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Scaling ice microstructures from the laboratory to nature: cryo-EBSD on large samples.

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Electron backscatter diffraction (EBSD) has extended significantly our ability to conduct detailed quantitative microstructural investigations of rocks, metals and ceramics. EBSD on ice was first developed in 2004. Techniques have improved significantly in the last decade and EBSD is now becoming more common in the microstructural analysis of ice. This is particularly true for laboratory-deformed ice where, in some cases, the fine grain sizes exclude the possibility of using a thin section of the ice.

Having the orientations of all axes (rather than just the c-axis as in an optical method) yields important new information about ice microstructure. It is important to examine natural ice samples in the same way so that we can scale laboratory observations to nature. In the case of ice deformation, higher strain rates are used in the laboratory than those seen in nature. These are achieved by increasing stress and/or temperature and it is important to assess that the microstructures produced in the laboratory are comparable with those observed in nature.

Natural ice samples are coarse grained. Glacier and ice sheet ice has a grain size from a few mm up to several cm. Sea and lake ice has grain sizes of a few cm to many metres. Thus extending EBSD analysis to larger sample sizes to include representative microstructures is needed. The chief impediments to working on large ice samples are sample exchange, limitations on stage motion and temperature control.

Large ice samples cannot be transferred through a typical commercial cryo-transfer system that limits sample sizes. We transfer through a nitrogen glove box that encloses the main scanning electron microscope (SEM) door. The nitrogen atmosphere prevents the cold stage and the sample from becoming covered in frost.

Having a long optimal working distance for EBSD (around 30mm for the Otago cryo-EBSD facility) , by moving the camera away from the pole piece, enables the stage to move without crashing into either the EBSD camera or the SEM pole piece (final lens). In theory a sample up to 100mm perpendicular to the tilt axis by 150mm parallel to the tilt axis can be analysed. In practice, the motion of our stage is restricted to maximum dimensions of $\sim$100 by 50mm by a conductive copper braid on our cold stage.

Temperature control becomes harder as the samples become larger. If the samples become too warm then they will start to sublime and the quality of EBSD data will reduce. Large samples need to be relatively thin ($\sim$5mm or less) so that conduction of heat to the cold stage is more effective at keeping the surface temperature low. In the Otago facility samples of up to 40mm by 40mm present little problem and can be analysed for several hours without significant sublimation. Larger samples need more care, e.g. fast sample transfer to keep the sample very cold. The largest samples we work on routinely are 40 by 60mm in size.

We will show examples of EBSD data from glacial ice and sea ice from Antarctica and from large laboratory ice samples.