

Functional Material

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HRTEM LaB₆: characterization of nano particles

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The transmission electron microscope is one of the central techniques used for characterization of nano-materials. The performance of an electron microscope strongly depends on the properties and characteristics of the electron source. In the absence of a field emission gun, a high resolution transmission electron microscope with a conventional LaB₆ gun can be very effective in performing most of the routine works aimed at the characterization of nano-materials structure. This presentation exhibits a few examples of employing a JEOL JEM-2100 high resolution electron microscope for structural characterization of nano-materials. The studies were performed in TEM, HRTEM, STEM, EDS mode, using conventional selected area diffraction (SAED), dark field (DF) imaging techniques, nano-beam diffraction (NBD) or Fourier transform analysis (FFT) of the high resolution images. While exploiting the high level of the atomic imaging capability of this microscope and using computational FFT we are characterizing nano-materials and receive information at the level close to the sub-nanometer level. Few examples of non - standard applications are summarized herein [1]:

Magnetic poly(divinyl benzene)/Fe₃O₄ microspheres with a narrow size distribution were produced by entrapping the iron pentacarbonyl precursor within the pores of uniform porous poly(divinyl benzene) microspheres prepared in our laboratory, followed by the decomposition in a sealed cell of the entrapped Fe(CO)₅ particles at 300°C under an inert atmosphere. Magnetic onion-like fullerene microspheres with a narrow size distribution were obtained by annealing the PDVB/Fe₃O₄ particles at 500, 600, 800, and 1100°C, respectively, under an inert atmosphere. Characterization of the PDVB/Fe₃O₄ microsphere is shown in figure 1 displaying a bright field image (BF) of a microsphere obtained by annealing the Fe(CO)₅ entrapped within the PDVB particles at 300°C.

Figures 2 exhibit TEM micrographs of the composite microsphere obtained at 500°C, displaying the carbon graphitic coating. The the (DF) image display the spatial distribution of the graphitic carbon, the onion-like fullerenes that coat the magnetic nano-particles in the microsphere. This amazing phenomenon of fullerene formation can be observed at all temperatures ranging from 500 to 1100°C.

1. Ron Snovski, Judith Grinblat, and Shlomo Margel, Langmuir, 27 (2011), p.11071-11080.

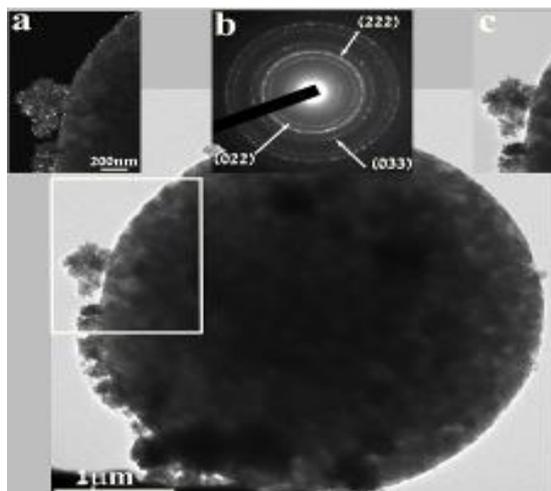


Figure 1. Bright field image of a microsphere obtained by annealing the $\text{Fe}(\text{CO})_5$ entrapped within the PDVB particles at 300°C . DF image taken in the (220 and 311) reflection for the area marked by the white square (a). SAED from a 300nm area showing the ring diffraction of iron oxide (b).

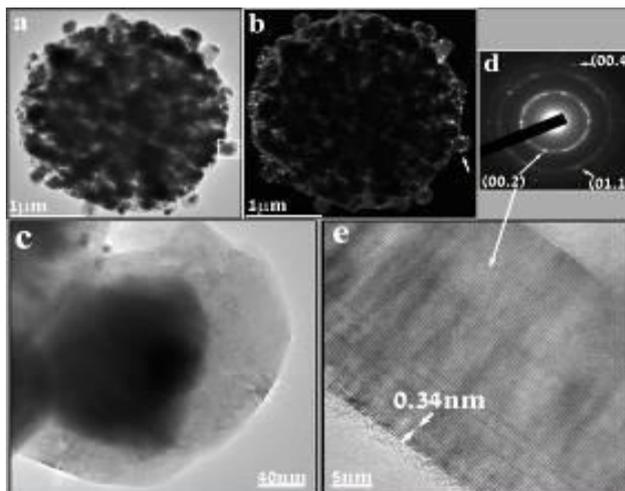


Figure 2. Bright field (a) DF (b) images of the $\text{C}/\text{Fe}_3\text{O}_4/\text{Fe}$ composite microsphere obtained at 500°C displaying the spatial distribution of the crystalline onion-like graphitic carbon. HRTEM image of the area marked by the white square, exhibiting the fullerene onion-like coating of the magnetic nano-particle (c). SAED from a 300nm area of the microsphere showing the ring diffraction pattern the hexagonal graphitic carbon (d). HRTEM image of the carbon coating (e).