

Observations of NaCl Crystal Growth in Solution Using Environmental Transmission Electron Microscopy

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Summary

Crystallization of atoms and/or molecules is utilized in various fields such as science and technology, biology, and the environment and it is important to elucidate its microscopic mechanism. In recent years, progress has been made in elucidating crystal nucleation in liquids at the atomic level using molecular dynamics simulations and it was reported that nucleation and crystallization in liquid consists of two steps: concentration fluctuation leading to a creation of high concentration region, followed by spatial ordering. However, in-situ observation of crystal growth in liquid with high spatial resolution is difficult and direct observation of high concentration regions in the two-step process has not been realized. In this study, we performed in-situ observation of NaCl crystal growth in liquid at nanometer scale using transmission electron microscopy. A liquid cell was used to hold solution samples in the electron microscope.

The observed crystals of several nanometers were constantly changing their shape due to reactions on the surface, but even after 150 seconds of electron irradiation at an electron dose of approximately $4 \times 10^4 \text{ e}^-/\text{nm}^2 \cdot \text{s}$, the structure remained unchanged. It was not significantly destroyed. This indicates that the NaCl crystals are in equilibrium under the current observed conditions. The observed lattice fringes were (200) or (220) lattice planes. Local crystal growth or disappearance was observed due to local structural fluctuations, but there were cases where the lattice fringes grew to lengthen at the edges of relatively large grains, and at the edges relatively small grains, growth was observed that increased the number of layers.

In this study, analysis was performed using stacked images that averages the intensity of six images to improve the image contrast. When we attempted analysis by reducing the number of stacked images in several fields of view, we discovered the possibility that the crystalline region was changing. In this analysis, the areas and boundary positions of crystalline regions were determined manually, but the noise was large and a more objective judgment was needed. Therefore, a future challenge is to introduce an automatic analysis method that automatically identifies crystal regions.