Supporting Information

Ir-Cu Nanoframes: One-pot Synthesis and Efficient Electrocatalysts for Oxygen Evolution Reaction

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Experimental Details

Chemicals and Materials: All the reagents used in this work, including various kinds of metal inorganic salts, oleylamine, oleic acid octylamine ethanol, and cyclohexane, were of analytical grade from the Beijing Chemical Factory. Iridium chloride hydrate ($IrCl_3·3H_2O$, 34.7% Ir basis), copper(II) acetylacetonate, and Nafion solution were purchased from Alfa Aesar. All chemicals were used as received.

Preparation of Ir-Cu nanoframes:

In a typical synthesis of Ir-Cu nanoframes, $IrCl_3·3H_2O$ (10mg), $Cu(acac)_2$ (15mg) and CTAB (50mg) was added into mixture solution which were incorporated with 0.5ml oleylamine, 0.5 ml oleic acid and 5 ml octylamine, respectively. Then the mixture was magnetically stirred for 30 minutes at room temperature until it turned homogeneous solution. The resultant solution was transferred to a 10 ml Teflon-lined stainless steel autoclave and the sealed vessel was kept at 170℃ for 24h. After it was cooled down to room temperature, the resulting black products were collected via centrifugation at 9000 rpm for 5 minutes and further purified by ethanol/cyclohexane mixture for three times.

Preparation of Ir-Cu nanoparticles:

Based on the preparation process of the nanoframes, merely needed to add 0.5 ml ethanol to the mixture solution.

Preparation of Ir-Cu-Au ternary nanoframes:

5 ml oleylamine and 1ml oleic acid were added into 25 ml single port round bottom flask, then the as-prepared Ir-Cu nanoframes solution and 0.025 ml $HAuCl_4$ (0.1M) aqueous solution were drew into the mixture. Then the flask was heated in an oil bath at 50℃ for 12 hours.

Instrumentation: The powder XRD patterns were recorded with a Bruker D8-advance X-ray powder diffractometer with CuKα radiation (λ= 1.5406 Å). The size and morphology of as-synthesized samples were determined by using Hitachi model H-

800 transmission electron microscope and JEOL-2010F high-resolution transmission electron microscope.

Electrochemical measurements: Electrochemical measurements were conducted on a computer-controlled electrochemical analyzer (CHI 660D, Chen hua, China). A conventional three-electrode cell was used, including a Ag/AgCl (KCl-saturated) electrode as the reference electrode, a platinum wire as the counter electrode, and a glassy carbon (GC) electrode as the working electrode. The GC electrode was first polished with 0.3 and 0.05 μm Al_2O_3 slurry on a polishing cloth and then sonicated in ethanol and water each for several times before use. To prepare the working electrode, an aqueous dispersion of Ir-based NCs was transferred onto the surface of the pretreated GC electrode using a microsyringe. After the electrode was dried, 35μL Nafion dilutes (5 wt%) was coated on the catalyst surface to enhance the samples' attachment. Aqueous solutions of 1 M KOH were used for the electrolytes. All electrochemical experiments were performed at room temperature.

Supplementary Figures

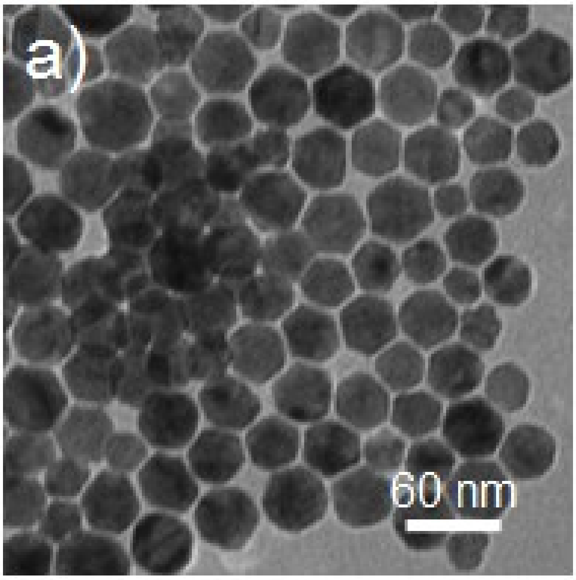
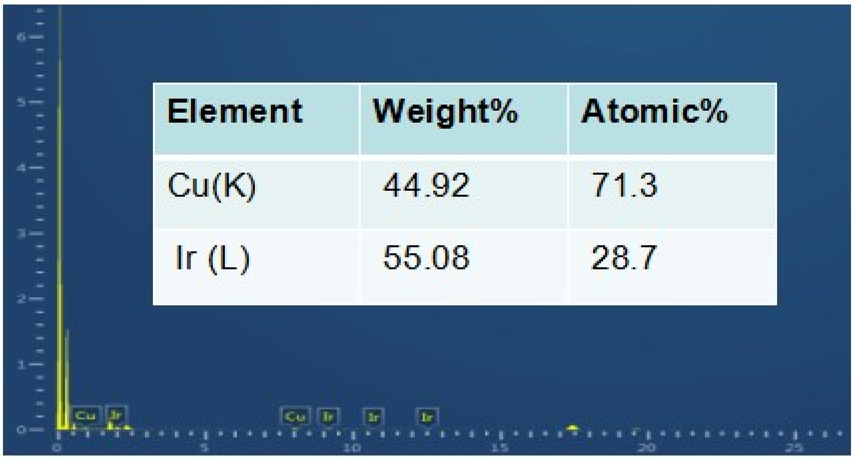


Figure S1. a) TEM image of Ir-Cu nanoparticles.



Element	Weight%	Atomic%
Cu(K)	44.92	71.3
Ir (L)	55.08	28.7

Figure S2. EDX spectrum of Ir-Cu nanoframes at 24 h

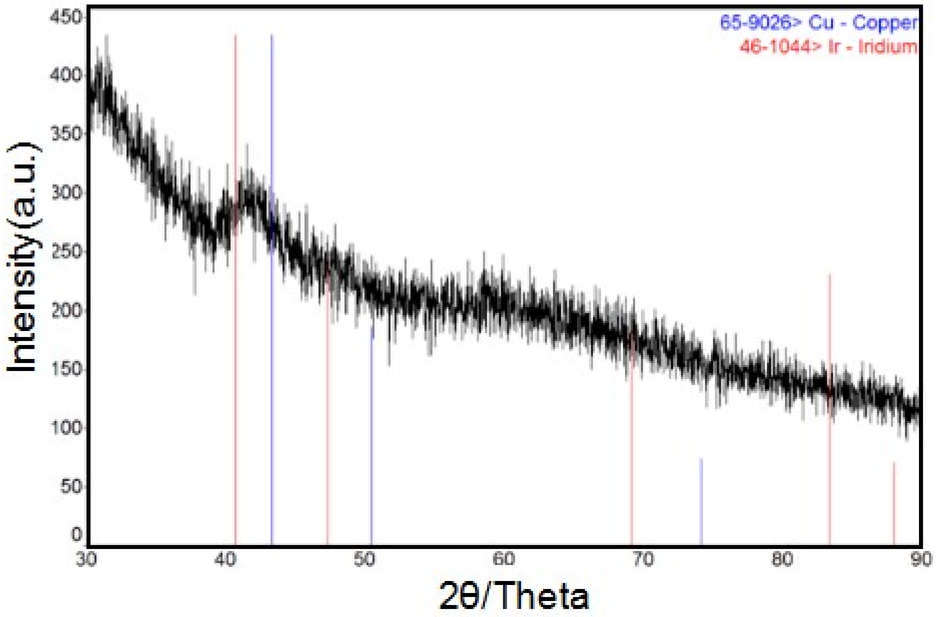
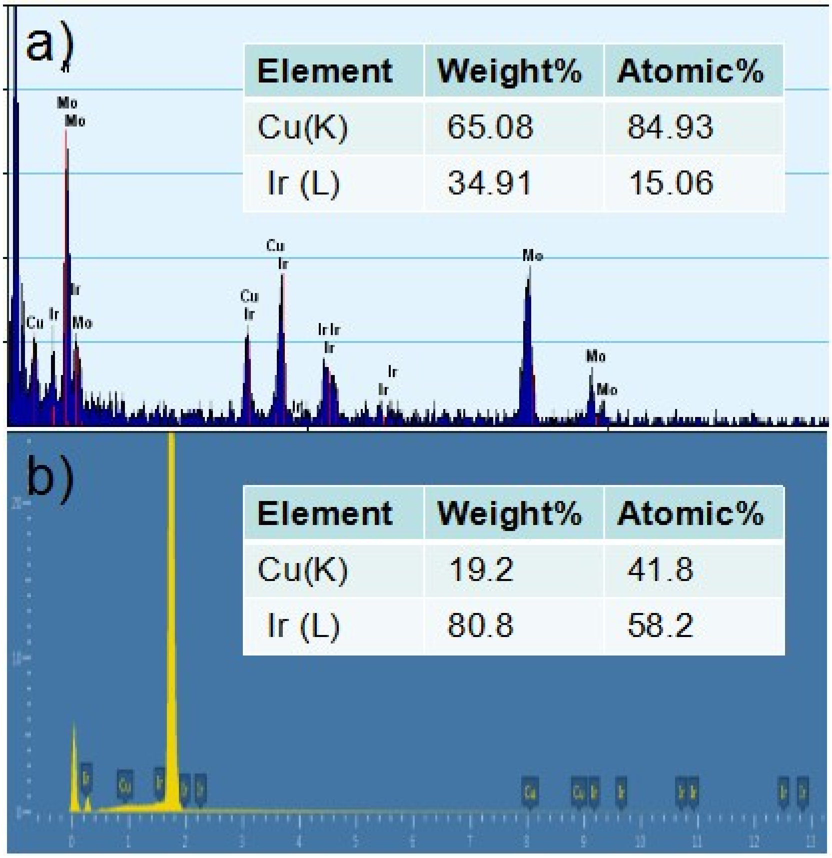


Figure S3. XRD pattern of Ir-Cu nanoframes at 24 h



a)

Element	Weight%	Atomic%
Cu(K)	65.08	84.93
Ir (L)	34.91	15.06

b)

Element	Weight%	Atomic%
Cu(K)	19.2	41.8
Ir (L)	80.8	58.2

Figure S4. EDX spectrum of Ir-Cu nanoframes at different time a):8h, b):16h.

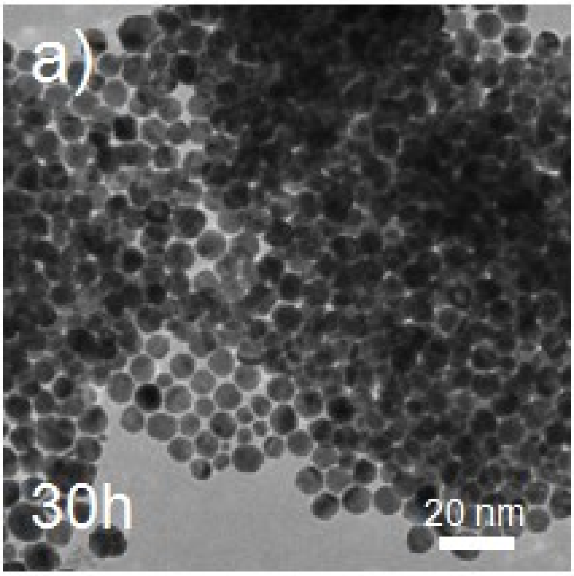


Figure S5.TEM images of the synthesized nanoparticles collected at 30h.

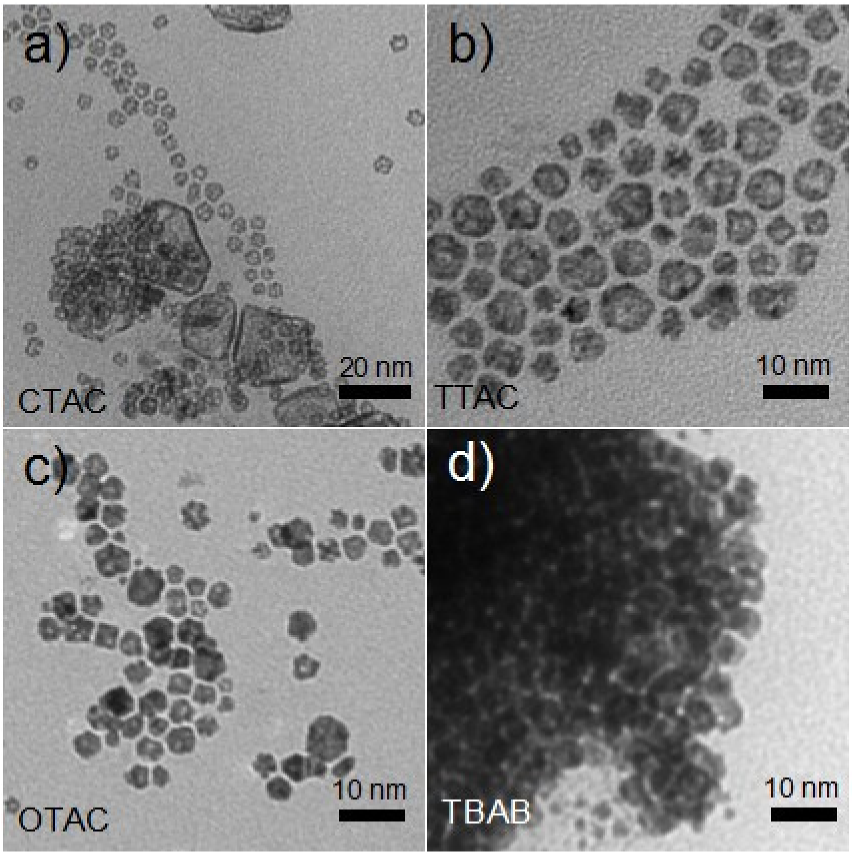


Figure S6. TEM images of the synthesized samples collected by transforming surfactant CTAB: a) CTAC, b) TTAC, c) OTAC, d) TTAB.

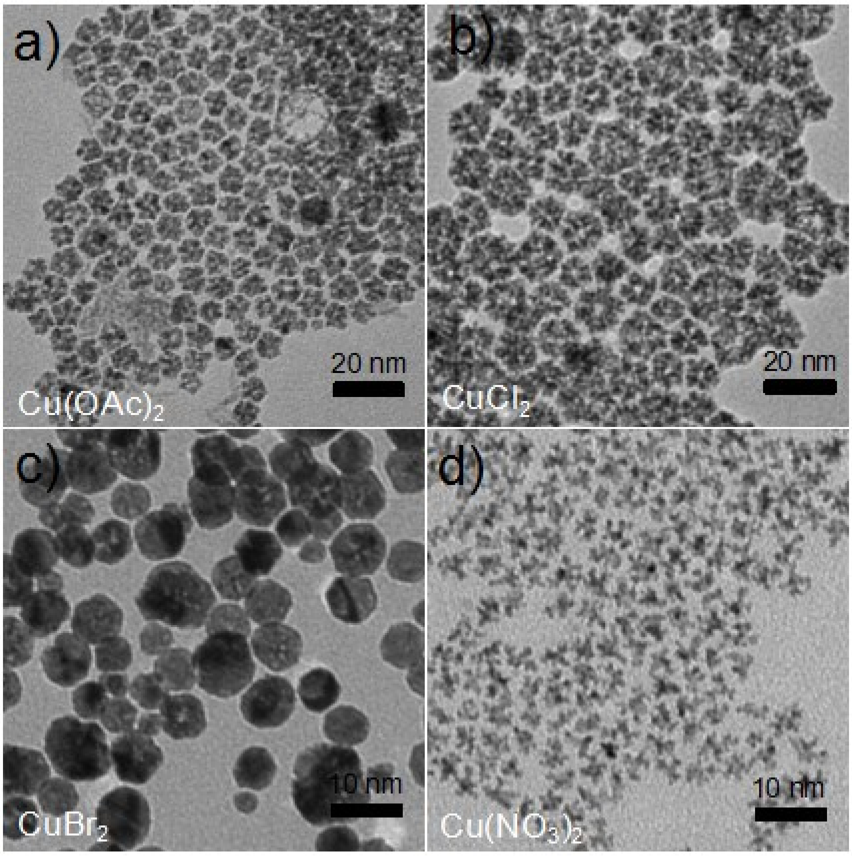
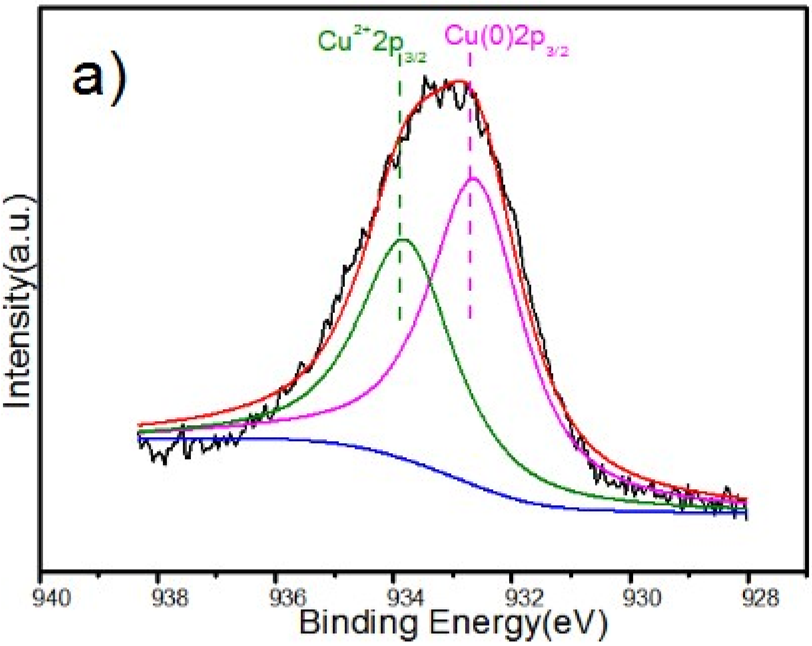


Figure S7. TEM images of the synthesized samples collected by transforming copper precursor: a) Cu(OAc)$_2$, b) CuCl$_2$, c) CuBr$_2$ d) Cu(NO$_3$)$_2$.



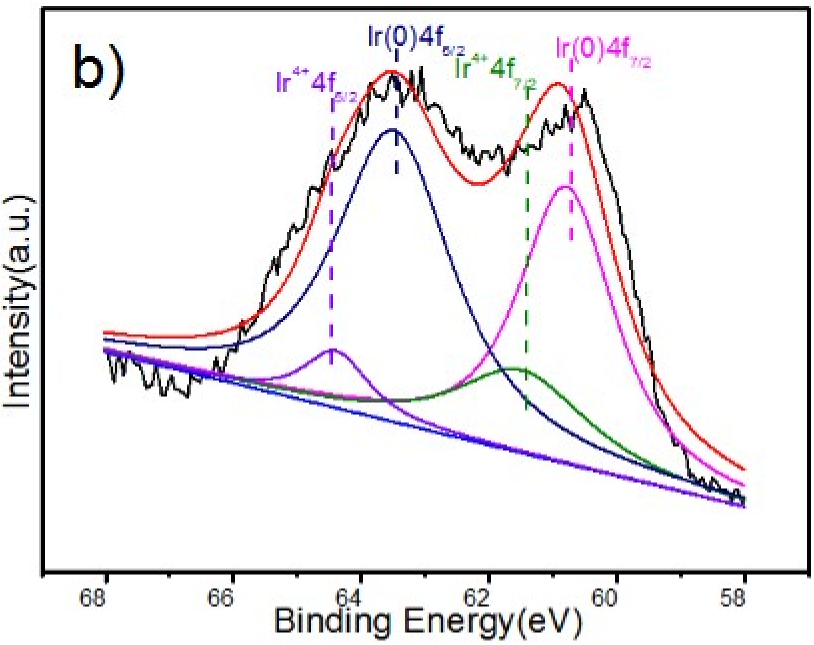


Figure S8. Representative high-resolution XPS spectra: a) Cu2p of Ir-Cu nanoframes; b) Ir 4f of Ir-Cu nanoframes.

Table s1 The relative content of different valence states of Cu2p

Element	Transition	Peak Energy (eV)	Peak Area (eV-cts/s)
Cu(0)	$2p_{3/2}$	932.62	19116(57.7%)
Cu^{2+}	$2p_{3/2}$	933.76	13987(42.3%)

Table s2. The relative content of different valence states of Ir4f

Element	Transition		Peak Energy (eV)	Peak Area (eV-cts/s)
$Ir4f_{7/2}$	$4f_{7/2}$	0^+	60.8	5490(71.2%)
		4^+	61.5	2222(28.8%)
$Ir4f_{5/2}$	$4f_{5/2}$	0^+	63.5	7005(90.2%)
		4^+	64.4	760(9.8%)

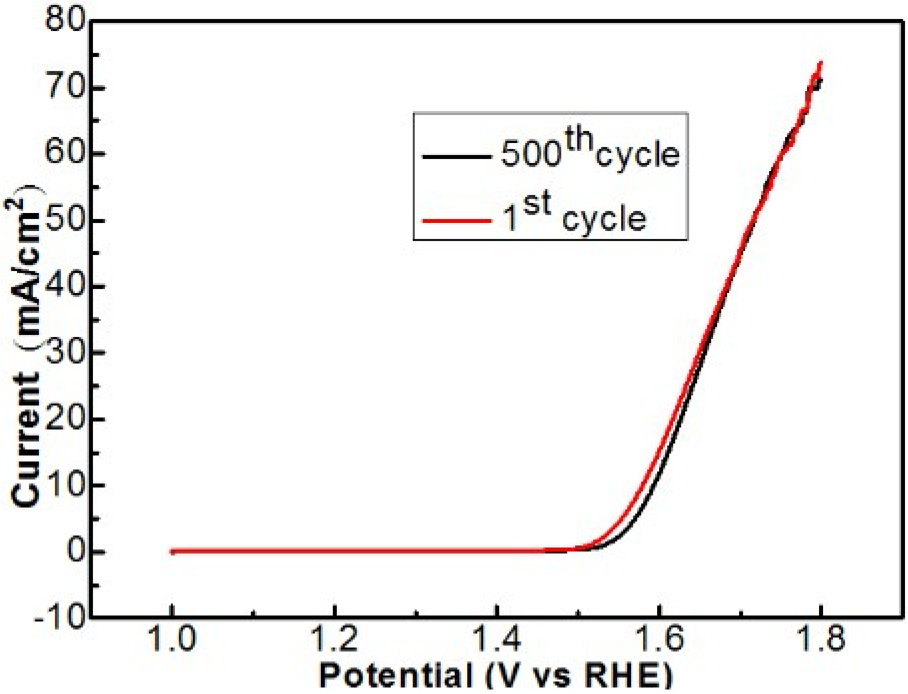


Figure S9. Electrochemical durability of Ir-Cu nanoframes, the red line was first cycle of LSV curves after CV curves and the 500th was black line.