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Short communication

# Tailoring phase composition in carbon-encapsulated iron nanoparticles

M. Bystrzejewski<sup>a,\*</sup>, A. Grabias<sup>b</sup><sup>a</sup> Warsaw University, Department of Chemistry, Pasteur 1 str., 02-093 Warsaw, Poland<sup>b</sup> Institute of Electronic Materials Technology, Wólczyńska 133 str., 01-919 Warsaw, Poland

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## ABSTRACT

A method in which the phase composition of carbon-encapsulated iron nanoparticles can be tailored in a controlled manner is reported. The proposed route incorporates oxygen in the synthesis process. Clear correlations between the quantities of bcc Fe, fcc Fe and Fe<sub>3</sub>C phases encapsulated in carbon shells and amounts of oxygen have been found.

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## 1. Introduction

Magnetic nanoparticles have attracted great attention of materials science community due to their outstanding size-dependent physical properties [1]. The intrinsic characteristics of magnetic nanoparticles result in their prospective applications that include materials science, catalysis and environmental protection [2–4]. However, high reactivity of magnetic nanoparticles due to their large surface area leads to agglomeration and oxidation at ambient conditions [5]. Oxidation is a serious problem, because metal oxides that are formed during corrosion may have a very different magnetic response in comparison to the bare nanoparticles. As an example, the saturation magnetization ( $M_s$ ) of bcc Fe is 224 emu/g, whilst the maximum moment achievable in

$\alpha$ -Fe<sub>2</sub>O<sub>3</sub> is 83 emu/g only. The encapsulation approach has been proposed to enhance the stability of magnetic nanoparticles. Carbon seems to be the best coating material, since it is light, non-toxic and inert in a wide range of oxidative environments.

It is widely accepted, that carbon-encapsulated magnetic nanoparticles (CEMNPs) are formed in a rapid quenching of gas containing carbon species and metal to be encapsulated [6]. There are two major problems that accompany the synthesis of CEMNPs: (i) formation of non-encapsulated particles and amorphous carbon, (ii) a diversity of metal-based encapsulated phases. The first problems are easy to overcome, e.g. via wet chemistry routes. The second disadvantage may become a serious drawback because various metallic phases can have completely different magnetic response. In the case of iron at least three kinds of phases

\* Corresponding author. Tel.: +48 22 822 02 11; fax: +48 22 822 59 96.  
E-mail address: [mibys@chem.uw.edu.pl](mailto:mibys@chem.uw.edu.pl) (M. Bystrzejewski).

can be encapsulated: bcc Fe, fcc Fe and iron carbides. The fcc Fe is the most unwanted phase, because it is paramagnetic and results in decreasing the overall magnetic moment in the CEMNP materials. Up to date there were no information dealing with the possibility of controlling the phase composition of carbon-encapsulated iron nanoparticles. In this communication we show that the inclusion of oxygen during synthesis of CEMNPs can be used to tailor the phase composition in a controlled manner. It has been reported in our previous work that oxygen significantly reduces the amount of amorphous carbon [7]. The rate of reduction of amorphous carbon changed monotonically with the oxygen content, and the complete elimination of amorphous carbon has been found when ethanol was used as a carbon source.

## 2. Experimental

Carbon-encapsulated iron nanoparticles were synthesized using an inductively coupled radio frequency thermal plasma torch. The detailed experimental procedure is described elsewhere [7]. In brief, iron powder and various carbon precursors were simultaneously evaporated and decomposed in a plasma jet. The inclusion of oxygen was realized by appropriate selection of carbon feedstock (e.g. an alcohol) or addition of gaseous oxygen to the plasma gas. Operational parameters and oxygen contents are summarized in Table 1. The as-obtained products were subjected to the purification treatment in order to leach all non-encapsulated metallic particles. This was a crucial step, because the non-encapsulated particles also contribute to the magnetic response of the studied samples. The morphology of CEMNPs was studied by scanning (SEM) and transmission (TEM) electron microscopy. X-ray diffraction patterns were acquired using Cu K-alpha radiation with a step of 0.05°. Mössbauer spectroscopy measurements were performed in a transmission geometry at room temperature. A constant-acceleration spectrometer with a 25 mCi  $^{57}\text{Co}$  source in a rhodium matrix was used. A proportional counter was employed to detect the transmitted Mössbauer  $\gamma$  radiation. The spectra were fitted assuming Lorentzian profile lines by a non-linear least-squares NORMOS program. Determination of the hyperfine parameters, such as magnetic hyperfine field and isomer shift for six-line spectral components, electric quadrupole splitting and isomer shift for quadrupole doublets, and isomer shift for single lines allowed us to identify the iron-containing phases. The relative fractions of the spectral

components related to the identified phases were calculated as a ratio of the area of the relevant subspectrum to the total spectral area, assuming similar Debye-Waller factors for each phase. Isomer shifts are given with respect to the  $\alpha$ -Fe standard.

## 3. Results

The morphology of Fe-contained CEMNPs is similar for all tests. Spheroid nanoparticles with the size below 100 nm (Fig. 1A) are dominant. More detailed morphological features are provided by a TEM image (Fig. 1B), which shows an example of a core-shell structure of an individual carbon encapsulate. Phase analysis by X-ray diffraction does not show any significant differences in all samples. The XRD pattern visualizes four phases to be present: semi-crystalline graphite, bcc Fe,  $\text{Fe}_3\text{C}$  and fcc Fe(C) solid solution (Fig. 2A). The samples were also burned in air in order to show if they contain any foreign phases or elements that were too small or too disordered to be detected in diffraction patterns. Burning caused gasification of all carbon particles, whilst metallic cores were oxidized to a solid residue. The XRD pattern of the as-obtained residue shows only one crystalline phase to be present —  $\alpha$ - $\text{Fe}_2\text{O}_3$  (Fig. 2B).

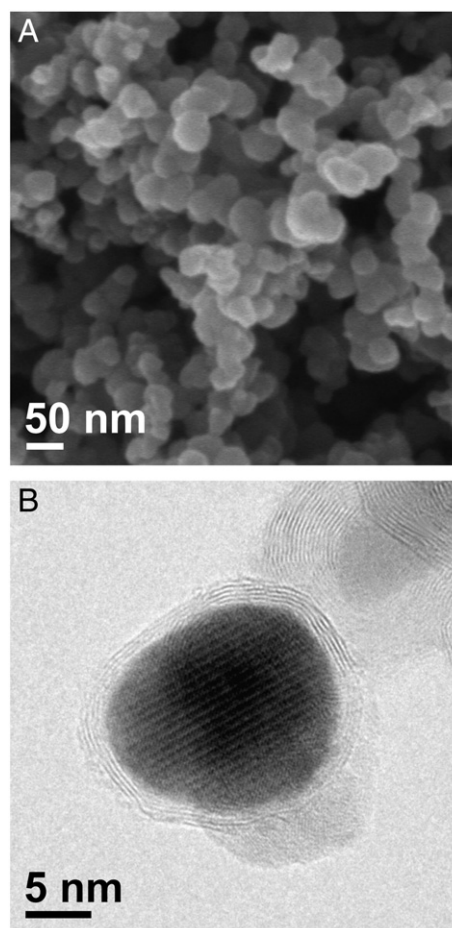


Fig. 1 – SEM and TEM images of product from Test 1.

Table 1 – Operational parameters and quantitative phase composition of products.

Test number	Carbon feedstock	O/C ratio	bcc Fe [%]	$\text{Fe}_3\text{C}$ [%]	fcc Fe + fcc Fe(C) [%]
Ar plasma	1 Hexane	0.00	25	49	26
	2 Toluene	0.00	27	56	17
	3 Hexanol	0.17	24	42	34
	4 Ethanol	0.50	34	40	26
Ar-O <sub>2</sub> plasma	5 Hexane	0.06	25	47	28
	6 Hexane	0.32	28	44	28
	7 Toluene	0.04	24	46	30
	8 Toluene	0.25	23	43	34

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