Radiolysis-induced crystallization process of salt in liquid cell transmission electron microscopy

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Crystallization in solution is a ubiquitous process in the earth and is important not only for the production of materials but also for understanding geochemical processes. However, the early stages of crystallization, including nucleation, are still mysterious because of difficulty in visualizing the processes due to the limitation of observation techniques. A promising method for observing these processes is liquid-cell transmission electron microscopy (LC-TEM), which has nanoscale spatial resolution and real-time temporal resolution. On the other hand, capturing the moment of nucleation using this technique is quite difficult because the observable area is very small and controlling supersaturation in the observing area is extremely difficult. We recently found that sodium chloride crystallized when the electron beam was irradiated on a mixture of a little amount of saturated aqueous solution of sodium chlorate and acetone. This experiment suggests that the electron beam causes radiolysis resulting in dissociation of chlorate ion into chloride ion, and acetone works as an antisolvent for sodium chloride and controls supersaturation of its crystal. Here we report further study for understanding the crystallization process using LC-TEM and different antisolvent.

We used the TEM holder which can enclose the vacuum-tight liquid cell. We enclosed a saturated antisolvent solution (e.g. ethanol) of sodium chlorate in the liquid cell. Then we observed the solution by TEM with a field-emission gun at an acceleration voltage of 200 keV. In this presentation, the detailed results of observation which include analysis of the nucleated crystal and mechanisms of the crystallization will be discussed.

Acknowledgement

This work was supported partly by Grant for Basic Science Research Projects from The Sumitomo Foundation (Grant Number 170738) and JSPS KAKENHI Grant Numbers JP15H05731 and JP18J01470.

Keywords: liquid-cell transmission electron microscopy, crystal growth, solution growth, antisolvent crystallization

