Preliminary Results on the Characterization of Firn Using SEM and Micro CT

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ABSTRACT

Traditionally, firn has been characterized using quantitative optical microscopy or stereology paired with thin-sectioning techniques utilizing infiltration with a polymer. In practice these methods are low resolution, time consuming and involve complex and destructive specimen preparation. By using a scanning electron microscope (SEM) equipped with a low temperature stage to view firn one can obtain images at high resolution. Crystallographic information and the compositions of impurities can also be acquired through the use of electron back-scattered patterns and energy dispersive X-ray spectroscopy in the SEM, respectively. Three-dimensional images of the pore structure in firn specimens can be obtained through the use of a micro X-ray computed tomography. By pairing the SEM and micro CT it is possible to compare data between the two media.

Keywords: SEM; Micro CT; firn

INTRODUCTION

It has been shown that a scanning electron microscope (SEM) coupled with energy dispersive spectroscopy (EDS) and electron back scattered patterns (EBSP) can be used to document the microstructural location of impurities and crystallographic orientation of grains in ice (Baker *et al*., 2007; Obbard *et al*., 2007). These methods together can be used to fully describe the 2-D microstructure. It is also of interest to relate the 3-D microstructure of firn to the already well-developed 2-D analysis. In the past infusion of firn with Dimethyl phthalate or other polymers has been used followed by sectioning and optical examination (Albert, 2002; Rick, 2004). However, problems occur with this method namely the viscous liquid may not infiltrate small or closed off pores, which increase in frequency with depth (Baker *et al*. 2007). Recently, micro X-ray computed tomography (micro CT) has been introduced to ice sciences as a method for nondestructive 3-D data acquisition (Coleou *et al.,* 2001; Freitag *et al.,* 2004; Kerbrat *et al.,* 2007; Schneebeli *et al.,* 2004). In order to accurately analyze the microstructure obtained using micro CT a correct visualization technique is required. In this paper preliminary work is presented which uses the method first proposed by Kerckhofs *et al.* (2008) to validate thresholding techniques by thresholding the micro CT data using high-resolution 2-D images of the same specimens.

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EXPERIMENTAL METHODS

The methods used here are similar to those of Kerckhofs *et al.* (2008) yet applied to a cold environment. Both the SEM and micro CT were used to examine each specimen. The micro CT was used first for two reasons; the SEM specimen size is limited, and there is less chance of degrading the specimen surface due to sublimation.

Three firn specimens from the 1999 U.S. ITASE were analyzed from Core 99-2 from a depth between 39-40m (Figure 1). Specimens, which had dimensions of 1 x 1 x 1.5 cm, were cut perpendicular to the core axis, and either used for immediate observation or sealed in a small container stored in a -20^{o}C cold room to await analysis.

First, a Skyscan 1172 micro CT, equipped with a home-built cold stage that utilizes dry ice (Figure 2) was used to image the specimens. A small pixel range and long working distance were used during data acquisition. The data were reconstructed with the use of NRecon. A series of processing techniques including Gaussian smoothing and beam-hardening correction were applied. These tools are employed to ensure minimal reconstruction artifacts. Thresholding is built into the beam-hardening correction as applied to the image histogram. Two peaks are available in the histogram, a strong low-index peak representing the background and a smaller high-index peak corresponding to the solid phase (Figure 3). The upper contrast limit should be placed near the tail of the solid phase peak while the lower limit can be zero. Image reconstruction yields a stack of horizontal images correlating to the specimen, which are uploaded into a second program, CT Analyzer, to acquire 2-D and 3-D measurements from each horizontal image and to create a 3-D plot of the specimen. Image processing is included in this analysis. Cropping is the main technique however, if a shorter working distance is needed during acquisition, a despeckle algorithm is applied to minimize the grainy nature of the images.

Next, we used a field emission gun (FEG) XL-30 environmental SEM equipped with a Gatan cold stage and cryotransfer system to examine the specimens. The SEM was operated at a 15kV accelerating voltage with a 0.15nA electron beam. The specimen was held at -130^{o}C ±5^{o}C to ensure minimal sublimation. The cooling system consists of a gold-plated stage, an airlock and a temperature controller. The airlock is fixed to the SEM chamber and has a separate vacuum component, which can be vented or pumped independently for changing specimens. This is used to maintain high vacuum conditions and to prevent frosting in the main chamber.

Initially, for SEM examination the specimens were shaved flat with a sterilized razor in a -20^{o}C cold room under a HEPA-filtered hood. This ensures a clean and flat specimen surface, which is needed to maximize the correlation to the horizontal micro CT image stack. After inserting into the SEM piecewise images are taken over the specimen surface. A 50x magnification and 12mm working distance were used to minimize spherical aberration. A program, Image J, was used to create a mosaic from these images. 2-D Porosity was measured by digitizing the pore space and percent areal porosity was determined by dividing the area of the void space by the total area of the image.

Since all structural values are dependent on thresholding it is determined the most critical aspect of the entire process. Finding a threshold value can be difficult. Therefore we use the method put forth by Kerckhofs *et al.* (2008) and validate our micro CT threshold by correlating it to a high resolution 2-D optical image. Unlike Kerckhofs *et al.* (2008) we applied this thresholding technique independently to each specimen instead of over a range of specimens.

RESULTS

Three ITASE 99-2 specimens were examined as described above. Figure 4 shows binarized composite SEM images, binarized 2-D micro CT images, and the 3-D models produced from the micro CT data for specimens 1, 2 and 3. Note the good match between the SEM images and the 2-D micro CT images. The porosity determined from both the SEM and micro CT images are listed in Table 1. Note the agreement between the values from the two different techniques. The Table

also shows the internal surface/volume ratio and density also determined from the micro CT analysis. The 3D images were a useful aid in the qualitative image analysis.

SUMMARY

Although only preliminary results are shown in this paper we were able to determine a good correlation between the two imaging methods i.e. micro CT and SEM. Thresholding of the micro CT data is the most important and difficult step in this analysis. Hence, we used an SEM image to aid in the thresholding. The resulting data showed porosity values within 15% error from the two techniques. 3-D projections can also be created which provide a qualitative knowledge of pore connectivity and which will be useful for understanding gas permeability.

In the future we hope to focus on differences between winter and summer layers, documenting pore close off, and applications to corresponding permeability measurements. We also hope to include crystallographic data from EBSP analysis and impurity microchemistry by utilizing EDS. Lastly, it may be advantageous to define a fixed thresholding value from the micro CT to process all specimens instead of treating them separately as performed by Kerckhofs *et al*. (2008).

ACKNOWLEGEMENTS

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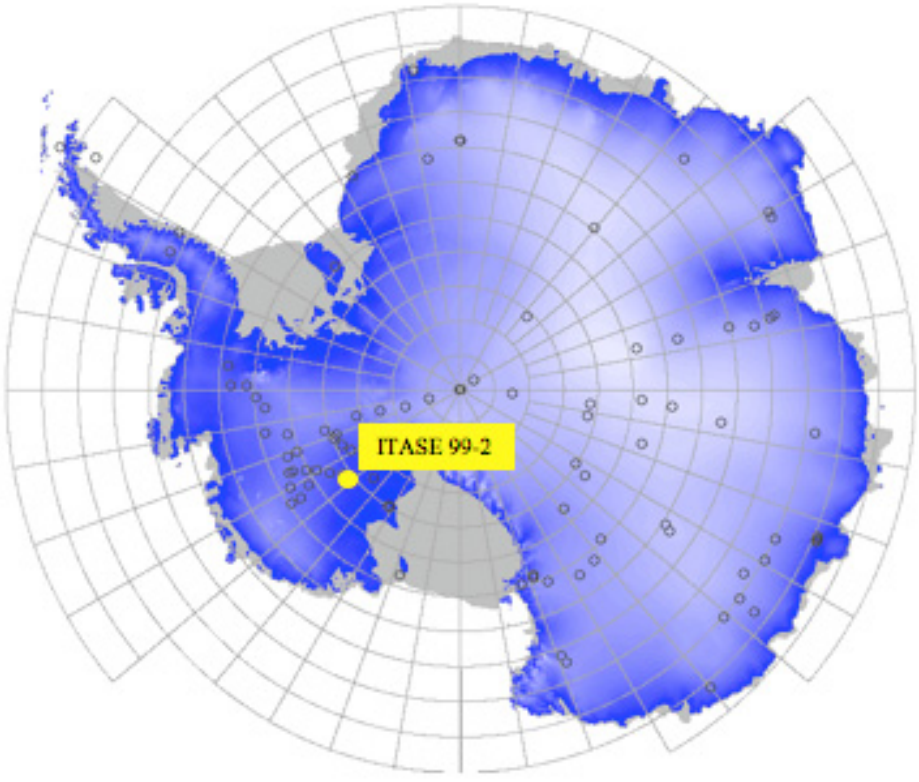

Figure 1: Location of the ITASE 99-2 core, used for analysis. From <http://www2.umaine.edu/itase/>

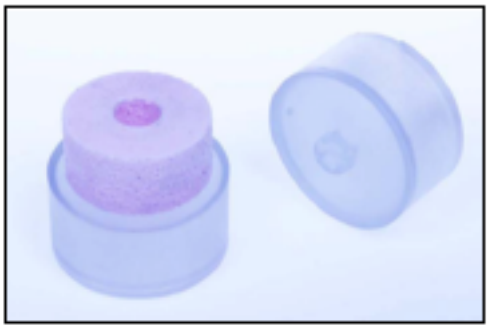

Figure 2: The micro CT cold stage. Dry ice is loaded in the top and bottom hollow containers and the specimen is place in the center region.

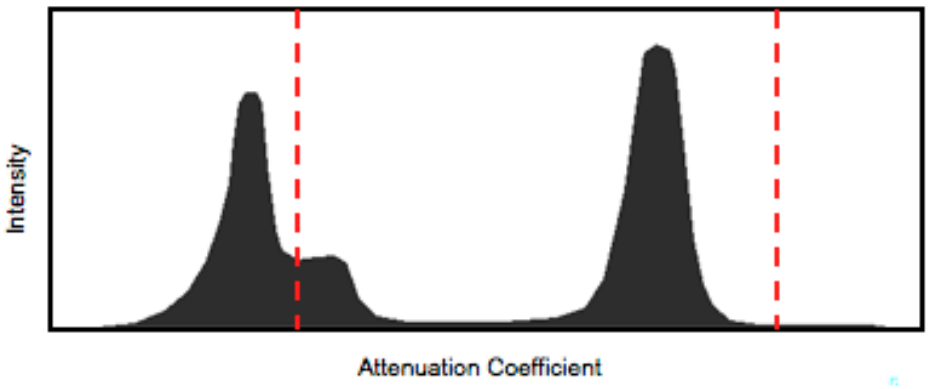

Figure 3: The image histogram thresholding technique from the NRecon program. Absorption values are on the x axes in units of attenuation coefficient, and the y axes has a relative intensity scale.

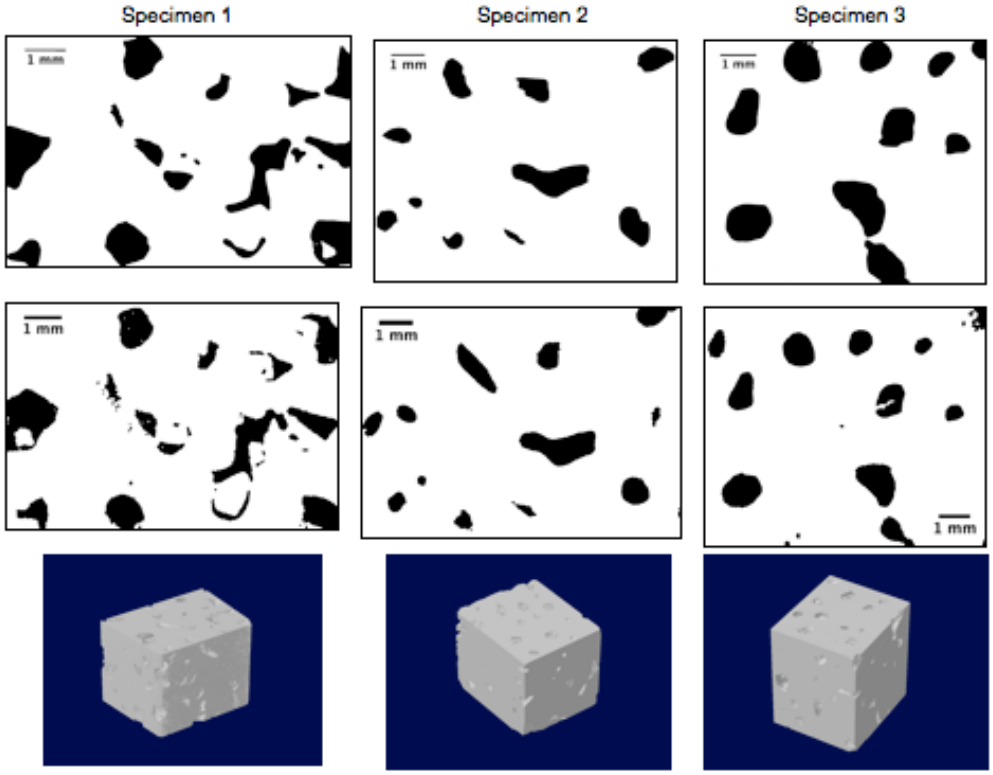

Figure 4: From top to bottom this shows the binarized montaged SEM image; the binarized micro CT image; and the 3D model produced from the micro CT data for specimens 1, 2, and 3 respectively.

Table 1. Comparison of the porosity determined from SEM and 2-D micro CT images.

Specimen	SEM % Porosity	Micro CT % Porosity	Surface / Volume (mm^{-1})	Density (kg/m3)
1	14.9	14.4	20.1	743
2	7.9	6.9	1.6	833
3	10.9	10	1.8	822

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