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Structure, Chemistry and Properties of Grain Boundaries in H_2SO_4-Doped Ice

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Polycrystalline ice containing various concentrations of H_2SO_4 from 10-1000 p.p.m. were grown from sulfuric acid-doped water. For examination in the SEM, the surface of each section was shaved flat with a clean razor blade under a HEPA-filtered, laminar-flow hood. The section was then frozen onto a brass plate and mounted onto a cold stage. The cold stage was cooled using cold, flowing nitrogen gas that had passed through a liquid nitrogen-cooled heat exchanger. The temperature of the stage was monitored using a thermocouple mounted near the specimen. The specimens were held at 158 K ± 5 K and examined at 10 kV using a JEOL 5310LV SEM, equipped with a PGT IMIX EDS system utilizing a pure germanium, aluminum-coated polyimide thin-window detector. The EDS was used in a light element optimization mode that enables elements as light as boron to be detected. The EDS data are all for 100,000 total counts. Secondary electrons were used for imaging. Although ice is non-conducting, at 158 K there is sufficient sublimation from the ice to produce enough ionized water molecules around the electron beam to neutralize charging of the specimen surface. Thus, the ice did not need to be coated. Prior to examination in the SEM, the ice specimens were allowed to sublimate at 253 K in small sealed containers Sublimation was also performed in the SEM chamber at temperatures between 158 K and 213 K.

SEM/EDS examination showed that the sulfuric acid strongly segregated to the grain boundaries during the ice growth. The sulfuric acid eutectic was evident as a bright region along the grain boundaries and as a sulfur peak in EDS data from this region, see FIG. 1.

Direct current electrical resistivity measurements were performed on both the grain boundaries and in the nearby lattice at 263 K in this polycrystalline, sulfuric acid-doped ice. Platinum-coated spade-shaped copper electrodes were placed ~10 mm apart and a one-volt potential was applied. The results show that the grain boundaries have a higher initial conductivity than the lattice by over an order of magnitude. The current flowing through the grain boundaries decays with increasing time, but even after one hour it is still larger than the current flowing through the lattice, by a factor of 3. FIG. 2 shows an example of this behavior. The results are discussed in terms of the polarization of the electrodes and the presence of a thin liquid layer at the grain boundaries. The relaxation time associated with the decay of grain boundary current was used to estimate the response to an alternating current. The applications of these measurements to natural ice are discussed.

In addition, single crystals of both sulfuric acid-doped ice and undoped ice were indented at 258 K with a steel ball. They were annealed at 271 K during which recrystallization occurred around the indentation. The results of attempts to measure, using EDS, whether sulfuric acid is swept up by the migrating grain boundaries during recrystallization will be presented. The implications of these observations both for the effects of impurities on recrystallization and for the redistribution of impurities by recrystallization will be discussed.

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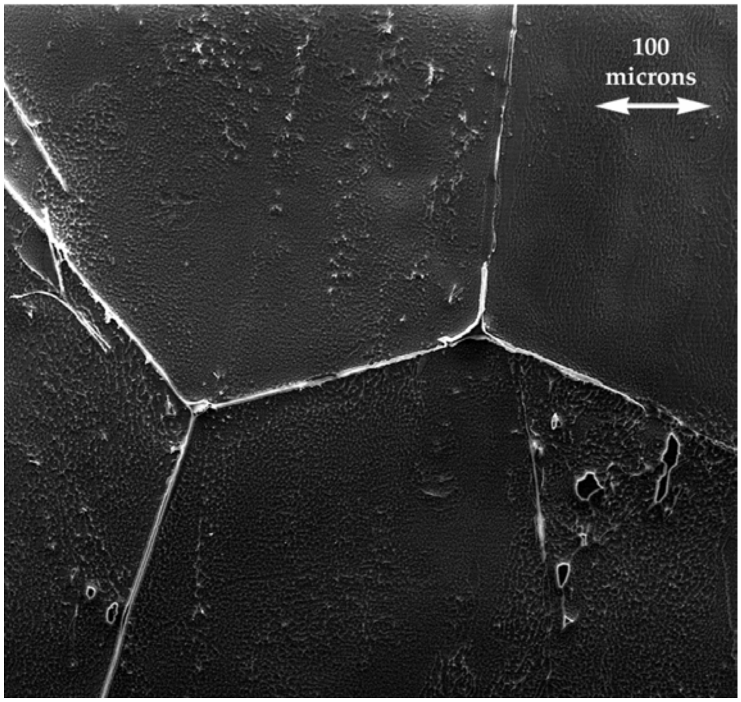

FIG. 1. SEM image of grain boundaries in ice doped with 1000 p.p.m. H_2SO_4.

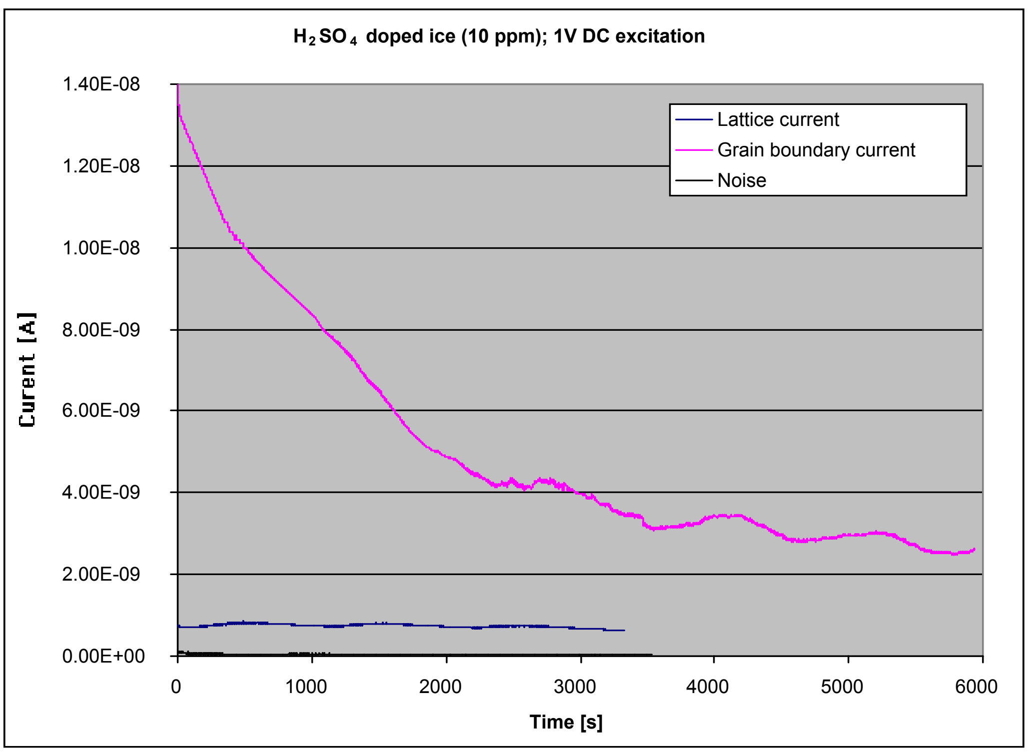

FIG. 2. Current as a function of time along a grain boundary and in the lattice in ice doped with 10 p.p.m. H_2SO_4 after a 1 V d.c. potential was applied through Pt electrodes spaced 10 mm apart.

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