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Synthesis of Nanoparticles $Ni_{0.55}Zn_{0.45}Fe_2O_4$ by Novel Precursor Method Showing Enhanced Resistivity

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Abstract. Nanoparticles $Ni_{0.55}Zn_{0.45}Fe_2O_4$ was synthesized at low temperature using novel precursor method. The single spinel phase crystallization of sample was confirmed using XRD and IR spectra. Nanosize particles were confirmed using XRD and TEM images. Isomer shift in Mossbauer spectral studies are consistent with Fe ions in trivalent state. DC resistivity was of the order of 10^8 ohm cm that is two orders higher than those reported for NiZn ferrites prepared by conventional ceramic method. The high resistivity was attributed to nanosize particles, absence of Fe^{+2} ions and method of preparation.

Keywords: Nanoparticles, XRD, TEM, Mossbauer spectroscopy, DC resistivity.
PACS: 61.05 a, 61.05c, 61.05cp, 61.05cf, 82.80Gk, 71.20Ps, 78.40Ck, 76.80.+y, 61.05.Qr

INTRODUCTION

Ni–Zn ferrites are a well-known class of technologically important ferrites particularly at high frequencies, because of its high resistivity and therefore low eddy current losses [1]. Ni–Zn ferrite prepared by conventional ceramic method involves solid-state reaction and has some serious limitations such as requires prolonged heating at high temperatures which may result in evaporation of some of the constituents and thereby changing the desired stoichiometry. Also, zinc volatilization at high temperatures results in formation of Fe^{+2} ions thereby increasing the electron hopping and reducing the resistivity. Also the grinding or milling operations involved in the process lead to loss of some material and to impurity pick-ups which result in non stoichiometric compositions. In the recent past, non conventional wet-chemical methods have been found to have distinct advantages over the conventional dry process [2].

EXPERIMENTAL

As per the requirement of composition of ferrite sample, calculated amount of ferrous nitrate, nickel nitrate and zinc nitrate (all salts of AR grade) were dissolved together in water and mixed thoroughly using magnetic stirrer. This mixture was then added to a solution containing appropriate amount of nitrilotriacetic acid and hydrazine hydrate (both AR grade). The entire solution was then heated on a hot plate maintained at temperature around 90 to 95^0C. Heating was continued till mixture gets converted into a solid sticky mass. This solid sticky mass was collected in a crucible and then decomposed using microwave oven. The thick paste containing precursor complex burns spontaneously with a dark red flame resulting into required ferrite powder. The X-Ray powder diffraction pattern, IR spectrum in KBr medium and TEM image was recorded on Rigaku X-ray diffractometer, Shimatzu FTIR 8900 spectrometer and Philips transmission electron microscope respectively. The room temperature Mossbauer spectra of the sample was recorded in constant acceleration mode using a ^{57}Co radioactive source. The ferrite powder was pressed into pellets of 10 mm diameter and 1.5 mm thickness at a pressure of 7.5 ton. The pellets were then coated with silver on both surfaces for having good electrical contact. Dc-resistivity was measured by two probe method using Keithley electrometer in the temperature range of 30–500 °C at

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an interval of 5 °C.

RESULTS AND DISCUSSION

The XRD pattern for $Ni_{0.45}Zn_{0.55}Fe_2O_4$ is as shown in fig1. The positions of peaks comply with JCPDS file no 8-234 for single phase cubic spinel structure of the sample. Average particle size calculated using Debye Scherrer formula was found to be 58 ± 14 nm. The lattice constant 'a' (8.3855 Å) is in good agreement with reported literature.

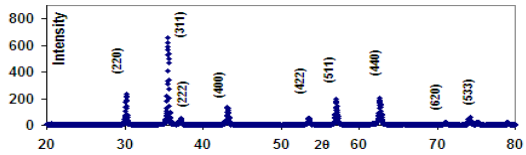

FIGURE 1. XRD pattern for $Ni_{0.55}Zn_{0.45}Fe_2O_4$.

IR absorption spectrum (fig.2) shows two absorption bands. The higher band is between 610-550 cm^{-1} and lower band is between 450-375 cm^{-1}. This also reveals the formation of single phase spinel structure having two sub lattices. The nanosize nature of sample was further confirmed by TEM images (fig3 (a) and (b)). The bright-field TEM images represent the basic morphology of the nanoparticle powder. The histogram depicts particle size distribution having maxima for 60-70nm and is comparable with the crystallite size calculated from XRD data. In Mossbauer spectrum (fig.4), the solid lines represent simulated curves and solid dots represent experimental data points. The two zeeman split sextets corresponding to Fe^{3+} ions at A and B-site sublattices indicating ferromagnetic behaviour of the sample. Third broad sextet showed typical sextet patterns with the signature of magnetic relaxation. Isomer shift values (0.272 to 0.353 mm/s) shows Fe ions are in trivalent state [3]. The absence of Fe^{+2} ions in the sample is one of the parameter to increase resistivity of the ferrites. The effect of temperature on DC electrical resistivity is presented in fig.5.Initially, at the relatively lower temperature resistivity increases with increase in temperature and then starts decreasing. This initial metallic behavior may be due might be due to phonon scattering or due to condensation of carriers to the density of localized states within the mobility gap that takes place in case of the disordered nanoparticles systems [4]. Similar transition was also seen in doped ferrites and layered perovskite oxides of 3d elements. This kind of behavior was attributed to the change in spin ordering from collinear to noncollinear in the B sites [5]. With further increase in temperature resistivity starts decreasing obeying Arrhenius characteristics typical of a semiconductor behavior. A change of slope was also observed at further higher temperature around 340-350 °C and is related to magnetic transition from ferrimagnetic to paramagnetic state. The highest resistivity value of the order of $3.244x10^8$ ohm-cm (at 50°C) was obtained in the present work which is two orders higher than that of the reported values for Ni–Zn ferrites prepared by conventional ceramic method [6].

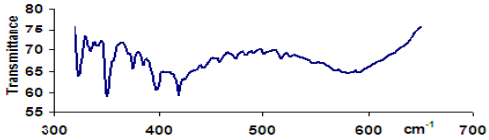

FIGURE 2. IR spectrum for $Ni_{0.55}Zn_{0.45}Fe_2O_4$.

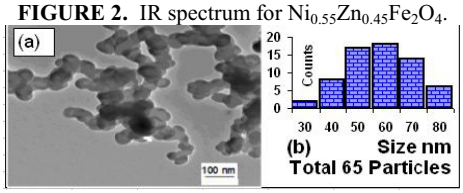

FIGURE 3. TEM image & Histogram of $Ni_{0.55}Zn_{0.45}Fe_2O_4$.

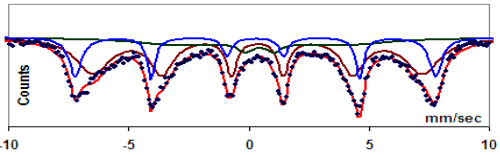

FIGURE 4. Mössbauer spectrum for $Ni_{0.55}Zn_{0.45}Fe_2O_4$.

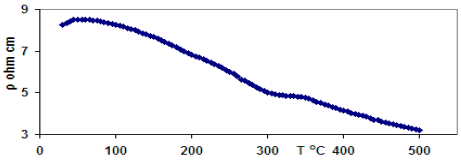

FIGURE 5. Variation of DC resistivity with temperature.

CONCLUSION

Nanoparticles $Ni_{0.55}Zn_{0.45}Fe_2O_4$ was successfully prepared at low temperature by novel precursor technique. The high resistivity is attributed to nanosize particles, absence of Fe^{+2} ions and method of preparation. This shows the promise of our method in preparing high performance ferrites suitable for high-frequency applications where eddy current losses are of paramount concern.

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