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In-situ TEM observation of dissolution processes in aqueous solutions using ”Poseidon”

FURUKAWA, Erika[1*] ; YAMAZAKI, Tomoya[1] ; KIMURA, Yuki[1]

[1]Tohoku University

Recently, we started a new project to observe crystallization and dissolution processes of crystals in a solution using two different methods under transmission electron microscope (TEM). To overcome the difficulties to introduce a solution into a TEM, Kimura et al. used ionic liquid to avoid evaporation of a solvent in the high-vacuum of a TEM [1]. As the result, several new insights were found: solubility-independent formation of polymorph; crystals do not dissolve smoothly but in a fluctuating manner; and new crystals form even in a totally dissolving system. Another advantage of this method is that the growing crystal does not have a hydrated layer on their surface. It has been believed that dehydration process has a largest potential barrier to incorporate a unit cell into the crystal. However, no one ever visualized the process and it has been totally veiled. The water free experiment using an ionic liquid may give us a new perspective on the dehydration process by comparison with experiments in general aqueous solutions. Now, we are forwarding a project to observe crystallization and dissolution processes in an aqueous solution in atomic-scale using the ”Poseidon”, which is a sample holder having a liquid cell for TEM observation. We call our TEM fluid-reaction TEM (FR-TEM) including both solution growth experiments using an ionic liquid and the Poseidon.

Poseidon (Protochips Inc.) give us the opportunity to visualize the three-dimensional process with several advantages compared with previous works using an atomic force microscope, which is able to observe only two-dimensional, and an optical microscope, which has much less lateral resolution. Growth and dissolution processes at the first top layer (surface) of a crystal have been energetically studied long time using these tools. However, the detail process in atomic scale has been observed very limited. Therefore, the aims of our project is understanding of three dimensional nucleation including Ostwald law of stages based on phase determination by electron diffraction, determination of very slow dissolution rates, and dissolution process in terms of an influence of defects. Here, we will show the first pictures about the movements of nanoparticles and dissolution of amorphous silica and crystalline silicate samples.

[1] Kimura, Y., Niinomi, H., Tsukamoto, K. & Garcia-Ruiz, J. M. In-situ live observation of nucleation and dissolution of sodium chlorate nanoparticles by Transmission Electron Microscopy. *J. Am. Chem. Soc.*, DOI: 10.1021/ja412111f. (2014).

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