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Direct observation of nucleation and dissolution processes in a solution using a TEM

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Since nucleation is a fundamental event to determine size, number and morphology of produced crystals, nucleation process must be clarified to form products efficiently. Direct approach to understand nucleation would be an atomic scale in-situ observation. A transmission electron microscope (TEM) may be a most powerful tool. Unfortunately, however, we can prepare limited experimental condition in a TEM and only have a few reports concerning in-situ observation of a nucleation process so far. In particular, since TEM needs a high vacuum, crystallization experiment in a solution was impossible. Recently, formation process of platinum nanoparticles from a solution was observed using a specially designed cell in a TEM [1]. Disadvantages of this noble method are difficulties of high-resolution observation and changing the temperature. Recently, we overcame the disadvantage by using ionic liquid, which has negligible vapor pressure and does not charge up by electron beam due to their relatively higher electron conductivity. Ionic liquid has been used for direct observation of organic materials such as protein and sea weed using mainly scanning electron microscope or occasionally TEM observations. Here, we report successful in-situ observation of nucleation, growth and dissolution processes of sodium chlorate in an ionic solution.

Supersaturated ionic solution of sodium chlorate was prepared on a TEM grid. After controlled induction time, sodium chlorate crystals nucleated and grew under TEM observations with an acceleration voltage of 200kV (Hitach H-8100 placed at the Tohoku University). Ionic solution could be observed stably under normal electron irradiation condition. Nucleated sodium chlorate crystals were floated with the Brownian motion. When two crystalline particles contacted, they fused together to be a larger single crystalline particle. Produced nanocrystals of sodium chlorate were heated up and were observed their dissolution process using a TEM with an acceleration voltage of 300 keV (H-9500 placed at the Hitachi Hitec). The nanocrystals were not only dissolved but also grown even in the totally dissolving system, i.e., most probably undersaturated condition.

[1] H. Zheng et. al., Science 324 (2009) 1309.

Keywords: Nucleation, nanoparticle, dust, in-situ observation, electron microscope, ionic liquid