Microstructural Characterization of Ice by Etch-Pitting Replication Method

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This study aims to develop a method for microstructural characterization of ice, by improving the etch-pitting replication method. Previous studies (Higuchi, 1957; Sinha, 1977) derived a method of reproducing surface features of ice by applying a dilute solution of polyvinyl formar (Formvar) in ethylene dichloride to the ice surface. When it dries, an inverse replica of the ice sample surface is created. Negative features like grain boundaries become ridges and favorably oriented dislocations intersecting the surface become protrusions, called whiskers. The method will allow us to use an ambient temperature SEM to image the replicas and characterize ice samples at high resolution. The SEM images can then be analyzed using imaging software to quantify grain size and dislocation density. In our study, we sought the optimal recipe for our sample dimensions and cold room conditions. Building upon Sinha's method, we ran through many iterations of the technique. To create the optimal film that captures microstructural details but is coherent enough to be peeled off the sample surface, we varied conditions of the process such as the concentration of the solution (0.25% to 5%), the number of layers (1 to 5 layers), the interval of drops to create each layer (a half hour to one day), and the surrounding environment (freezer temperature, humidity, vacuum). Our tests to date have determined that the optimal recipe involves a two-stage process of Formvar application: 1% solution allowed to dry for several hours, followed by application of a 3% solution. The replica is allowed to cure overnight in a walk-in freezer (-20°C) under vacuum. We will show microstructural images of the successfully created replica films observed by SEM, which show grain boundaries clearly and dislocations in crystals as whiskers, and will discuss future applications of the method.