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In-situ Observation of Gold Nano-particle Catalysts by High-Resolution Closed-type Environmental-Cell Transmission Electron Microscope

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In-situ observation is important to reveal real properties of materials. Especially, for catalysts, analysis during reaction is essential. “Environmental-cell (E-cell) electron microscopy” technique, which enables gas introduction around specimens, has been investigated actively[1]. The authors also have tried to develop the “closed-type” E-cell transmission electron microscope (TEM), aiming for the real condition, i.e. atmosphere pressure. In addition, high-resolution is tried to be simultaneously achieved, since our target is nano-size noble metal catalysts[2]. In the paper, we demonstrate potentials of our developed E-cell TEM system to observe dynamics of catalytic gold nano-particles.

Fig. 1 shows a schematic diagram of our E-cell TEM system. This consists of two developed apparatuses equipped with a conventional 200kV-TEM (H-8000, Hitachi). One is a closed-type E-cell specimen holder (see Fig. 2). This has a small gas room, called the E-cell, at its top and two pipes for the gas in/out. The gas in the E-cell is separated from vacuum with ultra-thin carbon films. The films are most important components of this system, because they have to both withstand the gas pressure and pass the electron beam easily for high-resolution. From this standpoint, we have developed a new method to make the ultra-thin carbon film with high strength. “8nm” thick films, less than half of conventional ones, enabling to withstand 2-atom pressure were achieved. This new ultra-thin film is one of the key points for high-resolution observation under the “real” reaction conditions. The other developed apparatus is a gas control unit. This is the hand-made equipment but enables fine tuning of introducing gas pressure and so on (see Fig. 2). The completed system realizes the catalytic reaction condition of the 1-atom gas atmosphere in the TEM.
In the present experiment, a specimen was nano-size gold particle supported on TiO_2 and the reaction gas was CO 1% in Air of 375 Pa. In-situ observation has been successful, resulting in dynamical deformation of surface structures of gold during about 3 min reaction. Figs. 3 show images (a) before and (b) after the reaction. They demonstrate changes of shape of the gold particle, e.g. size of a (111) facet at the bottom of the particle increases.

[1] P. L. Gai, Topics in Catalysis 21 (2002) 161
[2] M. Haruta, Catalysis Today 36 (1997) 153

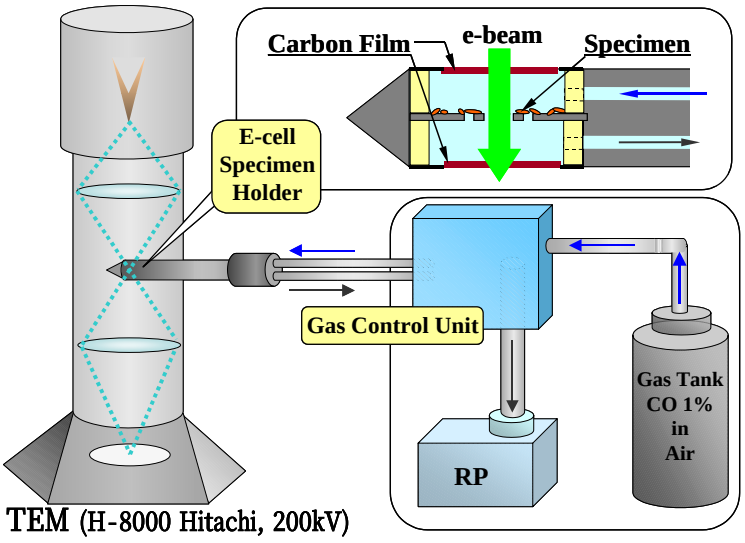


Fig. 1 Schematic diagram of the developed E-cell TEM system

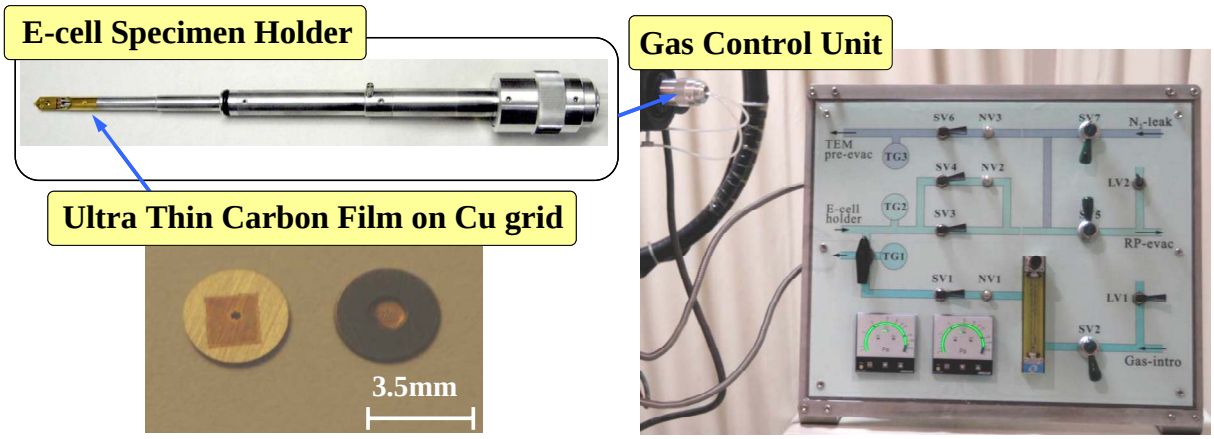
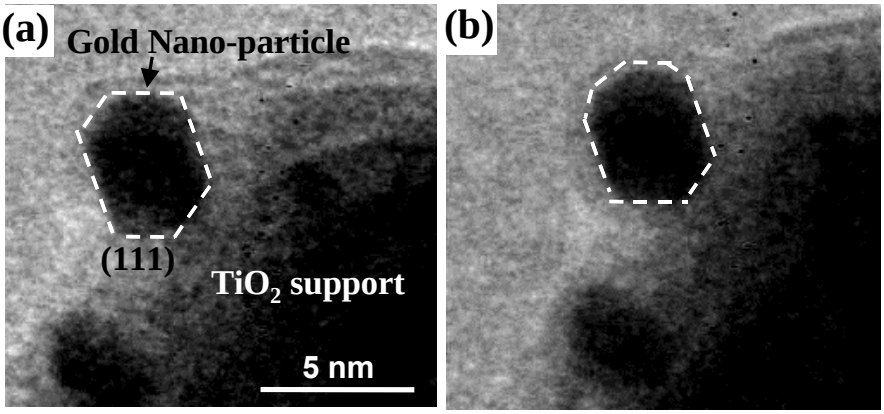


Fig. 2 Pictures of developed apparatuses and experimental parts



Figs. 3 Deformation of a gold nano-particle due to the reaction
(a) before and (b) after about 3 min reaction