linear relationship between uniaxial compressive strain after a single I-II cycle at 230 K and the uniaxial compressive stress [10]. After a single cycle, specimens were deformed to true strains as large as 18.5%.

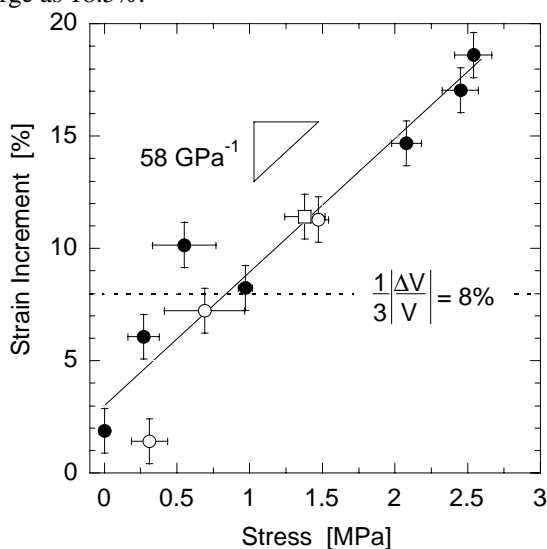


Fig. 2- True strain vs. true stress after a single I-II polymorphic pressure cycle ( $p_{\text{iso}}=0-300-0$  MPa). Empty symbols are for specimens subjected to a previous polymorphic cycle. The square symbol was measured at 220 K, all other points were measured at 230 K.

**Discussion:** The linear relationship in Fig. 2 is strong evidence that TP is the active deformation mechanism, as this linear relationship has been observed in all materials deforming by thermally induced TP and is incompatible with the power-laws expected for GBS [4] or dislocation creep [3]. This linear relationship is also predicted from theoretical treatments of TP [11]. Other indications for TP are the high strains achieved after transformation cycling (much larger than the value of 0.4% for a control specimen cycled at  $\sigma=2.1$  MPa without phase transformation), and the absence of temperature sensitivity for an experiment at 220 K (the dislocation-creep rate of ice I would be quadrupled from 220 to 230 K [3]; the GBS creep rate would be tripled [4]). A simple calculation demonstrates that at the conditions of our experiments, transforming ice is  $\sim 6$  orders of magnitude less viscous than either ice I or II at the same temperature.

Whereas the present study examined isothermal TP associated with the I-II transformation induced by pressure cycling, TP is expected for any transformation permitted by the  $\text{H}_2\text{O}$  phase diagram, i.e., under isobaric temperature variations (e.g., from ice II to III), or with simultaneous changes of both temperature and pressure along planetary profiles. For icy satellites large enough to contain ice isomorphs, many processes may be affected by TP during the formation and early evolution of the moons (accretion and differentiation dynamics), and during ongoing ice tectonics at the global scale (e.g., solid-state convection, spin and tidal bulging, coupling between rocky core and icy lithosphere).

Finally, our observations of pressure-induced TP in ice can serve as an analog for TP in planetary materials for which pressure-induced TP cannot be reproduced in the laboratory: olivine and spinel (e.g., in the mantle of the earth), iron (e.g., in the cores of some planets), and solid hydrogen (e.g., in the core of the gas giants).

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