## SR12 24

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## Specially Designed Cells Enabling In-Situ Observation of High-Current-Density Electrochemical Reactions with Multiple Spectroscopy Techniques

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Nowadays, the overuse of fossil fuel has brought up the urgency to efficient energy conversion and green energy consumption, which gives significance to high-value-added electrochemical reactions, such as hydrogen evolution reaction (HER), oxygen evolution reaction (OER), carbon dioxide reduction reaction (CRR), nitrate reduction reaction (NO3RR), and C-N coupling reactions. The application prospects of the aforementioned electrochemical reactions are highly related to the performance of electrocatalysts under high current density (HCD). Therefore, the observation of HCD electrochemical reactions is of great significance. Unfortunately, the focus and signal-to-noise ratio of multiple spectroscopy techniques (XAFS, Raman, FT-IR, etc.) are severely challenged by violent bubble desorption under HCD conditions.

In this talk, first, we will introduce our work related to the structure-performance relationship of HCD NiMo-based electrocatalyst revealed by in-situ XAFS combined with XRD, and Raman (Figure 1. a-c)[1,2]. These catalyst families (NiMo-Fe, NiMo-Ru, NiMo-Pt, NiMo-Ir) exhibit high efficiency in various HCD electrochemical reactions, for example, HER, OER, and NO3RR, and have been applied in anion-exchange membrane electrolyzer (Figure 1. d)[3,4,5].

Second, we will introduce our latest progress in specially designed cells enabling in-situ characterization of high-current-density electrochemical reactions with multiple spectroscopy techniques. We have achieved the observation of various electrochemical reactions via a specially designed membrane-assembly-structure insitu experimental cell. At present, it has been successfully applied to in-situ Raman as one of the important spectroscopy which gives high-quality results on the in-situ structural evolution of molybdate in solution under HCD of 1000 mA cm-2 (Figure 1. e). From this result, we observed a pH flipping phenomenon that had never been reported in in-situ testing under low current density. This also confirms the scientific importance of pursuing new research on HCD. The method can give other information closely related to the electrochemical reaction state such as electrocatalyst reconstruction (NiOOH in OER), reaction intermediates (NO2- n NO3RR), pH probe of micro-environment (HCO3-/CO32-), and H-O vibrations of water molecules. Recently, we also obtained high signal-to-noise ratio in-situ results under HCD through XAFS by this method, so it is universally applicable to multiple spectroscopy techniques.

This method can be further promoted to spectroscopy methods such as infrared SR-FTIR and XRS. Meanwhile, this technique will guide the electrochemical society to understand the HCD electrochemical processes further, and at the same time facilitate the industrial application of high-value-added reactions.

## References

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## I plan to submit also conference proceedings

Primary author: KANG, Xin (Tsinghua university)

**Co-authors:** Prof. LIU, Bilu (Tsinghua university); Prof. CHENG, Hui-Ming (Shenzhen Institute of Advanced Technology, Chinese Academy of Sciences); Prof. YU, Qiangmin (Tsinghua university); Prof. ZHANG, Shuo (Shanghai Synchrotron Radiation Facility, Shanghai Advanced Research Institute, Chinese Academy of Sciences); Dr ZHANG, Zhiyuan (Tsinghua university)

**Presenter:** KANG, Xin (Tsinghua university)

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