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In-situ TEM Observation of Nano-particulate Gold Catalysts under Reaction Gas and Non-reaction Gas Environments

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Abstract – The surface structure of catalytic gold nano-particles was observed dynamically during CO oxidation using an environmental-cell transmission electron microscope (E-cell TEM) system. In the developed system, the gold catalyst specimen can be set under the reaction gas condition by separating it from the vacuum using ultra-thin carbon films. We have developed a method capable of yielding for carbon films 8 nm thick capable of withstanding a gas pressure of 2 atmospheres. *In situ* observations using the above system indicated marked changes in the surface shape of the gold nano-particle catalyst during the reaction.

Gold exhibits catalytic activity when it is in the form of fine particles having a size of less than 10 nm and is tightly supported on specific metal oxides such as TiO_2, etc [1]. To reveal its mechanism, dynamic observation of the sample structures during the reaction by the transmission electron microscope (TEM) is quite essential. “Environmental-cell (E-cell) TEM” technique [2], which enables gas introduction around specimens, is one of the most powerful methods for the purpose. The authors also have developed the closed-type E-cell TEM [3], and had revealed that the shape of catalytic gold nano-particles are changing during reaction [4]. In the paper, we report about difference of the shape change of gold nano-particles between under reaction gas and non-reaction gas conditions.

In the present experiment, a specimen was nano-size gold particle supported on TiO_2. The gas introduced were 1% CO in N_2(78%) + O_2(21%) as a reaction gas and dry N_2 as a non-reaction gas. Their pressure around the specimen was set at about 750 Pa. Fig. 1 shows in-situ TEM images of a gold nano-particle picked up from among those recorded sequentially. In this case, the introduced gas was CO in dry air. Therefore, catalytic reaction happened on the catalyst surface; CO was oxidized into CO_2. As shown in these images, the shape of gold particle was dramatically changed. Various facets were appeared and disappeared. On the other hand, almost no change was observed in the case of non-reaction gas N_2 condition. Although slight shape changes might occur due to electron beam irradiation in this case, the difference of alterations of the particle shape can be clearly shown by comparing with Fig. 1. There results prove that the catalytic reaction affects the shape changes of the gold nano-particles.

References

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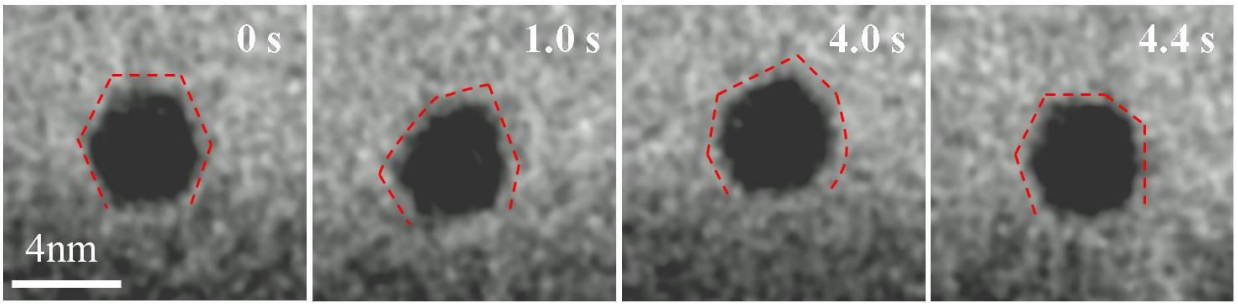

Figure 1: In-situ TEM images of a catalytic gold nano-particle during CO oxidation
(Reaction gas is CO with dry air; Pressure is about 750Pa)