

Supporting Information

Superior electrochemical properties of MoS₂ powders with MoS₂@void@MoS₂ configuration

You Na Ko,^{a,b} Yun Chan Kang^{*a} and Seung Bin Park^b

^aDepartment of Chemical Engineering, Konkuk University, 1 Hwayang-dong, Gwangjin-gu, Seoul 143-701, Korea; E-mail: yckang@konkuk.ac.kr

^bDepartment of Chemical and Biomolecular Engineering, Korea Advanced Institute of Science and Technology, 291 Daehak-ro, Yuseong-gu, Daejeon 305-701, Korea

EXPERIMENTAL DETAILS

Synthesis of yolk-shell and dense structured MoS₂ powders

Yolk-shell MoO₃ powders were synthesized by applying an ultrasonic spray pyrolysis process. The schematic illustration of the formation process of yolk-shell MoS₂ powders is shown in Figures S1. As the first step, MoO_x-carbon composite powders were synthesized by ultrasonic spray pyrolysis. Subsequently, yolk-shell MoO₃ powders were obtained by combustion of the prepared MoO_x-carbon composite powders in air at 400 °C for 1 h. The resultant yolk-shell MoO₃ powders were sulfidated at 400 °C for 6 h in the presence of 10% H₂/Ar mixture gas with thiourea as a sulfur source. A small alumina boat containing the yolk-shell MoO₃ powders was loaded into a larger alumina boat with a cover. An excess amount of thiourea powder was loaded on the outside of the small alumina boat for complete sulfidation of the yolk-shell powders. Decomposition of the melted thiourea under hydrogen/argon mixture gas supplied as the carrier gas produced hydrogen sulfide gas. MoS₂ powder was formed by sulfidation of the yolk-shell MoO₃ powders by the continuously generated hydrogen sulfide gas. The MoS₂ powders with dense structure were also prepared by the similar preparation process. The bare MoO₃ powders with dense structure obtained by spray pyrolysis at 600 °C from the spray solution without carbon source material transformed into the MoS₂ powders with dense structure after sulfidation process.

Characterizations

The morphology of the prepared samples was evaluated via scanning electron microscopy (SEM, JEOL JSM-6060), field emission scanning electron microscopy (FE-SEM, Hitachi S-4800), and transmission electron microscopy (TEM, JEOL-2100F). The crystal structures of the samples were investigated using an X-ray diffractometer (XRD, X'Pert PRO MPD) with Cu K α radiation ($\lambda = 1.5418 \text{ \AA}$). The XPS spectra of the rattle-type MoS₂ microspheres was

investigated using X-ray photoelectron spectroscopy (XPS, Theta Probe AR-XPS System) with Al K α radiation (1486.6 eV). The binding energy was calibrated with reference to the C 1s level of carbon (284.6 eV).

Electrochemical measurements

The electrochemical properties of the MoS₂ powders were analyzed by constructing a 2032-type coin cell. The anode was prepared by mixing the active material, carbon black, and sodium carboxymethyl cellulose (CMC) in a weight ratio of 7:2:1. Li metal and microporous polypropylene film were used as the counter electrode and the separator, respectively. The electrolyte was 1 M LiPF₆ dissolved in a mixture of ethylene carbonate/dimethyl carbonate (EC/DMC; 1:1 v/v). The discharge/charge characteristics of the samples were investigated by cycling in the 0.001–3 V potential range at various current densities. Cyclic voltammograms were measured at a scan rate of 0.1 mV s⁻¹.

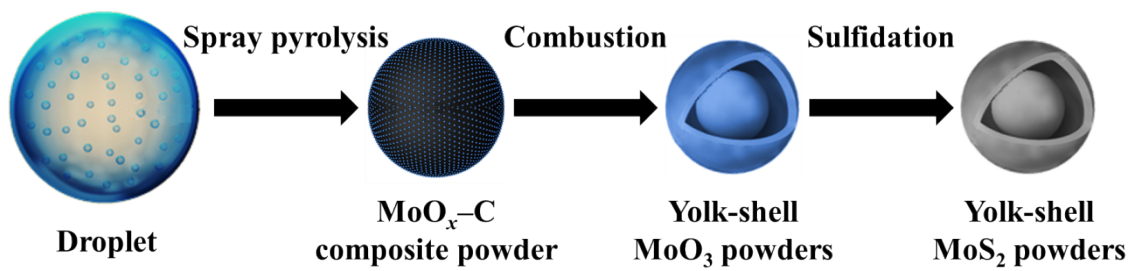


Figure S1. Schematic diagram of the formation process of yolk-shell MoS₂ powders.

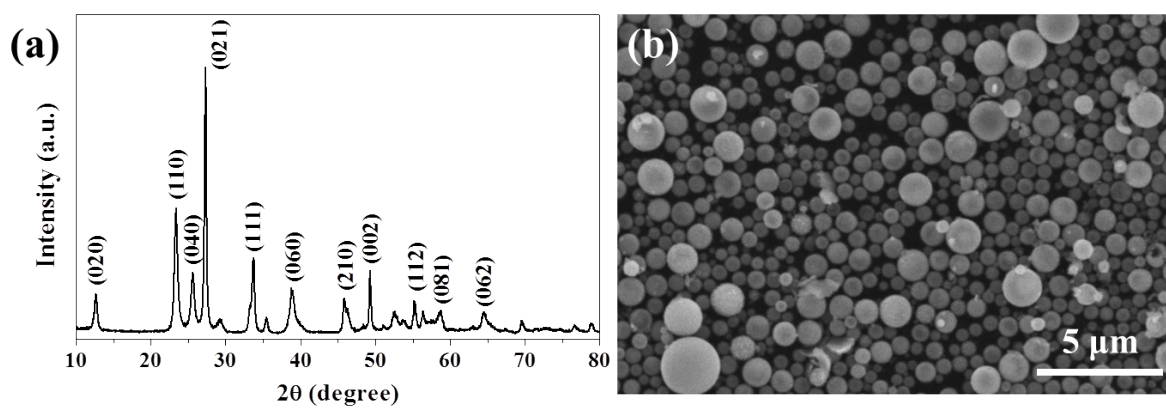


Figure S2. (a) XRD pattern and (b) SEM image of the yolk-shell MoO_3 precursor powders.

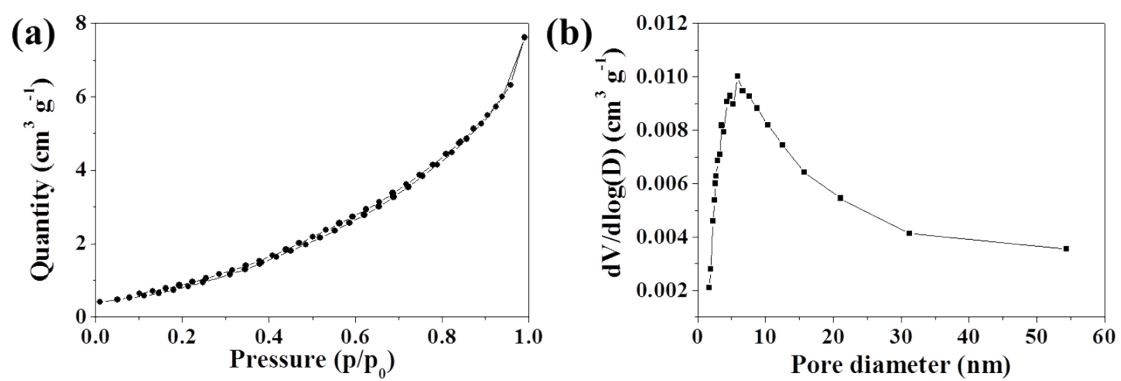


Figure S3. (a) Nitrogen adsorption-desorption isotherm plot and (b) pore size distribution curve of the yolk-shell MoS_2 powders.

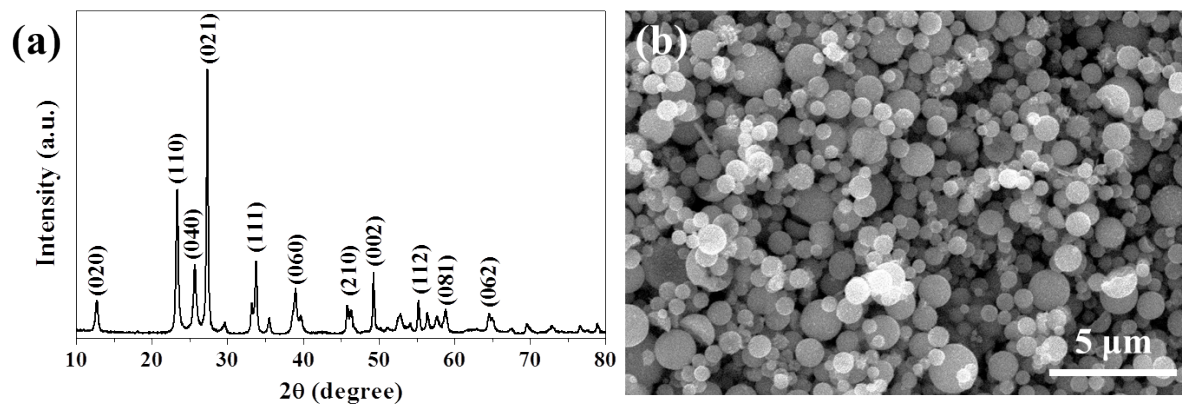


Figure S4. (a) XRD pattern and (b) SEM image of the dense MoO_3 precursor powders.

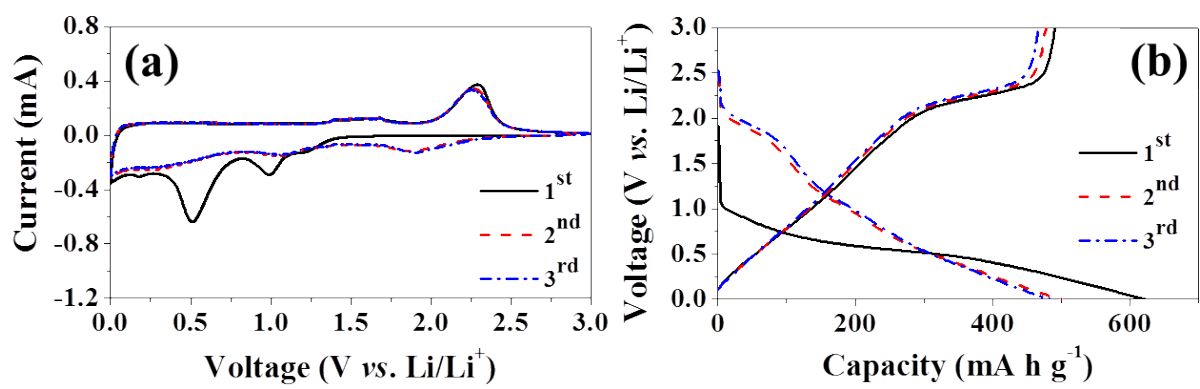


Figure S5. Electrochemical properties of the dense MoS₂ powders: (a) CVs and (b) discharge/charge voltage profiles.

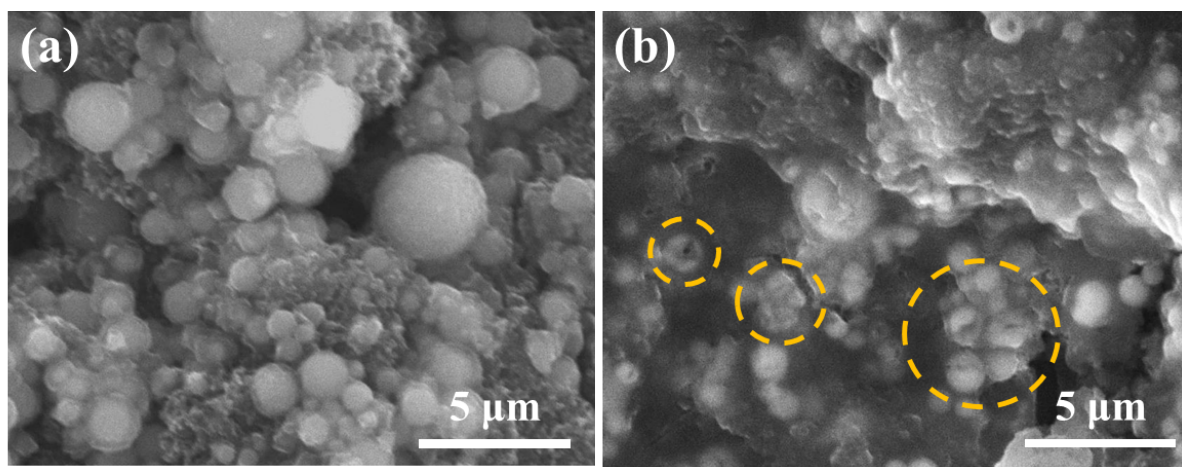


Figure S6. SEM images of the (a) yolk-shell and (b) dense MoS₂ powders after 100 cycles.

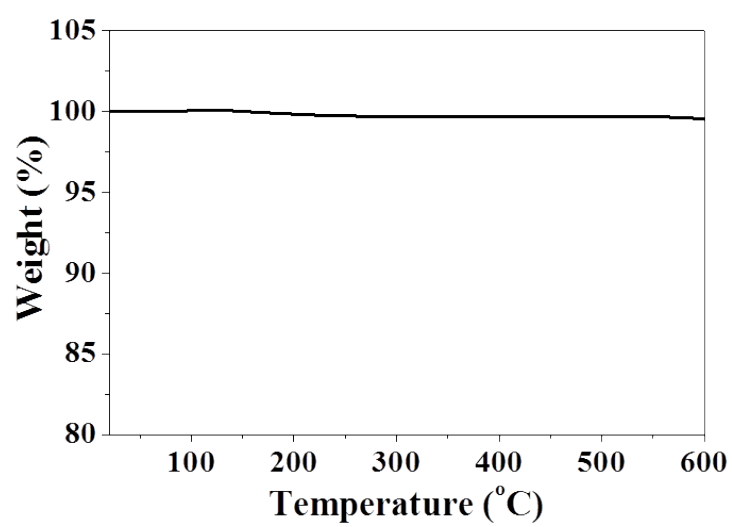


Figure S7. TG curve of the yolk-shell MoO₃ powders used as the precursor powders for MoS₂.