## Electrochemical Catalysis of Inorganic Fullerene-like MoS<sub>2</sub> Nanomaterials

J. Chen<sup>†,\*</sup>, N. Kuriyama<sup>‡</sup>, K. Tanaka<sup>‡</sup>, Q. Xu<sup>‡</sup>, H.T. Yuan<sup>†</sup>, and T. Sakai<sup>‡</sup>

<sup>†</sup>Institute of New Energy Materials Chemistry, Nankai University, Tianjin 300071, P.R.China <sup>‡</sup>National Institute of Advanced Industrial Science and Technology (AIST), Ikeda, Osaka 563-8577, Japan

(\*Corresponding author. E-mail: junchen\_1@sina.com)

Inorganic fullerene-like nanoparticles and nanotubes, first reported by Tenne and coworkers<sup>1</sup>, have engerated intense scientific interest owing to their promising electronic and mechanical properties. Substantial progress has been achieved in the use of WS<sub>2</sub> nanotubes as scanning tips<sup>2</sup>, microscope fullerene-like  $MoS_2$ nanoparticles as excellent lubricants<sup>3</sup>, and openended  $MoS_2$  nanotubes as electrochemical electrode for reversible hydrogen storage<sup>4</sup>. To date, various synthetic methods for the production of macroscopic amounts of MoS<sub>2</sub> and WS<sub>2</sub> nanotubes have been described.<sup>5-12</sup>

In the present work, we have studied the production of open-ended MoS<sub>2</sub> nanotubes using an improved floating catalyst approach, in which the ball-milled  $(NH_4)_2MoS_4$  was heated under  $H_2$ atmosphere. The as synthesized samples were analyzed by a combination of the following techniques: X-ray diffraction, high-resolution transmission electron microscopy, Brunauer-Emmett-Teller sorption, X-ray photoelectron spectroscopy, and thermal analyses. In comparison with the methods reported previously, the present work allows lower growth temperature and thus easier/safer control over the reaction process. Our improved route yielded large quantities of hollow and open-ended MoS<sub>2</sub> nanotubes with an average length of 5 micrometers, and an outside diameter of 30 nm, an internal diameter of 10 nm, and an interlayer spacing of 0.64 nm.

Furthermore, we have tested the electrochemical catalysis of open-ended MoS<sub>2</sub> nanotubes in aqueous and nonaqueous electrolytes. Very interesting results such as reversible hydrogen reduction/oxidation and alkali ion insertion have been obtained. As an example, in the measurement of aqueous electrolyte system the MoS<sub>2</sub> nanotubes (80 wt%) were mixed with 20 wt% Teflon acetylene black powder ( $\leq 1.0 \ \mu m$ ) in a slurry, pasted onto nickel foam matrix, and then followed by drying and pressing to construct the working electrode. Electrochemical characteristics of the electrodes were measured by using a sintered Ni(OH)<sub>2</sub>/NiOOH counter electrode (about 1000 mAh) and a Hg/HgO reference electrode in 5 M KOH solution at 20 °C. It is found that the cyclic voltammetric (CV) response exhibited excellent electrochemical activity, and the discharge capacity of 260 mAh/g was measured for MoS<sub>2</sub> nanotubes at 50 mA/g and 20 °C. After a preliminary test of 30 consecutive cycles of charging and discharging, the electrode capacity decreased by only about 2%. The high-rate dischargeability of these electrodes is also promising.

Our new results show that the nanotubes with much higher specific surface areas are responsible for the reversible hydrogen adsorption/desorption. Nevertheless, their further study may find wide applications such as electrochemical catalysis and high-energy batteries.

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