



Microstructure and magnetic properties of Y substituted M-type hexaferrite $\text{BaY}_x\text{Fe}_{12-x}\text{O}_{19}$

Yanjun Chen¹ · Yu Wang¹ · Yingli Liu¹ · Qian Liu¹ · Chongsheng Wu¹ · Binghao Qi¹ · Huaiwu Zhang¹

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Abstract

Y (yttrium)-substituted M-type hexaferrite of composition $\text{BaY}_x\text{Fe}_{12-x}\text{O}_{19}$ (x ranges from 0.1 to 0.8 with step of 0.1) was synthesized through ceramic process. Pure phase formation of M-type hexaferrite was confirmed by XRD patterns for prepared sample when $x < 0.4$. The second phase ($\text{Y}_3\text{Fe}_5\text{O}_{12}$) was detected when the substitution content exceeded 0.4. As the substitution dosage of yttrium increased, un conspicuous transform were observed in the microstructure of all samples $\text{BaFe}_{12-x}\text{Y}_x\text{O}_{19}$, such as compactness, size of grain. The bulk densities (ρ) of the samples decreased first and then increased with the increase of x . It means that the compactness of the sample receded first and then enhanced with rise of Y^{3+} substitution contents. Moreover, with increase of Y^{3+} amount, Y^{3+} substituted the Fe^{3+} in the octahedral position of m-type barium ferrite, the saturation magnetization (M_s) of the sample decreases rapidly and obtained a minimum of 55.63 emu/g, and then increased gradually attributed to the appearance of the second phase ($\text{Y}_3\text{Fe}_5\text{O}_{12}$). Appropriate values of M_s (56.57 emu/g) and H_c (1794.17 O_e) were obtained simultaneously when $x = 0.6$. These two characters made the material suitable for the applications infield of magnetic recording.

1 Introduction

M-type barium ferrite has been receiving continuous attention, and the researchers did a lot of research on it, since its discovery in the 1950s [1]. M-type barium ferrite ($\text{BaFe}_{12}\text{O}_{19}$) is an excellent permanent magnetic material with high coercive force and residual magnetization, good

temperature stability and corrosion resistance [2]. M-type barium ferrite has been proposed for applications in permanent magnets, self-biased microwave and millimeter wave devices (isolators, phase shifters and circulators) [3, 4]. However, with the development of electronic information technology, the traditional materials like M-type barium ferrite was also faced with the application in new field, such as magnetic recording. The magnetic head material in the field of magnetic recording require materials with high saturation magnetization and low coercive force, but pure m-type barium ferrite is difficult to meet the requirements. The M-type barium ferrite has high saturation magnetization, so the reduction of coercive force on the premise of ensuring the saturation magnetization is a direct method to make it more applied in the field of magnetic recording. Quantities investigations have concentrated on reducing the coercive force of M-type barium ferrite [5, 6]. Ion substitution is an effective technique to adjust the properties of M-type barium ferrite [7, 8]. Ion substitution is mainly through substituting ions to adjusting the crystal structure or magnetic structure of M-type barium ferrite, simultaneously the magnetic properties, electrical properties and microwave properties changed. Similarly, the density, grain size and porosity of doped M-type barium ferrite samples also have an important effect on the properties. So far, in the method of ion

✉ Yingli Liu
lyl@uestc.edu.cn

YanJun Chen
chenyanjun085@sina.com

Yu Wang
wangyu_dzkjdx@yahoo.com

Qian Liu
liuqianforxm@xina.com

Chongsheng Wu
wuchongsheng@sina.cn

Binghao Qi
2389626858@qq.com

Huaiwu Zhang
hwzhang@uestc.edu.cn

¹ State Key Laboratory of Electronic Thin Films and Integrated Devices, University of Electronic Science and Technology of China, Chengdu 610054, Sichuan, China

substituted the M type barium ferrite, the main method is to replace Fe^{3+} or Ba^{2+} with other ions, it mainly includes two methods of co-substitution [5, 9, 10] and single-ion substitution. The co-substitution method means the Fe^{3+} will be substituted by a divalent ion and a tetravalent ion. It has been proved in the past that the properties of M-type barium ferrite can be obviously changed by replacing the Fe^{3+} with trivalent nonmagnetic ions (Al^{3+} , Sc^{3+} , Ga^{3+} , etc.) [11–17].

In this paper, the substitution of Fe^{3+} with Y^{3+} in M-type barium ferrite has been studied. Yttrium is a rare earth element in the periodic table 39, and Y^{3+} is a nonmagnetic ion. The ionic radius of Y^{3+} (0.9 Å) is larger than that of Fe^{3+} (0.64 Å). Therefore, the single ion (Y^{3+}) substitution of M-type barium ferrite not only changed the magnetic structure of M-type barium ferrite, but also caused obvious distortion of lattice structure. Of course, because the radius of Y^{3+} is larger than that of Fe^{3+} , it is not easy for Y^{3+} to occupy the tetrahedral and hexahedral positions of M-type barium ferrite. Excessive yttrium ions which cannot enter the lattice of M-type barium ferrite, form the second phase ($\text{Y}_3\text{Fe}_5\text{O}_{12}$) with iron ions and oxygen atoms. The saturation magnetization, coercive force and bulk densities of the samples had obvious regular changed. This paper explored the influence of magnetic ion Yttrium Ion (Y^{3+}) substitution on the magnetic properties of M type barium ferrite, including the influence on magnetic structure, and coercive force.

2 Experiment

M-type barium ferrite substituted with Y for Fe, $\text{BaFe}_{12-x}\text{Y}_x\text{O}_{19}$ were synthesized using analytical grade BaCO_3 , Fe_2O_3 , Y_2O_3 via solid-state reactions. The X value was varied as X=0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8. To weigh the powdered raw material according to the chemical formula. The powders were mixed using a ball mill for 12 h in a steel vial with zirconia balls and deionized water as the milling media. The mixed powder was dried and then calcined at 1300 °C for 12 h in air. The calcined powder was milled again for 12 h in deionized water. After drying, the powder was granulated by adding 8 wt% of polyvinyl alcohol (PVA) as a binder and pressed into 2- to 3-mm thick plates or rings. The compacted samples were sintered at 1150 °C for 12 h.

The phase compositions of the samples determined using the X-ray diffractometer (XRD, DX-2700, Haoyuan Co) with Cu K α radiation. The bulk density of samples was measured by Archimedes method. The microstructures of the samples were presented using a scanning electron microscope (SEM, JEOL, JSM-6490). The basic magnetic properties were characterized using a vibrating sample magnetometer (VSM, MODEL, BHL-525).

3 Results and discussion

Figure 1 showed X-ray diffraction patterns for $\text{BaFe}_{12-x}\text{Y}_x\text{O}_{19}$ solid solutions with x ranging from 0.1 to 0.8. All the diffraction peaks were indexed to be the M-type barium ferrite phase (space group P63/mmc, JCPDS file number 43-002), demonstrating a single phase structure with X=0.1 to 0.3. The heterogeneity peak of second phase ($\text{Y}_3\text{Fe}_5\text{O}_{12}$) has been observed at X=0.4. The XRD patterns of the $\text{BaFe}_{12-x}\text{Y}_x\text{O}_{19}$ (X=0.1, 0.2, 0.3) matches well with the standard card of M-type barium ferrite at room temperature (300 K). It showed that when the substitution amount of Y^{3+} is small (X=0.1, 0.2, 0.3), the Y^{3+} enters the lattice of M-type barium ferrite.

The second phase ($\text{Y}_3\text{Fe}_5\text{O}_{12}$) was found in all samples (X=0.4–0.8), and the diffraction peak of the second phase became more and more obvious with increased of Y^{3+} substitution contents (X=0.4 to 0.8). The limiting substitutions amount that yttrium ions can enter into a lattice of M-type barium ferrite may be between x=0.3 and 0.4 without producing the second phase ($\text{Y}_3\text{Fe}_5\text{O}_{12}$).

XRD analysis and Rietveld refinement were performed on all the samples to estimate the microstructure parameters

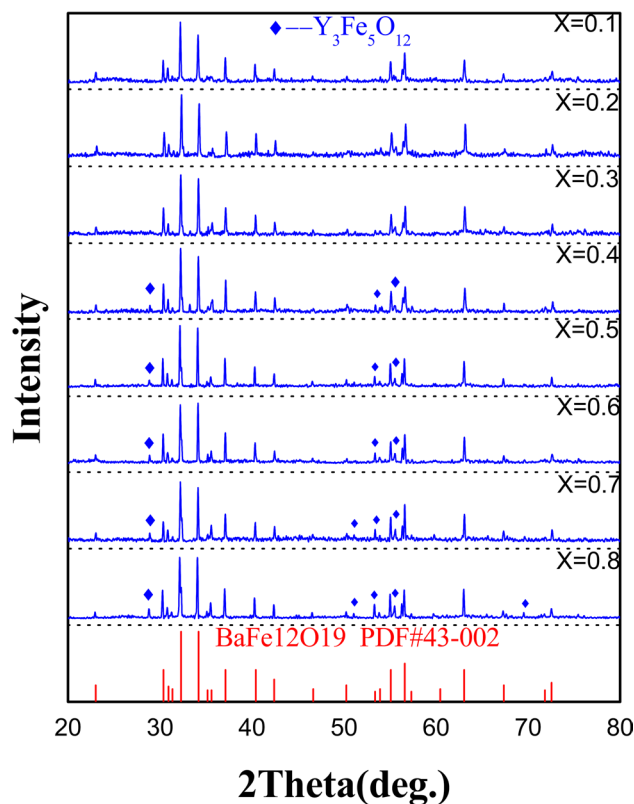


Fig. 1 X-ray diffraction patterns for $\text{BaFe}_{12-x}\text{Y}_x\text{O}_{19}$ solid solutions (X=0.1–0.8)

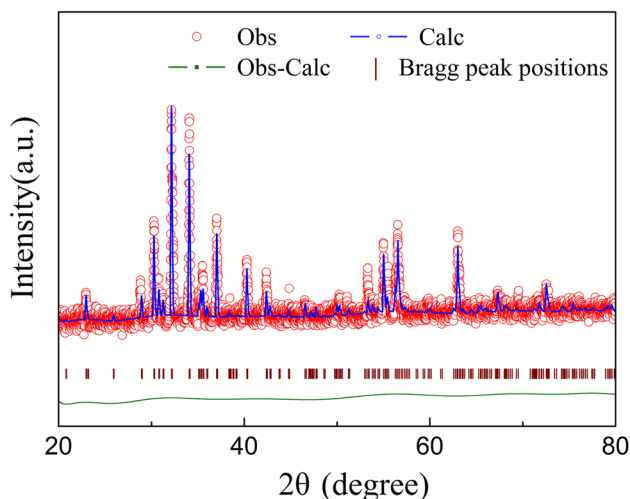


Fig. 2 The XRD pattern of the sample (X=0.8) was fitted by Rietveld

of the system. Figure 2 describes the experimental X-ray diffraction data of the sample (X = 0.8) and its corresponding Rietveld refinement mode. Table 1 shows the finishing parameters and lattice constants of each sample.

It was seen that with the increase of X, the unit-cell parameters *a* increases first, then decreases slowly, unit-cell parameters *c* increases steadily. This is due to the influence of the ionic radius. Y^{3+} is a nonmagnetic ion, the ionic radius of Y^{3+} (0.9 Å) is larger than that of Fe^{3+} (0.64 Å). When the substitution amount is too large ($X \geq 0.4$), some Y^{3+} unable to enter the crystal lattice of M-type barium ferrite, thus the second phase ($Y_3Fe_5O_{12}$) was produced and the unit-cell parameters *a* was reduced to a certain extent.

The bulk densities of all samples were measured by Archimedes and shown in Table 2. It can be clearly seen from the figure that with the increase of the substitution contents of yttrium ions (Y^{3+}), the bulk densities of the samples first decreases and then increases.

The reason for the decrease of $BaFe_{12-x}Y_xO_{19}$ (X = 0.1 to 0.3) bulk densities is that the small amount of large yttrium

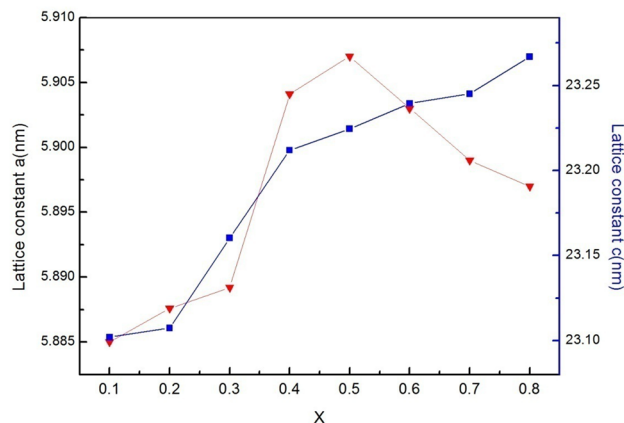


Fig. 3 The curves of the lattice constants *a* and *c* of the $BaFe_{12-x}Y_xO_{19}$ with X

ions (Y^{3+}) substituted iron ions (Fe^{3+}), which increases the volume of the crystal lattice. With the rise of the substitution amount of yttrium ions (X = 0.4 to 0.8), the second phase ($Y_3Fe_5O_{12}$) appeared in samples. Since the second phases ($Y_3Fe_5O_{12}$) has a denser structure than M-type barium ferrites, the bulk densities of the samples had limited reduced as shown in Fig. 3.

The SEM results were shown in Fig. 4. Hexagonal grain structure was obviously found in the SEM image of $BaY_xFe_{12-x}O_{19}$. From the SEM images, the density of the samples did not change significantly with the increase of the substitution contents of yttrium ions (Y^{3+}). This is consistent with the tiny change in bulk density. In addition, with the increase of substitution contents of yttrium ions (Y^{3+}), the grain size of the sample was increased. Moreover, the growth of grain size has a positive effect on reducing the coercive force of m-type barium ferrite as shown in Fig. 4.

Figure 5 shows the variation of the specific magnetization of the $BaY_xFe_{12-x}O_{19}$ with the external magnetic field. Saturation magnetization (*M_s*) and coercive force (*H_c*) extracted from the loops are given in Fig. 6 as functions of the x value. The maximum saturation magnetization of the sample had

Table 1 The crystal corresponds to the lattice parameters and Rietveld parameters of different quantities of cationic samples

X	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8
<i>A</i> (Å)	5.885	5.891	5.9001	5.907	5.910	5.904	5.902	5.899
<i>C</i> (Å)	23.1019	23.1073	23.1104	23.1219	23.1844	23.2213	23.2401	23.2499
<i>R_{wp}</i> , %	1.87	1.71	1.65	1.56	1.77	1.63	1.81	1.84
<i>R_{exp}</i> , %	1.48	1.48	1.42	1.44	1.22	1.38	1.36	1.47

Table 2 The variations of bulk density (ρ) as a function of Y substitution contents

X	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8
ρ (g/cm ³)	4.406	4.24	4.097	4.195	4.105	4.155	4.164	4.183

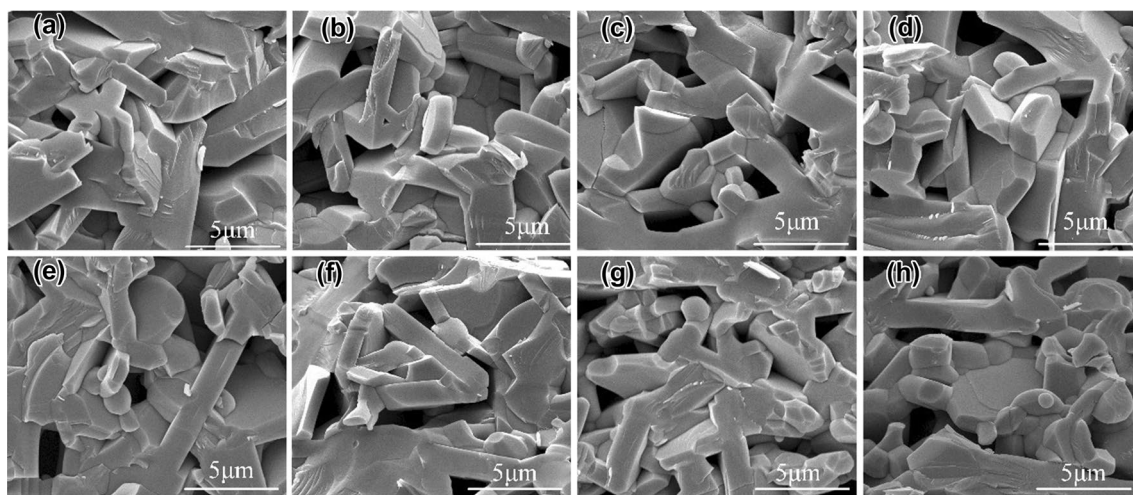


Fig. 4 SEM micrographs of sintered $\text{BaFe}_{12-x}\text{Y}_x\text{O}_{19}$ samples with different Y substitution contents: (a) $x=0.1$, (b) $x=0.2$, (c) $x=0.3$, (d) $x=0.4$, (e) $x=0.5$, (f) $x=0.6$, (g) $x=0.7$, (h) $x=0.8$

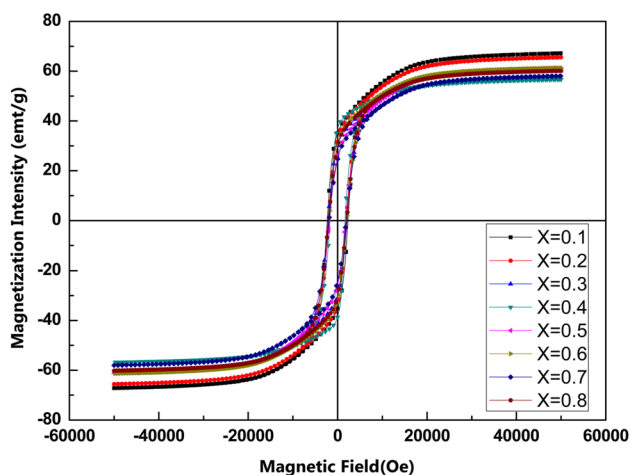


Fig. 5 Field dependence of the specific magnetization at $T = 300$ K for the $\text{BaFe}_{12-x}\text{Y}_x\text{O}_{19}$

obtained from the Fig. 6 at room temperature (300 K) was 67.19 emu/g at $x=0.1$. Figure 5 showed that before the point of $X=0.4$, the saturation magnetization decreases monotonously with the increase of substitution amount of Y^{3+} . This is also consistent with the results of other nonmagnetic ions substitution [12, 16]. But, after $X=0.4$, due to the formation of the second phase ($\text{Y}_3\text{Fe}_5\text{O}_{12}$), non-magnetic yttrium ions no longer occupy the iron position of M-type barium ferrite, so the total magnetic moment of the sample is recovered the saturation magnetization of the sample increases slowly.

The factors affecting the magnetic properties of ferrite include chemical composition, ion distribution in crystallographic sites and bulk densities [10]. In M-type barium ferrite, each unit cell contains two molecules ($\text{BaFe}_{12}\text{O}_{19}$),

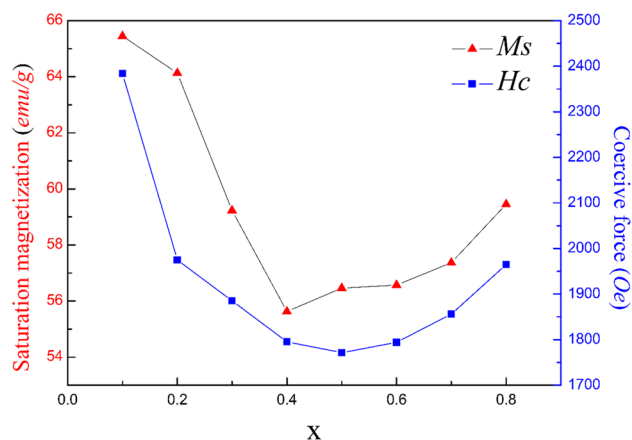


Fig. 6 The curve of saturation magnetization (M_s) and coercive force (H_c) with X value

and there are ten oxygen ion dense layers, of which two dense layers each contain one barium ion occupying oxygen potential. In M-type barium ferrite the magnetic Fe^{3+} ions are located in positions which have three octahedral site (Fe_{1-2a} , Fe_{4-4f_2} and Fe_{5-12k}), one tetrahedral (Fe_{3-4f_1}) and one bipyramidal (Fe_{2-2b}) oxygen environment [19]. The Fe^{3+} at 2a, 12k and 2b sites have upward spin configurations, while those at $4f_1$ and $4f_2$ have downward spin configurations. The iron ions in M-type barium ferrite are all trivalent iron ions, and their ion magnetic moment is $5\mu_B$. M-type barium ferrite, given that the molar ratio of Fe^{3+} ions in different sites is $\text{Fe}(12k):\text{Fe}(2a):\text{Fe}(2b):\text{Fe}(4f_1):\text{Fe}(4f_2)=6:1:1:2:2$ [18, 19], the total magnetic moment (m) of M-type barium ferrite can be computed as: $m = (6 + 1 + 1 - 2 - 2) \times 5\mu_B = 20\mu_B$. When the Y^{3+} substituted the Fe^{3+} , the total magnetic

moment and saturation magnetization of M-type barium ferrite will be affected by the different ionic magnetic moment of Y^{3+} and Fe^{3+} .

According to Ligand theory, ions site occupancy depends on d-configuration of electrons and the nature of other participating ions. Ions with d^1 , d^2 , d^3 and d^4 electrons prefer tetrahedral site, ions with d^6 , d^7 , d^8 and d^9 electrons prefer octahedral site, and ions with d^0 , d^5 and d^{10} have no preference [4]. Y^{3+} have the electronic structure of $4p^6$, so they tend to be octahedral in substitution. Fe^{3+} located at octahedral position in M-type barium ferrite and with upward spin (2a, 12 k) and downward spin ($4f_2$) configurations. According to the change curve of magnetization with external magnetic field, saturation magnetization decreases rapidly from 67.1872 emu/g to 56.7128 emu/g with X ranging from 0.1 to 0.4, indicating that Y^{3+} should be inclined to substituted 2b (\uparrow), 2a (\uparrow) and 12 k (\uparrow) sites. From the point of view of ionic electronegativity, Ions of high electronegativity have higher tendencies for octahedral sites. Yttrium Ion has a higher electronegativity (1.22) and tends to occupy octahedral position when replacing iron ion. Its seem that for Y substitution Y^{3+} ions first preferentially occupy the 12 k(\uparrow) and 2a (\uparrow). But, because of the ionic radius, when the Y^{3+} substitution reached to higher level, a second phase ($Y_3Fe_5O_{12}$) was produced.

As described in Stoner–wohlfarth's theory, the coercive force (H_C) of ferrite can be determined by saturation magnetization (M_S) and magnetocrystalline anisotropy constant (K_1) by the following equation [20]:

$$H_C = 0.64K_1/M_S$$

M_S and H_C are determined from VSM measurements. According to the law of approaching saturation, the magnetocrystalline anisotropy constant is calculated:

$$M_H = M_S(1 - a/H - b/H^2 - \dots) + \chi_p H$$

Where parameter a represents the crystal local inhomogeneous stress and inhomogeneity of doped region, χ_p is the high field paramagnetic susceptibility, and parameter b originates from the resistance of magnetocrystalline anisotropy on domain wall rotation [21], which can be expressed for the hexagonal crystal structure exhibiting uniaxial anisotropy as:

$$b = 4K_1^2/15\mu_0^2M_S^2$$

K_1 can be calculated by determining the value of parameter b. In the case of high H field region, the parameters a and χ_p are small enough to be negligible, M_H exhibited a linear tendency as a function of H^2 , which makes it possible to calculate the value of parameter b. Calculated K_1 versus increased Y substitution contents was present in Table 3.

Coercive force represents the additional magnetic field required to make M equal to 0 in the reverse magnetization

Table 3 The Variations of the magnetocrystalline anisotropy constant (K_1) and the coercive force (H_C) versus different Y substitution contents (x)

x	K_1 ($\times 10^5$ J/m ³)	M_S (emu/g)	H_C (Oe)
0.1	2.68	65.45	2383.96448
0.2	2.63	64.13	1974.75256
0.3	2.36	59.23	1885.02881
0.4	2.25	55.63	1795.32611
0.5	2.38	56.46	1771.44586
0.6	2.47	56.57	1794.17218
0.7	2.56	57.37	1856.13062
0.8	2.45	59.45	1964.62152

process. The irreversible magnetization is the cause of the hysteresis in the magnetization process. The reverse magnetization process, like the magnetization process, can also occur reversible and irreversible magnetization process. Therefore, the fundamental reason for the formation of hysteresis in the reverse magnetization process is the irreversible magnetization process caused by stress bullying, impurities and magnetic anisotropy in the ferromagnetic material. Yttrium ions (Y^{3+}) substituted the iron ions (Fe^{3+}) in M-type barium ferrite and changed the Magnetic crystal anisotropy and causes the coercive force changed. It can be found from Table 2 that the variation trend of coercive force is consistent with the variation rule of the anisotropic constant of magnetic crystals. When the grain size of the sample increases, the number of grain boundaries in the sample is reduced, the internal stress is fully released, and the coercive force is also reduced. Therefore, when the substitution amount of yttrium ions exceeded $x = 0.4$, the second phase ($Y_3Fe_5O_{12}$) was generated in samples, which increases the number of grain boundaries to a certain extent, increases the stress in the sample and increases the coercive force.

4 Conclusions

In this work, we have investigated the effects of Y substitution on the structure and magnetic properties of M-type barium ferrite. The new m-type barium ferrite ($BaFe_{12-x}Y_xO_{19}$) was synthesized by solid state reaction and sintering temperature of 1150 °C. It is found that single ion yttrium can adjust the magnetic anisotropic field of M-type barium ferrite and reduce the saturation magnetization intensity and coercive force of M-type barium ferrite ($T = 300$ k). Due to the large ionic radius of Y^{3+} , some yttrium ions cannot enter the lattice point of m-type barium ferrite lattice when x reached to a high value, thus forming the second phase ($Y_3Fe_5O_{12}$). According to the experimental results, it is suggested that the substitution content of Y^{3+} in the synthesis process should

be between $X=0.3$ and 0.4 . The appearance of the second phase will cause the saturation magnetization strength and coercive force of the original sharply decreasing samples to rise slightly. By adjusting the substitution content of Y^{3+} , the ratio of high saturation magnetization intensity and low coercive force can be found and applied in magnetic head materials.

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