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Structural and Magnetic Properties of NiCoZn Ferrite

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***Abstract*—Nickel-cobalt-zinc ferrite, $Ni_{0.5}Co_{0.2}Zn_{0.3}Fe_2O_4$ is interesting as it has the low coercivity and highest magnetization. We prepared $Ni_{0.5}Co_{0.2}Zn_{0.3}Fe_2O_4$ using the auto-combustion route. X-ray diffraction and Field Emission Gun Transmission Electron Microscope (FEG-TEM) studies were carried out for checking the crystal structure and morphology. Magnetic properties of the ferrite were studied by using SQUID Vibrating Sample Magnetometer (SVSM) under an applied magnetic field of ±5 Tesla. An examination of M-H loops revealed highest saturation magnetization of 82 emu/g with soft magnetic nature of the sample at different temperatures.**

***Index Terms*— NiCoZn ferrite, Auto-combustion route, XRD, Magnetization, FEG-TEM.**

I. INTRODUCTION

Ferrites are technologically important materials which can be used in the fabrication of electronic, magnetic and microwave devices [1]. Spinel ferrites are a compound with the chemical formula of MFe_2O_4, where M is a divalent cation and Fe is a trivalent cation. These ferrites exhibit a cubic crystal structure which can be considered as an interlocking network of positively and negatively charged metal ions and divalent oxygen ions (O^{2-}), respectively. The unit cell of spinel ferrites consist of 8 formula units of AB_2O_4, where A and B are the tetrahedral and octahedral sites in the spinel structure [2]. The cation distribution for the NCZFO can be explained on the basis of Neel's two sub-lattice model. According to this model, the A-site is occupied by (Fe^{3+} Zn^{2+}) ions and B-site is occupied by [$Fe^{3+}Co^{2+}$ Ni^{2+}] ions. Among the NiCoZn ferrites, $Ni_{0.5}Co_{0.2}Zn_{0.3}Fe_2O_4$ is one of the most important one due to its maximum saturation magnetization [3].
Many researchers have synthesized NCZFO by physical and chemical routes such as solid-state reaction [4], self-sustaining [5], chemical-combustion [6], co-precipitation [7], sol-gel [8] etc. The present work is focused on structural and magnetic properties of $Ni_{0.5}Co_{0.2}Zn_{0.3}Fe_2O_4$ synthesized by auto-combustion method for micropumps and biomedical applications [9, 10]. The main advantages of the auto-combustion are; fast synthesis process, low energy consumption and high purity of product [11]. Therefore, it is one of the best techniques for material synthesis.

II. EXPERIMENTAL

The composition $Ni_{0.5}Co_{0.2}Zn_{0.3}Fe_2O_4$ (NCZFO) was prepared by the auto-combustion route. In this method, analytical grade reagents; Ni nitrate [$Ni(NO_3)_2.6H_2O$], Co nitrate [$Co(NO_3)_2.6H_2O$], Zn nitrate [$Zn(NO_3)_2.6H_2O$], ferric nitrate [$Fe(NO_3)_3.9H_2O$], were used as oxidizing agents and glycine as a reducing agent. Stoichiometry of the redox mixture for auto-combustion was calculated based on the total reducing

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and oxidizing valencies of the fuel and oxidizer. Initially, the aqueous solution of both the; metal nitrates and glycine were prepared separately with the deionized water and then mixed with each other. The resultant solution was heated at 100 °C on magnetic stirrer. For the combustion, the solution was heated up to 350 °C. During the auto-combustion, large amount amount of gases are released and the product formed was a loose blackish powder. The powder was mixed with 2% PVA as a binder and pressed into disc shaped pellets (8 mm×2 mm) by using hydraulic press which is used to apply a uniform pressure of 6 tons. Finally, the pellets were sintered at 1250 °C for 2 hrs in the muffle furnace.

III. RESULTS AND DISCUSSION

A. Structural properties

We performed X-ray diffraction (Rigaku Smart Lab X-Ray diffractometer) measurements with Cu-Kα radiation on NCZFO powder calcined at 900 °C for 4 hrs. Fig. (1) showed the XRD results conforming to cubic spinel structure of Fd-3m (227) space group. The phase identification, lattice parameter and the particle size of the sample were also estimated from the XRD data. The X-ray diffraction confirmed the formation of single-phase of NCZFO powder at room temperature and revealed that the sample was polycrystalline in nature. The lattice parameter of the sample was determined using (1) [12],

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

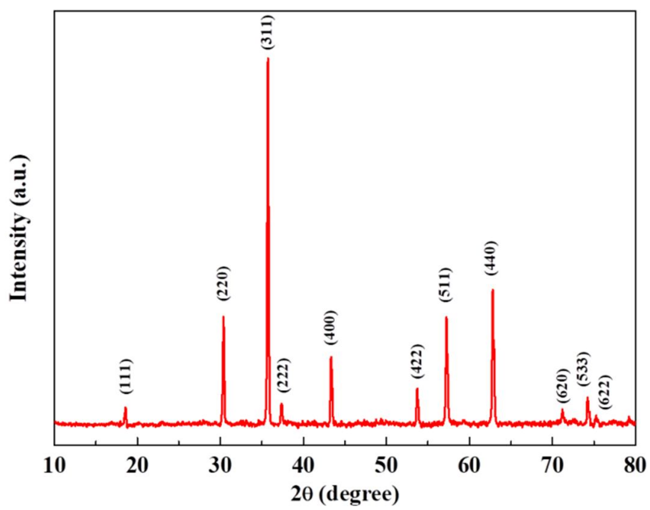

Fig. 1. XRD pattern of $Ni_{0.5}Co_{0.2}Zn_{0.3}Fe_2O_4$ calcined at 900 °C for 4 hrs

The lattice parameter was found to be 8.37 Å. The crystallite size D_{hkl} was calculated from the most intense peak (311) using (2) [12],

$$D_{hkl} = \frac{0.9\lambda}{\beta\cos\theta}, \quad (2)$$

where d_{hkl} is the distance between two planes having Miller indices ($h\ k\ l$), λ is the wavelength of X-rays (1.5406 Å), β is the full-width at half-maximum in radian, and θ the angle of diffraction. The average crystallite size was found about 22 nm. The X-ray density (ρ_x) of NCZFO was calculated using (3) given by Smith and Wijn [13],

$$\rho_x = \frac{nM}{N_A a^3}, \quad (3)$$

where M is the molecular weight of the sample, n is the number of formula units present in a unit cell (for NCZFO, n=8), N_A is the Avogadro's number and a is the lattice parameter. The X-ray density was found to be about 5.35 gm/cm^3. The bulk density (ρ_b) of NCZFO sample was measured using the standard formula, $\rho_b = m/v$, where m is the mass of the sample and v is the volume. Thus the obtained density was 4.81 g/cm^3 which was around 90% of that estimated by X-ray method. This 10% loss of density could be due to the presence of binder, possible porosity in the pellet and other anomalies arising from the material synthesis process.

B. Magnetic properties

The magnetic hysteresis, M-H loops of $Ni_{0.5}Co_{0.2}Zn_{0.3}Fe_2O_4$ sample obtained at different temperatures by using SVSM (7T SQUID magnetometer, Quantum Design, USA) under an applied magnetic field of ±5 Tesla. It was observed from the magnetization curves that the shapes of the loops reveal characteristic features of soft ferromagnetic nature of the sample (Fig.(2)). We found that the values of saturation magnetization (σ_s), remanence magnetization (σ_r) and coercivity (H_c) were decreased as the temperature increases. The measured values of σ_s, σ_r and H_c of NCZFO sample at different temperatures are summarized in table 1.The theoretical value of magnetic moment of NCZFO sample was calculated to be 4.6 μ_B using the cation distribution and their ionic magnetic moments of Ni^{2+} (2 μ_B), Co^{2+} (3 μ_B), Zn^{2+} (0 μ_B) and Fe^{3+} (5 μ_B) [2], The experimental value of the magnetic moment, per formula unit in the Bohr magneton was estimated to be 3.46 μ_B using (4) [13],

$$n_B = \frac{M\sigma_s}{5585}, \tag{4}$$

where M is the molecular weight of the sample and σ_s is the saturation magnetization.

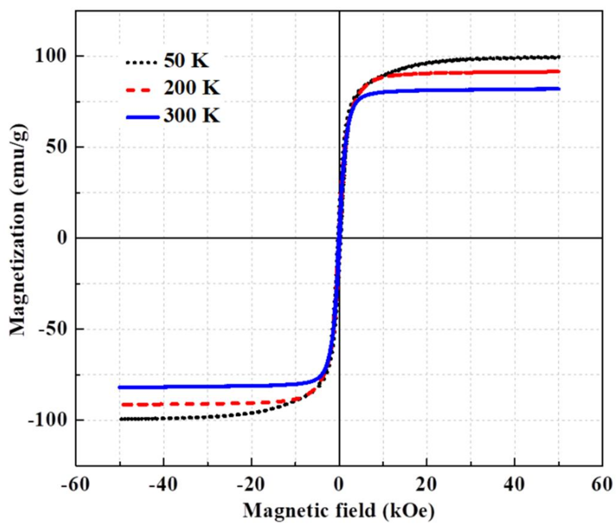

Fig. 2. Magnetic hysteresis loops of $Ni_{0.5}Co_{0.2}Zn_{0.3}Fe_2O_4$ sample measured at 50 K, 200 K and 300 K sintered at 1250 °C for 2 hrs

C. Microstructural analysis

FEG-TEM (JEM-2100F, JEOL) was used to study crystal structure and morphology of NCZFO powder calcined at 900 °C for 4 hrs. Fig.3(a) showed round-shaped NCZFO particles with the particle size of 1±0.2 μm. In Fig.3(b), the high-resolution TEM image showed the crystalline lattice fringes of NCZFO particles. An inter-atomic spacing of 0.25 nm coincides to the d-spacing of NCZFO (311) planes. Fig.3(c) presented the selected area electron diffraction pattern of NCZFO. The polycrystalline distribution characteristics were observed for NCZFO, consistent with the indistinct diffraction rings of the (111), (220), (311), (222), (400), (422), (511), (440), (620), (533) and (622) planes. XRD analysis of NCZFO exposed the signals for NCZFO metallic phase, representing the (111), (220), (311), (222), (400), (422), (511), (440), (620), (533) and (622) reflections, respectively, upon increasing the value of 2θ (Fig.1).

IV. CONCLUSION

$Ni_{0.5}Co_{0.2}Zn_{0.3}Fe_2O_4$ has been successfully synthesized by using the auto-combustion technique. XRD analysis confirmed the formation of single-phase and polycrystalline nature of NCZFO with an average crystallite size of approximately 22 nm. The M-H hysteresis curves revealed record high magnetization of 82 emu/g with soft magnetic nature of the sample at different temperatures. The FEG-TEM study showed the crystal structure and morphology of the particles. The particle size was found 1±0.2 μm. These ferrites are interesting for micropumps and biomedical applications.

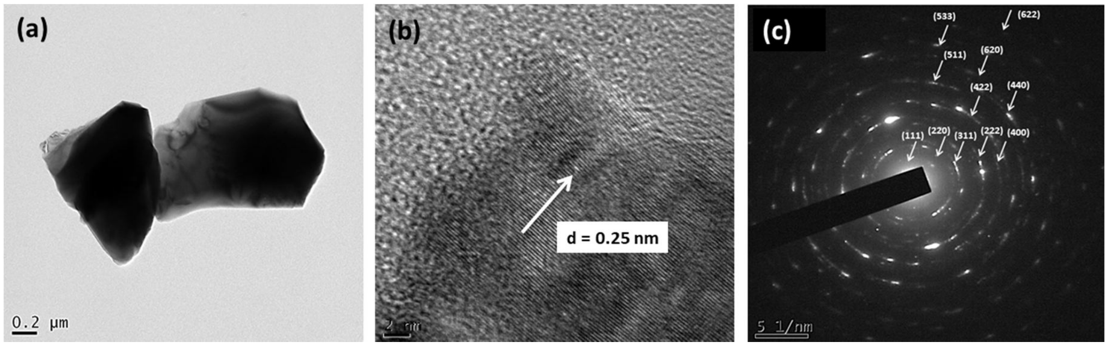

Fig. 3(a) TEM image of $Ni_{0.5}Co_{0.2}Zn_{0.3}Fe_2O_4$, (b) HR-TEM image, (c) Selected area electron diffraction (SAED) pattern

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