



# Metrics for interpreting the microstructure of sea ice using X-ray micro-computed tomography



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## ABSTRACT

As the character and dynamics of sea ice change in a rapidly changing climate, it is critical to have a detailed understanding of the fine microstructure of sea ice. Advances in X-ray micro-computed tomography ( $\mu$ CT) technology have enabled non-destructive three dimensional analysis of the brine channel morphology with resolution down to several microns. In this study, we examine six ice cores collected from the Ross Sea, Antarctica. Metrics were developed to describe the shape, size, and topology of the brine channels and air pockets in sea ice. A cubic sub-sample measuring 6.0 mm on edge was found to be the representative elementary volume for sea ice  $\mu$ CT analysis with these metrics. All samples were observed to have vertically oriented cylindrical brine channels, with increased branching and connectivity observed at lower depths. The highest degree of vertical anisotropy was detected through the middle, with increased variability near the top and bottom of each core. Air pockets were found to be mostly spherical in shape, except vertically elongated in multi-year ice.

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## 1. Introduction

The use of X-ray micro-computed tomography ( $\mu$ CT) has exploded over the last decade due to rapid advances in instrumentation technology and accessibility through commercially available benchtop  $\mu$ CT systems (Ketcham and Carlson, 2001; Stock, 2008). The primary advantage of  $\mu$ CT is that it provides a non-destructive three-dimensional visualization and characterization of the internal features of multiphase and porous materials with spatial resolution down to several microns. Once the phases of a given material have been segmented, the analysis of the  $\mu$ CT data then produces quantitative measurements on topology and structure of the material. This has natural applications for describing microstructure mathematically and modeling microstructure-dependent properties of different porous media in fields varying from sedimentary rock and sea ice (e.g., Golden et al., 2007; Obbard et al., 2009) in the geosciences to bone analysis in biology (e.g., Campbell et al., 2007) to engineered composite materials (Naik et al., 2006).

$\mu$ CT utilizes the fact that phases of different density have varying absorption and transmission of X-ray radiation. Through the photoelectric effect, heavier elements are significantly more absorbing of X-rays than lighter elements, allowing separate phases to be

differentiated (Ketcham and Carlson, 2001). Since sea ice consists primarily of three phases (ice, brine, and air) with quite different densities, it is well suited for  $\mu$ CT structural analysis.

Prior to the introduction of  $\mu$ CT analysis, microstructural study of sea ice was limited to either destructive techniques or indirect measurements on a particular property of the ice (Weeks and Ackley, 1982). This presents challenges for highly detailed characterization of both air and brine inclusions, which vary both spatially and temporally with the thermal evolution of the sea ice. Brine inclusions are of primary importance because they provide critical pathways for the exchange of heat, gases, salts, and other chemical species, affecting processes such as nutrient delivery to microorganisms (e.g., Krembs et al., 2011), snow photochemistry (e.g., Grannas et al., 2007), salinity profile evolution (e.g., Cox and Weeks, 1975), and melt pond formation (Polashenski et al., 2012). Early work utilized thin section optical microscopy and found brine inclusions to be more elongated than air pockets (Light et al., 2003; Perovich and Gow, 1996). Cole and Shapiro (1998) further characterized the brine channel network, quantifying size and aspect ratios over time, and noting that in addition to the large vertically oriented channels, there are a number of side branches. Weissenberger et al. (1992) found similar results using a freeze-casting technique in combination with scanning electron microscopy, and recorded increased branching in granular ice relative to columnar ice. Finally, Eicken et al. (2000) achieved similar inclusion statistics analyzing sea ice microstructure with nuclear magnetic resonance imaging, and examined the thermal evolution of

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the fluid inclusions. All of these techniques however, are highly constrained by stereological and resolution artifacts when quantifying the fine-scale morphology of the brine network (Eicken et al., 2000).

The use of cooling stages has permitted  $\mu$ CT to be used to study the pore structure of cryospheric samples such as snow (e.g., Wang and Baker, 2013; Wålin et al., 2014), firn (e.g., Freitag et al., 2004; Schneebeli and Sokratov, 2004; Gregory et al., 2014) and ice cream (Pinzer et al., 2012). Pringle et al. (2009) analyzed CsCl-doped laboratory-grown sea ice and observed changes in brine channel connectivity at varying temperatures. Results supported percolation-theory predictions of critical anisotropic connectivity thresholds (Golden et al., 2007; Pringle et al., 2009). More recently, methods which involved analyzing field-collected sea ice have been developed (Maus et al., 2009, 2013; Obbard et al., 2009). Maus et al. (2009) developed methods using synchrotron  $\mu$ CT by first centrifuging samples and storing at sub-eutectic temperatures. Although this protocol could not distinguish between closed, brine-filled pores and ice, the open porosity was removed of liquid and thus, less-likely to be altered during storage and transport (Maus et al., 2009). Further work with this method provided insight into pore size distribution, permeability, and applications to modeling oil entrapment, although no critical percolation threshold was observed (Maus et al., 2013).

The purpose of this study is to establish a robust methodology for analyzing sea ice microstructure using  $\mu$ CT and introduce metrics for quantifying its morphology. The considerations, protocols, and metrics presented here however, can be adapted to the  $\mu$ CT study of any porous media. To demonstrate the capabilities of this methodology, we examine the microstructure of natural first-year and multi-year sea ice cores that were used in two companion papers (Lieb-Lappen and Obbard, 2015; Obbard et al., 2016). Reactive bromine species play a large role in tropospheric ozone depletion events, and Lieb-Lappen and Obbard (2015) examines the role of the blowing snow in the activation of bromine over first-year Antarctic sea ice. The brine channels in the sea ice are critical in providing a transport mechanism for the salts to reach the sea ice surface. The precise locations for the first-year ice cores were chosen to maximize the contact of blowing snow with the ice surface, while the multi-year ice core was used for comparison purposes. Obbard et al. (2016) analyzes the microstructural location of particular salts within the sea ice cores. Here we use  $\mu$ CT to study the microstructure of the sea ice cores and the transport processes through the brine channels, whereby linking the location of the salts (Obbard et al., 2016) to the photochemistry on the sea ice surface (Lieb-Lappen and Obbard, 2015).

In Section 2 of this manuscript we will describe the sample preparation and  $\mu$ CT methodology. We will then introduce the metrics used for microstructural quantification in Section 3. In Section 4, we will present our results from the six ice cores analyzed, providing detailed microstructural descriptions for each phase segmented (brine, air, and ice). Additionally, we will present our results from both a spatial variability and temperature sensitivity analysis, as well as our calculation of the representative elementary volume required for this analysis. In Section 5, we will discuss the physical significance of our results and conclude the manuscript in Section 6.

## 2. Methods

### 2.1. Sample preparation

Sea ice cores were collected in October–November 2012 from six different locations in the Ross Sea, Antarctica, as shown in Fig. 1. The first two sites, named Butter Point and Iceberg Site, were located on first-year ice about 5 km from the open ocean at distances of 35 km and 55 km from the Ross Ice Shelf, respectively. The measured thickness of the ice at these two locations were 1.78 m and 1.89 m, respectively. The next three sites were also on first-year ice and

located on a transect at distances of 6, 12, and 18 km southeast from the ice edge, with thicknesses of 1.70, 1.80, and 1.82 m, respectively. To compare the microstructure of first-year sea ice to multi-year ice, an additional ice core measuring 1.96 m in length was extracted 700 m off-shore from Scott Base, Ross Island. Immediately following each core extraction, we recorded the temperature profile at 10-cm intervals, and stored the cores in a  $-20^{\circ}\text{C}$  freezer at McMurdo station prior to shipping. All sea ice cores were transported at a constant temperature of  $-20^{\circ}\text{C}$  back to Thayer School of Engineering's Ice Research Laboratory at Dartmouth College with temperature monitored with data loggers and stored in a  $-33^{\circ}\text{C}$  cold room.  $\mu$ CT analysis was then completed on cubic sub-samples measuring 1.5 cm on edge that were extracted every 10 cm along the length of each ice core. Minimal brine drainage was observed during core extraction as the brine volume fraction is relatively low in early Austral spring, and no brine drainage was observed during  $\mu$ CT processing 6–12 months later.

Vertical thin sections of each core were cut and imaged using cross-polarizing lenses. The frazil ice fractions were 7.8%, 15.9%, 45.0%, 8.2%, 33.9%, and 21.9% for the Butter Point, Iceberg Site, Scott Base, and the three transect cores (6 km, 12 km, and 18 km), respectively. The columnar ice fractions were 28.3%, 56.6%, 26.2%, 30.0%, 22.6%, and 19.7%, respectively and the platelet ice fractions were 63.9%, 27.5%, 28.7%, 61.8%, 43.5%, and 58.5%, respectively. A more thorough description of the relative percentages of different ice types in each core is provided in the companion paper to this manuscript (Obbard et al., 2016).

### 2.2. Scanning

The components of a  $\mu$ CT system are an X-ray source, sample stage, a scintillator, and a photo detector. For this work, we used a Skyscan 1172  $\mu$ CT scanner that uses a sealed microfocus X-ray tube with a spot size of  $5\ \mu\text{m}$  to produce a fixed conical, polychromatic X-ray source. In order to choose the accelerating voltage for the X-ray source, it is helpful to know the expected absorption of the specimen, and ensure minimum transmission does not drop below 20%. For 30 kV X-rays, the mass attenuation coefficient for water/ice is  $0.3756\ \text{cm}^2/\text{g}$ , while for 40 kV X-rays it is  $0.2683\ \text{cm}^2/\text{g}$  (Hubbell and Seltzer, 1995). Since sea ice has an approximate density of  $900\ \text{kg}/\text{m}^3$  (Hutchings et al., 2015; Timco and Frederking, 1996), this leads to linear attenuation coefficients of  $\mu = 33.804\ \text{m}^{-1}$  and  $\mu = 24.147\ \text{m}^{-1}$  for 30 kV and 40 kV X-rays, respectively. The resulting X-ray transmission is expressed by the Beer-Lambert law:

$$I = I_0 e^{-\mu x} \quad (1)$$

where  $I_0$  is the initial beam intensity,  $\mu$  is the linear attenuation coefficient, and  $x$  is the distance traveled through the specimen. Since  $x \approx 0.015\ \text{m}$  for all samples, roughly 40% of the X-ray signal is attenuated for 30 kV X-rays and 30% of the X-ray signal is attenuated for 40 kV X-rays. Ice cores collected from Butter Point, Iceberg Site, and Scott Base were scanned at an accelerating voltage of 30 kV and a current of  $100\ \mu\text{A}$ . By increasing the accelerating voltage to 40 kV with a current of  $250\ \mu\text{A}$  for samples from the transect cores, we were able to reduce acquisition time, while maintaining adequate contrast. A test sample was run at both settings, and by implementing appropriate segmentation as discussed below, we showed that differences between the two scanning setups were negligible.

Each sample was frozen onto a Peltier thermoelectric cooling stage that could maintain temperature at  $-20^{\circ}\text{C}$ . Each sample was then rotated  $180^{\circ}$  about its vertical axis in  $0.7^{\circ}$  steps, and the transmitted X-rays were imaged at each step. The geometry and spot size of the X-ray source determines the acquisition time required and the spatial resolution of the scan (Landis and Keane, 2010). For the setup

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