Chapter-V

Summary and Conclusions

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The electric and magnetic properties of nanocrystalline materials of both ferrimagnetic and ferromagnetic materials have attracted much attention in recent years. The number of techniques, both physical and chemical, have been developed to prepare nanosized magnetic materials. The nanocrystalline materials exhibit a variety of properties superior than those of conventional grained polycrystalline materials. The technical importance of ferrites lies in their high resistivity, low eddy current losses, large permeability and high value of magnetization. Recently the ferrites have found applications in new areas like environment protection and medicine.

Lithium ferrites are attractive because of the squareness of the hysteresis loop coupled with superior high temperature performance. Lithium ferrite has high resistivity and Curie temperature. It has been noted that properties of lithium ferrite can be suitably modified by addition of other substitutions. Hence the study of effects of substitution of Ni on the electrical and magnetic properties of nanocrystalline $Li_{0.5}Ni_{1.5x}Fe_{2.5-x}O_4$ ferrites with x = 0.1, 0.2, 0.3, 0.4 and 0.5 has been undertaken in the

present work. The work involves preparation, characterization and measurement of electrical and magnetic properties of these ferrites in the nanocrystalline form.

The present work is presented in five chapters, viz. introduction to nanocrystalline ferrites, synthesis and characterization of ferrites, electrical properties, magnetic properties and last chapter summary and conclusion.

The first chapter is introductory which includes, structure of ferrite, classification of nanocrystalline materials, properties of polycrystalline lithium ferrite, literature survey of lithium ferrite, applications of nanocrystalline material and orientation of the problem work. The references are given at the end of the chapter.

The second chapter is subdivided in to four Parts -Part A represents the synthesis of nanocrystalline ferrites. The nanocrystalline material preparation methods are discussed. The present ferrite samples were prepared by fast firing method (Pramanik method). The details of actual ferrite preparation process are given here. In Part B, the results of x- ray diffraction patterns recorded using Philips model PW 1700 using filtered CuKα radiations (λ = 1.5418 Å) are explained. The well-defined peaks corresponding to different reflecting planes confirm the formation of single-phase spinel structure of $Li_{0.5}Ni_{1.5x}Fe_{2.5-x}O_4$ ferrites (x = 0.1, 0.2, 0.3, 0.4 and 0.5). The lattice constant, bond lengths, site radii are determined. It is observed that these parameters increases with Ni^{2+} up to x = 0.1 to 0.3

and then decreases. For lithium ferrite the value of lattice parameter has been reported to be 8.33 Å by many workers. The present value of lattice parameter is 8.32 Å. In the case of Li-Ni ferrites lattice parameter value agrees with this value. The observed and calculated 'd' values are good agreement with each other.

The variation of lattice parameter with nickel content shows that the lattice parameter increases with nickel content up to x = 0.3. The Ni^{2+} ions have larger ionic radius (0.74 Å) than Fe^{3+} (0.65) ions and Li^{1+} (0.71 Å) ions. The Ni^{2+} ions successively replace Fe^{3+} on A-site. This results in increase of lattice parameter. The x- ray diffraction data was further used to calculate bind length R_A, R_B and site radii r_A, r_B.

The Part C of the chapter deals with scanning electron microscopic (SEM) studies of ferrites. It includes the aspect microstructure, such as porosity and grain growth. The SEM micrographs of the samples were taken on scanning electron microscope model JEOL-JSM 6360. It shows that grain size varies in the range 175 to 109 nm as content of Ni^{2+} increases in the lithium ferrite. The porosity is in the range of 18 to 23%. In part D the IR absorption spectra of the samples are discussed. The absorption spectra have been recorded at the room temperature in the range of 400-800 cm^{-1} in KBr medium on Perkin Elmer IR spectrometer Spectrum-1. The two main bands υ_1 and υ_2 are in the range of 620-590 cm^{-1} and 420-410 cm^{-1} respectively. The splitting of the principle bands have been

attributed to Jahn -Teller distortion in the lattice produced by Fe^{2+} ions which locally produces deformation in the lattices. The intensity of shoulders decreases with increasing Ni^{2+} content. Similar shoulder splitting and disappearance of IR absorption band has been reported.

Chapter III is devoted to electrical properties of ferrites. It is divided into two parts, Part A deals with DC. resistivity while the Part B with dielectric behaviour which covers variation of dielectric constant, loss tangent and a. c. conductivity with frequency. The d. c. resistivity measurements were carried out using two probe method from room temperature to 600^0 C. It has been observed that the resistivity decrease with increase in temperature. The plots of log ρ vs 1000/T linear, with breaks at the Curie temperature. The change in slope is observed when the material changes from ordered ferrimagnetic to disordered paramagnetic state which involve different activation energies [1]. The activation energy in paramagnetic region is higher than that in the ferrimagnetic region. The activation energy in ferromagnetic region has been observed to vary from 0.35 eV to 0.86 eV. Observed activation energies are greater than 0.2 eV. This gives an evidence of polaron hopping mechanism of conductivity in the present ferrites.

Part-B of the chapter deals with dielectric behaviour of the ferrite samples. Dielectric constant was measured at room temperature on LCR meter bridge HP 4284 A in the frequency range 10 Hz-1 MHz. The real

part of complex dielectric constant (ϵ') decreases rapidly with increasing frequency and remains constant at higher frequencies. The variations of ϵ' with frequency reveals dispersion due to Maxwell-Wagner [2, 3] type interfacial polarization in agreement with Koop's phenomenological theory [4]. The maximum dispersion of dielectric constant occurs for the sample with x = 0.1. The electronic polarization and heterogeneity of the sample may contribute the dispersion of ϵ'. At the higher frequency dielectric constant is non sensitive to heterogeneity effect and also polarization. Beyond a certain frequency of a. c. applied field the dielectric constant (ϵ') remains constant.

From the plots of tan δ vs frequency of the samples, it is observed that loss tangent decreases with frequency and also shows dispersion at lower frequencies. Similar results were observed by Reddy et al. [5] Variation of a. c. conductivity with frequency has also noted.

Chapter IV includes magnetic properties of the samples. The chapter is divided into two parts, Part-A deals with theory of magnetization and magnetic hysteresis while Part B- deals with ac susceptibility studies.

The hysteresis properties of ferrites were recorded on high field loop tracer developed by Magneta, Mumbai. The substitution of Ni^{2+} ions at B site transfers Fe^{3+} ions from B-site to A-site and affects the magnetic moment of an individual sublattices as well as the A-B interaction. This

results in the increase of saturation magnetization. Further Ni^{2+} substitution in lithium ferrite weaken the A-B interaction and prefer the B-B interaction and hence the net magnetic moment decreases. This results in the increase of Ms and η_B for the composition of x = 0.2 and later on it decreases. The decrease in saturation magnetization in the samples can be explained on the basis of the change in the magnetization M_A and M_B on tetrahedral and octahedral sub lattices respectively. The partial replacement of Fe^{3+}ions by Ni^{2+}ions results in lowering of values of M_A and M_B. The decrease of $[M_A-M_B]$ decreases the net magnetization and hence the magnetic moment. Saturation magnetization and magnetic moment can be explained on the basis of Neel's two sublattice model. The variation of η_B with content of Ni^{2+} is similar to the variation of Ms with Ni^{2+} content.

The AC susceptibility of the samples was recorded using double coil set up operating at 260 kHz in rms field of 7 Oe. The variation χ_{ac} versus T shows that the normalized ac susceptibility does not change appreciably with temperature and suddenly drops out at the Curie temperature and remains zero, for x = 0.4 and x = 0.5 indicating MD particle in the samples. However for x = 0.1, 0.2 and 0.3 normalized ac susceptibility decreases with temperature indicating SP particles.

The chapter V contains summary and conclusions.

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