Characterizing the Crystallographic Orientation of Snow Crystals with Electron Backscatter Diffraction

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Introduction

Electron backscatter diffraction (EBSD) is an advanced materials characterization technique that has been used to determine the crystallographic orientation of glacial ice specimens, along with a plethora of other inorganic crystalline materials. However, due to the specific sample preparation requirements necessary for performing EBSD (i.e., flat, smooth, clean surfaces), characterizing porous and ephemeral materials such as seasonal snow and/or firn has always been a challenge. Thus far, our results show that plate-shaped surface hoar grains grow preferentially along the primary prism axis, the most favorable and lowest energy face in the Ice 1h crystal structure. Depth hoar crystals were found to be more complex, such that growth may vary greatly depending on the temperature gradient, vapor flux, and supersaturation within the snowpack. EBSD results show that depth hoar can grow along each of the growth planes (basal, primary, and secondary planes), while secondary electron images and optical microscopy reveals the complex step-like features associated with depth hoar crystals grown along the basal growth plane due to vapor deposition.

Methods

Casting

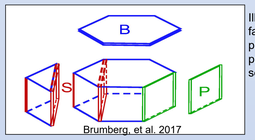
- Field collected depth hoar samples were casted with dimethyl phthalate (DMP) based on the methods of Schneebeli, M (2008) in the field location.
- Single grain crystals of field collected surface hoar, field collected depth hoar, and laboratory grown crystals were casted in a 20x20*mm* mold of DMP with added 20% carbon powder to visually accentuate the snow crystal during microtoming step

Sample Preparation

- Bulk samples are cut with a bandsaw to a size of ~ 20x20mm
- Bulk and single crystal samples are attached to a glass slide and microtomed to ~3-5mm, the optimum thickness to prevent charging during imaging and EBSD analysis.
- The sample is placed in a cooler containing dry ice and transported from the Subzero Research Laboratory (SRL) to the Imaging and Chemical Analysis Lab (ICAL) which houses the Field Emission Scanning Electron Microscope (FE-SEM).
- Samples are quickly slid onto the cryo-stage within the FE-SEM to prevent vapor deposition (frost) on the sample surface and immediately placed under vacuum
- Inherently there is some vapor deposition during transfer of the sample to the FEM, however it was observed that frost deposition was negligible to obtain EBSD patterning or sublimated off during the process of getting down to high vacuum (HV).

Technical

- Instrument: Zeiss Supra 55 VP FE-SEM
- EBSD System: Oxford HKL Channel 5
- Cryo-Stage Temperature: -160°C
- Working Distance: 15mm
- Forescatter Detector Distance: 175mm
- Accelerating Voltage: 20kV
- Imaging Detector: EBSD (backscatter) and Imaging (SE2)

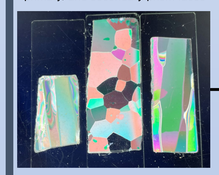


Brumberg, et al. 2017

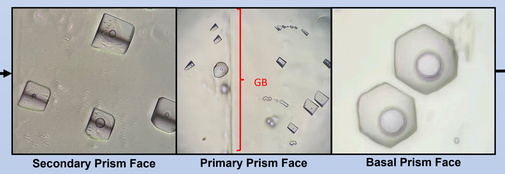
Illustration of major faces of a hexagonal prism. B, basal face, P, primary prism face, S, secondary prism face

Methodology for Laboratory Grown Depth Hoar

Laboratory grown bulk S1 columnar ice are cut into substrates along basal, primary, and secondary prism faces

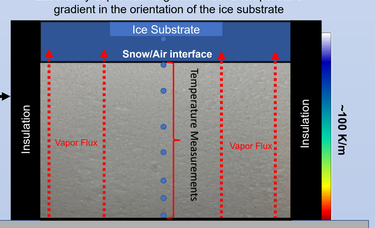


Samples undergo etch pitting to confirm ice crystal orientation



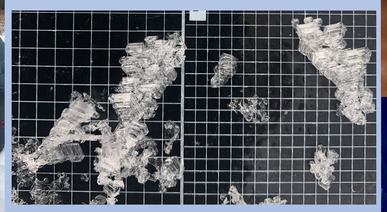
Secondary Prism Face | Primary Prism Face | Basal Prism Face

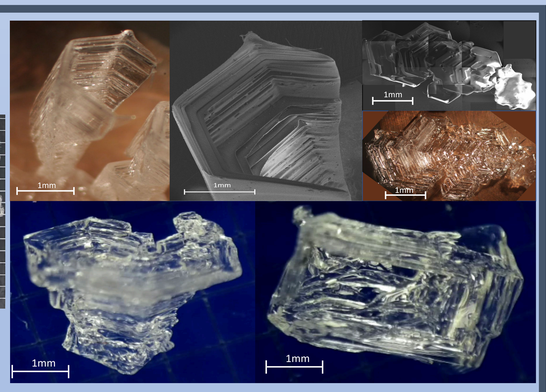
Laboratory depth hoar is grown under a temperature gradient in the orientation of the ice substrate



Temperature Table

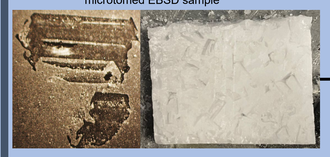
Field Collection and Microscopy (Optical & SEM)



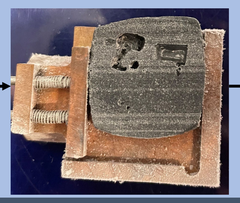


EBSD Sample Preparation

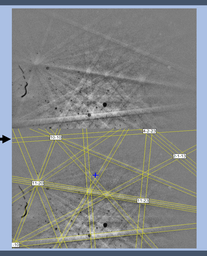
Single grain and bulk casted microtomed EBSD sample



Casted sample mounted on copper ingot ready for EBSD analysis



EBSD patterning results confirming viability of conducting EBSD on a porous material such as snow



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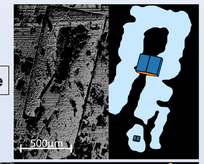
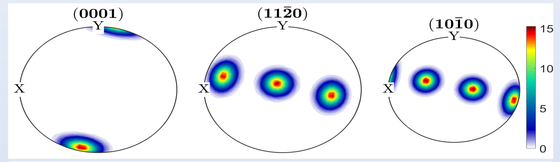
Results

EBSD on Bulk Casted Depth Hoar Crystals

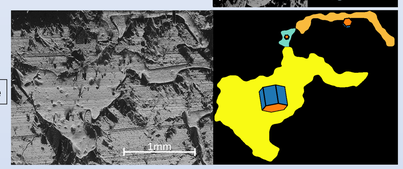
- Bulk field collected depth hoar samples were casted in the field and samples are cut and imaged via a backscatter detector to find desirable faces for EBSD mapping of grains.
- Analysis found depth hoar cups grown along the secondary and primary prism growth planes.

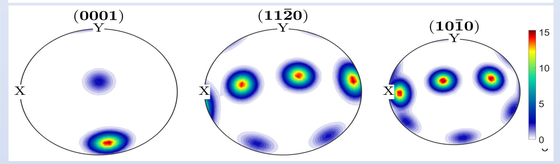
Secondary Prism Face





Basal Face

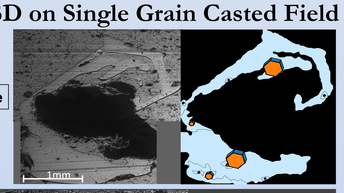


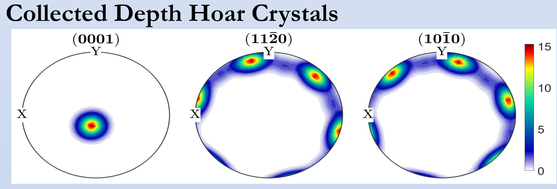


EBSD on Single Grain Casted Field Collected Depth Hoar Crystals

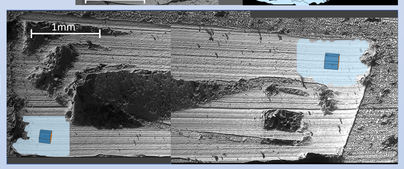
- Single grain field collected depth hoar with hexagonal and rectangular shaped cups were casted with the growth face oriented up.
- This analysis confirms that hexagonal cups grow along the basal face and rectangular shaped depth hoar cups can grow along the primary prism plane.

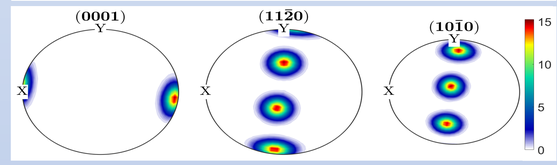
Basal Face





Primary Prism Face

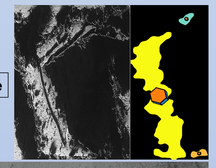


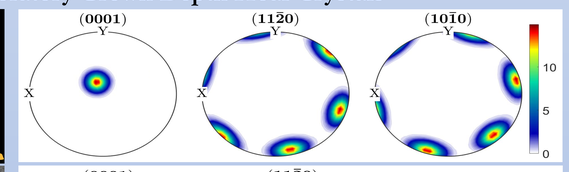


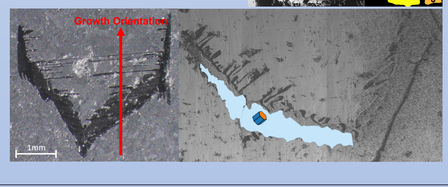
EBSD on Single Grain Casted Laboratory Grown Depth Hoar Crystals

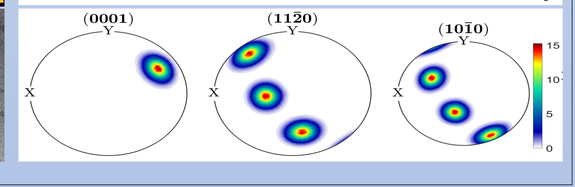
- Laboratory depth hoar was grown on a substrate oriented along the basal face.
- Due to the fragility of crystal attachment to the substrate, grains were taken off at the base and casted in the approximate orientation of growth and ~90° from the growth plane.

Basal Face



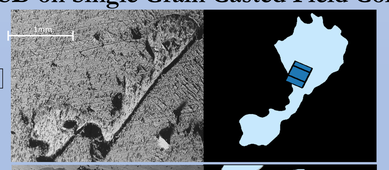


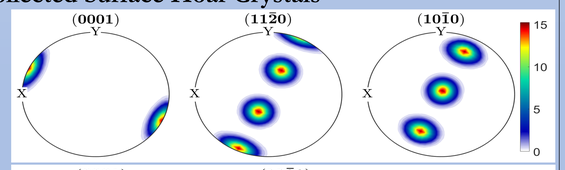




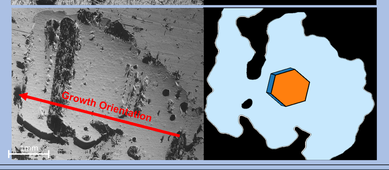
EBSD on Single Grain Casted Field Collected Surface Hoar Crystals

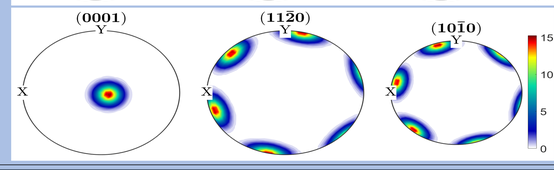
Primary Prism Face





- Confirmation of surface hoar growth along the energy preferred primary plane.
- Single grain depth hoar crystals were casted with a-axis oriented along growth plane and 90° (basal face).





Future Research in the SRL & ICAL

- EBSD mapping of different laboratory grown depth hoar on substrates with known orientations.
- With the addition of Montana State's new EBSD system, this will allow for increased mapping capabilities of bulk field collected depth hoar to understand depth hoar chain growth rather than single grain analysis.

References

1. A. Brumberg, et. al, "Single-crystal Ih ice surfaces unveil connection between macroscopic and molecular structure," Proceedings of the National Academy of Sciences, vol. 114, p. 5349–5354, 2017.
2. D. J. Prior,et. al, "Making EBSD on water ice routine," Journal of microscopy, vol. 259, p. 237–256, 2015.
3. M. Schneebeli, et. al, Casting Snow Samples with Phtalate, 2008.