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Transformation superplasticity of water ice and ice containing SiO_2 particulates

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[1] Transformation superplasticity is a deformation mechanism that occurs during the phase transformation of an externally stressed material. Pressure-induced transformation superplasticity, which has long been postulated to occur in olivine in the Earth's interior, is shown to take place in water ice. Pure ice specimens were subjected to hydrostatic pressure cycling between 0 and 300 MPa to reversibly induce the I/II transformation (at 220 or 230 K) or the I/III transformation (at 240 K). When a small uniaxial compressive stress was applied during cycling, the specimens exhibited a uniaxial compressive strain (as large as 22% after a single cycle) proportional to the applied stress, in agreement with observations and theory for transformation superplasticity of metals and ceramics, induced by polymorphic thermal cycling. Additionally, specimens of ice containing 10 vol% SiO_2 were deformed by this mechanism during the I/II transformation at 230 K. The presence of silica particles was found to enhance the deformation, rather than strengthen the ice. These experimental results are discussed both qualitatively and quantitatively in terms of existing models of transformation superplasticity and implications for the lithosphere rheology of the icy moons of the outer planets. *INDEX TERMS:* 3902 Mineral Physics: Creep and deformation; 3924 Mineral Physics: High-pressure behavior; 6218 Planetology: Solar System Objects: Jovian satellites; 8160 Tectonophysics: Evolution of the Earth: Rheology—general; *KEYWORDS:* Water ice, creep, phase transformation, superplasticity

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1. Introduction

[2] Transformation superplasticity is a deformation mechanism that occurs in externally stressed solids undergoing a polymorphic phase transformation as reviewed by *Dunand* [1997], *Meike* [1993], *Nieh et al.* [1997], and *Poirier* [1985]. During the transformation, density difference between the coexisting polymorphs leads to internal mismatch strains, which are accommodated by plastic flow. A small external deviatoric stress applied during the transformation biases the mismatch accommodation strains, resulting in a macroscopic plastic strain increment in the biasing direction after the transformation is complete.

[3] Two main features characterize transformation superplasticity. First, when the applied external stress is small compared to the internal mismatch stresses, the strain increment after each transformation is proportional to the applied stress, i.e., the average flow law is Newtonian. Second, when compared to creep deformation in the absence of a phase transformation, transformation superplasticity occurs significantly more rapidly. This combination of characteristics (stable Newtonian deformation at rapid rates) allows for strain increments to be accumulated upon multiple transformations up to very large strains, often in excess of 100% in tension for metals.

[4] Transformation superplasticity has been widely investigated for metals (e.g., Fe and Fe-alloys [*Clinard and Sherby*, 1964; *de Jong and Rathenau*, 1959, 1961; *Greenwood and Johnson*, 1965; *Zwigl and Dunand*, 1998b], Ti and Ti-alloys [*Dunand and Bedell*, 1996; *Greenwood and Johnson*, 1965; *Kot and Weiss*, 1967; *Sato et al.*, 1994; *Schuh and Dunand*, 2001b], and other allotropic metals [*Greenwood and Johnson*, 1965; *Stobo*, 1960; *Zamora and Poirier*, 1983; *Zwigl and Dunand*, 1998c]), ceramics (e.g., Bi_2O_3 [*Dunand and Grabowski*, 2000; *Johnson et al.*, 1975]), and composite materials (e.g., Ti-alloys reinforced with TiC [*Dunand and Bedell*, 1996; *Dunand and Myojin*, 1997; *Schuh and Dunand*, 1999] or TiB [*Schuh and Dunand*, 2001a], dual-phase salts [*McLaren and Meike*, 1996], and other systems [*Zwigl and Dunand*, 1998a, 2001]); in all of these cases the transformation is induced by a thermal excursion at ambient pressure. Transformation superplasticity has long been acknowledged as relevant to

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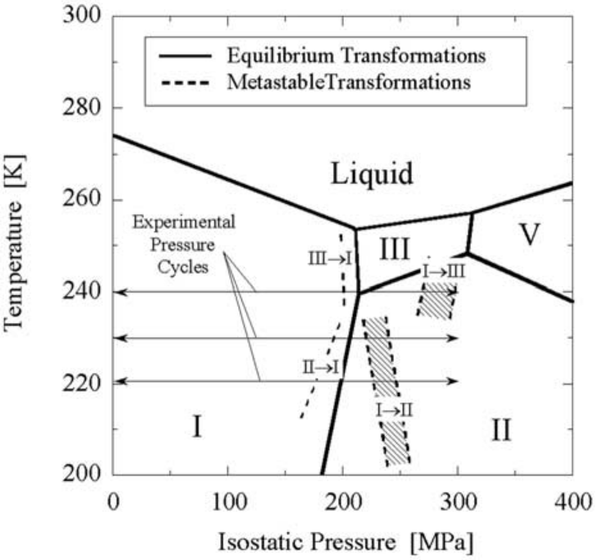

Figure 1. Schematic phase diagram for H_2O ice, showing the equilibrium and metastable boundaries between phase fields for ice I, II, III, V, and liquid, as well as the range of experimental pressure cycles [*Durham et al.*, 1997].

deformation within the Earth's mantle, through the pressure-induced solid-solid transformation of olivine [*Green and Burnley*, 1989; *Kirby et al.*, 1991]. Water ice is another geological material with a polymorphic transformation at pressures more accessible to laboratory experimentation. In a preliminary report, we have recently presented the first experimental observations of transformation superplasticity in H_2O ice during hydrostatic pressure cycles [*Dunand et al.*, 2001].

[5] In the largest icy moons of the outer planets, the internal hydrostatic pressure is believed to exceed the stability range of Ice I [*Durham et al.*, 1997; *Durham and Stern*, 2001], leading to high-pressure ice polymorphs (Figure 1). The tectonics of these moons are governed by the creep deformation of the appropriate ice polymorphs, which may occur by many physical mechanisms, including dislocation creep or grain-boundary sliding [*Durham et al.*, 1997; *Durham and Stern*, 2001; *Goldsby and Kohlstedt*, 1997]. However, within an icy moon, there is a critical depth at which the hydrostatic pressure induces the transformation of Ice I to Ice II or III. As for olivine within the Earth's mantle, transformation superplasticity can be an active deformation mechanism in the vicinity of this critical depth. Deviatoric stresses, which bias the transformation accommodation strains, may arise from mantle convection, tidal and spin forces, faulting, hot plumes, or meteoritic impact. Also, since several of the icy moons are expected to contain mineral inclusions [*Kirk and Stevenson*, 1987; *Mueller and McKinnon*, 1988; *Poirier*, 1982], mismatch may be increased by the transformation of an ice matrix around inert particulates, thus increasing the magnitude of transformation superplasticity.

[6] In this work, we present experimental data for transformation superplasticity of water ice during hydrostatic pressure cycling, with a superimposed uniaxial compressive stress. We discuss our original data for the I/II transformation [*Dunand et al.*, 2001], as well as new data for the I/II transformation in ice reinforced with 10 vol% SiO_2 particulates, in the light of existing micromechanical models for transformation superplasticity of metals and discontinuously reinforced composites. In addition, we present new results on deformation during the I/III transformation.

2. Experimental Procedures

[7] The materials investigated in this work were water ice in pure form or containing 10 vol% SiO_2 particulates. Cylindrical specimens were fabricated through a powder-processing route, described in detail by *Goldsby and Kohlstedt* [1997]. First, ice powders were produced by atomizing distilled water into liquid nitrogen, followed by wet-sieving in liquid nitrogen to <25 μm. Cylindrical billets were consolidated in an evacuated die 12.7 mm in diameter, for 2 h at 190 K, under a uniaxial pressure of 100 MPa, leading to fully dense ice with 25–40 μm grain size [*Goldsby and Kohlstedt*, 1997]. For the particulate-containing specimens, 10 vol% SiO_2 powder (<150 μm diameter, from Cerac, Milwaukee, WI) was dry-mixed with the sieved ice powder for 10 minutes min prior to hot pressing.

[8] Following *Stern et al.* [1997], the cylindrical specimens (12.7 mm diameter and 24–30 mm in length) were introduced into thin-walled indium tubes (0.15 mm wall thickness), which were subsequently sealed by soldering to copper-coated steel end-caps (Figure 2a). The indium jacket prevented gas penetration into the ice during pressurization, but crept rapidly enough to accommodate the deformation of the ice specimen without constraining it. The ends of the ice cylinder were covered with Teflon tape (25 μm thickness), to minimize lateral friction during the compression experiments. Prior to deformation experiments the length of the jacketed specimen assembly (typically 64–70 mm) was measured with a micrometer to ±5 μm.

[9] All experiments were conducted in a cryogenic pressure vessel [*Durham et al.*, 1983] using nitrogen gas as a pressure medium. Prior to deformation experiments, the specimen assembly was typically subjected to hydrostatic pressure of $P = 200$ MPa (well below the transformation pressures, Figure 1) for one minute, to insure that the ice specimen was well-seated against the compression surfaces. The specimen assembly was loaded into a steel tube and spring-loaded in compression using steel platens and custom steel springs that were calibrated at the test temperature. This arrangement allowed for the application of uniaxial compressive stresses up to $\sigma = 2.5$ MPa, which remained constant to within 10% during all experiments. The compressively stressed specimen was placed in the pressure vessel and allowed to thermally equilibrate for about 10 min. to the test temperature. Pressure cycles were performed between $P = 0$ and 300 MPa, with average ramp rates of 1 MPa/s, and a dwell of 1 min. at the upper pressure. After the pressure cycle, the compressive load was removed and the specimen assembly extracted from the pressure vessel. The length of the jacketed specimen assembly was again measured, and all deformation assigned to the ice specimen. In some cases, the specimen was subjected to a second pressure cycle with a different applied stress.

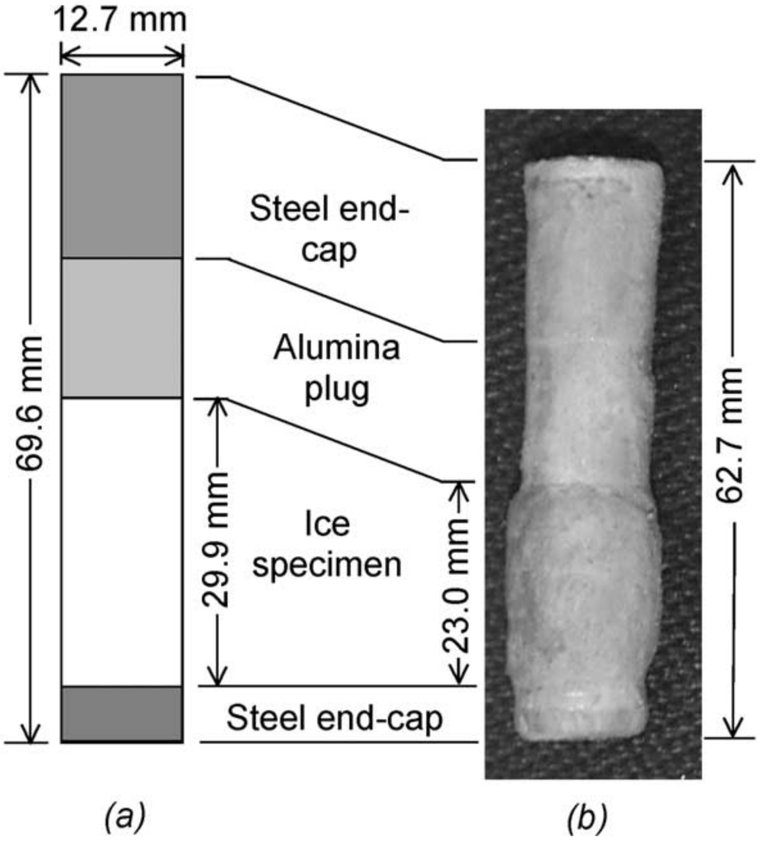

Figure 2. (a) Schematic of typical specimen assembly before deformation. (b) Photograph of the same assembly showing axial and radial strains for an ice specimen after two I–II pressure cycles (P = 0-300-0 MPa at 230 K) under an uniaxial compressive stress $\sigma = 2.1$ and 1.5 MPa, respectively.

[10] Two different ice phase transformations were examined in this work:

- A series of pressure cycling experiments was conducted at T = 230 K, to access the I/II transformation (Figure 1). This temperature was used for most of the experiments on ice, and all of the experiments on the ice/SiO_2 composites. In one case, for pure ice, the test temperature was lowered to T = 220 K (still in the range of the I/II transformation). Finally, two control experiments were conducted at T = 230 K, in which the specimen was subjected to a pressure cycle of the same duration as the other cycles, but with a maximum pressure of P = 200 MPa. Since this pressure is below the critical I/II transformation pressure (Figure 1), these specimens remained in the ice I field for the entire cycle, and thus deformed only by creep under the applied compressive stress $\sigma = 0$ or 2.1 MPa.
- Some limited experiments were conducted at T = 240 K, where ice I transforms to ice III during the pressure cycles used in this work (Figure 1), for purposes of comparison with the data for the I/II transformation.

3. Results

[11] During pressure cycling experiments, the phase transformations were observed as small discontinuities in the gas pressure history, in all cases having a duration of about 1 s. At T = 230 K, the transformation was observed at P = 257 ± 5 MPa on pressurization, and P = 195 ± 3 MPa on depressurization. The transformation pressures at T = 240 K were found to be P = 290 ± 5 and 195 ± 5, on pressurization and depressurization, respectively. For all of these transformations, the critical pressures are in good agreement with the metastable values reported by *Durham et al.* [1997] and shown in Figure 1. Furthermore, the transformation pressures on pressurization unambiguously identify the high-pressure polymorph produced during cycling; ice II at T = 230 K and ice III at T = 240 K. Finally, the speed of these transformations (~1 s) agree with prior experimental data on the kinetics of the ice phase transformations (W. B. Durham and S. H. Kirby, Lawrence Livermore National Laboratory, unpublished research, 1999).

[12] After pressure cycling, the specimens exhibited considerable plastic deformation, with a maximum true strain value of 22%, for I/III cycling at T = 240 K and $\sigma = 1.15$ MPa (in this paper, uniaxial compressive strains are written as positive, and uniaxial tensile strains as negative). An example of a deformed jacketed specimen is shown in Figure 2b. The ice specimen cross-section has increased due to the uniaxial compressive strain, which was not completely uniform. The specimens were also usually somewhat barreled, despite Teflon lubrication at the ends, but very rarely contained macrocracks. Also, the pure ice

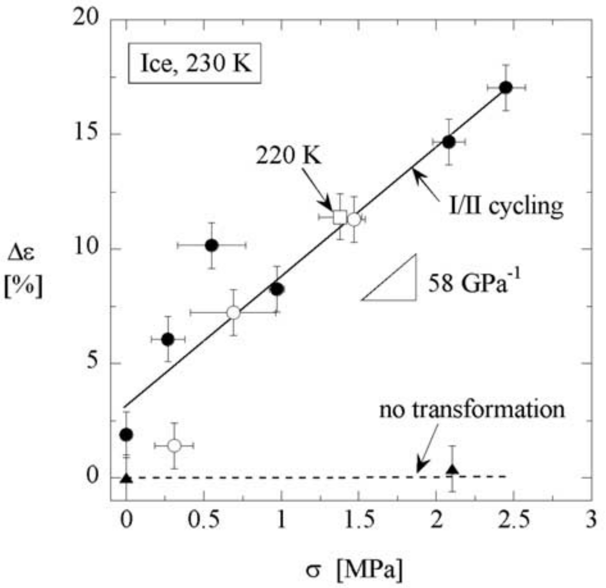

Figure 3. Uniaxial compressive strain increment $\Delta\varepsilon$ accumulated after a complete I/II transformation pressure cycle at 230 K, as a function of the applied compressive stress σ. Open symbols are from specimens that had been subjected to a previous transformation cycle, and the square symbol was measured at 220 K. The amount of strain expected from typical creep mechanisms without transformation is shown as a dashed line, and can be compared with the control experiments that were cycled between P = 0 and 200 MPa, without transformation (triangles).

specimens were optically clear after cycling, indicating that microcracking was not extensive (or had been healed by diffusion-bonding during the depressurization step).

[13] The uniaxial compressive strain increment $\Delta\varepsilon$ developed in pure ice after one pressure cycle at T = 230 K (through the I/II transformation range) is shown as a function of the applied deviatoric stress σ in Figure 3. Despite some scatter at the lower stresses, the data are consistent with a linear relationship, with an intercept near 3% at $\sigma = 0$. The single experiment conducted at T = 220 K and $\sigma = 1.4$ MPa resulted in a compressive strain increment ($\Delta\varepsilon = 11.5\%$) in good agreement with the trend of the data at T = 230 K. The slope of the relationship in Figure 3 is found to be $d(\Delta\varepsilon)/d\sigma = 58 \pm 11$ GPa^{-1}, much larger than typically observed for metals or alloys during thermal cycling through a polymorphic transformation (0.1–5 GPa^{-1} [*Nieh et al.*, 1997]).

[14] The very large uniaxial strains measured after pressure cycling were developed during a cycle of about 11 minutes. In the same amount of time, under compressive stress of $\sigma = 0$ or 2.1 MPa, the control specimens pressure-cycled entirely in the ice I phase field exhibited strains of 0.0% and 0.4%, respectively, as shown by the triangular symbols in Figure 3. Because ice II creeps more slowly than ice I at the same applied stress, the two triangular data points in Figure 3 represent an upper-bound on the amount of creep strain expected during a full pressure cycle with an excursion into the ice II field. The data in Figure 3 demonstrate that the I/II transformation results in an extraordinary deformation enhancement compared to creep outside the transformation range, which is negligible during full pressure cycles. Both the linear stress-dependence and the enhanced deformation (as compared to creep) observed in Figure 3 are defining characteristics of transformation superplasticity, and strongly suggest the operation of this deformation mechanism during pressure cycling.

[15] In Figure 4, the relationship between $\Delta\varepsilon$ and σ is shown for the ice specimens reinforced with 10 vol% SiO_2 particulates, subjected to pressure cycling at T = 230 K. Again, a linear relationship is observed, with a slope nearly twice that for pure ice (Figure 3), $d(\Delta\varepsilon)/d\sigma = 112 \pm 12$ GPa^{-1}. This result is consistent with work by Dunand and co-workers [*Dunand and Bedell*, 1996; *Schuh and Dunand*, 1999] on titanium reinforced with 10 vol% TiC particulates, for which the slope $d(\Delta\varepsilon)/d\sigma$ was about a factor of two higher than for unreinforced Ti during thermal cycling through the Ti α/β transformation.

[16] Figure 5 shows limited data acquired during pressure cycling at T = 240 K, through the I/III phase transformation, again showing the uniaxial strain increment $\Delta\varepsilon$ as a function of the applied compressive stress σ. Although only three data points are presented, they fall within experimental error on a line, and extend to very large strain increments, $\Delta\varepsilon \approx 22\%$ at $\sigma = 1.15$ MPa. The slope of a linear least squares fit to these data is $d(\Delta\varepsilon)/d\sigma = 248 \pm 19$ GPa^{-1}, larger than for either pure ice or the ice/SiO_2 composites during cycling at T = 230 K.

4. Discussion

4.1. Creep Deformation Outside the Transformation Range

[17] During a complete pressure cycle (P = 0-300-0 MPa), a specimen undergoes several distinct stages of deforma-

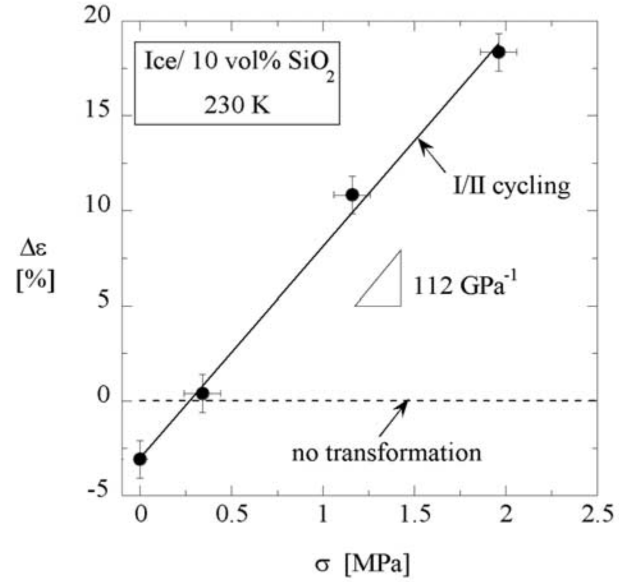

Figure 4. Uniaxial compressive strain increment $\Delta\varepsilon$ as a function of the applied compressive stress for ice/10 vol% SiO_2 composites, after a complete pressure cycle involving the ice I/II transformation at 230 K. Expected creep strain in the absence of transformation superplasticity is shown as a dashed line for comparison.

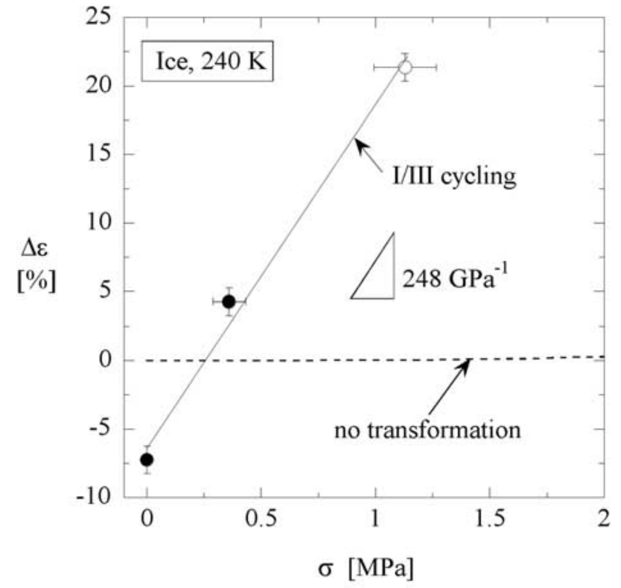

Figure 5. Uniaxial compressive strain increment after each ice I/III transformation cycle at 240 K, as a function of the applied stress σ. Open symbol was measured on a specimen that experienced a previous transformation cycle, and the amount of creep strain expected without an enhancement due to the phase transformation is shown as a dashed line.

tion. As the cycle begins, the specimen is in the ice I phase, and creeps under the action of the applied uniaxial stress. Although the creep rate decreases with hydrostatic pressure, this pressure dependency is weak [*Durham et al.*, 1997]. At the critical pressure $P_{I/II}$ = 257 MPa (or $P_{I/III}$ = 290 MPa), the specimen transforms in about 1 s, during which time it undergoes transformation superplasticity. After the transformation, up to the maximum hydrostatic pressure P = 300 MPa, the specimen is in the high-pressure ice II (or ice III) field, and further undergoes creep under the applied load. On the depressurization half-cycle, the above events are reversed, with the transformation occurring at a different critical pressure ($P_{II/I}$ = $P_{III/I}$ = 195 MPa). Thus, during a complete cycle, the total deformation experienced by the specimen is a sum of three independent contributions: creep of the low-pressure ice I phase, transformation superplasticity (due to two transformations), and creep of the high-pressure phase (ice II at 220 or 230 K, or ice III at 240 K).

[18] In the temperature and stress ranges of interest to the present work, ice I deforms by two possible mechanisms, i.e., dislocation creep or grain-boundary sliding [*Durham et al.*, 1997; *Goldsby and Kohlstedt*, 1997]. Using data from the literature [*Durham et al.*, 1997; *Goldsby and Kohlstedt*, 1997], the expected amount of creep deformation accumulated during a complete pressure cycle (without a transformation superplasticity contribution) is shown as a function of the applied stress (dashed line in Figure 3), accounting for creep of both phases, the change of pressure during the cycle, and using a grain size of 25 μm (expected from the powder-processing method used to fabricate the specimens [*Goldsby and Kohlstedt*, 1997]). The experimental control experiments, in which the specimens were cycled up to P = 200 MPa, without transformation, are in agreement with the calculated trend, and demonstrate the major enhancement in deformation induced by the phase transformation. Results of a similar calculation, again using constitutive laws from [*Durham et al.*, 1997], has been performed for the I/III pressure cycles; the result shown in Figure 5 exhibits the same essential features as in Figure 3, and also illustrates a strong deformation enhancement during transformation cycling.

[19] Isobaric creep of various ice/SiO_2 composites has been investigated by *Durham et al.* [1992], who found only a small effect of the reinforcement phase on the creep of ice I. These authors also present a constitutive law for creep of these composites, from which we have calculated the expected strain due solely to creep of the ice I/10 vol% SiO_2 and ice II/10 vol% SiO_2 materials during pressure cycling, as shown in Figure 4 (dashed line). Again, the expected creep strain is very substantially below the measured strain accumulated after transformation pressure cycles.

[20] To summarize this section, isobaric creep data are available in the literature for all of the materials used in this work. When integrated over the full pressure cycle, the amount of creep strain expected by typical creep mechanisms (i.e., dislocation creep and grain-boundary sliding) is found to be negligible compared to the very large strains measured during pressure cycles involving the I/II or I/III phase transformations.

4.2. Transformation Strain Ratcheting

[21] During pressure cycling without applied stress, specimens experienced a macroscopic longitudinal shrinkage of 2% for the I/II transformation cycles (Figure 3) and a longitudinal expansion 7% for I/III cycles (Figure 5). The composite materials also exhibited a longitudinal expansion of 3% for I/II cycles in the absence of applied stress (Figure 4). Macroscopic plastic deformation that is accumulated without external stress is referred to as ratcheting strain [*Stobo*, 1960], and is observed in metals and metal-matrix composites when internal mismatch stresses (e.g., from thermal-expansion or transformation mismatch) produce irreversible plastic strain. *Stobo* [1960] described the mechanism of strain ratcheting during an allotropic phase transformation, where the weaker phase deforms to accommodate the shape of the stronger allotrope. Strain ratcheting has been observed during thermal cycling of several allotropic metals without applied stress [*Buckley et al.*, 1958; *Frary et al.*, 2001; *Furushiro et al.*, 1987; *Stobo*, 1960; *Zwigl and Dunand*, 1998b], but, to our knowledge, there are no prior reports of transformation strain ratcheting induced by a pressure excursion.

[22] The strain ratcheting observed in ice in this work has several possible causes, including (i) crystallographic texture produced by uniaxial compaction of the specimens, (ii) propagation of a macroscopic transformation front, with local plastic deformation occurring at the phase boundary, or (iii) a small amount of transformation superplasticity, in which friction between the specimen and the compression platens provided a small biasing stress. According to the theory of thermally induced transformation strain ratcheting [*Buckley et al.*, 1958; *Greenwood and Johnson*, 1962; *Stobo*, 1960], the magnitude of macroscopic uniaxial deformation after a complete two-transformation cycle is usually at most one-third of the transformation volume change, $\Delta V/V$. For the I/II transformation, this upper bound is $\Delta V/3V \approx 8.0\%$

[*Durham et al.*, 1997], compared to the measured longitudinal ratchet of 2%; the I/III transformation produced a 7% ratchet, which is close to the upper-bound expectation of $\Delta V/3V \approx 7.3\%$ [*Durham et al.*, 1997]. Although the internal stress state of a transforming ice/SiO_2 composite is considerably more complicated than for pure ice, the measured ratcheting strain of 3% is still within the bounds expected from the existing theory.

[23] Finally, we note that the ratcheting strains measured for the I/III and I/II transformation cycles have opposite signs. The theory of transformation ratcheting [*Stobo*, 1960] predicts such a sign change if there is a change in the relative strengths of the two phases (i.e., the weaker phase becomes the stronger), or if the transformation volume change, $\Delta V/V$, changes sign. For both the I/II and I/III transformations, the ice I phase is the less dense polymorph; however, ice I is stronger than ice III, but weaker than ice II [*Durham et al.*, 1997]. Thus, the observed change of sign in the ratcheting strain for the I/II and I/III transformations is qualitatively expected based on theory, and each of the two ratchets (I/II and I/III) have the predicted sign.

4.3. Transformation Superplasticity

[24] The two defining features of transformation superplasticity expected at low applied stress levels, are (i) enhanced deformation compared to creep and (ii) a linear stress dependence of $\Delta\varepsilon$. As observed in Figures 3–5, both of these characteristics are present for the I/II and I/III transformations of pure ice, as well as for the I/II transformation in composite specimens. These observations are strong evidence that transformation superplasticity is the operative deformation mechanism during pressure cycling, as these characteristics have been observed in all metals and metal-matrix composites deforming by transformation superplasticity [*Dunand*, 1997; *Nieh et al.*, 1997], and the measured linear relationship between $\Delta\varepsilon$ and σ is incompatible with the power laws expected for grain-boundary sliding or dislocation creep in water ice, with respective stress exponents of 1.8 and 4 [*Durham et al.*, 1997; *Goldsby and Kohlstedt*, 1997]. Also, the lack of temperature sensitivity for the experiment performed at 220 K (compared with the experiments at 230 K, for the I/II transformation, Figure 3) is consistent with the weak temperature dependence of transformation superplasticity [*Kitazono et al.*, 1999; *Schuh and Dunand*, 2001b], but inconsistent with the strong temperature dependence of typical creep mechanisms (the dislocation-creep rate of ice I would be quadrupled from 220 to 230 K [*Durham et al.*, 1983]; the grain-boundary sliding creep rate would be tripled [*Goldsby and Kohlstedt*, 1997]).

[25] In the following sections, we consider our experimental results quantitatively in terms of the widely accepted model of transformation superplasticity by *Greenwood and Johnson* [1965], which has heretofore been used to describe this mechanism during thermal cycling. We first consider the I/II transformation, and subsequently examine the effect of SiO_2 reinforcement on transformation superplasticity. Finally, we discuss the I/III transformation, which we quantitatively compare to the I/II transformation.

4.3.1. Ice I/II Transformation

4.3.1.1. Transformation Superplasticity of Pure Ice

[26] *Greenwood and Johnson* [1965] developed a relationship for the strain increment $\Delta\varepsilon$ accumulated after a full polymorphic cycle (two phase transformations), for the case in which one phase accommodates the transformation by time-independent yield at its yield stress, σ_y:

$$\Delta\varepsilon \approx \frac{5}{3}\cdot\frac{\Delta V}{V}\cdot\frac{\sigma}{\sigma_y} \tag{1}$$

For the I/II transformation, ice I is significantly weaker than ice II [*Durham et al.*, 1997], and can therefore be assumed to accommodate the transformation mismatch. Furthermore, because the transformation is rapid (<1 s), it may be reasonable to take the accommodating flow of ice I to be time-independent at a constant flow stress σ_y, rather than time-dependent by a creep mechanism (which will be discussed later). The slope of the data in Figure 3 is $\Delta\varepsilon/\sigma = 58\ \mathrm{GPa}^{-1}$, from which a yield stress of $\sigma_y = 6.9$ MPa is calculated with equation (1) using $\Delta V/V = 24\%$ [*Durham et al.*, 1997]. Because the transformation strain evolves in about one second, the average accommodation strain rate is $\sim 0.1\ \mathrm{s}^{-1}$, for which the flow stress of ice I is expected to be about 59 MPa at T = 230 K and P = 228 MPa, close to the transformation pressure [*Durham et al.*, 1997]. The discrepancy between the expected flow stress and that found experimentally with equation (1) will be discussed in a later paragraph.

[27] For the more complex case where the transformation mismatch is accommodated by time-dependent power law creep, *Greenwood and Johnson* [1965] developed an alternate form of equation (1):

$$\Delta\varepsilon \approx \frac{4}{3}\cdot\frac{\Delta V}{V}\cdot\frac{5n}{4n+1}\cdot\frac{\sigma}{\sigma_o} \tag{2}$$

in which n is the creep stress exponent, and σ_o is the average internal equivalent stress during the transformation, and is given by:

$$\sigma_o = \left[\frac{2}{3}\cdot\frac{\Delta V}{V}\cdot\frac{1}{K}\cdot\frac{1}{\Delta t}\right]^{\frac{1}{n}} \tag{3}$$

where K is the temperature-dependent constant in the creep power law of the weaker phase (strain rate $\dot{\varepsilon} = K\sigma^n$), and Δt is the duration of the phase transformation.

[28] For the I/II transformation, the experimental data from Figure 3 are best fitted to equation (2) when the internal stress parameter takes a value of $\sigma_o \approx 6.5$ MPa, using a stress exponent of n = 4 for dislocation creep [*Durham et al.*, 1997] (given the high internal stress of 6.5 MPa, dislocation creep is expected to be the dominant transformation accommodation mechanism, rather than grain boundary sliding). We note that this internal stress is very close to that predicted by fitting the experimental data to equation (1) ($\sigma_y = 6.9$ MPa), which assumes time-independent yield accommodation of the transformation mismatch. In the present case, the fitted value of σ_o can be compared with the predictions of equation (3), using $\Delta t \approx 1$ s, and for creep of ice I at P = 250 MPa and T = 230 K, where $K = 9.6 \times 10^{-9}\ \mathrm{MPa}^{-4}$ and n = 4 [*Durham et al.*, 1997; *Goldsby and Kohlstedt*, 1997]. With these values as input, equation (3) predicts the internal stress to be $\sigma_o = 103$ MPa, much larger than the above fitted value ($\sigma_o = 6.5$ MPa).

[29] To summarize the above discussion, the rapid I/II transformation mismatch accommodation can be described either as time independent yield (equation (1)), or as rapid time-dependent power law creep (equation (2)). In either case, the models of *Greenwood and Johnson* [1965] can be fitted to the experimental data to predict a flow stress, or internal stress, due to the transformation, in the range 6.5–7.0 MPa. In contrast, previous literature data suggests that the internal stress or flow stress of the transformation accommodation should be about an order of magnitude higher. This discrepancy might be due to the operation of alternative deformation mechanisms with lower flow stresses, which include cracking (ice was found to crack in compression at $\sigma = 6.2$ MPa at $T = 233$ K and atmospheric pressure [*Hobbs*, 1974]), dynamic recrystallization (particularly likely to occur during and just after the phase transformation), or biasing of the transformation strains ($\Delta V/V$) to develop preferentially in the direction of the applied stress (the Magee effect [see, e.g., *Magee*, 1966]).

4.3.1.2. Effect of SiO_2 Reinforcement on Transformation Superplasticity

[30] Of the existing studies of transformation superplasticity in systems with only one transforming component, most have found that reinforcement leads to smaller strain increments $\Delta\varepsilon$ at a given stress σ. However, this strengthening effect is usually due to complex phenomena such as: load transfer to high aspect-ratio reinforcements [*Schuh and Dunand*, 2001a], increased strain-hardening around the inclusion phase [*McLaren and Meike*, 1996; *Zwigl and Dunand*, 1998b], or strengthening of the matrix by partial dissolution of the reinforcement phase [*Schuh et al.*, 1999; *Zwigl and Dunand*, 1998b]. In contrast, in commercial-purity titanium reinforced with 10 vol% equiaxed TiC particles, a significant enhancement of the strain increment $\Delta\varepsilon$ due to the presence of the inert reinforcement phase was observed [*Dunand and Bedell*, 1996; *Schuh and Dunand*, 1999]. In that system, the reinforcement was equiaxed (greatly reducing the amount of load transfer as compared to elongated or whisker reinforcement), the transformation temperature was high (eliminating the possibility of strain hardening), and the solubility of carbon in titanium is very small (reducing the possibility of solid-solution strengthening the titanium matrix with carbon). Thus, the above complicated phenomena that can lead to strengthening were absent from those experiments, and the direct effect of the reinforcement on transformation superplasticity was observed, i.e., a doubling of the superplastic slope, $\Delta\varepsilon/\sigma$, during thermal cycling through the matrix transformation.

[31] In qualitative agreement with these results during thermal cycling of the Ti/10 vol% TiC system, a similar increase is found in the present work upon the addition of 10 vol% SiO_2 particles to ice during pressure cycling; the superplastic slope increases from $d(\Delta\varepsilon)/d\sigma = 58\ GPa^{-1}$ (pure ice) to $d(\Delta\varepsilon)/d\sigma = 112\ GPa^{-1}$ (ice composites). As for the Ti/TiC specimens, our ice/silica specimens exhibit an equiaxed morphology of the reinforcement phase and were tested at a high homologous temperature, so that none of the complex strengthening mechanisms described above are expected for this system. Then, the measured enhancement of transformation superplasticity can be considered a direct effect of the presence of reinforcement particles. This enhancement is of practical importance to geological problems, since the addition of reinforcement typically results in a reduction of the isobaric creep rate [*Durham et al.*, 1992].

[32] In their original article on transformation superplasticity in Ti/TiC, *Dunand and Bedell* [1996] presented a simple model that they used to estimate the effect of reinforcement. They suggested that the transformation of the matrix phase around inert, non-transforming particulates led to an additional amount of internal strain mismatch, and therefore caused an enhancement of the observed transformation superplasticity. They estimated the magnitude of this effect by adapting a model by *Sato and Kuribayashi* [1993], which considers the deformation of a material containing a spherical inclusion, subjected to a volumetric mismatch between the particle and the matrix:

$$\frac{d(\Delta\varepsilon_c)}{d\sigma} = \frac{d(\Delta\varepsilon)}{d\sigma} + \int_{\Delta t} \frac{4n\cdot(n+4)}{2^{1/n}\cdot 5}\cdot\frac{\left(1-f^{1/n}\right)}{(1-f)^2}\cdot\left(f\cdot\dot{\varepsilon}_{mis}\right)^{1-1/n}\cdot K^{1/n}\cdot dt \qquad (4)$$

where $\Delta\varepsilon_c$ and $\Delta\varepsilon$ are the strain increments after a complete two-transformation cycle of the composite and the matrix, respectively, f is the volume fraction of inclusions, $\dot{\varepsilon}_{mis}$ is the rate at which the internal mismatch strains develop, and the accommodation occurs according to a power law with a stress exponent n and pre-stress constant K. In the present case of ice/SiO_2, the linear transformation mismatch strain is equal to $\Delta V/3V$, and develops over the time of the transformation, $\Delta t \approx 1$ s:

$$\dot{\varepsilon}_{mis} \approx \frac{1}{\Delta t}\cdot\left|\frac{1}{3}\cdot\frac{\Delta V}{V}\right| \qquad (5)$$

We note here that there is an additional source of mismatch due to the compressibility mismatch ($\Delta B \approx 0.1\ GPa^{-1}$ [*Dunand and Bedell*, 1996; *Hobbs*, 1974]) between ice I and SiO_2, giving a maximum mismatch strain of ~0.9%, and an average mismatch strain of ~0.4% during pressure cycling. Because this mismatch is small compared to the transformation mismatch of ~8%, it is neglected in the following discussion.

[33] Equation (4) can be simplified by assuming that the transformation mismatch in the matrix and the mismatch between particle and matrix during the transformation are both accommodated by the same mechanism (as also implicitly assumed by *Dunand and Bedell* [1996]), and combining equations (2)–(5) to yield:

$$\frac{d(\Delta\varepsilon_c)}{d\sigma} = \frac{d(\Delta\varepsilon)}{d\sigma}\cdot\left[1+\frac{(n+4)\cdot(4n+1)}{25}\cdot\frac{\left(1-f^{1/n}\right)}{(1-f)^2}\cdot f^{1-1/n}\right] \qquad (6)$$

which gives a prediction for the superplastic slope of the composite in terms of that for the pure matrix, with only two input parameters: the volume fraction of the reinforcement, f, and the stress-exponent of the accommodation mechanism, n. There are two physically reasonable mechanisms to accommodate the mismatch between the particle and the matrix, including power law creep (n = 4 [*Durham et al.*, 1997]) and grain-boundary sliding (n = 1.8 [*Durham et al.*, 1997; *Goldsby and Kohlstedt*, 1997]). Using the stress exponents for each of these possible accommodation mechanisms in equation (6) gives predicted values for the superplastic slope of $d(\Delta\varepsilon_c)/d\sigma$ = 88 or 93 GPa^{-1}, respectively. With either accommodation mechanism, the model predicts a significant enhancement due to the presence of reinforcement, and the predicted values are in reasonable agreement with the experimentally measured value of $d(\Delta\varepsilon_c)/d\sigma = 112 \pm 12$ GPa^{-1}.

4.3.2. Ice I/III Transformation

[34] The superplastic slope during the I/III transformation cycles at 240 K ($d(\Delta\varepsilon)/d\sigma$ = 248 GPa^{-1}, Figure 5) is significantly larger than for the I/II transformation ($d(\Delta\varepsilon)/d\sigma$ = 58 GPa^{-1}, Figure 3), although the temperature of these experiments differed by only 10 K. As already discussed, this slope is characteristic of the operative transformation, and depends on the volume mismatch, the creep properties of the weaker phase, and the duration of the transformation, according to equation (2). Both these transformations have similar volume changes ($\Delta V/V_{I/II}$ = 24% and $\Delta V/V_{I/III}$ = 22% [*Durham et al.*, 1997]) and similar durations ($\Delta t \approx 1$ s, as observed experimentally). The main difference between the two cases lies in the accommodation of the transformation strain, which, as previously discussed, occurs primarily in the weaker of the two polymorphic phases. For the I/II transformation, the accommodating phase is ice I; however, during the I/III transformation, ice III is substantially weaker than ice I (by about three orders of magnitude [*Durham et al.*, 1997]) and therefore accommodates the volume mismatch.

[35] Both ice I and III creep with a stress exponent $n \approx 4$ [*Durham et al.*, 1997], so the expected ratio Θ of the superplastic slopes for these two transformations can be found from equation (2) as:

$$\Theta \equiv \frac{(d(\Delta\varepsilon)/d\sigma)_{I/III}}{(d(\Delta\varepsilon)/d\sigma)_{I/II}} \approx \left(\frac{K_{III}}{K_I}\right)^{1/n} \cdot \left[\frac{\left(\frac{\Delta V}{V}\right)_{III}}{\left(\frac{\Delta V}{V}\right)_{I}}\right]^{1-1/n} \tag{7}$$

Using the values for ΔV/V described earlier, and creep parameters from [*Durham et al.*, 1997] ($K_{III} = 3.4 \times 10^{-7}$ $MPa^{-4}s^{-1}$ at T = 240 K and P = 290 MPa, and $K_I = 3.0 \times 10^{-10}$ $MPa^{-4}s^{-1}$ at T = 230 K and P = 257 MPa), equation 7 gives the ratio of superplastic slopes as Θ = 5.4. This value is in good agreement with the ratio of the experimental slopes, Θ = (248 ± 19)/(58 ± 11) = 4.3 ± 1.4. As discussed earlier with regard to the I/II transformation, power law creep may not be the only accommodation mechanism, and other contributions from, e.g., cracking or dynamic recrystallization may also occur during I/III transformation superplasticity. Consideration of such additional mechanisms would change the predictions of equation (7). However, the ratio of relative strengths of the accommodating phase (K_{III}/K_I) can be varied over a broad range (i.e., by a factor of three) without significantly changing the value of Θ (= 5.4 ± 1.7).

4.3.3. Geophysical Implications

[36] Whereas transformation superplasticity was examined here under isothermal conditions for the I–II and I–III transformations induced by pressure cycling, this deformation mechanism is expected for any transformation allowed by the H_2O phase diagram, i.e., under isobaric, thermal cycling conditions (e.g., from ice II to III), or for cases where both temperature and pressure are changed (e.g., along planetary profiles). Because hydrostatic pressures are sufficient to form ice isomorphs for the largest icy moons of the outer planets, transformation superplasticity may affect many geological processes in their ice lithospheres: during the formation and early evolution of the moons (accretion and differentiation dynamics, solidification of an early global ocean) and for ongoing ice tectonics at the global scale (e.g., motion of slabs, quakes, solid-state convection, spin and tidal bulging, coupling between rocky core and icy lithosphere). Small-scale geological features may also be affected, as reviewed by *Pappalardo et al.* [1999], who postulated a low-viscosity layer of creeping ice or liquid water to explain many local features on Europa: thermal plumes, faulting, spreading centers, diapirism, topographic flexing around massifs, cryovolcanism, and formation and relaxation of surface features such as impact craters, cracks and ridges. Transformation superplasticity could provide a low-viscosity solid layer in Europa and other large icy moons such as Ganymede, the surface features of which have also been discussed in terms of ice creep [*Kirk and Stevenson*, 1987]. Also, since the rheology of the icy lithosphere controls both heat dissipation (by convection) and heat creation (by tidal deformation) [*Pappalardo et al.*, 1999], transformation superplasticity may have important implications for the presence of a subsurface ocean on these moons. Additionally, transient phenomena, e.g., quakes, meteoritic impact and subsequent crater formation, may be affected by the presence of transformation superplasticity as a deformation mechanism.

[37] Finally, our experimental observations of pressure-induced transformation superplasticity in ice indicate that other polymorphic geological materials exhibit this deformation mechanism under appropriate conditions of temperature and pressure: other ices (e.g., ammonia, methane, carbon dioxide, and clathrates), salts (e.g., sodium chloride), olivine (e.g., in the mantle of the Earth and other rocky planets [*Green and Burnley*, 1989; *Kirby et al.*, 1991]), other silicates (e.g., majorite and pyrope [*Heinemann et al.*, 1997]), iron (e.g., in large meteorites and the cores of planetesimals, rocky moons and planets), and even solid hydrogen (e.g., in the core of giant solar and extrasolar planets).

5. Conclusions

[38] Transformation superplasticity is a deformation mechanism that occurs in polymorphic materials subjected to a small external stress, while simultaneously undergoing a polymorphic phase transformation. It is characterized by rapid deformation, in excess of the rates attainable by, e.g., dislocation creep or grain boundary sliding, and a linear flow law, in which the strain accumulated after a full polymorphic cycle (two transformations) is proportional to the applied external stress. In this article, we describe experiments on water ice subjected to the I/II or I/III transformations during a pressure excursion, as well as ice containing 10 vol% SiO_2 particles subjected to the I/II transformation.

[39] During hydrostatic pressure cycles over the range 0–300 MPa, ice exhibits the linear flow law and rapid deformations associated with transformation superplasticity, at 220 or 230 K (where the ice I/II transformation occurs) as well as 240 K (where the I/III transformation occurs). Combined with an earlier preliminary report [*Dunand et al.*, 2001], the present work represents the first experimental observation of transformation superplasticity in any material under conditions of pressure cycling. After a single 11-minute cycle, pure ice was deformed (without extensive cracking) to compressive strains as large as 22%. The commonly used theory of transformation superplasticity, due to *Greenwood and Johnson* [1965], agrees qualitatively with the experimental observations, and can quantitatively account for the observed differences between the I/II and I/III transformations. Plastic ratcheting strains were also observed during transformation cycling in the absence of applied deviatoric stress; both the sign and magnitude of these strains agree with theoretical expectations.

[40] Rather than strengthening ice during deformation by transformation superplasticity, the addition of inert equiaxed SiO_2 particles was observed to enhance the rate of ice deformation during pressure cycling, by about a factor of two. This doubling is in agreement with prior studies on titanium reinforced with 10 vol% TiC particulates subjected to thermal cycling through the transformation temperature of the matrix. An existing theoretical treatment is applied here to the case of ice/SiO_2 composites, and reduced to a more simplified form. The model indeed predicts a deformation enhancement due to the presence of inert particles, and is in reasonable quantitative agreement with the experimental results.

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