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A study on Nickel substituted Zirconium ferrite nanoparticles prepared by coprecipitation route

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Abstract: Magnetic nanoparticles of $ZrFe_2O_5$ and $Ni_{0.5}Zr_{0.5}Fe_2O_5$ have been synthesized by coprecipitation route. Particle size as estimated by full width half maximum (FWHM) of the strongest x-ray diffraction (XRD) peaks were found in the range of $25-35 \pm 4$ nm. SEM images reveal that the sample surface exhibit well defined crystalline nanoparticles of spherical shapes with small agglomeration. TEM was used to characterize the microstructure of the samples and particle size determination, which exhibited the formation of spherical nanoparticles.

Keywords: Nanostructured materials, Precipitation, Magnetization, Scanning electron microscopy (SEM), Transmission electron microscopy (TEM).

1.Introduction:

The possibility of preparing ferrites in the form of nano particles has opened a new and exciting research field, with revolutionary applications not only in the electronic technology but also in the field of biotechnology [1]. Ferrites have many technological applications in permanent magnets, magnetic drug delivery, medical diagnostics, microwave absorbing materials, high density information storage technology, broadband transformers, magnetic resonance and ferrofluid technology [2-6]. Nickel ferrite, a kind of soft magnetic materials is one of the most attracting class of materials due to its interesting and important properties and has many technical applications, such as in catalysis, sensors etc [7]. To the best knowledge of authors, $ZrFe_2O_5$ has never been doped with nickel and neither its properties nor tests like XRD, SEM, and TEM have been discussed.

2. Experimental Procedure:

Ferrites nanoparticles of $Ni_{0.5}Zr_{0.5}Fe_2O_5$ were prepared by co precipitation method. The starting materials used were Nickel chloride ($NiCl_2 \cdot 6H_2O$), Ferric chloride anhydrous ($FeCl_3$), zirconium (III) chloride ($ZrCl_3$) of 99.999% purity and sodium hydroxide (NaOH), all from Alfa Aesar and were of Analytical Grade. Polyethylene glycol – 400 (PEG -400) was used as a surfactant. The molarity of the coprecipitation agent (NaOH) was 3mol/l. The solution of $CoCl_2 \cdot 6H_2O$, $FeCl_3$, $ZrCl_3$, in their stoichiometry (100 ml of 0.1M $NiCl_2 \cdot 6H_2O$, 50 ml of 1.4M $FeCl_3$, 50 ml of 0.6M $ZrCl_3$, in the case of $Ni_{0.5}Zr_{0.5}Fe_2O_5$ and similar for other values of x) were mixed in double distilled de – ionized water. The salt solutions were mixed together with continues stirring. The neutralization is carried out with NaOH solution, and the pH is maintained around

12. Few drops of PEG – 400 were added to the precipitate and heated at 80°C using a hot plate with continuous stirring for 2 hrs. The resultant precipitate was then cooled to room temperature. The precipitate was washed and filtered several times with deionized distilled water. The precipitate was then dried at 100°C for overnight. The dried sample was sintered for 5 hrs at 500°C. After sintering the sample was grinded using the ball milling apparatus and the resulting powder sample was subjected to XRD, SEM and TEM characterization.

3. Results and Discussion

3.1. XRD Structural Analysis:

Fig (1) represents the XRD pattern of $ZrFe_2O_5$ and $Ni_xZr_xFe_2O_5$ (x : 0.5 and 0.5 at 500 °C). All the samples are in single phase cubic spinel structure. No other phase / impurity were detected.. The X-ray diffraction patterns of $Ni_xZr_xFe_2O_5$ (x : 0.5 and 0.5 at 500 °C) as shown in fig shows that the peaks at 24 and 42 were observed which are not present in the $ZrFe_2O_5$. This shows that the peaks at 24 and 42 correspond to NiO as an impurity. The formation of NiO is possible because of the two reasons, either due to the access oxygen that can cause the formation of intermediate products (NiO) instead of ferrites or due to the broken oxygen bonds at the surface causing incomplete exchange bonds resulting in the formation of nickel oxide. The average crystalline size of the powder sample was found to be 35 nm and 29 nm for the Zr- ferrite and for the Zr- ferrite containing 0.6 mol of Nickel.

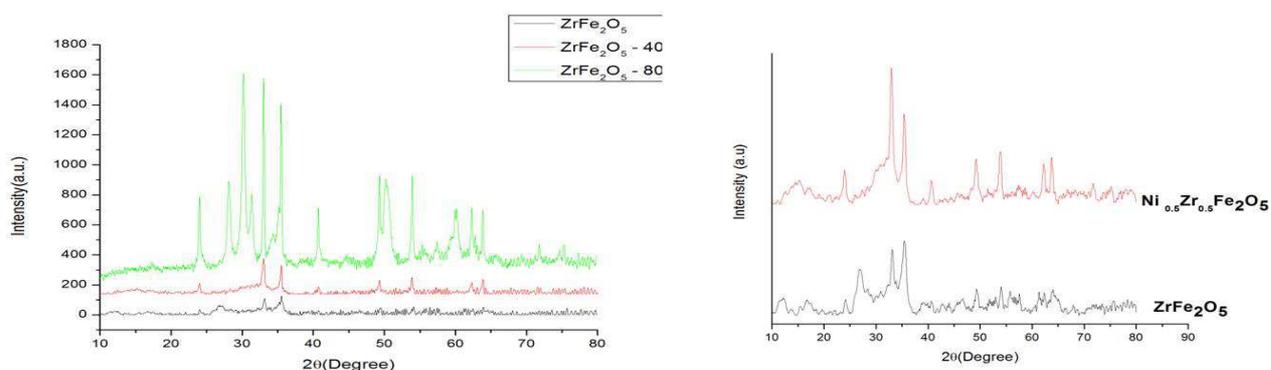


Fig: 1. XRD spectra of $ZrFe_2O_5$ and $Ni_xZr_xFe_2O_5$ (x : 0.5)

3.2. SEM Analysis:

The morphology (homogeneity and particle size) for the sample was determined using Field- Emission Scanning Electron Microscope (FE-SEM) using SUPRA 55-Carl ZEISS instrument. Figure (2) shows the SEM images of $ZrFe_2O_5$ and $Ni_xZr_xFe_2O_5$ (x : 0.5) nanoparticles. The crystalline size is around 24 nm which is nearer to the size obtained in XRD analysis.

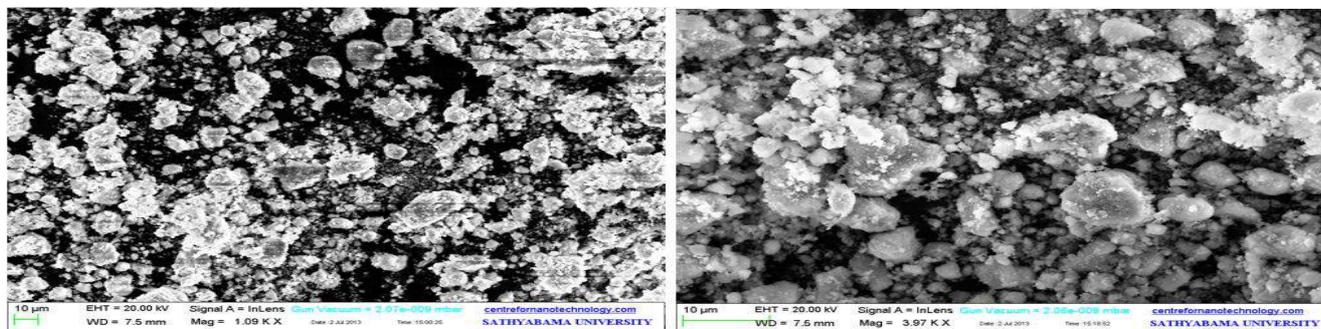


Fig: 2. SEM images of $Ni_xZr_xFe_2O_5$ (x : 0.5)

3.3. TEM Analysis:

The size distribution of $Ni_{0.5}Zr_{0.5}Fe_2O_5$ nanoparticles was calculated using the TEM images. It can be calculated only for the particles that are well separated from each other; for the agglomerated particles it is difficult to

estimate the particle size distribution. From the TEM image the size of the $\text{Ni}_{0.5}\text{Zr}_{0.5}\text{Fe}_2\text{O}_5$ is found to be 22nm. Most of the particles appear spherical in shape.

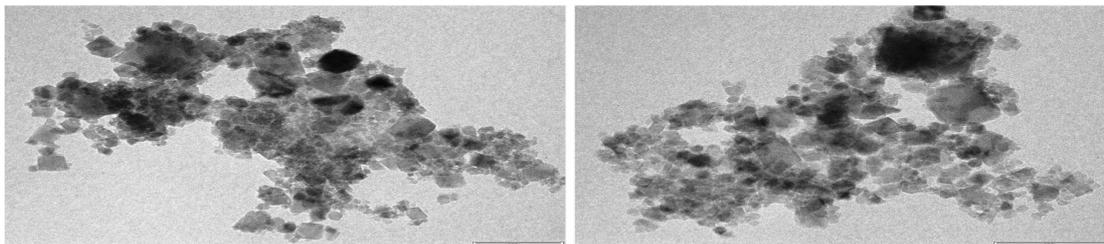


Fig 3: TEM images of ZrFe_2O_5 and $\text{Ni}_x\text{Zr}_x\text{Fe}_2\text{O}_5$ (x: 0.5)

3.4. VSM:

The magnetic hysteresis loops measured at room temperature for $\text{Ni}_{0.5}\text{Zr}_{0.5}\text{Fe}_2\text{O}_5$ at 800°C is shown in fig (4) with a maximum applied field upto 10 Oe. The value of remanent magnetization (M_r) is 33.78 emu/g, saturation magnetization (M_s) is 0.179 emu/g

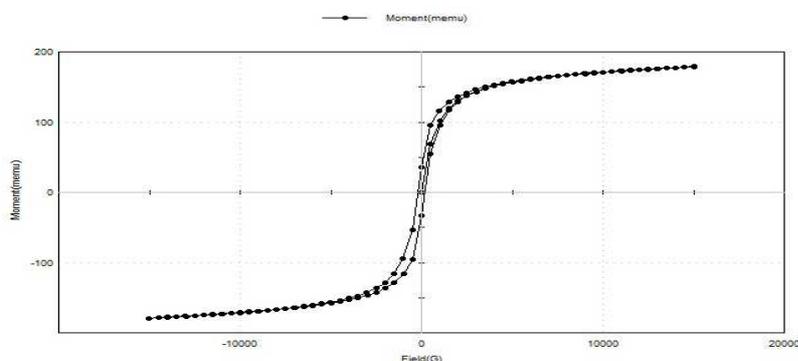


Fig 4: Hysteresis loop of $\text{Ni}_x\text{Zr}_x\text{Fe}_2\text{O}_5$ (x: 0.5) at 800°C

4. Conclusion:

Nickel substituted zirconium ferrites $\text{Ni}_{0.5}\text{Zr}_{0.5}\text{Fe}_2\text{O}_5$ are prepared by co-precipitation method and annealed at 500°C . The XRD spectra reveal the average crystallite size to be in the range of 35 -40nm for pure zirconium ferrites and 32-35nm for Nickel substituted zirconium ferrites. The morphology and the particle size was determined using FESEM and TEM. The observed coercivity (H_c), remanent magnetization (M_r), saturation magnetization (M_s) of the Ni substituted Zirconium ferrite decreases with the increase in Ni content when compared to pure Zirconium ferrite. The saturation magnetization decreases, whereas there is increase in the coercivity which is attributed to the decrease in grain size and increase in porosity.

Acknowledgment:

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