Microstructural Evolution of Polycrystalline Ice During Confined Creep Testing

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Abstract

The mechanical properties of polycrystalline ice I_h have been observed to change under an applied hydrostatic pressure comparable to that present near the bottom of kilometer-thick ice sheets. To help determine the cause of these changes, we conducted confined creep testing of laboratory-prepared polycrystalline ice at pressures up to 20 MPa (equivalent to ~ 2000 m of overburden) and subsequent microstructural analysis of specimens deformed by creep using optical microscopy and scanning electron microscopy, including extensive electron backscatter diffraction mapping of crystal orientations. Microstructural observations of the creep-deformed specimens revealed smaller median grain sizes, less regular, and more interlocked grain shapes in specimens deformed at higher pressure compared with those deformed at atmospheric pressure. Variable pressure testing reveals little change in strain rate for pressures less than 15 MPa, leading to alternative hypotheses regarding the influence of confining pressure on the dislocation dynamics and associated creep behavior of polycrystalline ice. Our central hypothesis is that widely dissociated basal dislocations in ice begin

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to constrict after the confining pressure reaches a critical value. This critical pressure depends strongly on the (currently unknown) lattice dilatation induced in ice by the presence of stacking faults.

Keywords: creep, dislocation, electron backscatter diffraction, grain boundary, stacking fault, polycrystalline ice

1. Introduction

The effects of hydrostatic pressure on the creep properties of ice I_h are of interest for terrestrial ice sheet flow modeling and to gain fundamental insight into the structure of ice and its crystalline defects. Presently, ice
 5 sheets on Earth have maximum depths of ~ 3000 m, yielding hydrostatic pressures P up to 30 MPa, and depressions of the melting temperature T_m down to -2.5°C using the rate of $-0.074^\circ\text{C MPa}^{-1}$ given in Hobbs (1974). The majority of laboratory experimental work has been performed within this range, both on single crystal and polycrystalline ice, and the
 10 work presented in this paper also focuses exclusively on the temperature and pressure range relevant to terrestrial ice. Following a review of the possible effects of hydrostatic pressure on creep and fabric development in ice, we review the experimental results to date to place this work in context.

1.1. Unique characteristics of ice I_h and hydrostatic pressure effects on creep

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Steady state creep strain rate as a function of hydrostatic pressure is often described (Frost and Ashby, 1982) by

$$\dot{\epsilon} = A\sigma^n \exp\left[-\frac{E^* + PV^*}{k_B T}\right] \quad (1)$$

where A is a constant, σ is the applied non-hydrostatic stress, n is the creep exponent, E^* is the creep activation energy, V^* is the creep activation volume, k_B is Boltzmann's constant and T is the absolute temperature. We emphasize that this expression uses the absolute temperature, and not the homologous temperature $T_h = T/T_m(P)$ where T_m is the melting temperature, which is a function of hydrostatic pressure. We will frequently refer to both types of temperatures in this paper. Comparisons of strain rate to ascertain pressure effects using Eq. 1 should be done at equal absolute temperatures T , as this expression does not include any provision for pressure melting effects.

Because most crystalline materials exhibit $V^* > 0$, application of high pressure results in reduced rates of creep (Butcher and Ruoff, 1961; Sherby et al., 1970), slower grain growth (Hahn and Gleiter, 1979) and lower rates of recrystallization (Tanner and Radcliffe, 1962; Syrenko et al., 1973). In contrast, ice displays *negative* activation volumes for grain growth (Azuma and Higashi, 1983) and for creep at $P > 15$ MPa (Jones and Chew, 1983b). The dominant point defect in ice is the interstitial (Hondoh et al., 1987) which increases in concentration with increasing P . Furthermore, ice has one of the lowest known stacking fault energies (γ_F), a result of its open lattice and the very small energy difference between hexagonal and cubic ice structures (Hondoh, 2000). Keeping these unique characteristics in mind, we now examine the possible effects of pressure on creep and the development of fabric and texture in polycrystalline ice.

Following Poirier (1985), the possible effects of increased hydrostatic pressure on creep and fabric development include:

1. Resistance to dislocation formation and multiplication can occur with increasing P , as the presence of a dislocation represents a less perfect and therefore slightly higher volume crystal compared to similar defect-free crystal (Poirier, 1985). In some polycrystals with very large *elastic* anisotropy, dislocations and associated transient plastic flow can be generated under application of high P (Margevicius and Lewandowski, 1991) but this type of dislocation generation is negligible for ice as it is nearly elastically isotropic (Schulson and Duval, 2009). Margevicius and Lewandowski characterized the relative isotropy of hexagonal materials via the ratio of c -axis to a -axis linear compressibilities, k_c/k_a . Using the compliance data of Gammon et al. (1983) for ice, we find k_c/k_a for ice is 1.14, compared with a values of 1.0 for a perfect elastically isotropic material and 8.7 for the strongly elastically anisotropic material cadmium.
2. Changes in dislocation production rate from Frank-Read sources can occur if the shear modulus G of the material changes with P . These sources have been observed to operate in single crystal ice (Ahmad et al., 1986), but are unimportant compared to the production of dislocations at grain boundaries (Liu et al., 1992, 1993). This effect is also likely negligible as G decreases by less than 0.2% from $P = 0.1$ to 20 MPa (Helgerud et al., 2009), yet another unusual characteristic of ice compared to most materials whose shear moduli typically increase with P .
3. Strengths of dislocation-dislocation and dislocation-obstacle interactions are also expected to increase linearly with G (Hirth and Lothe,

1982). Given the negligible change in G with P for ice as discussed above, little change in the dislocation interaction strength is expected in ice.

4. The pressure dependence of the Peierls stress, or lattice friction, can also act to hinder the motion of dislocations under high pressure (Poirier, 1985). Peierls stress variation with P has not been quantified for ice, though it seems that the essentially null result of Cole (1996) for basal slip and the noted absence of lattice friction for non-basal dislocations (Petrenko and Whitworth, 1999) suggests that this mechanism is unimportant for ice, though other opinions exist (Louchet, 2004). We note here that lattice friction is *not* the same as dislocation drag associated with proton disorder effects discussed below.

5. Dislocations in materials with low stacking fault energies often split into two partial dislocations bounding a ribbon of stacking fault between them (Hirth and Lothe, 1982). The width of these dissociated (or extended) dislocations can be described by $w \propto G/(\gamma_F + \alpha P)$ where α is a constant proportional to the lattice dilatation associated with the presence of the stacking fault (Fontaine and Haasen, 1969; Poirier, 1985). Increasing P will act to constrict dissociated dislocations and enhance the rate of recovery through dislocation climb and cross-slip (Weertman, 1965; Aladag et al., 1970; Nabarro, 2006). Given the extraordinarily low γ_F of ice, we expect that most dislocations are very widely extended on the basal plane (Tyson, 1971; Landgon, 1973; Hondoh, 2000). The pressure sensitivity for the width of these dissociated dislocations depends strongly on the lattice dilation associated with

stacking faults.

6. Dislocation motion in ice is thought by some to be controlled in some
95 way by proton disorder (Glen, 1968), though contrary theories have
been developed (Louchet, 2004) which point to lattice friction as the
rate limiting mechanism for basal dislocation motion. Experimental
work by Chan et al. (1965) on polycrystalline ice and Taubenberger
et al. (1973) on single crystal ice both demonstrate that proton re-
100 laxation times increase with increasing P . This effect is expected to
be quite small over the pressure range of our study, with a change
in proton relaxation time of less than 5% for a pressure change of
20 MPa. In thermodynamic regions where proton-disorder control of
basal dislocation glide is a likely rate-limiting mechanism, dislocation
105 motion and therefore strain rate may decrease slightly with increasing
pressure. Petrenko and Whitworth (1999) suggest that the barrier to
motion presented by the proton disorder mechanism should be much
higher for reconstructed dislocations than for partials.
7. Dislocation climb requires a supply of point defects. In the case of
110 ice at under pressure, the concentration of self-interstitials is slightly
higher (Hondoh et al., 1987; Hondoh, 2000) than at atmospheric pres-
sure (2.9 ppm at $P=20$ MPa vs 2.78 ppm at 0.1 MPa). In thermo-
dynamic regions where self-interstitial driven dislocation climb is the
rate-limiting process, we might expect a modest increase in recovery
115 and strain rate with increasing P .

We note that the above effects can be expected to have conflicting impacts

on both the strain rate and microstructural development of polycrystalline ice.

1.2. Review of Single Crystal Experiments

120 Studies of hydrostatic pressure effects on the dominant $(0001)\langle 11\bar{2}0 \rangle$ basal slip system in single ice crystals have found two very different behaviors. Rigsby (1958) found no pressure effect on basal creep rate at constant *homologous* temperature.

125 Cole (1996) found no pressure effect on the steady state creep rate at constant *absolute* temperature. These results, though somewhat contradictory, both emphasize that the pressure effect on the basal slip system, if it exists, is small within the P range relevant to ice on Earth.

1.3. Review of Polycrystal Experiments

Work to date on polycrystalline ice has shown a measurable effect on
130 creep strain rates due to elevated hydrostatic pressure. Early testing was performed by Haefeli et al. (1968) who compared creep strain rates both at constant absolute and constant homologous temperatures at pressures of 0.1 and 29.5 MPa. In contrast to single crystal behavior, they found that elevated hydrostatic pressure increased the creep rate at a fixed absolute
135 temperature, though conducting the test at the equivalent homologous temperature and atmospheric pressure increased it still more. No microstructural analysis of the creep-deformed specimens was reported and applied axial stresses were $< 0.1\text{MPa}$.

Jones and Chew (1983a) tested polycrystalline ice at a variety of pres-
140 sures from 0.1 up to 50 MPa, an applied axial stress of 0.47 MPa and a

constant absolute temperature of -9.6°C . Significantly, they found that secondary creep rates slowed with increasing P , passed through a minimum at ~ 15 MPa hydrostatic pressure, and then increased up to the maximum test condition of $P=60$ MPa. This result implies that the activation volume
145 for creep either *changes sign* or *changes from zero to a large negative value* at ~ 15 MPa. The creep rate increase at high pressure was attributed to the proximity of the system to the pressure melting point and therefore enhanced sliding along wetted grain boundaries. No microstructural analysis of the creep-deformed specimens was reported.

Durham et al. (1983) performed constant strain rate tests on polycrystalline ice specimens with the goal of characterizing the rheology of water ice in the outer solar system. A high pressure in this context is ~ 300 MPa, whereas a high pressure for natural ice on earth is ~ 30 MPa. The temperature and pressure ranges covered in Durham et al. (1983) are generally much
155 more extreme than those on Earth, though their data and the data of Jones and Chew (1983a) do overlap near $P=50$ MPa. In the region of overlap, the two groups have found roughly similar magnitudes but opposite signs for the activation volume V^* . Near $P=50$ MPa, Jones and Chew (1983a) found that creep rate increased with pressure (i.e. $V^* < 0$). Durham et al. (1983)
160 found that between 0.1 and 50 MPa pressure, increased pressure led to a slight hardening of the ice (i.e. $V^* > 0$). The discrepancy between these results may be related to the pre-test microfracturing issues detailed in the Discussion section of Durham et al. (1983).

Tests performed at confining pressures up to 31.5 MPa by Mizuno (1992)
165 found that the effects of P on secondary creep rate were limited to tem-

peratures above a critical temperature of -6°C . Applied axial stresses were high and ranged from 1 to 3 MPa. Analysis of optical thin sections taken from the creep-deformed specimens showed that, for specimens deformed above this critical temperature, rapid and extensive recrystallization and grain growth rendered the microstructure (in tertiary creep) independent of strain, while those deformed below this temperature featured microstructures which evolved more gradually with increasing strain. The resulting c -axis fabrics were girdle patterns centered on the compression direction; no fabric differences between specimens deformed at atmospheric and elevated hydrostatic pressures were reported.

1.4. *Motivation*

The varied and often conflicting results discussed above underscore the experimental difficulties associated with confined creep testing. However, taking these results together with the list of possible P effects in Section 1.1, we can surmise that

1. The observed changes of creep rate and activation volume with P in polycrystalline ice is not due to a fundamental change in the basal slip system dynamics.
2. The enhanced creep rates are derived from the presence of grain boundaries or stress heterogeneities (or both) found in polycrystals, and not in single crystals.
3. Microstructural analysis of the creep-deformed specimens, which was largely lacking in earlier work, may aid in separating the effects of temperature and hydrostatic pressure on creep deformation of ice.

190 In this paper we seek to (1) document the microstructural and mechanical differences between specimens deformed at $P=0.1$ and $P=20$ MPa, (2) discuss possible mechanisms for the observed microstructural differences and (3) argue that the observed effects of hydrostatic pressure on the creep of polycrystalline ice are not solely due to changes in the homologous temperature, but instead reflect pressure-induced changes in dislocation and grain
195 boundary dynamics.

We offer two possible hypotheses to explain the findings in the existing literature and the new results presented in this paper. Hypothesis (1): Increased strain rates are caused by the pressure-induced constriction of dis-
200 sociated basal dislocations at $P \geq$ some critical pressure P_c , Hypothesis (2): Basal dislocation drag is the rate controlling process at low pressures while climb, fueled by (but unlikely caused by) higher equilibrium self-interstitial concentrations, becomes rate controlling at high pressures.

Both hypotheses seek to explain the shift from positive (or zero) to neg-
205 ative V^* with increasing pressure via pressure effects on dislocation dynamics as an alternative to the pressure-melting hypothesis posed by Jones and Chew (1983a). At this point, the results of this on-going work are not definitive, but the above hypotheses are explored in an effort to stimulate continued interest in this important topic.

210 2. Methods

2.1. Test Specimens

We generated test specimens of polycrystalline ice with random crystal orientations using the methods of Cole (1979). The cylindrical ice specimens

were 34 mm diameter by 86 mm tall with 25 mm tall corrugated phenolic
215 endcaps frozen directly to the specimen ends during the fabrication process.
The endcaps ensure proper parallelism of the specimen ends and correct
axial alignment of the specimen within the uniaxial creep testing system.
Specimens were stored at -30°C for storage both before and after mechanical
testing to minimize microstructural changes while in storage.

220 2.2. *Confined Creep Testing*

The uniaxial creep testing system applies a constant load to the test
specimens via modular lead weights and machined steel plates at the in-
terface between ice and lead to provide a smooth and axially true loading
surface. The weights are constrained to move vertically by polished steel
225 guide rails and linear bearings. Two specimens can be tested simultane-
ously by building a stack from the bottom up, shown in Figure 1.

The load applied to the lower specimen is then the sum of the upper
and lower weights, while the upper specimen experiences the load from the
upper weights only. The modular weights allowed adjustment of the applied
230 stresses from 0.1 to 0.63 MPa; higher stresses are not possible given the
limited volume within the pressure vessel.

Creep displacement was measured (2.5 microstrain resolution) separately
on each specimen by vented, ± 6 mm range LVDTs (Macrosensors, Pennsauken,
NJ) mounted rigidly to the specimen endcaps. Specimen temperature was
235 monitored using a thermistor string (Thermometrics, Northridge, CA) ac-
curate to $\pm 0.01^\circ\text{C}$. System hydrostatic pressure was measured (690 Pa res-
olution) by a pressure transducer (Dynisco, Franklin, MA) and all voltages

Figure 1: Photograph of uniaxial creep testing system ready for installation into the pressure vessel; double specimen setup is shown here. A - upper weight stack, B - linear bearing, C - guide rail, D - upper specimen LVDT, E - thermistor, F - upper ice specimen, G - lower weight stack, H - lower specimen LVDT, I - lower ice specimen.

and temperatures were recorded at 60 sec intervals on a dedicated data acquisition computer.

240 Once set up, the creep testing system is placed into the pressure vessel, the vessel filled with Dow Corning 550 silicone oil, sealed, and pressurized using a hand pump. Pressurization/depressurization rates are limited to 150 kPa min⁻¹ to minimize adiabatic heating/cooling (typically less than 1°C) of the specimen. Pressure control (stable to within 1%) is achieved via a 3.8 L
245 hydraulic accumulator precharged to 15 MPa with N₂ gas, which supplies a sufficient volume of makeup oil for multi-month tests and minimizes pressure variations associated with temperature changes within the cold room.

The pressure vessel is then placed into a 200 L barrel of ethylene glycol which is maintained at a steady temperature ($\pm 0.5^\circ\text{C}$) by an Endocal
250 (Thermo-Neslab, Newington, NH) RTE-4 refrigerated circulator. The thermal mass of the pressure vessel itself typically limits specimen temperature variations to $\pm 0.1^\circ\text{C}$. The final testing conditions (i.e. steady T and P) are generally achieved within 4–6 h of first applying load to the specimens.

2.3. Specimen Microstructure Imaging

255 Specimen-scale images of the specimen microstructure for grain size and shape were recorded using thin sections digitally photographed in transmitted light between crossed polarizers. The resulting images (typical resolution 17 $\mu\text{m pixel}^{-1}$) were analyzed using the freely available FIJI (Schindelin et al., 2012) image processing software. Grain sizes were determined for each
260 grain by calculating the circular equivalent diameter, and we quantified the grain shapes by measuring circularity and solidity (Olson, 2011).

Smaller scale images were obtained on uncoated 1 cm by 3 cm specimens via SEM micrographs recorded on a FEI (FEI Inc., Hillsboro, OR) XL-30 environmental scanning electron microscope equipped with a Gatan (Gatan Inc., Pleasanton, CA) C1002 cryostage. Images were typically recorded under 0.2 Pa of N₂ gas, with a cryostage temperature of -160°C, using a 15 kV accelerating voltage and a large field gaseous secondary electron detector. Both the low temperature and slight N₂ gas pressure act to reduce specimen sublimation as discussed in Weikusat et al. (2011), thus preserving the specimen surface for several hours.

2.4. Electron Backscatter Diffraction Mapping

Electron Backscatter Diffraction (EBSD) determines the full a and c axis crystallographic orientation of ice grains and subgrains with an angular resolution $< 1^\circ$ and a spatial resolution down to $0.5\mu\text{m}$. The electron beam of the SEM is used as the source and the resulting backscattered diffraction pattern (EBSP) is recorded on a phosphor screen, imaged by a video camera. We used an Oxford-HKL Technology EBSD system to automate the beam scanning, stage motion, diffraction pattern recording and analysis, as has been done in several earlier studies (see Iliescu et al. (2004) and Obbard et al. (2006)).

The diffraction itself takes place in a few nm closest to the specimen surface, so specimen surface preparation and maintenance is critical. We microtomed the analysis surface and allowed further smoothing via sublimation in a closed container at -25°C for 6-12 h prior to analysis. However, a delay of more than 48 h under these sublimation conditions caused suffi-

cient surface roughening to negatively affect the EBSD analysis. This procedure was followed for all specimens, so we cannot say that microstructural changes did not occur between removing the specimen from the confined creep apparatus and time of analysis in the SEM.

290 Ice grains are quite large ($\sim 1\text{mm}$ or larger) compared to many other materials, requiring the use of low magnifications (typically 40X) and stitching together contiguous EBSD maps to fully cover the 3 cm^2 area of the SEM specimens. Large scale EBSD mapping was performed using an electron beam current of 20nA, enabling the collection of 3 orientation data points
295 per second with a $\sim 75\%$ successful EBSD indexing rate. Typically an 85 or $100\text{ }\mu\text{m}$ step size was used to generate large scale maps of useful resolution while still covering specimen area at a sufficient rate to ensure good surface quality throughout the analysis; approximately 1.25 h was required to analyze 3 cm^2 under these conditions. Extensive guidance for successful EBSD
300 on ice can be found in Prior et al. (2015).

The resulting orientation datasets were post-processed using both HKL Channel 5 software, as well as the open source MTEX toolbox (see Bachmann et al. (2011) and Bachmann et al. (2010)) to fill in non-indexed locations, determine grain boundaries and calculate average grain orientations
305 and Schmid factors (Azuma, 1995; Trickett et al., 2000).

3. Results

A total of ten specimens were tested under various conditions shown in Table 1. All were constant pressure tests except for S31MAY13, which was a variable pressure test.

Table 1: Listing of specimens and mechanical testing conditions. The date in the specimen designation indicates the date of fabrication. Temperature T is shown $\pm$ one standard deviation. (a) S27FEB13 and S01MAR13 were tested together in the double specimen setup. (b) S29MAR13 and S27MAR13 were tested together in the double specimen setup. (c) Variable pressure test conducted in secondary creep.

Specimen	P (MPa)	σ (MPa)	T ($^{\circ}\text{C}$)	T_h	$\dot{\epsilon}_{\min}$ (s^{-1})	Final ϵ	Duration (h)
S22OCT12	20.0	0.63	-5.06 ± 0.07	98.68%	1.7×10^{-8}	8.86%	622
S20NOV12	20.0	0.63	-5.04 ± 0.09	98.69%	1.8×10^{-8}	8.83%	554
S11DEC12	0.1	0.63	-4.94 ± 0.04	98.19%	1.4×10^{-8}	9.23%	760
S11JAN13	0.1	0.63	-3.60 ± 0.10	98.68%	3.5×10^{-8}	9.41%	388
S01FEB13	0.1	0.63	-2.48 ± 0.06	99.09%	1.0×10^{-7}	8.81%	304
S27FEB13 ^a	20.0	0.33	-4.74 ± 0.32	98.80%	5.0×10^{-9}	0.96%	335
S01MAR13 ^a	20.0	0.50	-4.74 ± 0.32	98.80%	2.4×10^{-8}	6.14%	335
S29MAR13 ^b	20.0	0.33	-9.67 ± 0.07	96.98%	1.5×10^{-9}	1.31%	1390
S27MAR13 ^b	20.0	0.50	-9.67 ± 0.07	96.98%	4.4×10^{-9}	6.18%	1390
S31MAY13 ^c	0.1 to 15	0.50	-4.80 ± 0.47	variable	1.2×10^{-8}	1.23%	180

310 *3.1. Mechanical Testing*

The mechanical testing showed that both secondary and tertiary strain rates were enhanced at $P=20$ MPa when compared to the same tests conducted at atmospheric pressure ($P=0.1$ MPa). Strain rate minima occurred at similar strains (roughly 1%) for both $P=20$ and 0.1 MPa which is interesting because the hydrostatic pressure induced a non-trivial shortening of the specimen (estimated at 0.09%) that apparently contributed to the transition from primary to secondary creep. Over half of this shortening can be attributed to elastic strain in the specimen and endcaps associated with the application of confining pressure. The remainder could potentially be associated with the collapse of very small bubbles in the specimen, yielding an upper bound on specimen porosity at roughly 0.0003.

Our constant pressure test results plotted in Figure 2 show that for equal absolute temperatures, a specimen deformed at $P=20$ MPa (e.g. S20NOV12) deforms faster than one deformed at $P=0.1$ MPa (S11DEC12). However, a specimen deformed at atmospheric pressure and at the equivalent homologous temperature (S11JAN12) deforms faster than the pressurized specimen (S20NOV12). These results are consistent with the findings (see Section 1.3) of Haefeli et al. (1968).

In testing any polycrystalline specimen, a certain amount of specimen-to-specimen variability is inevitable due to random differences in grain sizes, crystallographic orientations and initial dislocation densities that occur during specimen fabrication. Because of the long deformation times involved in creep testing, we have only two specimens, S22OCT12 and S20NOV12, that were deformed under the same P , T , and σ conditions, resulting in a

Figure 2: Strain rates vs. strain for specimens deformed at the same absolute temperature (S20NOV12 and S11DEC12) and for specimens deformed at the same homologous temperature of $T_h=98.7\%$ (S20NOV12 and S11JAN13). Specimen S01MAR13 was deformed with $\sigma=0.50$ MPa, while all other specimens were deformed with $\sigma=0.63$ MPa.

335 difference of $1 \times 10^{-9} \text{ s}^{-1}$ in the minimum strain rate. This value is in reasonable agreement with the $3 \times 10^{-9} \text{ s}^{-1}$ specimen-to-specimen variability observed between six specimens deformed at atmospheric pressure in Jones and Chew (1983a). Assuming that $3 \times 10^{-9} \text{ s}^{-1}$ is a reasonable estimate, the differences in strain rate attributed to pressure and temperature effects
 340 in our study are roughly 6-10 times larger than the specimen-to-specimen variability, and thus are readily distinguishable from it. Ideally, numerous repeat creep experiments would be run to better characterize this variability, but practical constraints on testing time prevent this.

Table 2: Variable pressure test schedule for S31MAY13 with $\sigma = 0.50$ MPa. At least 24 h elapsed between each pressure change to allow stabilization of temperature and strain rate. Temperature variations of 0.5°C were typical.

P (MPa)	T ($^\circ\text{C}$)	T_h (%)	ϵ	$\dot{\epsilon}$ (s^{-1})
0.1	-5.05	98.17	0.33%	1.6×10^{-8}
5.0	-4.95	98.30	0.53%	1.7×10^{-8}
10.0	-4.88	98.44	0.71%	1.4×10^{-8}
15.0	-4.85	98.57	0.85%	1.4×10^{-8}
0.1	-5.10	98.17	1.23%	1.3×10^{-8}

The variable pressure test (S31MAY13) was designed to reproduce that
of Jones and Chew (1983a), but at a higher homologous temperature. We
345 prepared the specimen by first deforming it at $P=0.1$ MPa until it reached
secondary creep, and then increased P in 5 MPa steps recording the strain
rate 24 h after the pressure change. The resulting strain rates are shown in
Table 2, indicating little change in strain rate with increasing pressure from
350 0.1 to 15 MPa. Temperature control for this experiment was not as good
as that of the constant pressure tests due to the varying P and mechanical
problems with the cold room.

3.2. Grain Size and Shape

The median grain diameter $\bar{d}$ for $P=20$ MPa specimens increased slightly
355 in the early stages of deformation ($\epsilon < 1\%$), as expected from the work of
Azuma and Higashi (1983). The grain size distribution was narrow, asym-
metric with a heavy tail towards larger grain sizes and mainly consisted of
equiaxed grains.

At strains approaching 6% we found a rapid decrease in $\bar{d}$. The grain
360 size distribution was again asymmetric, heavy tailed and very wide: a few
large irregularly shaped grains were surrounded by smaller, approximately

equiaxed grains.

At our highest strains, approaching 9%, the grains were large, heavily subgrained, amoeboid in shape and therefore extensively interlocked. The grain size distribution was narrower than that observed at $\epsilon = 6\%$, but still wider than the initial, unstressed state. Observations in the SEM shows significant amounts of grain growth directed along subgrain boundaries.

Specimens deformed to strains around 9% at $P=0.1$ MPa also demonstrated some amoeboid grains and, when observed in the SEM, less directed grain growth. $\bar{d}$ was larger than that of specimens deformed to similar strains at $P=20$ MPa, as shown in Table 3 and Figure 3.

3.3. Subgrain Boundaries

We observed the formation of subgrain boundaries in all specimens using sublimation-etched ice techniques similar to those discussed in Hamann et al. (2007) and Weikusat et al. (2011), and by performing a kernel averaged misorientation (KAM) analysis (Brewer et al., 2009) on one of our few high resolution EBSD datasets, shown in Figure 4. The spatial variability of subgrain boundary density (estimated by sublimation etching techniques) within a given specimen was significant, no doubt due to the well known inhomogeneity of the stress and strain fields within polycrystalline ice (Grennerat et al., 2012).

Individual subgrain boundaries can be observed in high resolution (~ 10 μm step size) KAM analyses and this technique can correctly identify high angle grain boundaries where contrast analysis of sublimation grooves fails (see Label d in Figure 4). Unfortunately, routine analysis of ice at this

Figure 3: Grain size distributions for the four most strained specimens, as determined by analysis of cross-polarized thin section images. N indicates the number of grains used in each analysis.

Table 3: Grain size and shape data for creep specimens. A value of ~ 10 or more grains across the specimen diameter ensures that the specimen is truly polycrystalline for the purposes of creep testing (Jones and Chew, 1983b).

Specimen	P (MPa)	σ (MPa)	T (°C)	T_h	Final ϵ	d mm	Average Solidity	Average Circularity	Grains across spec. diam
Unstrained	0.1	-	-	-	0%	2.30 ± 0.025	0.946 ± 0.001	0.805 ± 0.001	14.75
S01FEB13	0.1	0.63	-2.48	99.09%	8.81%	3.29 ± 0.035	0.891 ± 0.001	0.639 ± 0.001	10.3
S20NOV12	20.0	0.63	-5.04	98.69%	8.83%	1.52 ± 0.026	0.823 ± 0.001	0.585 ± 0.001	11.9
S22OCT12	20.0	0.63	-5.06	98.68%	8.86%	2.52 ± 0.028	0.812 ± 0.001	0.544 ± 0.001	10.9
S11JAN13	0.1	0.63	-3.60	98.68%	9.41%	2.70 ± 0.029	0.852 ± 0.001	0.608 ± 0.001	9.4
S11DEC12	0.1	0.63	-4.94	98.19%	9.23%	2.77 ± 0.033	0.873 ± 0.001	0.666 ± 0.001	9.6

Figure 4: Micrographs and KAM analysis of S11DEC12. Upper half shows secondary electron micrographs of the specimen surface, showing surface topography. Lower half shows a map of KAM values (in units of degrees misorientation) derived from a high resolution EBSD map of the same specimen. Labels a, b, and c show the locations of subgrain boundaries that are visible in the micrograph and detectable in the misorientation analysis. Label d shows a high angle grain boundary that is indistinguishable from subgrain boundaries on the basis of micrograph contrast alone. Label e shows locations where surface imperfections lead to higher values of misorientation without the presence of subgrain boundaries.

resolution is only possible on very small areas (about 4 mm^2) because of the sublimation rate of the ice surface during analysis in the SEM. This is especially problematic given the large grain size of ice and spatial variability of subgrain boundary densities within a single specimen.

390 3.4. Fabric

Ice fabrics were generally similar between $P=0.1$ and $P=20$ MPa specimens, as shown in Figure 5c, Figure 6c and Figure 7d. We were limited

in the number of grains available for analysis in the SEM due to the large grain size of ice, and limited sample volume from each specimen.

395 Figures 5 and 6 use a Schmid factor (Trickett et al., 2000; Wilson et al., 2014) coloration to demonstrate the relative magnitude of the resolved shear stress on the $(0001)\langle 11\bar{2}0 \rangle$ basal slip system of a given crystal orientation imposed by the axial load σ . The Schmid factor can range from 0.5 (typically shown in red) to 0 with the c -axis parallel to σ (typically shown in
400 blue). These maps indicate that, despite the overall similarity of the fabrics between $P=20$ MPa and $P=0.1$ MPa specimens, the spatial distribution and morphology of the grains making up that fabric can be different.

Figure 7 uses an inverse pole figure coloration to more clearly show the interlocked grain structure that develops in the early stages of tertiary creep.

405 4. Discussion

The results of our mechanical testing are consistent with the literature:

1. At the same T , secondary and tertiary strain rates are larger at $P=20$ MPa compared to $P=0.1$ MPa (i.e. comparing S20NOV12 with S11DEC12).

At the same T_h , strain rates are smaller at $P=20$ MPa compared to
410 $P=0.1$ MPa (i.e. comparing S20NOV12 with S11JAN13). Both results are consistent with (Haefeli et al., 1968). The fact that specimens deformed at equal homologous temperatures *do not* have equal strain rates is an important clue that processes other than the pressure melting effect are contributing to the observed changes in strain rate.

- 415 2. The enhancement factor $\dot{\epsilon}_{\text{tertiary}}/\dot{\epsilon}_{\text{min}} \approx 3$ for all P tested (Treverrow et al., 2012).

Figure 5: (a) EBSD map of specimen S22OCT12 strained 8.86% at $P=20$ MPa and $T = -5^\circ\text{C}$, Schmid factor coloration. (b) Schmid factor histogram for all data points. (c) c -axis pole figure, one point per grain. (d) Detail of grain boundary migration directed along subgrain boundaries and subsequent rotation recrystallization. (e) Detail of extensive subgrain development. (f) Detail of directed grain boundary migration along subgrain boundaries.

Figure 6: (a) EBSD map of specimen S11DEC12 strained 9.23% at $P=0.1$ MPa and $T = -5^{\circ}\text{C}$, Schmid factor coloration. (b) Schmid factor histogram. (c) c -axis pole figure, one point per grain. (d1) Detail of grain-subgrain interaction. (d2) High resolution EBSD map showing $\pm 3^{\circ}$ intragranular misorientations.

Figure 7: (a) EBSD map of specimen S01MAR13 strained 6.14% at $P=20$ MPa and $T = -5^{\circ}\text{C}$, Inverse Pole Figure coloration shown in (b). (c) Schmid factor histogram. (d) c -axis pole figure, one point per grain. (e) Detail of dissected grain. (f) Detail of grain boundary migration and bulging.

3. The minimum strain rate $\dot{\epsilon}_{\min}$ occurs around 1% for all P tested.
4. Strain rates for $P \leq 15$ MPa were essentially unchanged by the application of confining pressure in the variable pressure testing on S31MAY13.

420 It is significant that this result was observed at $98.17\% \leq T_h \leq 98.57\%$, nearly 2% higher than the variable pressure test conditions of Jones and Chew (1983a) which showed a similar lack of response for $P \leq 15$ MPa.

The pressure melting effect of ice complicates the interpretation of the
 425 mechanical results as it is difficult to separate the effects of changing the homologous temperature from those associated with pressure effects on the material properties or dislocation dynamics of ice. Jones and Chew (1983a) discuss a liquid-enhanced grain boundary sliding hypothesis to describe their results and is consistent with $V^* < 0$ at $P > 15$ MPa because the liquid
 430 state of water occupies less volume than the solid. If their hypothesis is true, and some critical value of T_h near 96.3% is the minimum required to activate liquid-enhanced grain boundary sliding processes, then our variable pressure test should have shown increasing strain rate with *any* increase in pressure over atmospheric. Further, our microstructural observations
 435 of heavily interlocked grains suggests that liquid-enhanced grain boundary sliding would not be an effective means of achieving bulk strain (Raj and Ashby, 1971). We therefore consider other mechanisms consistent with a negative V^* for $P > 15$ MPa.

4.1. Pressure-enhanced recovery in ice

440 Recovery is typically associated with the softening of dislocation-hardened crystals. Dislocation mobility is an essential part of recovery as it allows a dislocation the possibility of moving around obstacles, enabling further plastic deformation and lowering the stored strain energy (Lothe and Hirth, 1967).

445 The extreme plastic anisotropy of ice derives from the dissociation of dislocations on the basal plane (Fukuda et al., 1987); extended dislocations glide only on the basal plane and are unable to move unless constricted or fully recombined (Hondoh, 2000; Argon and Moffatt, 1981; Sherby and Burke, 1968). Thus recovery in ice is generally difficult and strain energy is
450 readily concentrated such that recrystallization becomes a viable means of reducing this energy (Duval et al., 2012; Ueki et al., 1987).

By making some simplifying assumptions regarding the variation of the energy of a stacking fault as a function of lattice dilatation, Fontaine and Haasen (1969) calculated the width of dissociated dislocations and deter-
455 mined that there should exist a *critical hydrostatic pressure* P_c at which point it is no longer energetically favorable for a dislocation to split. Fontaine and Haasen estimated that the critical pressure to begin observing pressure-induced constriction effects on dislocation width occurred when

$$\kappa_0 b P_c / 2 \approx F \gamma_0. \quad (2)$$

where b is the magnitude of the Burger's vector of the perfect dislocation,
460 γ_0 is the stacking fault energy at $P=0.1$ MPa, κ_0 is the lattice dilatation

associated with a stacking fault at $P=0.1$ MPa and F was chosen by the authors to be 0.1. In other words, pressure effects on dissociated dislocation width should become detectable when the PV energy of the stacking fault dilatated lattice at pressure P_c reaches 10% of the stacking fault energy of the material. Fontaine and Haasen explain their choice of this 10% constriction threshold to merely establish the order of magnitude of P_c . The critical pressures for the various stacking faults in ice as a function of κ_0 are shown in Fig. 8 where we plot curves both for Fontaine and Haasen’s 10% threshold and for the “100% threshold” ($F=1$) where pressure effects on w would be certain. We therefore regard the the 10% and 100% thresholds as lower and upper bounds for the range of P_c for the various stacking fault energies.

Values of κ_0 are not known for the various stacking faults in ice, but they are likely small given the low stacking fault energies observed in ice (Hondoh, 2013). The actual value of κ_0 is crucial to the viability of Hypothesis 1, both in terms of significant dissociated dislocation constriction and achievement of the critical pressure to begin that constriction. Taking the lowest energy stacking fault (0.31 mJ m^{-2} for the $\mathbf{p} + \mathbf{c}/2$ fault in Table 2 of Hondoh (2000)) as an example, we estimate the impact of different values of κ_0 and constriction threshold F on constriction and critical pressures:

- In the case of constriction for a 0.1 to 20 MPa confining pressure increase, we estimate 13% constriction for $\kappa_0=0.01$ and approximately 1.4% constriction for $\kappa_0=0.001$.
- The critical pressures could be consistent with the change in sign of V^* around $P=15$ MPa observed by Jones and Chew (1983a) if $0.006 <$

Figure 8: Critical pressure P_c vs lattice dilatation κ_0 for three possible stacking fault energies in ice. Solid curves show P_c for Fontaine and Hassen's 10% pressure effect threshold, while upper dot-dash curves show the full 100% threshold; the upper dot-dash curve for the highest energy stacking fault is outside the range of this plot. The lattice dilatation due to a stacking fault is not known for ice, but is likely small. The change from positive to negative V^* observed by Jones and Chew (1983a) occurs around $P=15$ MPa.

485 $\kappa_0 < 0.01$ and Fontaine and Haasen’s 10% constriction threshold is
correct. For the 100% constriction threshold, the lattice dilatation
would need to be an order of magnitude larger ($0.06 < \kappa_0 < 0.1$)
to explain the same change in sign. Figure 8 shows the effect of the
different values of stacking fault energy, κ_0 and constriction threshold
490 on the critical pressure.

The initial reduction in creep rate with pressure (i.e. $V^* > 0$) may
be caused by the usual diffusion-retarding effects of pressure (Butcher and
Ruoff, 1961; Hahn and Gleiter, 1979). However, we hypothesize (Hypothe-
sis (1) in Section 1.4) that the subsequent increase in creep strain rate with
495 pressure (i.e. $V^* < 0$) beyond ~ 15 MPa may be caused by the constriction
of dissociated dislocations, thus enhancing dislocation mobility and there-
fore strain rate in ice. Louchet (2004) argues that climb velocity of basal
dislocations is limited by the low concentration of jogs. Pressure-induced
constriction would act to reduce the energy required for jog formation, thus
500 increasing jog concentration and potentially shifting the rate limiting process
from one associated with dislocation drag to one associated with climb.

Our second hypothesis (Hypothesis 2 in Sec. 1.4) for increased creep
strain rate at high pressure is the higher equilibrium concentration of self-
interstitial defects available to fuel climb processes. However, this hypothesis
505 faces two significant challenges:

1. The increase in equilibrium self-interstitial concentration for a pressure
change of 20 MPa is very small, leading to a supersaturation of only 4%
relative to the equilibrium concentration at atmospheric pressures

(Hondoh, 2009); a temperature reduction of less than 0.2 C yields the
510 same supersaturation.

2. Monotonic changes in self-interstitial concentration with pressure are insufficient to explain the change in sign of V^* . Some change in rate controlling mechanism must be identified.

One possible argument is that the rate controlling process at lower pressures
515 is basal dislocation drag, perhaps controlled by proton-disorder effects (Glen, 1968; Taubenberger et al., 1973), or perhaps by lattice friction (Louchet, 2004). If at higher pressures we transition to a regime where dislocation climb becomes the rate controlling process, then increases in pressure would increase self-interstitial concentration, enhancing rates of climb and yielding
520 $V^* < 0$. However, such an argument does not provide a cause for the change of rate controlling mechanism unless we appeal to the dislocation constriction effects of Hypothesis 1. In summary, we acknowledge that the change in equilibrium self interstitial concentration is small for the 0.1 to 20 MPa pressure range. However, even a small change in self interstitial
525 concentration could be important *if* it occurs near a transition in the rate controlling process.

4.2. Fabric and Texture

Microstructurally, we observed an unexpected reduction in median grain size and less regular grain shapes for specimens deformed at $P=20$ MPa
530 compared to those tested at $P=0.1$ MPa. The reduced median grain size and irregular grain shape seems to be the result of grain dissection processes (Jessell, 1987) caused by enhanced grain boundary migration along subgrain

boundaries. As the driving force for this grain boundary migration is the reduction of interfacial energy, no particular orientation seems to be preferred for dissection as can be seen in Fig. 7. However, we note that higher-angle subgrain boundaries should have both a higher interfacial energy and lower velocity (Higashi and Sakai, 1961) suggesting that directed grain boundary migration may be more likely along these boundaries. The directed grain growth process leads to a different grain size distribution between $P=0.1$ and $P=20$ MPa specimens, but the fabrics are largely the same: a circle girdle around 40° as expected from prior work by Jacka and Maccagnan (1984).

Grain shapes for strains nearing 10% tend to be more equi-axed for $P=0.1$ MPa, but amoeboid at $P=20$ MPa, consistent with the directed grain growth along subgrain boundaries. The prevalence of interlocked amoeboid grains means that deformation by grain boundary sliding is less likely to contribute to the bulk strain of the specimen, though some local strain accommodation may be achieved through this method (Raj and Ashby, 1971).

4.3. Mechanical effects of enhanced recovery

The climb of basal dislocations provides two deformation systems independent of the basal slip system to, in the framework of the “self consistent” method, providing a total of four slip systems, which is sufficient for crystal plasticity (Ashby and Duval, 1985). Enhancing the climb recovery via hydrostatic pressure would act to lower the stress concentrations caused by grain incompatibility during deformation and generally act to increase the

creep rate by increasing the dislocation mobility (Barrett and Sherby, 1965).

Weertman (1965) analyzed this process by postulating that diffusion along a dislocation core is much faster than bulk diffusion in the case of perfect dislocations, and approaches the rate of bulk diffusion as the dissociated
560 dislocation width w becomes large. As climb depends on the formation and diffusion of point defects (and possibly on the concentration of jogs), the climb rate of an extended dislocation should depend on w . In turn, w decreases with increasing hydrostatic pressure and, if the dislocation is part of
565 an array within a subgrain boundary, with increasing misorientation angle of that boundary.

Continuing this line of thought, Weertman (1968) modified the Nabarro-Herring (NH) creep model for the presence of subgrains, using the subgrain size rather than the grain size to characterize the bulk diffusion distances
570 required for NH creep. This is especially important in ice as the nominal *grain* sizes are too large to allow the NH creep to play any significant role in the creep deformation of glacial ice (Duval et al., 1983). Using the subgrain size as the characteristic bulk diffusion distance and a G/σ subgrain size dependence, Weertman (1973) argues that subgrain-modified NH can result
575 in a creep law with $n = 3$ if the dislocations mainly exist evenly spaced within subgrain boundaries.

The purpose of this discussion is not to advocate a specific theoretical creep model, but instead to elaborate a plausible mechanism through which hydrostatic pressure, by changing the effective stacking fault energy, can
580 have a direct impact on dislocation mobility and thus creep rate in polycrystalline ice. Similar arguments have been made for the dependence of

climb velocity (Argon and Moffatt, 1981) and creep rate (Mohamed and Langdon, 1974) on the stacking fault energy.

In this discussion we have focused mainly on recovery via dislocation
585 climb, but cross-slip (Poirier, 1976; Landgon, 1973; Akimov et al., 1980)
is another possible recovery mechanism in ice that could be enhanced by
application of hydrostatic pressure. Poirier (1976) presents a constitutive
equation for high temperature creep which explicitly includes climb and
cross slip, where the activation energy for climb Q_C is equal to that of self
590 diffusion Q_D and the activation energy for cross slip Q_S is a function of
both σ and γ_F . In ice, with its widely extended basal dislocations, it seems
appropriate to also consider a σ and γ_F dependence for the climb activation
energy. In this case, the constitutive equation would look like

$$\dot{\epsilon} = \dot{\epsilon}_{0,C} \left(\frac{\sigma}{G} \right)^3 \exp \left(-\frac{Q_C(\sigma, \gamma_F)}{kT} \right) + \dot{\epsilon}_{0,S} \left(\frac{\sigma}{G} \right)^2 \exp \left(-\frac{Q_S(\sigma, \gamma_F)}{kT} \right) \quad (3)$$

where the effective stacking fault energy is related to the hydrostatic pres-
595 sure.

5. Conclusions

Our confined creep testing results are consistent with those reported
in earlier literature for both constant and variable pressure tests. We have
found significant microstructural differences, including median grain size and
600 grain morphology, between polycrystalline specimens deformed at $P=0.1$
and $P=20$ MPa. We conclude that these differences are caused by directed

grain growth along subgrain boundaries in specimens deformed at higher pressure, leading to a smaller median grain size and a dissection microstructure (Jessell, 1987).

605 We presented two hypotheses regarding the impact of confining pressure on creep strain rate in polycrystalline ice. We feel that Hypothesis 1 better explains our observations and the observations of others for the following reasons:

1. The change in sign of V^* around 15 MPa observed by Jones and Chew
610 (1983a) could explained by the existence of a critical confining pressure at which dissociated dislocations find it energetically favorable to constrict and thereby become more mobile and enhance creep strain rates.
2. Comparison of the variable pressure tests conducted in this work and
615 by Jones and Chew (1983a), combined with our observations of heavily interlocked grains, suggest that increased creep strain rate at $P \geq 20$ MPa is not a result of pressure melting effects and enhance grain boundary sliding. Both tests showed a negligible strain rate change for $0.1 < P < 15$ MPa, despite the fact that our variable pressure test
620 was conducted at a significantly higher T_h .

The notion that dissociated dislocations in ice I_h are sensitive to confining pressures is central to our hypothesis, and that sensitivity depends on both the unknown magnitude of lattice dilatation induced by stacking faults in ice, and the constriction threshold F (i.e the 10% threshold of Fontaine
625 and Haasen) at which confining pressure effects on dissociated dislocations

become important. Very small values ($\kappa_0 < 0.005$) of lattice dilatation and a large constriction threshold would preclude pressure-induced constriction of partial dislocations, at least in the 0.1 to 20 MPa pressure regime accessible to our equipment. A value of $0.06 < \kappa_0 < 0.2$ coupled with a 10%
630 constriction threshold would yield a critical pressure consistent with the 15 to 30 MPa range where Jones and Chew (1983a) observed the transition to a negative activation volume for creep. Molecular dynamics simulations of stacking fault behavior in ice (Pirzadeh and Kusalik, 2011; Malkin et al., 2015) at varying pressures may be one method to estimate these parameters.

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