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Real-Time Observation of Intermediates at the Solid-liquid Interface of Catalyst Electrode Using Wavelength-Dispersive Soft X-ray Absorption Spectroscopy

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A real-time measurement method by soft X-ray absorption spectroscopy (XAS) was developed to observe the chemical reaction under operando condition [1]. In addition, a dedicated cell for the method was prepared for real-time observation of the chemical reaction at the solid-liquid interface of the (photo)electrode for the oxygen evolution reaction (OER) of water electrolysis. Hydrogen generation through water electrolysis is one of the most popular research fields for achieving carbon neutrality. In particular, the electrode for OER is a performance bottleneck compared with the hydrogen evolution reaction electrode; therefore, improving the OER performance is important for enhancing the overall system performance. For the purpose, *operando* evaluation focusing on the solid–liquid interface conditions is key to elucidating the chemical states of the catalytic material surface and identifying the intermediate species at the surface.
In this study, CoO_x and TiO₂ as OER catalysts were observed in real time using fluorescence-yield soft X-ray XAS. Oxygen K-edge XAS was observed during linear sweep voltammetry for OER, and with UV light on or off (in the case of TiO₂). The spectra during the reaction were obtained every 3 s. Alternation of the spectra appeared during the potential sweep, and the peak intensity differed between the cases with UV light on and off for TiO₂. This spectral change can be attributed to the intermediates formed during the (photo)catalytic reaction at the solid–liquid interface.
The present technique can be applied to a wide range of analyses of (photo-) electrocatalysis and electrochemical reactions at solid–liquid interfaces to observe their products and intermediates during the reaction.

Figure 1 Schematic image of the dedicated electrochemical cell for measurement using wavelength-dispersive XAS. The XAS spectra of O K-edge were obtained in real time, and the spectrum changed according to the applied potential. The right side of the figure shows the change in intensity of O K-edge of CoO_x over time during the potential sweep. [2], [3]

References:
[1] K. Amemiya et al., Rev. Sci. Instrum. 91, 093104 (2020).
[2] K. Sakata, K. Amemiya, Chem. Lett. 50, 1710 (2021).
[3] K. Sakata, K. Amemiya, Electrochem. Commun. 157, 107627 (2023).

I plan to submit also conference proceedings

No

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