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In-situ TEM observation of structure changes of Pt particle on CeO_2 by exposing gases

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It is important to study the relations between the microscopic structure and function of materials in order to develop new functional materials. The materials for catalyst are widely studied by various research techniques. In situ TEM controlling environmental atmosphere has attracted considerable attention in dynamic observation of chemical reactions, crystal growth, and also applied for the observation of catalyst in reaction gases [1-3]. It is also important to investigate the fundamental interaction metal particle and reaction gases in nano- and atomic scale. In this experiment, the simple in-situ TEM system was constructed .for observation of interaction between metallic nano-particles and gases. In-situ TEM observation was tested for the Pt catalyst by introducing the gases. The advantage of this system is to switch the gases rapidly. Thus it is easy to observe the structure changes depending on the gases.

The system was constructed by modifying Hitachi H-9000NAR transmission electron microscope. The variable leak valves for ultra high vacuum type and quadro-pole mass spectrometer was attached at around the specimen chamber. The gases are introduced by controlling precisely via the variable leak valves manually. The gases can be introduced until total pressure of 10^{-3}Pa at present construction. The Pt/CeO_2 catalyst prepared by solid state gliding method was used for the observation as preliminary experiment. CO and O_2 gases are reserved in the small bottles connected to the variable leak valves. The TEM movies are recorded by hard disc recorder via TV camera. The Pt/CeO_2 powder sample dispersed on the carbon micro-grid supported on Cu-mesh. The double tilt heating specimen holder was used for observation and the temperature was set at 423K. The activity of the catalyst sample for CO oxidation is confirmed by the fix bet flow reactor, conversion of CO reaches 100% at 423K. The introduced gases are monitored by Q-mass, but reaction gases are not detected in this system because a small amount of sample is used for TEM observation.

Figure 1 shows HRTEM images of Pt particle on CeO_2 during gas exposure. The incident electron is parallel to the [110] zone axis for Pt crystal, and lattice fringe of fcc Pt crystal appeared. The Pt particle shows low index facets such as (111) surfaces while it includes the stacking fault. The reconstructed 2x1 structure is observed at Pt(110) surface of the particle when the O_2 is introduced. The lattice images of Pt become blur in fig1.c-e, it seems the electron beam direction deviates from zone axis by motion of Pt particle. The amorphous contrast also appears at perimeter between Pt and CeO_2 support after exposure to O_2 as indicated by an arrow in fig.1b. High resolution TEM observations can be carried out without significant disturbance by introducing gases. The reversible structure changes by gases were also observed by introducing CO

and O_2 gases. A reversible oxidation and reduction of Pt seemed to occur by gases. It is necessary to discuss details taking the irradiation effect by the electron beam into consideration.

References

[1] P.L.Gai, Topics in Catalysis 21(2002) 161.

[2] T.W. Hansen et al. Science 294 (2001) 1508.

[3] H. Yoshida et al. Nano Lett. 8(2008) 2082.

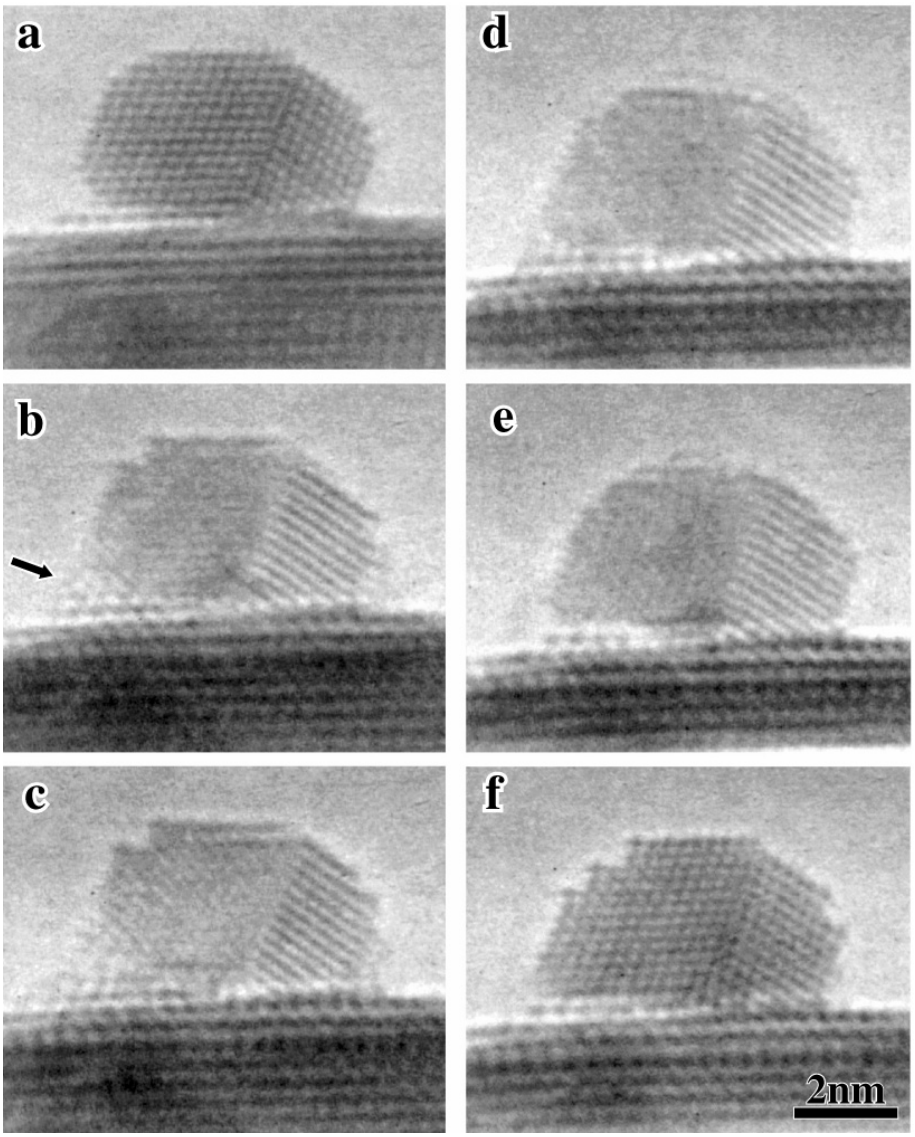

FIG. 1. Sequential TEM images of Pt particle on CeO_2 in the gases of 10^{-3}Pa of $CO+O_2$(a), O_2(b), in vacuum, CO(d), $CO+O_2$(e), O_2(f).