Inter-laboratory investigations of the effects of particulates on flow of fine-grained ice

D.L. Goldsby[1], W. B. Durham[2], A. V. Pathare[3], and L. A. Stern[4]

[1]Brown University (David_Goldsby@brown.edu), [2]Massachusetts Institute of Technology (wbdurham@mit.edu),
[3]Planetary Science Institute (avp@psi.edu), [4]U. S. Geological Survey, Menlo Park (lstern@usgs.gov)

Sample Fabrication and Creep Testing at MIT – Samples were prepared via the MIT method of mechanically mixing fine-grained ice and alumina dust in a mortar and pestle. In the MIT method, a 'standard ice' sample of grain size 180-250 μm is pressurized in the high-pressure gas apparatus into, then rapidly decompressed from, the ice-II field, with the cycle repeated 3 times. The samples are then disaggregated and ground fine and mixed with alumina powder by mortar and pestle. The resulting powder mixtures are then hot pressed in the gas apparatus and tested.

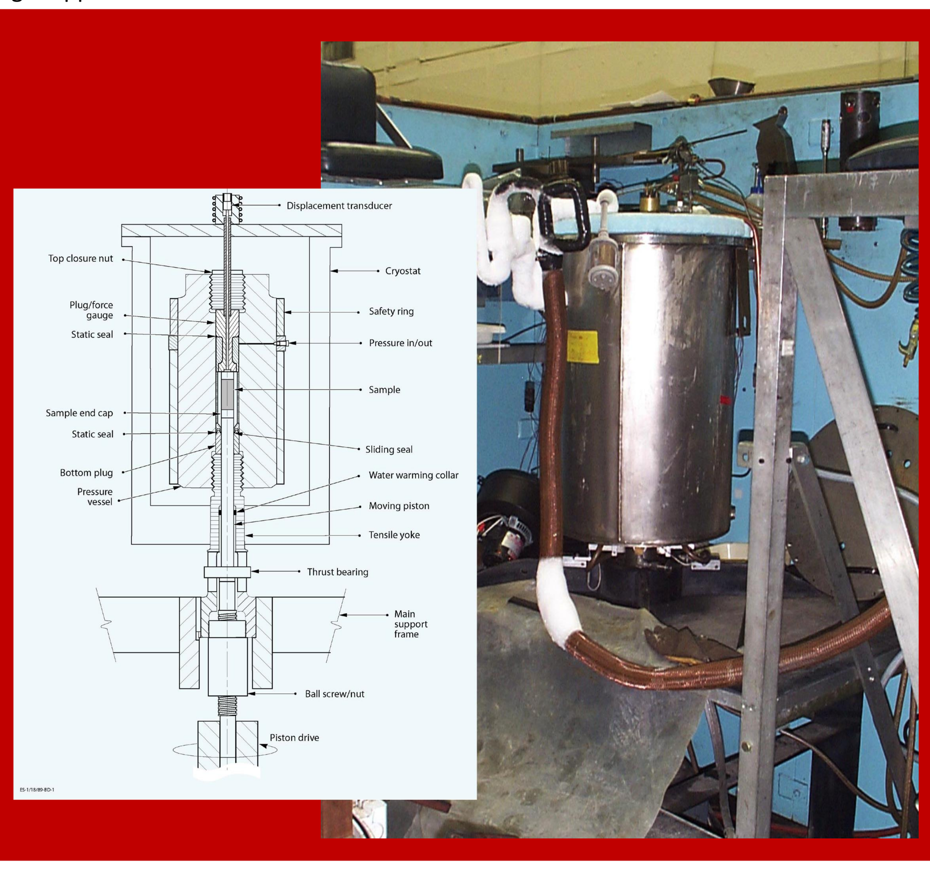


Fig. M1: Ice + dust samples are synthesized as solid cylinders approximately 20 mm in diameter x 60 mm in length, encapsulated in a jacket of indium and deformed axially under gas confining pressure in the above apparatus.

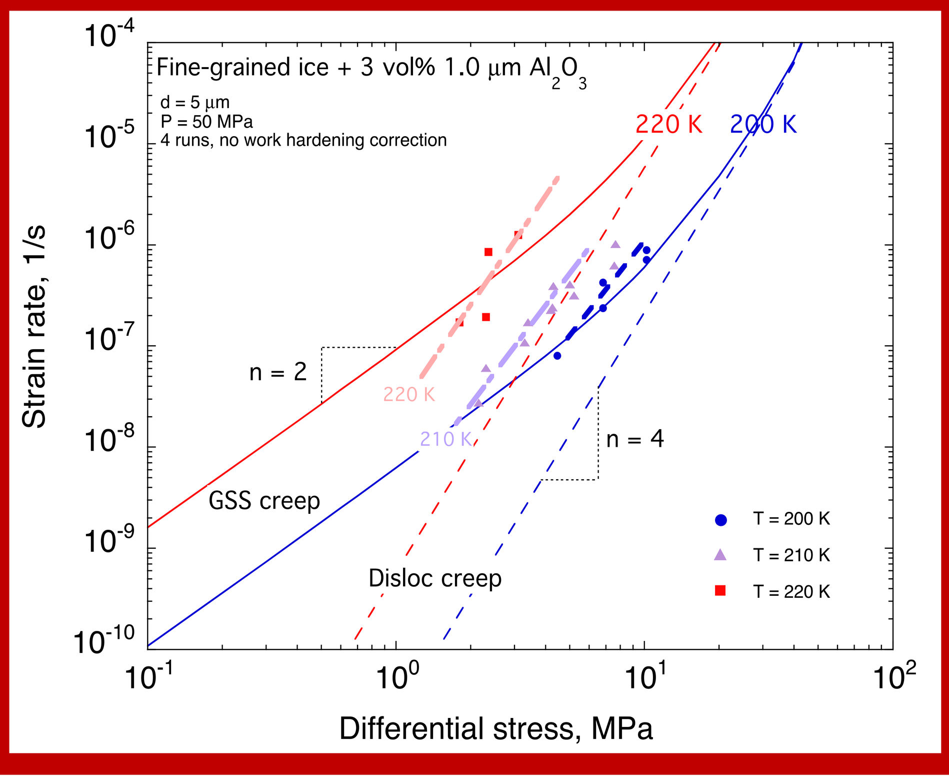


Fig. M2: Combined results from 4 runs [592 ,595, 599, 601] of nominally identical composition and preparation method. No correction made for strain hardening (see **Fig. M4**), which adds slightly to apparent scatter. Solid lines show the flow law for GBS-limited creep [4] at each T; dashed lines show the flow law for dislocation creep [5].

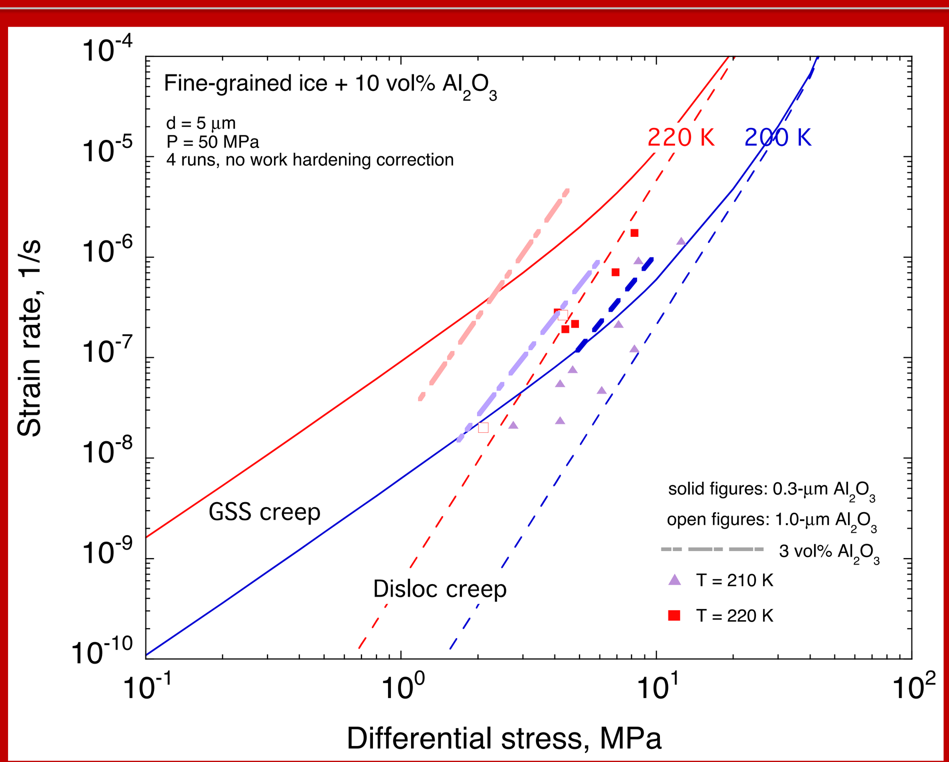


Fig. M3: Combined results from 4 additional runs [515,590,687,691] on samples of 10 vol% alumina of 0.3 and 1.0 μm grain size. Dash-dot lines from **Fig. M2** contrast the behavior of samples with 3% alumina of size 1 μm.

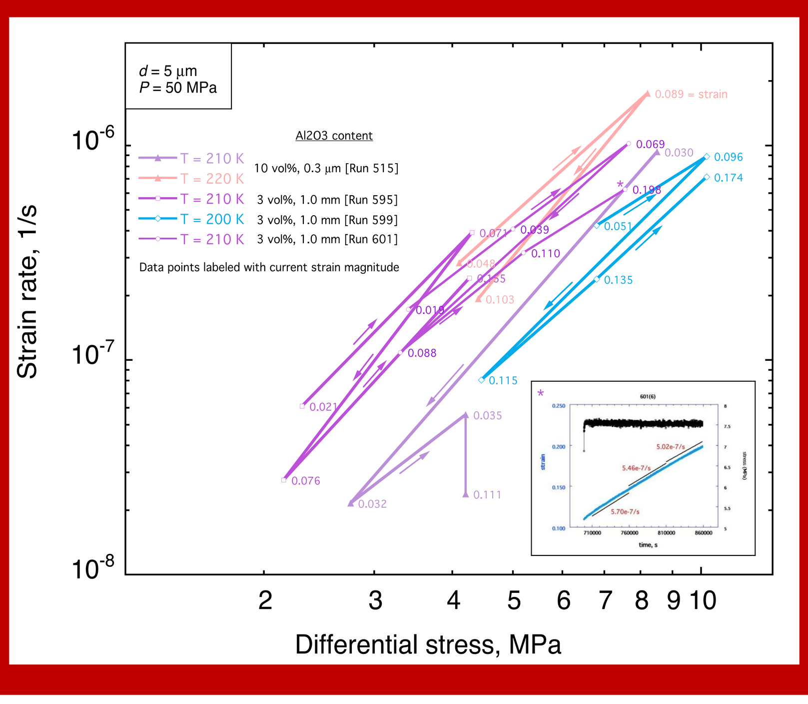


Fig. M4: Strain hardening (decreasing strain rate with increasing strain) is evident in the systematic downward and rightward stepping of data points in four runs where the testing sequence allowed such investigation. Data points are labeled by current strain; tie lines and arrows between data points indicate the test sequence for each run. Strain rate evolution with strain indicates that there is textural control of deformation and that texture is changing.

MOTIVATION

(A) Ground-penetrating Shallow Radar (SHARAD) sounding observations [1-3] indicate that Martian Polar Layered Deposits (PLD) and Lobate Debris Aprons (LDAs) are comprised of at least 90% pure water ice (e.g., Euripus Mons in **Fig. C1**).

(B) At Martian conditions, pure ice will undergo grain-size sensitive (GSS) creep characterized by a stress exponent of n≈2 stress exponent [4-5].

(C) But how will the presence of small quantities of dust (up to 10% by volume) affect the deformation of Martian PLD + LDA?

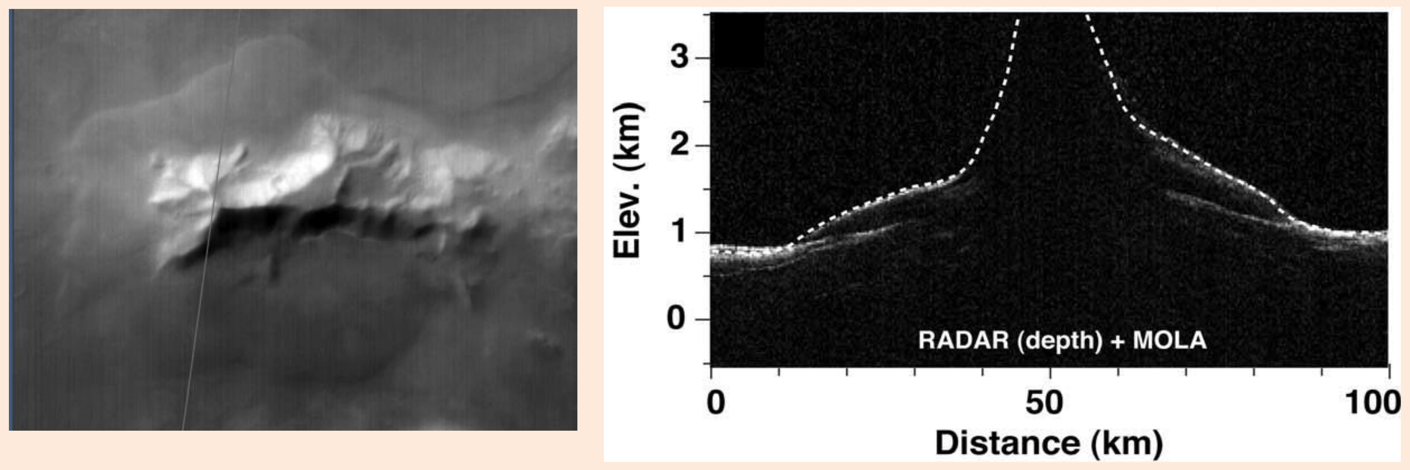


Fig. C1: MOC WA (Mars Orbiter Camera Wide Angle) image M0204416 of an LDA complex surrounding Euripus Mons massif in Eastern Hellas (45°S, 255°W), along with SHARAD radargram (taken from Holt *et al.* [2]).

RESULTS

(1) For small 3% dust content (**Fig. M2**), fine-grained ice can apparently exhibit high stress sensitivity (n=4) of grain-size independent (GSI) creep, but retains an overall strength closer to that of GSS creep.

(2) MIT tests on samples of 10% dust content (**Fig. M3**) indicate that flow of fine-grained ice appears to be indistinguishable from that of coarse ice undergoing dislocation creep, n=4.

(3) This finding is confirmed by Brown lab experiments on a 10% dust sample fabricated via the MIT method (red squares in **Fig. C2**):

(4) Texture matters: tests on stacked MIT+Brown samples (**Fig. C3**) show that the Brown sample deformed 3-4 times faster than the MIT sample, establishing the importance of texture. Prominent strain hardening (**Fig. M4**) is further evidence of the strong influence of texture on rheology.

(5) Threshold stress? (**Fig. B2**) The transition from n=2-2.4 to the threshold stress-like behavior for the DLG samples is consistent with a grain size ≤ 1 μm, not yet confirmed by cryo-SEM.

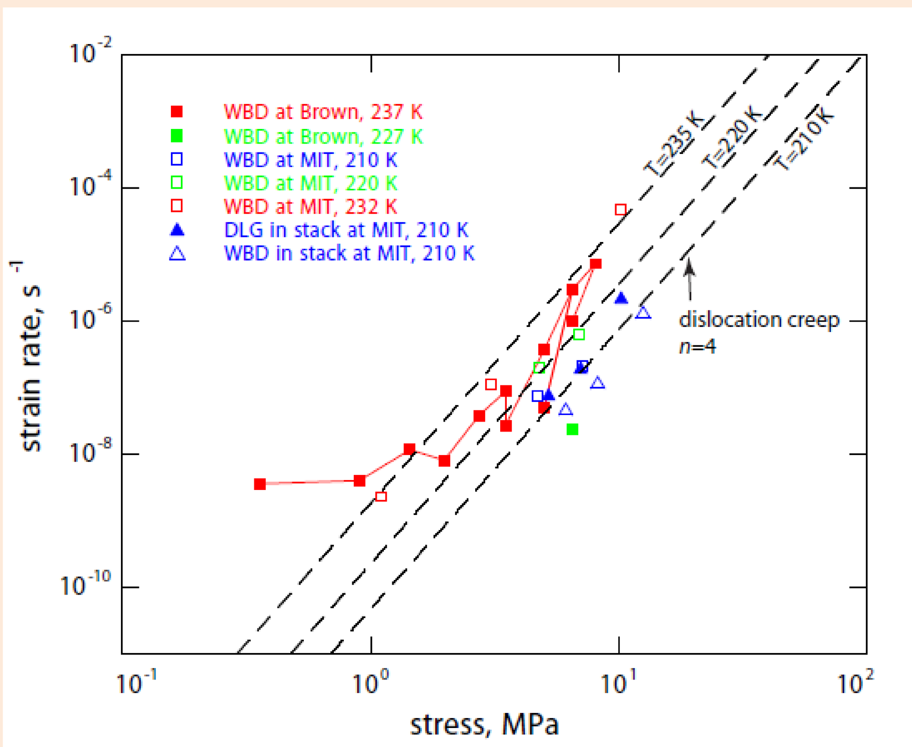


FIG C2: Plot of strain rate vs. differential stress (on logarithmic scales) for inter-lab experiments of various samples fabricated via MIT and Brown methodologies. The dislocation creep flow law at the indicated temperatures is plotted for comparison.

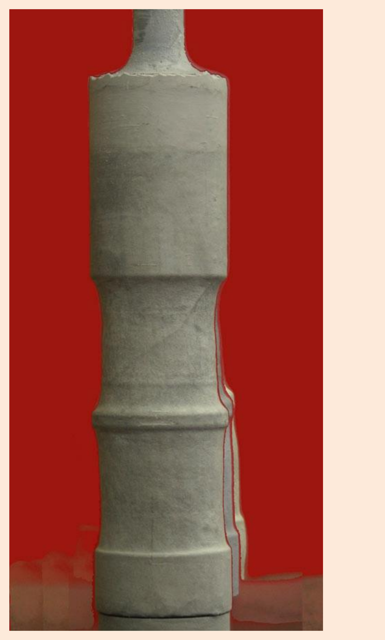


FIG C3: Overlay of three before-and-after photographs of the indium-jacketed sample stack for MIT Run 691, comprised of MIT-prepared above Brown-prepared material, the latter of which has clearly undergone more deformation despite having same dust content as upper MIT sample (see also triangles in **Fig. C2**)

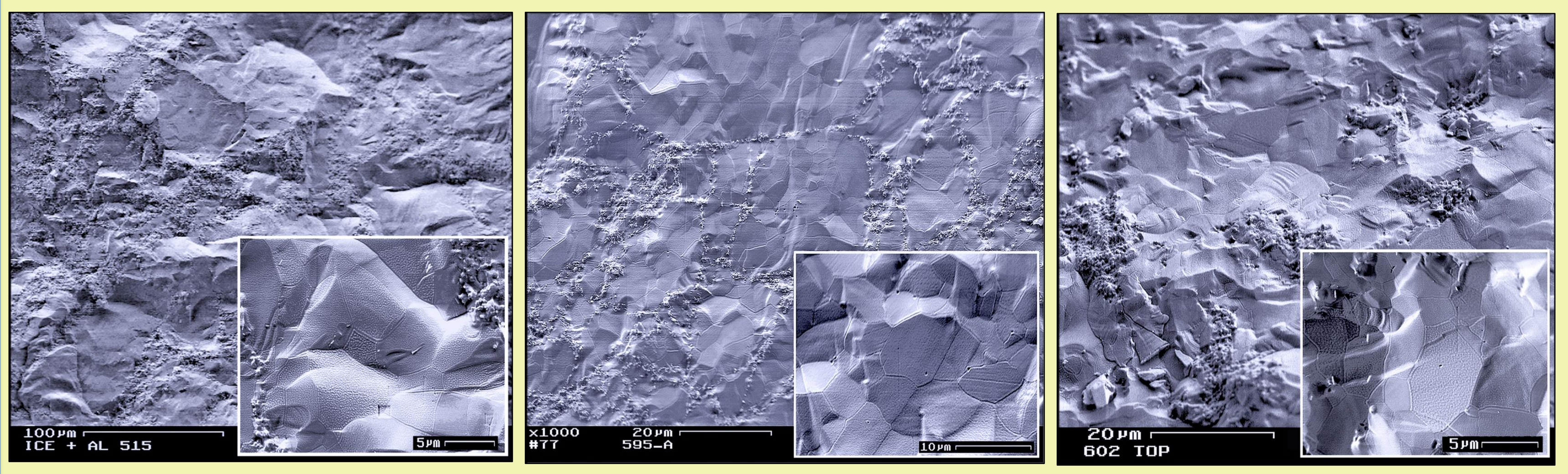
Fig. C4: Phase distribution and ice grain size as determined by cryo-SEM

Given the critical control that the textural arrangement of dust particles has over the rheological behavior of ice + dust, microstructural characterization of our material is essential to understanding the mechanisms of flow, and hence to the extrapolation of the behavior observed in these experiments to the Martian setting. Below are some examples of textural observations from CSEM studies conducted at the USGS of fine-grained ice + dust samples deformed at MIT.

Run 515 (left): 10 vol% 0.3-μm alumina dust (earliest run: predates "MIT method"). Lower magnification (background image) shows heterogenous mixing with clumps of alumina typically surrounding 20- to 100-μm-diameter "domains" of fine-grained ice (inset). Alumina appears as bright particles standing high in relief.

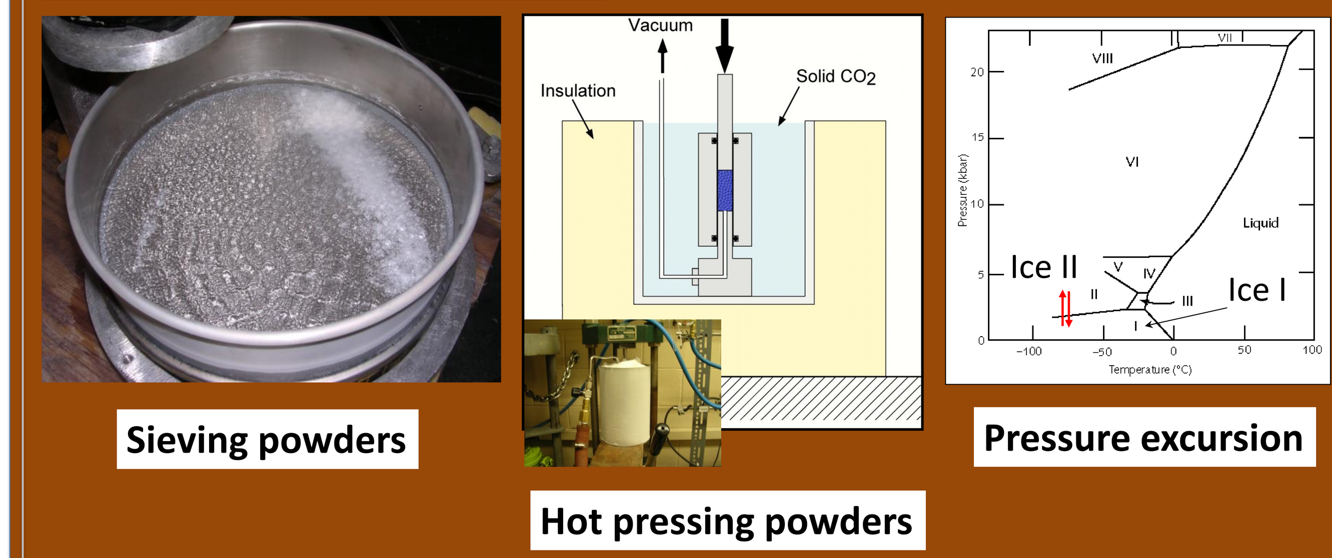
Run 595 (center): 3 vol% 1-μm alumina dust. Similar distribution as 515, but more homogeneous at the larger scale with less clumping of alumina and smaller "domains" of ice. But still little grain-scale dust structure; prominent configuration of dust is as coating around the domains of pure fine-grained ice (inset).

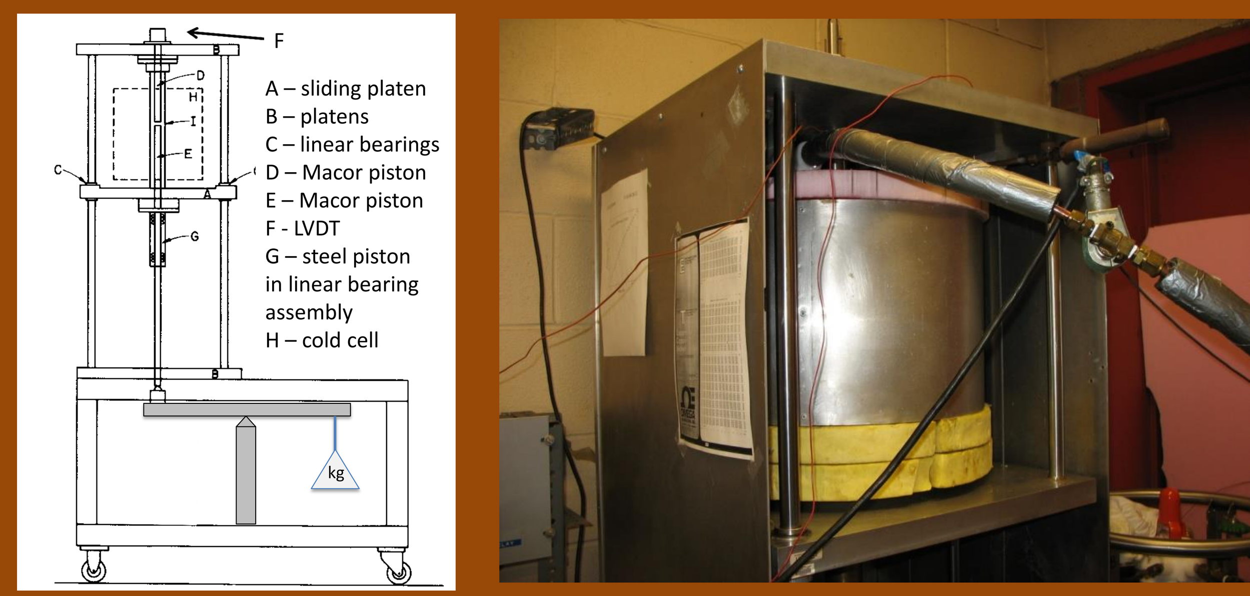
Run 602 (right): 3 vol% 1-μm alumina dust. Similar to 595 (with smaller domains of ice compared to 515) using the same synthesis method as 595.



Sample Fabrication and Creep Testing at Brown University – Ice samples were fabricated by the Brown method of hot pressing powders formed by spraying a fine mist of water and alumina into a reservoir of liquid nitrogen (LN) and sieving the powders in LN to a size <25 μm (see below), then subjecting the dense samples to ice I to II and ice II to I phase transformations. The texture as well as the ice grain size in the resulting samples has not yet been determined via cryo-SEM. Cylindrical samples ~1 cm in diameter by ~2 cm in length were then deformed at 1 atm in a dead-weight load, high-resolution creep apparatus, shown below.

Fig. B1 – **Experimental methods at Brown**





Schematic (LEFT) and photograph (RIGHT) of 1-atm, high-resolution creep rig in Brown Ice Lab.

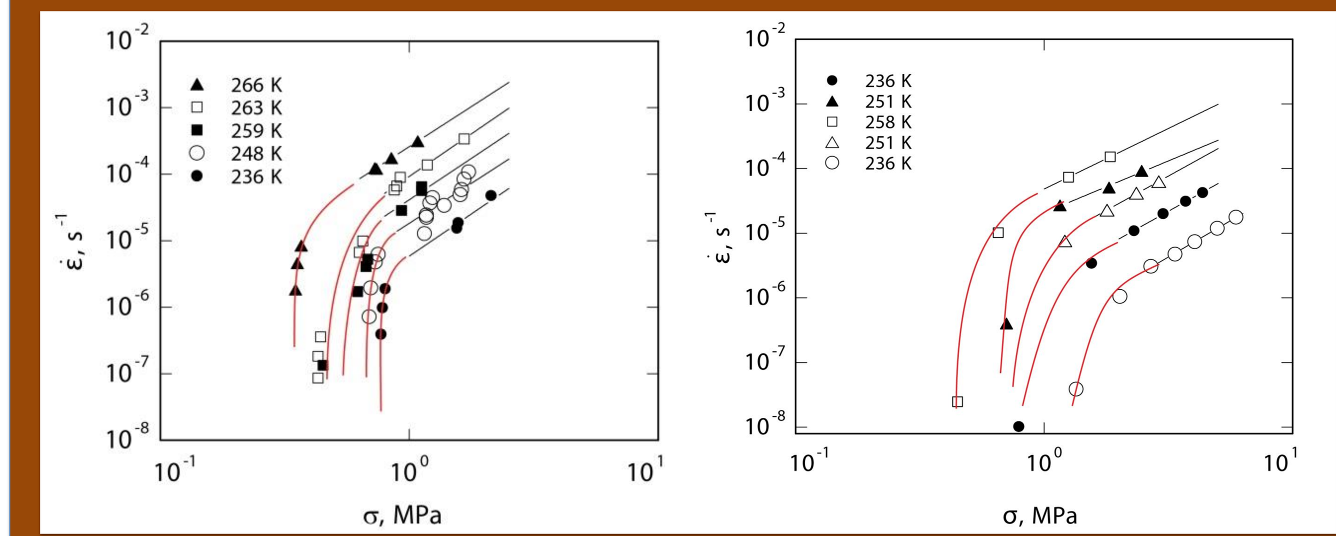


Fig. B2: Plots of strain rate vs. differential stress (on logarithmic scales) for (LEFT) a sample prepared by the Brown method and (RIGHT) a sample made by mechanically mixing powders and not subjecting the hot pressed mixture to the phase transformation. The data were acquired in the sequence (top to bottom) indicated in the legends. Note **threshold stress-like behavior** at lower stresses indicated schematically by the red lines in each figure. Data in the plot on the left yield a stress exponent $n \approx 2.4$ above a stress of ~1MPa. Data in the plot on the right yield n values of from 1.6 to 2.2 at higher stresses. **Strain hardening** is indicated on the plot on the right by the lower creep rates at a given stress for repeat tests at the same temperature.

CONCLUSIONS / DISCUSSION

Working hypothesis: Pure fine-grained ice at the conditions of these experiments (ice grain size, temperature, and stress) deforms by basal dislocation slip acting in concert with grain boundary sliding (GBS) [4]. In the mechanically mixed samples synthesized via the MIT method, dust particles are often located intergranularly (see **Fig. C4** micrographs), although ice-ice boundaries are also present. Smaller particle fractions harden the material (i.e., yield lower creep rates at a given stress) by slowing the rate of GBS, due, for example, to their impedance of dislocation motion in the ice. For larger particle fractions, the material hardens because GBS is slowed sufficiently that dislocation creep, an independent process with GBS creep, becomes the faster creep mechanism. The manner in which the Brown samples were synthesized suggests that these samples contain both intergranular and intragranular particles, both of which may impede dislocation motion in ice and hence GBS. This hypothesis, that particles slow the rate of GBS creep, is consistent with the following observations:

1. The apparent bulk stress sensitivity is n = 4 above an applied stress of ≈ 2 MPa (**Figs. C2,M2,M3**) and n ≈ 2 at lower stresses (**Fig. B2**).

2. At lower dust fractions (3%), the material is slightly stronger that pure fine-grained ice deforming via GBS creep, but still much weaker than large-grained ice deforming by dislocation creep (**Fig. M2**).

3. The higher the volume of dust particles, the closer the bulk rheological behavior (at stresses > 2 MPa) approaches that of dislocation creep (see data for ~10 vol% alumina, **Figs. C2, M3**).

4. The distribution of dust (i.e., the 'texture') affects the efficacy with which particles inhibit dislocation motion and slow GBS. Evolution of texture with strain causes strain hardening (**Fig. M4**), while differences in texture resulting from different sample preparation methods yield stark differences in strength for the same nominal particle size and fraction (**Fig. C3**).

5. Cryo-SEM analysis of samples (e.g., **Fig. C4**) will help elucidate the operative deformation mechanism(s) and the importance of texture in determining the rheological behavior.

6. *Implications:* Dislocation creep (n = 4) will often dominate the flow of dusty planetary ice masses for stresses > ~1 MPa and dust fractions ≥ ~10 vol.%. For lower stresses and/or particle fractions, GBS creep, with n ≈ 2, may dominate the creep rate. For natural dusty ice bodies of very fine grain size (microns), a threshold stress, below which intragranular dislocation slip may be severely limited, may yield extremely low strain rates at the lowest stresses, and perhaps a transition to diffusion creep.

References:

[1] Plaut J. et al., Subsurface radar sounding of the South Polar Layered Deposits of Mars, *Science*, 316, 92-95, 2007.
[2] Holt, J. et al., Radar sounding evidence for buried glaciers in the southern mid-latitudes of Mars, *Science* 322, 1232-1238, 2008.
[3] Plaut, J. et al., Radar evidence for ice in lobate debris aprons in the mid-northern latitudes of Mars, *GRL* 36, L02203, 2009
[4] Goldsby , D. and D. Kohlstedt, Superplastic deformation of ice. I. Experimental observations, *JGR*, 106, 11031-11042, 2001.
[5] Durham, W. et al., The rheology of ice I at low stress and elevated confining pressure, *JGR*, *106*, 11031-11042, 2001.