Focused electron beam induced deposition and Lorentz microscopy investigation of bi-magnetic core-shell nanostructures

Almeida, T.[1], McGrouther, D.[2] and McVitie, S.[3]

[1] University of Glasgow, United Kingdom, [2] School of Physics and Astronomy, University of Glasgow, United Kingdom, [3] SUPA School of Physics and Astronomy, University of Glasgow, Glasgow G12 8QQ, United Kingdom

The demand for improved functionality of modern magnetoelectronic devices has led to the rapid development of innovative magnetic materials comprising hard and soft magnets, commonly referred as exchange-spring magnets. In a bi-magnetic core-shell (CS) nanostructure, exchange coupling over the CS interface facilitates cooperative magnetic switching and provides for a novel nano-material system with tunable magnetic properties[1,2]. However, our current understanding of the exchange interaction between the core and shell is limited to bulk magnetic measurements or micromagnetic modelling, and thus not sufficient to explain their magnetic properties.

Next generation scanning electron microscopes (SEMs) incorporate systems for the injection of element-containing gases, acting as precursors for the localised deposition of magnetic nanopatterns (NPs) as small as ~ 10 nm in diameter, called focused electron beam induced deposition (FEBID)[3]. Deposition of reactive ferromagnetic metals like iron and cobalt acts as favourable sites for the formation of CS NPs through surface oxidation. Further, Lorentz microscopy encompasses several techniques within the transmission electron microscope (TEM) that allow imaging of nano-scale magnetism. In this context, we are now in a timely position to use state-of-the-art FEBID and TEM facilities to synthesise CS systems and image directly the effect of localised exchange coupling between core and shell in bi-magnetic nanostructures on their magnetic properties, for the first time.

In this study, we use a series of electron microscopy techniques to both synthesise CS NPs and characterise their chemical, magnetic and structural properties. Fig. 1a presents a SEM image of an array of Fe NPs produced by FEBID ranging from ~ 20 nm to ~ 50 nm in diameter. The bright field (BF) TEM image of Fig. 1b provides a higher magnification, localised view of an individual Fe NP (~20 nm in diameter) at 20°C, whilst the disperse diffraction rings in the corresponding selected area electron diffraction (SAED) pattern (Fig. 1c) reveals the Fe NP to be poorly crystalline. The scanning TEM image (Fig. 1d) and electron energy-loss spectroscopy (EELS) chemical maps (Fig. 1e,f) show the Fe distribution to be uniform across the NP (Fig. 1e) but exhibits an oxygen rich oxide shell (Fig. 1f), confirming the formation of an Fe / Fe-oxide CS nanostructure. Fig. 2a displays a slightly larger Fe NP (~ 50 nm in diameter) which is revealed by SAED (inset) to exhibit increased crystallinity, with more defined disperse diffraction rings. *In situ* heating within the TEM to 700°C promoted the coarsening of the Fe NP (Fig. 2b) into a well-crystallized structure, which is supported by the presence of the strong diffraction spots in the associated SAED pattern (inset). Lorentz microscopy techniques like differential phase contrast imaging and off-axis electron holography is performed on the CS NPs to examine their magnetic properties as a function of temperature, with emphasis placed on the investigation of exchange coupling between the core and shell.

References

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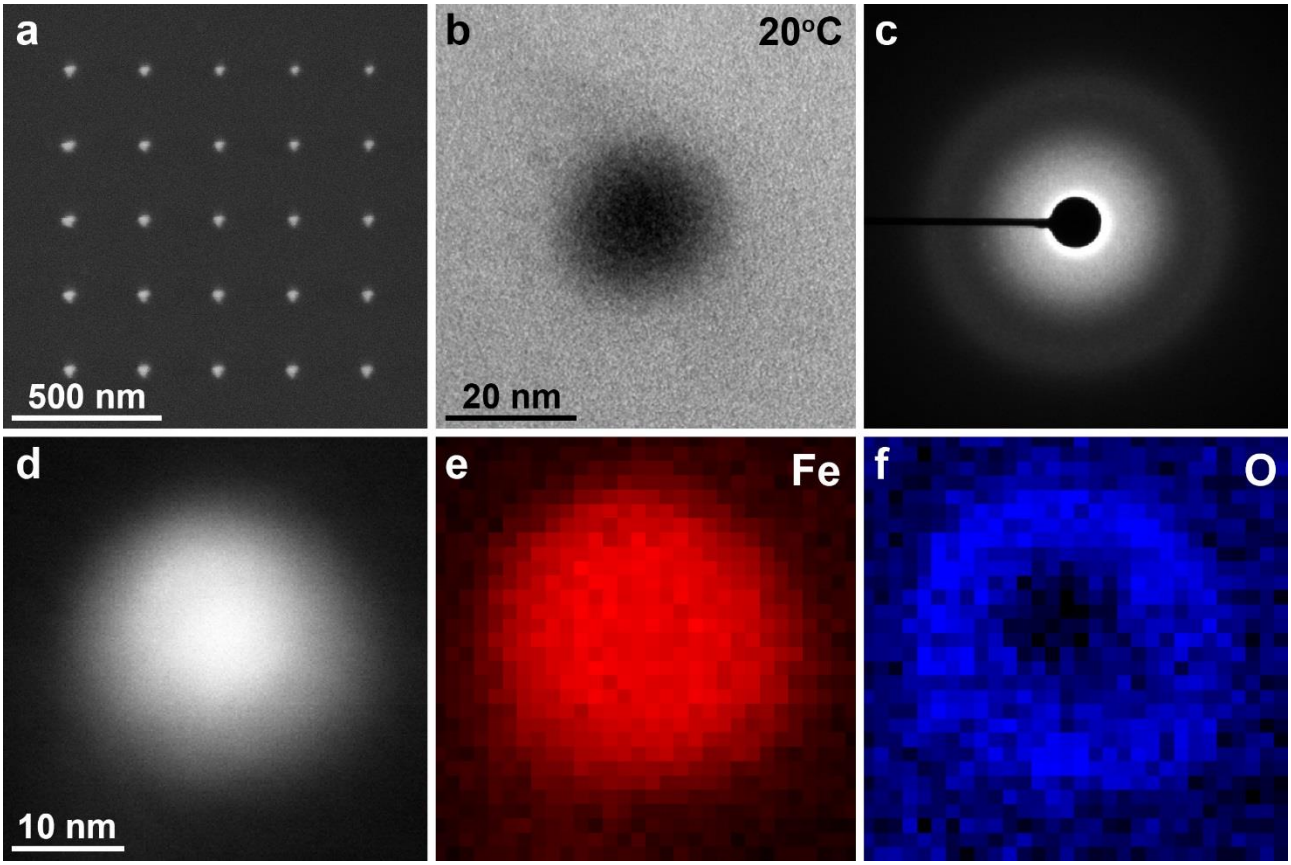

Fig. 1 (a) SEM image of an array of Fe NPs produced by FEBID, ranging from ~ 20 nm to ~ 50 nm in diameter. (b) BF TEM image of an individual Fe NPs (~ 20 nm in diameter) at 20°C; and (c) corresponding SAED pattern from the Fe NP in (b) displaying disperse diffraction rings. (d) Scanning TEM image of an individual Fe NP; and (e,f) EELS chemical maps showing its elemental distribution of (e) Fe; and (f) oxygen content.

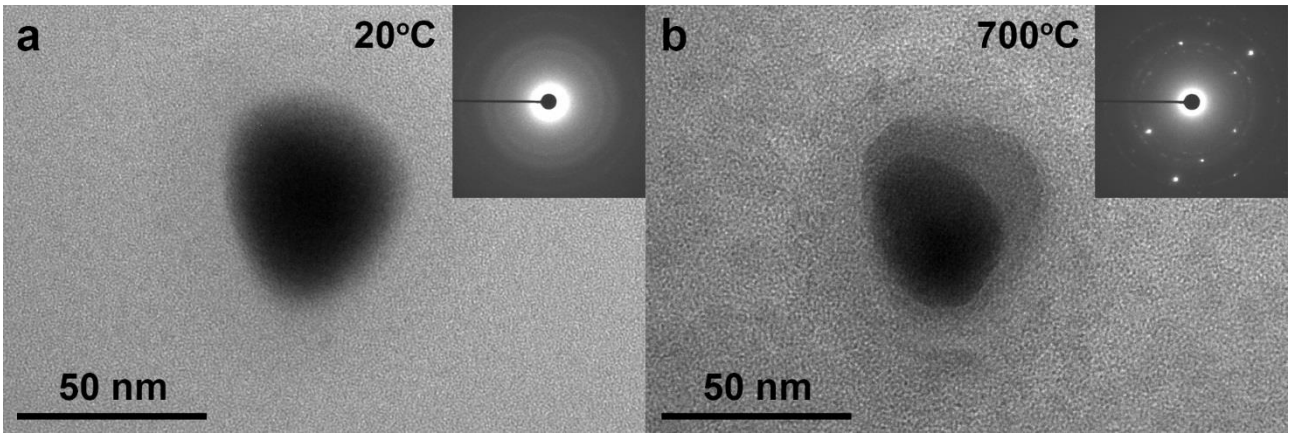

Fig. 2 (a,b) BF TEM images on individual Fe NP at (a) 20°C; and after *in situ* heating to (b) 700°C. The associated SAED patterns (inset) show an increase in crystallinity from (a) disperse diffraction rings; to (b) additional well-defined diffraction spots.