

# Easy nickel substrate-assisted growth of uniform carbon microspheres and their spectroscopic properties

Xuchu Ma <sup>a</sup>, Fen Xu <sup>b</sup>, Liyong Chen <sup>a</sup>, Yange Zhang <sup>a</sup>, Zude Zhang <sup>a,\*</sup>,  
Jue Qian <sup>a</sup>, Yitai Qian <sup>a</sup>

<sup>a</sup> Department of Chemistry, University of Science and Technology of China, Hefei, Anhui 230026, PR China

<sup>b</sup> Laboratory of Inorganic Materials Chemistry, The University of Namur (FUNDP), 61 rue de Bruxelles, B-5000 Namur, Belgium

Received 10 April 2006; accepted 3 June 2006

Available online 7 July 2006

**Keywords:** Graphitic carbon; Carbonization; Scanning electron microscopy

There is an immense interest in the fabrication of carbon materials, which stems from their unique structures, texture and properties. Especially, carbon spheres have attracted much attention due to their potential as lubricating materials, reinforcement materials for rubber, supports for catalysts, and the negative electrode in lithium secondary batteries. Some methods have been explored to prepare spherical carbons, such as CVD [1], hydrothermal [2], carbonization [3], pyrolysis [4], self-assembly template [5], carbonization of poly(furfuryl alcohol) [6]. However, most of them were carried out at a higher temperature (usually not lower than 600 °C) and/or with the need of catalysts or involving two-step procedure. Furthermore, the particle size could not be easily controlled owing to the encapsulation of catalyst into the products. Thus, simple methods for the synthesis of carbon spheres are especially welcome if they can produce samples with high purity and also high uniformity from inexpensive precursors. Herein, we report an easy substrate-assisted route where the carbonization of acetone at ~550 °C can be utilized to fabricate large quantities of uniform carbon microspheres with uniform sizes of the mean diameter of 5 μm.

The typical procedure was as follows: firstly, Ni foil (1 × 3 cm) was sonicated in acetone for 5 min before it was immersed into dilute HCl acid to remove the exterior oxide, then Ni foil was washed by distilled water and ethanol, and dried in vacuum at 30 °C for 6 h. Secondly, 8 mL acetone was filled into a titanium alloy autoclave with 10-mL-capacity, and then the processed Ni foil was loaded into the autoclave vertically. Finally, after being sealed and heated at 550 °C for 12 h, the autoclave was cooled to room temperature naturally. Ni foil was removed carefully, rinsed with distilled water and ethanol, and then

dried at 30 °C. The black products on Ni foil were scraped off for further characterization.

All the reflection peaks in XRD (Philip X' Pert PRO SUPER) pattern of samples on Ni foil (Fig. 1a) can be indexed to carbon (JCPDS File, No. 41–1487) and Ni (JCPDS File, No. 04–0850), respectively. The XRD pattern of pure black products removed from the surface of Ni foil (Fig. 1b) exhibits a wide diffraction peak at 26.2° ( $d = 3.40 \text{ \AA}$ ), which can be assigned to (002) plane of graphite carbon. The broad (002) peak shifts slightly to a lower angle compared to that of the graphite carbon. In the lattice, the (00*l*) reflections are associated with the orderly stacking along *c*-axis, and the slight shifting of this peak to a lower angle indicates a little lattice expansion between two adjacent graphite layers along the *c*-axis, which could be attributed to the introduction of the strain owing to the curvature of the layers. Moreover, the layers cannot be fully commensurate because the number of atoms in the layer increases with its radius, and this divergence can be partially relieved by the lattice expansion along the *c*-axis. So, this shift would also reflect the strain relief mechanism associated with folding of the initial graphite layers, similar to S–W–S layers [7].

Fig. 2a and b are FE-SEM (JEOL JSM-6700 F) images of the products. Fig. 2a displays the panoramic FE-SEM image, revealing that a large quantity of monodispersed carbon spheres have been grown on the surface of Ni foil. The proportion of uniform carbon microspheres in all as-produced carbon materials can be estimated to be ~90% from this FE-SEM image. Fig. 2b is another FE-SEM image with higher magnification, demonstrating that carbon spheres have uniform sizes with narrow diameter distribution of 4.5–5.5 μm. Additionally, Fig. 2b also indicates that samples exhibit fairly uniform sphericity with smooth surfaces across the entire substrate. Fig. 3a is a typical TEM (Hitachi Model H-800) image, showing that carbon spheres have uniform sizes with the mean diameter of

\* Corresponding author. Fax: +86 551 3601592.  
E-mail address: zhanglab@ustc.edu (Z. Zhang).

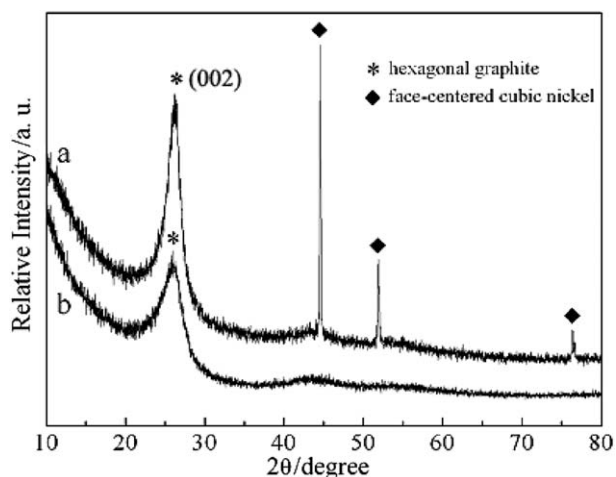


Fig. 1. XRD patterns of the products: (a) carbon microspheres (pointed by \*) on the Ni foil (denoted by ◆); (b) pure carbon materials removed from the surface of Ni foil.

5  $\mu\text{m}$ . Detailed structure information comes from HRTEM (JEOL 2010) image (Fig. 3b), where the lattice fringes of interplanar spacing between the adjacent carbon sheets can be identified in the figure. The average value of (002) spacing is 0.34 nm, which is close to the literature data and the XRD result. Several disorders also can be observed in the (002) planes. An EDX spectrum (Fig. 3c) demonstrates the high surface purity of the products.

A typical Raman spectrum (LABRAM-HR Confocal Laser) is shown in Fig. 4a, where a prominent peak at  $1599\text{ cm}^{-1}$  and another weak peak at  $1335\text{ cm}^{-1}$  can be clearly detected. The peak at  $1599\text{ cm}^{-1}$  (G-band) originates from the stretching modes of carbon  $\text{sp}^2$  bonds of the graphite ( $\text{E}_{2g}$  mode), while the peak at  $1335\text{ cm}^{-1}$  (D-band) is associated with the vibrations of carbon atoms with dangling bonds in disordered graphite planes [8]. Usually, the intensity ratio of  $I_D/I_G$  would decrease as the content of the amorphous carbon diminishes. In our case, the ratio of  $I_D/I_G = 0.70$  indicates the low graphitization degree of carbon spheres and the presence of disordered carbon, because the growth temperature ( $550\text{ }^\circ\text{C}$ ) is not high enough to improve the local order of the newly-formed

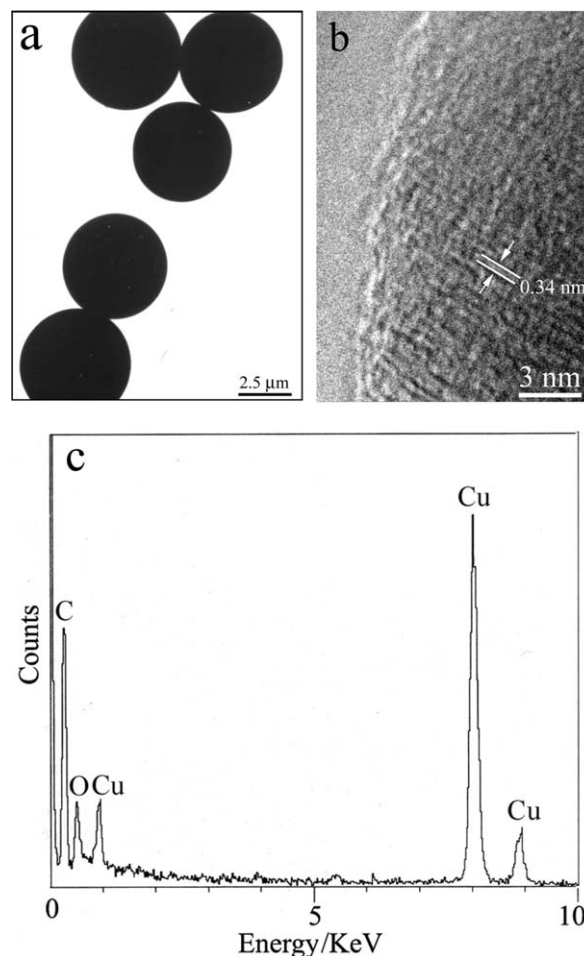


Fig. 3. (a) A typical TEM image of carbon microspheres; (b) HRTEM image taken on the surface of the carbon spheres; and (c) a typical EDX spectrum.

graphite layers. However, this data of  $I_D/I_G = 0.70$  could be comparable with those of the previously reported carbon spheres [2–4,6]. A further study on the carbon spheres is illustrated by FT-IR (EQUINOX 55) spectrum (Fig. 4b), which presents one peak at  $1630\text{ cm}^{-1}$  (C=C groups) and another weak peaks around the region of  $2920\text{--}2850\text{ cm}^{-1}$  (the surface C–H groups).

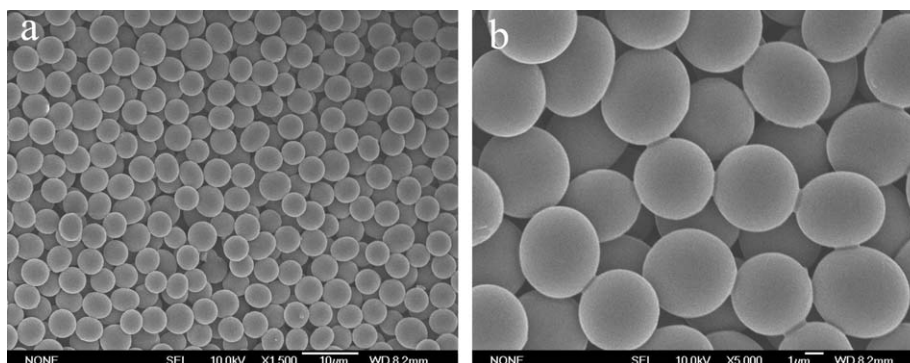


Fig. 2. (a) The panoramic FE-SEM image of carbon microspheres grown on the surface of Ni foil; (b) an enlarged FE-SEM image, illustrating the carbon spheres have the uniform size and sphericity.

Download English Version:

<https://daneshyari.com/en/article/1419700>

Download Persian Version:

<https://daneshyari.com/article/1419700>

[Daneshyari.com](https://daneshyari.com)