

# Energy-Filtered Imaging of Fe and Ni Nanoparticles in a Field Emission Gun Transmission Electron Microscope

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**Summary:** Two different kinds of nanoparticles have been analyzed using three-window elemental mapping in a transmission electron microscope equipped with a field emission gun and a post-column imaging filter. The first sample contained nanoparticles of Fe/FeO<sub>x</sub> synthesized by the “inert gas evaporation method”, while the second sample contained nanoparticles of Ni embedded in amorphous carbon and prepared using a sonochemical method.

## 1. Introduction

Transmission electron microscopes equipped with in-column or post-column energy filters are now routinely used to obtain energy-selected images or diffraction patterns of materials. Here, we use energy-filtered transmission electron microscopy (EFTEM) [1, 2] to form three-window background-subtracted elemental maps of crystalline nanoparticles that are between 5 and 20 nm in size [3]. The first sample contains an Fe nanocrystalline powder coated with iron oxide, while the second sample contains Ni crystals that are each encapsulated in amorphous carbon. Nanoparticles such as these are of great interest for applications in the magnetic recording industry [4].

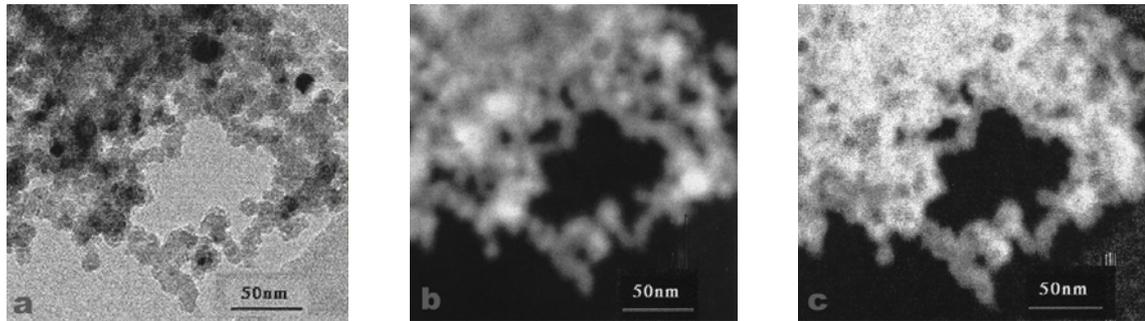
## 2. Experimental

Fe nanoparticles were synthesized by the “inert gas evaporation method” described by Fernández et al. [5], while Ni nanoparticles were prepared by sonication, as described by Koltypin et al. [6]. Samples for electron microscopy were prepared by dispersing a suspension of the resulting powders in acetone, followed by transfer onto a holey carbon film supported on a copper grid. The samples were examined at 300 kV using a JEOL JEM-3000F field emission gun TEM, which is equipped with a thermally assisted Schottky electron source and has a wide range of analytical facilities that include a Gatan 794 (1k×1k) CCD camera at the level of the plate camera and a GIF 2000 imaging filter with a (2k×2k) 794IF/20 CCD detector. The field emission gun provides a high brightness source that facilitates the formation of three-window background-subtracted elemental maps from small areas of each sample. This is a well-established technique that involves acquiring two images using electrons that lie just below a characteristic edge of the energy-loss spectrum, and a similar image just after the edge. The first two images are then used to extrapolate a background that can be subtracted from the third image to provide a two-dimensional chemical map of the sample for each element of interest.

## 3. Results and discussion

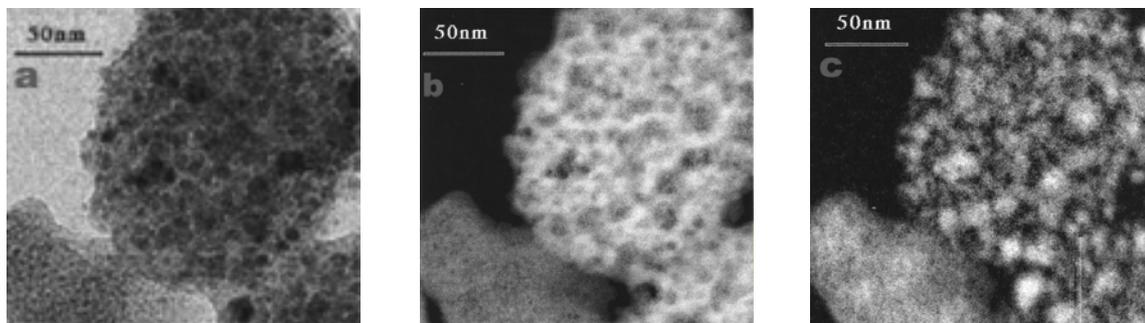
A representative zero-loss bright field energy-filtered image of the iron sample is shown in Fig. 1a. The sample is seen to consist of almost spherical grains, which link together form larger agglomerates but are each thought to contain a metal iron core surrounded by an outer iron oxide shell. Figures 1b and 1c show the corresponding iron and oxygen three-window elemental maps of the same region of the sample. These maps were formed from the Fe L<sub>2,3</sub> and O K edges, respectively, using a 20 eV wide energy-selecting slit. The maps confirm that the sample is strongly oxidized, while the differences between the maps demonstrate that the ratio of iron to oxygen is highly variable across the sample. In particular, it is apparent that Fe-rich regions vary in size

between small, isolated (~5nm) crystals and larger (~30nm) areas. This variability is of importance for magnetic applications of the material.



**Figure 1:** EFTEM images obtained from the iron sample. (a) Zero-loss bright field image; (b) Fe  $L_{2,3}$  and (c) O K three-window background-subtracted elemental maps.

Corresponding images from the Ni sample are shown in Fig. 2. Again, a zero-loss bright field energy-filtered image, in which the approximately spherical Ni particles appear dark, is shown in Fig. 2a. Figures 2b, 2c and 2d show three-window elemental maps obtained from the same region of the sample using the C K, Ni  $L_{2,3}$  and O K edges, respectively. The maps confirm that the Ni particles are embedded within an amorphous carbon matrix. The Ni crystallites vary in size between ~5 and ~20 nm, although this variation appears to be smaller than for the Fe sample. The oxygen map shows that, although the carbon matrix (or its surface) contains some oxygen, the oxygen concentration in the Ni particles themselves is below the detection limit of the technique (estimated to be 5-10% for the Ni particles in this sample). The chemistry (and thus the magnetic properties) of each Ni crystallite can be contrasted with that of the larger crystal seen at the bottom left of the image, which is not encased in carbon and whose oxygen content is much higher.



**Figure 2:** EFTEM images obtained from the nickel sample. (a) Zero-loss bright field image; (b) C K, (c) Ni  $L_{2,3}$  and (d) O K three-window background-subtracted elemental maps.

## References

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