

Effects of Pr-Co content on the microstructural and magnetic properties of the Pr-Co substituted M-type strontium hexaferrites

Yujie Yang*, Juxiang Shao, Fanhou Wang and Duohui Huang

Computational Physics Key Laboratory of Sichuan Province, School of Physics and Electronic Engineering, Yibin University, Yibin 644007, P. R. China

Pr-Co substituted M-type strontium hexaferrites with the chemical composition of $\text{Sr}_{1-x}\text{Pr}_x\text{Fe}_{12-y}\text{Co}_y\text{O}_{19}$ ($x = 0.00-0.50$; $y = 0.00-0.35$) were prepared by the solid-state reaction method. Microstructures of the M-type hexaferrite samples were investigated by the X-ray diffraction (XRD) and a field emission scanning electron microscopy (FE-SEM). The single-phase with hexagonal structure is obtained in the M-type hexaferrites with Pr-Co content ($0.00 \leq x \leq 0.20$, $0.00 \leq y \leq 0.14$). For the M-type hexaferrites with Pr-Co content ($x \geq 0.20$, $y \geq 0.14$), impurity phases are observed in the structure. The image of the representative magnet exhibits that the M-type hexagonal structure is formed in the magnet. A permanent magnetic measuring system was used to measure the magnetic properties of the M-type hexaferrite magnets. The remanence (B_r), maximum energy product $[(BH)_{\max}]$ and H_k/H_{cj} ratios increase with the increasing substitution content of Pr ($0.00 \leq x \leq 0.10$) and Co ($0.00 \leq y \leq 0.07$), and then decrease with increasing substitution content of Pr ($x \geq 0.10$) and Co ($y \geq 0.07$). The intrinsic coercivity (H_{cj}) and magnetic induction coercivity (H_{cb}) decrease with increasing Pr-Co content ($0.00 \leq x \leq 0.30$, $0.00 \leq y \leq 0.21$), and then increase with increasing substitution content of Pr ($x \geq 0.30$) and Co ($y \geq 0.21$).

Key words: M-type hexaferrite, Pr-Co substitution, X-ray diffraction, Magnetic properties.

Introduction

M-type hexaferrites have been widely investigated and used as permanent magnets due to their high intrinsic coercivity, high magnetization, high stability, high density and low price for production [1]. In the M-type hexaferrites crystals, the Fe^{3+} ions are situated in one tetrahedral site ($4f_1$), three octahedral ($2a$, $12k$ and $4f_2$) and one bipyramidal site ($2b$). In the magnetically ordered state of M-type hexaferrites, $2a$, $2b$ and $12k$ are spin up sites, whereas $4f_1$ and $4f_2$ are spin down sites [2].

There are many ways to modify the magnetic properties of M-type hexaferrites, such as preparation methods, variation of preparation parameters, ion substitution technique [3]. Various preparation methods like the solid-state reaction method [4], hydrothermal method [5], sol-gel method [6], citrate precursor method [7], sol-gel auto-combustion method [8], and chemical coprecipitation method [9] have been used to synthesize the M-type hexaferrites. The magnetic properties of M-type hexaferrites can be influenced by the partial substitution for Sr^{2+} or Ba^{2+} ions or Fe^{3+} ions, or both. The improvement of magnetic properties has been obtained by the substitution of Sr^{2+} or Ba^{2+}

ions by rare earth metal ions, such as La^{3+} , Pr^{3+} , Sm^{3+} , Nd^{3+} and Dy^{3+} , or Fe^{3+} ions by transition metal ions, such as Co^{2+} , Zn^{2+} , Cu^{2+} , Al^{3+} , Cr^{3+} and Bi^{3+} [10-20]. Studies on cation combinations, such as $\text{La}^{3+}\text{-Co}^{2+}$, $\text{La}^{3+}\text{-Cu}^{2+}$, $\text{La}^{3+}\text{-Zn}^{2+}$, $\text{Pr}^{3+}\text{-Ni}^{3+}$, $\text{Nd}^{3+}\text{-Co}^{2+}$ and $\text{Ce}^{3+}\text{-Co}^{2+}$, have been done [21-26].

In this article, in order to improve the magnetic properties of M-type strontium hexaferrites, we have chosen Pr^{3+} ions to substitute Sr^{2+} ions and Co^{2+} ions to substitute Fe^{3+} ions. In the present work, the solid-state reaction method was used to synthesize Pr-Co substituted M-type strontium hexaferrites. The influence of Pr-Co substitution on the microstructural and magnetic properties has been investigated.

Experimental Procedure

The solid-state reaction method was used to prepare the Pr-Co substituted M-type strontium hexaferrites with the chemical composition of $\text{Sr}_{1-x}\text{Pr}_x\text{Fe}_{12-y}\text{Co}_y\text{O}_{19}$ ($x = 0.00-0.50$; $y = 0.00-0.35$). The raw materials used in this study were SrCO_3 (99% purity), Pr_6O_{11} (99% purity), Fe_2O_3 (99% purity) and CoO (99% purity). These raw materials were used as such without further treatment. All the compositions were wet-mixed in a ball mill at a rotating speed of 80 rpm for 10 hrs. The mixed powders were dried in a drying oven in the air, and then calcined in a muffle furnace at 1260°C for 2 hrs in air. After that, the calcined samples were shattered to particles by a vibration mill, and then wet-

*Corresponding author:
Tel: +86 831 3531171
Fax: +86 831 3531161
E-mail: loyaltty-yyj@163.com

milled with suitable additives (CaCO_3 , SrCO_3 , SiO_2 , $\text{Ca}(\text{C}_6\text{H}_{11}\text{O}_7)_2$) for 18 hrs in a ball-mill. The finely milled slurry was pressed into disc-shaped pellets ($\Phi 30 \text{ mm} \times 16 \text{ mm}$) under 310 MPa in the magnetic field of 800 kA/m, which was parallel to the pressing direction. The pressed pellets were sintered in a muffle furnace at 1190 °C for 1.5 hrs in air. Finally, in order to measure the magnetic properties, of the sintered pellets were polished in the faces perpendicular to the pressing direction.

Phase compositions of the magnetic powders were investigated by using a PANalytical X'Pert Pro diffractometer in continuous mode with Cu K_α radiation in the range of $2\theta = 20\text{--}80^\circ$. The morphology of the magnets was examined by a field emission scanning electron microscopy (FE-SEM, Hitachi S-4800). The magnetic properties of the sintered pellets were measured at room temperature by a permanent magnetic measuring system (NIM-2000HF, made by the National Institute of Metrology of China).

Results and Discussions

Fig. 1 shows the XRD patterns of M-type hexaferrite $\text{Sr}_{1-x}\text{Pr}_x\text{Fe}_{12-y}\text{Co}_y\text{O}_{19}$ ($x = 0.00\text{--}0.50$; $y = 0.00\text{--}0.35$) magnetic powders. It is seen that for the M-type hexaferrite magnetic powders with the substitution of Pr ($0.00 \leq x \leq 0.20$) and Co ($0.00 \leq y \leq 0.14$), the XRD patterns belong to the M-type strontium hexaferrite

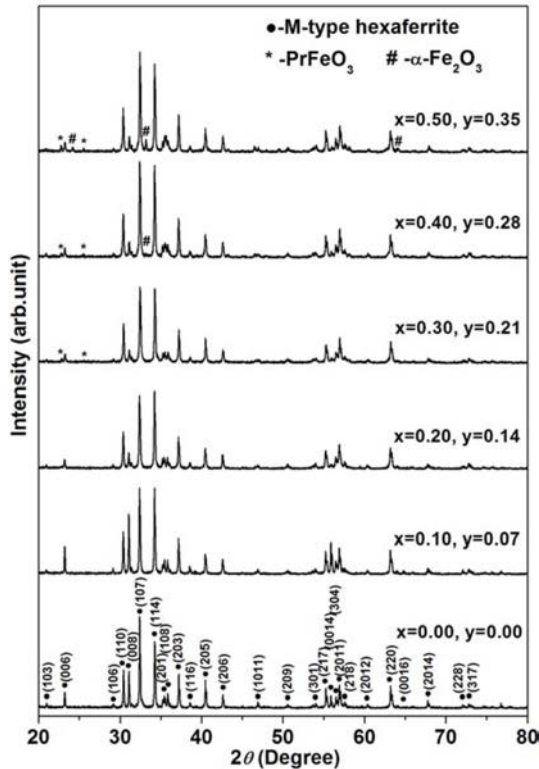


Fig. 1. XRD patterns of M-type hexaferrite $\text{Sr}_{1-x}\text{Pr}_x\text{Fe}_{12-y}\text{Co}_y\text{O}_{19}$ ($x = 0.00\text{--}0.50$; $y = 0.00\text{--}0.35$) magnetic powders.

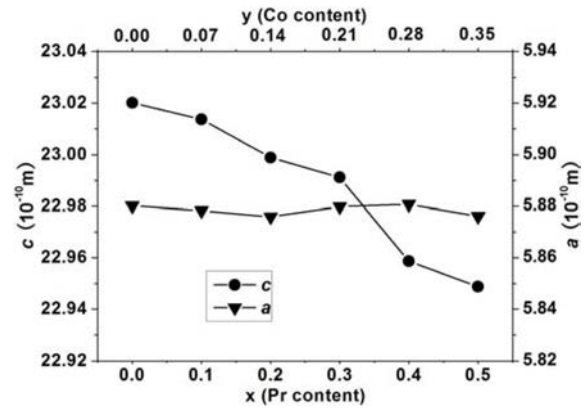


Fig. 2. Lattice constants c and a of M-type hexaferrite $\text{Sr}_{1-x}\text{Pr}_x\text{Fe}_{12-y}\text{Co}_y\text{O}_{19}$ ($x = 0.00\text{--}0.50$; $y = 0.00\text{--}0.35$) magnetic powders.

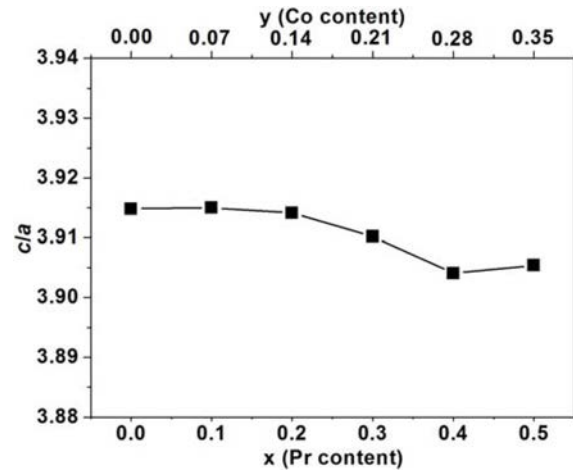


Fig. 3. c/a ratios as a function of Pr-Co content for the Pr-Co substituted M-type hexaferrite $\text{Sr}_{1-x}\text{Pr}_x\text{Fe}_{12-y}\text{Co}_y\text{O}_{19}$.

(JCPDS card no. 80-1198). For the magnetic powders containing Pr ($x \geq 0.30$) and Co ($y \geq 0.21$), PrFeO_3 is detected in the structure. For the magnetic powders containing Pr ($x \geq 0.40$) and Co ($y \geq 0.28$), PrFeO_3 and $\alpha\text{-Fe}_2\text{O}_3$ are detected in the structure.

The lattice constants c and a of hexagonal ferrites are calculated by the following equation:

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

where d_{hkl} is an inter-atomic spacing and h , k and l are the Miller indices. The variation of lattice constants c and a of M-type hexaferrite $\text{Sr}_{1-x}\text{Pr}_x\text{Fe}_{12-y}\text{Co}_y\text{O}_{19}$ ($x = 0.00\text{--}0.50$; $y = 0.00\text{--}0.35$) magnetic powders is shown in Fig. 2. It is evident that the lattice constant a keeps almost constant with the increasing substitution of Pr-Co, while the lattice constant c decreases with the substitution of Pr-Co contents ($0.00 \leq x \leq 0.50$, $0.04 \leq y \leq 0.35$). The decrease of lattice constant c can be interpreted in terms of the difference in the ionic radii (Δr) of the metal ions and the number of ionic substitutions of each species. Substitution of Sr^{2+} ($r = 1.180 \text{ \AA}$) by Pr^{3+} ($r = 1.013 \text{ \AA}$) makes a negative

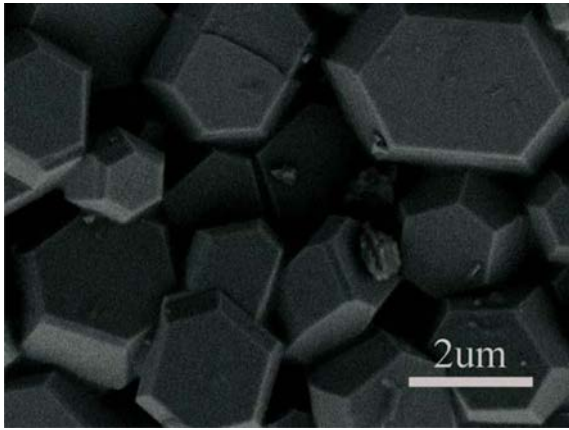


Fig. 4. A representative FE-SEM image of the M-type hexaferrite $\text{Sr}_{1-x}\text{Pr}_x\text{Fe}_{12-y}\text{Co}_y\text{O}_{19}$ magnet for $x = 0.20$, $y = 0.14$.

difference in the ionic radii of $\Delta r = -0.167 \text{ \AA}$. The ionic radius of Co^{2+} was $0.745 \text{ \AA}$ and that of Mn^{2+} was $0.67 \text{ \AA}$, giving an average value of $0.7075 \text{ \AA}$ for equiatomic CoMn co-substitution. Thus, substitution of Fe^{3+} ($r =$

$0.645 \text{ \AA}$) by Co^{2+} ($r = 0.745 \text{ \AA}$) makes a positive difference in the ionic radii of $\Delta r = +0.1000 \text{ \AA}$. In the Pr-Co substituted M-type hexaferrites, in order to keep the electroneutrality, some Fe^{3+} ions ($r = 0.645 \text{ \AA}$) will change into Fe^{2+} ions ($r = 0.780 \text{ \AA}$) at $2a$ or $4f_2$ site, this makes a positive difference in the ionic radii of $\Delta r = +0.135 \text{ \AA}$. As in the case of substitution of Pr ($x = 0.20$) and Co ($x = 0.14$), the total content of both Co^{2+} and Fe^{2+} ions is equal to that of Pr^{3+} ions, and thus, the lattice constant c is decreased.

The c/a ratios and cell volume (V_{cell}) as a function of Pr-Co content for the Pr-Co substituted M-type hexaferrite $\text{Sr}_{1-x}\text{Pr}_x\text{Fe}_{12-y}\text{Co}_y\text{O}_{19}$ are shown in Fig. 3. It is reported that the c/a ratio could be used to quantify the structure type, and the M-type hexagonal structure may be assumed if the c/a ratio is lower than 3.98 [27]. In this study, the c/a ratios are in the range from 3.9041 to 3.9150, which shows the formation of M-type hexagonal structure.

Fig. 4 shows a representative FE-SEM image of the M-type hexaferrite $\text{Sr}_{1-x}\text{Pr}_x\text{Fe}_{12-y}\text{Co}_y\text{O}_{19}$ magnet for

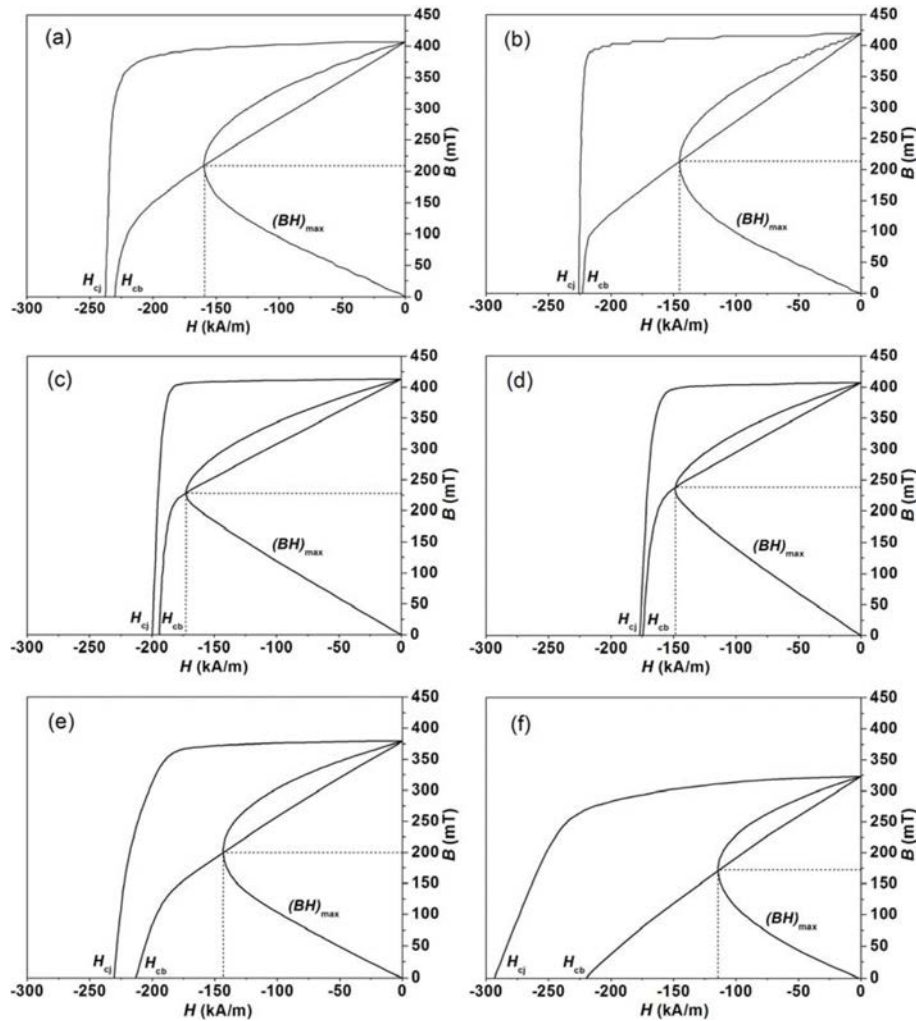


Fig. 5. Demagnetizing curves of M-type hexaferrite $\text{Sr}_{1-x}\text{Pr}_x\text{Fe}_{12-y}\text{Co}_y\text{O}_{19}$ ($x = 0.00-0.50$; $y = 0.00-0.35$) magnets measured at room temperature.

$x = 0.20$, $y = 0.14$. As seen from the FE-SEM image, grains are regular hexagonal structures with homogeneous grain with size between 2.0 and 4.0 μm .

The Demagnetizing curves of M-type hexaferrite $\text{Sr}_{1-x}\text{Pr}_x\text{Fe}_{12-y}\text{Co}_y\text{O}_{19}$ ($x = 0.00-0.50$; $y = 0.00-0.35$) magnets measured at room temperature are shown in Fig. 5. The remanence (B_r), the intrinsic coercivity (H_{ci}), magnetic induction coercivity (H_{cb}) and maximum energy product $[(BH)_{\max}]$ are extracted from the demagnetizing curves. Fig. 6 shows the variation of remanence (B_r) as a function of Pr-Co content for the M-type hexaferrite $\text{Sr}_{1-x}\text{Pr}_x\text{Fe}_{12-y}\text{Co}_y\text{O}_{19}$ magnets. It is observed from Fig. 6 that B_r initially increases with the substitution of Pr-Co content ($0.00 \leq x \leq 0.10$, $0.00 \leq y \leq 0.07$), and later on shows decreasing trend with increase in Pr-Co substitution. $3d^5$ configuration of Fe^{3+} ion attributes the $5 \mu_B$ of the magnetic moment for each Fe^{3+} ion. In the M-type hexaferrites, 2a, 2b and 12k are spin up sites, whereas $4f_1$ and $4f_2$ are spin down sites [2]. Wang et al have reported that Pr^{3+} ions are substituted for Sr^{2+} ions in the octahedral $4f_2$ and 2a sites [11]. It has been reported that Co^{2+} ($3.7 \mu_B$) ions are substituted for Fe^{3+} ($5 \mu_B$) ions in the $4f_1$, $4f_2$ and 2b sites [28]. In the Pr-Co substituted M-type hexaferrites, in order to keep the electroneutrality, some Fe^{3+} ions will change into Fe^{2+} ions at 2a or $4f_2$ site. The increase of remanence (B_r) with the substitution of Pr-Co content ($0.00 \leq x \leq 0.10$, $0.00 \leq y \leq 0.07$) can be due to the following two reasons. Firstly, the large magnetic moment for Pr^{3+} ion ($3.5 \mu_B$) enhances the exchange interactions and the net magnetic moment of hexagonal structure unit cell [29]. This results in the increase of remanence (B_r). Secondly, the occupancy of $4f_1$ and $4f_2$ sites by Co^{2+} ions is positive in inhibiting the offsetting of magnetic moment and leads to the increase of bulk magnetic moment. This causes the remanence (B_r) to increase. The decrease of remanence (B_r) with the substitution of Pr-Co content ($0.10 \leq x \leq 0.50$, $0.07 \leq y \leq 0.35$) can be due to the below three reasons. Firstly, the doping of Fe^{3+} ($5 \mu_B$) ions in the 2b sites by Co^{2+} ($3.7 \mu_B$) ions and the change of Fe^{3+} ($5 \mu_B$) ions to Fe^{2+} ($4 \mu_B$) ions result in the decrease of bulk magnetic moment. This results in the decrease of remanence (B_r). Secondly, with the substituting of Pr^{3+} ions for Sr^{2+} ions and Co^{2+} ions instead of Fe^{3+} ions, the $\text{Fe}^{3+}\text{-O-Fe}^{3+}$ superexchange interaction is weakened, which causes to magnetic collinearity to collapse. This results in the degradation of remanence (B_r). Thirdly, for the Pr-Co substituted M-type hexaferrites with the substitution of Pr-Co content ($0.30 \leq x \leq 0.50$, $0.21 \leq y \leq 0.35$), the decrease of remanence (B_r) is found for the appearance of impurity phases as shown in Fig. 1. In M-type hexaferrites, the impurity phases have almost no contribution to the increase of remanence (B_r).

Fig. 7 shows the variations of intrinsic coercivity (H_{ci}) and magnetic induction coercivity (H_{cb}) of M-type hexaferrite $\text{Sr}_{1-x}\text{Pr}_x\text{Fe}_{12-y}\text{Co}_y\text{O}_{19}$ ($x = 0.00-0.50$; $y = 0.00-0.35$) magnets. As seen from Fig. 7, it can found

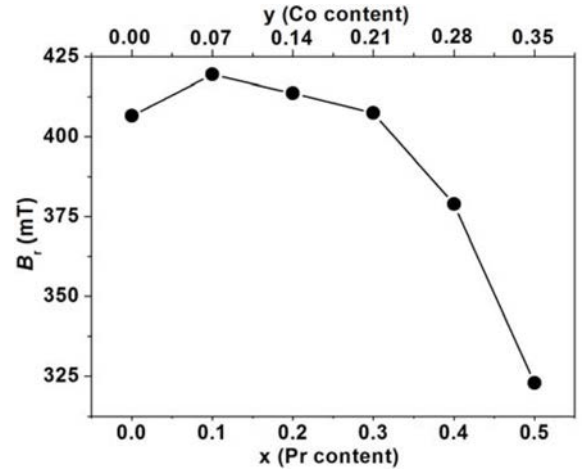


Fig. 6. Rmanence (B_r) of M-type hexaferrite $\text{Sr}_{1-x}\text{Pr}_x\text{Fe}_{12-y}\text{Co}_y\text{O}_{19}$ ($x = 0.00-0.50$; $y = 0.00-0.35$) magnets.

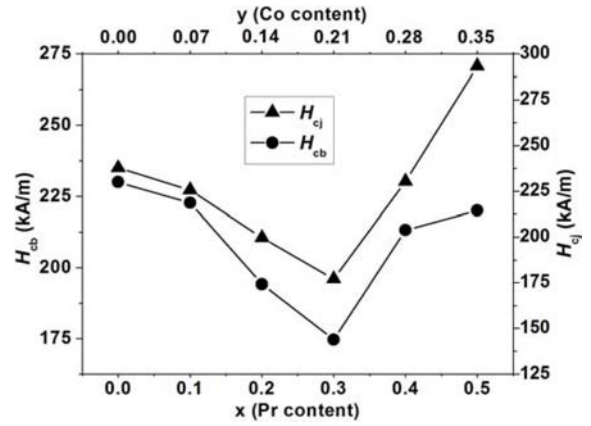


Fig. 7. Intrinsic coercivity (H_{ci}) and magnetic induction coercivity (H_{cb}) of M-type hexaferrite $\text{Sr}_{1-x}\text{Pr}_x\text{Fe}_{12-y}\text{Co}_y\text{O}_{19}$ ($x = 0.00-0.50$; $y = 0.00-0.35$) magnets.

that H_{ci} and H_{cb} decrease with the substitution of Sm-CrAl content ($0.00 \leq x \leq 0.30$, $0.00 \leq y \leq 0.21$), and then begin to increase with the increasing substitution content ($0.30 \leq x \leq 0.50$, $0.21 \leq y \leq 0.35$). The decrease of H_{ci} should be due to the decrease of uniaxial anisotropy constant because of the occupancy of the $4f_2$ and 2b sites by Co^{2+} ions. According to the Stoner-Wohlfarth theory, the coercivity in the M-type hexaferrite could be estimated by the below equation [30]:

$$H_c = 0.64 \frac{2K}{M_s} \quad (2)$$

where K is the magnetocrystalline anisotropy constant and M_s is the saturation magnetization. Therefore, according to the equation (2), the increase of H_{ci} can be due to the decrease of remanence (B_r) with the increasing substitution content ($0.3 \leq x \leq 0.50$, $0.21 \leq y \leq 0.35$) as shown in Fig. 6.

Fig. 8 shows the change of the maximum energy product $[(BH)_{\max}]$ with Pr-Co content for H_k/H_{ci} ratios of M-type hexaferrite $\text{Sr}_{1-x}\text{Pr}_x\text{Fe}_{12-y}\text{Co}_y\text{O}_{19}$ magnets. It

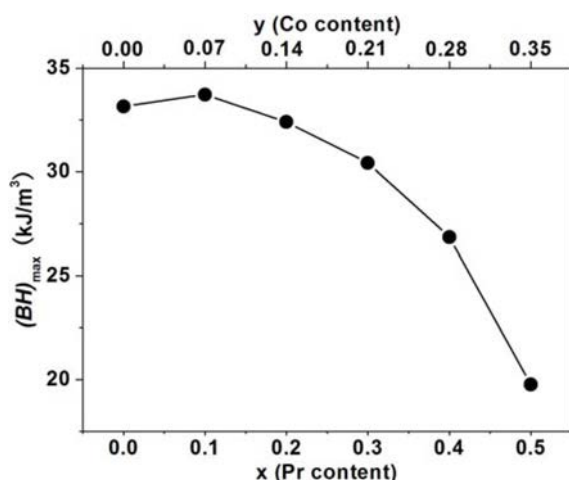


Fig. 8. Maximum energy product $[(BH)_{\max}]$ of M-type hexaferrite $\text{Sr}_{1-x}\text{Pr}_x\text{Fe}_{12-y}\text{Co}_y\text{O}_{19}$ ($x = 0.00-0.50$; $y = 0.00-0.35$) magnets.

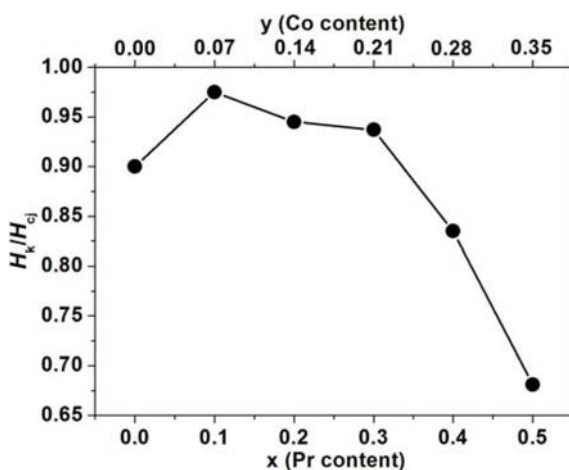


Fig. 9. H_k/H_{cj} ratios of M-type hexaferrite $\text{Sr}_{1-x}\text{Pr}_x\text{Fe}_{12-y}\text{Co}_y\text{O}_{19}$ ($x = 0.00-0.50$; $y = 0.00-0.35$) magnets.

is observed that the value of $(BH)_{\max}$ initially increases with the substitution of Pr-Co content ($0.00 \leq x \leq 0.10$, $0.00 \leq y \leq 0.07$), and then shows decreasing trend with increase in Pr-Co substitution. The maximum energy product of the hexaferrite magnets can be estimated by the product between the remanence (B_r) and coercivity field. As seen from Fig. 6 and Fig. 8, the changing trend of $(BH)_{\max}$ is agreement with that of the remanence (B_r).

The variation of H_k/H_{cj} ratios with Pr-Co content for H_k/H_{cj} ratios of M-type hexaferrite $\text{Sr}_{1-x}\text{Pr}_x\text{Fe}_{12-y}\text{Co}_y\text{O}_{19}$ magnets is shown in Fig. 9. Wang et al. [31] have reported that the H_k/H_{cj} ratio is one of the important indicators of the magnetic properties for the permanent magnets, and can intuitively indicate the rectangularity of the demagnetizing curves for the magnets. As seen from Fig. 9, It is clear that the ratio H_k/H_{cj} ratio initially increases with the substitution of Pr-Co content ($0.00 \leq x \leq 0.10$, $0.00 \leq y \leq 0.07$), and then shows decreasing trend with increase in Pr-Co substitution.

Conclusions

The solid-state reaction method was used to synthesize the Pr-Co substituted M-type strontium hexaferrites with the chemical composition of $\text{Sr}_{1-x}\text{Pr}_x\text{Fe}_{12-y}\text{Co}_y\text{O}_{19}$ ($x = 0.00-0.50$; $y = 0.00-0.35$). There is a single magnetoplumbite phase in the M-type hexaferrites with Pr-Co content ($0.00 \leq x \leq 0.20$, $0.00 \leq y \leq 0.14$). For the M-type hexaferrites with Pr-Co content ($x \geq 0.20$, $y \geq 0.14$), impurity phases are observed in the structure. The image of the representative magnet exhibits that the M-type hexagonal structure is formed in the magnet.

The magnetic properties of the M-type hexaferrite magnets were measured at room temperature by a permanent magnetic measuring system. The values of B_r , $(BH)_{\max}$ and H_k/H_{cj} ratios increase with the increasing substitution content of Pr ($0.00 \leq x \leq 0.10$) and Co ($0.00 \leq y \leq 0.07$), and then decrease with increasing substitution content of Pr ($x \geq 0.10$) and Co ($y \geq 0.07$). H_{cj} , and H_{cb} decrease with increasing Pr-Co content ($0.00 \leq x \leq 0.30$, $0.00 \leq y \leq 0.21$), and then increase with increasing substitution content of Pr ($x \geq 0.30$) and Co ($y \geq 0.21$).

Acknowledgments

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