Fabrication Of M-Type Barium Ferrite Nano-Powder With Citrate Sol-Gel Precess

Sun Chang

Abstract: Nano-powder of M-type barium ferrite ($BaFe_{12}O_{19}$) were prepared by citrate sol-gel process with the starting materials of stoichiometric amounts, such as $Ba(NO_3)_2$•$6H_2O$, $Fe(NO_3)_3$•$9H_2O$, and citric acid. The phase analysis was characterized by x-ray diffraction (XRD) . The microscopic morphology of the as prepared nano-powder was observed with transmission electron microscope (TEM). The average crystallite size of M-type barium ferrite nano-powder was found to be about 50 nm. The results show that nano-$BaFe_{12}O_{19}$ powders was obtained by citrate sol-gel process.

Index Terms: M-type barium ferrite, citrate sol-gel process, nano-powder, preparation, phase analysis, microscopic morphology, sintering.

1 INTRODUCTION

Due to their unique physical properties, M-type barium ferrites have been intensively studied as one of the most important hard magnetic materials for high density recording media, permanent magnets, drug delivery, and microwave absorbers[1-4]. For example, M-type barium ferrites BaFe12O19 possesses a high saturation magnetization and strong uniaxial anisotropy[5]; Moreover, the planar structure of hexagonal ferrites is the best structure for microwave absorber[6]. There are several preparing method to obtain high quality M-type ferrites, such as aqueous colloidal precipitation, sol–gel, solid state reaction, and hydrothermal method[7-10]. Because of well-known inherent advantages at generating glass, glass–ceramic and ceramics powders, which include homogeneous molecular mixing, low processing temperature, the ability to generate nanosized particles and the tremendous flexibility to generate nanocrystalline powders, bulk amorphous monolithic solids, and thin films, sol–gel approaches have recently received much concern[11]. In this paper, M-type barium ferrite nano-powder was synthesized by citrate sol-gel process with stoichiometric amounts of Ba(NO3)2•6H2O, Fe(NO3)3•9H2O, and citric acid as the starting materials. The phase analysis was characterized by x-ray diffraction (XRD). The microscopic morphology of the as prepared nano-powder was observed with transmission electron microscope (TEM).

2 EXPERIMENTAL DETAILS

M-type barium ferrite nano-powder $BaFe_{12}O_{19}$ was prepared by sol-gol method. In this paper, all regents, which were of analytical purity, used without further purification. $Ba(NO_3)_2$•$6H_2O$, and $Fe(NO_3)_3$•$9H_2O$ were used to incorporate metal ions needed. The synthesis procedure was as follows. A stoichiometric amount of $Ba(NO_3)_2$•$6H_2O$, and $Fe(NO_3)_3$•$9H_2O$ was dissolved in a citric acid aqueous solution under stirring. The molar ratio of nitrates to citric acid was 1:1. A homogenous transparent solution was achieved within a few minutes. An appropriate amount of ammonia hydroxide solution was added to the solution to adjust the PH value to about 7. During this process, the solution was continuously stirred using a magnetic agitator. After the precursor mixture was heated by water bath at 80℃ and stirring for 3 h the gel formed. Then the gel was put into drying cabinet at 120℃,and dried gel was got after 1-2 days. Then the dry gel was milled in a mortar The dried gel sharply burnt and give out bright flame when it was calcined at 210℃ in silicon carbide furnace in air so as to remove the organic substance. Finally, they were calcined at different temperature and the M-type barium ferrite nano-powder was obtained. Phase analysis of the as prepared powder was conducted using primarily X-ray diffraction employing a X-ray powder diffractometer (RIGAKUD/Max-A) using Cu Kαradiation(λ=1.5405). X-ray powder diffractometer was operated at 60kv and 40 mA at a 2θrange of 10-80^o employing a step size of 0.02 and a speed of 5 deg/min. The microstructure of the as synthesized powder was observed by transmission electron microscope (TEM, HITACHI-2500).

3 RESULTS AND DISCUSSION

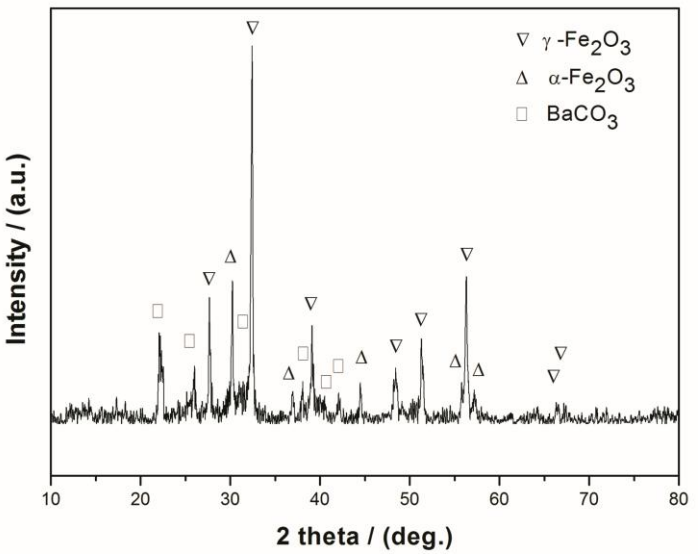

Figure 1. *XRD pattern of $BaFe_{19}O_{12}$ prepared at 600℃*

The crystalline phases of the as prepared barium ferrite power were determined by XRD. Figure 1 shows XRD pattern of the powder prepared at 600℃ for 0.5 hour. It can be seen that at 600℃, the major phase was γ-Fe_2O_3 and small amount of α-Fe_2O_3 and $BaCO_3$ phase. At 800℃, γ-Fe_2O_3 ,α-Fe_2O_3 and $BaCO_3$ phase all disappeared and single phase of hexagonal $BaFe_{12}O_{19}$ formed, which can be seen from the Figure 2.

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Moreover, Figure 2 shows the X-ray diffraction patterns for $BaFe_{12}O_{19}$ prepared for various holding time. It can be seen that there was no change in the XRD patterns except the intensity of the peaks due to $BaFe_{12}O_{19}$. Therefore, the study of the effect of the holding time on the formation $BaFe_{12}O_{19}$ revealed that a stable M-type hexagonal ferrite can be obtained at relatively short holding time by sol-gel process, while its formation required longer time by solid state reaction process from the corresponding oxide precursors. The XRD pattern of the as prepared powder prepared at 800, 1100℃ for 5h is shown in Fig. 2. The sharp and single diffraction peaks indicate homogeneity and better crystallization of the samples. The X-ray analysis implied the as prepared powder was of single phase with hexagonal structure.

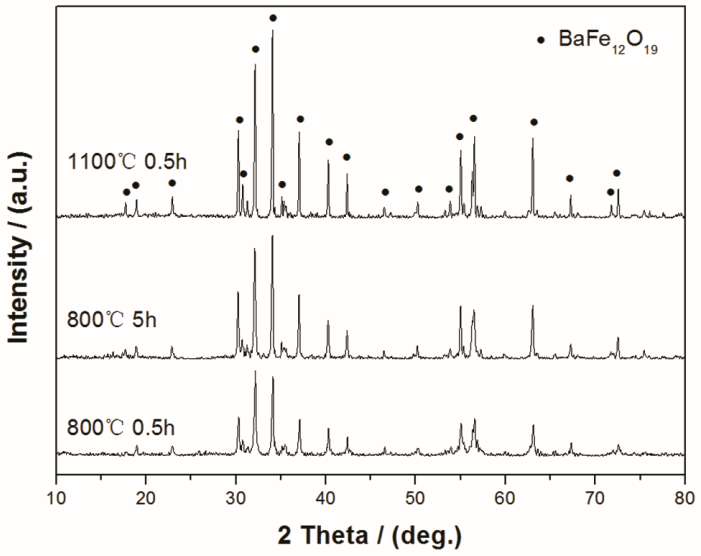

Figure 2. *XRD pattern of $BaFe_{19}O_{12}$ prepared at 800, 1100℃*

The average diameter (Dhkl) of the ferrite particles was calculated from the XRD line broadening of the (114) XRD-peaks by using Scherrer's equation:

$$D_{hkl} = \frac{0.89\lambda}{\beta_i \cos\theta} \quad (1)$$

where λ is the incident wavelength of Cu Kα radiation of the XRD, βi is the peak width at midheight and θ is the considered angle. The crystallite size of the as synthesized powders is about 40-70nm, which is consistent to the result discussed later from TEM. Transmission electron microscopy (TEM) was employed to observe the microstructure of the as prepared barium ferrite nano-powder. Figure 3 is the typical TEM image of the as–prepared sample obtained after heated- treatment at 800 ℃ for 0.5 h. From the image, the crystallites or aggregates of the smallest visible particles can be identified. It's can be seen that BaFe12O19 nanoparticles disperse on the surface of grid. The picture clearly indicated that most particles are hexagonal in structure. The average particle size is about 50 nm, which is consistent to the result from the Scherrer formula of XRD.

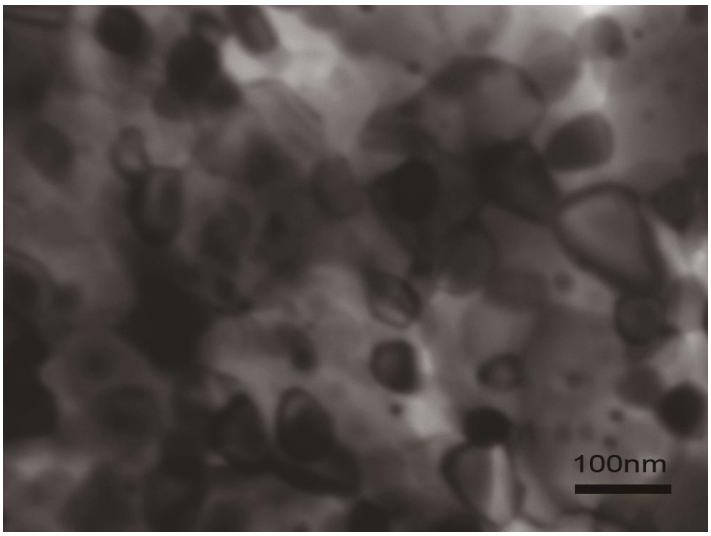

Figure 3. *TEM image of the as prepared $BaFe_{12}O_{19}$*

4 Conclusion

M-type barium ferrite nano-powder ($BaFe_{12}O_{19}$) were synthesized by citrate sol-gel process with the starting materials of stoichiometric amounts, such as $Ba(NO_3)_2$•$6H_2O$, $Fe(NO_3)_3$•$9H_2O$, and citric acid. The average crystallite size of M-type barium ferrite nano-powder was found to be about 50nm. The results show that $BaFe_{12}O_{19}$ nano-powder with the average grain size of about 50nm can be prepared at 800℃.

Acknowledgment

This work was supported by the Scientific Research Fund for Doctors of Shandong Jianzhu University (XNBS1258), China Postdoctoral Science Foundation (No. 200804401138) and Shandong Province Postdoctoral Innovation Foundation (No. 200902030).

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