Environmental transmission electron microscopy (ETEM) recently attracts a strong interest of many materials researchers, because the observation of chemical reaction process with gases and liquids becomes important for the practical use. However, the samples to be observed are only thin edges of films and particles due to the limitation of electron transmission in ordinary TEM up to 300 kV accelerating voltage. ETEM observations of samples of a few micron thickness in gas environment of hydrogen or oxygen are crucial for development of practical fuel cells and solar batteries, and various kinds of chemical and structural materials. 3D observation of samples is also necessary for clarifying the morphologies related to the chemical properties such as catalysis.

We have developed 1MV TEM/STEM with an open-type environmental cell which enables observation in 100 Torr atmosphere of gases, named "Reaction Science HVEM (RSHVEM)"[1]. High-transmission of electrons insures the point-to-point resolution less than 0.2 nm even in gas-environment of 100 Torr. High-speed beam blanking system reduces irradiation effects of incident electrons on the samples.

Figure 1 shows an external view of the RSHVEM with a box-shaped environmental cell inserted in the pole-piece-gap of an objective lens from the left-hand side. Figure 2 shows in-situ atomistic observation of oxidation process of a copper(Cu) particle followed by reduction by hydrogen. The repeated reaction process is monitored also by in-situ EELS. Figure 3 shows in-situ observation of porous gold (Au) catalyst in reaction, whose inner surface with zigzag atom arrangement enhances the catalytic reaction with carbon-mono oxide[2,3]. Figure 4 shows the world-first in-situ observation of hydrogen brittleness of a copper(Cu)/silicon(Si) interface as well as in-situ measurement of stress/strain curves in a mixed gas of hydrogen and nitrogen[4]. The present microscope can be used also for in-situ high-resolution STEM-EELS mapping[5] for elemental, chemical and physical analysis, and 3D electron tomography of large inorganic particles and biological cells[6] is possible.

[6]Y. Murata et al., to be submitted (2014), and in this conference.

Acknowledgement: The present authors would like to acknowledge many collaborators and graduate students for help to the present studies.
Fig. 1: Front-view of RSHVEM in Nagoya University.

Fig. 2: In-situ observation of oxidation of a copper particle followed by the reduction.

Fig. 3: In-situ HRTEM of reaction change of a (011) surface of gold with CO gas.

Fig. 4: In-situ TEM of fracture of a Cu/Si interface in H₂ + N₂ gas.