

In-situ TEM observation of electrochemical growth

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It is important to understand the transfer of electrons and/or ions at the interface between the electrode material and liquid electrolyte in the field of storage device such as battery. A transmission electron microscope (TEM) is a powerful tool to obtain local structure information at such an interface. Ross et al. developed an electrochemical cell for TEM and observed copper deposition process on gold surface [1], but there are a few reports about such an in-situ TEM observation.

In this study, a new-type electrochemical cell for a transmission electron microscope (TEM) has been developed as shown in Fig.1. The cell is made by quartz, and three electrodes (working, reference and counter) are placed in it. The working electrode surface, which contact with liquid electrolyte, can be observed through very thin barrier membrane (50 nm silicon nitride film). The working and reference electrodes were gold film and the counter one, copper film. The electrolyte contained 0.2M $\text{CuSO}_4 + 0.05 \text{ M H}_2\text{SO}_4$.

Using the developed cell, electrochemical deposition and desorption of copper ions were observed on the working electrode surface simultaneously during cyclic voltammetry measurement (Fig.2). The cycle speed was 0.025 V per second. When the applied voltage to the working electrode decreased gradually from 0 V, copper ions started to be deposited on gold surface. On the other hand, when the voltage was around 0.15 V, copper ions were dissolved distinctly.

[1] M. J. Williamson, R. M. Tromp, P. M. Vereecken, R. Hull and F. M. Ross, Nat. Mater. 2 (2003) 532.

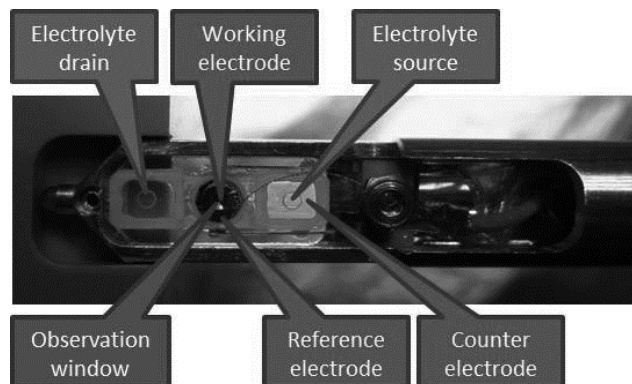


Fig. 1 A photo of our developed electrochemical-cell for a TEM holder

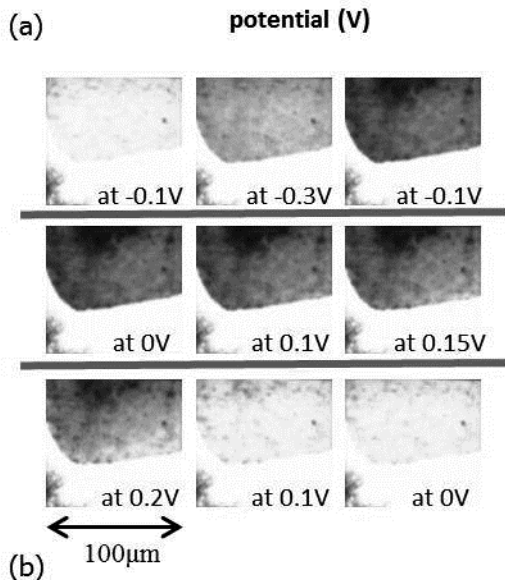
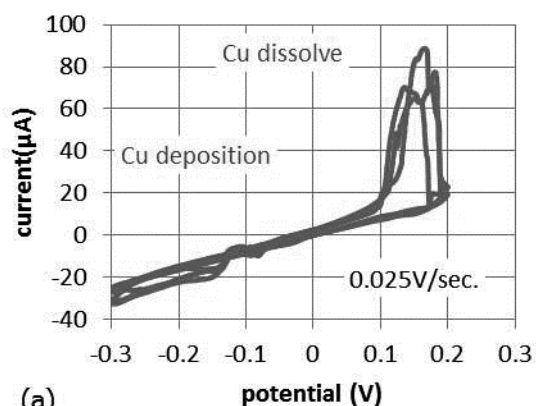


Fig. 2 (a) Typical cyclic voltammetry results. Copper ions are deposited at the negative voltage, while they, dissolved at the positive voltages.(b) A series of optical microscope images, which were obtained during the cyclic voltammetry.