Materials Chemistry and Physics 120 (2010) 213–216

Contents lists available at ScienceDirect

Materials Chemistry and Physics

journal homepage: www.elsevier.com/locate/matchemphys

Formation and magnetic properties of M-Sr ferrite hollow fibers via organic gel-precursor transformation process

Fuzhan Song, Xiangqian Shen*, Jun Xiang, Haojie Song

School of Materials Science and Engineering, Jiangsu University, Xuefu Road, Zhenjiang 212013, Jiangsu, PR China

ARTICLE INFO

Article history:
Received 8 June 2009
Received in revised form 27 October 2009
Accepted 29 October 2009

Keywords:
M-Sr ferrite
Hollow fibers
Magnetic property
Shape anisotropy

ABSTRACT

The M-Sr ferrite hollow fibers have been successfully prepared by the organic gel-precursor transformation process. The phase formation process of M-Sr ferrite is analyzed by FTIR, XRD and consists of the gel-precursor thermal decomposition and the subsequent ferrite phase formation from strontium oxide and iron oxide. The M-Sr ferrite hollow fibers obtained are characterized with SEM and XRD, and show a high aspect ratio, fine diameters around 4 μm and a ratio of the hollow diameter to the fiber diameter being about 1/2. The optimized M-Sr ferrite hollow fibers are composed of nanograins with a hexagonal plate morphology. Magnetic properties are measured with VSM under a maximum applied of 1194 kA m^{-1}. The M-Sr ferrite hollow fibers formed at 1100 °C for 2 h with the specific saturation magnetization of 53.5 A m^2 kg^{-1} possess a shape anisotropy characteristic and the coercivity for the aligned hollow fibers parallel and perpendicular to the applied field is correspondingly 385 and 357 kA m^{-1}.

1. Introduction

The strontium ferrite (M-Sr), $SrFe_{12}O_{19}$, is a uniaxial ferromagnetic compound with the hexagonal magnetoplumbite structure. The hexagonal ferrites are well-known permanent magnets with a great technical importance and attracted an extensive attention for the last few decades. They are widely used in the fabrication of commercial permanent magnets, magnetic recording media and certain microwave devices owing to their high maximum saturation magnetization, high Curie temperature, theoretical maximum coercivity, high magneto-crystalline anisotropy and excellent chemical stability [1–3]. However, they are quite heavy as in bulk materials, which restricts their applications in many high-tech fields such as advanced microwave absorbents.

The studies showed that short fibers with a high aspect ratio could bring a much higher magnetic permeability than the same volume of materials in a non-fibrous form [4,5]. Fibrous forms of ceramic materials can be made stronger and often stiffer in mechanics than the bulk ceramic, and this will be an advantage if the M-Sr ferrite fibers are used in large composite magnets. The ferrite hollow fibers can reduce the specific density with a large specific area and will exhibit some unique characteristics.

Common processes produce polycrystalline ferrites with a large grain size of multi-domains and yield non-homogeneous mixtures and lattice strains in the material [6]. For a magnetically optimized crystallite a grain size of single-domain is required, with 80% of the theoretical maximum coercivity reported for grains of 0.1 μm. In order to achieve highly homogeneous single-domain ferrites, several techniques have been used, such as citrate-precursor [7,8], chemical co-precipitation [9], glass-crystallization [10], microemulsion [11,12] and hydrothermal [13,14] methods. Zhan et al. [15] prepared $ZnFe_2O_4$ ferrite nanometer fibers by electrospinning. The $NiFe_2O_4$ and Mn–Zn ferrite fibers were synthesized in the authors' laboratory using the organic gel-thermal decomposition process and the results have been previously reported [16,17]. Pullar and Bhattacharya [18] have prepared M-Ba ferrite fibers and M-Sr ferrite fibers by the aqueous organic gel method, but the formation process and relation between the structure and magnetic characteristics for these fibers are not fully understood.

The aim of this investigation therefore was to determine the feasibility of utilizing the organic gel-precursor transformation process to prepare the M-Sr ferrite hollow fibers, analyze the formation and characterize the microstructure and magnetic properties of the M-Sr ferrite hollow fibers.

2. Experimental

The raw materials were analytical grade $Sr(NO_3)_2$, $Fe(NO_3)_3{\cdot}9H_2O$ and citric acid. According to the stoichiometry, organic acid and metal nitrates were dissolved in deionized water to form aqueous solutions with a continuous magnetic stirring. The final solution was magnetically stirred for 20–24 h at room temperatures and was transferred to a rotary evaporator and evaporated in a vacuum at 60–70 °C to remove surplus water until a viscous liquid was obtained. The gel fibers were drawn from the spinnable gels by the domestic machine and dried in a vacuum oven at 80 °C for about 24 h. The dried gel fibers were then put in an alumina crucible

* Corresponding author. Te.: +86 511 88791964.
E-mail address: shenxq@ujs.edu.cn (X. Shen).

0254-0584/$ – see front matter © 2009 Elsevier B.V. All rights reserved.
doi:10.1016/j.matchemphys.2009.10.048

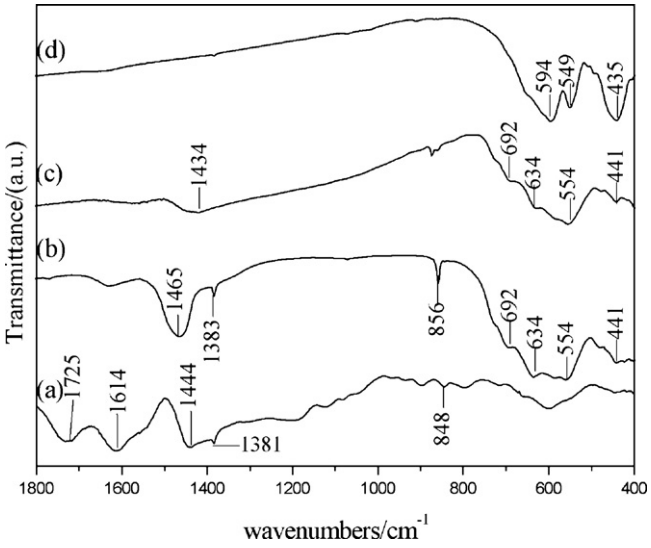

Fig. 1. FTIR spectra of gel precursor and products calcined at different temperatures, (a) gel precursor, (b) 600 °C, (c) 900 °C and (d) 1000 °C.

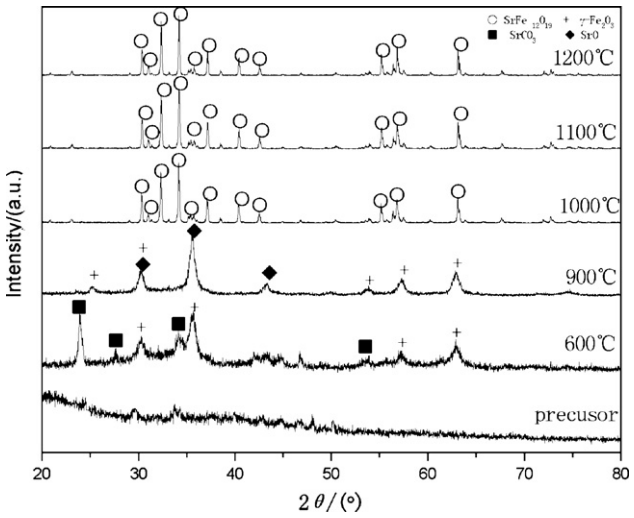

Fig. 2. XRD patterns of gel precursor and products calcined at different temperatures.

and subsequently were calcined at different temperatures for 2 h under an ambient atmosphere to form the ferrite fibers.

The structure, composition and morphology of the gel precursors and the products derived from thermal decomposition of these precursors at different temperatures were examined by Fourier transform infrared spectroscopy (FTIR) using a model of Nexu670 spectrometer, X-ray diffraction (XRD) using a D/max2500PC diffractometer (RIGAKU), and scanning electron microscopy (SEM) using a XL-30ESEM instrument (PHILIPS). The magnetic properties of the hollow ferrite fibers were measured using a vibrating sample magnetometer (VSM) at room temperature with applied field of 1194 kA m^{-1} (15 kOe).

3. Results and discussion

3.1. Phase transformation process of gel precursor

Fig. 1 shows the FTIR spectra for the gel precursor and fibers calcined at different temperatures. The spectrum of the gel precursor (Fig. 1a) indicates the anti-symmetrical and symmetrical stretching vibration bands of $RCOO^-$ related to the organic acid are located at 1614 and 1444 cm^{-1} while the characteristic absorbing band for the C–O vibration is around 1725 cm^{-1}. A set of bands appear at 1381 and 848 cm^{-1}, which are attributed to the N–O stretching and bending vibrations of NO_3^-, respectively [18]. When fired to 600 °C (Fig. 1b), the absorption bands related to γ-Fe_2O_3 (692, 634, 554 and 441 cm^{-1}) and CO_3^{2-} (1465, 1383 and 856 cm^{-1}) occur [19,20]. At 900 °C (Fig. 1c), the characteristic bands for CO_3^{2-} are no longer detected, whilst the band appear at 1434 cm^{-1} due to the Sr–O. When calcined at 1000 °C (Fig. 1d), the bands related to Sr–O and γ-Fe_2O_3 have completely disappeared, and the absorption bands at 594, 549 and 435 cm^{-1} are identified as M-Sr ferrite.

To aid further interpretation of the phase formation process, the FTIR spectra analysis is supplemented by the XRD analysis. Fig. 2 shows XRD patterns of the gel precursor and hollow ferrite fibers calcined at different temperatures for 2 h. It can be seen that the gel

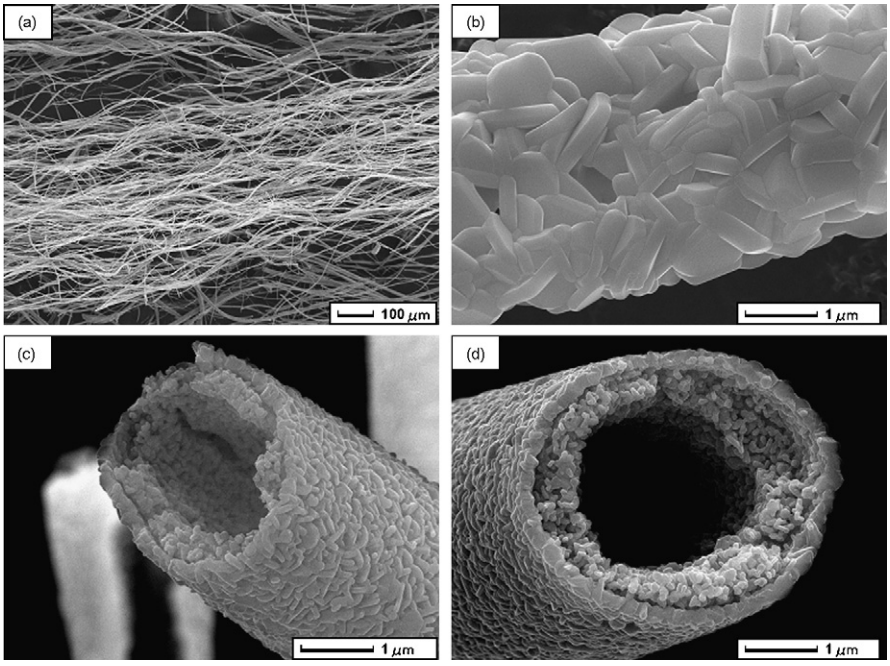

Fig. 3. SEM morphologies of the M-Sr ferrite hollow fibers.