

Electronic supplementary information (ESI) for Catalysis Science & Technology

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***In-situ* reconfiguration of plasma-engineered copper electrodes
towards efficient electrocatalytic hydrogenation**

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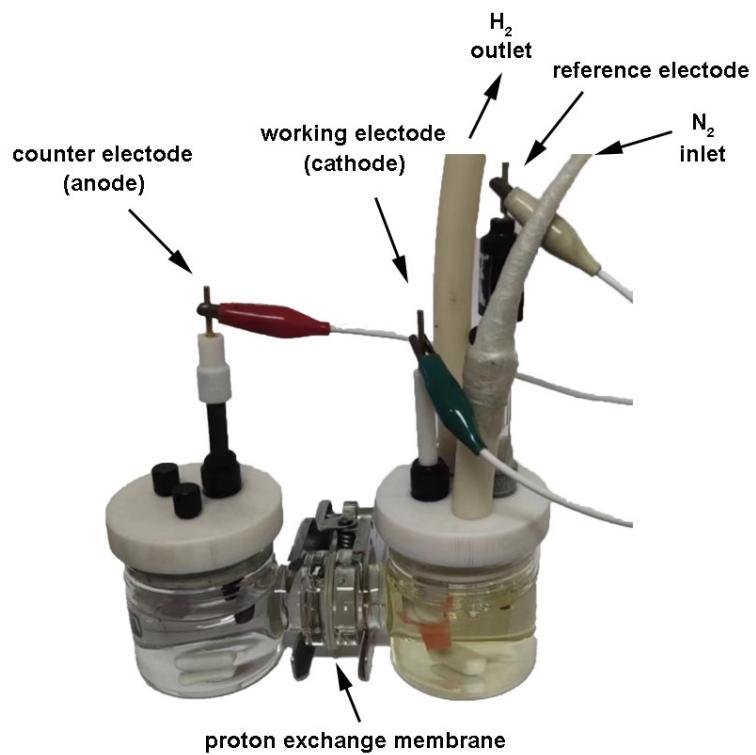


Fig. S1 Photograph of the setup for ECH experiment.

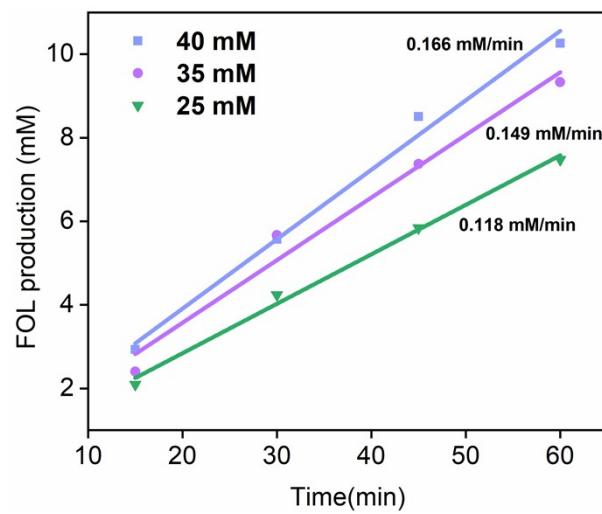


Fig. S2 FOL production with time under different initial FAL concentration during the initial electrolysis.

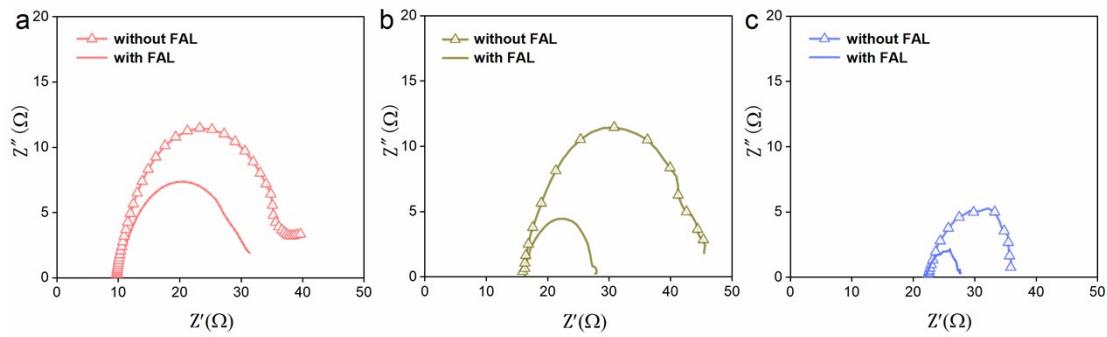


Fig. S3 Electrochemical impedance spectroscopy of (a) EP-Cu, (b) AP-Cu and (c) OP-Cu electrocatalysts without and with 35 mM FAL.

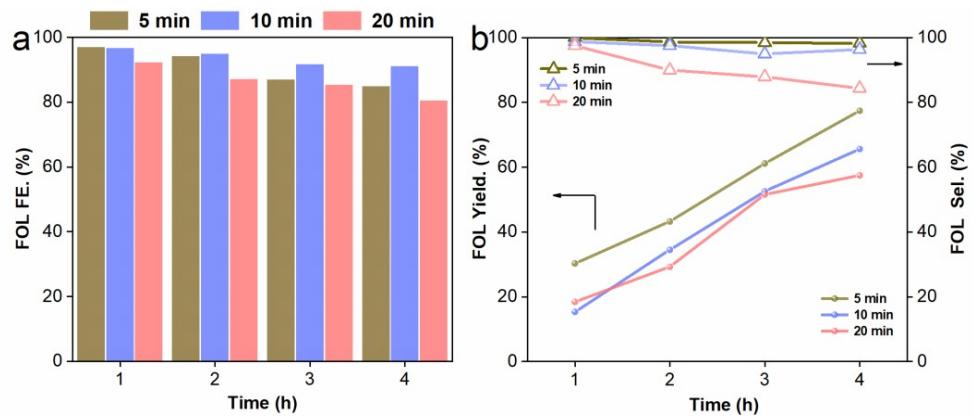


Fig. S4 (a) FE and (b) yield and selectivity of FOL on OP-Cu received with different O_2 -plasma etching time (5, 10 and 20 min).

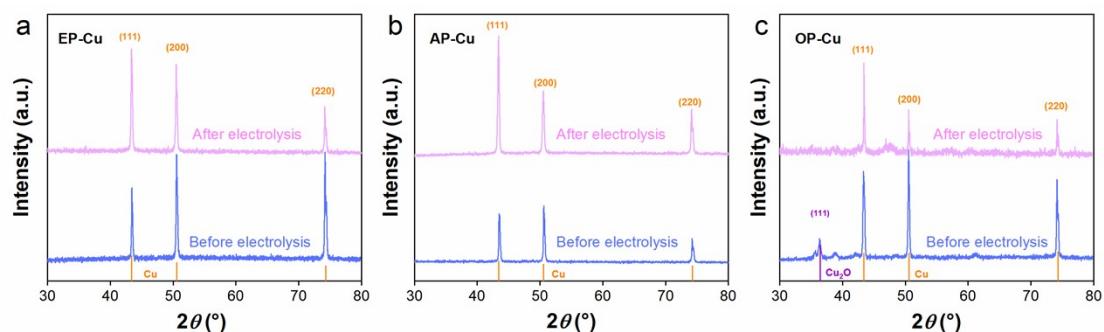


Fig. S5 XRD patterns of EP-Cu, AP-Cu and OP-Cu before and after the ECH of FAL at -0.56 V vs. RHE for 4 hours.

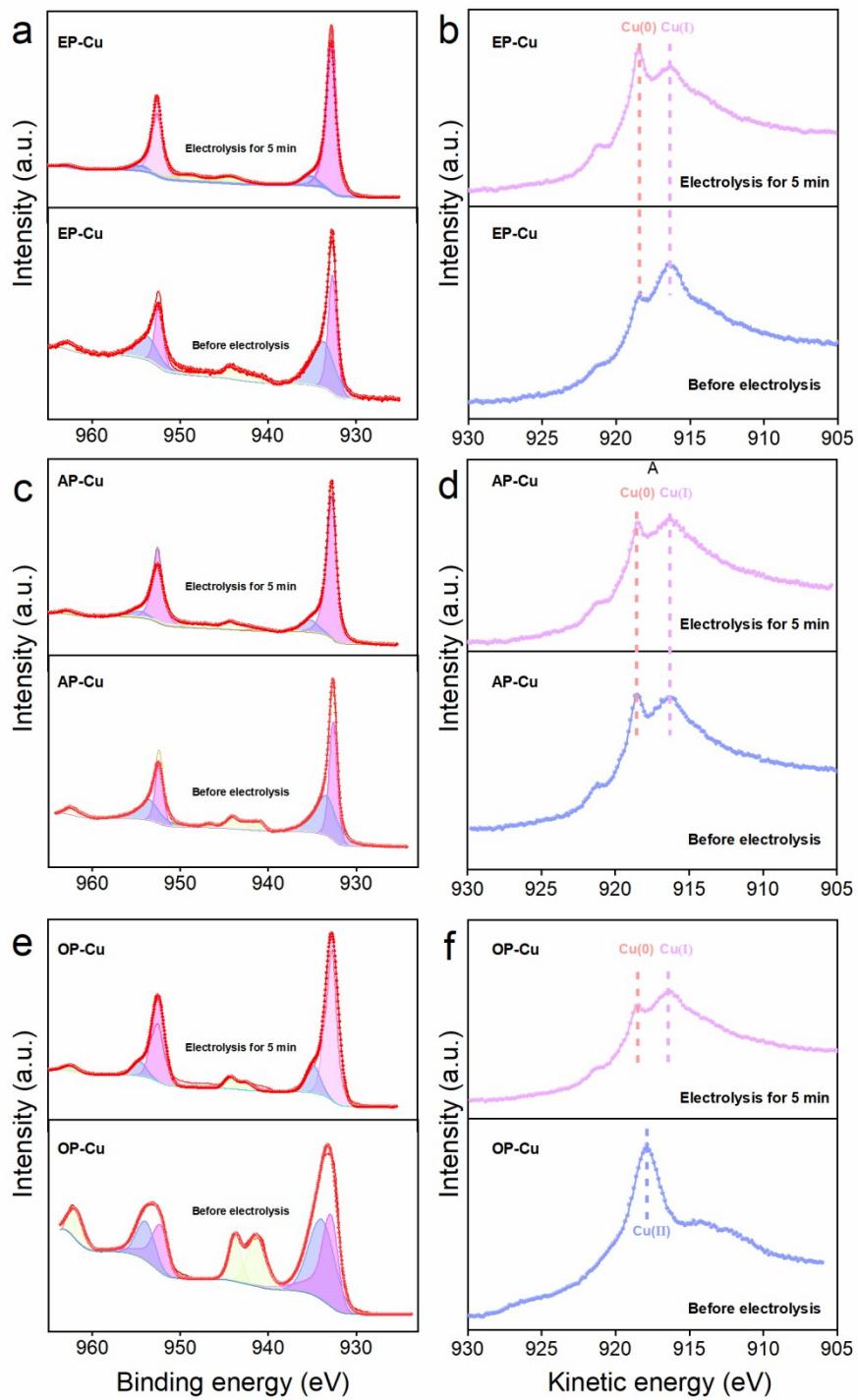


Fig. S6 XPS and Auger Cu LMM profiles of (a) and (b) EP-Cu, (c) and (d) AP-Cu, (e) and (f) OP-Cu before and after 5 min ECH at -0.56 V vs. RHE, respectively

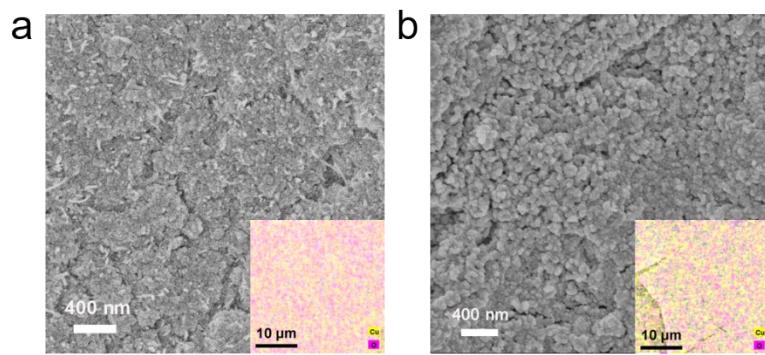


Fig. S7 SEM images of OP-Cu (a) before and (b) after 5 min ECH at -0.56 V vs. RHE.

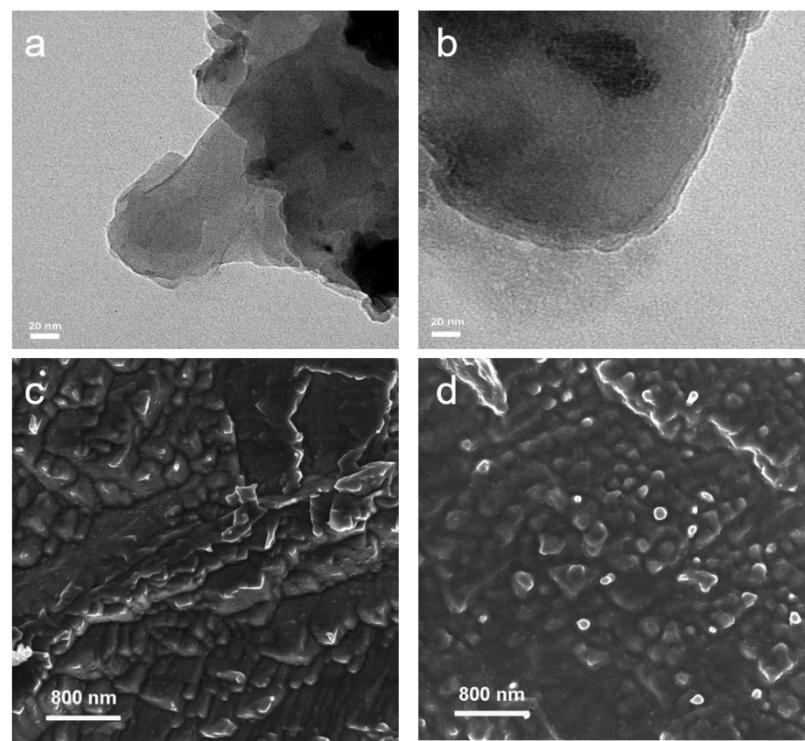


Fig. S8 (a and b) TEM and (c and d) SEM images of (a and c) EP-Cu and (b and d) AP-Cu after electrolysis (5 min) at -0.56 V vs. RHE.

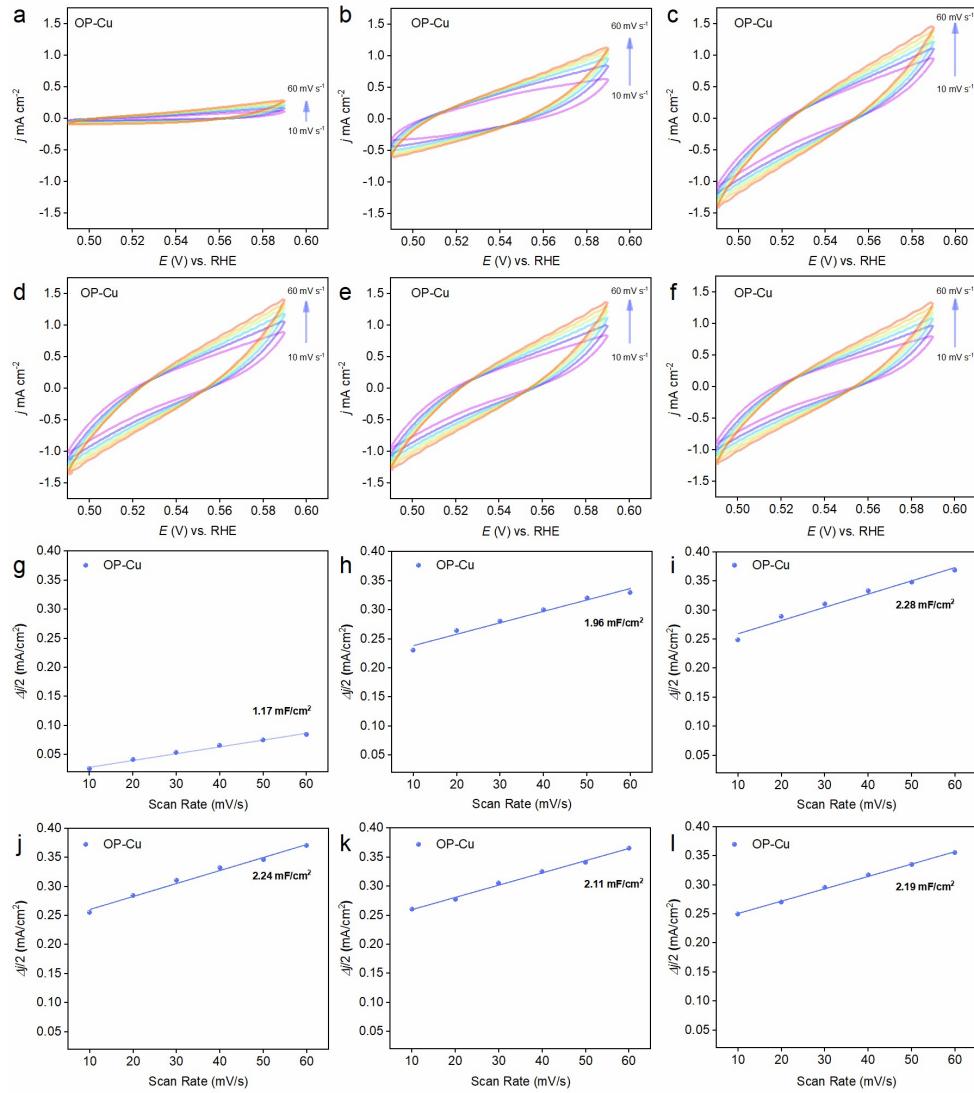


Fig. S9 (a ~ h) CV curves collected in N_2 -saturated 0.25 M phosphate buffer solution (pH 6.8) with different scan rates and (g ~ l) the corresponding charging current density variation ($\Delta j = (j_a - j_c)/2$, at 0.54 V vs RHE; data obtained from the CV in Fig. S9a – S9f) vs. scan rate plots of OP-Cu after the electrolysis for (a, g) 0 min, (b, h) 2 min, (c, i) 5 min, (d, j) 10 min, (e, k) 30 min, (f, l) 1 h.

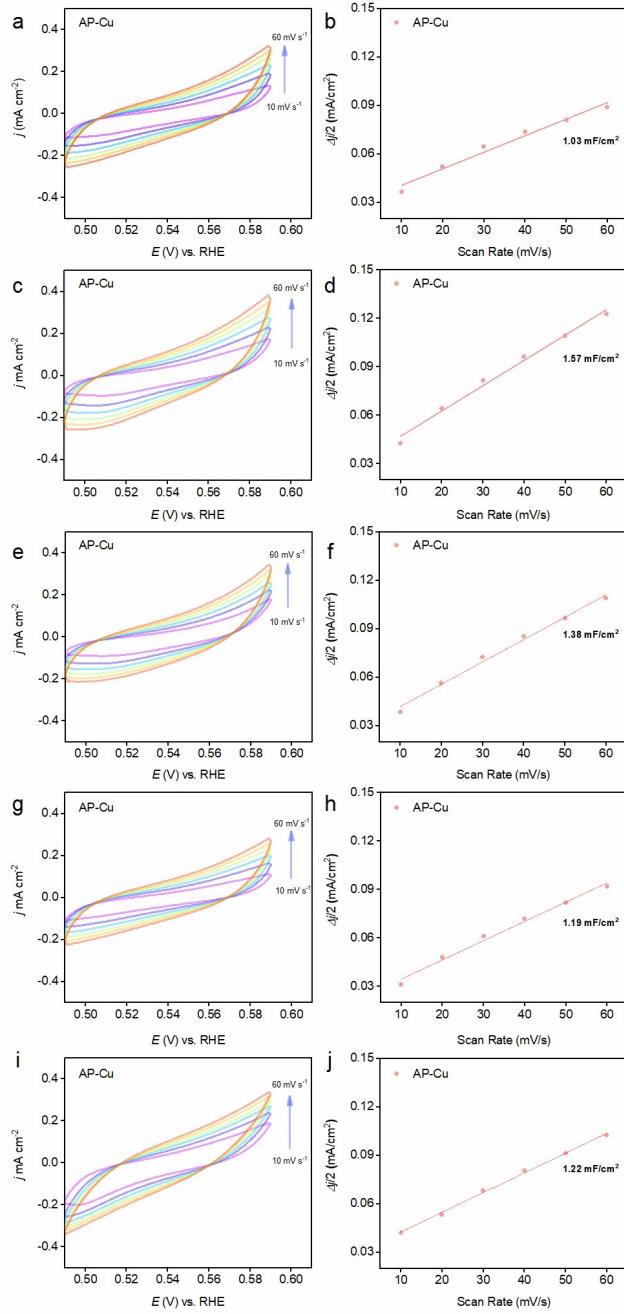


Fig. S10 CV curves collected in N_2 -saturated 0.25 M phosphate buffer solution (pH 6.8) with different scan rates and the corresponding charging current density variation ($\Delta j = (j_a - j_c)/2$, at 0.54 V vs RHE; data obtained from the CV in Fig. S10a, S10c, S10e, S10g and S10i) vs. scan rate plots of AP-Cu after electrolysis for (a, b) 0 min, (c, d) 5 min, (e, f) 10 min, (g, h) 30 min, (i, j) 1 h.

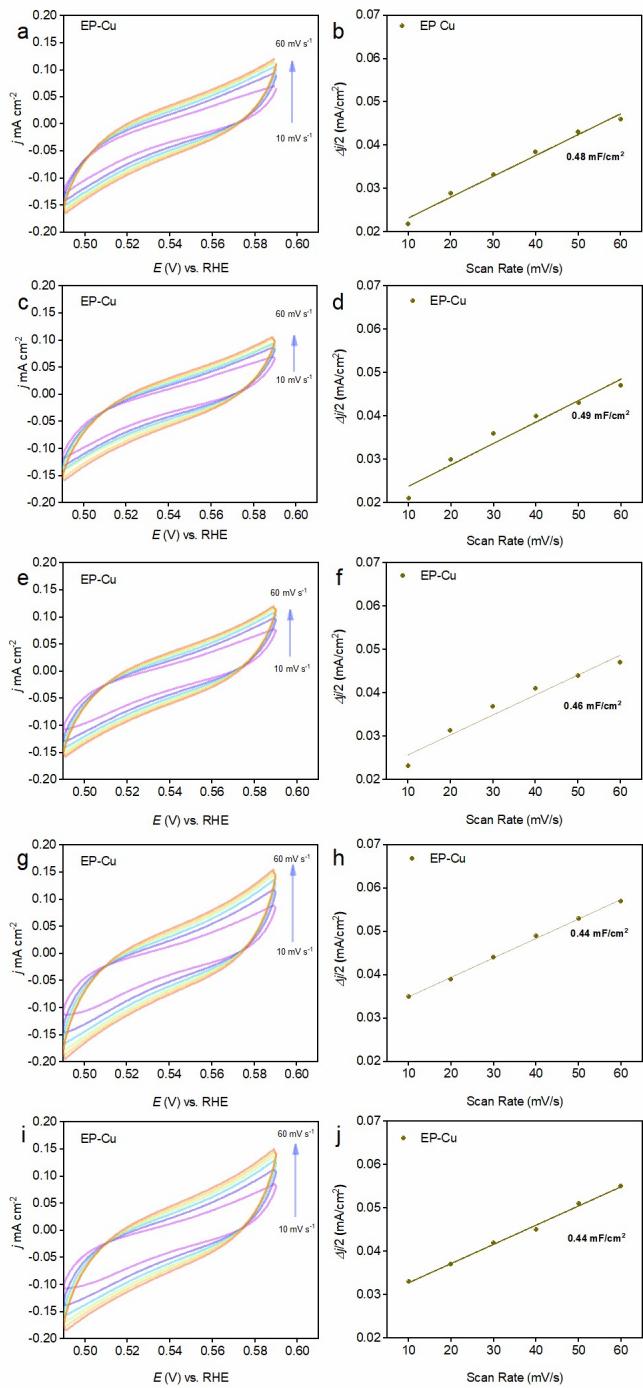


Fig. S11 CV curves collected in N_2 -saturated 0.25 M phosphate buffer solution (pH 6.8) with different scan rates and the corresponding charging current density variation ($\Delta j = (j_a - j_c)/2$, at 0.54 V vs RHE; data obtained from the CV in Fig. S11a, S11c, S11e, S11g and S11i) vs. scan rate plots of EP-Cu after electrolysis for (a, b) 0 min, (c, d) 5 min, (e, f) 10 min, (g, h) 30 min, (i, j) 1 h.

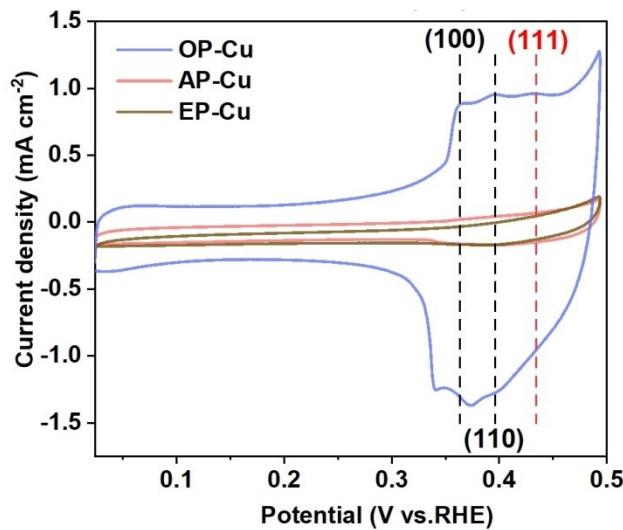


Fig. S12 CV curves collected in N_2 -saturated 0.1 M KOH for EP-Cu, AP-Cu and OP-Cu after 5 min electrolysis.

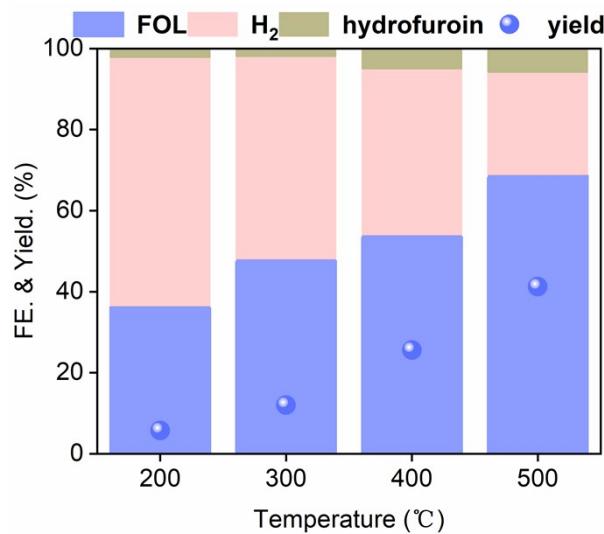


Fig. S13 FE and yield of FOL over heated Cu foils under a O_2 flow at different temperature. The electrolysis was conducted at -0.56 V vs. RHE for 4 hours in 0.25 M phosphate buffer solution (pH 6.8) containing 35 mM FAL.