Dislocation density measurement in artificial polycrystalline ice by X-ray diffraction

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Ice sheets in polar regions such as Antarctica and Greenland flow into the ocean relating to plastic deformation caused by the gravitational effect of its own weight. Therefore, there are concerns about sea-level rise and associated global environmental changes. The understanding of the ice sheet flow is hence important in predicting the future global environment. It is usually accepted that the ice sheet is flowing due to creep deformation[1)]. When ice undergoes the creep deformation, dislocations are introduced into the crystals as the origin of strain. So far, the dislocation density in ice has been confimed by an image analysis technique, so-called X-ray topography, but it could not be applied when the dislocation density is higher than 1×10^7 m^{-2} [2)]. Therefore, an alternative method of the dislocation density measurement by X-ray diffraction is highly expected. In this study, the dislocation density in artificial ice is measured to investigate the mechanical properties of polycrystalline ice.

We prepared artificial ice samples with pure water (18.2 MΩ·cm). The samples were prepared by spraying pure water maintained in a spray bottle onto liquid nitrogen to prepare powdered ice and consolidating by applying pressure at 70 MPa and temperature at −10 ˚C for 1 h. For comparison of dislocation density measurements, creep tests of the samples were performed using a uni-axis creep test device. The apparatus was installed in a freezer maintained at -20 ˚C. The samples were set at the apparatus and deformed by applying a constant stress of 2 MPa. The samples before and after the creep test were measured using an X-ray diffraction (XRD) instrument (Rigaku, UltimaIV) with Cu-Kα radiation (0.15406 nm), and the obtained X-ray peak profiles were fitted with a Lorentz function. From the full width at half-maximum, the size and strain in the crystals are theoretically separated; the latter provides us the information of the dislocation density.

A diffraction peak of 102 was detected by the XRD. However, depending on the used ice samples, an extra peak which usually did not appear in Powder Diffraction File (PDF : 01-085-1394) of the ideal ice crystal (I_h, space group: P63/mmc, a = 0.4506, c = 0.7346 nm and c/a = 1.63) was detected on the vicinity of the 102 peak as a superimposed continuous peak. In order to understand the origin of the appearance of an extra peak, the relationship between the stage height and the Bragg angle was carefully analyzed. By changing the height of the sample stage, it is found that the related peak positions are shifted towards higher or lower Bragg angles; when the height is reduced, the peak is shifted to lower Bragg angle.

In this presentation, we discuss the possibility of the application of the dislocation density measurement using the conventional X-ray diffractometer.

References

1) Faria, S.H., Weikusat, I., and Azuma, N., 2014, The microstructure of polar ice. Part II：State of the art, J. Struct. Geol., 61, 21-49.
2) Cullen, D., Baker, I., 2002, Observation of sulfate crystallites in Vostok accretion ice, Mater. Charact., 48, 263-269.