

XRD And AFM Study Of Zirconium Substituted Zn-Ni Ferrite Using Solution Combustion Method

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Abstract

$Zr_xZn_{0.5-x}Ni_{0.5}Fe_2O_4$ ($x=0.00, 0.05$ and 0.15) has been synthesized successfully using solution combustion method with the use of high purity nitrates and fueling agent urea. XRD study of obtained sample confirms the cubic spinel structure formation. AFM reveals the surface morphology and microstructure of well dispersed Zn-Ni ferrite nanoparticles to be spherical.

Keywords: *Zn-Ni ferrite nanoparticles, combustion method, surface morphology and microstructure*

1. INTRODUCTION

Zinc-nickel ferrites are so-called normal ferrites, in which the Ni^{2+} and Zn^{2+} ions are located at tetragonal sites, while the Fe^{3+} ions occupy octagonal sites [1]. If the devices are to be miniaturized, investigation on the properties of the nano-scale nickel-zinc ferrites is required. Size reduction in magnetic nanomaterials leads to novel properties, such as superparamagnetism [2], spin glass [3], spin canting [4], and ionic redistribution [5]. Polycrystalline Ni-Zn ferrites could be used in power applications at frequency over 1 MHz as they offer various attractive properties for high frequency usage [6].

Zinc nickel ferrites nanoparticles usually exhibit high saturation magnetization [7].

These ferrites are soft magnetic material that is mostly used as various inductance components, such as transformers, antenna and video magnetic heads and so on [8]. It has also found increased applications in pulse magnetic devices such as pulse power transformers & in magnetic pulse compressor network [9].

The present investigation deals with synthesis of Zn-Ni ferrite. The Zn-Ni ferrite with composition $Zn_{0.5}Ni_{0.5}Fe_2O_4$ was synthesized by solution combustion method. In this work, Zn-Ni ferrite presented a relatively high saturation magnetization and effects in the microstructure, electrical and magnetic properties of the Zn-Ni ferrites were investigated to improve the performance of Ni-Zn ferrites for power field use.

2. PREPARATION AND CHARACTERIZATION OF Zn-Ni FERRITE NANOPARTICLES

2.1 Preparation Method

The metal salts of 0.5 mole Nickel (II) Nitrate ($\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$), 0.5-x mole Zinc Nitrate ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$), x mole Zirconium oxynitrate ($\text{ZrO}(\text{NO}_3)_2 \cdot x\text{H}_2\text{O}$) and 2 mole Iron (III) Nitrate ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$) were dissolved in distilled water and then add 1 mole of urea in it. The resulting solution was mixed and heated at 70°C till it starts boiling. The solution was then heated in muffle furnace at till it ignites. The final obtained product is calcined at temperature of 600°C to 800°C for an hour. Resultant product was grounded in pestle mortar for an hour.

2.2 Characterizations

X-Ray Diffraction (XRD) Cu $K\alpha$ radiation with a JEOL (Panalytical X'pert Pro) operated at 35kV and 25mA in the range of $0-160^\circ$ of the 2θ was used for analysis of crystal structure of prepared sample. The surface morphology and microstructure of the prepared samples were observed using Atomic Force Microscopy (AFM).

3. RESULTS

3.1 Crystal Structure

The XRD patterns of $\text{Zr}_x\text{Zn}_{0.5-x}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$ of Zn-Ni ferrite is shown in figure below.

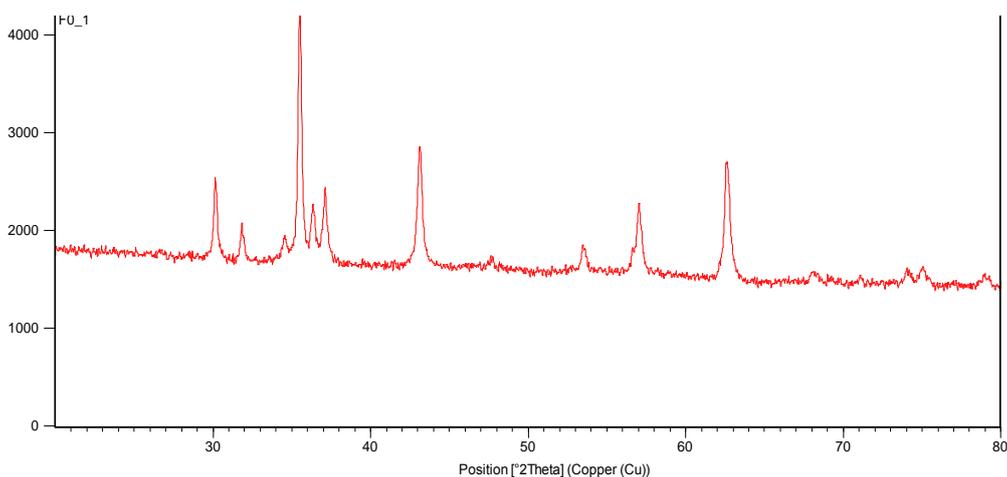


Fig 1a: XRD pattern of $\text{Zn}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$

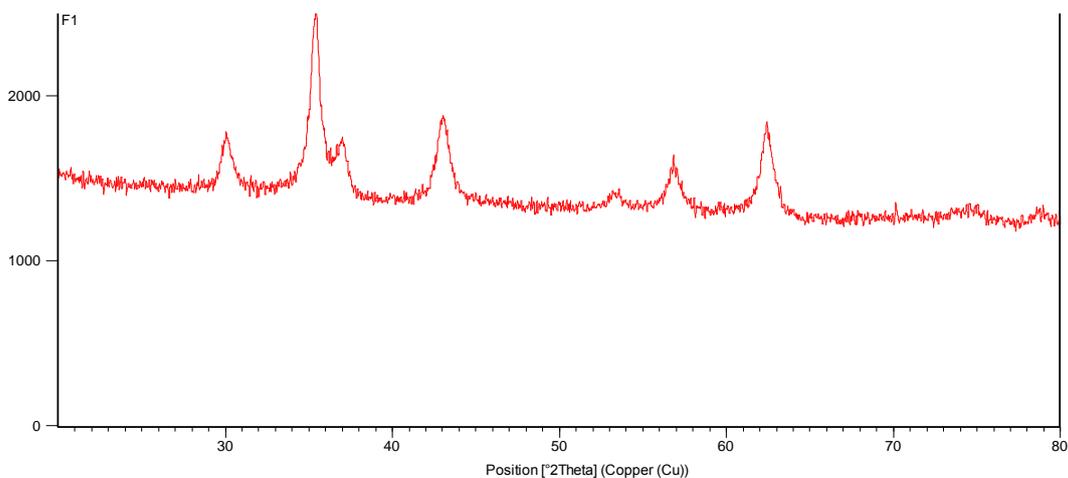


Fig 1b: XRD pattern of $\text{Zr}_{0.15}\text{Zn}_{0.35}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$

The cubic spinel structure of zinc-nickel ferrite is shown in figure. Crystallite size D was calculated using the Debye–Scherrer equation [10]:

$$D = 0.9\lambda / \beta \cos\theta,$$

where ' λ ' is the wavelength of used radiation, ' β ' is the full width half maximum (FWHM) of diffraction peak, and ' θ ' is the Bragg angle.

The average crystallite size D is calculated to be 61 nm and 41 nm of prepared samples of $Zn_{0.5}Ni_{0.5}Fe_2O_4$ and $Zr_{0.15}Zn_{0.35}Ni_{0.5}Fe_2O_4$. However, all the peaks perfectly match with the crystalline phase of cubic spinel structure of Zn-Ni ferrite (JCPDS Card No. 008-0234) [11]. The diffraction peak shifts towards the

minimum angle a little with increase in Zr substitution.

3.2 AFM Study

It is well known that AFM is one of effective ways for the surface analysis due to its high resolution and powerful analysis software [12]. The AFM studies revealed uniform surface without any valleys, due to the homogenously distribution of the ferrite particles. Atomic force microscope (AFM) is widely used to visualize individual particles and groups of particles and unlike other microscopy techniques; it offers visualization in three dimensions also

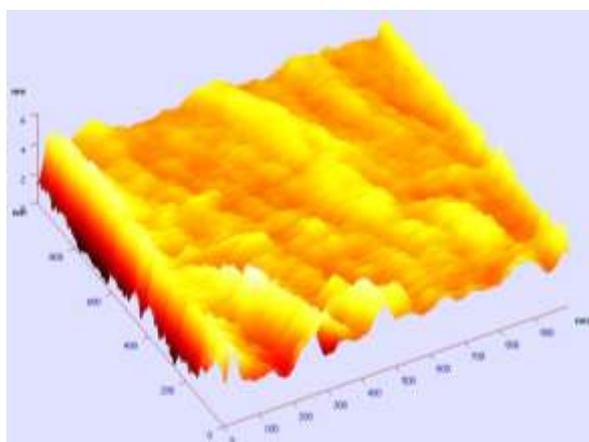


Fig 2a: 3D view $Zn_{0.5}Ni_{0.5}Fe_2O_4$.

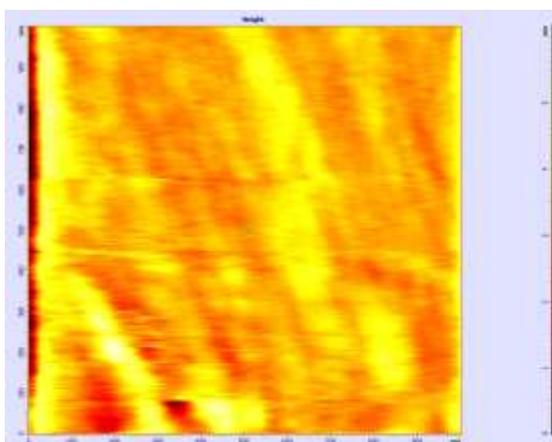


Fig 2b: 2D view $Zn_{0.5}Ni_{0.5}Fe_2O_4$.

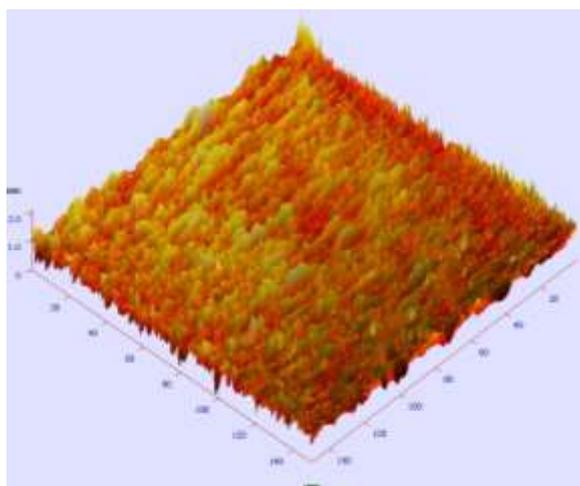


Fig 2c: 3D view $Zr_{0.05}Zn_{0.45}Ni_{0.5}Fe_2O_4$.

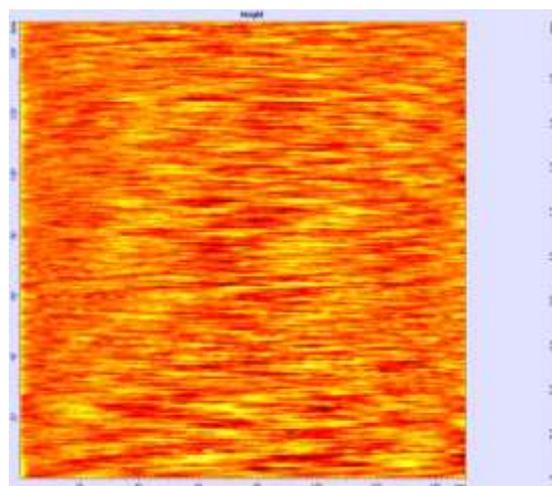


Fig 2d: 2D view $Zr_{0.05}Zn_{0.45}Ni_{0.5}Fe_2O_4$.

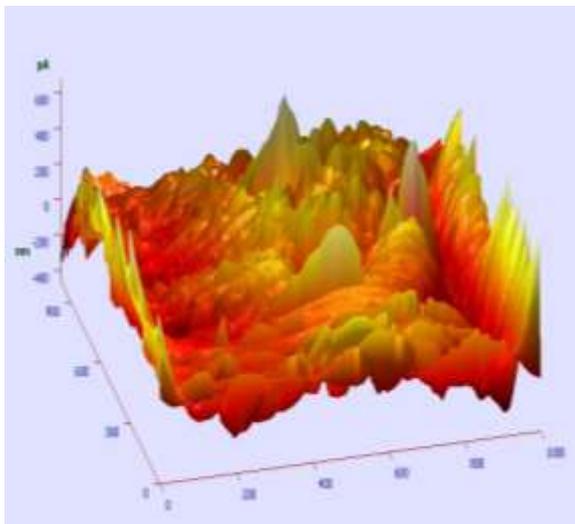


Fig 2e: 3D view $Zr_{0.15}Zn_{0.35}Ni_{0.5}Fe_2O_4$.

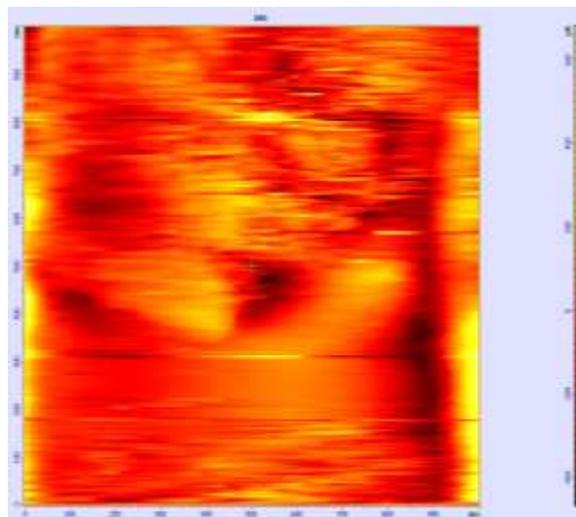


Fig 2f: 2D view $Zr_{0.15}Zn_{0.35}Ni_{0.5}Fe_2O_4$.

The lateral sizes of the nanoparticles varied from image to image because of the variation in powdered samples. However, the height was found to be relatively 2-20nm.

In the figure 2 (a,c,e), the 3D AFM height images are shown, respectively, in a $1\ \mu\text{m} \times 1\ \mu\text{m}$ scanning area, whereas in figure 2 (b,d,f) the 2D height and phase images from the “hill” and the “valley” in 3D images with a scanning area of $1\ \mu\text{m} \times 1\ \mu\text{m}$. A substantial difference in the microstructures of each sample between the “hill” and the “valley” regions can be seen clearly in the prepared samples. The “valley” region is relatively smooth; on the other hand, the “hill” region consists of many crystal-like structures that exhibit certain orientations [13]. These “hills” are probably composed of mostly the byproducts which are the produced during reaction between Zinc and the air.

4. Conclusion

$Zr_xZn_{0.5-x}Ni_{0.5}Fe_2O_4$, show the shifting of diffraction peaks towards the lower angle. Moreover the particles were found to be spherical in shape and the obtained size was found to be 66 nm, 53 nm and 41 nm. The surface morphology and microstructure of well dispersed Zr doped Zn-Ni ferrite nanoparticles revealed that the surface is spherical.

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