Development of a new non-exposure transfer holder for an environmental HVTEM

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Environmental transmission electron microscopy (E-TEM) is one of the effective research methods for nano-materials in practical use such as catalysts and battery electrodes[1]. The important points for the electron microscopy are (1) gas-atmospheres in actual usage, (2) easily heating, (3) thicker samples prepared from actual devices and circuits and (4) non-exposure to air for decrease of oxidation and hydration. The problems of ordinary environmental TEM still remain after the development of MEMS chip holders. For example, the electrodes of lithium battery should be FIB-fabricated and transferred to TEM instruments without exposure to air, and the actual samples are rather thicker, which do not give clear-cut images by using medium-voltage TEM. In the present study we have developed a new transfer system from a glove box to an E-HVTEM.

The E-HVTEM used is a 1 MV instrument developed in Nagoya University in collaboration with JEOL since 2008, where various kinds of in-situ observation such as heating, cooling, mechanical operation, gas reactions, ice-embedding are available with the maximum TEM resolution of 0.1 nm and STEM resolution of 1 nm[2]. In the present study we have developed a special holder for non-exposure transfer as shown in Fig. 1. The holder has a special closing sleeve (arrow) for non-exposure of samples to air. Also a special vessel was manufactured in order to transfer from a FIB apparatus to the glove box filled with dry inert gases. Figure 2 shows E-HVTEM images of tin particles heated in 523 K after transferring with exposure to air. The image with the exposure showed very thin oxide layers on the particle surface. Then, after heating/melting, oxidation process was in-situ observed with injection of oxygen of 5 x 10⁻⁴ Pa. Figure 3 and 4 show high-resolution images of rhodium/graphite and platinum/graphite electrodes transferred by this system, and observed at RT and over 700K in vacuum, respectively.

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References
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FIG. 1. Photos of the transfer holder with non-exposure to air for E-HVTEM.

FIG. 2. E-HVTEM images of oxidation process of tin particles with injection of $5 \times 10^{-4}$ Pa of oxygen.

FIG. 3. High resolution E-HVTEM image of Rh/graphite at RT, transferred by this system.

FIG. 4. High resolution E-HVTEM image of Pt/graphite heated around 700K, showing movement and coagulation of small clusters.