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Chief Editor

Aabiskar Bhusal

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Cover: Ball-and-stick model of MOF-5. © Roshani Sharma. Printed with permission.
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Complex impedance analysis of soft chemical synthesized NZCF systems

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Abstract: The soft chemical method was adopted to prepare of cobalt-substituted nickel-zinc ferrites (Ni₀.⁹₅₋ₓZn₀.⁰₅COₓFe₂O₄ for x = 0.01, 0.02 and 0.03). We have recently studied their structural, morphological, and magnetic properties, initial permeability, and dielectric constant. They were found to be with cubic ferromagnetic spinel structure, the morphology of which is suitable for high-density recording media. The impedance has a major role in characterizing the electrical and magnetic properties of the sample, which are dependent on their permeability and dielectric constant. So, this study will verify the values obtained before. Next, the energy stored in a capacitor is directly proportional, and its size is inversely proportional to the dielectric constant. Similarly, the resistance offered by a material to the magnetic field to pass through it is related to permeability. This study will focus on the variation of impedance with Co²⁺ concentration. The results were derived with the equivalent circuit model. The impedance analysis is also important in the biological field having a name of Bio-Impedance Analysis (BIA), for determining nutritional status, and many more.

Keywords: Ni-Zn ferrites • Initial permeability • Dielectric constant • Magnetocrystalline anisotropy

I. Introduction

Impedance has basically two components: resistance and reactance. The reactance is the resistance offered by the inductor and capacitor. The complex impedance deals with the interdependent voltage and currents with sinusoidal waves comprising the circuit’s electrodynamics. It is frequency sensitive. The fluctuation in current and voltage in the ac circuit stores or release the energy in the form of an electric or magnetic field. The resistance is the real part and the reactance is the imaginary part of the impedance. The imaginary parts with inductive reactance are denoted as +jX_L related to the magnetic field and the capacitive reactance related to the electric field is denoted as -jX_C. So the impedance has two values: R + jX_L and R - jX_C. Initially, the dielectric materials

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were studied with Impedance spectroscopy [1]. Now, it is used for alloys [2] and wires [3, 4] of ferromagnetic nature. The frequency response by the material is the main observation in this study.

The memory and recording media need higher magnetic saturation, permeability, and lower loss of energy. The soft ferrites are under exploration for these purposes [5]. Ni-Zn ferrites have the Curie temperature and higher resistivity of 570°C [6, 7] and 10^6 Ωm respectively. Nickel-Zinc soft ferrites are mostly applied in EM noise absorbers, inductors, and converters [5, 8]. The addition of extrinsic elements like Co^{2+} ion to these Ni-Zn elaborate their range of applications to microwave absorbers etc [9].

B. Parvatheswara et al. [10] found improved magnetic saturation and reduced initial permeability of Ni–Zn–Co ferrites. In Ni_{0.22}Zn_{0.78-x}Co_{x}Fe_{2}O_{4}, the cobalt content strongly affects the coercivity but the magnetic saturation remains independent [8]. Kulikowski [11] found the increasing domain wall energy and decreasing permeability with cobalt concentration. Shinde et al. prepared the Co_{0.4}Ni_{0.3}Zn_{0.3}Fe_{2}O_{4} ferrites nanoparticles by mechanical alloy process and studied the effect of sintering temperature on the permeability which was found proportional to each other [12]. Kim and Koh [13] prepared (Ni_{0.4}Co_{0.1}Zn_{0.5}) Fe_{2}O_{4} ferrites nanoparticles by co-precipitation method and found as an excellent absorber of electromagnetic waves. Shimba et al. [14] found (Fe–B)/(Ni–Zn–Co) nano-composite with a good absorber of microwaves.

We have prepared cobalt substituted Ni_{0.95–x}Zn_{0.05}Co_{x}Fe_{2}O_{4}, (where x = 0.01, 0.02, and 0.03) by easy and cheap soft chemical method and carried their impedance analysis along with dielectric dispersion [15–31]. The structural, morphological, and magnetic properties along with their initial permeability and dielectric constant Ni-Co-Zn were studied recently and published [18, 32, 33].

II. Experimental Procedure

The Ni_{0.95–x}Zn_{0.05}Co_{x}Fe_{2}O_{4} (where x = 0.01, 0.02, 0.03) were prepared by soft chemical route method [34–37]. In the process, the nitrates of Nickel, Zinc, and Cobalt are mixed with Ferric citrate in a molar ratio of 1:1 [37, 38]. The ammonium hydroxide is added until the solution became neutral and is dried for 10 to 12 hours with the addition of ethylene glycol until a puffy and porous dry gel is obtained. The self-ignition gives its fine powder. The carbon content in the powder is removed by its sintering at 800°C for 2 hours. The sintering binds the particles strongly by eliminating pores and completing the reaction.

The as-prepared sample was used for the structural, morphological, and functional tests (whose results are already under publication process). For the magnetic, dielectric, and electrical properties, we have to prepare the pallets (toroids in this case) with a diameter of 12 mm and thickness of 2 mm under 5 tons of hydraulic pressure. The toroids are then sintered in the range of (900-1200)°C for 2 hours and wounded with 100 turns of 30 SWG enameled copper wire.

A computer aided impedance analyzer modeled HP4192A was used for the frequency measurement ranged 5 Hz–13 MHz, with 94 discrete values in the temperature range of 110-450K with preset temperature variation of
Figure 1. Flowchart of the experimental method following the soft chemical method.

Figure 2. Puffy and porous gel dried into a powder.
III. Results and Discussion

Figure 3. Real and imaginary part plot of the impedance at room temperature (NZCF) for (a) x= 0.01, (b) x =0.02 and (c) x =0.03

There has always been a problem for the evaporation of Zn for sintering in higher temperature for longer time. So, Zn content are related with the sintering time. Appropriate stoichiometry can fix the problem [40].

The real part against the imaginary part plot of an impedance for x = 0.01 at room temperature (NZCF) according to equations 1-3 is as shown in Fig. 2(a). The data were taken in the frequency range 20 Hz–1 MHz. A well-structured semicircle was obtained. The open squares and solid triangles indicate the calculated and observed values of $Z'$ and $Z''$. The observed and calculated complex impedance data match each other fairly well. The semicircle size depends on the grain size. If the grain size is increased, the size of the semicircle is decreased, and the grain boundary is increased. This gives rise to the larger impedance of the circuit [41]. Similarly, the plot for the concentration x= 0.02 and 0.03 are shown in Fig. 2(b) and Fig. 2(c) respectively. A single semicircle indicates the conductivity due to the bulk grain at higher frequencies, and the resistivity is indicated by the diameter of the semicircle [42]. The conductivity increases with the cobalt content indicated by the reduced diameter of the semicircle and hence the bulk resistance.

Figure 4. The RC equivalent circuit with Cole-Cole semi-circle.

The resistance and capacitance offered by the bulk grains denoted by (Rg) and (Cg) respectively can be incorporated through the equivalent circuit connected in parallel as shown in Fig. 4 for x= 0.01 to 0.03 samples of NZCF which is in agreement with the Cole-Cole plot in a semicircular arc and with the fitting model by Kleitz et al. [43].

The calculations of real ($Z'$) and imaginary ($Z''$) parts of the complex impedances were done with the help
Complex impedance analysis of NZCF systems

of the following relations [42]:

\[ Z = Z' - jZ'' \]  \hspace{1cm} (1)
\[ Z' = \frac{R_g}{1 + R_g^2 \omega^2 C_g^2} \]  \hspace{1cm} (2)
\[ Z'' = \frac{R_g^2 C_g}{1 + R_g^2 \omega^2 C_g^2} \]  \hspace{1cm} (3)

where \( \omega \) is the angular frequency.

IV. Normalization of the Result

The material of each composition prepared is incorporated in the circuit as above, the first without inductance coil and then with the coil and tested at a temperature higher than the Curie temperature. From this, the frequency response of the ferrite in a simple equivalent circuit model [40] can be studied. Further, the circuit measurement shows the grain size distribution affecting the difference in the values from their ideal ones. Similarly, the microstructure-dependent relaxation frequency \( K1 \) is obtained by directly measuring the macroscopic properties [40].

V. Conclusions

We successfully prepared the sample with the compositions \( x = 0.0 \) to \( 0.3 \) in the \( \text{Ni}_{0.95-x}\text{Zn}_{0.05}\text{CO}_x\text{Fe}_2\text{O}_4 \) ferrites and used them in the electrical circuit for the impedance measurement. The plots of the real and imaginary parts of the complex impedance in the frequency range of \( 20 \) Hz–\( 1 \) MHz gave a perfect semicircular arc for each composition. The increase in cobalt concentration decreases the diameter of the semicircle, indicating an increase the conductivity. The observed data are fitted in an equivalent circuit with the bulk resistance (\( R_g \)) and capacitances (\( C_g \)). The observed semicircle can be modeled by an equivalent circuit consisting of a parallel combination of bulk resistance and bulk capacitance which are fitted well with each Cole–Cole semicircle.

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