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# Influence of Mn substitution on the magnetic and microstructural properties of the Sr<sub>0.75</sub>La<sub>0.25</sub>Fe<sub>12.00-x</sub>Mn<sub>x</sub>O<sub>19</sub> hexaferrites

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A series of Mn substituted M-type hexaferrites having the chemical compositions of  $Sr_{0.75}La_{0.25}Fe_{12.00-x}Mn_xO_{19}$  (where  $0.00 \le x \le 0.60$ ) were prepared by the conventional ceramic processing method. X-ray diffraction (XRD) was used to evaluate the hexagonal phase evolution for the M-type hexaferrite magnetic powders. The XRD results show that  $Mn^{3+}$  ions completely enter into the hexaferrite structure and all magnetic powders show M-type hexagonal structure. Magnetic properties of the powders were studied by a physical property measurement system-vibrating sample magnetometer (PPMS-VSM). The saturation magnetization (M<sub>s</sub>) and remanent magnetization (M<sub>r</sub>) first slightly increase with Mn content (x) form 0.00 to 0.24, and then decrease with Mn content (x) form 0.24 to 0.60. The coercivity (H<sub>c</sub>) of the magnetic powders first increases with Mn content (x) form 0.12 to 0.36, and increases with the increase of Mn content (x) when Mn content (x)  $\ge 0.36$ .

Key words: Hexagonal ferrites, Mn substitution, X-ray diffraction, Magnetic properties.

#### Introduction

M-type hexaferrites represents a class of materials with promising technological importance in various fields such as magnetic recording, microwave devices, telecommunications and permanent magnets [1]. These hexaferrites are available at low cost and possess large magneto-crystalline anisotropy, moderate energy product, high Curie temperature, excellent chemical stability, high corrosion and electrical resitivity [2]. In order to utilize these materials in a number of applications, many studies have been done to tailor the magnetic properties of Mtype hexaferrites. In the M-type hexaferrite structure, the Fe<sup>3+</sup> ions occupy five different crystallographic sites, three octahedral (2a, 12k and  $4f_2$ ), one tetrahedral site  $(4f_1)$  and one bipyramidal site (2b). In addition, 2a, 2b and 12k are spin up sites, while  $4f_1$  and  $4f_2$  are spin down sites [3]. Partial substitutions of  $Sr^{2+}$  or  $Ba^{2+}$  ions by  $La^{3+}$ ,  $Pr^{5+}$  and  $Sm^{3+}$  ions, and , and of  $Fe^{3+}$  ions by  $Co^{2+}$ ,  $Ti^{4+}$ ,  $Mn^{3+}$ ,  $Ni^{2+}$ ,  $Al^{3+}$  and  $Cr^{3+}$  ions have been investigated [4-13]. On the other hand, M-type strontium ferrites with combined substitution such as La-Co, La-Zn, La-Cu, Zn-Mn, etc. have been synthesized [14-17]. The magnetic properties of the substituted hexaferrites strongly depend on the electronic configuration of substituted cations. Abbas et al. have reported that for

the Pr-substituted M-type hexaferrites Ba<sub>0.25</sub>Sr<sub>0.75</sub>Pr<sub>x</sub>  $Fe_{12-x}O_{19}$  (0.00  $\le x \le 0.25$ ), at x = 0.10, the sample exhibits maximum saturation magnetization (M<sub>s</sub>) and a few hundreds of coercivity  $(H_c)$  [5]. Teh et al. have synthesized the Co<sup>2+</sup> doped M-type barium hexaferrites  $Ba_{1-x}Co_xFe_{12}O_{19}$  (x = 1.0) and found that both the saturation magnetization and coercivity decreased for x = 1.0  $Co^{2+}$  doping [7]. Sharma et al. have reported that for the Mn<sup>3+</sup> ions doped barium hexaferrites Ba<sub>1-x</sub>Fe<sub>12-x</sub> Mn  $_xO_{19}$  (0.0  $\le$  x  $\le$  2.0), Mn<sup>3+</sup> ions occupies all iron sites, decreasing the magnetization and increasing the coercivity [10]. Katlakunta et al. have studied the microwave-sintered Cr3+ doped strontium hexaferrites synthesized by microwave hydrothermal route and found that with increasing Cr<sup>3+</sup> doping the saturation magnetization systematically decreased and the coercivity increased [13]. Yang et al. have synthesized the La-Co substituted Sr-Ca hexaferrites by the solid state reaction method and found that with increasing La-Co contents the remanence continuously increased, and the coercivity first increased and then decreased [14]. Arab et al. have investigated the magnetic properties of MnZn-substituted strontium ferrite SrMn<sub>x</sub>Zn <sub>x</sub>Fe<sub>12-2x</sub>O<sub>19</sub>  $(0 \le x \le 1.0)$  nanoparticles via conventional ceramic technique followed by a high energy ball milling and found that by the increasing of cation concentions, the magnetizations of samples are increased up to 0.6 and then they are decreased [17].

Many methods are used to prepare the M-type hexagonal ferrites, such as the ceramic process [18], the citrate precursor method [19], the hydrothermal

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method [20], the chemical coprecipitation method [21] and the sol-gel method [22]. The ceramic process is employed to synthesize the hexaferrites because of its simplicity, highly productive, well controllable grain size and cheaper than other methods [23]. Therefore, in the present work, the conventional ceramic processing method was used to prepare Mn substituted Sr-La Mtype hexaferrites. The influence of Mn substitution on the microstructural and magnetic is investigated herein.

## **Experimental**

The conventional ceramic processing method was employed to synthesize the hexagonal ferrites with the chemical composition ( $Sr_{0.75}La_{0.25}Fe_{12.00-x}Mn_xO_{19}$ , where Mn content (x) = 0.00, 0.12, 0.24, 0.36, 0.48, 0.60). The starting materials, i.e.  $SrCO_3$ ,  $La_2O_3$ ,  $Fe_2O_3$  and  $Mn_3O_4$ were powders of analytical grade. In a stoichiometric ratio, required weights of different starting materials for various compositions were weighted in approximate amounts, and wet-mixed for 6 hrs in a ball mill. The mixed powders were calcined at 1255 °C for 2 hrs in the air in an electric furnace. The calcined samples were shattered to particles less than 60 µm using a vibration mill, and then annealed at 780 °C for 1 hr in an electric furnace in order to relieve the stress.

All the obtained magnetic powders were characterized by X-ray diffraction (XRD) in a continuous mode with Cu  $K_{\alpha}$  radiation source ( $\lambda = 1.5406$  Å) (SmartLab 9 kW, Rigaku Corporation). Magnetization studies were carried out at room temperature using a physical property measurement system-vibrating sample magnetometer (PPMS-VSM, Quantum Design). The saturation magnetization ( $M_s$ ), remanent magnetization ( $M_r$ ) and coercivity ( $H_c$ ) of the magnetic powders were measured with a maximum applied magnetic field of 20000 Oe.

# **Results and Discussion**

The XRD patterns of the hexaferrite  $Sr_{0.75}La_{0.25}Fe_{12.00-x}$  $Mn_xO_{19}$  magnetic powders with Mn content (x) from 0.00 to 0.60 are shown in Fig. 1. Density intensity was measured with a 20 range from 20 ° to 80 ° with scan size step of 0.02°. The peak position and relative intensity of all diffraction peaks are observed to be matching well with powder diffraction file of JCPDS card no. 80-1198, thereby confirming the P63/mmc space group. Using the JCPDS data (card no. 80-1198), the peaks of X-ray diffraction patterns were indices for hexagonal structure. For all the samples, free Mn<sup>3+</sup> ions are not seen from the diffraction patterns, indicating that Mn<sup>3+</sup> ions enter into the hexaferrite structure. The sharp lines of the diffraction patterns are observed for all the samples, indicating that the samples consist of large crystallites.

The lattice parameters '*a*' and '*c*' of the hexaferrite  $Sr_{0.75}La_{0.25}Fe_{12.00-x}Mn_xO_{19}$  magnetic powders with Mn



Fig. 1. XRD patterns of the Mn doped hexaferrite  $Sr_{0.75}La_{0.25}Fe_{12.00-x}Mn_xO_{19}$  magnetic powders.

content (x) from 0.00 to 0.60 were calculated from the the peaks (107) and (114) using the following equation [24]:

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

where  $d_{hkl}$  is the inter-planer spacing value, and the values of *h*, *k* and *l* are the Miller indices. The crystallite size of the hexaferrite Sr<sub>0.75</sub>La<sub>0.25</sub>Fe<sub>12.00-x</sub> Mn<sub>x</sub>O<sub>19</sub> magnetic powders with Mn content (x) from 0.00 to 0.60 is calculated by using the relation [25]:

$$D = \frac{K\lambda}{\beta\cos\theta} \tag{2}$$

where  $\lambda$  is the X-ray wavelength,  $\beta$  the broadening of diffraction lines at half-width of maximum intensity,  $\theta$  the Bragg angle and the value of shape factor K = 0.89. The unit cell volume ( $V_{cell}$ ) was calculated according to the following formula [24]:

$$V_{cell} = \frac{\sqrt{3}}{2}a^2c \tag{3}$$

The calculated values of lattice parameters '*a*' and '*c*', c/a ratios, crystallite size and unit cell volume ( $V_{cell}$ ) are listed in Table 1. It can be observed that from the values of lattice constants '*a*' and '*c*' remain almost unchanged in all Mn substituted Sr-La M-type hexaferrites. This is mainly due to the fact that the ionic radius of Mn<sup>3+</sup> ions (0.645 Å) is almost the same as that of Fe<sup>3+</sup> ions (0.64 Å). From Table 1, it is also

693.4

694.3

695.3

694.5

56.4

56.6

58.2

58.2

(D) and cell volume ( $V_{cell}$ ) for the hexaferrite Sr <sub>0.75</sub> La <sub>0.25</sub> Fe <sub>12.00-x</sub> Mn <sub>x</sub> O <sub>19</sub> magnetic powders with different Mn content (x).								
Mn content (x)	c (Å)	a (Å)	c/a	D (nm)	V (Å <sup>3</sup> )			
0.00	23.072	5.892	3.916	54.9	693.6			
0.12	23.076	5.893	3.916	56.6	694.0			

3.917

3.916

3.914

3.913

5.891

5.894

5.898

5.896

23.073

23.079

23.082

23.069

0.24

0.36

0.48

0.60

**Table 1.** Lattice parameters c and a, ratio of c/a, crystallite size

seen	that	with	increasi	ng Mn	conten	it (x), 1	the a	c/a ratios
and	unit	cell	volume	$(V_{cell})$	of the	magne	etic	powders
basic	cally	keep	constan	t.				

Fig. 2 shows the magnetic hysteresis loops of the hexaferrite Sr<sub>0.75</sub>La<sub>0.25</sub>Fe<sub>12.00-x</sub>Mn<sub>x</sub>O<sub>19</sub> magnetic powders with Mn content (x) from 0.00 to 0.60. The characteristic magnetic parameters such as the saturation magnetization  $(M_{\rm s})$ , remanent magnetization  $(M_{\rm r})$  and coercivity  $(H_{\rm c})$ 

Table 2. Data of  $M_s$ ,  $M_r$ ,  $H_c$  and  $M_r/M_s$  of the hexaferrite Sr<sub>0.75</sub>La<sub>0.25</sub>Fe<sub>12.00-x</sub>Mn<sub>x</sub>O<sub>19</sub> magnetic powders with different Mn content (x) from 0.00 to 0.60.

Mn content (x)	M <sub>s</sub> (emu/g)	M <sub>r</sub> (emu/g)	H <sub>c</sub> (Oe)	$M_{ m r}/M_{ m s}$
0.00	67.70	37.18	3429	0.549
0.12	67.86	37.31	3735	0.550
0.24	67.93	37.44	3278	0.551
0.36	66.82	36.80	3262	0.551
0.48	64.69	35.59	3687	0.550
0.60	64.56	35.45	3761	0.549

were determined experimentally and are listed in Table 2. Fig. 3 shows the variation of the saturation magnetization  $(M_s)$  and remanent magnetization  $(M_r)$  as a function of Mn content (x) for the hexaferrite Sr<sub>0.75</sub> La<sub>0.25</sub>Fe<sub>12.00-x</sub>Mn<sub>x</sub>O<sub>19</sub> magnetic powders. It is observed that the value of  $M_{\rm s}$  and  $M_{\rm r}$  for the magnetic powders first slightly increase with Mn content (x) from 67.70 and 37.18 emu/g at x = 0.00 to 67.93 and 37.44 emu/g



Fig. 2. Magnetic hysteresis loops of the M-type hexaferrite  $Sr_{0.75}La_{0.25}Fe_{12.00-x}Mn_xO_{19}$  magnetic powders for compositions of (a) x = 0.00, (b) x = 0.12, (c) x = 0.24, (d) x = 0.36, (e) x = 0.48, and (f) x = 0.60.



**Fig. 3.** Variation in the saturation magnetization ( $M_s$ ) and remanent magnetization ( $M_r$ ) with Mn content (x) for the hexaferrite Sr<sub>0.75</sub>La<sub>0.25</sub>Fe<sub>12.00-x</sub>Mn<sub>x</sub>O<sub>19</sub> magnetic powders.



Fig. 4. Variation in the coercivity (Hc) with Mn content (x) for the hexaferrite  $Sr_{0.75}La_{0.25}Fe_{12.00-x}MnxO_{19}$  magnetic powders.

at x = 0.24, respectively, and then decrease with Mn content (x) from 0.24 to 0.60. The variation of  $M_s$  with Mn content (x) is not in agreement with that reported by Sharma et al. [26]. This may be due to the different preparing methods. In the crystalline structure of Mtype hexaferrites, twenty-four Fe<sup>3+</sup> ions occupy interstitial positions in five different crystallographic sites and are distributed as follows. Three octahedral (12k, 4f<sub>2</sub> and 2a) and site 4f<sub>1</sub> have a geometric tetrahedron formed by oxygen atoms, and site 2b has a bipyramidal geometry with a hexahedral triangular base formed by five oxygen atoms around the Fe<sup>3+</sup> ion. The Fe<sup>3+</sup> ions with up-spin are distributed on the 2a, 2b and 12k, and  $Fe^{3+}$ ions with down-spin are located on the  $4f_1$  and  $4f_2$ sites. For the M-type hexaferrite, the magnetic moments of Fe<sup>3+</sup> ions are arranged collinearly because of the existence of super-exchange interaction. Sharma et al. [26] have reported that manganese occupies all  $Fe^{3+}$  ion sites. The magnetic moments of  $Fe^{3+}$  ion and  $Mn^{3+}$  ion are 5  $\mu_B$  and 3.5  $\mu_B$ , respectively [27]. The increase in  $M_{\rm s}$  and  $M_{\rm r}$  of magnetic powders with Mn content (x) from 0.00 to 0.24 may be due to the fact that the site occupation percent in spin-down sites is slightly higher than that in the spin-up sites. This will cause an increasing number of Fe<sup>3+</sup> ions in spin-up sites compared with spin-down sites, and, as a result, there appears an increase of magnetization with the increase of x from 0.00 to 0.24. When Mn content (x) $\geq$  0.24, the decrease in  $M_{\rm s}$  and  $M_{\rm r}$  of magnetic powders with Mn content (x) can be attributed to the following two reasons. The first reason is the fact that the site occupation percent in spin-up sites is higher than that in the spin-down sites. This may reduce the magnetic moment, and thus, results in the decrease in magnetization. As is well known, the collinear arrangement of magnetic moment is settled by the superexchange interaction between  $\mathrm{Fe}^{3+}$  ions in the hexaferrites. The doping of  $\mathrm{Mn}^{3+}$ ions causes the superexchange interaction between the octahedral and tetrahedral sites to reduce, and then, results in the deviation to the collinear arrangement of magnetic moment and a destruction of magnetic structure. This may be the second reason for the decrease in magnetization. The  $M_r/M_s$  ratio which is known as squareness ratio is also calculated and is listed in Table 2. It can be seen from the Table 2 that  $M_r/M_s$  ratios with the increase of Mn content (x) basically keep constant about 0.550. This shows that Mn doping can not affect the squareness of magnetic hysteresis loops greatly.

The variation of the coercivity ( $H_c$ ) as a function of Mn content (x) for the hexaferrite Sr<sub>0.75</sub>La<sub>0.25</sub>Fe<sub>12.00-x</sub> Mn<sub>x</sub>O<sub>19</sub> magnetic powders is shown in Fig. 4. It can be seen that the value of  $H_c$  for the magnetic powders first increases with Mn content (x) from 0.00 to 0.12, and then decrease with Mn content (x) from 0.12 to 0.36, and increases with the increase of Mn content (x) when Mn content (x)  $\geq$  0.36. In the M-type hexaferrites, the magnetocrystalline anisotropy is mainly from the single ion anisotropy of the Fe<sup>3+</sup> ions on the 4f<sub>2</sub> and 2b sites [28]. Sharma et al. [26] have reported that manganese occupies all Fe<sup>3+</sup> ion sites. As is known,  $H_c$  is inversely proportional to  $M_s$  theoretically and can be described by the following formula [29]:

$$H_c = \alpha \times \frac{2K}{M_s} \tag{4}$$

where  $\alpha$  is the orientation factor,  $M_s$  is the saturation magnetization and K is the magneto-crystalline anisotropy constant. As mentioned, the value of  $M_s$  for the magnetic powders slightly increases with Mn content (x) from 0.00 to 0.24. Therefore, according to the formula (4), the increase of  $H_c$  with Mn content (x) from 0.00 to 0.12 should be due to the increase of magnetocrystalline anisotropy constant. When Mn content (x) is increases from 0.12 to 0.36, some Mn<sup>3+</sup> ions may substitute Fe<sup>3+</sup> ions on the 4f<sub>2</sub> and 2b sites. This can result in the decrease of magnetocrystalline anisotropy, therefore, the decrease of  $H_c$  in the Mn substituted hexaferrite magnetic powders. When Mn content (x)  $\geq$  0.36, according to the formula (4), the decrease in  $M_{\rm s}$  for the magnetic powders with Mn content (x) results in the increase of  $H_{\rm c}$ .

### Conclusions

M-type hexaferrites Sr<sub>0.75</sub>La<sub>0.25</sub>Fe<sub>12.00-x</sub>Mn<sub>x</sub>O<sub>19</sub> with Mn content (x) from 0.00 to 0.60 have been sythesized by the conventional ceramic processing method. The XRD analysis indicates that Mn<sup>3+</sup> ions completely enter into the hexaferrite structure and all magnetic powders show M-type hexagonal structure. The magnetic properties of the magnetic powders with different Mn content (x) were investigated systematically.  $M_{\rm s}$  and  $M_{\rm r}$  first slightly increase with x from 0.00 to 0.24, and then decrease with x from 0.24 to 0.60.  $M_{\rm s}$  and  $M_{\rm r}$  reache to the maxmum values of 67.93 and 37.44 emu/g at x = 0.24, respectively.  $M_r/M_s$  ratios with the increase of Mn content (x) basically keep constant about 0.550.  $H_c$ of the magnetic powders first increases with x from 0.00 to 0.12, and then decrease with x from 0.12 to 0.36, and increases with the increase of x when Mn content (x)  $\geq$  0.36. H<sub>c</sub> reaches to the minimum value of 3262 Oe at x = 0.36.

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