

Fe-B Nanocapsules Characterized by Analytical Electron Microscopy

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Magnetic metal particles in nanometer scale provide a unique dimensionally confined system, exhibit particle size related properties and have great potential in drug delivery, magnetic fluid and magnetic recording. To protect the fine metal particles from oxidation, magnetic nanocapsules with a core/shell structure have been developed. Most of the nanocapsules were prepared in carbon system, that is, the particles were encapsulated in a carbon shell with graphite crystal structure. Three years ago, we fabricated magnetic nanocapsules in boron system, consisting of various crystalline Fe-B cores and amorphous B-O shells [1]. In order to investigate if the cores are protected by the amorphous B-O shells and understand the formation of crystalline cores, the Fe-B nanocapsules were recently re-examined using high-resolution transmission electron microscopy (HRTEM), energy-filtered transmission electron microscopy (EFTEM), energy dispersive X-ray (EDX) microanalysis and micro-diffraction (MD). A combination of these techniques proves powerful to characterize complex structural features at the nanometer scale.

The well-dispersed nanocapsules were prepared by arc discharge in diborane [1]. Bright field micrograph and EFTEM elemental maps (Fig.1) show the shell/core morphology of the nanocapsules in which oxygen is still only located in the shells.

The crystalline cores still remain unchanged and contain several phases. Fe₂₃B₆ (Figs. 2 and 3) with fcc structure ($a=1.07$ nm) has been newly identified, together with α -Fe(B), FeB, Fe₃B and γ -Fe(B). A close relationship between Fe₂₃B₆ and γ -Fe(B) phases has been found from both diffraction patterns and images, suggesting a possible phase transformation (Fig. 3). The cores can be one of Fe-B phases or a combination of two or more phases. Statistics of micro-diffraction data indicate that the cores containing metastable Fe₃B or Fe₂₃B₆ phases which are normally formed during quenching account for 80% of the total cores.

An amorphous or partially crystallized iron oxide layer has been identified between the boron oxide shell and the core, and is attributed to poor vacuum in the sample preparation chamber instead of post-oxidation.

These recent results indicate that the shell can well protect the core from oxidation even the shell is amorphous and contains oxygen, and the quenching procedure in sample preparation plays an important role in forming the core phases.

[1] Z.D. Zhang, J.L. Yu, J.G. Zheng, I. Skorvanek, J. Koac, X.L. Dong, Z.J. Li, S.R. Jin, H.C. Yang, Z.J. Guo, W. Liu, X.G. Zhao, Phys. Rev. B., 64 (2001), 244041

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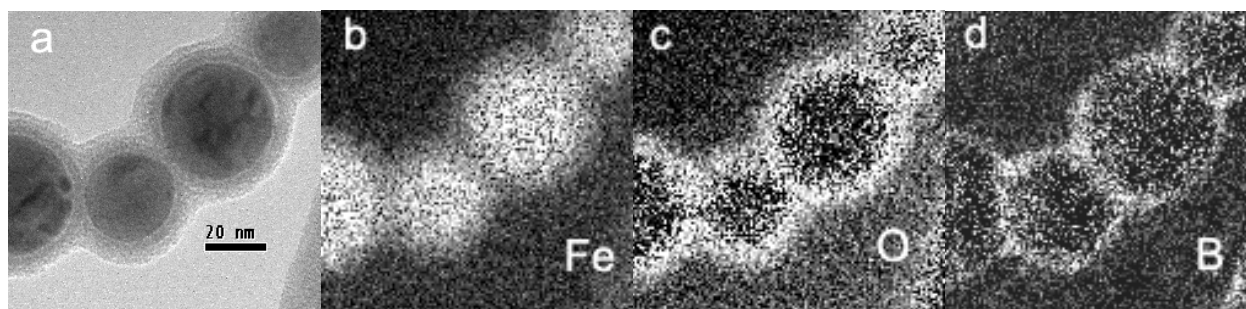


Figure 1 (a) Bright field TEM micrograph of Fe-B nanocapsules. (b)-(d) EFTEM images show elemental maps of Fe, O and B, respectively.

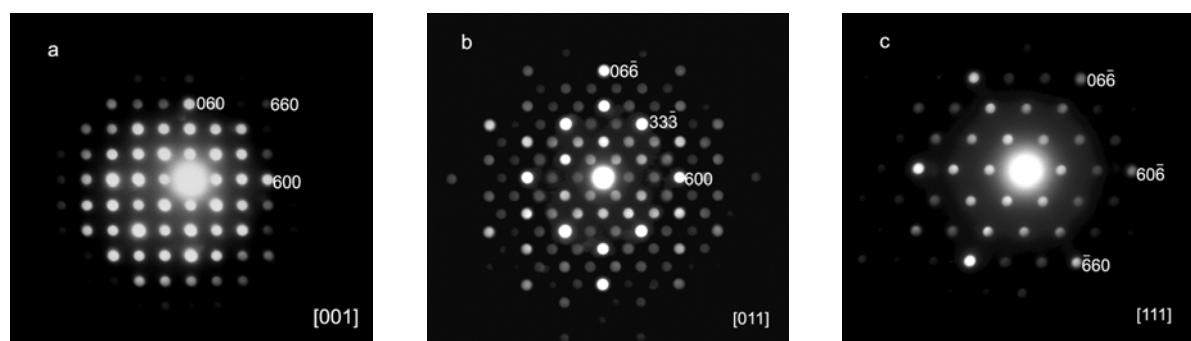


Figure 2. (a)-(c) Micro-diffraction patterns recorded from a Fe_{23}B_6 core along the [001], [011] and [111] zone axes, respectively.

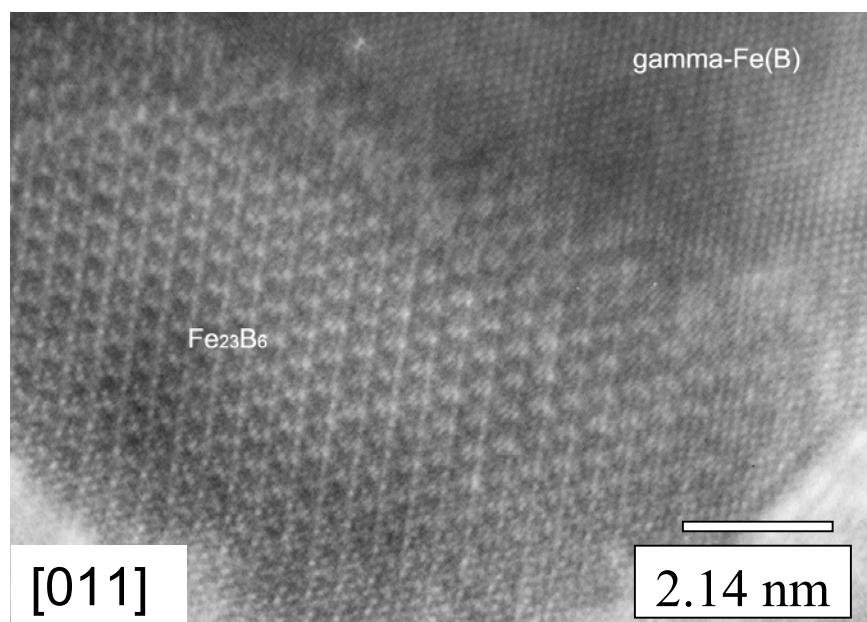


Figure 3. HRTEM image of a core containing $\gamma\text{-Fe(B)}$ and Fe_{23}B_6 phases recorded along the [011] zone axis.