

## Magnetic properties of La-substituted Ni–Zn–Cr ferrites via rheological phase synthesis

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### ABSTRACT

Nanocrystalline ferrites  $\text{Ni}_{0.6}\text{Zn}_{0.4}\text{Cr}_{0.5}\text{La}_x\text{Fe}_{1.5-x}\text{O}_4$  ( $x=0-0.10$ ) were prepared by a rheological phase-reaction process. The influence of the La content on microstructure and magnetic properties of samples were investigated. The structure, morphology and magnetic properties of the obtained products were characterized by X-ray diffractometer (XRD), transmission electron microscope (TEM), vibrating sample magnetometer (VSM) and network analyzer (NA). XRD results showed that the substituting  $\text{La}^{3+}$  ions had a solubility limit in the spinel lattice. The present work showed that the crystallite size and magnetic properties could be tuned over by introducing  $\text{La}^{3+}$  ions into the Ni–Zn–Cr ferrites. The crystallite size, saturation magnetization of samples decreased with the increase of La content, whereas the variation of coercivity was reverse. Moreover, the microwave absorption ability of the samples can be improved by changing the components of the ferrites.

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### 1. Introduction

Ni–Zn ferrites are one of the most versatile soft magnetic materials. Recently, the technological application of materials has been studied extensively [1–4], primarily due to their applicability in many electronic devices, with high permeability at high frequency, remarkably high-electrical resistivity, low-eddy current loss and reasonable cost [5–8]. The replacement of  $\text{Fe}^{3+}$  by  $\text{Cr}^{3+}$  in the Ni–Zn ferrite could effectively reduce porosity and grain size whereas the bulk density increases [9], so the appropriate content of Cr substitution is propitious to obtain a less grain size. It is well known that rare earth ions with larger ionic radius affect the physical properties of substituted Ni–Zn ferrite drastically. When these ions enter the octahedral (B site), they can partly replace  $\text{Fe}^{3+}$  ions [10]. In this case, the difference in the ionic radii will lead to microstrains, which may cause domain wall motion resulting in deformed spinel structure. For instance, the relative density of sintered bodies decreases with increasing R content in  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_{2-x}\text{R}_x\text{O}_4$  ( $\text{R}=\text{La}$  or  $\text{Gd}$ ,  $x=0-0.04$ ), while the variation of lattice parameter is reverse [11]. The addition of superfluous rare earth ions in  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{R}_{0.02}\text{Fe}_{1.98}\text{O}_4$  ( $\text{R}=\text{Y}$ ,  $\text{Gd}$  or  $\text{Eu}$ ) would enlarge local distortion and disorder [12]. Our

previous works indicated that the particle sizes and magnetic properties of  $\text{Zn}_{0.6}\text{Cu}_{0.4}\text{Cr}_{0.5}\text{Sm}_x\text{Fe}_{1.5-x}\text{O}_4$  ( $x=0.000, 0.025, 0.050, 0.075$  and  $0.100$ ) powders were strongly affected by Sm content [13]. However, the effect of La substitution on the structure, morphology and magnetic properties for Ni–Zn–Cr ferrites was little reported. Therefore, we had the motivation to synthesize La-substituted Ni–Zn–Cr ferrite which was suitable for soft magnet applications.

Many methods can be used to prepare soft ferrites, such as mechano-chemical method [14], chemical co-precipitation [15], hydrothermal method [16], sol-gel method [17], microemulsion method [18], rheological phase-reaction method [19], and so on. Compared to other methods, rheological phase-reaction method is an economical, effective, simple route for preparing materials from a solid–liquid rheological system, without stirring and repeated heat treatment. In virtue of uniform distribution and close contact between solid and liquid particles in rheological mixture, heat exchange can be carried out easily and quickly, and the solid particles can be utilized efficiently, so the reaction is more complete. Besides, it can reduce the quantity of generated solid waste, and has great potential application in green chemical reaction. In our early researches, Gd-doped Li–Ni ferrites and La-, Sm-substituted Zn–Cu–Cr ferrites were prepared by this method [13,20,21]. In present work, La-substituted Ni–Zn–Cr ferrites had been obtained via rheological phase-reaction method, and the structure, morphology and magnetic properties of the obtained products were investigated.

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## 2. Experimental

The starting materials, ZnO, Cr<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, NiO, La<sub>2</sub>O<sub>3</sub> and H<sub>2</sub>C<sub>2</sub>O<sub>4</sub>·2H<sub>2</sub>O, were all analytical grade. The Ni<sub>x</sub>Zn<sub>1-x</sub>Cr<sub>0.5</sub>Fe<sub>1.5</sub>O<sub>4</sub> and Ni<sub>0.6</sub>Zn<sub>0.4</sub>Cr<sub>0.5</sub>Fe<sub>1.5-x</sub>La<sub>x</sub>O<sub>4</sub> ( $x = 0.00, 0.02, 0.04, 0.06, 0.08$  and  $0.10$ ) were synthesized by the rheological phase-reaction method. For the preparation of Ni<sub>0.6</sub>Zn<sub>0.4</sub>Cr<sub>0.5</sub>Fe<sub>1.48</sub>La<sub>0.02</sub>O<sub>4</sub> (0.01 mol), the stoichiometric amounts of NiO (0.006 mol), ZnO (0.004 mol), Cr<sub>2</sub>O<sub>3</sub> (0.0025 mol), Fe<sub>2</sub>O<sub>3</sub> (0.0074 mol), La<sub>2</sub>O<sub>3</sub> (0.0001 mol) and H<sub>2</sub>C<sub>2</sub>O<sub>4</sub>·2H<sub>2</sub>O (0.04 mol) were thoroughly mixed by grinding in an agate mortar for 30 min, then 15 ml anhydrous ethanol was added to form the mixture in rheological state. The mixture was sealed in a teflonlined stainless-steel autoclave, and maintained at 100 °C for 48 h in an oven. The obtained precursors were washed several times with deionized water and anhydrous ethanol, respectively, dried at 60 °C for 12 h, and sintered at 1000–1200 °C for 2 h in air, followed by cooling in furnace to room temperature with 5 °C min<sup>-1</sup> cooling rate, then the well-designed sample was obtained. Other samples were synthesized as described above.

The chemical compositions of samples were determined by an IRIS Intrepid II XDL inductively coupled plasma atomic emission spectrometer (ICP-AES). The structure of samples was determined by Philips-PW3040/60 model X-ray diffractometer (XRD) using Cu K $\alpha_1$  radiation ( $\lambda = 0.15405$  nm) at a scanning speed of 4° min<sup>-1</sup> in the range of  $2\theta = 15$ –80°. Magnetization measurements were performed by using a vibrating sample magnetometer (VSM, Lakeshore 7404) in 10 kOe applied field at room temperature. The morphology and particle sizes of the obtained products were characterized by transmission electron microscope (TEM, JEOL-2010). The magnetic loss and dielectric loss were obtained by network analyzer (NA, HP4991A) in the range of 2–18 GHz.

## 3. Results and discussion

### 3.1. XRD analysis

The XRD patterns of Ni<sub>x</sub>Zn<sub>1-x</sub>Cr<sub>0.5</sub>Fe<sub>1.5</sub>O<sub>4</sub> ferrites ( $x = 0$ –1.0) calcined at 1000 °C for 2 h were shown in Fig. 1. All samples had characteristic peaks of the spinel Ni–Zn ferrite reported in the standard card (ICDD PDF #80-1687). Meanwhile, the second phase Fe<sub>2</sub>O<sub>3</sub> ( $2\theta = 33.28^\circ$ , ICDD PDF #86-2368) increased with the zinc content. It could be seen from Fig. 1 that a well-crystalline single phase (pattern d) occurred in Ni<sub>x</sub>Zn<sub>1-x</sub>Cr<sub>0.5</sub>Fe<sub>1.5</sub>O<sub>4</sub> at  $x = 0.6$ , the intensity of the characteristic peaks decreased and the width increased, compared with that of other patterns in Fig. 1. It indicated that the sample of pattern d had smaller particle size, so Ni<sub>0.6</sub>Zn<sub>0.4</sub>Cr<sub>0.5</sub>Fe<sub>1.5</sub>O<sub>4</sub> was the best suitable composition in Ni–Zn ferrite.

The X-ray diffraction patterns of Ni<sub>0.6</sub>Zn<sub>0.4</sub>Cr<sub>0.5</sub>La<sub>0.02</sub>Fe<sub>1.48</sub>O<sub>4</sub> ferrites sintered at different temperatures were shown in Fig. 2. When the as-obtained precursors were treated at 1000 and 1100 °C

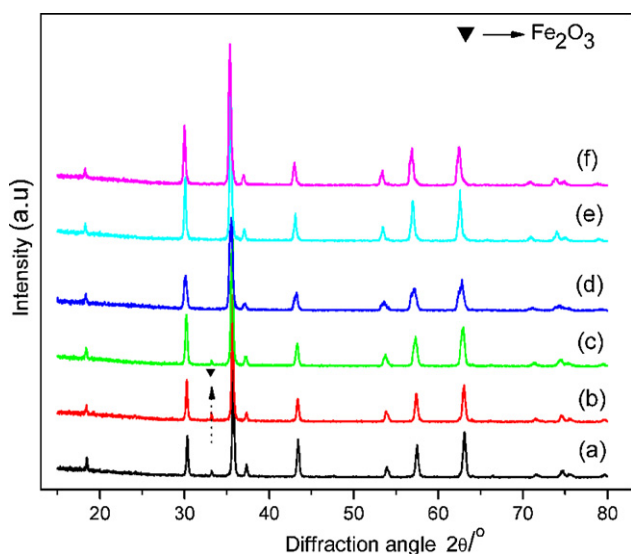


Fig. 1. XRD patterns for Ni<sub>x</sub>Zn<sub>1-x</sub>Cr<sub>0.5</sub>Fe<sub>1.5</sub>O<sub>4</sub> sintered at 1000 °C: (a) 0.0, (b) 0.2, (c) 0.4, (d) 0.6, (e) 0.8 and (f) 1.0.

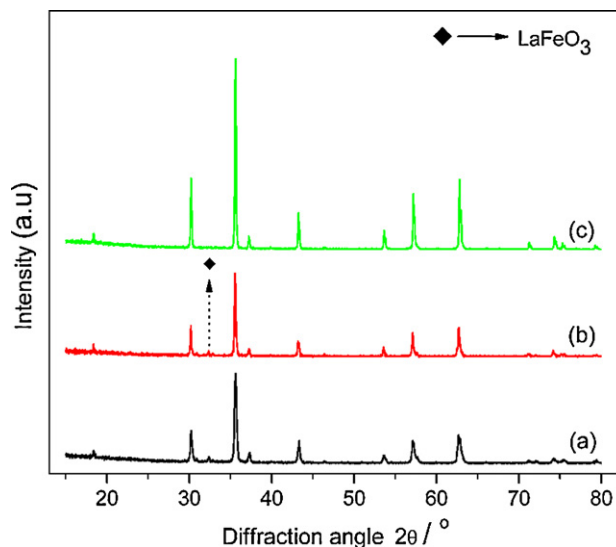


Fig. 2. XRD patterns for Ni<sub>0.6</sub>Zn<sub>0.4</sub>Cr<sub>0.5</sub>La<sub>0.02</sub>Fe<sub>1.48</sub>O<sub>4</sub> sintered at different temperatures: (a) 1000 °C, (b) 1100 °C and (c) 1200 °C.

the samples had a small amount of secondary phases identified as LaFeO<sub>3</sub> (ICDD PDF #74-2203) except for the cubic spinel phase. While the sintering temperature was up to 1200 °C, there was no second phase left in the samples. Meanwhile, the growth of crystallites led to the intensity of the characteristic peaks increasing and their width decreasing as shown in Fig. 2(c) [22]. Herein, for the sake of avoiding the LaFeO<sub>3</sub> phase, the best suitable sintering temperature to prepare La-substituted Ni–Zn–Cr ferrites was 1200 °C.

Fig. 3 was the XRD patterns of Ni<sub>0.6</sub>Zn<sub>0.4</sub>Cr<sub>0.5</sub>Fe<sub>1.5-x</sub>La<sub>x</sub>O<sub>4</sub> (with  $x = 0, 0.02, 0.04, 0.06, 0.08$  and  $0.10$ ) ferrites calcined at 1200 °C for 2 h. It can be learned from Fig. 3 that the intensity of LaFeO<sub>3</sub> phase increased gradually at La content  $\geq 0.06$ . The crystallite size calculated by the Debye–Scherrer formula decreased with the increase of La content shown in Fig. 4. The presence of the secondary phase in the NiZnCrLa ferrites indicated that the La substitution had a solubility limit in the spinel lattice. A possible explanation was that the radius of La<sup>3+</sup> ions (0.106 nm) was larger than that of Fe<sup>3+</sup> ions (0.064 nm), and the amount of Fe<sup>3+</sup> ions substituted by La<sup>3+</sup> ions had a limit [23], which obeyed the Vegard's law, so the redundant

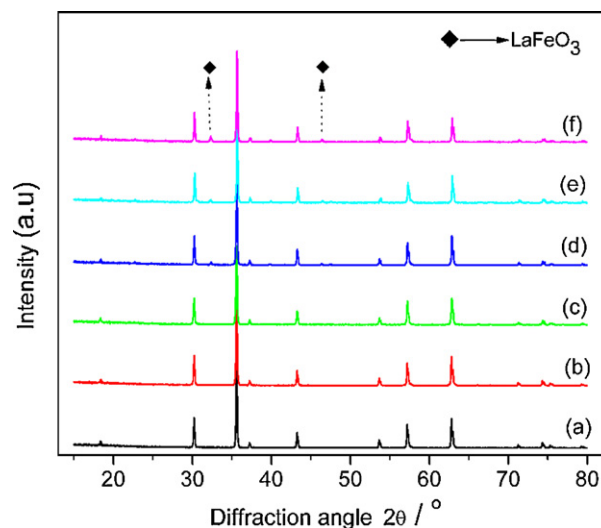


Fig. 3. XRD patterns for Ni<sub>0.6</sub>Zn<sub>0.4</sub>Cr<sub>0.5</sub>Fe<sub>1.5-x</sub>La<sub>x</sub>O<sub>4</sub> sintered at 1200 °C: (a) 0.00, (b) 0.02, (c) 0.04, (d) 0.06, (e) 0.08 and (f) 0.10.

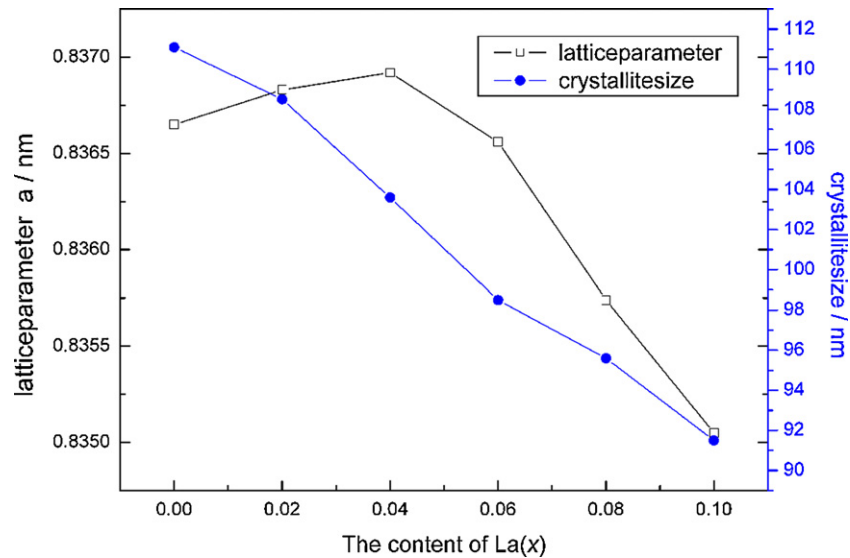


Fig. 4. The lattice parameter 'a' and crystallite size depend on the content of La.

$\text{La}^{3+}$  ions will aggregate on grain boundaries forming the secondary phase ( $\text{LaFeO}_3$ ).

The lattice parameter 'a' shown in Fig. 4 increased with the  $\text{La}^{3+}$  ions ( $x \leq 0.04$ ) and subsequently decreased with the increase of La content. It can be explained by the introduction of the larger  $\text{La}^{3+}$  ions into the spinel lattice, making the lattice expand. While a plausible explanation for decrease in 'a' was the compression of the spinel lattice induced by the secondary phases due to the differences in the thermal expansion coefficients [24].

### 3.2. Morphology analysis

Fig. 5 presented the size and morphology of as-synthesized  $\text{Ni}_{0.6}\text{Zn}_{0.4}\text{Cr}_{0.5}\text{Fe}_{1.5}\text{O}_4$  and  $\text{Ni}_{0.6}\text{Zn}_{0.4}\text{Cr}_{0.5}\text{La}_{0.04}\text{Fe}_{1.46}\text{O}_4$  particles sintered at  $1200^\circ\text{C}$  observed by TEM. The crystallite size was in the range of 90–120 nm, consistent with the results calculated by XRD patterns using the Scherrer formula. It could be seen from Fig. 5 that the particles were spherical and appeared to be a little agglomerated. Meanwhile, the crystallite size observed in Fig. 5(b) was

smaller than that shown in Fig. 5(a), so it was indicated the introduction of  $\text{La}^{3+}$  led to the decrease in crystallite size of the samples [25]. The corresponding ED patterns for the  $\text{Ni}_{0.6}\text{Zn}_{0.4}\text{Cr}_{0.5}\text{Fe}_{1.5}\text{O}_4$  and  $\text{Ni}_{0.6}\text{Zn}_{0.4}\text{Cr}_{0.5}\text{La}_{0.04}\text{Fe}_{1.46}\text{O}_4$  particles were also given in the inset of Fig. 5(a) and (b). It was obvious that the rings of ED patterns in Fig. 5(a) were clearer and brighter than that shown in Fig. 5(b), which indicated the crystallization of the former sample was more complete than that of latter one. Due to the larger bond energy of  $\text{La}^{3+}-\text{O}^{2-}$  as compared to that of  $\text{Fe}^{3+}-\text{O}^{2-}$ , the more energy was needed to make  $\text{La}^{3+}$  ions enter into lattice and form the bond of  $\text{La}^{3+}-\text{O}^{2-}$ . Therefore, La-substituted ferrites had higher thermal stability relative to pure Ni–Zn–Cr ferrite, and more energy was needed for the La-substituted samples to complete crystallization and grow grains [26].

### 3.3. Magnetic property analysis

The magnetic hysteresis loops of the samples with different contents of La substitution sintered at  $1200^\circ\text{C}$  were shown in Fig. 6. The

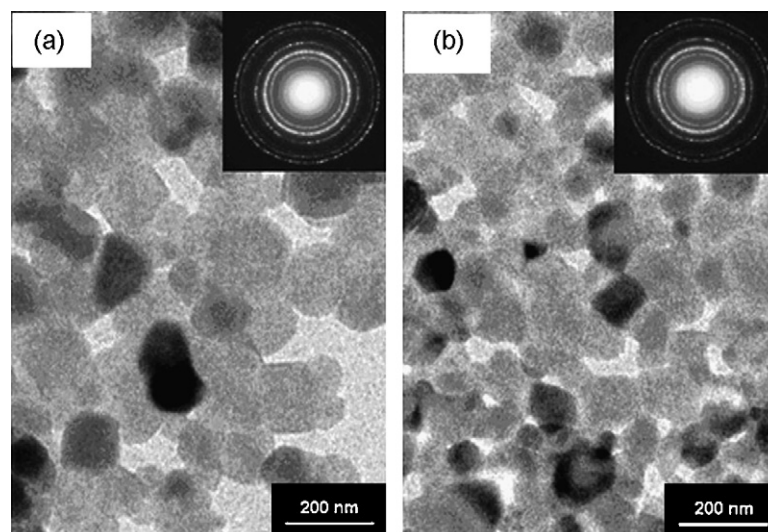
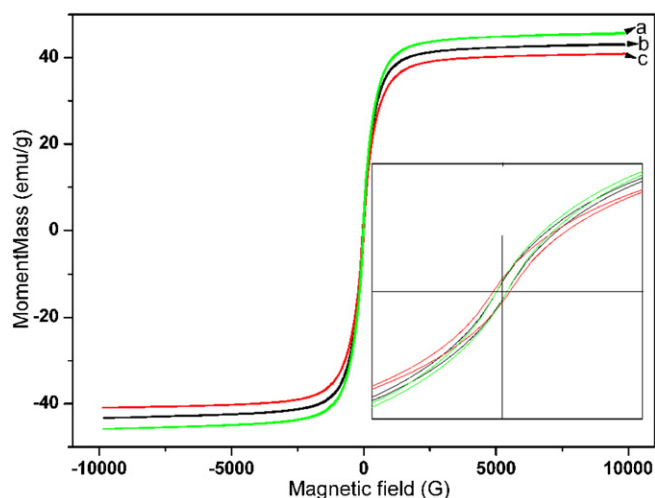


Fig. 5. TEM images and SAED ring patterns of samples: (a)  $\text{Ni}_{0.6}\text{Zn}_{0.4}\text{Cr}_{0.5}\text{Fe}_{1.5}\text{O}_4$  and (b)  $\text{Ni}_{0.6}\text{Zn}_{0.4}\text{Cr}_{0.5}\text{La}_{0.04}\text{Fe}_{1.46}\text{O}_4$ .

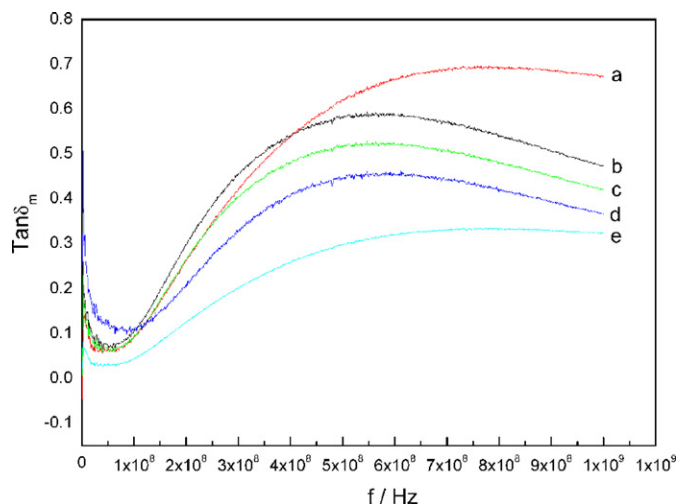


**Fig. 6.** Hysteresis loops of  $\text{Ni}_{0.6}\text{Zn}_{0.4}\text{Cr}_{0.5}\text{La}_x\text{Fe}_{1.5-x}\text{O}_4$  sintered at  $1200^\circ\text{C}$ . (a)  $x=0.00$ , (b)  $x=0.02$  and (c)  $x=0.04$  at sintering temperature  $1200^\circ\text{C}$ .

value of  $H_c$ ,  $M_s$  was listed in Table 1. It was found from Fig. 6 that the saturation magnetization  $M_s$  decreased with the increase of La content, while the coercive force  $H_c$  increased. It was well known that the magnetic properties of ferrites with  $\text{AB}_2\text{O}_4$  spinel crystal structure depended on the composition of cation in tetrahedral A site and octahedral B site. The radius of  $\text{La}^{3+}$  ions was larger than that of others metal ions in the as-obtained products and the inter-spaces of tetrahedral sites (A) was smaller than that of octahedral site (B), so  $\text{La}^{3+}$  ions was prior to occupy the octahedral sites (B). The main contribution of magnetic properties came from  $\text{Fe}^{3+}$  on B sites of ferrosinell, the net overall magnetic moment ( $m = m_B - m_A$ ) decreased when the non-magnetic  $\text{La}^{3+}$  ions replaced  $\text{Fe}^{3+}$  ions on B sites, meanwhile the superexchange interactions would decrease with the increase of doped  $\text{La}^{3+}$  ions content. Besides, spin canting is usually caused by rare earth ions. The colinear ferrimagnetic order transforming into non-colinear arrangement and disruption of ferrimagnetic order occurs because of introducing  $\text{La}^{3+}$  into the octahedral site, leading a decrease in  $M_s$ . As a result, the saturation magnetization decreased as the non-magnetic  $\text{La}^{3+}$  ions content increased. Moreover, the symmetry of crystal would be lower after the samples were substituted by  $\text{La}^{3+}$  ions. On the one hand, the lower symmetry of crystal would lead to strong magnetocrystalline anisotropy, inducing the high coercivity [27]. On the other hand, the coercivity depended on the crystallite size. It was known that there was no domain wall in the single-domain grains. The magnetization mechanism was a domain rotation process. The  $H_c$  was in direct proportion to the size of single-domain grains. Therefore,  $H_c$  gradually increased with the size of single-domain grains. However, when the size arrived to the critical diameter of a single domain, the grains became multi-domains, the coercivity started decreasing [28]. One of the main reasons was that the domain-wall displacement was dominant in the magnetization process of the multi-domain grains. Therefore, the coercivity decreased with the growing of multi-domain grains. It can be seen from Fig. 5 that the crystallite size of La-substituted Ni–Zn–Cr ferrites was smaller

**Table 1**  
The values of  $M_s$  and  $H_c$  for samples

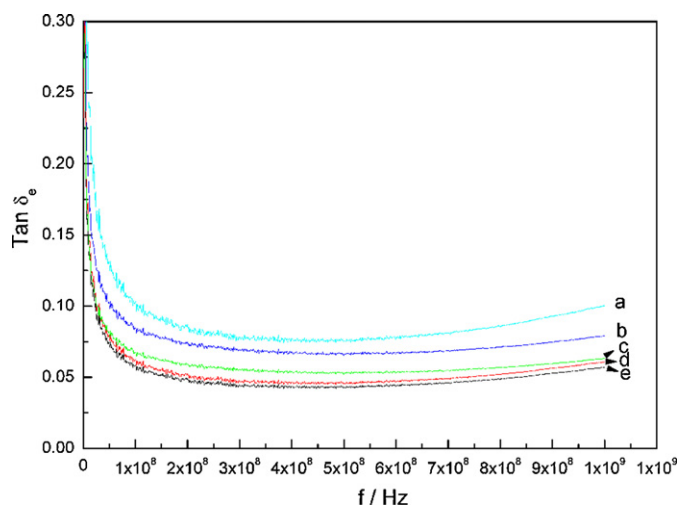
| The composition of samples                                                                | Magnetization ( $M_s$ ) ( $\text{emu g}^{-1}$ ) | Coercivity ( $H_c$ ) (Oe) |
|-------------------------------------------------------------------------------------------|-------------------------------------------------|---------------------------|
| $\text{Ni}_{0.6}\text{Zn}_{0.4}\text{Cr}_{0.5}\text{Fe}_{1.5}\text{O}_4$                  | 45.884                                          | 15.545                    |
| $\text{Ni}_{0.6}\text{Zn}_{0.4}\text{Cr}_{0.5}\text{La}_{0.02}\text{Fe}_{1.48}\text{O}_4$ | 40.925                                          | 18.187                    |
| $\text{Ni}_{0.6}\text{Zn}_{0.4}\text{Cr}_{0.5}\text{La}_{0.04}\text{Fe}_{1.46}\text{O}_4$ | 36.037                                          | 27.637                    |



**Fig. 7.** Frequency dependence of permeability loss ( $\tan \delta_m$ ) for  $\text{Ni}_{0.6}\text{Zn}_{0.4}\text{Cr}_{0.5}\text{La}_x\text{Fe}_{1.5-x}\text{O}_4$  as a function of La content: (a)  $x=0.00$ , (b)  $x=0.02$ , (c)  $x=0.04$ , (d)  $x=0.06$  and (e)  $x=0.08$ .

than the noumenon ferrites, and the crystallite size of as-obtained samples was larger than the critical size of a single domain at higher sintering temperature, so the coercivity increased with the decrease of the crystallite size for the multi-domain grains. Namely, the coercivity increased with the La content. The value of coercivity indicated that the La-substituted Ni–Zn–Cr ferrites were suitable for soft magnet applications.

Figs. 7 and 8 represented the magnetic loss and dielectric loss of the ferrites with different La contents calcined in  $1200^\circ\text{C}$ . It is well known that the spinel ferrites possessing the characteristics of dielectric loss and magnetic loss is one of excellent microwave absorbent. As was seen from the figures, in the range of 1 MHz–1 GHz, the magnetic loss tangent ( $\tan \delta_m$ ) increased first, and then decreased with the increase of La content, while the dielectric loss tangent ( $\tan \delta_e$ ) at first decreased, and then trend towards gentleness. Obviously, the magnetic loss and dielectric loss could be affected by the La content, and the magnetic loss was bigger than dielectric loss. It was known that the dielectric loss for ferrites originates from the electron transferring between  $\text{Fe}^{2+}$  and  $\text{Fe}^{3+}$ , and the magnetic loss comes from the movements and spin



**Fig. 8.** Frequency dependence of permittivity loss ( $\tan \delta_e$ ) for  $\text{Ni}_{0.6}\text{Zn}_{0.4}\text{Cr}_{0.5}\text{La}_x\text{Fe}_{1.5-x}\text{O}_4$  as a function of La content: (a)  $x=0.00$ , (b)  $x=0.02$ , (c)  $x=0.04$ , (d) 0.06 and (e) 0.08.

relaxation of magnetic domains [29]. The colinear ferrimagnetic order transforming into non-colinear arrangement and disruption of ferrimagnetic order occurs because of introducing  $\text{La}^{3+}$  into the octahedral site, leading a decrease in electron transferring between  $\text{Fe}^{2+}$  and  $\text{Fe}^{3+}$  the dielectric loss decreased with the increase of La content. Meanwhile, spin relaxation of magnetic domains increased, the crystallite size of multi-domain grains decreased with the increase of La content, movements of magnetic domains become easier, leading a increase in electromagnetic wave loss. Therefore, the microwave absorption ability could be improved by adjusting the components of the ferrites.

#### 4. Conclusions

In summary, the La-substituted Ni–Zn–Cr ferrites had prepared successfully by the rheological phase reaction. XRD patterns confirmed that the amount of  $\text{Fe}^{3+}$  ions substituted by  $\text{La}^{3+}$  ions had a limit ( $x < 0.06$ ) for  $\text{Ni}_{0.6}\text{Zn}_{0.4}\text{Cr}_{0.5}\text{Fe}_{1.5-x}\text{La}_x\text{O}_4$ . Lattice parameter decreased with the increase of zinc and lanthanum content. Saturation magnetization decreased with the increase of La substitution content, whereas crystallite size and coercivity increased. The value of coercivity indicated that the La-substituted Ni–Zn–Cr ferrites were suitable for use as a soft magnetic material. Moreover, the microwave absorption ability can be improved by adjusting the components of the ferrites.

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