

Supporting Information for

Self-assembled MoS₂-carbon nanostructures: influence of nanostructuring and carbon on lithium battery performance

Shyamal K. Das,^a Rajesh Mallavajula,^a Jayaprakash Navaneethakrishnan,^a and Lynden A. Archer^{*a}

Experimental details

Synthesis of MoS₂-carbon: The MoS₂-carbon composites with varying carbon weight fractions were synthesized by a hydrothermal method. Resorcinol/formaldehyde (Sigma-Aldrich) and ammonium tetrathiomolybdate (Sigma-Aldrich) were used respectively as carbon and MoS₂ precursors. A desired concentration (0.076 M) of aqueous solution of ammonium tetrathiomolybdate was added to another aqueous solution containing resorcinol, formaldehyde and sodium carbonate under continuous stirring. The ratios of resorcinol to formaldehyde and to sodium carbonate were kept at 0.185 g ml⁻¹ and 251 respectively calculated on a molar basis for all MoS₂-carbon composites. However, the concentrations of resorcinol, formaldehyde and sodium carbonate were varied to obtain various carbon loadings in the final product. The intense violet color sol was transferred to a Teflon-lined stainless steel autoclave of capacity 100 ml (70% filling) and heated at 180 °C for 12 h and then cooled to room temperature. The resultant black product was recovered by centrifugation and washed with deionized water and freeze dried. The dried product was further calcined at 550 °C for 4 h in an atmosphere of 5% H₂ balanced with Ar at a heating rate of 5 °Cmin⁻¹. Pure MoS₂ was synthesized by hydrothermal treatment of ammonium tetrathiomolybdate (180 °C for 12 h, calcination at 550 °C for 4 h under H₂/Ar), but without any addition of resorcinol and formaldehyde. The materials were designated as MS-0, MS-11, MS-22, MS-32 and MS-41 corresponding to 0, 11, 22, 32 and 41 wt % of carbon in the MoS₂-carbon composites.

Characterizations: The crystallographic phase identification was performed using powder x-ray diffraction (Scintag theta–theta PAD-X-ray Diffractometer; Cu-K_α radiation, λ = 1.5406 Å). The morphology was observed by scanning electron microscopy (SEM, LEO

1550 FESEM) and transmission electron microscopy (TEM, FEI Tecnai G2 T12). Specific surface area (BET) was obtained from nitrogen adsorption-desorption isotherms (Micromeritics ASAP 2020). Estimation of carbon content in MoS₂-carbon composites was done using thermogravimetric analysis (TGA, TA Instruments Q5000). TGA experiments were performed by heating the sample in air from room temperature to 700 °C at a heating rate of 10 °Cmin⁻¹. For the working electrode, slurry of the active material and carbon black (Super P Timacal) was prepared with PVdF (Sigma) in a weight ratio of MoS₂:CB:PVdF = 90:0:10, 80:10:10, 65:10:10, 40:50:10 in *N*-methyl-pyrrolidone (NMP). The slurry was cast on a copper foil and dried in vacuum at 120 °C for 12 h. Room temperature cyclic voltammetry (CV, CH608 CH Instruments) and galvanostatic charge/discharge cycling (Maccor) were done in 2032 coin-type cells with pure metal Li (Aldrich) as anode, Whatman glass fibre as separator and 1M LiPF₆ in ethylene carbonate (EC, Aldrich) and dimethyl carbonate (DMC, Aldrich) (1:1 w/w) as an electrolyte.

Supplementary Figures

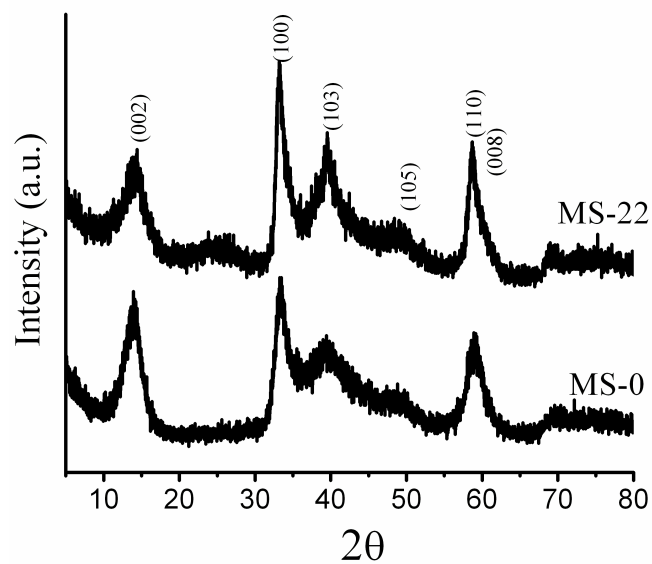


Figure S1. X-ray diffraction patterns of pure MoS₂ and MoS₂-carbon (22 wt %) composite.

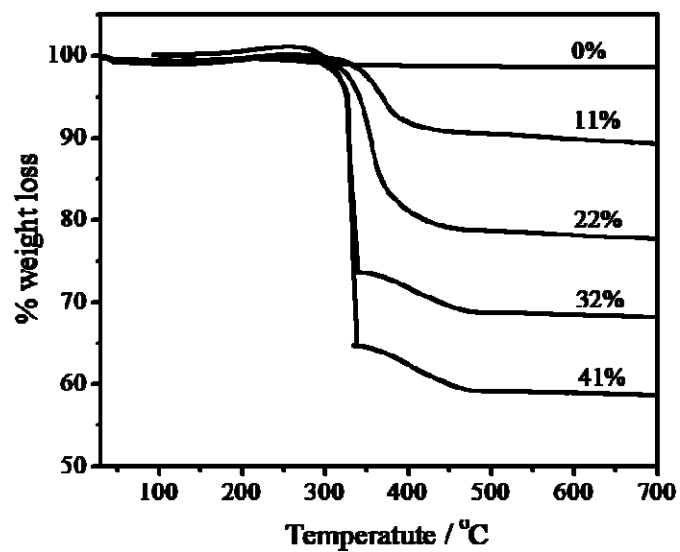


Figure S2. Thermogravimetry analysis of pure MoS₂, MS-11, MS-22, MS-32 and MS-41.

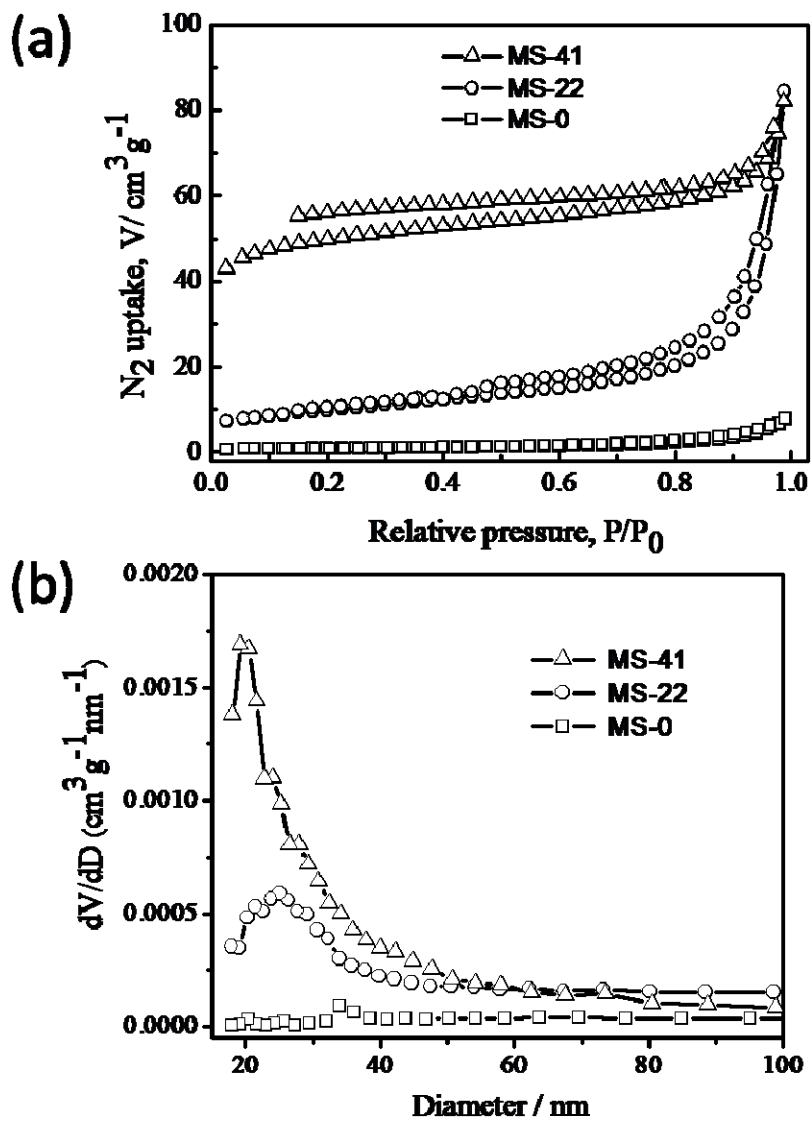


Figure S3. (a) N₂ adsorption/desorption isotherms and (b) pore size distribution of pure MoS₂, MS-22 and MS-41.

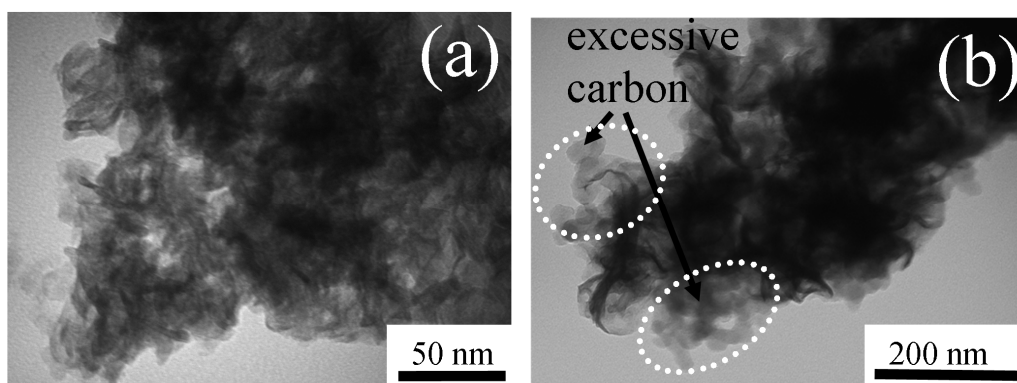


Figure S4. Transmission electron micrographs of (a) MS-11 and (b) MS-32.

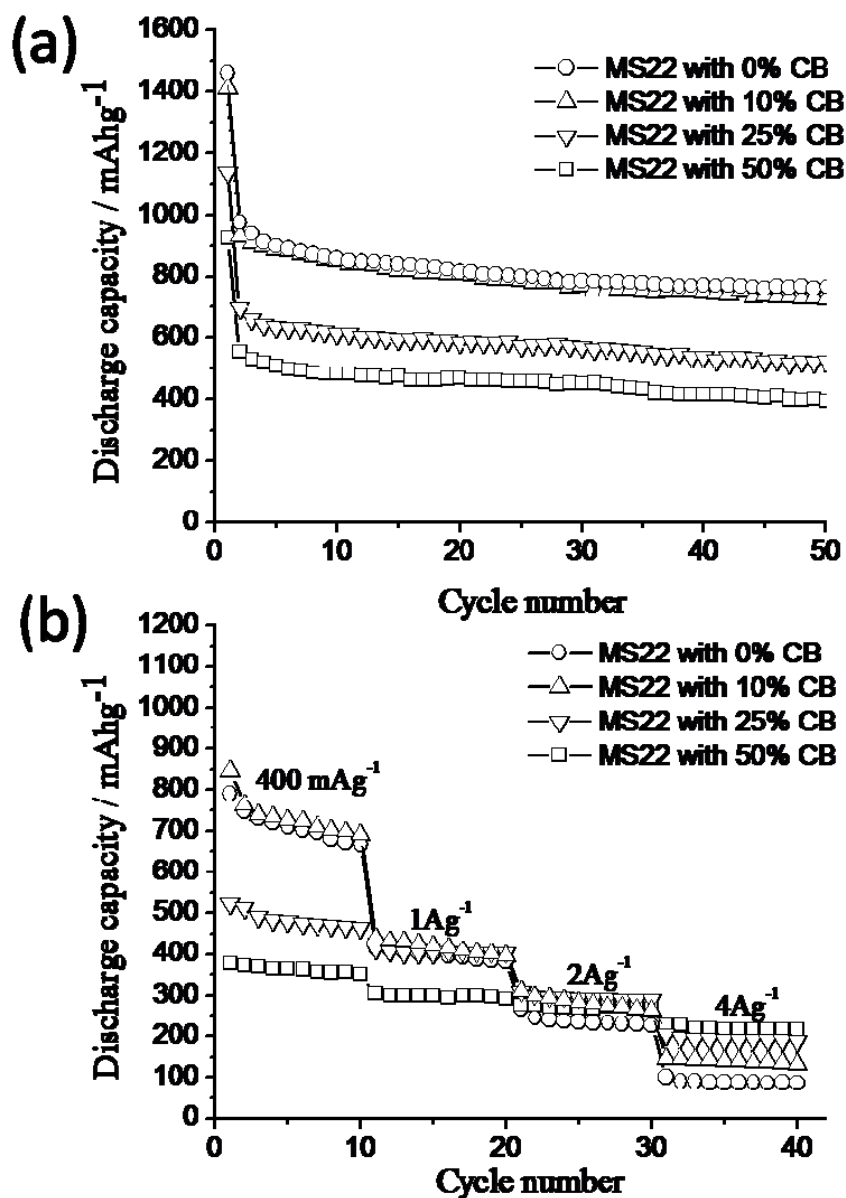


Figure S5. (a) Cycling stability of pure MS-22 with 0%, 10%, 25% and 50% carbon black in the electrode at a current rate of (a) 100 mA g⁻¹; (b) at various current rates in the range of 0.4-4 Ag⁻¹.

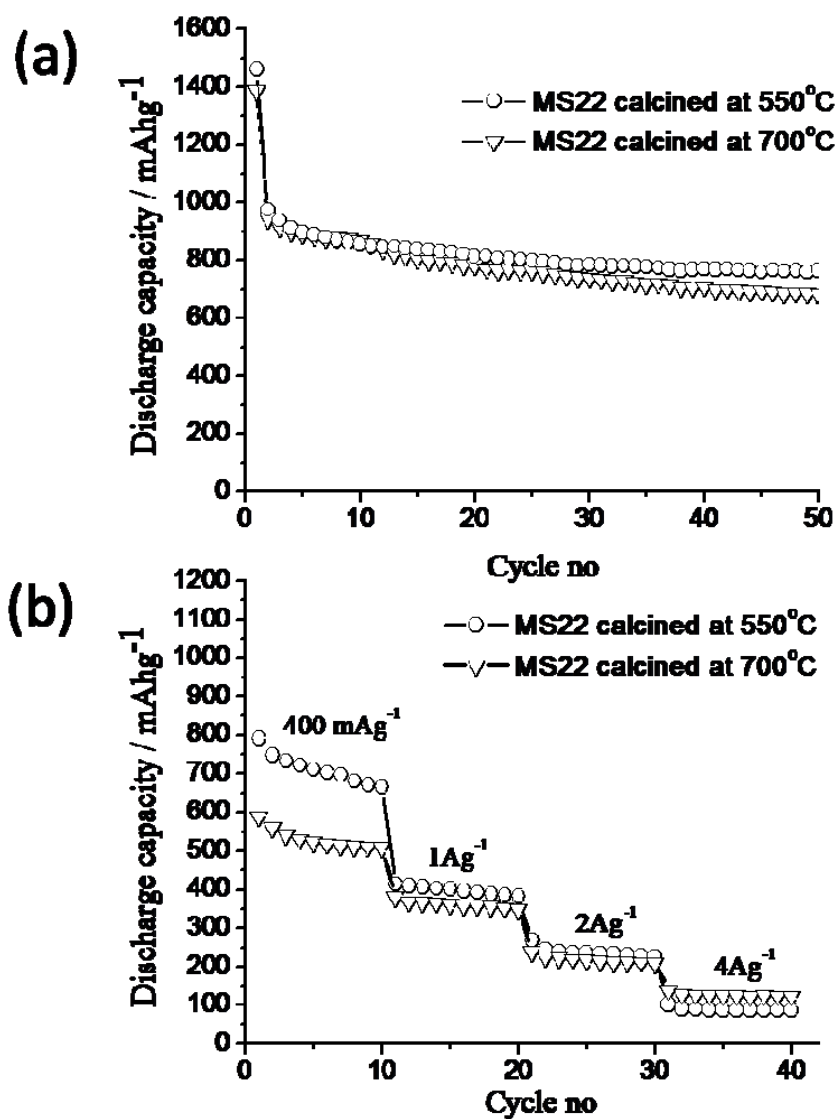


Figure S6. (a) Cycling stability of 550 °C and 700 °C calcined MoS₂-carbon (22 wt %) composite at a current rate of (a) 100 mA g⁻¹; (b) at various current rates in the range of 0.4-4 Ag⁻¹.

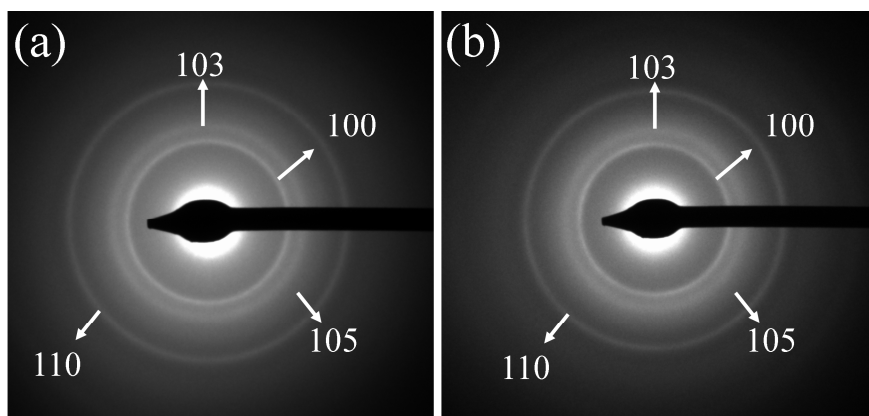


Figure S7. SAED patterns of (a) pure MoS₂ and (b) MoS₂-carbon (22 wt %) composite.