Application of small angle X-ray scattering to fine particles dispersed in crept artificial ice samples

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It has been reported that flow of ice sheet is related to plastic deformation of ice, and the ice is basically deformed by slip which is mainly occurred on the basal plane of the ice crystal in the case of ice I_h (Faria et al., 2014). When the slip is activated, dislocations start moving on the basal plane in addition to non-basal planes when the deformation temperature is increased. If the grain size is large enough, then Glen's flow low could be the rate controlling mechanism, indicating that the stress exponent, n, appearing in the strain rate equation generally becomes 3 (Glen, 1955). However, recently it has been clarified that "cloudy bands" appear in deep ice cores such as EDML in which relatively small grains are formed due to presence of small impurities relating to mineral and other origins. This is due to the fact that the impurities segregate at the grain boundaries (GBs), preventing the grain growth during creep deformation. Such fine grains usually cause soft behavior with creep deformation, and therefore the deformation behavior of the impurity containing ice samples should be addressed. Although impurities having > 500 nm can be detected using optical microscopy and other techniques, tiny particles are usually difficult to distinguish due to the resolution issue. Hence, we have prepared artificial ice samples with nanoparticulates using a sintering technique which is composed by amorphous SiO_2 (0.01, 0.1 and 1 wt.%), and then applied creep deformation to them at 0.5 MPa of compressive stress and -5ºC. The average grain diameters of the ice matrix are approximately less than 100 μm: the grain size in the cloudy bands is simulated.

Interrupted creep deformation was carried out so as to identify the size of the nanoparticulates using small angle X-ray scattering (SAXS). This technique can detect nanoparticulates (Guinier, 1994) whose diameter is ranging from a few nm to 100 nm. The used sample sizes for the SAXS measurements are fixed at a diameter of 23 mm and the height of 2 mm. The used X-ray source was Cu Kα and the SAXS measurements were carried out by Rigaku Nanoviewer. It is found that the strain rate increases when the concentration of SiO_2 in artificial ice increases. However, the strain rates do not increase in all the SiO_2 concentration range during creep, although the strain increases. This behavior is totally different from those observed in coarse ice grains which obey Glen's flow low. The grain growth of the ice matrix does not occur during the creep, but the agglomeration of SiO_2 particles appear even in the as-sintered samples. The agglomeration is once decreased when the strain reaches 5% due to the fragmentation, and then again the size increases, for example 70 nm in the 0.1 wt. % SiO_2 added sample. These results indicate that "superplastic-like" deformation with GB diffusion in addition to GB sliding may occur in the fine grained artificial ice samples—the creep mechanism is different from Glen's flow low.

References

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