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ABSTRACT

The W-type ferrites doped with Pr³⁺, BaCoNiPr_xFe_{16-x}O₂₇ (x=0–0.20), were prepared by a sol-gel method. The structure and electromagnetic properties of the samples are studied using powder X-ray diffraction, field emission scanning electron microscope, vibrating sample magnetometer and vector network analyzer. All the samples are hexagonal platelet-like W-type barium ferrite. These synthesized samples exhibit paramagnetism and strong magnetism. The saturation magnetization (*M_s*) increases with the increase of Pr³⁺ content. The real part of complex permittivity (ϵ') decreases and the imaginary part (ϵ'') increases with Fe³⁺ replaced by Pr³⁺. The imaginary part of complex permittivity (μ'') increases and the real part (μ') decreases after Pr³⁺ is doped. Furthermore, the doped Pr³⁺ improves the microwave absorptency.

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1. Introduction

Increasing attention has been devoted to hexagonal ferrites, the well-known ferrimagnetic materials, due to the high resistivity and permeability suitable for various applications, such as magnetic recording media, absorbers, microwave and mm-wave devices [1–6]. For the hexagonal structure, there are six possible different types designated M, W, X, Y, Z and U. The W-type hexaferrites, BaMe₂Fe₁₆O₂₇ (where Me is any divalent element), have a crystalline structure built up as a superposition of RSSR*S*S*, where R is a three-oxygen-layer block with composition BaFe₆O₁₁, S (spinel block) is a two-oxygen-layer block with composition Fe₆O₈ and the asterisk means that the corresponding block has been turned 180° around the hexagonal axis [7–9].

The saturation magnetization, coercivity and ferromagnetic resonant frequency can be effectively adjusted by ions substitution of Ba²⁺ or Fe³⁺ [10,11]. Rare-earth elements (RE) have typical relaxation characteristics, which can affect the electromagnetic properties of ferrite magically when chosen as substituted ion [12]. Effects of RE substitution on the structures and electromagnetic properties of the W-type barium ferrites, Ba_{1-x}R_xMe₂Fe₁₆O₂₇ with R=Nd, Er, La and Sm, were investigated [13–16]. Ahmed et al. [17,18] have studied the structure and magnetic properties of W-type barium ferrites Ba_{0.95}R_{0.05}Mg_{0.5}Zn_{0.5}CoFe₁₆O₂₇ where R=Y, Er, Ho, Sm, Nd, Gd and Ce. Extensive studies concerning the ions substitution of the Ba and Me ions in the structure to change the

magnetic properties, microstructures and absorbing performance of W-type hexaferrite have been reported in many literatures [19–23]. However, few studies are available on the rare earth ions substitution of Fe³⁺ in the ferrites, which is the main subject discussed in this work.

The aim of this work is to study the structure, electromagnetic properties and microwave absorption of the hexaferrites, BaCoNiPr_xFe_{16-x}O₂₇ (where x=0, 0.05, 0.10, 0.15 and 0.20), and tries to illustrate the effect of the Pr³⁺ substitution for Fe³⁺ on these properties. The synthesized hexaferrites were characterized by X-ray powder diffraction (XRD), field emission scanning electron microscope (FE-SEM), vibrating sample magnetometer (VSM) and vector network analyzer (VNA).

2. Experimental

Stoichiometric amounts of iron nitrate, cobalt nitrate, nickel nitrate and barium nitrate (praseodymium nitrate was added for preparing BaCoNiPr_xFe_{16-x}O₂₇) were dissolved in citric acid solution and the proper amount of citric acid was added to complex the metal ions completely. The pH value of the sol was adjusted to approximately 7.0 using quadrol. Then, the sol was stirred gently to produce gel. After that operation, the gel was dried immediately in an oven. Finally, the citrate precursor was decomposed at 450 °C for 2 h, and subsequently annealed at 1300 °C for 5 h.

The crystalline phases were characterized by XRD (D/max-IIIC, Japan) at room temperature. The data were recorded using Cu K α radiation at 40.0 kV and 20.0 mA in the region of $2\theta=15\text{--}85^\circ$ with a scanning speed of 4 °/min. The particle morphologies were

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examined by FE-SEM (SUPRA55, Germany) operating at 20.0 kV. The magnetic properties were measured by VSM (Lake-Shore 7307, USA). The frequency dependence of permeability and permittivity was measured by VNA (HP8720ES, USA). The samples were prepared as follows: the particles and the paraffin were mixed together (7:3, mass ratio) and pressed to be cyclic samples with internal diameter 3.04 mm, external diameter 7.00 mm and height 3.00–4.00 mm.

The real part (ϵ') and imaginary part (ϵ'') of complex permittivity, together with the real part (μ') and imaginary part (μ'') of complex permeability were determined in the frequency range of 2–18.0 GHz.

3. Results and discussion

3.1. Microstructure identification

Fig. 1 shows the X-ray diffraction patterns of the investigated samples. Compared with the standard card (PDF#19-0098), all the patterns show that the samples have single phase W-type hexaferrites without notable residuals of the original constituent oxides. Since the XRD patterns of Pr_2O_3 phase cannot be found in Fig. 1, we can draw a conclusion that Pr^{3+} has entered the lattice of hexaferrite successfully.

The lattice parameters of the samples, (a) and (c), are calculated by the computer program (Jade 5), given in Table 1. The results show that the V values slightly decreased as the content of the Pr^{3+} substitution increased. As is known, the ionic radius of Pr^{3+} and Fe^{3+} is 1.086 Å and 0.67 Å, respectively [24]. Therefore, when Fe^{3+} was replaced by Pr^{3+} , the lattice constant should aggrandize as the content of the Pr^{3+} increased. The results, however, were just the opposite. This may account for the change of the lattice distortion when Pr^{3+} was doped.

Fig. 2 exhibits the SEM images of $\text{BaCoNiPr}_x\text{Fe}_{16-x}\text{O}_{27}$ ($x=0-0.20$). As seen in Fig. 2, all the ferrites showed hexagonal

platelet-like shapes. The particle sizes are somewhat reduced with the increase of the Pr^{3+} substitution content. The grain size is about 6–9 μm (mainly about 6–7 μm) of the $\text{BaCoNiFe}_{16}\text{O}_{27}$ sample, while that of the $\text{BaCoNiPr}_{0.2}\text{Fe}_{15.8}\text{O}_{27}$ sample is only about 3–4 μm . It might be attributed to the suppression of Pr^{3+} on the grain growth. The lattice distortion suppressed the abnormality of grains growth available [25]. This result agrees well with the XRD patterns that the V values decrease with the Pr^{3+} substitution content increasing, resulting in a decrease of the unit cell volume (Table 1).

3.2. Magnetic properties

The “S” shaped hysteresis loops of the substituted hexaferrites show the behaviors of the soft magnets, as shown in Fig. 3. The saturation magnetization (M_s) was very high, while both coercive field (H_c) and remanent magnetization (M_r) were comparatively low, meaning that the samples exhibited paramagnetism and strong magnetism. The M_s values are given in Table 2.

Table 2 shows the increase of M_s values in the compositional range $x=0-0.20$. For W-type ferrites, the dominant contributions to this rise may be the three factors, the $\text{Re}^{3+}-\text{Fe}^{3+}$ interactions, the augment of net magnetization and the enhancement of hyperfine fields at 12k and 2d sites. Generally [3], the magnetic behavior of the ferrimagnetic hexaferrite material is largely governed by the distribution of iron ions on the crystallographic lattice sites and the $\text{Fe}^{3+}-\text{Fe}^{3+}$ exchange interactions (the spin coupling of the 3d electrons). The magnetization of the hexaferrite material varies with the factors influencing the strength of these exchange interactions. In the rare-earth substituted ferrite materials, the $\text{Re}^{3+}-\text{Re}^{3+}$ interactions are very weak [26] and the $\text{Re}^{3+}-\text{Fe}^{3+}$ interactions exist via 4f–3d couplings, which can cause changes in magnetic properties.

As we know, the magnetic moment of Pr^{3+} ($3.58\mu_B$) is lower than that of the Fe^{3+} ($5\mu_B$). The substitution of Fe^{3+} in the spin-up states (12k, 6g, $4f_3$ and 2d) appears to cause reduction in magnetization while the substitution in the spin-down states ($4e$, $4f_1$ and $4f_2$) may lead to an increase in the net magnetization [27]. Therefore, if Fe^{3+} in the spin-down states were replaced by Pr^{3+} , the net magnetization in upward spin is expected to increase the total magnetic moment and hence strengthen the magnetization, just as the results show. Besides, the substitution of Pr^{3+} to Fe^{3+} induces a perturbation in both electron-density and symmetry around the 2d lattice site, which could strengthen the exchange interactions and hence strengthen the magnetization [28]. According to the above discussion, through the Pr^{3+} substitution in the spin-down states, the $\text{Fe}^{3+}-\text{O}-\text{Fe}^{3+}$ superexchange interaction was strengthened. Hyperfine fields at 12k and 2d sites were enhanced by the higher net magnetization, which was meaningful to M_s [28]. The increase in the magnetization of the synthesized ferrites validates their possible use in magnetic data storage devices.

3.3. Dielectric and magnetic parameters

Fig. 4a and b shows the frequency dependence of the complex permittivity for different Pr^{3+} doped samples in the frequency range from 2.0 GHz to 18.0 GHz. It indicates that the real part (ϵ') decreases and imaginary part (ϵ'') increases with the Pr^{3+} substitution content increasing. From Fig. 4b, a particular peak appeared at around 8.0 GHz when $x=0.15$. The ϵ' when $x=0.15$ is the smallest while the ϵ'' is the biggest, meaning that the dielectric loss of $\text{BaCoNiPr}_{0.15}\text{Fe}_{15.85}\text{O}_{27}$ is the most excellent.

The reason that brings the difference of the complex permittivity of the ferrites may be the significant contribution of Pr^{3+} . Dielectric properties of polycrystalline ferrite arise mainly due to

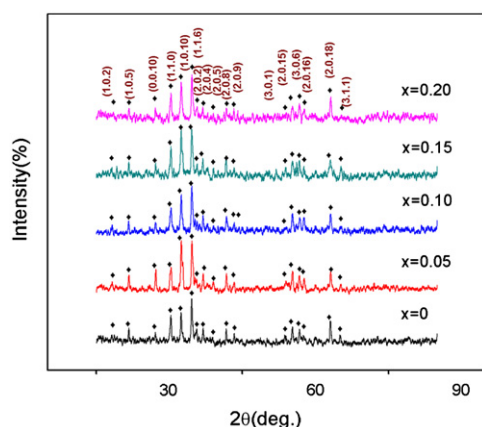


Fig. 1. XRD patterns of $\text{BaCoNiPr}_x\text{Fe}_{16-x}\text{O}_{27}$ ($x=0-0.20$).

Table 1

Lattice parameters of hexaferrites $\text{BaCoNiPr}_x\text{Fe}_{16-x}\text{O}_{27}$ ($x=0-0.15$).

Sample	Chemical formula	Calculated cell parameters		Unit cell volume $V=a^2c$ (Å ³)
		a (Å)	c (Å)	
$x=0$	$\text{BaCoNiFe}_{16}\text{O}_{27}$	5.89092	32.87373	987.98
$x=0.05$	$\text{BaCoNiPr}_{0.05}\text{Fe}_{15.95}\text{O}_{27}$	5.89156	32.81473	986.42
$x=0.10$	$\text{BaCoNiPr}_{0.10}\text{Fe}_{15.90}\text{O}_{27}$	5.88838	32.80256	984.99
$x=0.15$	$\text{BaCoNiPr}_{0.15}\text{Fe}_{15.85}\text{O}_{27}$	5.89058	32.74277	983.93
$x=0.20$	$\text{BaCoNiPr}_{0.20}\text{Fe}_{15.80}\text{O}_{27}$	5.88443	32.8070	983.80

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