Electronic Supplementary Material (ESI) for Journal of Materials Chemistry C. This journal is © The Royal Society of Chemistry 2015

The Transformation of α -Fe Nanoparticles into Multi-Domain FeNi-M₃O₄ (M = Fe, Ni) Heterostructures by Galvanic Exchange

Supplemental Information

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Supporting Figures

Figure S1. XRD results for NPs after oxidation by opening reaction vessel to air at 100 °C for 5h, the α -Fe core NPs before (a) and after Ni addition at Ni:Fe ratios of 1 (b), 2 (c), 5 (d), and 7 (e). Inset: Overlay of a and e showing shift in a-Fe <110> position due to FeNi alloying. Reference diffraction are shown for Ni (FCC), Fe₅₀Ni₅₀ (FCC), α -Fe (BCC), and Fe₃O₄.



Figure S2. Representative TEM of additional α -Fe core NPs $d = 12.3 \pm 2.3$ nm (a) and $d = 17.8 \pm 2.3$ nm (b) used in the synthesis of Ni:Fe = 5, and 1 and 2 respectively.



Figure S3. Representative TEMs for the NiFe-M₃O₄ (M = Fe, Ni) heterostructures formed at Ni:Fe ratios of 1 (a), 2 (b), 5 (c), and 7 (d), collected after opening the reaction vessel to air and heating at 100 $^{\circ}$ C for 5h.



Figure S4. FTIR of the α -Fe core NP (a) and for NiFe-M₃O₄ NPs prepared at Fe:Ni of 1 (a), 2 (b), 5 (c), and 7 (d) before (i) and after (ii) oxidation.



Figure S5. Additional HRTEM of FeNi- M_3O_4 heterostructures formed at Ni:Fe reaction ratio of 1 (a) 2 (b) and 7 (c), along with a STEM-EDS line scan for a two domain heterostructure from Ni:Fe of 7 (d). The inset shows the particular NP studied.



Figure S6. XRD of NP products produced during a control synthesis using thermal decomposition of $Ni(CO)_2(PPh_3)_2$ at α -Fe cores at [Ni]:[Fe] = 3. The α -Fe NPs (a) are crystalline as in Figure 2, and after deposition of Ni via $Ni(CO)_2(PPh_3)_2$ show improved resistance to oxidation, as indicated by the lower intensity of M_3O_4 diffraction planes (b). XPS analysis revealed Ni:Fe of 73.3% nickel and 26.7% iron in final NP, further confirming improved nickel deposition as a result of the system not relying on galvanic exchange for nickel deposition, unlike when $Ni(acac)_2$ is added.