

Morphology-Controlled MoS₂ by Low-Temperature Atomic Layer Deposition

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Electrochemical Measurements

Electrochemical measurements were performed using a Bio-Logic VMP3 electrochemical workstation equipped with a three-electrode system and a 3 mm diameter glassy carbon rotating disc electrode (RDE). RE-1B (Ag/AgCl, 3M KCl) and a Pt wire were employed as reference electrode and counter electrode, respectively. The glassy carbon (GC) working electrode with a surface area of 7.069 mm² was rotated under a rotating speed of 1600 rpm. The catalyst inks were prepared by dispersing 2 mg sample of MoS₂/CNTs component and 16 μL Nafion (5 wt %, Sigma-Aldrich) in 384 μL of water/ethanol mixture (1:1 volume ratio) with 30 min sonication. Then, 3 μL catalyst ink was dropped on a GC electrode and dried at room temperature. The mass loading of MoS₂/CNTs on the GC electrode for the electrochemical catalytic reaction is 0.212 mg/cm². The electrochemical measurements were evaluated in 0.5 M H₂SO₄ at 25 °C and linear sweep voltammetry (LSV) curves were measured in the potential window -0.6 to 0 V (vs. RHE) with a scan rate of 5 mV/s.

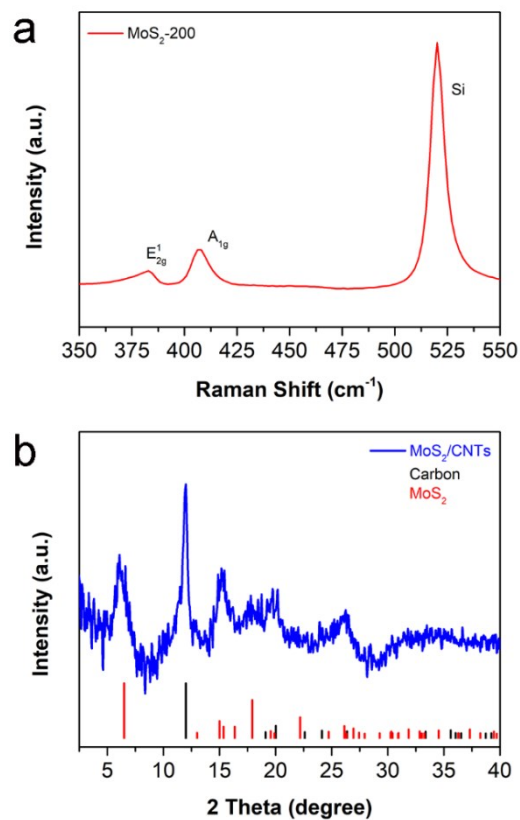


Fig. S1. (a) Raman spectrum of MoS₂-200 (MoS₂ deposited on Si wafer at 200 °C). (b) XRD of MoS₂/CNTs deposited at 200 °C with 500 ALD cycles.

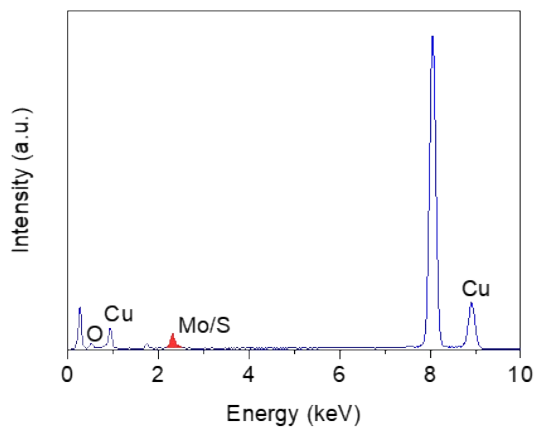


Fig. S2. EDX spectra for the MoS₂/CNTs-200 sample.

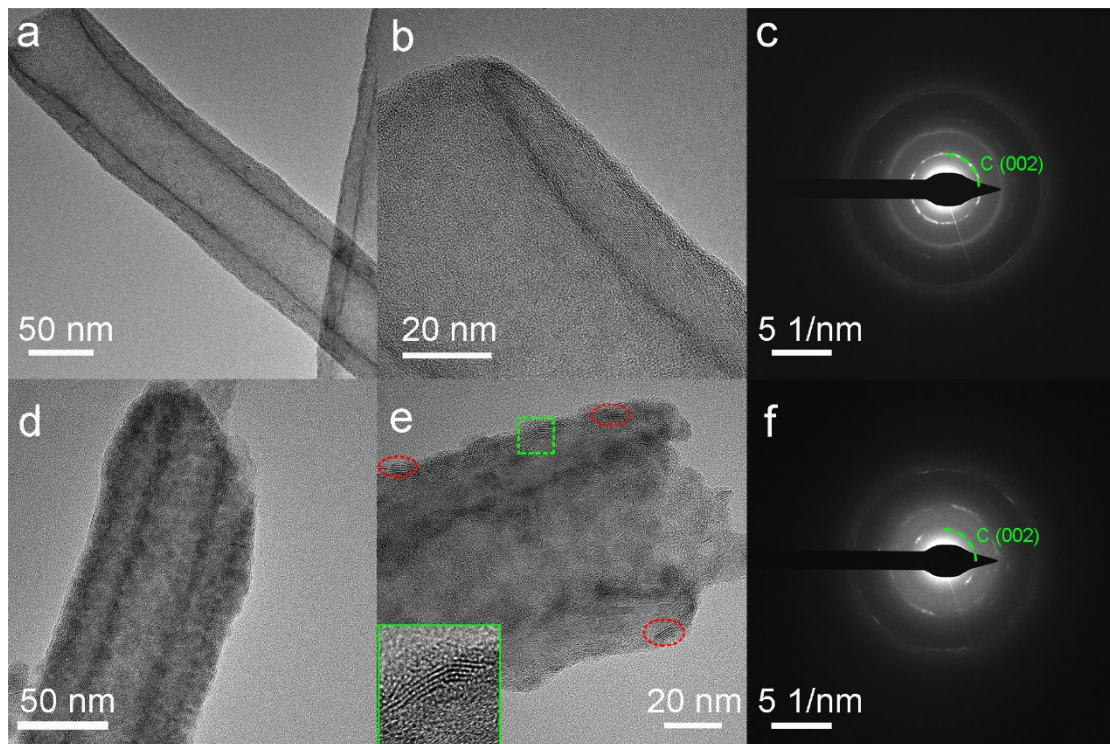


Fig. S3. TEM images of as-deposited MoS₂/CNT samples at 200 °C (a-b) and 250 °C (c-d).

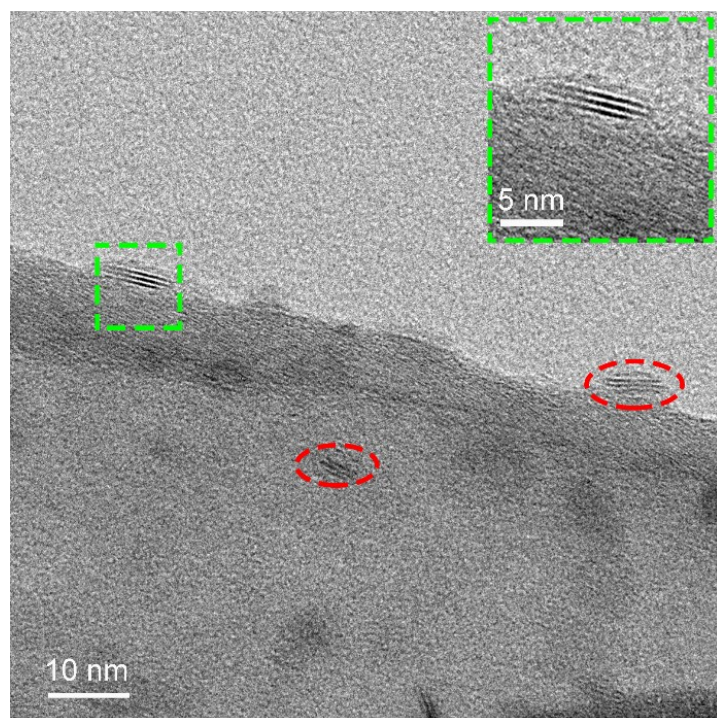


Fig. S4. High-resolution TEM image for MoS₂/CNT deposited at 300 °C without post-annealing.

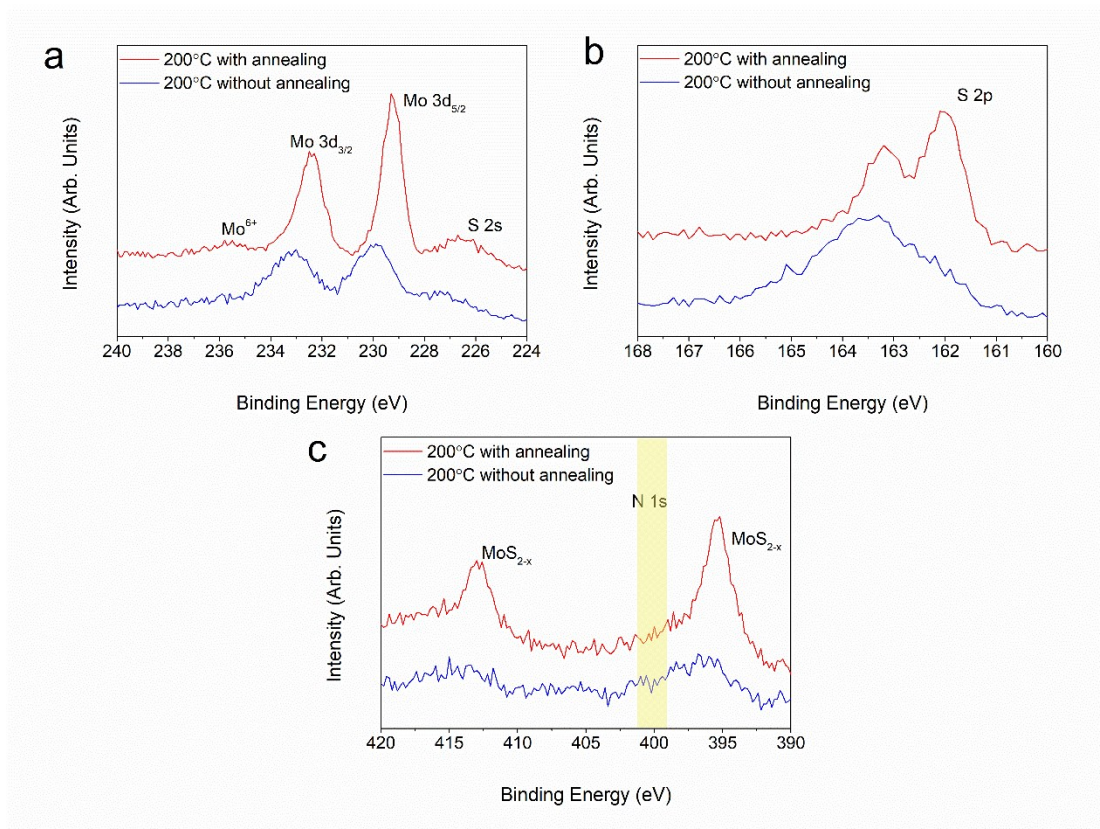


Fig. S5. XPS of Mo 3d (a), S 2p (b) and N 1s and Mo 3p (c) of MoS₂ deposited at 200 °C with (sample of MoS₂/CNTs-200) and without post-annealing.

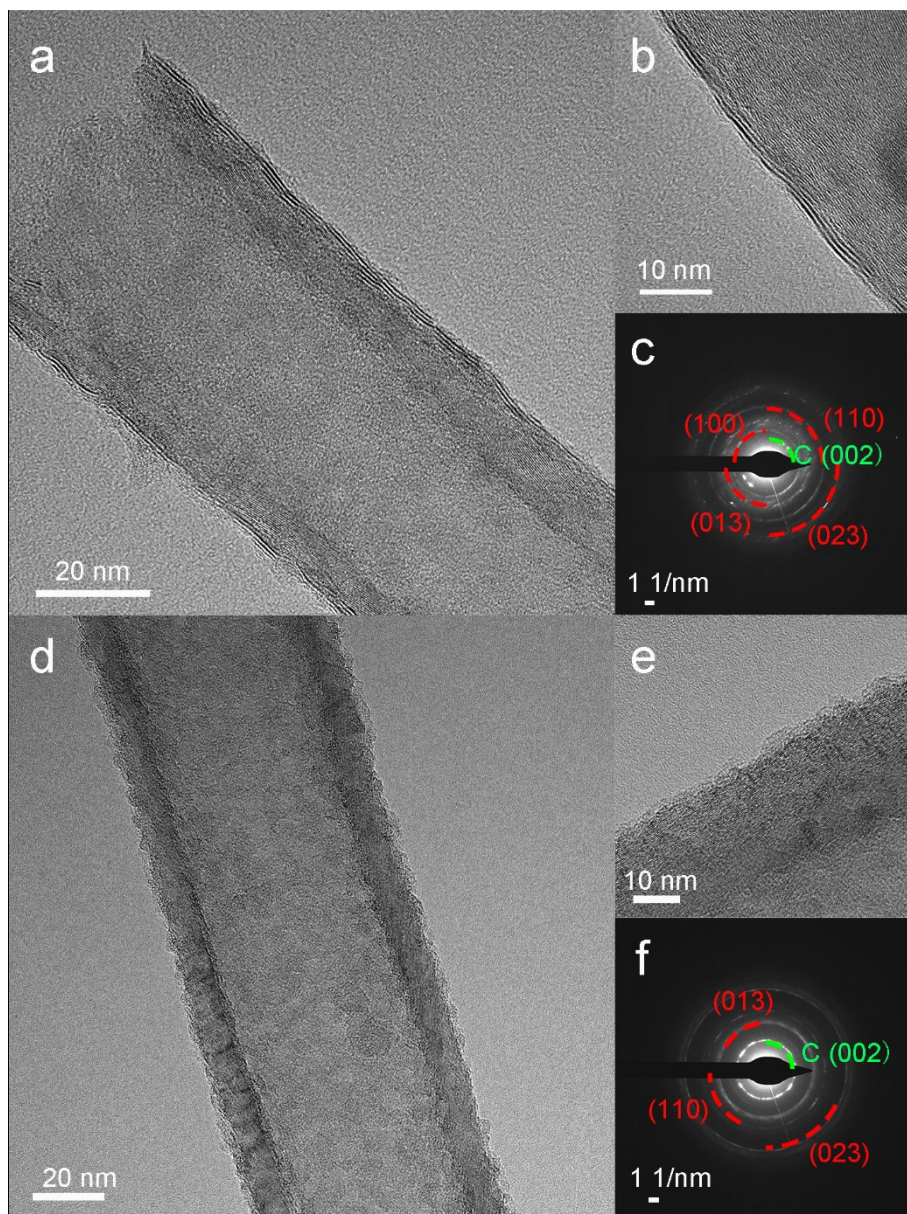


Fig. S6. TEM images of MoS₂ deposited on CNTs at 200 °C with 200 ALD cycles and annealed at 700 °C under Ar flow (a-c) and under forming gas (d-f) for 2h.

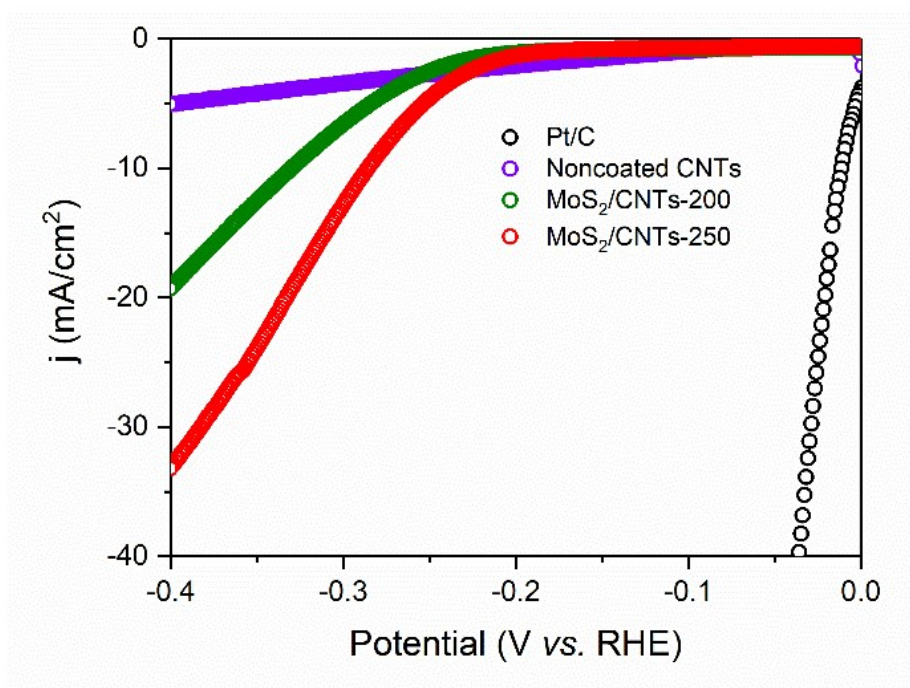


Fig. S7. HER performance of $\text{MoS}_2/\text{CNTs-200}$ and $\text{MoS}_2/\text{CNTs-250}$.