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Preparation and crystallographic property of Mg- and Cu-doped barium ferrite

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Abstract

This paper presents preparation and crystallographic property of $BaFe_{12-x}Mg_{x/2}Cu_{x/2}O_{19}$ ferrite magnetic compound where x varies from 0 to 2 in steps of 0.5 synthesized by sol-gel method. For preparation, the reactants are firstly mixed together by magnetic stirrer, after which an adjustment is made of appropriate pH value and homogenous solution with ammonia and citric acid, respectively. Next, provide heated at 80 ^{o}C for 1 day. Precursors are gel after that eliminate moisture by evaporation at 150 ^{o}C for 1.5 hrs and anneal at 800 ^{o}C for 3 hrs. Finally, the compound is powder. For characterization, the crystalline structure, particle sizes and shapes of these compounds were investigated by X-ray diffraction (XRD) and scanning by electron microscope (SEM). The results show that all samples are magnetic and dielectric materials. The structure of $BaFe_{12}O_{19}$ and $BaFe_{12-x}Mg_{x/2}Cu_{x/2}O_{19}$ are hexagonal. In addition, its average grain size is 120-250 nm.

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Keywords: barium ferrite; hexagonal ferrite; sol-gel

1. Introduction

Ferrite compounds are used in several technologies and applications such as microwave absorber [1, 2], magnetic recording [3-6], cores of inductors, motor [7], transformers, electromagnets and electronics devices. Recently, many research groups have reported the characteristics of doped ferrite compounds e.g. (i) electromagnetic properties and microwave absorbing characteristics of Mn, Co and Co, Ti doped strontium ferrites [8], Mn, Cu and Ti doped barium hexaferrite [9], Mg and Ti doped barium hexaferrite [10], Mn and Ti doped strontium hexaferrite [11], Mn, Co and Sn doped barium hexaferrite [12]; (ii) static and dynamic magnetic characteristics of Mn, Co and Ti doped barium hexaferrite [13], Mn, Co and Zr doped barium hexaferrite [14]; (iii) structural analysis of Ni doped cobalt ferrite [15], Mn, Co and Zr doped strontium hexaferrite [16].

For the synthesis, ferrite compounds can be prepared in several methods. The popular methods are solid-state method [8, 10, 11], ceramics sintering method [1, 9] and sol-gel method [12, 14, 16, 17]. Ceramics sintering

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method is easy process but it takes a long time and high annealing temperature. Solid-state method is quite complex process, many devices, long time and high annealing temperature. On the other hand, sol-gel method is process which changed fluid (sol) is suspendible solution of which average size is 0.1-1 μm to solid (gel). It is easy process, appropriate time, preparation at room temperature and atmospheric pressure and medium annealing temperature.

From above reasons, this paper presents the preparation and crystallographic property of Mg and Cu doped barium ferrite ($BaFe_{12-x}Mg_{x/2}Cu_{x/2}O_{19}$) where 0 to 2 in step of 0.5 was prepared by sol-gel method. After that, gel was eliminated moisture and annealed at appropriate temperature. The crystallographic property is analyzed by X-ray diffraction (XRD) and the grain size and crystal shape is evaluated by scanning electron microscope (SEM).

2. Experiments

$BaFe_{12-x}Mg_{x/2}Cu_{x/2}O_{19}$ ferrite compound where x varies from 0 to 2 in step of 0.5 were prepared by sol-gel method. The X-ray diffraction (XRD) was used to analyze crystallographic property and scanning electron microscope (SEM) was used to evaluated grain size and shape.

2.1 The preparation of composite

The $BaFe_{12-x}Mg_{x/2}Cu_{x/2}O_{19}$ ferrite was prepared by sol-gel method as shown in Fig. 1. The starting materials were $Fe(NO_3)_3.9H_2O$ for Fe, $Ba(NO_3)_2$ for Ba, $Cu(NO_3)_2.6H_2O$ for Cu and $Mg(NO_3)_2.6H_2O$ for Mg**.** Firstly, the reactants were mixed together by magnetic stirrer after that adjusted pH value around 1.0 and homogenous solution with ammonia and citric acid, respectively. Next, heat at 80 ^oC for 1 day. Precursors were gel after that eliminated moisture by evaporation at 150 ^oC for 1.5 hrs and annealed at 800 ^oC for 3 hrs. Finally, the compound was powder which was the magnetic and dielectric material. Samples were named according with proportion of magnesium and copper present in $BaFe_{12-x}Mg_{x/2}Cu_{x/2}O_{19}$: $BaFe_{12}O_{19}$ (A) was not annealed and $BaFe_{12}O_{19}$ (B), $BaFe_{11.5}Mg_{0.25}Cu_{0.25}O_{19}$ (C), $BaFe_{11}Mg_{0.5}Cu_{0.5}O_{19}$ (D), $BaFe_{10.5}Mg_{0.75}Cu_{0.75}O_{19}$ (E) and $BaFe_{10}MgCuO_{19}$ (F) were annealed at 800 ^oC.

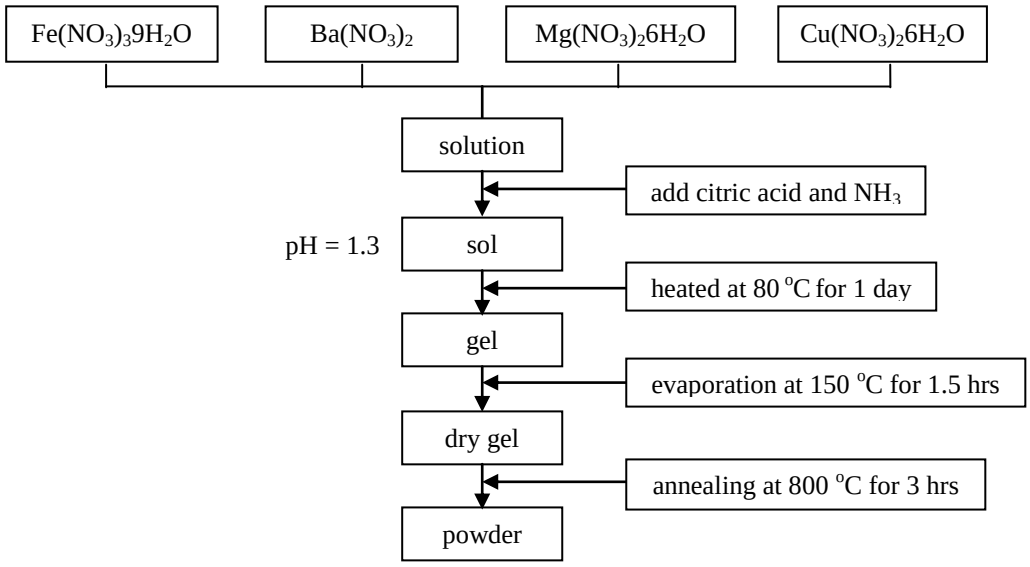

Fig. 1. Sol-gel method for preparation of $BaFe_{12-x}Mg_{x/2}Cu_{x/2}O_{19}$; x = 0-2

2.2 The analysis crystallographic property and evaluation grain size and shape

The X-ray diffraction (XRD) was used to analyze crystallographic property, using a CuKα radiation detector (λ = 0.15408981 nm). The ferrite powder was scanned through the 2θ angle range of 20°-60° and scanning rate of 0.5°/min. Scanning electron microscope (SEM) was used to evaluated grain size and shape, using a Philips XL 1000, 15000 and 30000. Using sputtering technique, specimens were sprinkled on carbon tape substrate and coated by an Au film before observation in SEM.

3. Results and discussions

3.1 Crystallographic property

Fig. 2 shows XRD pattern of undoped and doped ferrites annealed at 800°C for 3 hrs. Ferrite "A" was undoped barium ferrite and was not annealed. It is amorphous due to coordination of intensity. Ferrite "B" was doped barium ferrite and was annealed at 800°C. It is crystal because it has peak of intensity at 107 and 114 miller indices as ferrite "C", "D", "E" and "F" that were doped barium ferrite and was annealed at 800°C. The distance between the planes of atoms in the crystal for (*hkl*) plane, d_{hkl}, the lattice parameters a and c are calculated by eq. (1) and (2) [18], respectively. The average crystallite size in nanometer, $<D_{hkl}>$ is evaluated by eq. (3). The unit cell volume, V_{cell} is computed by eq. (4).

$$2d_{hkl}\sin\theta = n\lambda \tag{1}$$

$$\frac{1}{d_{hkl}^2} = 4\left(\frac{h^2+hk+k^2}{3a^2}\right)+\frac{l^2}{c^2} \tag{2}$$

$$< D_{hkl} >= \frac{K\lambda}{\beta\cos\theta} \tag{3}$$

$$V_{cell} = 0.8666a^2c \tag{4}$$

where h, k and l are miller indices, K is scherrer constant in the range of 0.87–1 (K = 0.89) [19], λ is wavelength of X-ray in nanometer, θ is diffraction angle in degree and β is full width at half maximum in radian. By using four equations, the structural parameters are shown in Table 1. The most, structural parameter "a" increases when parameter "c" decreases from 2.1995 to 2.2704 nm. The c/a is almost the same. This is in agreement with the fact that a/c of all hexagonal types is constant [20]. The V_{cell} is in range from 0.6078 to 0.7022 nm^3.

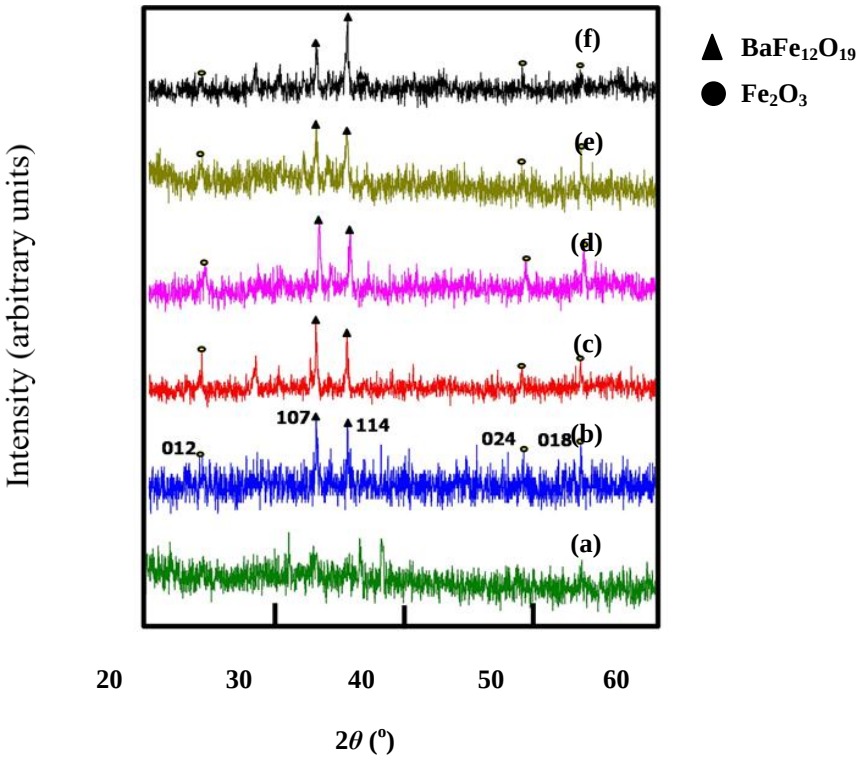

Fig. 2. XRD patterns of doped barium ferrites: (a) ferrite ''A'', (b) ferrite ''B'', (c) ferrite ''C'', (d) ferrite ''D'', (e) ferrite ''E'' and (f) ferrite ''F''

Table 1. Lattice parameters, *c/a* and unit cell volume for undoped and doped barium ferrite were annealed at 800 °C

x	d_{107} (nm)	d_{114} (nm)	$\langle D_{hkl} \rangle$ (nm)	*a* (nm)	*c* (nm)	*c/a*	V_{cell} (nm^3)
0	0.2698	0.2516	108.1641	0.5614	2.2704	4.0441	0.6200
0.5	0.2698	0.2519	341.6128	0.5623	2.2686	4.0345	0.6216
1.0	0.2678	0.2499	113.9484	0.5577	2.2525	4.0389	0.6078
1.5	0.2697	0.2523	115.4811	0.5636	2.2651	4.0190	0.6236
2.0	0.2697	0.2657	59.9000	0.6070	2.1995	3.6236	0.7022

3.2 Microstructure

Fig. 3 shows the microstructure and crystal shape of ferrite powders. Grain size and shape of ferrite "A" are random and it is also amorphous. The grains of ferrite "B", "C", "D", "E" and "F" are hexagonal structure with grain sizes of about 200-250 nm.

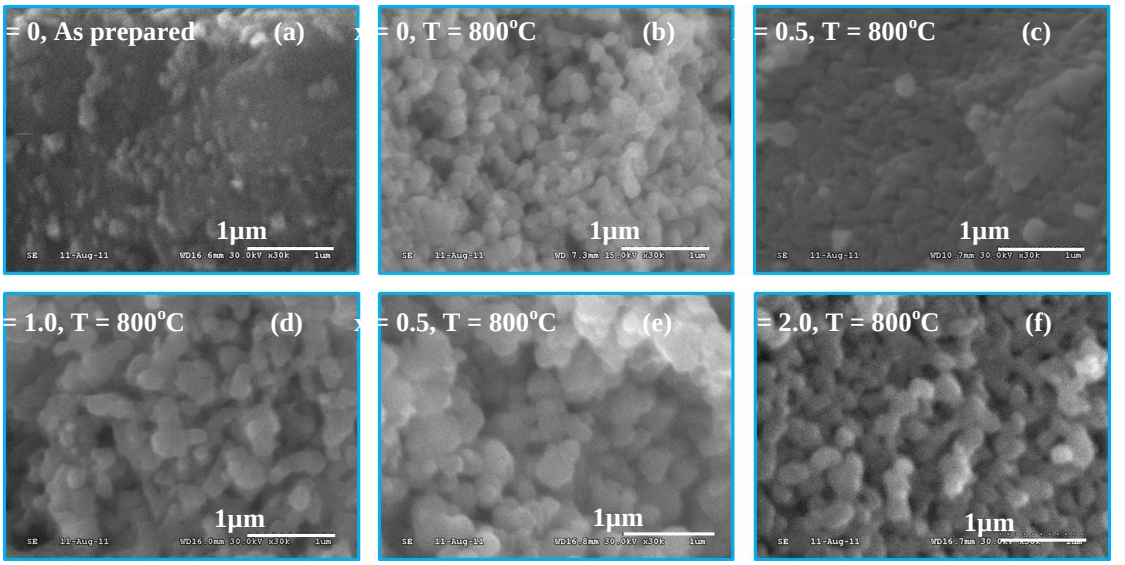

Fig. 3. Scanning electron microscopy image of doped barium ferrites: (a) ferrite ‘‘A’’, (b) ferrite ‘‘B’’, (c) ferrite ‘‘C’’, (d) ferrite ‘‘D’’, (e) ferrite ‘‘E’’ and (f) ferrite ‘‘F’’

4. Conclusions

$BaFe_{12-x}Mg_{x/2}Cu_{x/2}O_{19}$ ferrite compound where x varies from 0 to 2 in steps of 0.5 were prepared by sol-gel method. $BaFe_{12}O_{19}$ (x = 0) was not annealed, it is nonmagnetic material, amorphous and crystal shape are random. $BaFe_{12}O_{19}$ (x = 0), $BaFe_{11.5}Mg_{0.25}Cu_{0.25}O_{19}$ (x = 0.5), $BaFe_{11}Mg_{0.5}Cu_{0.5}O_{19}$ (x = 1), $BaFe_{10.5}Mg_{0.75}Cu_{0.75}O_{19}$ (x = 1.5) and $BaFe_{10}MgCuO_{19}$ (x = 2) were annealed at 800°C for 3 hrs. They are magnetic material and the grain shape is hexagonal structure with grain sizes of about 200-250 nm.

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