SUPPORTING INFORMATION

One-Pot Synthesis of Bi-Disperse FePt Nanoparticles and Size-Selective Self-Assembly into AB₂, AB₅, and AB₁₃ Superlattices

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Synthesis Details

Synthesis. A mixture of 0.5 mmol of Pt (acac)₂, 0.5 mmol of Fe(acac)₃, and 0.025 mmol of Cu(acac)₂ was added to 25 ml of octyl ether in a 250 mL three-neck round bottom flask. The solution was sonicated for approx. 30 minutes. Next, 5 mmol of 1,2-hexadecanediol was added and the solution was again sonicated for 10 minutes. After purging with Ar for approx. 20 minutes, 160 μ L of oleic acid and 170 μ L of oleylamine were added and the solution was purged for 10 more minutes. The solution was then heated in a heating mantle to 250 °C after which the temperature was lowered down to 210°C. The flask was maintained at this temperature for the next 40 minutes before cooling down to room temperature under the blanket of Ar. After this step, the reaction mixture was handled in air.

Purification. About 40 mL of ethyl alcohol was added to the flask and the mixture was centrifuged at 4000 rpm. The supernatant was discarded and the precipitate was redispersed in ~10 mL of hexane. The hexane mixture was again centrifuged for about 6 minutes, first at 1000 rpm and then at 4000 rpm, to remove any undissolved solids. About 40 mL of ethanol was added to the supernatant and shaken for about one hour. The solution was again centrifuged at 4000 rpm and the precipitate was dispersed, washed with ethanol, and redispersed in hexane. Approx. 16 μ L of oleic acid and 17 μ L of oleylamine were added to aid in redispersing the particles.

Characterization. Most of the TEM analyses were performed on a JEOL 2010 TEM (w/EDS and SAED) using copper or nickel grids coated with formvar carbon. TEM grids were prepared by drop casting a hexane solution of the mixture, to which 16 μ L of oleic acid and 17 μ L oleylamine were added. The grids were dried in air for 30 min. High resolution electron microscopy was performed on a Philips CM30-UT-FEG. EDS data were acquired on a Tecnai 200 by using STEM mode with spot size less than 0.5 nm, which guaranteed that the measured spectra were only coming from each single particle. Sample drift was checked after the collection of a spectrum by correlation of the images before and after acquiring the EDS. (Spectra with large image drift were ignored.)

Size distributions (obtained from analyzing HRTEM micrographs)



Random nanoparticles

(no superlattices; included all Fe_xPt_{1-x} particles after larger Pt particles were removed)

	Large particles	Small particles
Min	3.00 nm	1.70 nm
Max	5.80 nm	3.20 nm
Mean	3.88 nm	2.04 nm
Stdev	0.50 nm	0.29 nm



AB₂ Superlattice

	Large particles	Small particles
Min	3.40 nm	1.80 nm
Max	5.10 nm	2.50 nm
Mean	4.13 nm	2.12 nm
Stdev	0.39 nm	0.18 nm



AB₅ Superlattice

	Large particles	Small particles
Min	3.80 nm	2.00 nm
Max	5.20 nm	2.70 nm
Mean	4.52 nm	2.28 nm
Stdev	0.25 nm	0.15 nm



AB₁₃ Superlattice (icosahedral)

	Large particles	Small particles
Min	3.50 nm	1.90 nm
Max	5.50 nm	2.80 nm
Mean	4.51 nm	2.30 nm
Stdev	0.44 nm	0.22 nm

Representative data for single-particle EDS analysis

(data acquired for samples immobilized on a Cu grid)



STEM micrograph and EDX spectra of AB_{13} nanoparticle superlattice. Panel (a) shows the image recorded under STEM mode. The EDX spectra recorded from positions 1, 2, 3, and 4 are shown in (b) – (e) respectively. Under STEM mode, the spot size can be controlled to less than 0.5 nm, which guarantees that the measured spectra only come from each single particle.

Large-area EDS analysis taken with the AB₁₃ sample immobilized on a Ni grid.

(Some Cu is detected, but it is difficult to discern if it is an intrinsic part of the individual particles, or whether it is a result of microscopic contamination.)

