Low-Temperature EBSP and EDAX analyses on ice and gas hydrate microstructures

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Cryogenic experiments with the Electron microscope on ice and gashydrates became a standard analytical tool in geoscience during the last years [1]. Cryogenic experiments down to –190°C at varying temperatures and pressures are carried out in a FE-SEM Quanta 200 FEI, which is equipped with EDX and EBSP detectors in order to investigate structural properties (Fig.1) The FE-SEM allows experimental conditions at low acceleration voltage without charging and damage of the sample surfaces on anorganic and organic materials. Different examples of the use and the analytical possibilities of cryogenic in-situ experiments at varying temperature and vaccum conditions will be given: (1) The study of natural and synthetic ice surfaces and grain boundary properties at the nanometer scale and their in-situ alteration by sublimation give insight into the possible mechanisms of snow crystal metamorphism and ice recrystallization, (2) structural changes in the microstructures of natural and synthetic gas hydrates. Recently, dynamic investigations with the cryo SEm allow the observations of sturctural changes during the experimental run: (3) in-situ study of structural changes in cooled biological samples at low vacuum.

In order to distinguish the specific microstructures of ice and gas hydrates it was necessary to have efficient EDAX analyses of light elements e.g., carbon. Therefor a lot of studies have been carried out to get informative EDAX analyses of carbon at a high spatial resolution and at low temperatures down to –190°C.

For the EBSP analyses of ice a critical problem had to be solved to avoid the EBSP detector acting as an anticontaminator. Therefor, a pretilted cryo sample holder has been constructed in the Department, which works at 15° and 20°. So, for the critical EBSP pretilting of the sample at an angle of 70° it is only necessary to tilt the whole cryo stage assemblage at an angle of 55° resp. 50°. This is efficient in order to get a sample surface which is free of secondary ice contamination as well as to avoid ice condensation along the EBSP detectors and finally to get a good EBSP pattern of ice.

1. W.-F. Kuhs, A. Klapproth, F. Gotthardt, K. Techmer and T. Heinrichs (2000): The Formation of Meso- and Macroporous Gas Hydrates, Geophys. Res. Lett **27(18)** (2000) 2929 - 2932.
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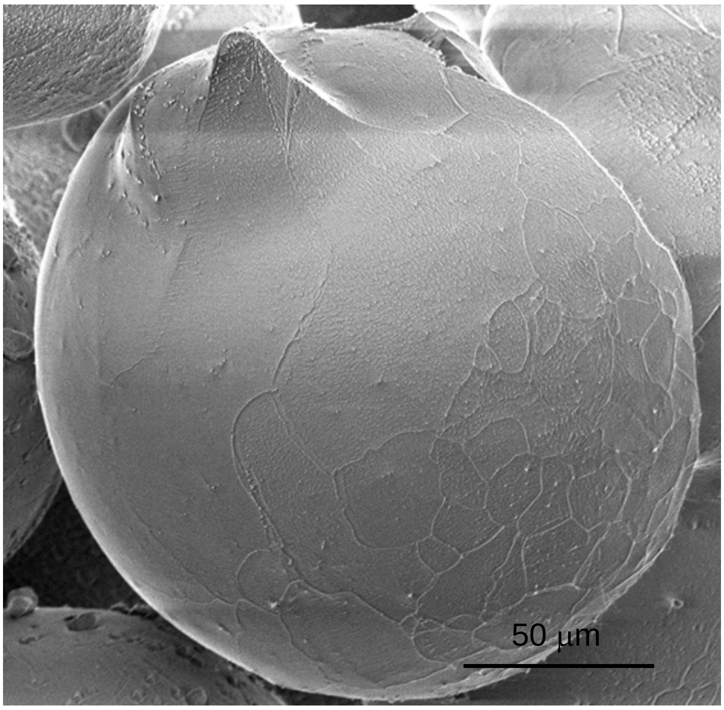

Figure 1 Ice spheres with decorated grain boundaries

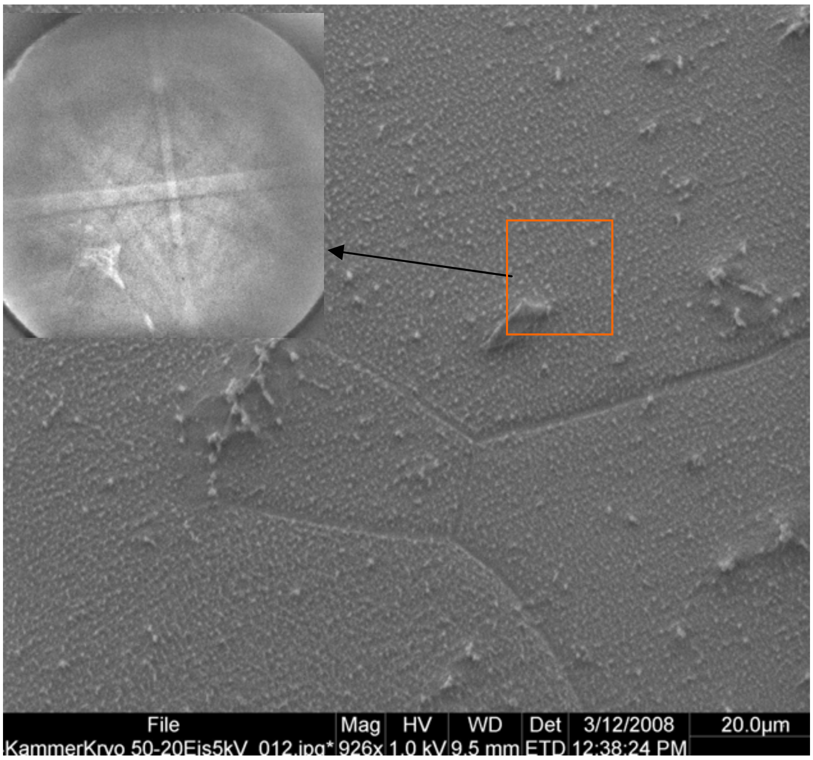

Figure 2 EBSP pattern of ice grains.

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