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Analysis of magnetic disaccommodation in La^{3+}–Co^{2+}-substituted strontium ferrites

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A B S T R A C T

M-type strontium ferrites substituted by La^{3+}–Co^{2+}($Sr_{1-x}La_xFe_{12-x}Co_xO_{19}$) were prepared by ceramic process. Effects of the substituted amount of La^{3+} and Co^{2+} on structure and magnetic properties of $Sr_{1-x}La_xFe_{12-x}Co_xO_{19}$ compounds have systematically been investigated by X-ray diffraction (XRD), vibrating sample magnetometer (VSM) and magnetic disaccommodation. In the measurement range from 80 to 500 K, the magnetic disaccommodation is represented by means of isochronal curves. It is well known that magnetic disaccommodation cannot be obviously found in the M-type of pure strontium ferrites. However, three peaks were observed in $Sr_{1-x}La_xFe_{12-x}Co_xO_{19}$, and this behavior is explained in terms of the presence of Fe^{2+} cation and to the site occupation by the magnetic Co^{2+} ionic within the hexagonal structure.

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1. Introduction

M-type strontium hexaferrites have been used as permanent magnets for years. Moreover, they also have been widely used in microware devices and magneto-optic or high density recording media [1,2]. The magnetic properties of the ferrites, such as intensity of self-magnetization, coercive force or the magnetic anisotropy are due to the composition and ionic distribution. Several kinds of techniques can be used to prepare M-type ferrites, such as the ceramic process [3], the sol–gel method [4] or the chemical deposition technique [5]. However, the ceramic process is usually applied in industry manufacture. In order to fulfill various applications, the performance of the M-type hexaferrites must be improved. Ion substitution technique is used to enhance the performance of the M-type hexaferrites. Doping or combinatorial doping is used to carry out ion substitution. This is one of the important techniques which can be used to investigate the exchange function of the magnetic materials, the magnetic anisotropy, some other intrinsic properties and improve the performance of the permanent magnets. In 1970s, scientists discovered that appending rare-earth La^{3+} and transition Co^{2+} can improve the performance of the permanent magnetic hexagonal ferrites [6,7].

In this work, we have prepared La–Co substituted M-type strontium hexaferrite ($Sr_{1-x}La_xFe_{12-x}Co_xO_{19}$) by applying the ceramic process, then the samples were processed with demagnetization at temperatures in the $80\,K<T<500\,K$ range. We have systematically studied the influences of La^{3+}–Co^{2+} substituted amount on the magnetic disaccommodation of the samples. The magnetic disaccommodation is a relaxation phenomenon, well known in spinel ferrites [8,9] due to the simultaneous presence of ferrous cations and lattice vacancies, which promotes electronic and ionic reorientations in the Bloch walls in order to minimize the free-energy. As the electrons or cations diffuse to the advantageous sites, the magnetic domain walls are stabilized in the potential pits, and these processes induce the relaxation of the initial permeability, leading to a time-dependent decrease of initial permeability after demagnetization. This undesirable effect has been proved to be useful in the detection of minute impurities [9] and there are works that have extended the application range of this technique to hexaferrites and magnetic garnets [10].

2. Experimental

In this study, the initial materials, $SrCO_3$ (98%, industry purity), La_2O_3 (98%), Fe_2O_3 (99%), and Co_2O_3 (98.5%) were mixed together in the composition of $Sr_{1-x}La_xFe_{12-x}Co_xO_{19}$, where x varies from 0.05 to 0.20 with 0.05 increment. The mixtures of these raw materials were milled in water for 2 h with an angular velocity of 200 rpm and a ball to power weight ratio of 8:1. The milling

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processes were performed in a centrifugal planetary ball mill using three sizes of hardened steel balls with diameters of 8, 10, and 12 mm. The mixed powder was dried, crushed, and sifted. These samples were shaped into balls of about Φ8 mm, presintered in a furnace at temperatures up to 1250–1300 °C in air, and then were cooled in the furnace down to room temperature. The pre-calcined samples were dry-milled using a vibration mill, then wet-milled with additives ($CaCO_3$, SiO_2, Al_2O_3 or Cr_2O_3) using an attritor. The finely milled slurry with a diameter of about 0.8 μm was pressed into disk-shaped compacts in the magnetic field of 8×10^2 kA/m parallel to the pressing direction. The pressed compacts were sintered in a muffle from 1207 to 1231 °C in air atmosphere, keeping the top temperature about 1 or 2 h. Finally, they were quenched in the muffle.

The powder X-ray diffraction (XRD) patterns were collected on a Mac Science (MXP18AHF) powder X-ray diffractometer (using Cu-Kα radiation 0:15418 nm). By the way, we measured the magnetic properties of the samples with a Riken Denshi (BH-55) vibrating sample magnetometer (VSM). Magnetic disaccommodation measurements were carried out with a computer-aided system based on an automatic impedance meter (LCR bridge), by measuring the time evolution of both real and imaginary parts of the impedance of a coil wound around the sample after AC demagnetization process with a decreasing alternating field with top amplitude adjusted to overcome coercive field of the sample. The magnetic relaxation is measured at fixed temperatures in the 80–500 K range, and then represented as isochronal curves by plotting the relative variation of the initial permeability after sample demagnetization between an initial time $t_1 = 2$ s and different window times $t_2 = 54$, 8, 16, 32, 64, and 128 s in the form

$$\frac{\mu(t_1, T) - \mu(t_2, T)}{\mu(t_1, T)} (\%) \tag{1}$$

When the time window (t_2–t_1) is of the same order of magnitude as the relaxation time at a specified temperature, this curve exhibits a maximum. In this way, isochronal spectra reveal the different after-effect processes in the temperature range tested.

3. Result and discussion

X-ray diffraction patterns for doped samples $Sr_{1-x}La_xFe_{12-x}Co_xO_{19}$ ($x = 0.05-0.20$) sintered in air at 1215 °C are shown in Fig. 1. It is clear that the samples are composed of single phase of magnetoplumbite crystal structure, i.e., hexagonal M-phase ferrite. A representative hysteresis loop measured at room temperature, corresponding to the sample with doping rate $x = 0.15$ ($Sr_{0.85}La_{0.15}Fe_{11.85}Co_{0.15}O_{19}$) sintered at 1215 °C, is shown in Fig. 2. It can be induced from Fig. 2. This samples have a relative high remanent magnetization ($Mr = 500$ emu/cm^3) regarding pure Sr hexaferrite, together with a low coercive field ($Hcj = 0.58$ kOe). Moreover, we can see that the hysteresis loop presents rectangle shape, so it suits to the application of microwave device. With this result in mind, we have adjusted the top demagnetizing field in the magnetic disaccommodation measurements. In addition, it can be inferred that with lower coercive fields the magnetic relaxation effects analyzed below become more prominent, as the hexaferrites analyzed are magnetically softer, and magnetic disaccommodation effects are connected to wall dynamics.

The isochronal curves for doped samples $Sr_{1-x}La_xFe_{12-x}Co_xO_{19}$ ($x = 0.05$–0.20) sintered in air at 1215 °C are shown in Fig. 3. It can be made out that magnetic disaccommodation phenomena occurs in the doped samples, i.e., the relaxation process takes place in the samples. We observe three peaks (A, B, A_0) at different temperatures: a peak is centered at about 300 K, and its amplitude keeps almost constant in the compositions analyzed. The B peak is centered at about 180 K, and its amplitude makes this process almost the preponderant one. A_0 peak is not centered in the $80\,K < T < 500\,K$ range, but we can affirm its existence and we can see that its amplitude raises as the substituted amount x increases. These three peaks represent three different relaxation processes.

The magnetic after-effect phenomena are strongly coupled to the presence of both anisotropic cations, usually ferrous cations in octahedral sites, and lattice vacancies. This type of magnetic relaxation processes is due to anisotropic reorientations of vacancy and interstitial-type point defects in the crystal lattice (orientational processes), as well as diffusion of anisotropic cations via lattice vacancies (diffusional processes) [8]. The reorientation of point defects within the Bloch walls creates a

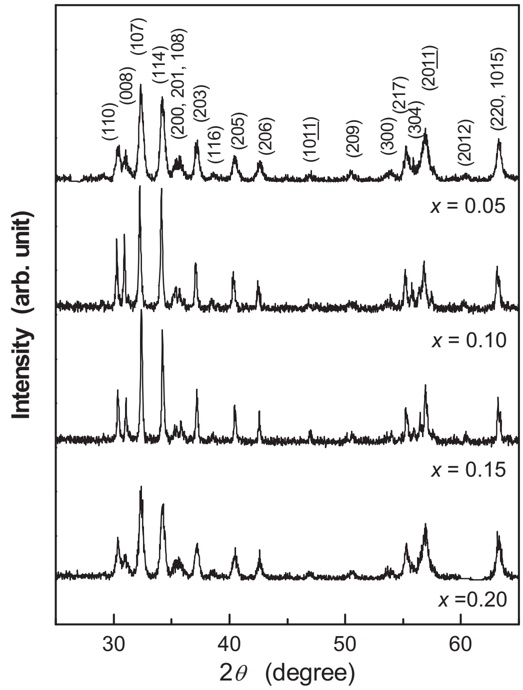

Fig. 1. X-ray diffraction patterns of the sintered powders of $Sr_{1-x}La_xFe_{12-x}Co_xO_{19}$ ($x = 0.05$–0.20).

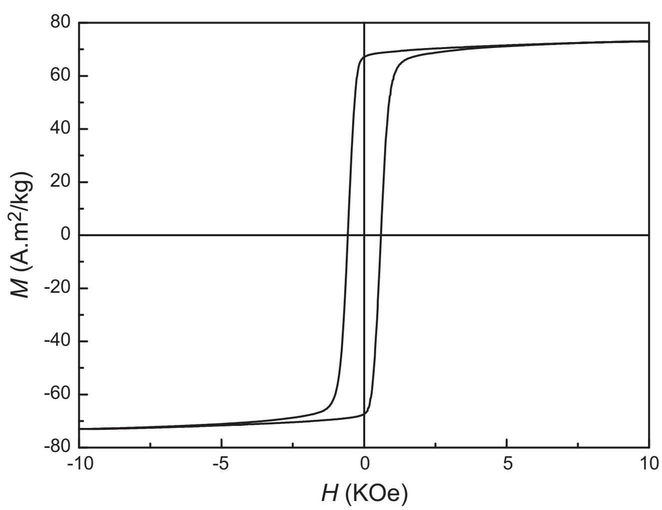

Fig. 2. Hysteresis loop for the sample $Sr_{1-x}La_xFe_{12-x}Co_xO_{19}$ ($x = 0.05$–0.20) ($x = 0.15$) measured at room temperature under the sintering temperature with 1215 °C.

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