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ScienceDirect

Ceramics International 40 (2014) 16529–16534

CERAMICS INTERNATIONAL

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Structural and magnetic properties of nickel ferrite nanoparticles synthesized via a template-assisted sol–gel method

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Received 22 October 2013; received in revised form 1 August 2014; accepted 2 August 2014
Available online 11 August 2014

Abstract

Spinel nickel ferrite ($NiFe_2O_4$) nanoparticles with high specific area and excellent magnetic properties were synthesized via a template-assisted sol–gel method. The effects of template type on the structural properties, magnetic properties, and heating rate are investigated. Results of XRD and TEM indicate that the $NiFe_2O_4$ particles synthesized using templates exhibit a typical single phase spinel structure in nano size. The sample using sponge or cotton as template presents the best saturation magnetization (66.6 emu/g) and the lowest coercivity (137.7 Oe), respectively, and their specific surface area is enhanced greatly from 20.1 m^2/g to 55.7 m^2/g. It is found that the calculated hysteresis loss agrees well with the measured radio frequency heating rate.
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Keywords: Nickel ferrite; Template-assisted sol–gel method; Specific surface area; Magnetic properties

1. Introduction

Spinel $NiFe_2O_4$, one of the most important ferrite materials, has been widely studied because of its application in many fields, such as ferrofluids, catalysts, microwave devices, gas sensors, magnetic materials, and so on [1–7]. $NiFe_2O_4$ is an inverse spinel in which a unit cell consists of 8 $NiFe_2O_4$ molecules. Half of the ferric ions preferentially occupy the tetrahedral sites (A-sites) and the others occupy the octahedral sites (B-sites). Therefore, $NiFe_2O_4$ can be represented by the formula $(Fe^{3+})_A[Ni^{2+}Fe^{3+}]_BO_4^{2-}$, where A and B represent the tetrahedral and octahedral sites, respectively [8]. The remarkable electrical and magnetic properties of $NiFe_2O_4$ depend on the nature, the charges, and the distribution of metal ions. However, $NiFe_2O_4$ shows ferromagnetism that originates from the magnetic moment of anti-parallel spins between Fe^{3+} ions in A sites and Ni^{2+} ions in B sites [9].

Nanoparticles with controlled size and composition have attracted more and more fundamental and technological interests. The synthesis method determines the morphology as well as the physical properties of products. $NiFe_2O_4$ ferrite is usually synthesized via the solid state method [10]. However, this method has some unconquered disadvantages, such as high sintering temperature, inhomogeneity of the final product, growth of large crystallites, and so on. To avoid these disadvantages, various wet chemical routes, such as co-precipitation [11], sol–gel [12], microwave [13], aerosolization [14], and reverse micelle technique [15] were developed to synthesize nanocrystalline $NiFe_2O_4$. The sol–gel technique is feasible for tailoring physical properties by optimizing synthesis conditions, such as gelation time, temperature, reagent concentration, media pH value, and so on. The particles obtained via a sol-gel process are high phase purity, chemically homogeneous, and flexible to control the morphology of grains. Template-assisted synthesis is an effective approach to synthesize size/shape-controlled nanoparticles [16]. However, most templates need to be fabricated by elaborate and time-consuming procedures in advance. Moreover, the complete removal of template is still a challenge [17]. Recently, some

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http://dx.doi.org/10.1016/j.ceramint.2014.08.006
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biological materials are identified as the ideal templates for the synthesis of nanoparticles [16,18]. In this study, $NiFe_2O_4$ nanoparticles were synthesized via a sol–gel method using medical absorbent cotton and sponge as the template, respectively. The effects of different template on morphology, specific surface area, magnetic properties, heating rate at 295 kHz, and hysteresis loss are studied.

2. Experimental process

2.1. Synthesis of samples

Spinel $NiFe_2O_4$ ferrites were synthesized by a sol-gel method. Iron (III) nitrate nonahydrate, nickel nitrate hexahydrate, citric acid, ethanol, and ammonia solution (Sigma Aldrich, ACS grade) were used as the starting materials. The stoichiometric metal nitrates and the citric acid were separately dissolved in ethanol. The acid solution was then added to the nitrates solution until the pH value was adjusted to 2, accompanied with a rigorous stirring for 4 h. The sol obtained was kept stirred rigorously for 24 h and then divided into three parts. The first part was dried at 353 K to obtain a dry gel which was milled and calcined at 1073 K for 1 h with an air flow of 50 mL/min to produce the $NiFe_2O_4$ ferrite. The remaining two parts were firstly absorbed by small blocks of medical absorbent cotton and sponge, respectively, and then dried and calcined through the same process as described above for the first part. Both medical absorbent cotton and sponge were obtained from Changsha Antaeus Fine Chemical Industrial Co., Ltd, China.

The obtained $NiFe_2O_4$, $NiFe_2O_4$-cotton, and $NiFe_2O_4$-sponge are denoted as sample 1♯, 2♯, and 3♯, respectively.

2.2. Characterization

The phase composition of samples was determined with a Bruker D8 advance X-ray diffractometer (XRD) using Cu K_α radiation (wavelength λ=1.5418 Å). The morphology of samples was obtained on a Philips Tecnan F20 transmission electron microscope (TEM). The BET specific surface area was determined on a Quantachrome Nova 1000E nitrogen adsorption apparatus at 77 K. The magnetic hysteresis loops were measured at room temperature on a LakeShore7407 vibrating sample magnetometer (VSM) with a maximum external field H_m=20 kOe. The radio frequency (RF) heating rates of sintered samples were measured in a quartz tube (i.d.=3 mm) inserted along the center axis in a 50 mm-long RF coil connected to an RF system operated at a current of 200 A and a frequency of 295 kHz (Easy Heat, Ambrell). Prior to the measurements, slurry of the materials (10 mg) in deionized water (0.08 mg) was prepared. The temperature of the medium was monitored with a fiber optic sensor, and the RF heating rate was calculated by considering the specific heat capacities of the slurry and the ferrite.

3. Results and discussion

3.1. Microstructure of the templates

SEM images of the selected template materials are shown in Fig. 1. As natural fibers, the medical absorbent cotton used is composed of 96–98 wt.% cellulose, and has the morphology of twisted long fibers (Fig.1(a)), while the sponge used exhibits a honeycomb-like porous structure (Fig.1(b)).

3.2. XRD patterns of samples

XRD patterns of samples 1♯, 2♯, and 3♯ are shown in Fig. 2. Seen from the figure, the typical diffraction peaks from spinel are found in all the XRD patterns, which means that $NiFe_2O_4$ is the main phase in all samples. It is found that some impurity peaks from cubic-Fe_2O_3 are also observed in the pattern of sample 1♯, while no impurity peak can be found in the patterns of samples 2♯ and 3♯. This difference could be attributed to the slight composition segregation in the drying process when the sol was not absorbed by the templates [19].

The average crystal size D was obtained from the (311) peaks in XRD patterns using the Scherrer equation [20]:

$$D = \frac{k\lambda}{B \sin \theta} \quad (1)$$

where k is the Scherrer constant (0.89), B is the full-width at half maximum (FWHM) of a diffraction peak, and θ is the Bragg's angle. Also, from the position of the (311) peak ($2\theta_{(311)}$) in the XRD patterns, the $d_{(311)}$ interplanar spacing can

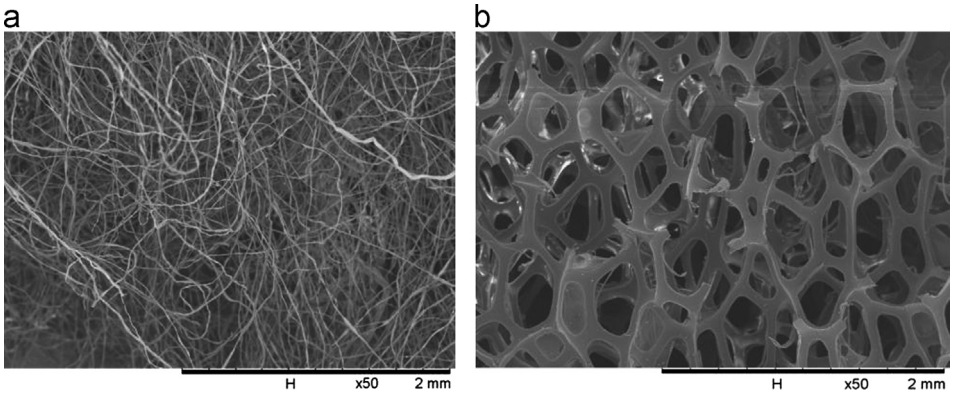

Fig. 1. Typical SEM images of the templates. (a) Absorbent cotton (b) Sponge.

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