

Characterization and Mössbauer Study of $\text{Ni}_{0.45}\text{Zn}_{0.55}\text{Fe}_2\text{O}_4$ Nanoparticles Prepared by Novel Precursor Method

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Abstract. Nanoparticles of $\text{Ni}_{0.45}\text{Zn}_{0.55}\text{Fe}_2\text{O}_4$ were chemically synthesized by novel precursor method at low temperature. Average particles size was found to be 37nm. The Mössbauer spectroscopy studies were carried out on the sample for the investigation of magnetic and structural arrangement of Fe in the nano-crystalline ferrite samples. The values of isomer shift are consistent with Fe ions in trivalent state. The low values of the quadrupole splitting identifies that the local symmetry of the magnetic phase of A-sites is close to cubic while that of B-site is close to trigonal. The broad collapsing sextet B_1 can be correlated to the presence of relaxation effect in the sample.

Keywords: Nanoparticles, XRD, Mössbauer spectroscopy, Isomer shift, Quadrupole splitting.

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INTRODUCTION

Ni-Zn ferrites having spinel crystal structure are extensively used in a number of electronic devices because of their high permeability at high frequency, remarkably high electrical resistivity, mechanical hardness, chemical stability and reasonable cost [1]. Superparamagnetism, collective magnetic excitations, low saturation magnetization, metastable cation distributions etc., are some of the phenomena which have been observed in nanoparticles of various ferrites. In the present article the findings of the Mössbauer spectroscopy studies carried out in order to investigate magnetic and structural arrangement of Fe in the nano-crystalline ferrite sample $\text{Ni}_{0.45}\text{Zn}_{0.55}\text{Fe}_2\text{O}_4$ prepared via novel precursor route is being reported.

EXPERIMENTAL

Nanoparticles of $\text{Ni}_{0.45}\text{Zn}_{0.55}\text{Fe}_2\text{O}_4$ were prepared by chemical method of preparation using auto combustion. The precursor method employed uses metal salts in form of metal nitrates. Corresponding

metal salts were taken in stoichiometric proportions along with a suitable complexing agent. The XRD pattern was recorded on Rigaku X-ray diffractometer using Cu-K_α radiation. IR spectrum was obtained on Shimadzu FTIR 8900 spectrometer in KBr medium. The room temperature Mössbauer spectrum of the sample was recorded in constant acceleration mode using a ⁵⁷Co Mössbauer source.

RESULTS AND DISCUSSION

The X-ray diffraction pattern for $\text{Ni}_{0.45}\text{Zn}_{0.55}\text{Fe}_2\text{O}_4$ is shown in fig1. The positions of the observed peaks are in agreement with the peaks reported in JCPDS file no 8-234. The peaks indexed to (220), (311), (222), (400), (422), (511), (440), (620), (533), (622) and (444) planes are of cubic unit cell, which confirms the single phase cubic spinel structure of the sample. The average particle size was estimated by using Debye Scherrer formula for seven major peaks and is found to be 37 nm. The lattice constant 'a' (8.4064 Å) is in good agreement with literature reported [2]. IR absorption spectrum shows two broad absorption

bands as shown in figure 2. The higher band ν_1 is between $610\text{-}550\text{ cm}^{-1}$ and lower band ν_2 is between $450\text{-}375\text{ cm}^{-1}$. These two absorption bands is a common feature for all the ferrites in general. This reveals the formation of single phase spinel structure with two sub lattices [3]. The band ν_1 corresponds to intrinsic stretching vibrations of the metal at the tetrahedral site whereas ν_2 band is assigned to octahedral metal stretching.

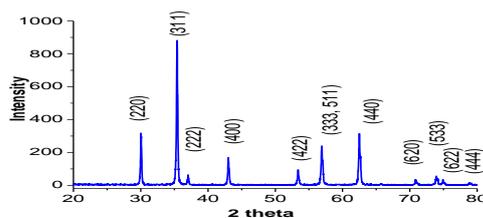


FIGURE 1. XRD pattern of $\text{Ni}_{0.45}\text{Zn}_{0.55}\text{Fe}_2\text{O}_4$.

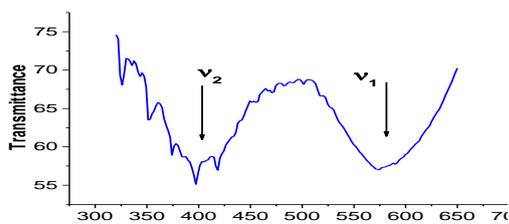


FIGURE 2. IR spectrum of $\text{Ni}_{0.45}\text{Zn}_{0.55}\text{Fe}_2\text{O}_4$.

The Mössbauer spectrum (fig. 3) was fitted with three magnetic sextets which arise due to Zeeman splitting. The solid lines represent the simulated curves and the solid circles represent the experimental data points. The two zeeman split sextets corresponding to Fe^{3+} ions at A-site (inner) and B-site (outer) sublattices is an indication of ferromagnetic behaviour of the sample [4]. The existence of the six-line magnetic pattern is due to the superexchange interaction between the magnetic ions at A and B-sublattices. The third broad sextet B_1 showed typical sextet patterns with the signature of magnetic relaxation characterized by a significant reduction of the magnetic hyperfine field [5]. Mössbauer parameters are given in table 1.

The results of the isomer shift shows that all the components are due to Fe^{3+} ions. The smaller value of A-site isomer shift is due to a large covalency at the A site. The observed low values of the quadrupole shift of A and B magnetic pattern for the sample indicates that, the local symmetry of the magnetic phase of A-sites is close to cubic while that of B-site is close to trigonal [6]. For a Fe ion at A-site, its metallic ion neighbors at B-sites are all magnetic. This ratio, on an average is of the order of 3Fe ions to 1Ni ion. Therefore, the hyperfine field at the A-site Fe nuclei is

higher. For Fe at B-site, however, its six metallic ion neighbors at A- sites may include any number of nonmagnetic Zn^{+2} ions (zero to six). Thus there is a reduction in hyperfine field due to the presence of Zn^{+2} ions [7]. The correct amount of Fe^{3+} ions occupying A- and B-sites could be estimated from the area under the Mössbauer absorption spectrum.

TABLE 1. The hyperfine Field values (H_{hf}), isomer shift (δ), quadrupole splitting (ΔE_Q), Outer linewidth (I) and areas in percentage of tetrahedral (A) and octahedral (B) sites of Fe^{3+} ions for $\text{Ni}_{0.45}\text{Zn}_{0.55}\text{Fe}_2\text{O}_4$. δ values are relative to $\alpha\text{-Fe}$.

Site	(H_{hf}) kG	(ΔE_Q) mm/s	(δ) mm/s	(I) mm/s	Area (%)
A	435.68	0.0139	0.2711	0.8150	18.29
B	382.73	0.0015	0.3117	1.9828	41.37
B_1	134.30	0.0289	0.3003	4.1608	40.34

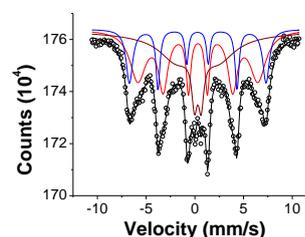


FIGURE 3. Room temperature Mössbauer spectrum of $\text{Ni}_{0.45}\text{Zn}_{0.55}\text{Fe}_2\text{O}_4$.

CONCLUSION

Nanoparticle $\text{Ni}_{0.45}\text{Zn}_{0.55}\text{Fe}_2\text{O}_4$ sample was prepared by using novel precursor method. The average crystallite size was 37 nm. The room temperature Mössbauer spectrum exhibits some relaxation effects due to superparamagnetic particles but their strength is considerably weak compared to that of ordered particles. Cation distribution may be further estimated using area under the Mössbauer absorption spectrum.

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