Electronic Supplementary Information

Synthesis of CoFe alloy nanoparticles embedded in a MgO crystal matrix using a single-source inorganic precursor

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Supporting Information: Experimental details.

Materials.

The following analytical grade chemicals were purchased from Beijing Yili Fine Chemical Reagent Co. and used without further purification: NaOH, Na_2CO_3, $Mg(NO_3)_2·6H_2O$, $Co(NO_3)_2·6H_2O$ and $Fe(NO_3)_3·9H_2O$. Deionized water with a conductance below 10^{-6} S cm^{-1} was used in synthesis and purification steps.

Synthesis of MgCoFe LDH precursors.

Ternary $Mg_xCo_{1.5}Fe$ (x = 3.3, 2.4, 1.5, and 0.6) LDH precursors with interlayer CO_3^{2-} anions were prepared by a procedure developed in our group involving separate nucleation and aging steps (the SNAS method): A mixture of $Mg(NO_3)_2·6H_2O$, $Co(NO_3)_26·H_2O$ and $Fe(NO_3)_3·9H_2O$ with Mg/Co/Fe molar ratios of 3.5/1.5/1, 2.5/1.5/1, 1.5/1.5/1, or 0.5/1.5/1 was dissolved in 100 mL of deionized water to form a clear solution ($[Mg^{2+}] + [Co^{2+}] + [Fe^{3+}]) = 1.0$ M). The aqueous solution of the metal nitrate salts and 100 mL of NaOH and Na_2CO_3 solution ($[OH^-]$ = 1.6 M, $[CO_3^{2-}]$)= $2[Fe^{3+}]$) were simultaneously added to a colloid mill with rotor speed of 3000 rpm and mixed for 1 min. The resulting suspension was removed from the colloid mill and transferred into a Teflon-lined autoclave. The autoclave was then tightly sealed and maintained at 100 ^{o}C for a period of 48 h. The resulting suspension was filtered and washed thoroughly with deionized water. The final precipitate was directly dried in an oven at 60 °C for 24 h to collect the solid $Mg_xCo_{1.5}Fe$ LDH products.

Characterization.

Elemental analysis for metal ions was performed using a Shimadzu ICPS-7500 inductively coupled plasma emission spectrometer (ICP-ES) on solutions prepared by dissolving the samples in dilute HNO_3.

X-ray diffraction (XRD) patterns of the samples were recorded using a Shimadzu XRD-6000 diffractometer under the following conditions: 40 kV, 30 mA, Cu Kα_1 radiation (λ = 0.15406 nm). The samples were step-scanned in steps of 10°/min in the 2θ range from 3 to 90°. The observed diffraction peaks were corrected using elemental Si as an internal standard. The samples were ground into powders before measurement.

Transmission electron microscopy (TEM) was performed on a Hitachi H-800 microscope with an accelerating voltage of 150 kV. The sample was ultrasonically dispersed in an appropriate amount of ethanol and a drop of the resulting suspension was deposited on a carbon-coated Cu grid followed by evaporation of the solvent in air.

Magnetic properties of samples was measured at room temperature on a locally made JDM-13 vibrating sample magnetometer.

Supporting Information: Figures.

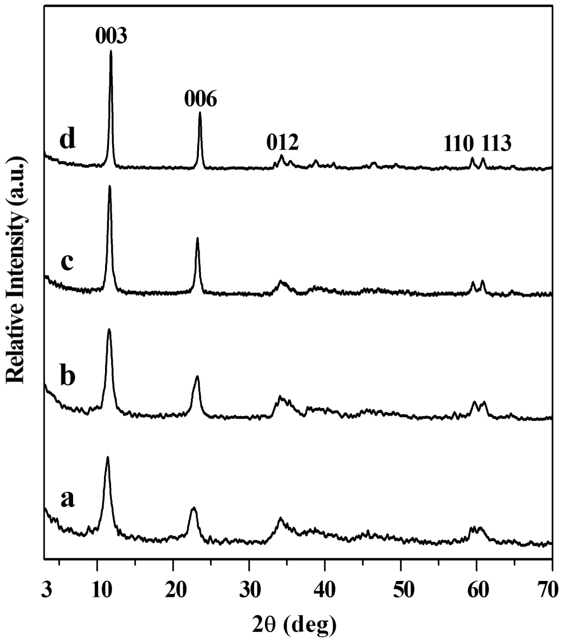

Fig. S1 Powder XRD patterns of (a) $Mg_{3.3}Co_{1.5}Fe$, (b) $Mg_{2.4}Co_{1.5}Fe$, (c) $Mg_{1.5}Co_{1.5}Fe$, and (d) $Mg_{0.6}Co_{1.5}Fe$ LDH precursors.

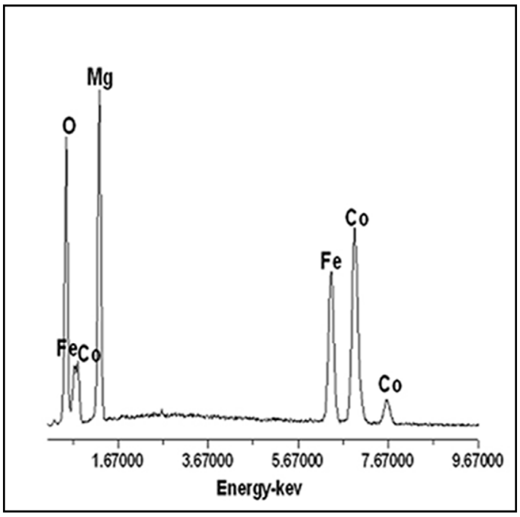

Fig. S2 EDX spectrum of the CoFe/MgO nanocomposite by reducing the $Mg_{3.3}Co_{1.5}Fe$ LDH precursor.