Electrochemical Mapping of Hydrogen Evolution Reaction on Transition Metal Dichalcogenide Nanosheets

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Due to a recent increase in interest of two-dimensional (2D) layered transitional metal dichalcogenides, molybdenum disulfide (MoS_2) has received a great amount of research attentions. MoS_2 is one of the most promising precious rare metal-free catalysts for the hydrogen evolution reaction (HER). In order to improve the catalytic activity of MoS_2, significant efforts have been made in terms of conductivity improvement and chemical doping. However, most of the research characterizes the macroscopic catalytic activity of a large number of MoS_2 nanosheets, while quantitatively identifying and characterizing catalytically active sites in MoS_2 are critically important for understanding the catalysis of MoS_2. Therefore, it is necessary to develop a measurement technology to connect the relationship between the spatial distribution and the electrochemical activity of the HER active sites. Scanning electrochemical cell microscopy (SECCM), which uses a nanopipette as a probe in a local and movable electrochemical cell, is an effective tool for characterizing surface structures electrochemically in a submicroscale spatial resolution. The advantages of SECCM are that it is a reproducible and reliable technique for fabricating nanoprobes together with fast electrochemical characterization due to its small capacitive current[1] and its ability to prevent sample contamination during scanning. In this presentation, I will introduce about recent our 1H-MoS_2 monolayer nanosheet research result.[2] Our data provides information about the local catalytic properties as well as electrochemical images of the HER current, Tafel slope, and overpotential by measuring the cyclic voltammograms (CVs) at all measurement points during the imaging (Fig.1). These SECCM measurement unveiled heterogeneous reactivity, relationship of layer number and HER activity, and aging effect.

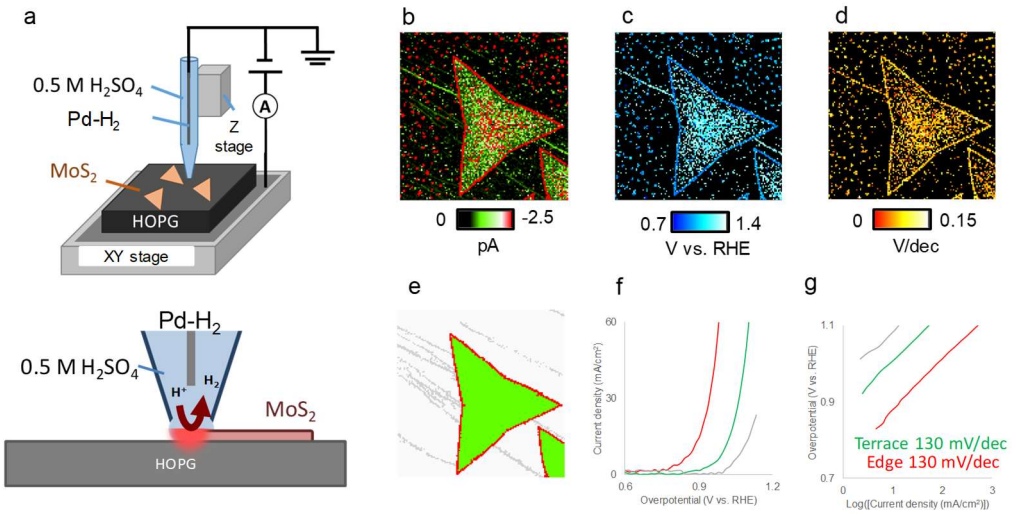

Figure 1. (a) Schematic illustration of SECCM measurements of MoS_2 nanosheets. Pd-H_2 used as a quasi-reference electrode. Nanopipette filled with 0.5 M H_2SO_4. SECCM (b) current, (c) overpotential (30 mA/cm^2), (d) Tafel slope images of 1H MoS_2 nanosheets on HOPG substrate. Scansizes were 15 × 15 μm^2. Scan rate is 130 V/s. Sweep Voltage were -1.3 V vs. RHE. (e) MoS_2 nanosheets edge(red), terrace(green), and HOPG edge(grey) tricolor images. Graphs showing the (f) overpotential and (g) Tafel slope on the 1H

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[2] Y. Takahashi, Y. Kobayashi, Z. Wang, Y. Ito, M. Ota, H. Ida, A. Kumatani, K. Miyazawa, T. Fujita, H. Shiku, Y. E. Korchev, Y. Miyata, T. Fukuma, M. Chen, T. Matsue, *Angewandte Chemie International Edition*, **2020,** *59* (9), 3601-3608.