Research Article

Characterization of $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ and Permittivity of Solid Material of NiO, ZnO, Fe_2O_3 , and $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ at Microwave Frequency Using Open Ended Coaxial Probe

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This paper describes a detailed study on the application of an open ended coaxial probe technique to determine the permittivity of $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ in the frequency range between 1 GHz and 10 GHz. The x compositions of the spinel ferrite were 0.1, 0.3, 0.5, 0.7, and 0.9. The $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ samples were prepared by 10-hour sintering at 900°C with 4°C/min increment from room temperature. Particles showed phase purity and crystallinity in powder X-ray diffraction (XRD) analysis. Surface morphology measurement of scanning electron microscopy (SEM) was conducted on the plane surfaces of the molded samples which gave information about grain morphology, boundaries, and porosity. The tabulated grain size for all samples was in the range of 62 nm–175 nm. The complex permittivity of Ni-Zn ferrite samples was determined using the Agilent Dielectric Probe Kit 85070B. The probe assumed the samples were nonmagnetic homogeneous materials. The permittivity values also provide insights into the effect of the fractional composition of x on the bulk permittivity values $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$. Vector Network Analyzer 8720B (VNA) was connected via coaxial cable to the Agilent Dielectric Probe Kit 85070B.

1. Introduction

Electromagnetic (EM) waves at microwave frequencies have many applications in various fields such as wireless telecommunication system, radar, local area network, electronic devices, mobile phones, laptops, and medical equipment [1, 2]. The effect of growth in various applications has led to electromagnetic interference (EMI) problems that have to be suppressed to acceptable limits. EMI reducing materials (absorbers) may be dielectric or magnetic [3] and the design depends on the frequency range, the desired quantity of shielding, and the physical characteristics of the devices being shielded. Thus it is important to determine their high frequency characteristics for the applications of EM in the high GHz ranges [4, 5]. Ni-Zn ferrite ceramics are the preferred ceramic material for high frequency applications in

order to suppress generation of Eddy current [6]. Although Ni-Zn ferrite ceramics have high electrical resistivity to prevent Eddy current generation, they have moderate magnetic permeability compared to Mn-Zn ferrites. However, the electrical and magnetic properties of these ferrite ceramics are heavily influenced by its microstructural features such as grain size, nature of grain boundaries, nature of porosity, and crystalline structure. The microstructural features of interest could be attained via chemical composition and high temperature processing [7]. However, the detailed electrical properties of Ni-Zn ferrite at different Ni-Zn ratio in a wideband frequency using open ended coaxial probe have not been studied yet. Thus, the aim of this work is to determine electrical properties of Ni-Zn ferrites prepared at different chemical composition based on chemical formula $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ with $0.1 \leq x \leq 0.9$ that sintered at constant

temperature. The variations in the microstructures, surface morphology, and alterations in reflection coefficient as well as their electrical properties of the Ni-Zn ferrites are the concern of this study.

2. Basic Principle

2.1. Loss Mechanism by Oscillating Electric Field. Materials can be categorized into two types which are the nonmagnetic materials and the magnetic materials. The core loss mechanisms for nonmagnetic materials are dielectric (dipolar) loss and conduction loss. The conduction and dipolar losses usually occur in metallic, high conductivity materials and dielectric insulators, respectively. The loss mechanisms for magnetic materials are also the conductive loss with addition magnetic loss such as hysteresis, eddy current, and the resonance losses (domain wall and electron spin). Loss condition of the materials is greatly influenced by microwave absorption.

The microwave absorption is caused by external electrical field and related to the material's complex permittivity ϵ :

$$\epsilon^* = \epsilon_o (\epsilon' - j\epsilon''), \quad (1)$$

where ϵ_o is the permittivity of free space ($\epsilon_o = 8.86 \times 10^{-12}$ F/m) and the real part ϵ' and the imaginary part ϵ'' are the relative dielectric constant and the effective relative dielectric loss factor, respectively. The real part of permittivity controls the amount of electrostatic energy stored per unit volume for a given applied field in a material. The imaginary part defines the energy loss caused by the lag in the polarization upon wave propagation when it passes through a material.

The translational motions of free or bound charges and rotating charge complexes are induced by the internal field generated when the microwaves penetrate and propagate through a material. These induced motions are resisted by inertial, elastic, and frictional forces, thus causing energy losses.

2.2. Open Ended Coaxial Probe. For the open ended coaxial probe measurement technique the complex relative permittivity is determined by inverting the expression of $Y(\epsilon^*)$ where Y is the aperture admittance of the probe [8]:

$$Y = Y_o \frac{1 - \Gamma}{1 + \Gamma}, \quad (2)$$

where Y_o is the characteristic admittance of the coaxial line and Γ is the reflection coefficient at the aperture. The aperture admittance of open ended coaxial probes has several analytical expressions which contains the complex permittivity and can be compared to the measured admittance [9–12]. Some are from the computational points which may contribute to convergence problems because of the presence of multiple integrals, Bessel functions, and sine integrals when numerically solved. The expression for the aperture admittance is

given by [13], found by matching the electromagnetic field around the probe aperture, and can be adopted:

$$Y = \frac{\sqrt{\epsilon_r^*} \gamma_o}{\sqrt{\epsilon_{cl}^*} \ln(b/a)} \left\{ \int_0^{\pi/2} \left[J_o \left(\gamma_o a \sqrt{\epsilon_r^*} \sin \theta \right) - J_o \left(\gamma_o b \sqrt{\epsilon_r^*} \sin \theta \right) \right]^2 \frac{d\theta}{\sin \theta} + \frac{j}{\pi} \cdot \int_0^{\pi} \left[2\text{Si} \left(\gamma_o a \sqrt{\epsilon_r^*} \sqrt{1 + \frac{b^2}{a^2} - 2\frac{b}{a} \cos \theta} \right) - \text{Si} \left(2\gamma_o a \sqrt{\epsilon_r^*} \sin \frac{\theta}{2} \right) - \text{Si} \left(2\gamma_o b \sqrt{\epsilon_r^*} \sin \frac{\theta}{2} \right) \right] d\theta \right\}, \quad (3)$$

where ϵ_r^* is the complex relative permittivity of the material under test, ϵ_{cl}^* is the relative permittivity of the coaxial line, a and b are the inner and outer radii of the coaxial line, respectively, γ_o is the absolute value of the propagation constant in free space, and Si and J_o are the sine integral and the Bessel function of zero order, respectively. This integral expression can be evaluated numerically by series expansion as in [10, 11] or numerical integration.

A different procedure for the extraction of material parameters involves minimizing the distance between the calculated aperture admittance (3) and the corresponding measured quantities through fitting algorithms, which may be based on either deterministic or stochastic optimization procedures. The minimization can be performed over the whole frequency range or on a point-by-point basis (i.e., at individual frequency points). Optimization procedure is needed to determine parameters for the point-by-point basis since it consists of modelling the complex relative permittivity and magnetic permeability with a prespecified functional form. Laurent series can be used for complex relative permittivity and magnetic permeability models [14], as well as dispersive laws, such as Havriliak-Negami and its special cases Cole-Cole and Debye to model dielectric relaxation [15], or the Lorentz model for both dielectric and magnetic dispersion [16]. The Havriliak-Negami model is an empirical modification of the single-pole Debye relaxation model:

$$\epsilon_r^* = \epsilon_{\infty} + \frac{\epsilon_s - \epsilon_{\infty}}{[1 + (j\omega\tau)^{1-\alpha}]^{\beta}}, \quad (4)$$

where ϵ_s and ϵ_{∞} are the values of the real part of the complex relative permittivity at low and high frequency, respectively, τ is the relaxation time, and α and β are positive real constants ($0 \leq \alpha, \beta \leq 1$). From this model, the Cole-Cole equation can be derived setting $\beta = 1$; the Debye equation is obtained with $\alpha = 1$ and $\beta = 1$. This empirical model has the ability to give a better fit to the behaviour of dispersive materials over a wide frequency range.

FIGURE 1: Flowchart of sample preparation to characterization.

3. Method

3.1. Sample Preparation and Structural and Morphological Characterization. The materials required for preparing $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ samples were obtained from Alfa Aesar: Iron(III) Oxide (99.500%), Nickel(II) Oxide (99.000%), and Zinc Oxide (99.900%). The sample preparation procedures are roughly illustrated in the flowchart as in Figure 1.

3.2. Complex Permittivity Measurement. In this work, the permittivities of the samples were measured using Agilent 85070B Dielectric Probe Kit. Air and short and distilled water were used as standard materials for calibration as recommended by the manufacturer. The Dielectric Probe Kit automatically determined the complex permittivity of the materials under test by measuring both the magnitude and phase of the reflection coefficients. The measurement was started with the standard test materials which consist of air, Teflon, RT-duroid 5880, and Perspex. Then, the measurement continued with the Iron(III) Oxide (Fe_2O_3), Nickel(II) Oxide (NiO), and Zinc Oxide (ZnO) materials. The materials already prepared by the manufacturer (Alfa Aesar) in powder form were pressed into cylindrical mold at 4 tons

FIGURE 2: XRD profile of $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ samples sintered at 900°C.TABLE 1: d -spacing for $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ samples at the main (3 1 1) hkl plane.

x	2θ (°)	d -spacing (Å)
0.1	35.317	2.543
0.3	35.319	2.536
0.5	35.412	2.526
0.7	35.426	2.518
0.9	35.778	2.511

using mechanical pressing machine. The measurement was continued with $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ samples that have different fractional compositions of x . The sintered mixture powder of $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ samples was pressed into cylindrical mold at 4 tons using mechanical pressing machine as well.

4. Results and Discussion

4.1. Structure Characterization and Morphology of $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$

4.1.1. XRD Profiles. Figure 2 presents the XRD patterns of $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ samples after sintering at 900°C for 10 hours with heating rate of 4°C/min. The patterns showed distinct diffraction lines with the highest peaks at 35.317, 35.319, 35.412, 35.426, and 35.778 of the 2θ (°) for all samples with an increment of x which in turn decrease the d -spacing accordingly (Table 1). The d -spacing was linearly decreased as the fractional composition of x increased as shown in Figure 3. The distinct diffraction lines could be observed for the powders sintered at 900°C meaning that the intensity of XRD peaks increased as the amorphous phase transformed into the crystalline phase for $\text{Ni}_{0.1}\text{Zn}_{0.9}\text{Fe}_2\text{O}_4$ sample. This could be related to the development of crystal growth of the entire particles. The peaks for (2 2 0), (3 1 1), (2 2 2), (4 0 0), (4 2 2), (5 1 1), and (4 4 0) occurred at the reflections planes originated at the 2θ (°) values 30.003, 35.317,

FIGURE 3: d -spacing against fractional composition of x for $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$.

36.931, 42.905, 53.172, 56.680, and 62.228, yielding to the d -spacing [Å] values of 2.978, 2.543, 2.434, 2.108, 1.723, 1.624, and 1.492 consecutively thus indicating that a pure cubic ferrite phase formed according to the reference spectrum of Ni-Zn ferrite (Joint Committee of Powder Diffraction Standards). The XRD profiles of different x are also presented in Figure 2 that showed the same behaviors as described above for $\text{Ni}_{0.1}\text{Zn}_{0.9}\text{Fe}_2\text{O}_4$ sample with slight difference in the intensity of 2θ (°) and decreased pattern for d -spacing [Å] as x increased in the fractional composition.

4.1.2. Lattice Constant. The lattice constant a was obtained as a function of fractional composition of x substitution in $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ calculated from the combination of Bragg's equation and d -spacing expression:

$$\frac{1}{d_{hkl}^2} = \frac{h^2}{a^2} + \frac{k^2}{a^2} + \frac{l^2}{a^2} \quad (5)$$

for cubic system equation. The calculation of lattice constant for all samples was considered at the single phase crystallite (3 1 1) hkl planes and thus the value of lattice constant was established. A linear relationship with negative sensitivity could be obtained between lattice constant and x for $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ sample as shown in Figure 4. Other studies also found that the lattice constant a decreased with the increasing of x concentration [17, 18].

4.1.3. Density. The X-ray densities (D_x) of the $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ samples were calculated using

$$D_x = \frac{8 \times M}{N \times a^3} \text{ g} \cdot \text{cm}^{-3}. \quad (6)$$

It was found that the density of the $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ samples increased linearly with increasing of the substituted amount of x inside the Ni-Zn ferrite sample (Figure 5). Every reduction in number of molecular masses for all compositions gave a higher density value (Table 2).

TABLE 2: Calculated true X-ray density of $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ samples.

Composition (x)	Molecular mass (g/mole)	Lattice constant a (Å)	X-ray density (g/cm^3)
0.1	240.4234	8.4334	5.3250
0.3	239.0802	8.4105	5.3386
0.5	237.7370	8.3789	5.3689
0.7	236.3938	8.3500	5.3942
0.9	235.0506	8.3280	5.4062

FIGURE 4: Lattice constant against fractional composition of x for $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$.

FIGURE 5: X-ray densities against fractional composition of x for $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$.

4.1.4. SEM Morphologies. The microstructural properties of the molded $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ samples were obtained by scanning electron microscope as in Figure 6. The raw mixture in the form of powder was first sintered at 900°C for 10 hours before being poured into mold and compacted using mechanical pressing machine. The measurement was conducted on the plane surfaces of the molded samples which gave information in terms of grain morphology, grain boundaries, and porosity. The grain size of each sample was randomly selected through 60000 magnifications from the morphology picture so that the grain size could be seen clearly. The tabulated grain size for all samples was in the range of 62 nm–175 nm. Lots of pores could be seen from the morphology

FIGURE 6: SEM micrograph of (a) $\text{Ni}_{0.1}\text{Zn}_{0.9}\text{Fe}_2\text{O}_4$, (b) $\text{Ni}_{0.3}\text{Zn}_{0.7}\text{Fe}_2\text{O}_4$, (c) $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$, (d) $\text{Ni}_{0.7}\text{Zn}_{0.3}\text{Fe}_2\text{O}_4$, and (e) $\text{Ni}_{0.9}\text{Zn}_{0.1}\text{Fe}_2\text{O}_4$ sample at 60000 magnifications.

and that was probably due to inhomogeneous size of particle; thus there would be air gaps between the particles. If the sintering time is increased, the pores will reduce because of the formation of strong bonds between the adjacent particles [19].

4.2. Permittivity Results

4.2.1. Standard Material. The measurement procedure to determine complex permittivity using the Agilent Dielectric Probe Kit 85070B was described above. The permittivity values also provide insights into the effect of the fractional composition of x on the bulk permittivity values $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$. Vector Network Analyzer 8720B (VNA) was connected via coaxial cable to the Agilent Dielectric Probe Kit 85070B.

The technique was done by pressing the dielectric probe against the sample material. The microwave signal launched by the VNA was reflected by the sample. The reflected wave was received by the VNA which then used the wave to calculate the dielectric constant and loss factor.

The dielectric constant and loss factor of air and several standard materials including Teflon, RT-duroid 5880, and Perspex with thickness of 20 mm, 19.05 mm, and 20 mm were measured in the frequency range between 1 GHz and 10 GHz as shown in Figure 7. The dielectric constant values for all the samples were almost constant for the whole frequency range with slight dispersion toward the higher end of the frequency range except for air which was lossless. The slight dispersion for all the samples at the higher frequency end was due to the increase of the loss factor because of higher

FIGURE 7: Complex permittivity of standard samples measured with Agilent 85070B Dielectric Probe Kit.

absorption loss. The dielectric constants of air, Teflon, RT-duroid 5880, and Perspex at 10^5 Hz to 1 MHz were found to be 1, 2.1 (Teflon PTFE, Technical Datasheet), 2.2 (Rogers Corporation, Technical Datasheet), and 2.6 (Goodfellow Group, Technical Information-Polymethylmethacrylate) which were in very good agreement with available data.

The slight dispersion for all the samples at the higher frequency end was probably due to several factors. Firstly, the minimum sample thickness recommended by the manufacturer ($d = 30 \text{ mm}/\sqrt{\epsilon'}$) for the 85070B Dielectric Probe Kit should be more than 20 mm for $\epsilon' = 2.05$. The higher the dielectric constant is, the lower the required minimum thickness shall be based on the higher dielectric. Small errors could be attributed to the fact that Dielectric Probe Kit 85070B was designed for liquid materials. The permittivity computation for the Dielectric Probe Kit 85070B was a simplified version of Debye model obtained from empirical fitting of several known liquids [20]; thus the permittivity calculations were less accurate for solid materials.

FIGURE 8: Complex permittivity of raw materials measured with Agilent 85070B Dielectric Probe Kit.

4.2.2. Pure NiO, ZnO, and Fe_2O_3 . The dielectric constant and loss factor consisting of Nickel(II) Oxide (NiO), Zinc Oxide (ZnO), and Iron(III) Oxide (Fe_2O_3) are shown in Figure 8. It could be clearly observed from the graph that NiO had both higher dielectric constant and loss factor compared to ZnO and Fe_2O_3 . The dielectric constants for both ZnO and Fe_2O_3 were almost stable for the whole frequency range. However the dielectric constant of NiO was gradually decreased from 5.5 at 1 GHz to 4 at 10 GHz. Interestingly, it could be observed clearly that NiO had loss factor approximately 5 times larger than ZnO and Fe_2O_3 thus qualifying it to be categorized as a highly loss material.

4.2.3. $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$. The measurement of complex permittivity of $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ samples using open ended coaxial probe with different fractional composition of x was also performed. The thickness of the all samples was 8 mm. Figure 9 shows the results for each $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ sample, where $x = 0.1, 0.3, 0.5, 0.7$, and 0.9 .

FIGURE 9: Complex permittivity of $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ samples measured with Agilent 85070B Dielectric Probe Kit.

The high uncertainties in both ϵ' and ϵ'' at frequencies below 2 GHz were due to multiple reflection effect within the sample. The samples must be infinitely thick to avoid reflection from the end face of the sample. The lower the operating frequencies, the longer the wavelengths and thus the higher the uncertainties due to incident wave reflected at the end surface of the sample. These effects were reduced at higher frequencies especially beyond 3 GHz due to shorter probing wavelength. Generally Figure 9 suggests higher fractional composition of x would result in higher values of the dielectric constant of $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$. At 5 GHz, the value of ϵ' increased from approximately 3.1 to 3.8 for $x = 0.1$ to 0.9. This was expected as Figure 8 showed the dielectric constant of NiO was much higher than both ZnO and Fe_2O_3 . Similarly, the loss factor ϵ'' values for all $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ samples increased with increasing values of fractional composition of x especially at frequencies above 3 GHz.

The effect of fractional composition of x on the dielectric constant in the frequency range between 3 GHz and 10 GHz

TABLE 3: Mean value of dielectric constant $\Delta\epsilon'$ in the frequency range from 3 GHz to 10 GHz.

Symbol, x	Mean value of $\Delta\epsilon'$
0.2	0.1145
0.4	0.3332
0.6	0.4395
0.8	0.7020

FIGURE 10: Variation in $\Delta\epsilon'$ with frequency for various fractional composition values of x .

can be observed clearly in Figure 10 by defining the change in dielectric constant:

$$\Delta\epsilon' = \epsilon'_{x(i)} - \epsilon'_{x=0.1}, \quad (7)$$

where $\epsilon'_{x(i)}$ is the dielectric constant of $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ with $x(i) = 0.3, 0.5, 0.7$, and 0.9.

The mean values $\Delta\epsilon'$ for the whole frequency range from 3 GHz to 10 GHz are summarized in Table 3. A slight change from $x = 0.1$ to 0.3 would give a change of approximately 0.11 in the value of ϵ' and could be as high as 0.70 if x increased from 0.1 to 0.9. The higher the NiO content is, the higher the dielectric constant and loss factor of $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ will be.

5. Conclusion

The permittivity of $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ in the frequency range between 1 GHz and 10 GHz was successfully determined using an open ended coaxial probe technique as higher fractional composition of x would result in higher values of the dielectric constant of $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$. It was found that the lattice constant of the $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ samples decreased linearly with increasing of the substituted amount of x inside the Ni-Zn ferrite sample. The tabulated grain size for all samples was in the range of 62 nm–175 nm.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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