THE INFLUENCE OF PARTICLES ON THE RHEOLOGICAL BEHAVIOR OF ICE. D.L. Goldsby[1], C. Qi[1], T.A. Werts[1], L.A. Stern[2], W.B. Durham[3], and A. Pathare[4], [1]Earth and Environmental Sciences, University of Pennsylvania, Philadelphia, PA, 19104; [2]U.S. Geological Survey, Menlo Park, CA, 94025; [3]Earth, Atmospheric and Planetary Sciences, MIT, 02139; [4]Planetary Science Institute, Pasadena, CA

Introduction: The influence of embedded particles on the rheological behavior of polycrystalline ice is not well known. Although numerous experimental studies on 'dirty ice' have been conducted, these studies often yield inconsistent to contradictory results regarding the effect of particles on flow behavior [1]. This knowledge gap is compounded by the recent discovery that ice deforms not by a single deformation mechanism, as classically assumed, but rather by multiple creep mechanisms, each of which dominates the flow behavior of ice over different regimes of grain size, temperature and stress [2,3]. Given the different microphysics of the primary deformation mechanisms in ice - dislocation creep and grain boundary sliding (GBS) creep – the presence of particles will affect these mechanisms differently, depending on their size, fraction, and distribution within the ice. Intergranular particles may slow the rate of GBS creep, whereas intragranular particles may impede the motion of lattice dislocations, slowing both dislocation creep and GBS creep. To help fill this knowledge gap, we conducted experiments on particle-bearing ice samples and deformed them in both creep regimes.

Methods: Samples with ice grain sizes of ~10 µm containing intragranular graphite particles were fabricated from powders created by misting a water/graphite slurry into liquid N_2 using a pneumatic nozzle. Samples with ice grain sizes of ~10 µm containing intergranular particles were fabricated by mechanically mixing ice powders (created by misting pure water into liquid nitrogen) with graphite particles. In both cases, loose powders were packed into and sealed within cylindrical indium jackets and subsequently isostatically hot pressed into fully dense creep specimens at a confining pressure P=20 MPa and a temperature T=236 K in a high-pressure gas-medium apparatus. For the samples prepared by spraying ice + particles, particle sizes of 0.1 and 1 µm were used, in concentrations of up to 4.4 vol.%. Mechanically mixed samples were prepared for a particle size of 1 µm, in concentrations of up to ~7%.

After hot pressing, samples were removed from the high-pressure apparatus, measured, and placed back in the apparatus for mechanical testing. Samples were deformed at nominally constant strain rate in axial compression at P=20 MPa and T=236 K in the gas apparatus. Strain-rate steps were conducted on a given sample to determine the value of the stress exponent n, where $\dot{\varepsilon} \propto \sigma^n$, $\dot{\varepsilon}$ is strain rate, and σ is differential stress. After each rate step, deformation was continued until a roughly constant stress was obtained before stepping to a higher strain rate.

After deformation, samples were quenched in liquid nitrogen for long-term storage. A subset of the deformation samples were analyzed in a field emission gun scanning electron microscope (SEM) to determine the ice grain size and the location and fraction of particles present in the ice.

Results: For samples containing an intragranular dispersion of <1% of either 0.1-µm or 1-µm graphite, a value of n=1.6-1.8 is observed at lower stresses (<10 MPa) and a value of n=4 is observed at higher stresses. These values of n, and the magnitudes of the stress at a given strain rate, are consistent with GBS creep and dislocation creep of pure water ice. In Fig. 1, for example, data for two samples of ice containing 0.4 vol.% of 0.1-µm graphite particles are shown to be in

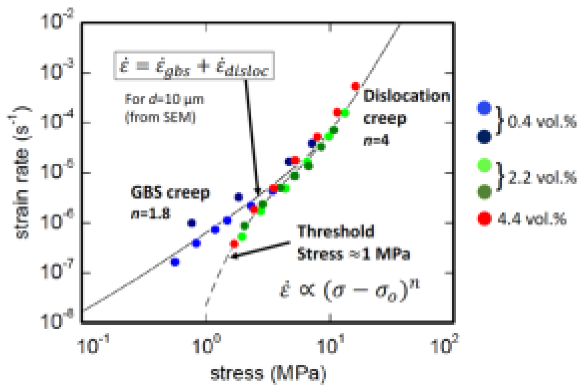

Figure 1 – Rheological data for samples with an ice grain size of 10 µm containing an intragranular dispersion of 0.1-µm graphite powders. For particle concentrations of 2.2 and 4.4 vol.%, the data deviate from the composite flow law for GBS creep and dislocation creep at low stresses, interpreted as a threshold stress for creep of ~1-2 MPa.

excellent agreement with the composite flow law for GBS creep and dislocation creep of Goldsby and Kohlstedt [2]. For samples containing 2.2 and 4.4 vol.% of 0.1-µm graphite, however, the data show a deviation from the predictions of the composite law at low stresses, which we attribute to the existence of a threshold stress for creep of ~1-2 MPa (see below).

For mechanically mixed samples of sufficiently high particle contents, however, the rheological data reveal dramatically different behavior compared to pure-water ice. For a sample containing intergranular particles 1 µm in size at a concentration of 6.6 vol.%, a value of the stress exponent of 4.6 is obtained, indicating dislocation creep as the operative deformation mechanism (see below).

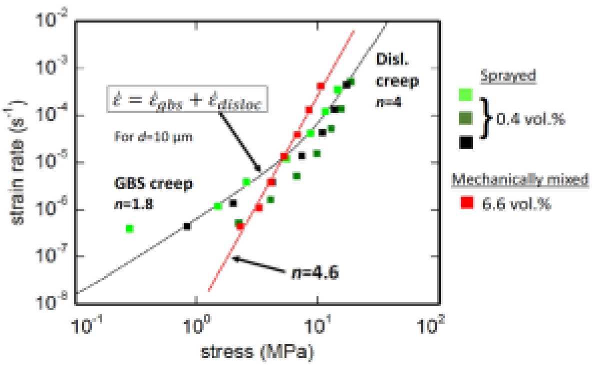

Figure 2 – Rheological data (red data points) for a sample with an ice grain size of 10 µm containing intergranular 1-µm graphite powders, compared with data (black and green data points) for samples of sprayed ice with 1-µm graphite in a concentration <1 vol.%. The red data appear to show dislocation creep behavior, with no indication of GBS creep at low stresses

Interpretation: The data from the experiments reveal two prominent features – 1) the appearance of a threshold stress for creep for samples that bear intragranular dispersions of fine graphite particles, and 2) the disappearance of GBS creep for samples containing a significant fraction of intergranular particles.

Threshold stress. Dislocation-accommodated GBS in ice involves the coupled processes of GBS and basal dislocation slip, so that the slowing of either process due to the presence of particles may affect the creep rate. The emergence of threshold stress-like behavior at low stresses in the GBS creep regime in our samples is thus consistent with the interaction of gliding basal dislocations with intragranular particles. The threshold stress can be approximated by calculating the stress needed to bow out a dislocation line between particles that pin the dislocation. This so-called Frank-Read stress is given by

$$\sigma_{F-R} = \frac{Gb}{l-r}$$

where G is the shear modulus, b is the Burgers vectors, l is the interparticle spacing in a regular array of particles, and r is the particle radius. For a particle fraction of 2.2 vol.%, G= 3 GPa, b=0.45 nm, an estimated particle spacing of 0.3 µm and a particle radius of 0.05 µm, we estimate a threshold stress of ~6 MPa, comparable to our observed value of 1-2 MPa.

Suppression of GBS creep. The suppression of GBS creep may occur if intergranular particles of sufficiently large size and in sufficiently high concentration can impede sliding and migration of grain boundaries. As shown in the SEM micrograph of Figure 3, the particles

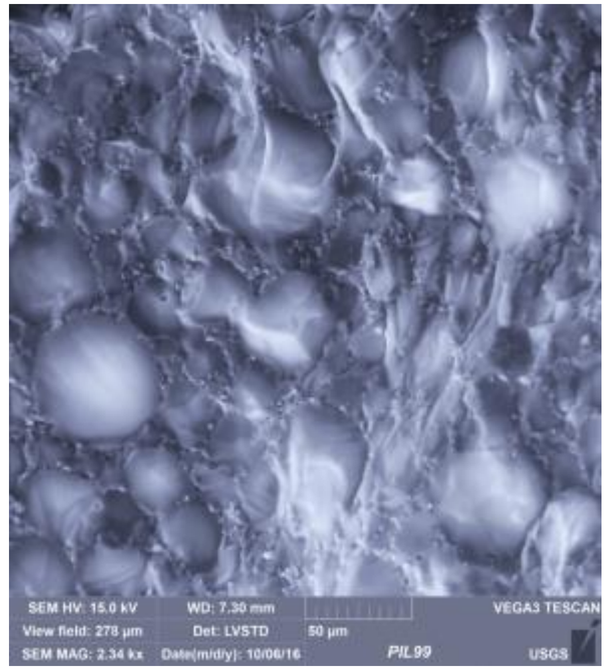

Figure 3 – SEM micrograph of a fractured surface from a deformed sample containing 6.6 vol.% of 1 µm graphite. Note that the graphite particles are segregated to the grain boundary regions between ice grains dislocation creep behavior, with no indication of GBS creep at low stresses.

in the sample containing 6.6 vol.% of 1 µm graphite are segregated to the grain boundary regions between the ice grains. In this configuration, the particles effectively 'lock-up' the microstructure against GBS, so that the creep rate is dominated by dislocation creep. Because the particles are located between the ice grains, they do not provide barriers to dislocation motion within the grains, and dislocation creep can proceed readily. The enhancement of the dislocation creep rate compared to expections from the dislocation creep flow law, as shown in Figure 2, are not yet well understood.

References:

[1] Moore, P.M. (2014), *Rev. Geophys., 52,* 435-467. [2] Goldsby, D.L. and Kohlstedt, D.L., *JGR, 106,* 11,017-11,030. [3] Stern, L.A., Durham, W.B., Kirby, S.H., *JGR, 102,* 5313-5325.