Contribution ID: **107** Type: **Oral presentations**

Operando X-ray absorption spectroscopic studies on high-pressure and electrochemical heterogonous catalytic reactions towards CO2 utilization

Thursday, 18 June 2015 16:20 (20 minutes)

To level off the atmospheric concentration of CO2, the molecule largely responsible for global warming, several CO2 utilization pathways have been actively developed in the past years. Among others, heterogeneous catalytic conversion of CO2 has a strong potential to convert large amount of CO2 in short time span. For the rational improvement of heterogeneous catalytic reactions, it is of prime importance to elucidate active state of catalyst materials under realistic operating conditions. X-ray absorption fine structure (XAFS) allows investigation of gas, liquid, and solid samples in wide pressure and temperature ranges to understand the local structure around an absorber together with its electronic structures such as oxidation states.
In this work, we studied CO2 conversion and related reactions (H2 production for CO2 reduction) by XAFS to gain insights about the oxidation state of active catalysts under challenging and realistic operating conditions. The first experiment was on high pressure CO2 hydrogenation to methanol to examine the state of copper using a conventional Cu/ZnO/Al2O3 catalyst. A high pressure (up to 200 bar) and high temperature (up to 280 ℃) micro-reactor XAFS cell with a unique coiled heating system was fabricated using the polyimide coated fused silica capillary. It was found that the active copper component of the catalyst remain in the metallic state under working reaction conditions of 200 bar and 260 ℃. [1] The second studied reaction is water electrolysis using polymer electrolyte membrane (PEM) using a novel material, cobalt oxide, for hydrogen evolution reaction (HER). A conventional PEM electrolyser was modified with X-ray transparent windows and XAFS studies was performed under operando electrochemical conditions. The study clarified a dynamic oxidation state change of active Co material under electrochemically working conditions.
These experiments were performed at BL22-CLÆSS beamline at ALBA Synchrotron Light Facility with the collaboration of beamline staff members.

References

[1] A. Bansode, G. Guilera, V. Cuartero, L. Simonelli, M. Avila, A. Urakawa, Performance and characteristics of a high pressure, high temperature capillary cell with facile construction for operando x-ray absorption spectroscopy, Rev. Sci. Instrum., 85 (2014) .

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Session Classification: Session 8 - II ALBA User's Meeting 2015

Track Classification: II ALBA User's Meeting