Synthesis of $SrFe_{12}O_{19}$ by sol-gel method and its morphology and magnetic properties

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ABSTRACT

In this research, strontium hexa-ferrite nanoparticles (SrFe12O19) were synthesized by sol-gel method. The crystal structure, morphology and magnetic properties of nanoparticles were investigated using X-ray Diffraction (XRD), Scanning Electron Microscope (SEM) and Vibrating Sample Magnetometer (VSM). The XRD patterns confirmed the formation of single phase M-type hexagonal crystal structure for powders which was calcined above 700oC. The product shows the magnetization of 66 emu/g, which is consistent with pure hexa-ferrite obtained by other methods, and the magnetic coercivity of 6,145 kOe higher than expected for this hexa-ferrite. The powder morphology is composed of aggregates of hexagonal particles with an average particles size of above 100nm.

Keywords: *Strontium hexa-ferrite, sol-gel method, magnetic material.*

1. INTRODUCTION

M-type hexagonal ferrites are important permanent magnetic materials which was widely studied since their discovery in the 1950s [1]. As a kind of hard magnetic material, the hexagonal ferrite material efficiency has the chemical formula of MFe12O19 (M = Ba, Sr, Pb). Due to the magnetic properties and cost efficiency, hexa-ferrites was considered as a material with promising scientific and technological applications such as permanent magnets, electrical and microwave devices, data storage and recording, plastoferrites [1,2], RAM and microwave/EM wave absorption, magneto-electric (ME) and multiferroic (MF) application and a multitude of other applications [1].

The formation of M-type hexagonal ferrites is an extremely complicated process, and the mechanisms involved are not fully understood

despite having been investigated by many researchers for over 50 years [1]. Various synthesis methods of M-type hexagonal ferrites were developed, including standard ceramic techniques [1,3], co-precipitation [4,5], ion exchange [6], sol-gel method [7], citrate synthesis [1,8], hydrothermal synthesis [9], glass crystallization [1,10], the combustion method [11,12], self-propagating high temperature synthesis (SHS) [1,13], spray drying [1], water-in-oil micro-emulsions [1,14] and industrial manufacture of hexagonal ferrites [1,15].

$SrFe_{12}O_{19}$ is a hard magnetic material due to its high coercivity. $SrFe_{12}O_{19}$ plays a relatively special role due to its appropriate magnetic properties, chemical stability, corrosion resistivity and cost efficiency in comparison with rare-earth compounds [1,15].

The sol–gel method has been already established as an alternative route for magnetic ferrite synthesis. This method allows the preparation of $SrFe_{12}O_{19}$ with high crystalline perfection and small particle sizes, resulting in the favorable properties for several technological applications [15].

Therefore, in this paper, strontium hexa-ferrite nanoparticles were synthesized with the Fe/Sr ratio of 12/1 by sol–gel method prior to the study regarding the effect of annealing temperature on phase formation, microstructure and magnetic properties of strontium hexa-ferrite nano powders.

2. EXPERIMENTAL

2.1. Materials

Iron (III) nitrate nonahydrate (Fe(NO3)3.9H2O (≥98%) and strontium nitrate (Sr(NO3)2) (≥99%) were used as inorganic reactants, while ethylenediamine tetraacetic acid (EDTA) (99%) was used as chelating agent, ammonium hydroxide (25 wt.%) and distilled water were used to prepare strontium hexa-ferrite nanoparticles. All starting precursors were of high purity compounds and were purchased from Xilong Chemical (China).

2.2. Preparation by sol-gel method

The starting precursors ($Fe(NO_3)_3.9H_2O$, $Sr(NO_3)_2$; Fe/Sr = 1/2) with a stoichiometric amount of metal nitrates were dissolved in deionized water under continuous stirring. The sols were prepared by dissolving the metal salts and ethylene diamine tetra acetic acid at which the molar ratio of EDTA to total metal cations were 1:1. Subsequently, ethylene glycol was added to the solution while the ratio of Fe3+ to ethylene glycol was 1:1 in moles of pure substance. pH of the solution was also adjusted to 7± 0.5 using ammonia under continuous stirring. The temperature of Fe–Sr precursor solution was maintained at 80^{o}C to obtain the desired viscosity. In the next step, drying the obtained gel at 150^{o}C from 2 to 4 hours sufficiently dehydrated the product. Finally, the product was annealed at different temperatures (700, 800, 900 and 1000^{o}C) for 2h.

2.3. Characterizations

The X-ray Diffraction (XRD) patterns of powders that prepared at various annealing temperatures were recorded by the D8 Advance Bruker system using Cu-Kα radiation (λ = 0.154056 nm) with 2θ ranging from 20 to 80o. Scanning Electron Microscopy (SEM) images were obtained using the Hitachi S-4800 which is operated at 0.5 - 30kV. A Vibrating Sample Magnetometer (VSM, Microsense EV11) was

used to measure the magnetic properties at room temperature.

3. RESULTS AND DISCUSSION

Basing on ($Fe(NO_3)_3.9H_2O$, $Sr(NO_3)_2$, EDTA, EG; $SrFe_{12}O_{19}$ was synthesized by sol-gel method. The gel was treated at different temperature from 700 to 1000°C, 2 hours in the air.

3.1. XRD analysis

Figure 1 presents the XRD patterns of SrFe12O19 calcined powders at temperatures from 700 to 1000°C. As can be seen, the distinct peaks in both XRD patterns appeared at 32.35, 34.18, 37.12, 40.38, 56.85 and 63.13° attributed to (107), (114), (203), (205), (2011) and (220) reflections for the standard pattern of M-type hexagonal SrFe12O19 crystals (JCPDS card no. 33-1340) [15]. Furthermore, as the annealing temperature increases (from 700 to 1000°C), the intensity of peaks, specially, that at 2θ = 34.18 (114), is found to increase suggesting the improvement in the degree of crystallinity of the annealed powders at further temperate.

On the other hand, annealing at 700°C was not sufficient to obtain the single phase of hexagonal crystal structure. The characteristic peaks appeared completely when the sample was annealed at 900°C 2 hours at which the crystallinity was enhanced to form single phase $SrFe_{12}O_{19}$.

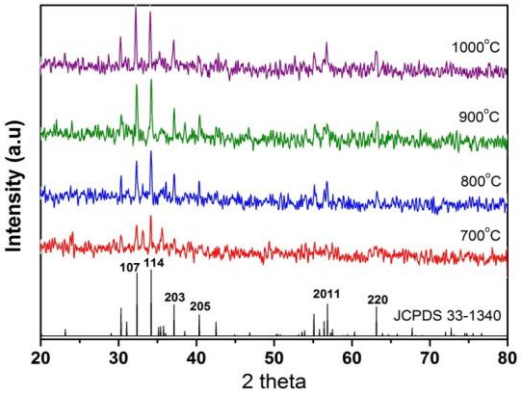

Figure 1. XRD patterns for $SrFe_{12}O_{19}$ powders for 2 hours in air

3.2. SEM results

Figure 2 presents SEM images of the prepared samples with EDTA at 900oC. It can be observed that most of the particles share hexagonal shape and the average particlesis in the proximity of 100nm. That results are also observed with sol-gel process [16].

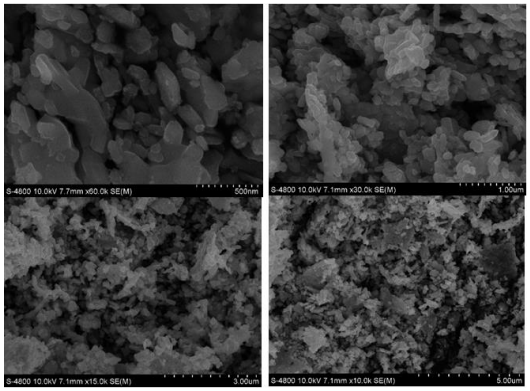

Figure 2. SEM images of $SrFe_{12}O_{19}$ powders annealed at 900°C for 2 hours in air

3.3. VSM results

Magnetization curves of the $SrFe_{12}O_{19}$ powders which was calcined at 900°C for 2 hours is shown in Figure 3. The specific saturation (Ms) and coercivity (Hc) of sample are also obtained from VSM measurement. The

specific saturation magnetization of synthesized $SrFe_{12}O_{19}$ is about 69.50 emu/g. The intrinsic coercivity of the sample is about 5696 Oe and it exhibits characteristics of single magnetic domains (Mr/Ms = 0.56), confirming the isotropic characteristic of this sample that makes $SrFe_{12}O_{19}$ a promising candidate for various applications.

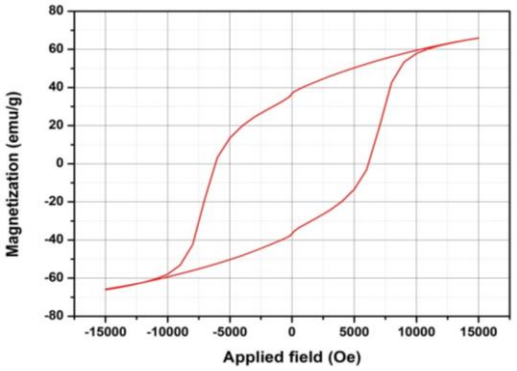

Figure 3. Magnetization curves of the $SrFe_{12}O_{19}$ powders annealed at 900°C for 2 hours

4. CONCLUSIONS

In this paper, the effect of calcination temperature (T = 700-1000°C) on structural, morphological and magnetic properties of $SrFr_{12}O_{19}$ nanoparticles which was prepared by sol–gel method have been studied in details. Although the formation of the crystal starts at 700oC, highly-structured single crystalline strontium hexa-ferrite phase was sufficiently formed at 900°C as proved by XRD results.

The SEM images of the produced ferrite present a low agglomeration rate of particles as well as the particle sizes in the proximity of 100 nm.

From the magnetization curves of the $SrFe_{12}O_{19}$ powders which calcined at 900°C, the specific saturation magnetization of 69.50 emu/g along with the instrinsic coercivity of 5696 Oe are identified.

Tổng hợp $SrFe_{12}O_{19}$ bằng phương pháp sol-gel và kết quả hình thái và tính chất từ tính của vật liệu.

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TÓM TẮT

Trong phạm vi nghiên cứu này, các hạt nano hexa-ferrite (SrFe12O19) đã được tổng hợp bằng phương pháp sol-gel. Cấu trúc tinh thể, hình thái được phân tích bằng các phép đo Nhiễu xạ tia X (XRD), Kính hiển vi quét (SEM) và tính chất từ tính của các hạt nano được đo bằng Từ kế mẫu rung (VSM). Các kết quả phổ XRD cho thấy việc hình thành đơn pha cấu trúc tính thể loại M dạng lục giác khi mẫu nung ở nhiệt độ trên 700^{o}C 2 giờ trong không khí. Mẫu cho kết quả độ từ hóa là 66 emu/g, phù hợp với các phương pháp nghiên cứu về hexa-ferrite tinh khiết. Lực kháng từ là 6,145 kOe cao hơn so với các kết quả từ các nghiên cứu trước đó về hexa-ferrite. Hình thái của các hạt nano lục giác có kích thước trung bình khoảng 100nm.

Từ khóa: *Strotium hexa-ferit, phương pháp sol-gel, vật liệu từ tính.*

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