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ID-12-P-1501 Gas environment study of Fe nanoparticles using in-situ aberration corrected E-(S)TEM

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Fe and Fe-oxide nanoparticles have a series of promising potential applications in physical and medical sciences. These include magnetic storage devices, catalysis, sensing, contrast enhancement in magnetic resonance imaging and magnetic hyperthermia [1-3]. Understanding of the Fe-Oxide NPs reduction to metal and the oxidation processes down to atomic scale is paramount for the control of the quality and the optimization of their applications.

A recently modified double aberration corrected JEOL 2200FS (S)TEM [4] has demonstrated the possibility of the analysis of metallic nanoparticles in gas environment at temperature allowing single atom visualisation by HAADF STEM in controlled gas reaction environment [5].

In this study, thin films of iron were deposited by sputtering on C films supported by standard TEM Cu grids as in Figure 1a). Nanoparticles were produced by annealing in-vacuum the films within the microscope column (pressure ~1.0 * 10^{-5} Pa) at temperatures from room temperature to up to 600 °C using an in-house designed Gatan heating holder (Fig 1b). Nanoparticle formation and size distribution was monitored in-situ as a function of time and temperature by HAADF STEM imaging. After annealing nanoparticles were shown to consist of single crystal metallic Fe, composition confirmed by EDX analysis. The Fe nanoparticle samples interaction with Hydrogen (Fig 2a) and Oxygen (Fig 2b) gases were studied in-situ at 300 °C with a differential pressure at the specimen in the range of 2.5-3.0 Pa. The interaction of the nanoparticles with the gases, as well as the substrate, will be discussed in terms of the changes in nanoparticle geometry, composition, size distribution, crystallinity and microstructural defects.

Fe to Fe_xO_y phase change was identified by HAADF/BF STEM imaging, and EDX line scans (Fig 3). These show uniform Fe/O composition within each particle with comparable particle sizes, but complex morphologies for the metallic and the oxide phases. The mechanism is being developed with single atom sensitivity.

References:

[1] B D Terris and T Thomson, J. Phys. D 38, R199–R222 (2005)
[2] H. Galvis et al. Science 335, 835–838 (2012)
[3] Q A Pankhurst, N T K Thanh, S K Jones, and J Dobson, J. Phys. D 42, 224001 (2009)
[4] P L Gai and E D Boyes, Microscopy Research and Technique 72, 153 (2009)
[5] E D Boyes, M R Ward, L Lari, and P L Gai, Annalen der Physik 525, 423 (2013)

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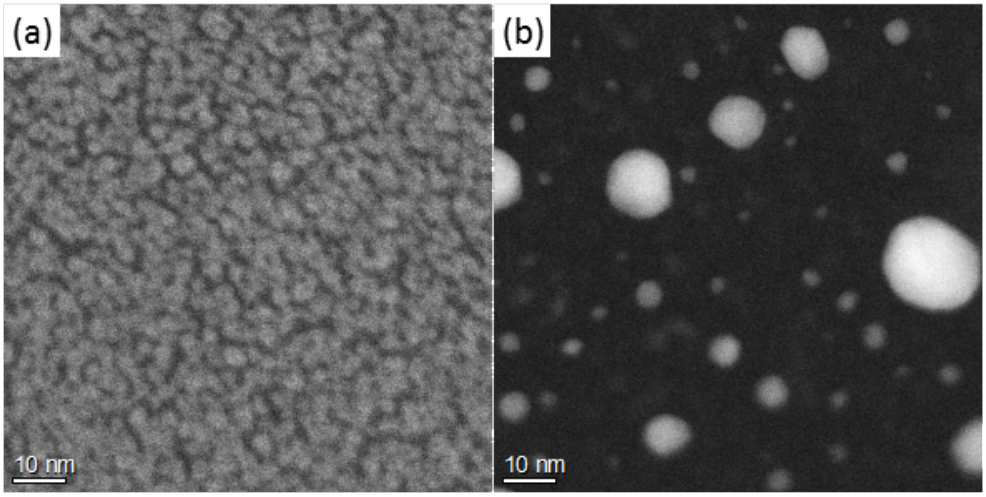

Fig. 1: (a) Initial sputtered Fe film, left in air for two days before annealing in the microscope up to 600 °C to form nanoparticles (b).

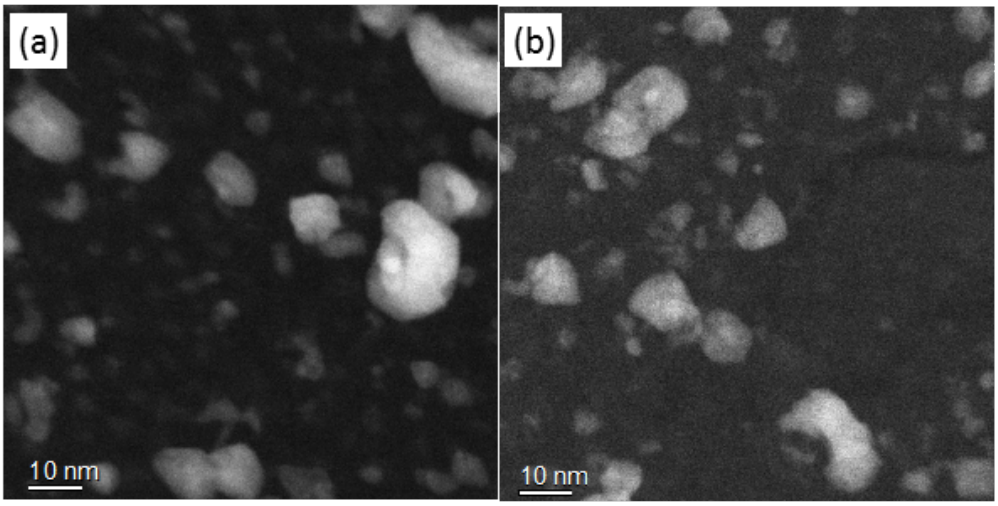

Fig. 2: (a) Overview of Sample A after exposure to H_2 gas environment with a pressure at the specimen of 3 Pa and with a sample temperature of 300 °C. (b) Overview of Sample B after exposure to O_2 gas environment with a pressure at the specimen of 2.5Pa and with a sample temperature of 280 °C.

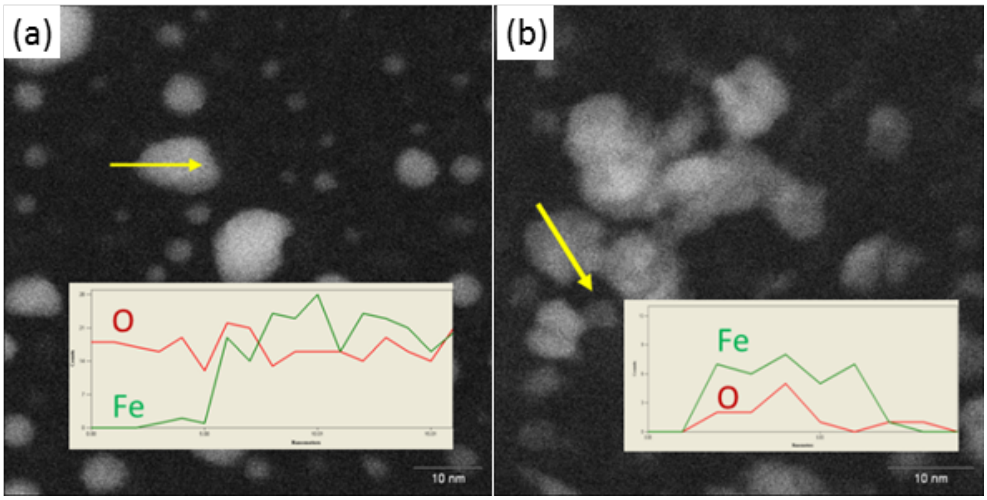

Fig. 3: (a) EDX line scan of Fe nanoparticles at of 300 °C in vacuum showing O signal at background in the particle as well as on the support. (b) EDX line scan of a Fe_xO_y nanoparticle done in O_2 environment at 2.5 Pa and 300 °C (scale bar 10nm).