In-situ chemical analysis using 300 kV aberration corrected scanning transmission electron microscope with gas-cell specimen holder

Ohnishi, I.[1], Suzuki, T.[1], Fukunaga, K.[1], Sasaki, T.[1], Sawada, H.[1] and Okunishi, E.[2]

[1] JEOL Ltd., Japan, [2] JEOL Ltd., Japan

Recent progress of gas-cell type specimen holder made by micro electro mechanical system (MEMS) allows us to conduct highly stable and reproducible in-situ experiments under high gas pressure condition in transmission electron microscope (TEM). Well designed tip of the latest holder can realize in-situ chemical analysis using energy dispersive X-ray spectroscopy (EDS) and electron energy loss spectroscopy (EELS). However, compared to EELS analysis, there are still some restrictions for EDS analysis. Since the infrared radiation from the heated sample increases the background noise and degrades energy resolution of EDS detector, the allowable temperature for measurement by using a silicon drift detector (SDD) with no window is limited to <400 degrees. Absorption of X-ray by gas and a window made of Si_xN_y decreases X-ray signals. Since the Si substrate supporting the Si_xN_y windows completely blocks X-ray signal, specimen should be highly tilted to the EDS detector if take-off angle of the detector is low. Thus, chemical analysis using EELS is common for in-situ experiments so far, compared to EDS.

JEM-ARM300F is a 300 kV aberration corrected TEM with a cold field emission electron gun. This microscope with a wide gap objective lens pole piece (WGP) can be equipped with two windowless SDDs, whose sensor size is selectable to be 158 or 100 mm^2 to realize highly sensitive X-ray analysis. When we use a gas-cell type specimen holder, only one SDD (SDD1), which is located on the right side of specimen holder, is available. Even in such a case, the collection solid angle reaches ~1.1 sr with the 158 mm^2-sized SDD. The take off angle is as high as ~30 degrees. It can minimize shadowing of specimen holder. Thus, it is expected that in-situ EDS analysis can be performed under gas environment with high sensitivity. In this paper, we demonstrate in-situ experiments using the microscope equipped with the gas-cell type specimen holder (Protochips Inc. (Atmosphere 200)). The combination can provide not only atomic resolution imaging under 1 atmosphere condition but also highly sensitive elemental analysis as well as EELS analysis.

Figure 1 shows the results of redox reaction experiment of Cu powder. The sample temperature was fixed at 300 °C. To observe the morphologies during the oxidation and deoxidization, the gas condition was changed under three conditions: firstly (1) N_2, 10^3 Pa, secondly (2) H_2, 10^4 Pa, and finally (3) O_2, 10^4 Pa. The scanning TEM (STEM) images revealed that the sample morphology significantly changed depending on the gas conditions. EDS/EELS analyses also revealed a change in sample stats: mixing of oxidized and metallic copper in (1), metallic copper in (2) and oxidized copper in (3). Moreover, EDS maps revealed small particles of oxidized iron in the copper sample and the particle keeps oxidized state under all gas conditions in this experiment. This result suggests that oxidized iron is more stable to oxygen and hydrogen gas than the oxidized or metallic copper. In the presentation, we will show the results of EELS analyses and other analytical applications.

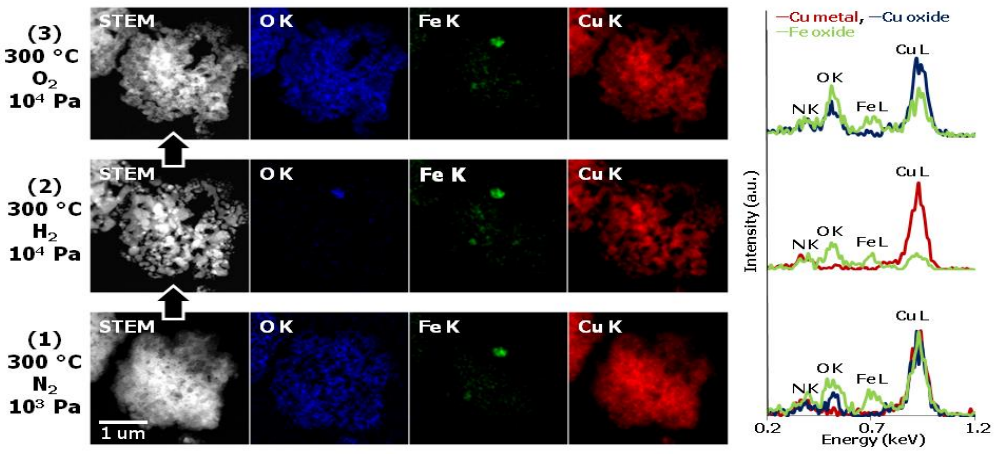

Figure 1: Redox reaction experiment of Cu powder under three gas conditions, (1) N_2, 10^3 Pa, (2) H_2, 10^4 Pa, and (3) O_2, 10^4 Pa at 300 °C.