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Orientation Mapping in Polycrystalline Ice Using Electron Backscatter Patterns

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We recently demonstrated, that the orientations of crystals in polycrystalline ice could be determined with a higher angular and spatial resolution and more rapidly than any currently-used method by using electron back-scattered patterns (EBSPs) acquired from uncoated polycrystalline ice in a cold-stage equipped scanning electron microscope (SEM) [1]. The technique was originally developed using an older Zeiss 932 thermionic emission SEM operated in high vacuum mode (5 x 10^{-4} Pa) and equipped with a custom-built cold stage cooled by cold N_2 gas.

The acquisition of a new FEI XL30 field emission gun environmental SEM capable of delivering a much smaller but lower-current electron probe and the requirement that the SEM be run in environmental mode (13-17 Pa) when investigating ice necessitated a reassessment of the EBSP-based technique. We focused primarily on enhancing the quality of the EBSPs obtained with a smaller-current probe in a significantly poorer vacuum by designing a new type of cold stage (Figure 1) that allowed a series of procedural changes. The particular characteristics and advantages of the EBSP-based orientation method are discussed in [1]. We note that while the quality of the EBSPs obtained with the FEG SEM in environmental mode is evidently poorer compared to that of the patterns obtained in the Zeiss SEM in high vacuum mode, the EBSPs in the former case are still well defined and suitable for automatic indexing.

Three grains meeting at triple junction in fresh-water laboratory ice were imaged in the FEG SEM using a 15 KV beam (Fig. 2A). The pressure in the chamber was 17 Pa. Prior to imaging the surface of the specimen was shaved flat and was free of scratches and other macroscopic imperfections. The specimen (20 mm x 10 mm x 3-4 mm) was stored in dry ice for 3-4 h before being imaged. The temperature of the cold stage and removable brass holder (Fig. 1B) were maintained constant at a constant temperature of -75^oC (within $\pm 1^oC$) during the entire period using a PID controller. Given the pressure in the SEM chamber (17 Pa), this temperature ensured that charge from the 15KV electron beam did not build up excessively on the specimen surface but was largely removed through sublimation. These conditions allowed both manual and automatic collection of EBSPs.

Figure 2B shows an orientation map from the area marked in Fig. 2A. To create the map the beam was translated on the specimen's surface in 10 μm increments across a 69 x 54 point rectangular grid. At each point, an EBSP was automatically acquired and indexed to determine the local lattice orientation. Three indexed EBSPs corresponding to the bulk of each grain are shown in Figure 2C.

References

[1] D. Iliescu et al., Microscopy Research and Technique. 63 (2004) 183.
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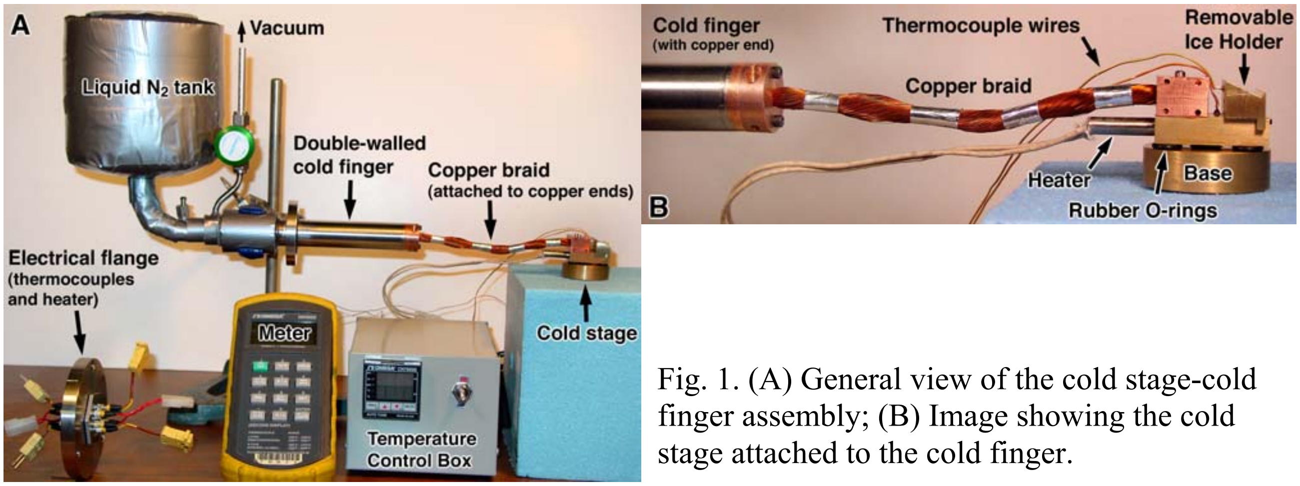
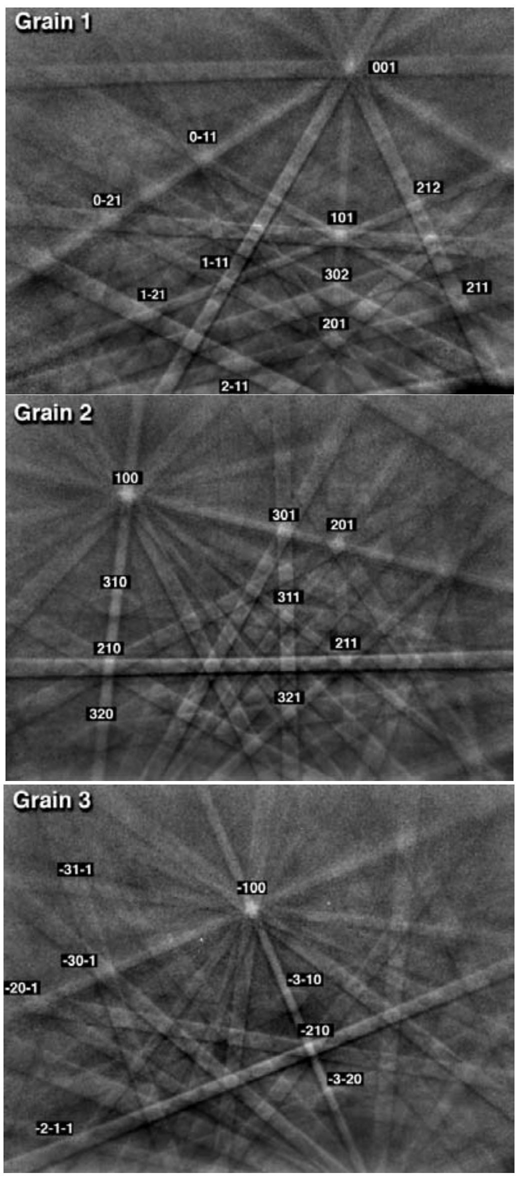


Fig. 1. (A) General view of the cold stage-cold finger assembly; (B) Image showing the cold stage attached to the cold finger.



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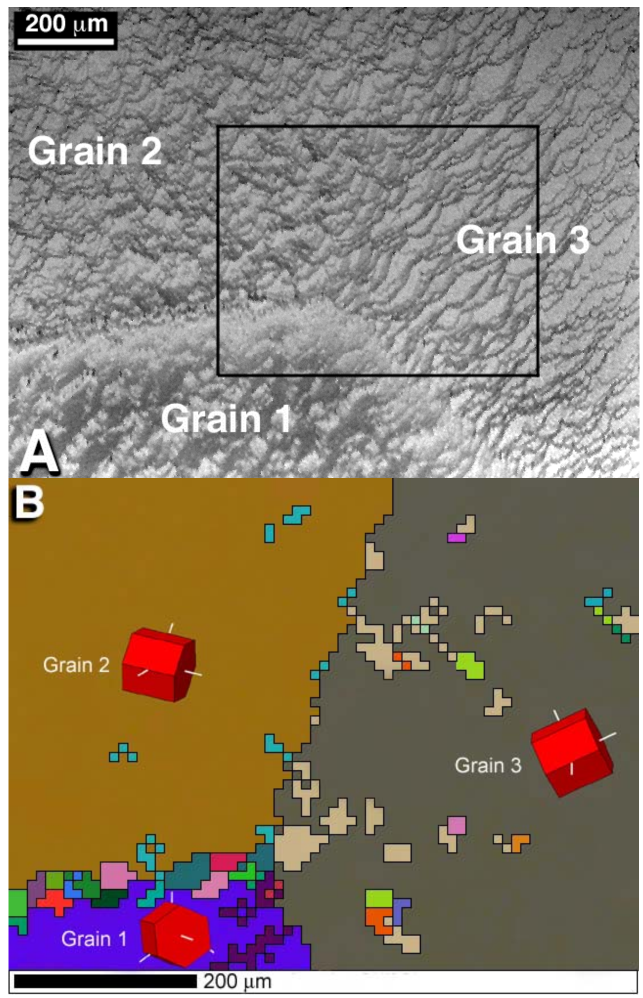

Fig. 2. (A) SEM image of a triple junction in laboratory ice; (B) orientation image from the rectangular area marked in (A). The orientation of the hexagonal unit cell in each grain is shown in insets. The orientations were computed from the EBSPs shown in (C) with respect to the specimen's surface coordinate system.