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Program Title (English) :In-situ XAFS study of noble metal high entropy alloy nanoparticles with high stable and excellent activity under CO oxidation conditions

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## 1. Introduction

The purpose of this beam time is to observe the electronic structure change of metal alloy nanoparticles from monometallic to high-entropy alloy nanoparticles during the gas atmosphere.

## 2. Experimental

First, we observed the ex situ XAFS of the samples in the pellet form. For the in situ measurement, focusing on the In K-edge and Mn K-edge, the samples were pressurized and fitted into the in situ holder provided by Dr. Matsumura. The XAFS spectra were obtained under different atmospheres including vacuum, $H_2$ (1 bar and 9 bar), $CO_2$ (1 bar and 9 bar) with varying temperatures (RT to 773 K).

## 3. Results and Discussion

During this beamtime, we have confirmed that all the liquid metal-based nanoparticles are covered by metal oxide layers on their surfaces. The thickness of the surface oxide layers is highly dependent on the particle size. Small NPs have thicker layers. We also found that the formed oxide layers have shorter M-O bond lengths than their corresponding crystalline metal oxides. These results are in line with the XPS as well as the XRD results. As for the liquid metal loaded on support, we found that the liquid metal tends to get lattice oxygen from the supported metal oxides and forms the amorphous liquid metal oxides. We also find that the coordination of metals is changed by changing the gas atmosphere. Moreover, $MnO_2$ was found to be unstable during the $CO_2$ and $H_2$ atmospheres. With a long-term gas phase treatment, $MnCO_3$ was found. This suggests the activity of $MnO_2$ for $CO_2$ hydrogenation but also the instability.