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Materials Research Bulletin 43 (2008) 1549–1554
Materials Research Bulletin
www.elsevier.com/locate/matresbu

Formation, characterization and magnetic properties of carbon-encapsulated iron carbide nanoparticles

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Received 27 October 2006; received in revised form 13 April 2007; accepted 3 June 2007
Available online 9 June 2007

Abstract

Fe_3C nanoparticles encapsulated in carbon shell with a size range of 20–50 nm were obtained in large scale by reacting anhydrous $FeCl_3$, hexamethylenetetramine and metal Na in an autoclave at 650 °C. Magnetization measurements show that the as-obtained materials display superparamagnetic properties at room temperature. A possible formation mechanism of the core–shell nano-structures was discussed.

Keywords: A. Carbides; A. Magnetic materials; B. Chemical synthesis; D. Magnetic properties

1. Introduction

Great interest has been focused on carbon–magnetic material systems due to the possibility of obtaining magnetic nanoparticles encapsulated by crystalline or amorphous carbon. The encapsulated magnetic nanoparticles can be widely applied in areas such as magnetic data storage and magnetic resonance imaging [1,2]. Furthermore, the core–shell structure is of considerable significance, not only because the encapsulated species are likely to be immune to environmental effects or degradation owing to the protective carbon shells around them, but also because it offers an opportunity to investigate dimensionally confined systems [3].

The iron carbide (Fe_3C, cementite) nanoparticle is a very important compound due to its potential applications in the preparation of materials used for construction of machines or equipment [4] and in the production of magnetic supports of information [5]. Up to now, many methods have been employed to synthesize Fe_3C. Gedanken and co-workers have prepared Fe–Fe_3C nanocrystalline particles by sonochemistry and investigated their properties, but their samples need to be annealed at 700 °C [6,7]. Nelson and Wagner reported the synthesis of high-surface area Fe_3C nanoparticles using alkalide reduction method, but the sample also needed to be annealed at 950 °C [8]. Narkiewicz et al. prepared Fe_3C by carburisation of nanocrystalline iron with methane, where the magnetite alloy needed to be reduced under hydrogen at 500 °C first, and then the as-obtained Fe nanoparticles reacted with methane under hydrogen flow at 520 °C [9]. Zhang et al. developed a novel CVD method to synthesize Fe_3C nanowires filled in carbon nanotubes with titanate modified palygorskite as catalyst [10]. All these preparation techniques need either higher temperature or complicated operation. Recently, Song et al. synthesized the carbon-encapsulated iron carbide

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0025-5408/$ – see front matter © 2007 Published by Elsevier Ltd.
doi:10.1016/j.materresbull.2007.06.017

nanoparticles by co-carbonization of durene with ferrocene, which was a relatively simple method for the synthesis of iron carbide up to now [11].

In this paper, we also describe a facile method to synthesize carbon-encapsulated Fe_3C nanoparticles by reacting anhydrous $FeCl_3$, hexamethylenetetramine (HMT) and metal Na at a relatively low temperature. In the present route, the chemical reaction mechanism is similar to previous reports [9,10], but the operation is easier. The high pressure in the autoclave coming from the pyrolysis and vaporization of reagents may facilitate the reaction process, thereby the reaction even can be initiated at 550 °C.

2. Experimental

In a typical experiment, 1.622 g anhydrous $FeCl_3$, 1.0 g metal Na and 2.8 g HMT were put into a 50 mL stainless steel autoclave. The autoclave was sealed, heated at a rate of 10 °C/min to 650 °C and kept at the temperature for 12 h. It was then cooled to room temperature naturally. The product was collected and washed with distilled water and pure ethanol several times to remove NaCl and other impurities. After that, the product was dried in vacuum at 50 °C for 4 h.

The X-ray powder diffraction (XRD) patterns were collected on a Bruker D8 advance X-ray diffraction solutions equipped with graphite monochromatized Cu Kα radiation (λ = 1.5418 Å). High-resolution transmission electron microscopy (HRTEM) images, selected area electron diffraction (SAED) patterns and energy dispersive X-ray spectroscopy (EDS) were recorded on a JEOL JEM2100 transmission electron microscope. Magnetization measurements were performed with a MicroMag 2900 at room temperature.

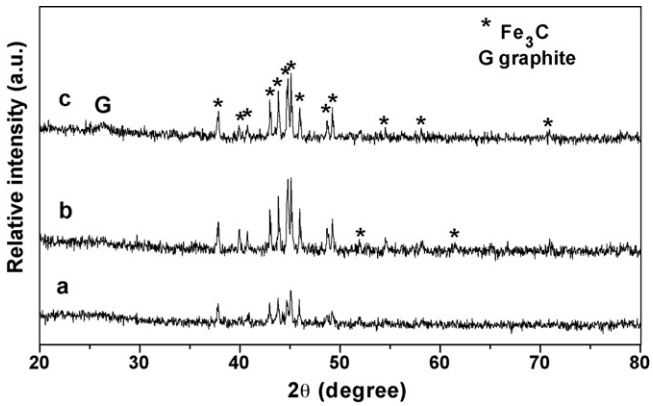

Fig. 1. XRD patterns of the products prepared with HMT 2.8 g at (a) 550 °C, (b) 600 °C and (c) 650 °C for 12 h.

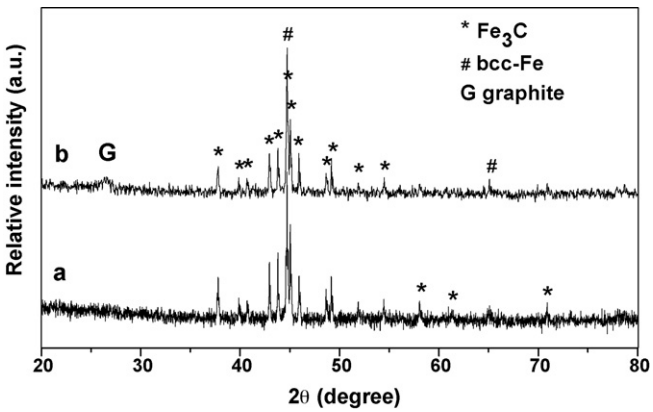

Fig. 2. XRD patterns of the products obtained at 650 °C with (a) HMT 1.4 g and (b) HMT 2.1 g for 12 h, respectively.