Quantifying Damage in Polycrystalline Ice via X-Ray Computed Micro-tomography

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ABSTRACT

The use of X-ray computed micro-tomography (micro-CT) is presented here as a useful tool for the analysis and quantification of damage in polycrystalline ice. Although known to be useful for characterizing damage in many other materials, the use of micro-CT has not yet been adapted to the non-trivial case of also characterizing damage in polycrystalline ice. Samples of polycrystalline ice were tested in uniaxial compression at six different strain rates, spanning four orders of magnitude, from $1 \times 10^{-6} \text{ s}^{-1}$ to $1 \times 10^{-3} \text{ s}^{-1}$, and two different testing temperatures of -10°C and -20°C . The extent of cracking from each test is characterized via micro-CT imaging and is quantified via a newly proposed variant of the crack density tensor, which accounts for any anisotropy in the mean crack orientation and is shown to be equivalent to the materials anisotropy tensor. To account for anisotropy in the distribution of cracks, an eigenanalysis is also performed. The results show that micro-CT can be a useful tool for both visualizing and quantifying damage in polycrystalline ice and that a 3-D analog of the traditional second-rank crack density tensor can be readily calculated via commercially available software.

KEYWORDS: Damage, Fracture, Ice, X-ray, micro-CT

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1. INTRODUCTION

Over the last two decades, the use of X-ray computed micro-tomography (micro-CT) has become a widely used laboratory technique for many applications in materials science and engineering [1,2]. Underlying much of this broad appeal is the ability of micro-CT to nondestructively characterize material microstructures at a micron resolution in three dimensions. To date, micro-CT has been successfully employed to characterize an extensive variety of materials and processes, such as bone porosity [3], brine channels and air inclusions in sea ice [4], the fracture and damage of concrete [5], the microstructural evolution of snow under a temperature gradient [6], fatigue crack propagation in cast-iron [7], etc. Although not nearly an exhaustive list, one area of study where the advantages of micro-CT have not yet been fully realized, is the study and 3-D characterization of damage in strained polycrystalline ice. Developing such an understanding is thought to be of critical importance to better predicting the fracture mechanics of both land-based and sea-based ice flows and is thus the primary focus of this work.

Like many other materials, ice can sustain damage in the form of non-propagating cracks that result from localized strains [8]. The presence of these cracks, in turn, can have dramatic affects on the material properties of the ice, even though the larger body remains fully intact. Understanding the role of deformation and damage in polycrystalline ice, including columnar ice, has been the topic of many laboratory, field, and theoretical studies spanning the past five decades [9,10,11,12,13,14,15,16]. This interest is derived from the broad application of such knowledge, including calving rates of glaciers and ice sheets [17,18], predicting sea ice behavior in the Arctic and Antarctic

oceans [19,20,21,22], structural engineering of off-shore structures in the polar regions [23,24,25], and interpretation of the surface characteristics of icy terrestrial bodies [26,27,28].

In order to quantify and validate the predictions of traditional fracture mechanics, as it would apply to ice I_h , there is often an imaging component of the analysis required for either the investigation of field-collected samples or laboratory tested specimens. This imaging component requires that the actual cracks be imaged such that they can be manually counted, measured, and characterized. This is an important component of the analysis, as the size, type, and number of cracks in a sense act as benchmarks detailing the mechanical history of the ice. For example, the observation of “comb-cracks” versus “wing-cracks” along a fault may be indicative of the destabilization mechanism of the body [15], whereas evidence of intergranular versus transgranular fracture may be more telling about the response of the ice to either elastic or plastic deformation [29], respectively.

Similar to many polycrystalline metals and alloys, the study of polycrystalline ice via micro-CT has previously been thought of as a poor choice of materials for micro-CT analysis as no other microstructural information can be elucidated and x-ray attenuation in ice can be quite high [4]. Given sufficient voiding, cracking, or pore space is present within the material, the use of polychromatic x-ray sources has been shown to be useful, mostly limited only by camera resolution. This has been evidenced by other recent micro-CT studies pertaining to sea ice inclusions [4], the bonding of ice spheres [30], and the metamorphism of ice-snow interfaces [31].

2. BACKGROUND

2.1 Crack Density Tensor

A very recent and detailed review of the various ways to measure and quantify damage in polycrystalline ice is given in Snyder et al. (2015) [8] and is not repeated here. To summarize, however, most approaches involve first the concession of a 3-D perspective to a 2-D perspective, such that all cracks within a 2-D plane can be differentiated and then manually measured and counted. Making such measurements can be quite tedious as both the crack length and the normal orientation of the crack from a fixed point of reference is required. With this information, a dimensionless 2-D crack density tensor α_{2-D} can be derived

$$\alpha_{2-D} = \frac{1}{A} \sum_i (c^2 \vec{n} \vec{n})_i \quad (1)$$

where A is the cross-sectional area of the sample, c is the crack half-length, and $\vec{n}$ is the unit vector normal to the i th crack of length $2c$ [32,8]. The vector product $\vec{n} \vec{n}$ denotes the dyadic, which when summed over all cracks and all orientations, gives a measure of the anisotropy of the measured crack orientation in a tensorial format as a second-rank tensor [8]. If extending to the 3-D case, Eq. (1) simply becomes

$$\alpha_{3-D} = \frac{1}{V} \sum_i (a^3 \vec{n} \vec{n})_i \quad (2)$$

where V is the sample volume and a is the radius of the i th circular-shaped crack [33,8]. In either format, the benefit of the tensorial representation is that it lends a continuum

approach to understanding the damage mechanics of the material [34]. In the case of an isotropic or random distribution of crack orientations, Eq. (1) and Eq. (2) simplify to the scalar (first invariant of the tensor), which is the trace of alpha ($tr(\alpha) = \rho_c$).

2.2 Stereological Analysis

Of the many variables that can be calculated with the Bruker CTan software, the 3-D stereological analysis is focused on here as perhaps the most beneficial in handling anisotropy and crack density distributions. Originally developed for the analysis of pore space in bone, the stereological analysis makes use of a statistical technique in which a mean intercept length is calculated in 3-D from within a spherical volume encompassing the user-defined volume of interest (VOI). Within this encompassing sphere, lines can be drawn in up to 1024 different orientations with a spacing that is user-defined, but can be as small as one pixel or “voxel” (a 3-D pixel) apart. Once drawn, the orientation of each line is recorded and the length of each line is divided by the number of times it intercepts an object (voxels equal to 1 on a binary scale), this quotient defines the mean intercept length l . It should be noted, however, that whether or not the line drawn traverses a lone 1 pixel or ten 1 pixels, as long as these pixels are connected, this would only count as one intercept. Thus, the mean intercept length could also be thought of as a metric for the length between cracks and not necessarily the crack length itself.

For the mean intercept length analysis presented in this study, the number of randomly selected orientations was set to 512 with a $90\ \mu\text{m}$ (5 pixel) spacing. For perspective, this allowed for 53,625 lines to be drawn per orientation within a VOI of $4213\ \text{mm}^3$. Once complete, the number and length of all intercepts over all orientations

are related back to the principal axes of the encompassing sphere, such that three principal mean intercept lengths, l_1 , l_2 , and l_3 and vector orientations, $\vec{e}_1$, $\vec{e}_2$, and $\vec{e}_3$ can be determined. These orientations are solved for via the statistical fitting of an ellipsoid encompassing all mean intercept length values when each intercept is plotted as a radius with each line anchored at the origin. Originally given in Harrigan and Mann (1984) [36], this process creates the surface of an ellipsoid of the general form

$$Ax_1^2 + Bx_2^2 + Cx_3^2 + 2Dx_1x_2 + 2Ex_1x_3 + 2Fx_2x_3 = 1 \quad (3)$$

where A , B , C , D , E , and F are dimensionless coefficients and x_1 , x_2 , and x_3 are the principal axes of the original Cartesian coordinate system of the VOI. $\vec{e}_1$, $\vec{e}_2$, and $\vec{e}_3$ can then be solved for via an eigenanalysis, determining the rotation of the orthogonal principal axes of the ellipsoid from the original axes of the VOI, such that $\vec{e}_1$, $\vec{e}_2$, and $\vec{e}_3$ are the eigenvectors representing the orientations of l_1 , l_2 , and l_3 relative to the original Cartesian coordinate system of the VOI. Additionally, the eigenvalues E_1 , E_2 , and E_3 , from the eigen analysis can be used as a measure of the degree of anisotropy DA of the VOI, where $DA = 1 - (\frac{E_{min}}{E_{max}})$. It was also cleverly pointed out in Harrigan and Mann (1984) [35] that the coefficients of Eq. (3) can equally be given as a dimensionless tensor,

which they name the materials anisotropy tensor M , where $M = \begin{bmatrix} A & D & E \\ D & B & F \\ E & F & C \end{bmatrix}$. Given that

M is indiscriminate towards the material to which it is being applied, it is proposed in this study that it could be used as an analog to the tensorial component of Eq. (2). From within the constructs of the commercially available CTan software for micro-CT analysis,

all the variables described above (i.e. $l_1, l_2, l_3, \vec{e}_1, \vec{e}_2, \vec{e}_3, E_1, E_2, E_3, DA$, and M) can be calculated and given as outputs as part of a 3-D stereological analysis, further demonstrating the advantages of micro-CT.

To more plainly demonstrate and test the applicability of the CTan 3-D stereological analysis for application to problems concerning crack orientation and distribution in solids, two test-cases were created and are presented here. The first, a stack of binary images alternating in layers of pixel values set to either 0 or 1, and oriented such that the 1 layers were perpendicular to the x_3 axis is shown in the upper panel of Figure X. The second example shown in the lower panel of Figure X, is the same as that shown in the upper panel, but rotated 90 degrees, such that the layers with pixel values equal to 1 are parallel to the x_3 axis. These binary matrices were created such that they were 100x100x100 pixels in size, with each pixel equaling 100 μm in resolution, resulting in a 1 cm cube. The stacks were analyzed with the 3-D stereological analysis in CTan, as discussed above.

As would be expected, it was found that the eigenvectors with the minimum mean intercept length were normal to the layering of the matrix, while the other two mean intercept lengths were much larger and nearly identical. Also shown in the upper and lower panels of Figure X, are illustrations of the best fitting ellipsoid about the mean intercept lengths, representing the obvious nature of the layering and corresponding anisotropy. Calculated values for the mean intercept length, eigenvectors, and eigenvalues are also given in this figure.

3. EXPERIMENTAL METHODS

3.1 Ice Preparation

Following Cole (1979) [36], right-cylindrical specimens of pure polycrystalline ice (using Milli-Q 18.2 M Ω cm⁻¹ deionized water) were created via a radial freezing technique, where 0.8 mm seed grains were loaded into an aluminum mold, flooded with chilled and deaerated water, and then slowly frozen radially from the outside-inwards via a temperature controlled glycol wrap placed around the exterior of the mold. Following this approach, polycrystalline specimens of ice with a mean grain diameter of 1 mm and a cylindrical aspect ratio of 3:1 were created. An example of the initial polycrystalline microstructure is shown in Figure 1, as photographed between crossed polarizing filters. An initial microstructure of randomly oriented grains ranging from 0.8-1.2 mm in diameter was obtained, as ascertained with polarized light microscopy and a manual calculation of the mean intercept length [37]. Following Cole (1979) [36], this initial microstructure was found to be reproducible. Specimen dimensions were 3.8 cm in diameter by 11.4 cm in length.

3.2 Mechanical Testing

Uniaxial compression tests were carried out using a MTS servo-hydraulic testing apparatus housed in a cold room. At a test temperature of -10°C (+/- 0.2°C), six constant strain rates of 1x10⁻³ s⁻¹, 1x10⁻⁴ s⁻¹, 1x10⁻⁵ s⁻¹, 5x10⁻⁶ s⁻¹, 2.5x10⁻⁶ s⁻¹, and 1x10⁻⁶ s⁻¹ were applied. At a test temperature of -20°C (+/- 0.2°C), two constant strain rates of 1x10⁻⁵ s⁻¹ and 1x10⁻⁶ s⁻¹ were applied. All compression tests were terminated at an engineering strain of 5% as the peak stress had always been obtained by that point. It should also be noted that, apart from the samples tested at 1x10⁻³ s⁻¹, these are the same samples as those

used for the mechanical testing data shown in Hammonds & Baker (2016) [38] for pure polycrystalline ice, thus the results from the mechanical tests will not be repeated here. At the completion of each mechanical test, two cross-sectional volumes of ice were cut with a high precision bandsaw for damage analysis via micro-CT that were at least 2 cm in height and 3.8 cm in diameter. This was done for two specimens per each applied strain rate and temperature, resulting in a total of 32 cross-sectional volumes to be analyzed via micro-CT. Between the periods of mechanical testing and micro-CT analysis, all samples were stored in a -30°C ($\pm 0.2^{\circ}\text{C}$) cold room to prevent any significant changes in the microstructure.

3.3 X-ray Computed Micro-Tomography (micro-CT)

For the damage analysis and imaging of cracks, a Bruker Skyscan 1172 desktop micro-CT housed in a -10°C cold room was used. For all samples, the X-ray beam was set to 40 kV and 250 mA. The sample was placed on the scanning stage such that the long cylindrical axis (z-axis or x_3) was oriented perpendicular to the X-ray beam, as shown in Figure 3. The sample was then rotated 180° in the x-y plane (or x_1 - x_2 plane) at 0.7° increments while being scanned. Attenuation profiles were captured using a 1.3 Mp, 12-bit, cooled CCD Hamamatsu camera. A $15\text{ }\mu\text{m}$ resolution was used, allowing for the actual scanned inner volume of the sample to be 1.9 cm in diameter by 1.5 cm tall. If using software to stitch together reconstructions, it would be possible to scan the entire sample, but that was not done here, as the volume of interest (VOI) from one individual scan was large enough to capture what is thought to be a representative volume of cracks and orientations.

Image post-processing and three dimensional image reconstruction were performed using NRecon software, which included thermal drift correction, post alignment, removal of ring artifacts, Gaussian pixel smoothing, and beam hardening correction. Because the resulting images from this reconstruction are on a 0–255 grayscale, they must then be converted to binary images, such that they can be analyzed as individual pixels made up of either 0's (ice) or 1's (air/crack) on a binary scale. To allow for absolute control of the binary conversion, grayscale images were converted using Matlab's image processing toolbox, although CTan could have also been used. As per visual inspection, this yielded satisfactory results overall, but it should be noted that the threshold chosen for the conversion will ultimately create noise in some images while potentially deleting parts of the objects of interest in others. Therefore, for each sample scanned, a representative set of images must be visually inspected upon conversion to assure that the objects of interest (cracks) are being accounted for while the noise is kept to a minimum. To illustrate this point, a typical binary conversion is shown in Figure 2, where two different thresholds were chosen for the same 2-D grayscale image taken from a sample tested at -10°C and $1 \times 10^{-3} \text{ s}^{-1}$ (Figure 2a). Notice that neither images Figure 2b or Figure 2c perfectly capture the full and unbiased extent of the micro-cracks observed in the grayscale image, but Figure 2b would certainly yield more accurate results than Figure 2c when performing a 2-D or 3-D quantitative analysis based on the number and location of binary pixels (0 or 1), thus a threshold is manually chosen that is applied to the entire stack of images.

4. RESULTS & DISCUSSION

4.1 Damage

The most easily obtainable metric for imparted damage D from a binary micro-CT reconstruction is a simple cavity volume fraction of air to ice (i.e. $D = V_{air}/V_{ice}$) from within a given VOI. This approach can be useful if considering the evolution of damage within a material over periods of varying stress, strain, or time [39,8]. Expressed as a percentage, D is shown in Figure 4 as the mean value for all scans performed of all samples over a given strain rate. In this figure, D monotonically increases upon increasing the applied strain rate at -10°C and -20°C , as would be expected. What is missing from this analysis, however, is additional information pertaining to the number of cracks, their orientation, their distribution about the volume, and whether or not any anisotropy is present, as may also be expected from a compression test conducted in a uniaxial mode [34]. Although an individual object analysis can be performed within CTan, such that each individual crack becomes an object, the focus here is on only the distribution of cracks about the VOI and the presence of any anisotropy within this distribution of crack orientations. To further elucidate this point, Figure 5 shows images for tests conducted at $1 \times 10^{-4} \text{ s}^{-1}$ and $1 \times 10^{-5} \text{ s}^{-1}$ at temperatures of -10°C and -20°C , respectively, when viewed along the loading direction x_3 (top view) vs. perpendicular to the loading direction x_1 or x_2 (side view). In these images, a clear example of anisotropy exists in the crack orientation, such that most cracks formed parallel to the loading direction. This trend was apparent throughout all specimens analyzed with the micro-CT, except at the lower strain rates ($\leq 5 \times 10^{-6} \text{ s}^{-1}$), where virtually no cracking was observed. Previously, such information about the 3-D anisotropy of damage within a specimen would have been difficult to obtain, normally extrapolated from a 2-D analysis, but with

micro-CT imagery and reconstruction such characteristics become readily apparent. To account for this anisotropy in calculations, the implementation of a crack density tensor, and not just a scalar variable, would be most beneficial. Additional micro-CT images and views, as well as micro-CT animations, are provided in the supplementary material.

4.2 Extension of the Crack Density Tensor

In order to account for all x, y, and z components of the crack orientation distribution and resultant anisotropy, a variant of α_{3-D} (Eq. 2) is proposed here, which to the author's knowledge has never before been proposed or applied to ice. Key to this variant of α_{3-D} , is the 3-D stereological analysis introduced in Section 2.2 and the concept of a mean intercept length. It could also be interpreted as an inverse crack density tensor, but it will be shown in this section that the materials anisotropy tensor is of an equal measure. Given that all crack lengths and orientations within a particular VOI can be reasonably and statistically represented as an ellipsoid, it would seem that Eq. (2) could be modified such that a_i becomes l_i and $\vec{n}\vec{n}$ becomes $\vec{e}_i\vec{e}_i$, where l_i is the mean intercept length in each of the three orthogonal directions of the ellipsoid, represented by the three eigenvectors $\vec{e}_i$. Making this substitution yields a volume-averaged and inverse variant of the crack density tensor $\langle \alpha_{3-D} \rangle_V$, where Eq. (2) becomes that given in Eq. (4).

$$\langle \alpha_{3-D} \rangle_V = \frac{1}{V} (l_1^3 \vec{e}_1 \vec{e}_1 + l_2^3 \vec{e}_2 \vec{e}_2 + l_3^3 \vec{e}_3 \vec{e}_3) \quad (4)$$

In this equation, the importance of crack orientation and length is conserved, but it should be noted that $\vec{e}_i$ is in the same orientation as that in which the mean linear intercept length

l_i was measured. Again, if a scalar representation of crack density is desired, then $tr\langle\alpha_{3-D}\rangle_V$ yields such a result, as the first invariant of $\langle\alpha_{3-D}\rangle_V$. To show the effectiveness of $\langle\alpha_{3-D}\rangle_V$ in demonstrating the anisotropy in damage observed over all compression tests, the ratios $\langle\alpha_{3-D}\rangle_{V_{22}}/\langle\alpha_{3-D}\rangle_{V_{11}}$, $\langle\alpha_{3-D}\rangle_{V_{33}}/\langle\alpha_{3-D}\rangle_{V_{11}}$, and $\langle\alpha_{3-D}\rangle_{V_{33}}/\langle\alpha_{3-D}\rangle_{V_{22}}$ are shown in Figure 6a as functions of the applied strain rate at 10°C. For comparison to the material anisotropy tensor M (see Section 2.2), the ratios M_{22}/M_{11} , M_{33}/M_{11} , and M_{33}/M_{22} are also shown in Figure 6b. In these figures, it can be seen that very little anisotropy exists between l_1 and l_2 and their respective orientations $\vec{e}_1$ and $\vec{e}_2$. However, between l_1 and l_3 and l_2 and l_3 , and their respective orientations an anisotropy exists, as for a perfectly isotropic medium all ratios would have been equal to 1. Also, and perhaps not surprisingly, the trends observed in Figures 6a and 6b are quite similar, as they are both based on the mean intercept length and orientation, suggesting that M is a direct measurement of anisotropy in crack orientation, without the need of calculating $\langle\alpha_{3-D}\rangle_V$. If allowing for this substitution, Eq. (4) then gives

$$\langle\alpha_{3-D}\rangle_M = \frac{1}{V}(l_1^3 M + l_2^3 M + l_3^3 M)$$

which simplifies to

$$\langle\alpha_{3-D}\rangle_M = \frac{M}{V}(l_1^3 + l_2^3 + l_3^3) \quad (5)$$

where $\langle \alpha_{3-D} \rangle_M$ remains a unitless second-rank tensor representation of the crack orientation and mean intercept length. For comparison in tensor form, one example of $\langle \alpha_{3-D} \rangle_V$ and $\langle \alpha_{3-D} \rangle_M$ from a single compression test at each strain rate and at -10°C are given in Table 1.

4.3 Crack Density Distribution

While both the traditional crack density tensor and the proposed variant of the crack density tensor give information about the anisotropy of crack orientation, they equally lack information about the three-dimensional distribution of cracks about the entire VOI. For this, a simple eigenanalysis is more useful. In addition to obtaining eigenvectors with CTan, eigenvalues (E_1, E_2, E_3) are also computed. These eigenvalues correlate directly to the coefficients of the ellipsoid given in Eq. (3). Because the eigenvalues represent the geometric transformation of the axes of the ellipsoid ($\vec{e}_1, \vec{e}_2, \vec{e}_3$) back to the principal axes of the scanned volume (x_1, x_2, x_3) of unit length, a larger eigenvalue corresponds to a greater degree of “stretching” or “compressing” of that axis. Thus, large eigenvalues correspond to large geometric transformations of that axis. Figure 7a shows the mean eigenvalues ($\overline{E}_1, \overline{E}_2, \overline{E}_3$) calculated for each constant strain rate test at -10°C. In this figure, the similarities in $\overline{E}_1$ and $\overline{E}_2$ demonstrate the relative isotropy observed with respect to the x_1 and x_2 axes. At the same time, decreasing values of $\overline{E}_3$ with increasing strain rate demonstrates an increasing number of intercepts in x_3 . This occurs because at low strain rates, cracking begins in only select regions and is not uniform over the entire sample volume. This creates an anisotropy in the crack orientation and relative distribution about the VOI, as evidenced by taking the trace of

Eq. (5), $tr\langle\alpha_{3-D}\rangle_M$, as shown in Figure 7b. Here, the decreasing trend of $tr\langle\alpha_{3-D}\rangle_M$ with increasing strain rate provides evidence of the crack distribution becoming more uniform as more cracks begin filling out the VOI.

4.4 Discussion

The results presented in this section are thought to be of significance, as similar previous laboratory studies investigating the mechanical properties of polycrystalline ice [8,14,16] have not yet been able to quantify many of the three-dimensional characteristics of the damage which they had observed. For example, quantifying the crack shape, connectivity, orientation, and spatial distribution of cracks could allow for a more rigorous 3-D model to be developed relating the elasticity and porosity of ice [33,8]. Although this was beyond the scope of this study, it has now been shown to be well within the realm of what is possible with micro-CT. Additionally, as shown in Figure 4 for the data collected at -10°C, it is possible that an exponential relationship may exist between the rate of cavity growth and the imposed strain rate in polycrystalline ice. If these experiments were to be repeated as a function of strain instead of strain rate, for instance, it is possible with the approach presented here that such information could lend additional insight into the mechanics of the cavity development and whether there is a strain dependency indicative of a more ductile or brittle regime of deformation occurring locally [40]. With the introduction of the quantitative techniques presented in this study, combined with the many other recent advances in the quantification of damage and deformation in other polycrystalline materials via micro-CT [41], it is thought that a new paradigm in the laboratory study of polycrystalline ice is possible.

CONCLUSIONS

It has been shown that the use of X-ray computed micro-tomography can be extended to the study of damage and the onset of micro-cracking in polycrystalline ice. This was demonstrated through the detailed analysis of a set of experiments where laboratory-prepared samples of polycrystalline ice, of a 1 mm mean grain diameter, were strained at six strain rates spanning four orders of magnitude and two different test temperatures. It was found that a clear anisotropy exists in both the orientation and distribution of micro-cracks and that these features could be quantified and measured via existing and commercially available software for micro-CT. Additionally, it was found that two individual measurements of anisotropy are necessary for measuring the anisotropy of crack orientation vs. crack distribution, and that these can both be determined with a mean intercept length analysis and quantified with the materials anisotropy tensor, as first given in Harrigan & Mann (1984) [35]. Using this approach, a new and alternative form of the traditional crack density tensor was introduced. In future work, the mechanisms responsible for the damage observed in these test specimens, as well as their connectivity as a function of strain, will be addressed.

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FIGURES

Figure 1. Initial microstructure of a cylindrical specimen prepared for mechanical testing as viewed through crossed polarizing filters. Mean grain diameters were typically on the order of 1 mm. Adapted from Hammonds & Baker 2016.

Figure 2. Conversion of grayscale image generated by micro-CT reconstruction (a) to an optimal binary image (b) and an example of a much less optimal binary image (c). Specimen was strained to 5% at a constant strain rate of $1 \times 10^{-3} \text{ s}^{-1}$ at -10°C . View is looking down the loading axis (x_3).

Figure 3.

Figure 4. Mean volume fraction of air to ice, calculated from 3-D binary reconstructions over all strain rates and temperatures. The mean cavity volume fraction could be considered as a metric for the volumetric crack density. Error bars represent one standard deviation from the mean with n=4 samples.

Figure 5. Micro-CT reconstructions of binary images taken from specimens tested at constant strain rates of $1 \times 10^{-4} \text{ s}^{-1}$ at -10°C (upper) and $1 \times 10^{-3} \text{ s}^{-1}$ at -20°C (lower). This view was generated with micro-CT software that maximally limits attenuation, giving a “see-through” appearance. Distance between blue dots encompassing image denote 1 mm scale. Top view is looking down the loading direction (x_3).

Figure 6. Principal component ratios from (a) the proposed volumetric crack density tensor $\langle \alpha_{3-D} \rangle_V$ (see Eq. 4) and (b) the materials anisotropy tensor M (see Section 2.2) at -10°C .

Figure 7. Mean eigenvalues for all constant strain rate tests at -10°C (a) and $tr\langle \alpha_{3-D} \rangle_M$ for all constant strain rate tests (b).

Strain Rate	$\langle \alpha_{3-D} \rangle_V = \frac{1}{V} (l_1^3 \vec{e}_1 \vec{e}_1 + l_2^3 \vec{e}_2 \vec{e}_2 + l_3^3 \vec{e}_3 \vec{e}_3)$	$\langle \alpha_{3-D} \rangle_M = \frac{M}{V} (l_1^3 + l_2^3 + l_3^3)$
$1 \times 10^{-3} \text{ s}^{-1}$	$\begin{matrix} 8.30\text{e-}07 & 1.79\text{e-}08 & -3.79\text{e-}09 \\ 1.79\text{e-}08 & 8.09\text{e-}07 & 2.33\text{e-}09 \\ -3.79\text{e-}09 & 2.33\text{e-}09 & 2.64\text{e-}07 \end{matrix}$	$\begin{matrix} 8.26\text{e-}05 & -2.44\text{e-}06 & -8.21\text{e-}07 \\ -2.44\text{e-}06 & 8.40\text{e-}05 & 1.28\text{e-}06 \\ -8.21\text{e-}07 & 1.28\text{e-}06 & 1.77\text{e-}04 \end{matrix}$
$1 \times 10^{-4} \text{ s}^{-1}$	$\begin{matrix} 1.23\text{e-}06 & -2.74\text{e-}08 & 3.61\text{e-}09 \\ -2.74\text{e-}08 & 1.16\text{e-}06 & 2.20\text{e-}09 \\ 3.61\text{e-}09 & 2.20\text{e-}09 & 2.28\text{e-}07 \end{matrix}$	$\begin{matrix} 8.76\text{e-}05 & 2.72\text{e-}06 & -8.68\text{e-}07 \\ 2.72\text{e-}06 & 9.10\text{e-}05 & -1.32\text{e-}06 \\ -8.68\text{e-}07 & -1.32\text{e-}06 & 2.69\text{e-}04 \end{matrix}$
$1 \times 10^{-5} \text{ s}^{-1}$	$\begin{matrix} 4.38\text{e-}07 & -1.11\text{e-}08 & -5.34\text{e-}10 \\ -1.11\text{e-}08 & 4.62\text{e-}07 & 2.67\text{e-}10 \\ -5.34\text{e-}10 & 2.67\text{e-}10 & 7.32\text{e-}08 \end{matrix}$	$\begin{matrix} 6.47\text{e-}05 & 2.09\text{e-}06 & -1.96\text{e-}07 \\ 2.09\text{e-}06 & 6.24\text{e-}05 & 4.31\text{e-}07 \\ -1.96\text{e-}07 & 4.31\text{e-}07 & 2.13\text{e-}04 \end{matrix}$
$5 \times 10^{-6} \text{ s}^{-1}$	$\begin{matrix} 4.85\text{e-}06 & -3.18\text{e-}07 & 7.58\text{e-}09 \\ -3.18\text{e-}07 & 4.64\text{e-}06 & 1.29\text{e-}08 \\ 7.58\text{e-}09 & 1.29\text{e-}08 & 1.01\text{e-}06 \end{matrix}$	$\begin{matrix} 1.41\text{e-}04 & 1.28\text{e-}05 & -1.88\text{e-}06 \\ 1.28\text{e-}05 & 1.45\text{e-}04 & -1.13\text{e-}06 \\ -1.88\text{e-}06 & -1.13\text{e-}06 & 3.98\text{e-}04 \end{matrix}$
$2.5 \times 10^{-6} \text{ s}^{-1}$	$\begin{matrix} 1.76\text{e-}07 & -4.15\text{e-}08 & 2.73\text{e-}10 \\ -4.15\text{e-}08 & 1.73\text{e-}07 & 6.06\text{e-}11 \\ 2.73\text{e-}10 & 6.06\text{e-}11 & 2.16\text{e-}08 \end{matrix}$	$\begin{matrix} 4.68\text{e-}05 & 1.50\text{e-}05 & -2.27\text{e-}07 \\ 1.50\text{e-}05 & 4.72\text{e-}05 & -5.37\text{e-}07 \\ -2.27\text{e-}07 & -5.37\text{e-}07 & 1.83\text{e-}04 \end{matrix}$
$1 \times 10^{-6} \text{ s}^{-1}$	$\begin{matrix} 1.72\text{e-}07 & 9.85\text{e-}09 & 8.58\text{e-}10 \\ 9.85\text{e-}09 & 1.90\text{e-}07 & 5.10\text{e-}10 \\ 8.58\text{e-}10 & 5.10\text{e-}10 & 1.20\text{e-}08 \end{matrix}$	$\begin{matrix} 4.64\text{e-}05 & -3.25\text{e-}06 & -1.20\text{e-}06 \\ -3.25\text{e-}06 & 4.35\text{e-}05 & -2.36\text{e-}06 \\ -1.20\text{e-}06 & -2.36\text{e-}06 & 2.74\text{e-}04 \end{matrix}$

Table 1. Variants of the crack density tensor calculated with Eq. (4) and Eq. (5) from one test at each constant applied strain rate at -10°C.