Electronic Supporting Information

Ultrafast and Surfactant-Free Synthesis of Sub-3 nm Nanoalloys by Shear-Assisted Liquid Metal Reduction

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Fig. S1 TEM images and average particle size statistics for Pt-Cu-vxc72 synthesized by 10 μ L of reducing agent at 10 s and 10 min.



Fig. S2 (a) TEM, (b) HRTEM images and (c-e) HAADF-STEM images and its corresponding EDS elemental maps of Pt-Cu NPs. (f-h) HAADF-STEM images and its corresponding EDS elemental maps of Pt-Co NPs.



Fig. S3 XPS of Pt-Cu-Fe-vxc72 catalyst: (a) survey spectrum. (b-d) high-resolution spectra for XPS spectrum, (b) Pt 4f, (c) Fe 2p and (d) Cu 2p.



Fig. S4 The current-time plot of the Pt-Cu-vxc72 sample (as-synthesized by 160 mL of reducing agent at 1 min) in 0.5 M H_2SO_4 at an overpotential of 28 V vs RHE.



Fig. S5 HER polarization curves initial and after 5000 cycles of CV scanning for (a) Pt/C, (b) Pt-Cu-vxc72 and (c) Pt-Cu-Fe-vxc72. CV curves for (d) Pt/C, (e) Pt-Cu-vxc72 and (f) Pt-Cu-Fe-vxc72 initial and after 5000 cycles between +0.6 and -0.2 V vs. RHE.

We selected the Pt-Cu-vxc72 sample with the best catalytic performance for CV test, and calculated the ECSA value. Through calculation, it is found that the ECSA value of Pt-Cu is 5.26 m^2/g_{Pt} , which is slightly larger than the standard HER catalyst (the electrochemically active area value of Pt/C 20% is $3.11 m^2/g_{Pt}$), and Pt-Cu-Fe-vxc72 has the largest ECSA value ($6.416 m^2/g_{Pt}$). We further carried out a 5000 electrochemical test cycles. The change in the hydrogen adsorption peak area of the CV curve before and after the test can approximate the loss of ECSA. It can be seen from the Fig. S5 that the change in the peak area of the alloy prepared by adding transition metal is less than that of Pt/C. It shows from the side that its stability is stronger.



Fig. S6 HER polarization curves of Pt/C, Pt-Cu-vxc72 and Pt-Cu-Fe-vxc72 in 0.5 M H₂SO₄.

We also calculated the mass specific activity of the sample and the standard HER catalyst (Fig. S6). The result is shown in the picture. Compared with Pt/C, the alloy sample we synthesized has better catalytic performance.



Fig. S7 The variation of lattice parameters with Pt content.

According to Vigard's law, the lattice constant of a solid solution formed by atoms with the same crystal structure is an intermediate value of the two lattice constants. As can be seen from the Fig. S7, we have calculated the lattice parameters of the synthesized PtCu alloy with different Pt mole fraction. As the amount of reducing agent increases, the degree of alloying also increases.