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X-ray Crystal Truncation Rod Measurements of Calcite/Water Interfaces at Elevated Temperature

Hiroshi Sakuma[1,*] Henning O. Sørensen[2], Jun Kawano[3], Naoki Nishiyama[1], and Hironori Nakao[4]
[1]National Institute for Materials Science, Tsukuba 305-0044, Japan
[2]University of Copenhagen, Universitetsparken 5, DK-2100 Copenhagen, Denmark
[3]Hokkaido University, N21 W10 Kitaku, Sapporo, Hokkaido 001-0021, Japan
[4]Condensed Matter Research Center and Photon Factory, Tsukuba 305-0801, Japan

1 Introduction

The structure of mineral/liquid interfaces can be modified at elevated temperatures by adsorption of dissolved ions on the mineral surface. Structural changes may alter the wettability of mineral surfaces from water wet to oil wet [1], and are therefore especially important for enhanced oil recovery. We have determined the structure of mineral/liquid interfaces using the technique X-ray crystal truncation rod (CTR) scattering. This method is a powerful tool for observing the electron density profile vertical to the interface.

In this study we measured the CTR profiles of the calcite {10.4} surface in a NaCl solution at 298 and 358 K. Calcite ($CaCO_3$) is the main constituent of chalk and other limestone rocks.

2 Experiment

Calcite spar single crystals were cleaved prior to the measurements and were immediately immersed in $CaCO_3$ saturated 0.5 M NaCl aqueous solution for a few hours. The liquid was degassed before the measurements to avoid bubble formation in the sample cell at elevated temperature. We have developed a thin-film sample cell for controlling the temperature of the sample up to 373 K. This cell reduced the evaporation of liquid compared to our previous sample cell [2]. The cell was covered using a polyimide film 7.5 μm thick to prevent the solution from evaporating.

Specular X-ray CTR scattering measurements were performed at the BL-4C beamline at the Photon Factory, KEK, Japan. A monochromatic X-ray beam of 11.0 keV was used. The specular X-ray CTR profile was measured for the natural cleavage plane {10.4}. The integrated and background corrected scattering intensities were subsequently corrected for Lorentz, polarization, attenuation, area factors, and rod interception effect. The intensities were converted to structure factors, $|F|$. The analysis was performed using in-house program based on the ANA-ROD code [3].

3 Results and Discussion

The specular CTR profiles of calcite/0.5 M NaCl solution interface measured at 298 and 358 K are plotted in Fig.1. No significant difference can be seen between the profiles determined at 298 and 358 K, indicating that the interfacial structure at 358 K was similar to the one at 298 K. This is consistent with the experimental results, where no difference in oil recovery was found by the imbibition of NaCl solution into a porous chalk rock [4]. Our results also imply that the sample cell can hold aqueous solution during the CTR measurements at elevated temperature.

The effect of temperature under various salt concentrations and chemical compositions will be investigated using this sample cell in future experiments.

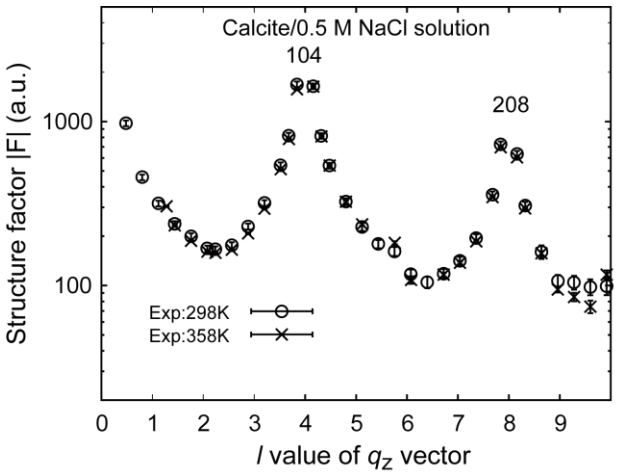

Fig. 1: Specular X-ray CTR profiles of calcite/0.5 M NaCl solution interface at 298 and 358 K.

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* SAKUMA.Hiroshi@nims.go.jp