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Cryogenic EBSD: a technique to preserve a stable surface in a low pressure SEM to characterize ice microstructure

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Electron backscattered diffraction (EBSD) is an invaluable method for the characterization of microstructures in all kinds of materials. For example, characteristic geometries of subgrain boundaries in combination with their lattice rotation configurations can be interpreted in terms of dislocation activity.

The probable slip systems responsible for subgrain boundaries in ice can be determined using EBSD, providing the subgrain boundaries can be localized in the coarse grained natural ice and small scanning electron microscope (SEM) samples, and providing the subgrain boundary geometry can be characterized precisely. This can be realized by observation of etch features imaged with light microscopy (LM microstructure mapping[1]). The surface then has to be retained during EBSD data acquisition in an SEM.

Retention of the etch features requires that the ice surface is stable. Depending on the pressure and temperature, sublimation of ice can occur. The equilibrium temperature for a low pressure SEM ($\sim$1x10^{-6} hPa) is about -112°C. Operating at higher temperatures causes sublimation. Although charging of uncoated ice samples is reduced by sublimation, which was used as the charge minimizer in previous EBSD studies on ice, important information contained in the etch features are removed as the surface sublimes.

We present an alternative technique for collecting EBSD data on stable ice surfaces in a low pressure SEM using a phase diagram for selection of measurement conditions[2]. We found that operating at temperatures $<$–112°C reduced sublimation so that the original etch surface features were retained. Charging, which occurred at temperatures $<$–112°C and low pressures, was reduced by defocusing the beam. Spatial resolution and angular resolution with the EBSD and SEM conditions of our alternative technique were examined (0.5 μm to 3 μm, and $<$0.7° after orientation averaging). Excellent agreement was obtained between light microscopy etch features and EBSD mapped microstructures.

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[2]Weikusat, I.; de Winter, D. A. M.; Pennock, G. M.; Hayles, M.; Schneijdenberg, C. T. W. M. Drury, M. R. Cryogenic EBSD on ice: preserving a stable surface in a low pressure SEM. J. Microsc., 2010, doi: 10.1111/j.1365-2818.2010.03471.x