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Supporting Information

High Resolution Electrochemical Additive Manufacturing of Microstructured Catalyst Materials: Case study of MoS_x for Hydrogen Evolution Reaction

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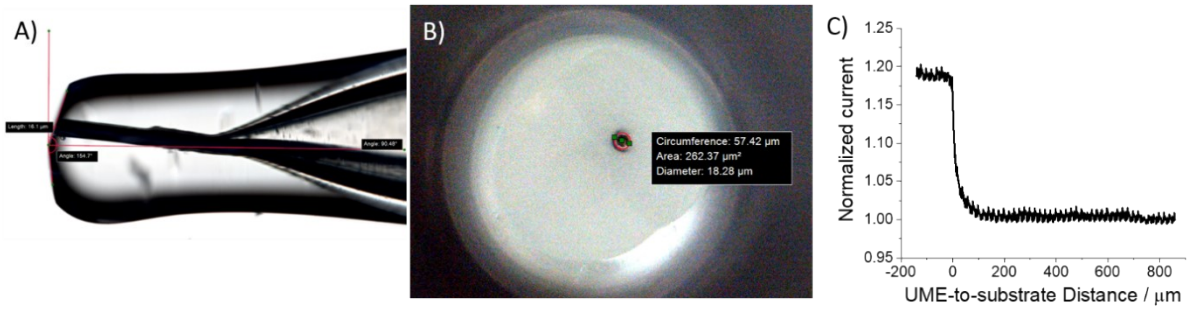

Figure S1: Optical images of the side (A) and bottom (B) view of the UME. The probe approach curve of the UME C). Electrochemical measurements were recorded in 1.5 mM FCMeOH with 0.2 M KNO_3 mediator solution. The max. approach speed of the UME was 2 µm s^{-1}. The insets in A) showing the values (from left to right) of Length: 16.1 µm, Angle 154.7°, Angle 90.48°.

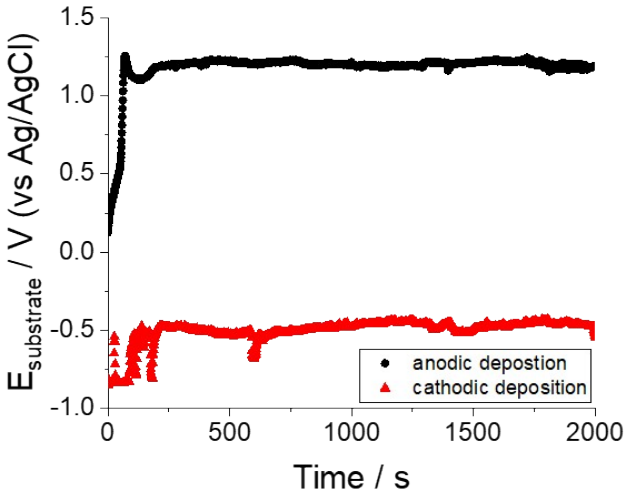

Figure S2: Chronopotentiograms of electrochemically printed MoS_x using direct mode-SECM with constant distance. The current settings for anodic deposition were $I_{Substrate}$ = 100 µA and for cathodic deposition $I_{Substrate}$ = -100 µA.

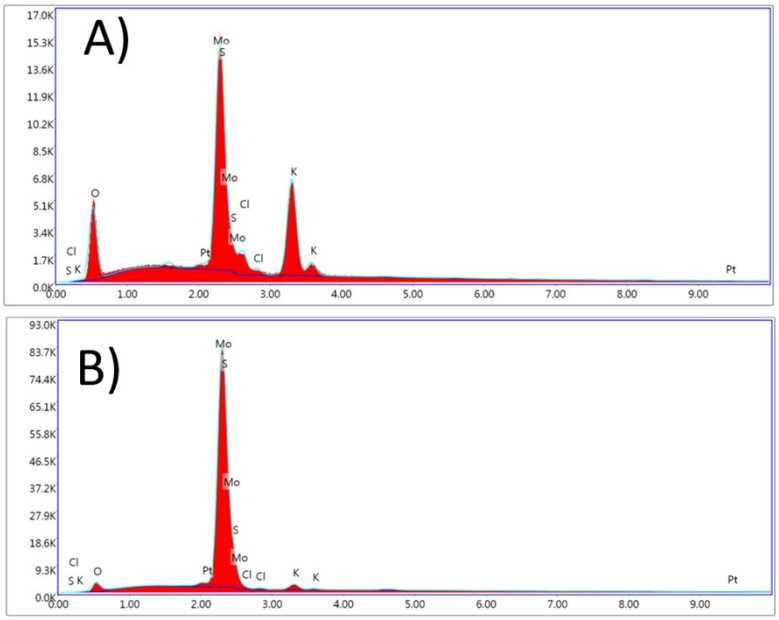

Figure S3: Energy dispersive X-ray spectroscopic spectra of anodic (A) and cathodic (B) printed MoS_x.

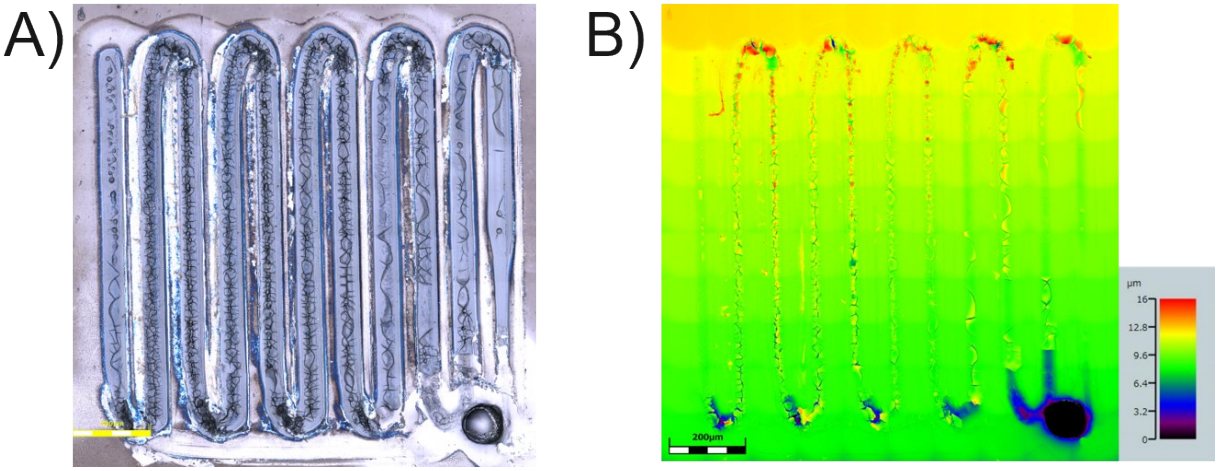

Figure S4: Optical image of the anodic printed MoS_x recorded with CLSM A) and CLSM height profile given in false color. The scale bars show 200 µm.

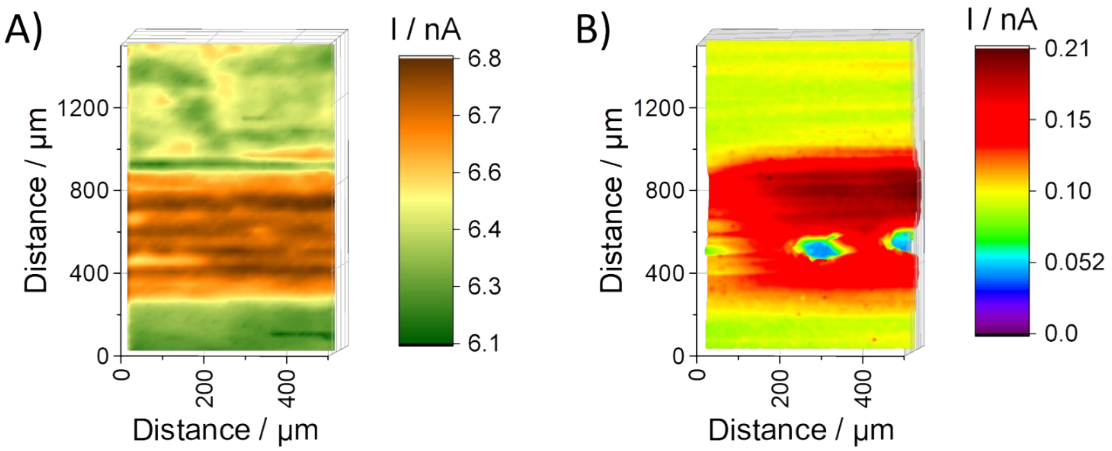

Figure S5: Scanning electrochemical microscopic characterizations of in subtractive mode printed cathodic MoS_x. (A) SECM micrograph recorded in feedback mode in 1.5 mM FcMeOH with 0.2 M KNO_3 with E_{UME} = 0.6 V and $E_{Substrate}$ = 0 V. (B) SECM micrograph recorded in SG/TC mode for the HER activity recorded in 0.5 M H_2SO_4 with E_{UME} = 0.3 V and $E_{Substrate}$ = -0.65 V. SECM images were recorded subsequently in constant height with a 25 µm diameter Pt UME, a pixel size of 10 x 20 µm, a max. scan speed of 100 µm s^{-1} and a waiting time of 4 ms.

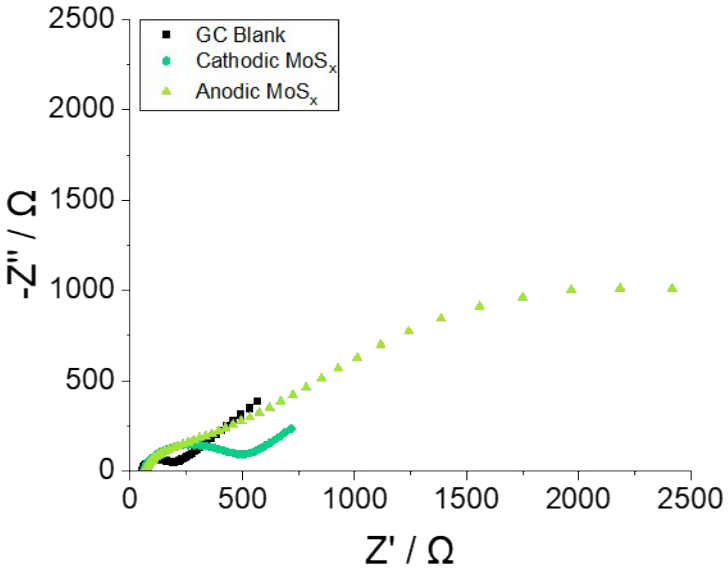

Figure S6: Electrochemical impedance spectroscopy. Nyquist plot of the bare GC electrode, anodic and cathodic printed MoS_x.

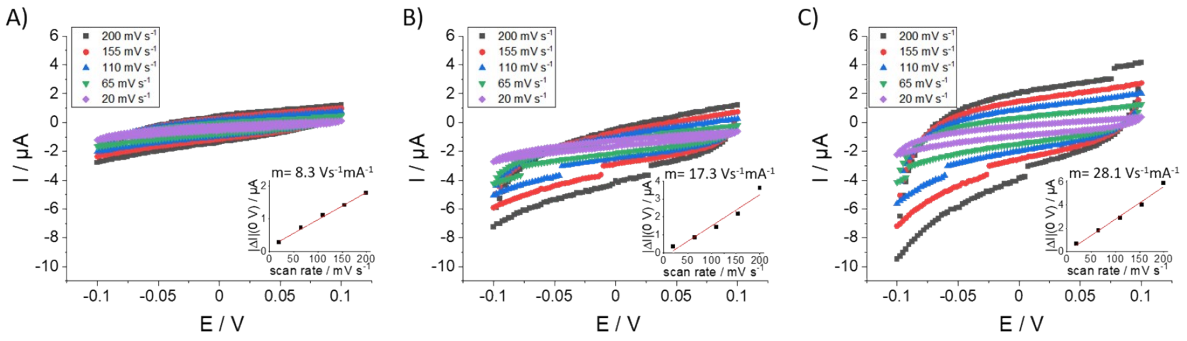

Figure S7: Cyclic voltammograms of the bare GC electrode (A), anodic (B) and cathodic (C) printed MoS_x with different scan rates in non-faradaic region in 0.5 M H_2SO_4 for characterization of the electrochemical surface area. The insets show the slope m of the linear correlation of the absolute current difference at $E_{Substrate}$ = 0 V.