



Effect of Ti–Zn substitution on structural, magnetic and microwave absorption characteristics of strontium hexaferrite



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ABSTRACT

SrFe_{12-x}Ti_{x/2}Zn_{x/2}O₁₉ ($x = 0-2.5$) powders were synthesized by use of chlorides through co-precipitation method. The obtained powders were then milled by high energy ball mill to crush hard agglomerates and achieve nanoparticles. In order to evaluate microwave absorption versus frequency, composites including ferrite, as a filler, and matrix of polyvinylchloride (PVC) with weight ratio of 70% ferrite were prepared. X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), field emission scanning electron microscopy (FE-SEM), vibrating sample magnetometer (VSM) and vector network analyzer (VNA) were employed to study the structural, magnetic and microwave absorption properties of samples. The XRD patterns showed that the hexagonal single phase were obtained only if $x \leq 2$. FE-SEM micrographs indicated that the particle sizes were almost less than 40 nm. With increasing amount of dopant from $x = 0$ to $x = 2.5$, the saturation magnetization and coercivity decreased from 56 emu/g and 1350 Oe to 16 emu/g and 88 Oe, respectively. The maximum loss of electromagnetic waves was -36.13 dB obtained by ferrite with maximum amount of substitution. The obtained results indicated that the proposed composites can be introduced as electromagnetic wave absorption materials.

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1. Introduction

Quite a few electronic systems, such as the mobile phones, the wireless local area network (LAN) systems, and the bluetooth technologies, operate in the microwave frequency range [1–4]. Notwithstanding all the merits, widespread applications of electromagnetic devices precipitate the electromagnetic pollution crisis [4,5]. So as to address the problem posed by electromagnetic interference (EMI), thinner electromagnetic wave absorbers which enjoy wide absorbing bandwidth and have an ability of absorbing unwanted electromagnetic signals can be a good solution [4]. In this regard, Dielectric materials and magnetic materials can be considered as the best options [6]. Ferrites—as a member of magnetic materials—are much better electromagnetic interference suppressors in comparison to dielectric counterparts [2]. Large electrical resistivity, low density, low cost, high stability, and high microwave magnetic loss are some of the outstanding features that make hexaferrites applicable to microwave band [5–8]. Strontium hexaferrite possesses high magnetocrystalline anisotropy, leading it to have high resonance frequency. In order to reduce the resonance frequency of pure hexaferrites, substitution of cation which has lower magnetic moment than ferric ions

for ferric is a well-known fact. In this regard, the influence of several cation substitutions on microwave absorption characteristics of strontium hexaferrite was investigated in recent years [6–13]; however, the Ti–Zn doped strontium ferrite microwave properties have been rarely reported. In the present attempt, Ti–Zn substituted strontium hexaferrites were first prepared by co-precipitation technique and nano sized powders of them were later achieved after the ball milling process. The effects of Ti–Zn substitution on structural, magnetic and microwave absorption properties were surveyed. The findings suggested that the doped strontium hexaferrites were suitable candidates for microwave absorbers in K_u band.

2. Material and methods

FeCl₃·6H₂O (Merck, >98%), SrCl₂·6H₂O (Merck, >99%), TiCl₄ (Merck, >99%), ZnCl₂·4H₂O (Merck, >99%) and NaOH (Merck, >99%) were used as the starting materials to synthesize M-type hexaferrite powders with the compositions of SrFe_{12-x}Ti_{x/2}Zn_{x/2}O₁₉ (where x varies from 0 to 2.5 in steps of 0.5) by means of co-precipitation technique. Stoichiometric amount of the salts were dissolved in distilled water to achieve aqueous solution; subsequently, NaOH (1 M) was gradually added while stirring so as to form the precipitate until the solution pH reached a value of 12. The aqueous suspensions were further stirred for another 1 h to homogenize the system. Having been filtered, the ferrite precursors were washed several times with distilled water, dried at 100 °C and calcined at 900 °C for 1 h. A high energy ball mill machine (SPEX Certprep-M8000) equipped with hardened steel balls was used in order to reduce the calcined powders particle size. The

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stearic acid, as a process control agent, was added in milling procedure. The ball to powder weight ratio of 10:1, shaking frequency of 50 Hz, and milling time of 1.5 h were applied during the process.

Phase analysis of synthetic powders was investigated using X-ray powder diffractometer with Cu K α radiation in the 2θ range from 20° to 70° . The FTIR spectra for the all samples were achieved by Nicolet 100IR spectrometer over the wave number range $400\text{--}4000\text{ cm}^{-1}$. The particle size and morphology of powders was studied by JSM-7000F JEOL field-emission scanning electron microscopy (FE-SEM). Vibrating sample magnetometer (VSM) (TM-XYZTB-SIH) was employed to characterize the magnetic properties with maximum applied magnetic fields of 9000 Oe. Reflection loss variation measurements in the frequency range of 12–18 GHz were performed through HP8722ET vector network analyzer. In order to measure the reflection loss, homogenous mixture of ferrite powders and polyvinylchloride (PVC) with the weight ratio of 70:30 were plasticized, and fired at 220°C and 5 MPa pressure to form cylindrical composites with the constant thickness of 1.8 and diameter of 40 mm.

3. Results and discussion

XRD patterns of the $\text{SrFe}_{12-x}\text{Ti}_x/2\text{Zn}_x/2\text{O}_{19}$ powders with different Ti–Zn substitution levels are shown in Fig. 1. It shows that when substitution amount, x , is equal or less than 2, the powders solely contain desirable M-type hexagonal strontium ferrite phase. On the contrary, when x reaches 2.5, $\alpha\text{-Fe}_2\text{O}_3$ peaks are also detectable. The values of the lattice parameters (a and c) were calculated for all the samples using Eq. (1), and their variations as a function of dopant content are depicted in Fig. 2.

$$\frac{1}{d^2} = \frac{4}{3} \left(\frac{h^2 + hk + k^2}{a^2} \right) + \frac{l^2}{c^2} \quad (1)$$

The ionic radius of the Fe^{3+} , Ti^{4+} and Zn^{2+} are 0.64, 0.60 and 0.74 Å, respectively. The observed variations can be ascribed to the difference in the ionic radius of the metal ions as well as the amount of substituted Ti–Zn ions in the structure. Furthermore, Fang et al. [14] reported that the lattice parameters might be affected by the change of exchange energy after ions substitution. When x reaches 2.5, on account of $\alpha\text{-Fe}_2\text{O}_3$ presence, the number of cationic and anionic vacancies increases; as a result, both a and c lattice parameters decrease.

Fig. 3 shows the FTIR spectra of strontium hexaferrite with various quantities of the dopant. In the case of all the samples, there are two absorption bands in the area between 400 cm^{-1} and 800 cm^{-1} , which are attributed to Fe–O stretching vibration band in octahedral and tetrahedral clusters. The tetrahedral and octahedral bands occur in the range of $500\text{--}700\text{ cm}^{-1}$ and $400\text{--}500\text{ cm}^{-1}$, respectively [15]. The difference in $\text{Fe}^{3+}\text{--O}^{2-}$ distances for octahedral and tetrahedral sites results in the band positions variation

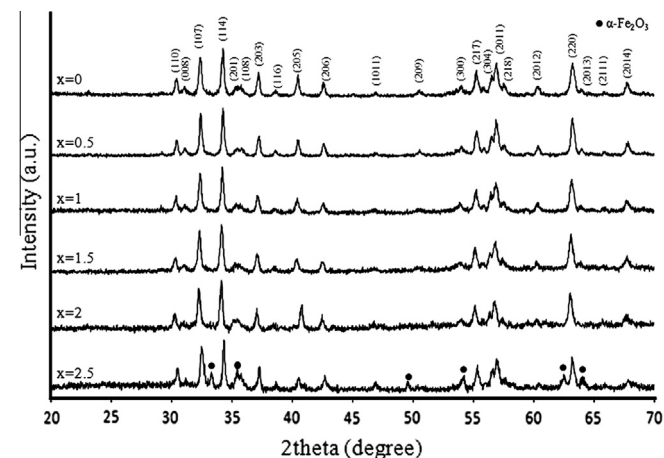


Fig. 1. XRD patterns of as calcined $\text{SrFe}_{12-x}\text{Ti}_x/2\text{Zn}_x/2\text{O}_{19}$ at various amounts of substitution.

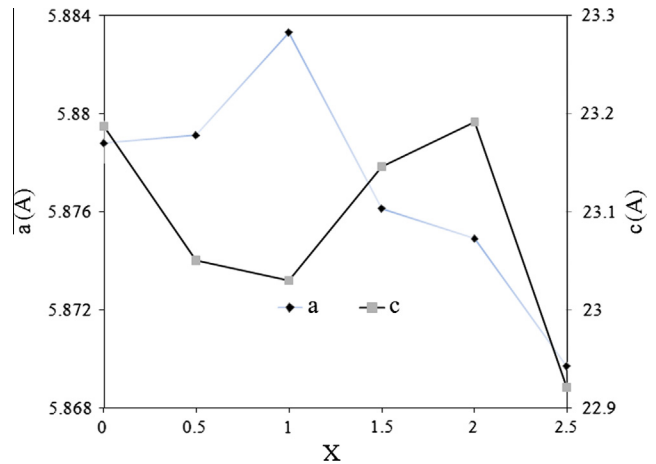


Fig. 2. Variation of lattice parameters (a and c) as a function of composition (x).

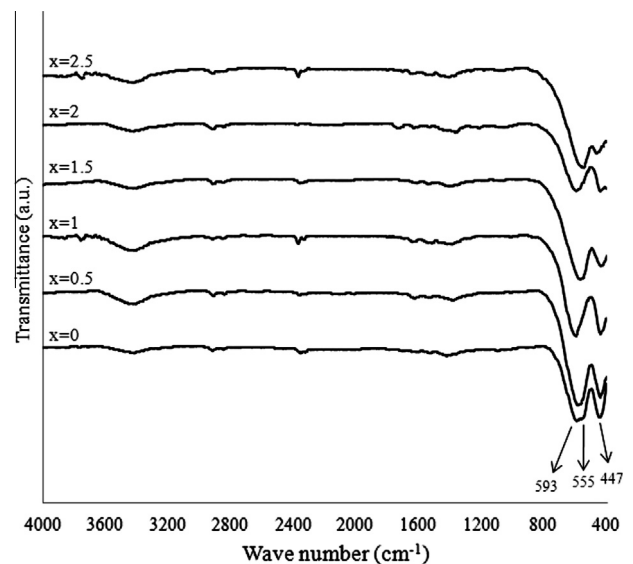


Fig. 3. FTIR spectra of milled $\text{SrFe}_{12-x}\text{Ti}_x/2\text{Zn}_x/2\text{O}_{19}$ at various amounts of substitution.

[16]. Moreover, as it can be identified in Fig. 3, the positions of absorption peaks were shifted as a consequence of Ti and Zn replacements due to the variation in $\text{Fe}^{3+}\text{--O}^{2-}$ distances caused by substitution. As for undoped ferrite, the absorption peak at about 555 cm^{-1} is distinguishable associated with Sr–O stretching vibration band. The above-mentioned peak cannot be observed in the doped samples on the grounds that it overlapped with shifted characteristic peaks of hexaferrites.

Fig. 4 displays the FE-SEM micrographs of ball milled $\text{SrFe}_{12-x}\text{Ti}_x/2\text{Zn}_x/2\text{O}_{19}$ nanoparticles. The powders have irregular non spherical morphologies. It stands to reason that the final morphologies strongly is affected by high energy ball mill processing, and the addition of Ti and Zn has not a major effect on the final microstructures. The particle size of all the samples was estimated to be 15–40 nm.

The impact of Ti–Zn substitutions on coercivity and saturation magnetization is indicated in Fig. 5. The outcomes represents that the coercivity is decreased from 1350 to 88 Oe with the increasing of x value from 0 to 2.5. As reported, coercivity is contingent upon magnetocrystalline anisotropy and particle size; furthermore, the particle size of the powders is approximately in the same range,

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