Soft X-ray Absorption Spectroscopic Analysis on Water Electrolysis

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Renewable energy sources, such as solar cells and wind power generation generally have large fluctuation of supply, and cannot be stored as electrical energy. Power to gas system produces hydrogen from renewable energy by water electrolysis and store the energy as chemical energy has attracted attention as one of the next-generation carbon-free strategies. For electrolyzer in which electrolytic reaction proceeds, the durability against a load variation of renewable energy with high electrolytic efficiency in a long period of operation is required. In particular, the analysis of the mechanism of high durability and activity is important for developing an electrocatalyst. Under such background, an analytical method of electrocatalyst for water electrolysis using soft X-ray beamline of SR center was established in Advancement of Hydrogen Technologies and Utilization Project/ Development of Fundamental Technology for Advancement of Water Electrolysis Hydrogen Production/ Development of Alkaline and Solid Polymer Electrolyte Water Electrolysis of New Energy and Industrial Technology Depelopment Organization (NEDO). In the alkaline water electrolysis and polymer electrolyte membrane electrolysis, nickel-based oxides and iridium-based materials were used as anode catalysts, respectively. To build databases of X-ray absorption spectra (XAS) required for the analysis of electrocatalysis, beamlines BL-2 and BL-11, Ritsumeikan SR center were used to measure XAS at Ni L-edge and O K-edge of typical nickel compounds, and O K-edge of iridium-based materials in total electron yield and fluorescence yield modes. Additionally, the measurements of electrocatalysts and operated electrodes studied in the project are performed, and the degradation mechanism was discussed.

X-ray absorption near edge structure (XANES) at Ni L3,L2-edge of typical nickel compounds show the doublet absorption peak at Ni L3-edge around 855 eV. The peak at higher energy of trivalent β-NiOOH and $LiNiO_2$ shows higher intensity than divalent NiO and β-$Ni(OH)_2$, indicating that the difference in valence of Ni can be discussed by comparing the two peak intensities. It is also possible to discuss the spin state of transition metals by the ratio of L3- and L2-edge. When comparing O K-edge XANES of β-NiOOH and $LiNiO_2$ classified as trivalent, the intensities of the observed pre-edge peaks around 528 eV differ. In the comparison between NiO and β-$Ni(OH)_2$, a peak is observed near 531 eV in only NiO. In other words, even compounds of the same valence are quite different in their O K-edge shapes, so that the compound can be identified by having Ni L-edge and O K-edge XANES. XAS analysis of degradation catalysts produced according to the proposed degradation protocol from the project was performed. Moreover, the analysis of nickel-based oxide catalysts with high durability to give information on the mechanism for durability enhancement [1].

References

[1] Y. Ren, R. Yamaguchi, T. Uchiyama, Y. Orikasa, T. Watanabe, K. Yamamoto, T. Matsunaga, Y. Nishiki, S. Mitsushima, Y. Uchimoto, *ChemElectroChem* **2021**, *8*, 3.

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