Supporting Information

Controlled Synthesis and Assembly into Anisotropy Arrays of Magnetic Cobalt-Substituted Magnetite Nanocubes

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Materials and Methods

Chemicals and Materials. Oleylamine (OAm, >70%), Benzyl ether (BE, technical grade 99%), oleic acid (OAc, technical grade, 90%), cobalt(II) acetylacetonate ($Co(acac)_2$) hydrate and iron(III) acetylacetonate ($Fe(acac)_3$) (99%), hexane (98.5%), ethanol (100%) were all purchased from Sigma Aldrich.

50 nm Hard Magnetic $Co_{0.4}Fe_{2.6}O_4$ Nanoparticles (NCs) Synthesis

$Fe(acac)_3$ (1.33 mmol), $Co(acac)_2$ (0.665 mmol), OAc (2.5 mL), and BE (20 mL) were mixed and magnetically stirred under a flow of nitrogen at room temperature. Then the solution was heated to 290 ^oC at a heating rate of 20 ^oC/min kept at the synthesis temperature for 30 min. The heating source was then removed, and the solution was cooled to room temperature, after which the solution was exposed to air. A black product was precipitated by adding 40ml of ethanol, and separated by centrifugation. Finally, the NPs were dispersed in hexane.

Characterization. X-ray diffraction (XRD) characterization was carried out on a Bruker AXS D8-Advanced diffractometer with Cu *K*α radiation (λ =1.5418 Å). The Inductively coupled plasma-atomic emission spectroscopy (ICP-AES) measurements were carried on a JY2000 Ultrace ICP Atomic Emission Spectrometer equipped with a JY AS 421 autosampler and 2400g/mm holographic grating. Samples for transmission electron microscopy (TEM) analysis were prepared by depositing a single drop of diluted clusters dispersion in hexane on amorphous carbon coated copper grids. TEM images were obtained with a Philips CM 20 operating at 200 kV. High-resolution TEM (HRTEM) and the atomically resolved scanning transmission electron microscopy- electron energy-loss spectroscopy (STEM-EELS) images were obtained on a Fei Tecnai Osiris with an accerating votalage of 200 kV. Magnetic studies were carried out using a Lakeshore 7404 high sensitivity vibrating sample magnetometer (VSM) with fields up to 12.5 kOe at room temperature.

Figures

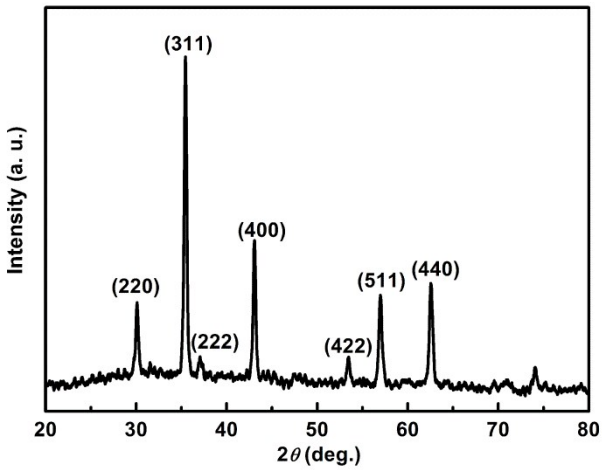

Figure S1. XRD pattern of the 50 nm $Co_{0.4}Fe_{2.6}O_4$ NCs synthesized at 290 ^oC for 30 min with a heating rate of 20 ^oC/min

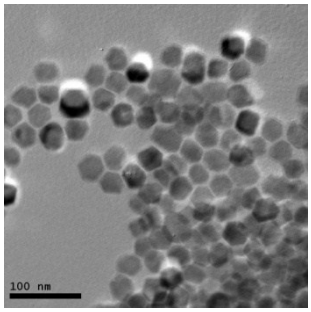

Figure S2. TEM image of the 35 nm $Co_{0.4}Fe_{2.6}O_4$ NPs with polyhedral shape.

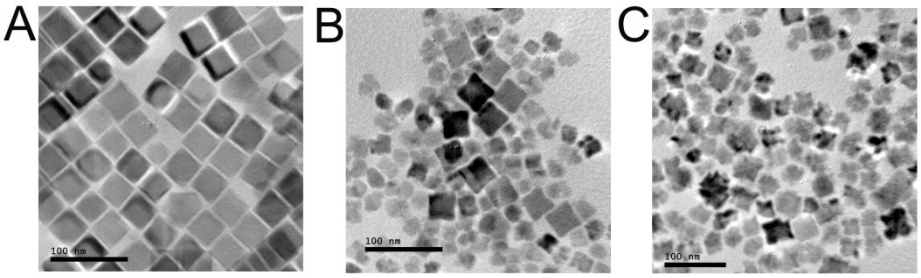

Figure S3. TEM images of the as-synthesized $Co_xFe_{3-x}O_4$ NPs with OAm (**A**) 0.5 mL, (**B**) 1.0 mL, and (**C**) 2.0 mL.

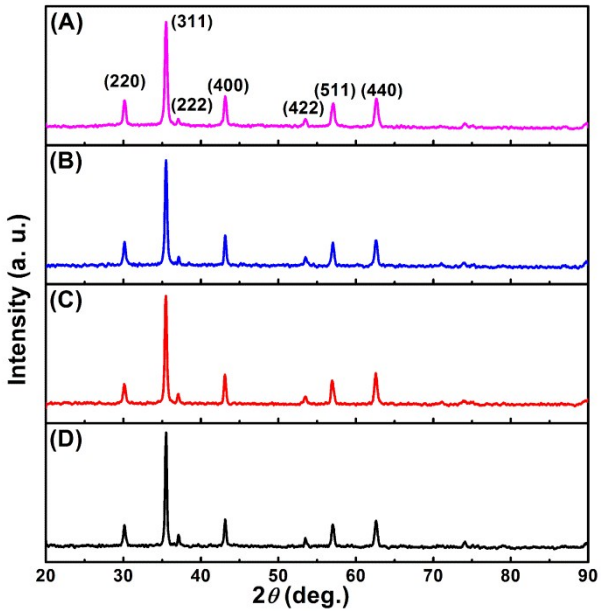

Figure S4. XRD patterns of the as-synthesized $Co_xFe_{3-x}O_4$ NCs with OA (**A**) 3.1 mL, (**B**) 1.27 mL, (**C**) 0.9 mL, and (**D**) 10 mL BE and 1.27 mL OA.