## Supplementary information

# **One-Pot Synthesis of Hexagonal and Triangular Ni-Cu Alloy Nanoplates and Their Magnetic and Catalytic Properties**

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### 1. Synthesis of spherical Ni-Cu alloy nanoparticles

A mixture of 7 mL of oleylamine, 1 mmol of Ni(acac)<sub>2</sub>, and 0.2 mmol of copper(II) acetate was decanted into a three-necked flask and kept under a flow of high-purity argon gas at 110 °C for 20 min with strong magnetic stirring before 1 mmol of TOP was injected. After stirring for 5 min, the resulting solution was then slowly heated up to 220 °C directly, aged at this temperature for another 60 min and then cooled to room temperature. The products were separated from the solution by centrifugation, washed several times using the mixture of hexane and ethanol or acetone, and dried in a vacuum.

#### 2. Synthesis of spherical Ni nanoparticles in the present of KCl

In order to confirm the indispensable role of the joint reaction of Cl<sup>-</sup> and Cu<sup>2+</sup> for the generation of anisotropy Ni-Cu nanoplates, KCl was chosen to replace CuCl<sub>2</sub>•2H<sub>2</sub>O. In a typical synthesis, a mixture of 7 mL of oleylamine, 1 mmol of Ni(acac)<sub>2</sub>, and 0.2 mmol of KCl was decanted into a three-necked flask and kept under a flow of high-purity argon gas at 110 °C for 20 min with strong magnetic stirring before 1 mmol of TOP was injected. After stirring for 5 min, the resulting solution was then slowly heated up to 220 °C directly, aged at this temperature for another 60 min, and then cooled to room temperature. The remaining steps are the same as those described in section 1.

#### 3. Synthesis of monodisperse Ni nanoparticles

1 mmol of Ni(acac)<sub>2</sub> was added to 7 ml of oleylamine and the mixture was stirred magnetically under a flow of high-purity argon gas at 110 °C for 20 min. Then, 1 mmol of TOP was injected. The mixture was then rapidly heated to 220 °C, aged at this temperature for additional 40 min, and then cooled to room temperature. The remaining steps are the same as those described in section 1.

#### 4. Synthesis of Cu nanoparticles

0.8 mmol of copper(II) nitrate trihydrate was added to 7 ml of oleylamine and the mixture was stirred magnetically under a flow of high-purity argon gas at 110 °C for 20 min. The mixture was then rapidly heated to 280 °C, aged at this temperature for 90 min, and then cooled to room temperature. The remaining steps are the same as those described in section 1.



**Fig. S1** TEM image of the as-synthesized hexagonal nanoplates. The insert shows the statistic result of particle size distribution determined from Fig. S1 and Fig. 1a.



**Fig. S2** TEM image of the as-synthesized triangular nanoplates. The insert shows the statistic result of particle size distribution from Fig. S2 and Fig. 1b.



**Fig. S3** XRD patterns of the as-synthesized hexagonal (a), triangular (b) Ni-Cu alloy nanoplates and Ni nanoparticles (c). Standard pattern of Cu (black solid peak-sticks, JCPDS No. 7440-50-8) is included for comparison (d). The used Ni nanoparticles have a spherical morphology and a mean size of 21 nm (see TEM image in Fig. S15) synthesized using the method mentioned in page 2 (No. 3).



**Fig. S4** EDS spectra of triangular (top) and hexagonal (bottom) nanoplates. The Au signals are due to the gold TEM grid. The results of Ni: Cu ratios and synthetic conditions are available in Table S1 (see No. 4 (hexagonal) and No.7 (triangular)).



**Fig. S5** HAADF image (a), STEM-EDS line-scanning profile (b) taken along the red line in image (a), and STEM-EDS elemental maps (c and d) of a single hexagonal Ni-Cu alloy nanoplate.



**Fig. S6** XRD pattern of spherical Ni-Cu alloy nanoparticles synthesized using copper(II) acetate as precursor compound.



Fig. S7 TEM image of spherical Ni-Cu alloy nanoparticles synthesized using copper(II) acetate as precursor compound.



Fig. S8 EDS spectrum taken from the spherical Ni-Cu alloy nanoparticles synthesized using copper(II) acetate as precursor compound.



Fig. S9 TEM image of the mixed nanocrystals of NiO and Cu.



Fig. S10 XRD pattern of the mixed nanocrystals of NiO and Cu.



Fig. S11 TEM image along with SAED pattern (inset) of Ni nanoparticles synthesized in the present of KCl.



**Fig. S12**. EDS spectra taken from the Ni-Cu nanocrystals generated at different reaction stages in the typical synthesis of hexagonal nanoplates.



Fig. S13 TEM image of Ni-Cu alloy NCs synthesized without TOP.



Fig. S14 ZFC and FC curves of the Ni-Cu alloy nanoplates synthesized using equivalent molar quantities of Ni(acac)<sub>2</sub> and CuCl<sub>2</sub>·2H<sub>2</sub>O (see Table 1 No. 14). The measuring field and cooling field are 100 Oe.



Fig. S15 TEM image of the as-synthesis monodisperse Ni nanoparticles. The insert shows the statistic result of particle size distribution.



Fig. S16 XRD pattern of the as-synthesized monodisperse Ni nanoparticles.



**Fig. S17** TEM image of the as-synthesized Cu nanoparticles. The insert shows the statistic result of particle size distribution.



Fig. S18 XRD pattern of the as-synthesized Cu nanoparticles.

Synthetic Conditions							Atomic Ratio (%)	
No.	Oleylamine (mL)	Dibenzyl Ether (mL)	Ni(acac) <sub>2</sub> (mmol)	CuCl <sub>2</sub> ·2H <sub>2</sub> O (mmol)	Reaction Temperature (°C)	Reaction Time (min)	Cu	Ni
1	7	0	1	0.2	175	60	74.0	26.0
2	7	0	1	0.2	190	60	51.6	48.4
3	7	0	1	0.2	210	60	22.8	77.2
4	7	0	1	0.2	220	60	19.2	80.8
5	7	0	1	0.2	230	60	19.5	80.5
6	7	0	1	0.2	240	60	16.2	83.8
7	3	7	1	0.2	200	60	23.3	76.7
8	2	8	1	0.2	200	60	22.4	77.6
9	1	9	1	0.2	200	60	29.5	70.5
10	7	0	1	0.2	200	0	93.8	6.2
11	7	0	1	0.2	210	0	60.6	39.4
12	7	0	1	0.2	210	15	51.5	48.5
13	7	0	1	0.2	210	30	22.1	77.9
14	7	0	0.2	0.2	200	60	48.4	51.6
15	7	0	0.4	0.8	220	60	35.9	64.1

Table S1 The results of EDS analyses on the samples generated at different synthetic conditions.