Influence of Cr doping on the microstructural and magnetic properties of the Sr$_{0.35}$Ca$_{0.30}$La$_{0.35}$Fe$_{12.00-\chi}$Cr$_\chi$O$_{19}$ hexagonal ferrites

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Cr-doped hexagonal ferrites, with chemical composition Sr$_{6.35}$Ca$_{4.30}$La$_{3.30}$Fe$_{12.00-\chi}$Cr$_\chi$O$_{19}$ (where 0.00 $\leq \chi \leq 1.05$), were synthesized using the traditional ceramic route. An X-ray diffractometer (XRD) and a vibrating sample magnetometer were used to characterize the phase composition and magnetic properties of the magnetic powders, respectively. The XRD data show that when the Cr content ($\chi$) is less than 0.60, the magnetic powders show a magnetoplumbite structure. When the Cr content ($\chi$) is greater than 0.60, the magnetic powders present a CrFe$_2$O$_3$ structure. The coercivity increases with increasing Cr content ($\chi$) from 0.00 to 0.60, and then decreases slowly with increasing Cr content ($\chi$) above 0.60.

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1. Introduction

Permanent magnets have been an important class of functional materials because of their wide technological applications, such as small motors, actuators, generators, medical and microwave devices, and more recently, emerging applications related to automobile industry.$^{1-3}$ Permanent ferrites with magnetoplumbite (also known as M-type hexaferrites) occupy a major place in the permanent magnet market because of their competitive cost per unit of stored energy, their good chemical stability, and their high Curie temperature (usually above 600 K).$^{4-6}$ In the hexagonal structure, there is an overlap of cubically and hexagonally packed sections. In the M-type hexaferrite structure, the Fe$^{3+}$ ions occupy five different crystallographic sites such as three octahedral (2a, 12k and 4f$_1$), one tetrahedral site (4f$_2$), 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.$^7$

Many studies have been done to improve the magnetic properties of hexaferrites in order to utilize these materials in a number of applications. It has been reported that the coercivity ($H_c$) of M-type hexaferrites can be improved by partial substitutions of Sr$^{2+}$ or Ba$^{2+}$ ions by La$^{3+}$, Pr$^{3+}$, Dy$^{3+}$, Sm$^{3+}$, Nd$^{3+}$ and Ho$^{3+}$ ions, and of Fe$^{3+}$ ions by Al$^{3+}$ and Cr$^{3+}$ ions.$^{8-16}$ Liu et al. have reported that for the M-type strontium ferrites, the suitable amount of La$^{3+}$ addition may remarkably increase saturation magnetization ($M_s$) and intrinsic $H_c$.$^8$ Buzinario et al. have synthesized the samarium-doped M-type strontium hexaferrites with chemical formula Sr$_{1-x}$Sm$_x$Fe$_{12}$O$_{19}$ (0 $< x < 0.15$) and found that the $H_c$ increases with Sm$^{3+}$ concentration.$^{17}$ Liu et al. have reported that for the

Al$^{3+}$ ions-doped nanocrystalline Sr$_{x}$Fe$_{12-\chi}$Al$_{\chi}$O$_{19}$ (0 $\leq x \leq 12$), with increase of Al$^{3+}$ ion content, the room temperature $M_s$ values continuously reduced while $H_c$ value increases to a maximum value of 18100 Oe at $x = 4.14$, Praveena et al. have studied the microwave-sintered Cr$^{3+}$-doped Strontium hexaferrites and found that the $M_s$ and $H_c$ values increase with increasing Cr$^{3+}$ doping, but the $H_c$ increased.$^{16}$ It has also been reported that La–Co substitutions can improve both the $M_s$ and remanence $H_r$, while La–Zn or La–Cu substitutions lead to the increase of the magnetization and remanence and the decrease of the $H_r$. Many different methods have been used to synthesize the M-type hexaferrites with better magnetic properties, such as the sol-gel method,$^{20}$ the ceramic process,$^{21}$ the hydrothermal method,$^{22}$ the citrate precursor method$^{23}$ and the chemical coprecipitation method.$^{24}$ Among these methods, the ceramic process is typically used to prepare the magnetic oxides because of its simplicity, highly productive, well controllable grain size and cheaper than other methods.$^{25}$

In this work, series of hexagonal ferrites, Sr$_{6.35}$Ca$_{4.30}$La$_{3.30}$Fe$_{12.00-\chi}$Cr$_\chi$O$_{19}$ (0.00 $\leq \chi \leq 1.05$), have been synthesized by the traditional ceramic route. The microstructural and magnetic properties of Cr-doped Sr–Ca hexaferrites were investigated systematically.

2. Experimental

Hexagonal ferrite samples have been prepared by the traditional ceramic route. Compositions were chosen to according to the chemical composition Sr$_{6.35}$Ca$_{4.30}$La$_{3.30}$Fe$_{12.00-\chi}$Cr$_\chi$O$_{19}$, where Cr content ($\chi$) varied from 0.00 to 1.05 with about 0.15 increment. All the precursor materials used (CaCO$_3$, SrCO$_3$, La$_2$O$_3$, Fe$_2$O$_3$ and Cr$_2$O$_3$) were powders of analytical grade. All the precursor
Materials were weighted in approximate amounts, and wet-mixed for 9 h in a ball mill. The mixed powders were calcined in a laboratory furnace at 1255°C for 2.0 h in the air. The calcined samples were shattered to particles less than 100 μm using a vibration mill, and then annealed in a laboratory furnace at 780°C for 1.0 h in order to relieve the stress.

The phase composition of the magnetic powders was characterized by an X-ray diffractometer in a continuous mode with Cu Kα radiation source (λ = 1.5406 Å) (PAanalytical X’Pert Pro). The particle sizes of the magnetic powders were measured by a sub-sieve sizer (WLP-205, Huayu Instrument). The magnetic properties of the magnetic powders were measured by using a vibrating sample magnetometer (VSM) (VSM, Lakeshore 7401) in applied field up to 10000 Oe at room temperature.

3. Results and discussion

3.1 Structure determination

Figure 1 shows the X-ray diffraction patterns of the hexaferrite Sr0.35Ca0.30−xFe12−xO36−x−Cr0.05O19 magnetic powders with Cr content (x) from 0.00 to 1.05. Density intensity was measured with a 2θ range from 20 to 80° with scan size step of 0.02°. Using the JCPDS data (card no. 80-1198), the peaks of X-ray diffraction patterns were indices for hexagonal structure. When the Cr content (x) ≤ 0.60, the diffraction peak of non-magnetic phase, α-Fe2O3, is obviously observed in Fig. 1. The strength of the magnetic powders with Cr content (x) from 0.00 to 1.05 are listed in Table 1. It is seen that the lattice parameters a and c that both lattice parameters fluctuate and basically keep constant. This is mainly ascribed to the fact that the ionic radius of Cr3+ ions (0.63 Å) is almost the same as that of Fe3+ ions (0.64 Å). Thus, the variation in the lattice parameters can mainly result from the effect of ion size, and also might be due to the change of exchange energy after Cr3+ doping.

The values of the c/a ratios for the hexaferrite Sr0.35Ca0.30−xFe12−xO36−x−Cr0.05O19 magnetic powders with Cr content (x) from 0.00 to 1.05 are calculated from the XRD data using the following equation:

\[
\frac{1}{d_{hkl}} = \frac{4}{3} \cdot \frac{h^2 + hk + k^2}{a^2} + \frac{\mu^2}{c^2}
\]

(1)

where \(d_{hkl}\) is the inter-planer spacing value, and the values of h, k and l are the Miller indices. The values of lattice parameters a and c are listed in Table 1. It is evident from the values of lattice constants a and c that both lattice parameters fluctuate and basically keep constant. The variation in the lattice parameters can mainly result from the effect of ion size, and also might be due to the change of exchange energy after Cr3+ doping.

The values of the c/a ratios for the magnetic powders with different Cr content (x) range from 3.905 to 3.911, which are well within the ratio range of magnetoplumbite structures.

The unit cell volume (\(V_{cell}\)) and X-ray density (\(\rho_{X-ray}\)) are calculated according to the following formulae:

\[
V_{cell} = \frac{\sqrt{3}}{2} a^2 c
\]

(2)

\[
\rho_{X-ray} = \frac{2M}{N_A V_{cell}}
\]

(3)

where M is the molar mass, NA is Avogadro’s number (6.02 × 10²³/mole), and \(V_{cell}\) is the unit cell volume. The values of the unit cell volume \(V_{cell}\) and X-ray density \(\rho_{X-ray}\) for the hexaferrite Sr0.35Ca0.30−xFe12−xO36−x−Cr0.05O19 magnetic powders as a function of Cr content (x) are given in Table 1. The variations in applied field up to 10000 Oe at room temperature.

Table 1. Lattice parameters c and a, ratio of c/a, crystallite size (D), cell volume (Vcell), X-ray density (\(\rho_{X-ray}\)) and particle size (d) for the hexaferrite Sr0.35Ca0.30−xFe12−xO36−x−Cr0.05O19 magnetic powders with different Cr content (x)

| Cr content (x) | c (Å) | a (Å) | c/a | D (nm) | Vcell (Å³) | \(\rho_{X-ray}\) (g cm⁻³) | d (μm) |
|---------------|------|------|-----|--------|------------|----------------|-------|
| 0.00          | 23.006 | 5.889 | 3.907 | 50.1 | 691.4 | 5.120 | 7.6 |
| 0.15          | 23.032 | 5.884 | 3.914 | 48.6 | 691.0 | 5.120 | 7.3 |
| 0.30          | 23.013 | 5.885 | 3.911 | 50.3 | 690.7 | 5.119 | 7.4 |
| 0.45          | 23.027 | 5.888 | 3.911 | 52.3 | 691.8 | 5.108 | 7.2 |
| 0.60          | 22.989 | 5.874 | 3.914 | 53.9 | 687.4 | 5.138 | 7.5 |
| 0.75          | 22.978 | 5.882 | 3.907 | 53.9 | 688.9 | 5.124 | 7.3 |
| 0.90          | 22.935 | 5.873 | 3.905 | 54.4 | 685.5 | 5.147 | 7.2 |
| 1.05          | 22.948 | 5.876 | 3.903 | 53.4 | 686.6 | 5.136 | 7.4 |
in $V_{cell}$ and $\rho_{X-ray}$ are obviously slight and are in agreement with that reported by Dhage et al.\textsuperscript{29)} The crystallite size of the hexaferrite $Sr_{0.35}Ca_{0.30}La_{0.35}Fe_{12.00-\delta}Cr_{\delta}O_{19}$ magnetic powders with Cr content ($x$) from 0.00 to 1.05 is calculated by using the relation:\textsuperscript{30)}

$$D = \frac{K\lambda}{\beta \cos \theta} \quad (4)$$

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 values of the crystallite size for the magnetic powders with different Cr content ($x$) are listed in Table 1. The variations in $V_{cell}$ and $\rho_{X-ray}$ are not in agreement with that reported by Dhage et al.\textsuperscript{29)} The average crystallite size is in the range from 48.6 to 54.4 Å, which are larger than that reported by Dhage et al.\textsuperscript{29)} This may be due to the difference in preparation method.

The values of particle size for the magnetic powders with different Cr content ($x$) are also listed in Table 1. The particle sizes basically keep constant and are in the range from 7.2 to 7.6 μm.

### 3.2 Magnetic measurements

Magnetic hysteresis loops have been recorded at the room temperature for all the samples using VSM. The magnetic measurements were performed in an applied field of 10000 Oe. The magnetic hysteresis loops of the hexaferrite $Sr_{0.35}Ca_{0.30}La_{0.35}Fe_{12.00-\delta}Cr_{\delta}O_{19}$ magnetic powders with Cr content ($x$) from 0.00 to 1.05 are shown in Figs. 2 and 3. Various magnetic parameters including the $M_r$, remanent magnetization ($M_r$) and $H_c$ are calculated from the magnetic hysteresis loops. The values of $M_r$, $M_s$, $H_c$ and $M_s/M_r$ are tabulated in Table 2.

The variations in the $M_r$ and $M_s$ as a function of Cr content ($x$) for the hexaferrite $Sr_{0.35}Ca_{0.30}La_{0.35}Fe_{12.00-\delta}Cr_{\delta}O_{19}$ magnetic powders are shown in Fig. 4. It can be seen that the value of $M_s$ for the magnetic powders linearly decreases from 45.53 to 35.62 emu/g with the increase of Cr content ($x$) from 0.00 to 1.05, while the value of $M_s$ for the magnetic powders first increases with Cr content ($x$) from 18.59 emu/g at $x = 0.00$ to 25.71 emu/g at $x = 0.45$, and then keeps about 26 emu/g with Cr content ($x$) form 0.45 to 0.75, and begins to decrease when Cr content ($x$) $\geq 0.75$. The variation of $M_s$ with Cr content ($x$) is in agreement with that reported by Katalkunta et al.\textsuperscript{15)} and Praveena et al.\textsuperscript{16)} In the M-type hexaferrite structure, Fe$^{3+}$ ions are distributed on five different crystallographic sites such as three octahedral (2a, 12k and 4f$_2$), one tetrahedral site (4f$_1$), and one bipyramidal site (2b). In the magnetically ordered state of M-type hexaferrite, 2a, 2b and 4f$^2$ are spin up sites, while 4f$_1$ and 4f$_2$ are spin down sites. For the M-type hexaferrite, the magnetic moments of Fe$^{3+}$ ions are arranged collinearly because of the existence of super-exchange interaction. Ounnunkad et al.\textsuperscript{11)} and Kim et al.\textsuperscript{32)} have reported that Fe$^{3+}$ ions prefer to distribute on spin up Fe sites known as 2a and 12k. Thus, the decrease in $M_s$ of magnetic powders with Cr content ($x$) can be ascribed to the magnetic moment of constituent ions. The doping of higher magnetic moment Fe$^{3+}$ $(5 \mu_B)$ by lower magnetic moment Cr$^{3+}$ $(3 \mu_B)$ results in the decrease of bulk magnetic moment. More Cr$^{3+}$ doping leads to the weakening of superexchange interaction of type Fe$_s^{3+}$-O–Fe$_B^{3+}$, which causes to magnetic collinearity to collapse. The variation of $M_s$ with Cr content ($x$) are not in agreement with that reported by Katalkunta et al.\textsuperscript{15)} and Praveena et al.\textsuperscript{16)} This difference may be due the different preparation method. In the references 15) and 16), Cr-doped Sr-hexaferrites (SrFe$_{12-x}$Cr$_x$O$_{19}$) were prepared by microwave-hydrothermal method. The variation of $M_s$ with Cr content ($x$) can be attributed to the variation of squareness of magnetic hysteresis loops. From the Figs. 2 and 3, we can see that the squareness of magnetic hysteresis loops increases with the increasing Cr content ($x$) from 0.00 to 1.05. The $M_s/M_r$ ratio which is known as squareness ratio is also calculated and is listed.

![Fig. 2. Magnetic hysteresis loops of the hexaferrite $Sr_{0.35}Ca_{0.30}La_{0.35}Fe_{12.00-\delta}Cr_{\delta}O_{19}$ magnetic powders for compositions of (a) $x = 0.00$, (b) $x = 0.15$, (c) $x = 0.30$ and (d) $x = 0.45$.](image-url)
in Table 2. It can be seen from the Table 2 that $M_r/M_s$ increases with Cr content ($x$) from 0.408 at $x = 0.00$ to 0.687 at $x = 1.05$. This shows that Cr doping can affect the squareness of magnetic hysteresis loops greatly. With the increase of Cr content ($x$), the rising speed of squareness is much faster than the falling speed of $M_s$. Thus, $M_r$ of the magnetic powders first increases with Cr content ($x$) from 0 to 0.45, and then keeps constant with Cr content ($x$) from 0.45 to 0.75, and decreases when Cr content ($x$) $\geq$ 0.75.

Our results on the $M_r/M_s$ of Cr-doped hexaferrites magnetic powders are in agreement with that reported for the Al-Cr co-substituted M-type barium hexaferrites.33)

Figure 5 shows the variation of the $H_c$ as a function of Cr content ($x$) for the hexaferrite Sr$_{0.35}$Ca$_{0.30}$La$_{0.35}$Fe$_{12.00-x}$Cr$_x$O$_{19}$ magnetic powders. It is clear that $H_c$ of the magnetic powders initially enhances significantly with the increasing of Cr content ($x$) until it reaches to the maximum value of 3925 Oe at Cr content ($x$) = 0.60, and then decreases slowly when Cr content ($x$) $\geq$ 0.60. $H_c$ of the magnetic powders reaches the maximum value at Cr content ($x$) = 0.60. As is known, $H_c$ is inversely

| Cr content ($x$) | $M_r$ (emu/g) | $M_s$ (emu/g) | $H_c$ (Oe) | $M_r/M_s$ |
|------------------|---------------|---------------|------------|-----------|
| 0.00             | 45.53         | 18.59         | 1154       | 0.408     |
| 0.15             | 42.35         | 23.02         | 1760       | 0.544     |
| 0.30             | 41.19         | 23.76         | 2143       | 0.577     |
| 0.45             | 40.56         | 25.71         | 3693       | 0.634     |
| 0.60             | 39.93         | 25.83         | 3925       | 0.647     |
| 0.75             | 39.12         | 26.03         | 3596       | 0.665     |
| 0.90             | 37.15         | 25.27         | 3558       | 0.680     |
| 1.05             | 35.62         | 24.46         | 3533       | 0.687     |

Fig. 3. Magnetic hysteresis loops of the hexaferrite Sr$_{0.35}$Ca$_{0.30}$La$_{0.35}$Fe$_{12.00-x}$Cr$_x$O$_{19}$ magnetic powders for compositions of (a) $x = 0.60$, (b) $x = 0.75$, (c) $x = 0.90$ and (d) $x = 1.05$.

Fig. 4. Variation in the $M_t$ and $M_r$ with Cr content ($x$) for the hexaferrite Sr$_{0.35}$Ca$_{0.30}$La$_{0.35}$Fe$_{12.00-x}$Cr$_x$O$_{19}$ magnetic powders.
increases with Cr content (where the magnetoplumite structure, and only a small trace of fraction patterns demonstrate that the magnetic powders show obviously observed. When the Cr content (the amount of 3925 Oe at Cr content (the traditional ceramic route. The XRD analysis indicates that the non-magnetic phase, when Cr content (0.15, the 0.60, the dif-fraction patterns demonstrate that the magnetic powders show the magnetoplumite structure, and only a small trace of α-Fe₂O₃ with the increase of Cr content (α-Fe₂O₃, is the orientation factor, Mₓ is the amount of anisotropy constant. 35) When Cr³⁺ ions do the Fe³⁺ ions at 2a sites, there is also another notable change in the quadrupole splitting at 4f₁ site. 36) It is reported that the quadrupole splitting decreases at 4f₁ site and the magnetocrystalline anisotropy constant also decreases. 36) These changes are responsible for the decrease of the Hₓ with the increase of Cr content (α) when Cr content (α) ≥ 0.60. In this work, when Cr content (α) ≥ 0.15, the Hₓ values lie in the range from 1760 to 3925 Oe, which are above 1200 Oe. Thus, the prepared Cr-doped hexaferrite magnetic powders could be suitable for applications in the permanent magnets and recording media.

4. Conclusions

M-type hexagonal ferrites Sn₁₅Ca₃₀La₃₀Fe₁₂₀₋ₓCrₓOₓ₆ with Cr content (x) from 0.00 to 0.60 have been prepared by the traditional ceramic route. The XRD analysis indicates that when the Cr content (x) ≤ 0.60, non-magnetic phase, α-Fe₂O₃, is obviously observed. When the Cr content (x) ≥ 0.60, the diffraction patterns demonstrate that the magnetic powders show the magnetoplumite structure, and only a small trace of α-Fe₂O₃ is observed. The magnetic properties of the magnetic powders were investigated systematically. Mₓ linearly decreases with the increase of Cr content (x) from 0.00 to 1.05. While Mᵧ first increases with Cr content (x) from 0.00 to 0.75, and then decreases when Cr content (x) ≥ 0.75. Hₓ initially increases significantly with Cr content (x) from 0 to 0.60 and then decreases slowly when Cr content (x) ≥ 0.60. The Hₓ reaches to the maximum value of 3925 Oe at Cr content (x) = 0.60. Therefore, the prepared Cr-doped hexaferrites magnetic powders could be suitable for applications in the permanent magnets and recording media.

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