



Magnetic and microstructural properties of Al substituted M-type Ca–Sr hexaferrites



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ABSTRACT

In the current study, Al substituted M-type Ca–Sr hexaferrites, with composition $\text{Ca}_{0.6}\text{Sr}_{0.1}\text{La}_{0.3}\text{Fe}_{12-x}\text{Al}_x\text{O}_{19}$ ($0 \leq x \leq 1.4$), have been prepared using the conventional ceramic techniques. The phase composition of the magnetic powders was characterized by X-ray diffraction (XRD). The XRD data show that the magnetic powders with Al content (x) from 0 to 0.8 show $\alpha\text{-Fe}_2\text{O}_3$ as a second phase, while the magnetic powders with x from 1.0 to 1.4 consist of pure magnetoplumbite without any other impurity phases. A field emission scanning electron microscopy (FE-SEM) was used to observe the morphologies of the magnetic powders. FE-SEM images of the magnetic powders show the hexagonal platelet-like shape. The magnetic properties of the magnetic powders were measured by a physical property measurement system–vibrating sample magnetometer (PPMS–VSM). The saturation magnetization (M_s) linearly decreases with Al content (x) from 0 to 1.4. While the remanent magnetization (M_r) first increases with Al content (x) from 0 to 0.2 and then decreases when Al content (x) ≥ 0.2 . However, the coercivity (H_c) increases with Al content (x) from 0 to 1.4.

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1. Introduction

M-type hexaferrites $\text{MFe}_{12}\text{O}_{19}$ ($\text{M} = \text{Ba}, \text{Sr}, \text{Pb}$) are increasingly a popular topic in technological applications [1]. They have been widely used in many fields, such as permanent magnets, microwave devices, magneto-optics and magnetic recording media [2]. M-type hexaferrites are preferred due to their large uniaxial magnetocrystalline anisotropy, excellent chemical stability, corrosion resistivity and low price [3,4]. Magnetic properties of hexaferrites are influenced by the synthesis method, changing of chemical composition and cation distribution in the crystal lattice. As it is well known, the M-type hexaferrite structure is represented by alternate stack of hexagonal and spinel layers, $\text{MFe}_6\text{O}_{11}^{2-}$ and $\text{Fe}_6\text{O}_8^{2+}$, respectively. In the structure of M-type hexaferrite, the unit cell contains 38 O^{2-} ions, 2 M^{2+} ions and 24 Fe^{3+} ions. The Fe^{3+} ions occupy five different crystallographic sites such as three octahedral (2a, 12k and 4f), one tetrahedral site (4f), and one bipyramidal site (2b). In addition, 2a, 2b and 12k are spin up sites, while 4f and 4f are spin down sites [1]. These sites are coupled by the $\text{Fe}^{3+}\text{--O--Fe}^{3+}$ super-exchange interaction leading to the ferromagnetic structure [5].

Therefore, any further enhancement to the magnetic properties of M-type hexaferrites is of relevance for technological innovation and larger market. Many attempts have been done to improve the magnetic properties of M-type hexaferrites by cation substitution [6–18]. It has been reported that the coercivity of M-type hexaferrites can be increased by partial substitutions of Sr^{2+} or Ba^{2+} ions by La^{3+} , Nd^{3+} and Sm^{3+} ions, and of Fe^{3+} ions by Cr^{3+} and Al^{3+} ions [5–13]. Babu et al. have reported that the sintered product of barium hexaferrite powders with La_2O_3 has higher coercivity force than that of barium hexaferrite powders without La_2O_3 [6]. Luo et al. have reported that the specific saturation magnetization (M_s) and the coercivity of strontium ferrites could be improved by substitutions of Nd^{3+} ions on Fe^{3+} ions basis sites [7]. Katlakunta et al. have studied the magnetic properties of Cr^{3+} doped $\text{SrFe}_{12}\text{O}_{19}$ and found that with Cr^{3+} doping saturation magnetization systematically decreased and coercivity increased [9]. Wang et al. have reported the improvement of the coercivity of strontium induced by substitution of Al^{3+} ions for Fe^{3+} ions [11]. Nga et al. have studied the magnetic properties of ultrafine Al-substituted strontium hexaferrite particles prepared by citrate sol-gel method and found that the saturation magnetization decreases while the coercivity increases with increasing the Al content [13]. It is also reported that La–Co substitutions can improve both the saturation magnetization or remanence and coercivity, while La–Zn or La–Cu substitutions lead to the increase of the magnetization

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or remanence and the decrease of the coercivity [16–18]. It is well known calcium is more abundant than strontium and barium on the earth. Using cheap Ca^{2+} ions to replace Sr^{2+} or Ba^{2+} ions is a useful way to reduce cost and expand the material sources. There are few previous reports about M-type hexaferrites with calcium substitution [19–22]. Li et al. [19] have prepared the calcium substituted SrLaCo hexaferrite magnetic powder by the ceramic process. Yang et al. have studied the influence of calcium content on the structural and magnetic properties of $\text{Sr}_{0.70-x}\text{Ca}_x\text{La}_{0.30}\text{Fe}_{11.75}\text{Zn}_{0.25}\text{O}_{19}$ hexaferrites by the ceramic process [22].

M-type hexaferrites have been synthesized by various methods, which include the ceramic process [23], the hydrothermal method [8], the sol–gel method [24], the chemical coprecipitation method [25], the citrate precursor method [26] and the self-propagating high temperature synthesis (SHS) route [27]. However, 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 [28].

In this study, the M-type hexaferrite $\text{Ca}_{0.6}\text{Sr}_{0.1}\text{La}_{0.3}\text{Fe}_{12-x}\text{Al}_x\text{O}_{19}$ magnetic powders with Al content (x) from 0 to 1.4 have been prepared using the conventional ceramic techniques. We have systematically investigated the influence of Al content on the microstructural and magnetic properties of M-type Ca–Sr hexaferrites.

2. Experimental procedure

M-type hexaferrite samples have been synthesized by the conventional ceramic techniques. Compositions were chosen to according to the chemical composition $\text{Ca}_{0.6}\text{Sr}_{0.1}\text{La}_{0.3}\text{Fe}_{12-x}\text{Al}_x\text{O}_{19}$, where Al content (x) varied from 0 to 1.4 with about 0.2 increment. All the starting materials used (CaCO_3 , SrCO_3 , La_2O_3 , Fe_2O_3 and Al_2O_3) were powders of analytical grade. Firstly, mixed powders of the precursor materials prepared by the wet-mixing method were calcined in a muffle furnace at 1260 °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 muffle furnace at 750 °C for 1.0 h in order to relieve the stress.

The phase composition of the magnetic powders was characterized by a PANalytical X'Pert Pro diffractometer in continuous mode using $\text{Cu K}\alpha$ ($\lambda=1.5406 \text{ \AA}$). Morphologies of the magnetic powders were observed by a HITACHI S-4800 field emission scanning electron microscopy (FE-SEM). Magnetic hysteresis loops of the magnetic powders were measured at room temperature using a physical property measurement system-vibrating sample magnetometer (PPMS-VSM, Quantum Design) with a magnetic field of 15,000 Oe.

3. Results and discussion

3.1. X-ray diffraction analysis

The X-ray diffraction patterns of the hexaferrite $\text{Ca}_{0.6}\text{Sr}_{0.1}\text{La}_{0.3}\text{Fe}_{12-x}\text{Al}_x\text{O}_{19}$ magnetic powders with Al content (x) from 0 to 1.4 are given in Fig. 1. It can be seen that the XRD patterns of the magnetic powders with Al content (x) from 0 to 0.8 show $\alpha\text{-Fe}_2\text{O}_3$ as a second phase, while the magnetic powders with Al content (x) ≥ 1.0 are in good agreement with the M-type strontium ferrite JCPDS card no. 80-1198 and show a single phase hexagonal structure. This result indicates that Al^{3+} ions are all incorporated into the lattice of M-type hexaferrite. The presence of $\alpha\text{-Fe}_2\text{O}_3$ for the magnetic powders with Al content (x) from 0 to

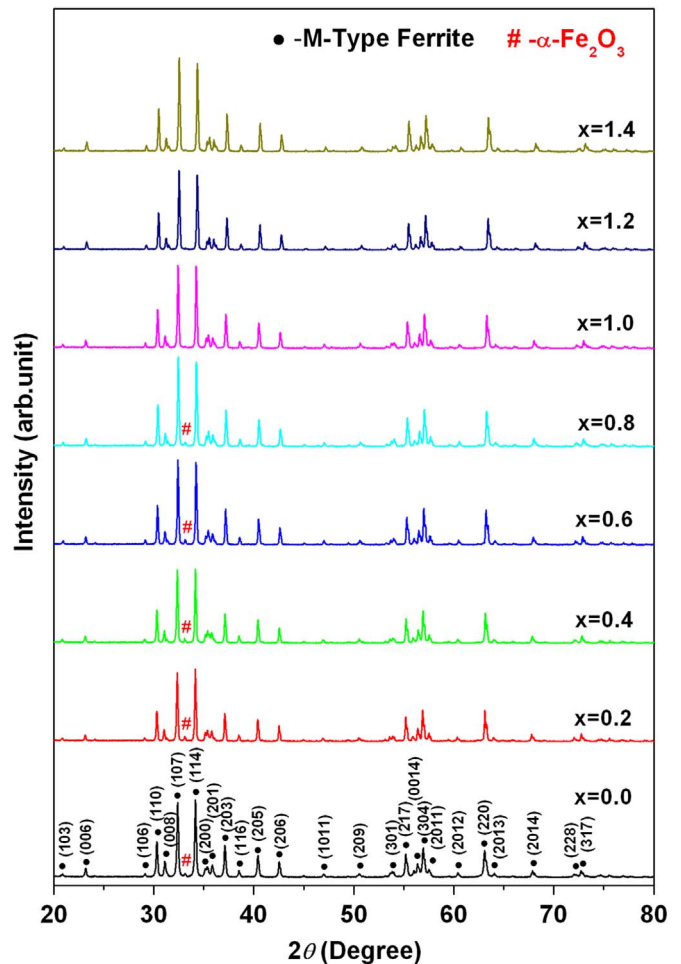


Fig. 1. X-ray diffraction patterns of the hexaferrite $\text{Ca}_{0.6}\text{Sr}_{0.1}\text{La}_{0.3}\text{Fe}_{12-x}\text{Al}_x\text{O}_{19}$ magnetic powders with Al content (x) from 0 to 1.4.

0.8 might be attributed to the incomplete reaction under synthesis conditions.

The lattice constants a and c of the hexaferrite $\text{Ca}_{0.6}\text{Sr}_{0.1}\text{La}_{0.3}\text{Fe}_{12-x}\text{Al}_x\text{O}_{19}$ magnetic powders with Al content (x) from 0 to 1.4 are calculated using the following equation:

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

where d_{hkl} is the inter-planer spacing value, and the values of h , k and l are the Miller indices. Fig. 2 displays the lattice constants a

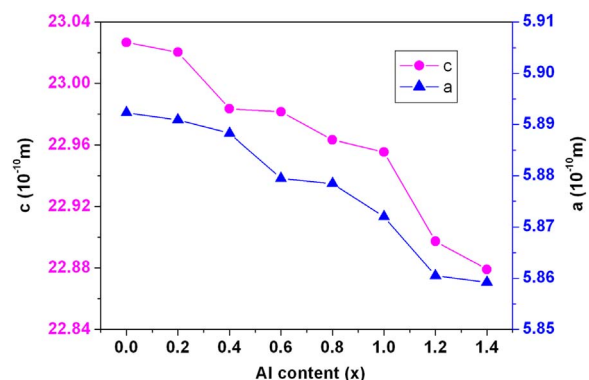


Fig. 2. Lattice constants a and c of the hexaferrite $\text{Ca}_{0.6}\text{Sr}_{0.1}\text{La}_{0.3}\text{Fe}_{12-x}\text{Al}_x\text{O}_{19}$ magnetic powders with Al content (x) from 0 to 1.4.

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