

フルイド反応TEMを用いた炭酸カルシウム核生成過程のTEM中“その場”観察 In-situ observation of nucleation process of calcium carbonate by the fluid-reaction TEM

山崎 智也^{1*}; 古川 えりか¹; 木村 勇気¹
YAMAZAKI, Tomoya^{1*}; FURUKAWA, Erika¹; KIMURA, Yuki¹

¹ 東北大学大学院理学研究科地学専攻

¹Tohoku University

Recent studies have reported achievements of in-situ observation of the nucleation and crystallization studies using transmission electron microscope (TEM), and several new perspectives for non-classical pathway of crystallization [1-4]. Calcium carbonate generates a lot of attention because of complex nucleation due to appearance of various polymorphs in addition to availability for industrial materials such as paper and paint, and reservoir of carbon dioxide, and biomineralization. We also focus on the calcium carbonate in view of selection of polymorph in nucleation process. In case of nucleation from relatively higher supersaturated solution, nucleation of amorphous phase prior to crystalline phase has been known [5,6]. Kawano et al. have been reported an in-situ observation of solution-mediated phase transition from amorphous phase into crystalline phase under optical microscope [6]. The Ostwald law of stages has been believed to occur in many cases. Contribution of prenucleation cluster, which was confirmed by using the cryo-TEM [8], to the nucleation has also been reported [7]. However the generality or solution condition to take these processes is still not obvious.

Now, we have performed energetically a project to observe crystallization and dissolution processes in an aqueous solution using ionic liquid instead of water or the "Poseidon" (Protochips Inc.), which is a sample holder having a liquid cell for TEM observation. We call our TEM fluid-reaction TEM (FR-TEM), which is able to perform crystallization experiments in a solution including both methods. Here, we have been tried to observe whole the process of crystallization of calcium carbonate via amorphous phase using fluid-reaction TEM and actually observed a solid-state phase transition from amorphous phase into a crystalline phase by in-situ observation.

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