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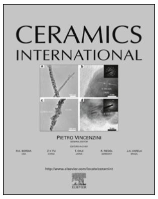

Structural, electrical, and magnetic properties of Zn substituted magnesium ferrite

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ABSTRACT

Zinc substituted magnesium (Mg–Zn) ferrites with the general formula $Mg_{1-x}Zn_xFe_2O_4$ ($x=0.00$, 0.25, 0.50, 0.75, and 1.00) were prepared using the solution combustion route. The dried powder after calcination (700 °C for 2 h) was compacted and sintered at 1050 °C for 3 h. The structural, morphological, dielectric and magnetic properties of the sintered ferrites were studied using X-ray diffraction (XRD), scanning electron microscopy (SEM), impedance spectroscopy, and vibration sample magnetometry (VSM). The XRD analysis of sintered samples confirmed that the expected spinel cubic phase was formed for all samples. The crystallite sizes evaluated using Scherre's formula were found to be in the range of 47–80 nm. SEM analysis showed homogeneous grains with a polyhedral structure. The electrical conductivity increased with increasing frequency, which is normal dielectric behavior for such materials. The dielectric constant, dielectric loss tangent, and AC conductivity were found to be lowest for $x=0.50$. The VSM results showed that the zinc concentration had a significant influence on the saturation magnetization and coercivity.

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1. Introduction

In recent years, there has been growing interest from the magnetic, electronic, and microwave fields for developing spinel ferrite materials with improved electromagnetic properties at higher frequencies. Ferrites are considered superior to other magnetic materials for use at higher frequencies, because of their low dielectric losses and high electrical resistivity [1]. Spinel ferrites are technologically important materials and have potential applications, including high-density magnetic storage, electronic and microwave devices, gas sensors, and magnetically guided drug delivery [2].

Mixed zinc ferrites are technologically interesting materials with applications ranging from magnetic storage devices to catalytic water splitting [3]. Mg–Zn ferrites are used in power transformers, microwave devices, and telecommunications due to their high permeability and electrical resistivity [4]. The structural, magnetic, electrical, and dielectric properties of ferrites depend on the method of preparation, chemical composition, sintering time, sintering temperature, and level of doping [5]. Ferrite nanoparticles are generally prepared by physical and chemical methods, such as ceramic processing, co-precipitation, hydrothermal methods, and via the fabrication of reverse, or, normal micelles [4,6,7]. For the past two decades, the solution combustion method has attracted a great deal of attention for synthesizing oxides with well controlled compositions. The solution combustion method is a popular method because it provides a fast, self-sustaining, and exothermic chemical reaction between metal salts and a suitable organic fuel. In this technique, most of the heat required for synthesis is supplied by the reaction itself [8–10]. This study focuses on the synthesis and electromagnetic properties of Zn^{2+} substituted magnesium ferrites prepared by the solution combustion method using urea and sugar as the fuel.

2. Material and methods

Various Mg–Zn ferrites were prepared with different levels of Zn substitution using the solution combustion method. A stoichiometric mixture of AR grade ferric nitrate ($Fe(NO_3)_3 \cdot 9H_2O$), magnesium nitrate ($Mg(NO_3)_2 \cdot 6H_2O$), zinc nitrate ($Zn(NO_3)_2 \cdot 6H_2O$) 99.9% pure MERK and the sugar and urea fuel mixture was used: the detailed synthesis procedure is reported elsewhere [11]. A dark brown powder was obtained and then calcined at 700 °C for 2 h at a heating rate of 10 °C/min. This calcination step is necessary to improve the crystallization and homogeneity of the cation distribution in the spinel. A fixed amount of calcined powder was mixed with a few drops of 3 wt% aqueous polyvinyl alcohol (PVA) as a binder and compacted at a pressure of 75 kg/cm^2 for 5 min to form pellets with a diameter of 13 mm and a thickness of 2 mm. These pellets were sintered at

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1050 °C in air for 3 h and then the furnace was allowed to cool to room temperature. Silver paste was painted on both sides of the pellets to prepare parallel plate capacitor geometry with the ferrite material. The pellets were dried in a hot air oven (50 °C) before the characterization measurements to avoid the effect of humidity.

The phase purity of the sintered ferrite pellets was studied using a PANalytical X'pertPRO X-ray diffractometer with Cu Kα radiation (λ=1.5406 Å). Scanning was performed from 20° to 70° with a step size of 2 °/min. The lattice constant, crystallite size and X-ray density were determined from the XRD data. The morphological characteristics of fractured surfaces of the samples were studied using scanning electron microscopy (SEM, JEOL-JSM Japan). The elemental composition of the samples was calculated using energy dispersive X-ray spectroscopy (EDS). The number of moles of each element present in the composition $Mg_{1-x}Zn_xFe_2O_4$ was calculated using the observed wt% of each element from EDS data as follows:

$$\text{No. of moles of Me} = \frac{\text{Observed wt\% of Me from EDS Molecular wt of sample}}{\text{Atomic wt of Me} \times 100} \tag{1}$$

An Alpha-A high performance frequency analyzer (NOVACONTROL GmbH) was used to carry out the dielectric measurements at room temperature in the frequency range of 10^2 Hz to 10^7 Hz. The dielectric constant (ε') was calculated using the following relationship:

$$\varepsilon' = C_p t / \varepsilon_0 A \tag{2}$$

where the permittivity of free space $\varepsilon_o = 8.854 \times 10^{-12}$ F/m, t is thickness of the pellet, A is the cross sectional area of the pellet and C_p is the capacitance of the pellet.

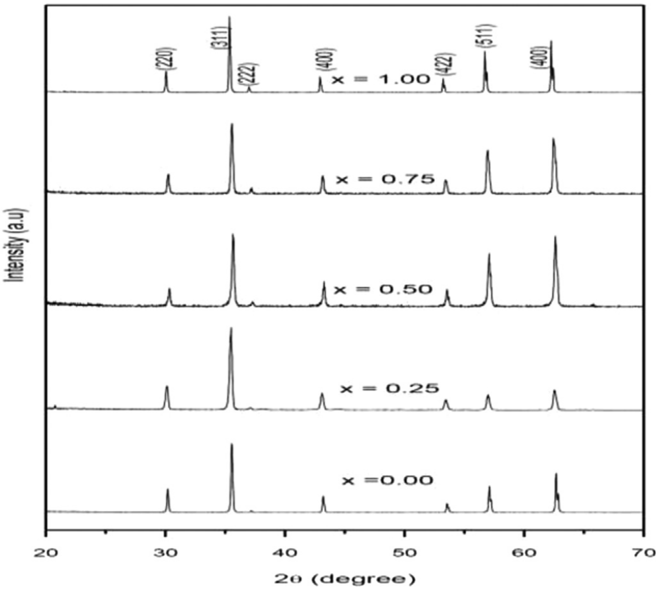

Fig. 1. Powder XRD patterns of $Mg_{1-x}Zn_xFe_2O_4$ ferrites sintered at 1050 °C.

The loss tangent (tan δ) was obtained using the following relationship:

$$\tan\delta = 1/2\pi f R_p C_p \tag{3}$$

where R_p is the resistance of the pellet. The AC conductivity was calculated using the following formula:

$$\sigma_{ac} = 2\pi f \varepsilon' \varepsilon_o \tan\delta \tag{4}$$

where f is frequency.

The magnetic properties of the samples were measured using a vibrating sample magnetometer (VSM, Model Lakeshore 7410) using applied magnetic fields of ± 15 kOe at room temperature. The saturation magnetization (M_s) and coercivity (H_c) were determined from the obtained hysteresis loops.

3. Results and discussion

3.1. XRD analysis

Fig. 1 shows the X-ray diffraction patterns of $Mg_{1-x}Zn_xFe_2O_4$ (x=0.00, 0.25, 0.50, 0.75 and 1.00) ferrites sintered at 1050 °C. The XRD patterns of the sintered samples characteristic were of the cubic spinel structure and no impurity peaks were observed. The lattice parameters were determined by indexing the XRD patterns. The lattice parameter (a) of the sintered samples was found to increase with increasing Zn content obeying Vegard's law [12]. This is attributed to the larger ionic radius of Zn^{2+} (0.82 Å) compared with Mg^{2+} (0.66 Å) [6]. The lattice parameters for the samples with different Zn^{2+}contents are shown in Table 1. A similar trend was reported previously for Mg–Zn ferrites [13–15]. The crystallite size (D) was calculated from the (311) peak using Scherrer's formula [16]. This peak narrowed with increasing doping, indicating a decrease in the d-spacing (increase in the lattice parameter). The crystallite sizes of the sintered ferrites were in the range of 47–80 nm. The X-ray density d_x was determined using the relationship $d_x = ZM/Na^3$ [17], where M is the molecular weight of the sample, N is Avogadro's number, and Z is the number of chemical units per unit cell, (8 for cubic spinel). The bulk density d_b of the samples was calculated using the formula $d_b = m/\pi r^2 h$ where, m is the mass, r is the radius and h is the height of the pellet. It can be seen from the data shown in Table 1 that the bulk density increased with increasing Zn^{2+} content. The increase in the density is associated with the increased atomic weight resulting from the substitution of Mg^{2+} ions with Zn^{2+} ions. The atomic weights of Zn^{2+} and Mg^{2+} are 65.38 and 24.31 amu, respectively. The apparent porosity (P) was calculated from the difference between the bulk and X-ray densities, i.e., $P = (1 - d_b/d_x)$. From Table 1 it is evident that the calculated porosity increased with Zn^{2+} concentration. The larger atomic weight of zinc increases the bulk density, which may be responsible for the increase in porosity.

Table 1
Composition dependence of lattice parameter (a), crystallite size (D), grain size, X-ray density (d_x), bulk density (d_b), apparent porosity, magnetization (M_s) and coercivity (H_c) of $Mg_{1-x}Zn_xFe_2O_4$ at room temperature.

Composition (x)	a (Å)	D (nm)	Grain size from SEM (nm)	d_x (gm/cc)	d_b (gm/cc)	% Theoretical density	Apparent porosity ($1-d_b/d_x$) (%)	M_s (emu/g)	H_c (Oe)
0.00	8.301	53	179	4.64	3.944	85	15	22.44	46.91
0.25	8.332	64	220	4.829	4.076	84.4	15.6	42.33	9.60
0.50	8.347	69	237	5.037	4.298	85.33	15.67	46.97	2.78
0.75	8.379	80	317	5.166	4.335	83.91	16.09	9.90	160
1.00	8.414	47	154	5.379	4.491	83.49	16.51	2.48	176

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