CHAPTER-IV

SUMMARY AND CONCLUSIONS

The continuous struggle against the eddy current losses during the past several years opened an avenue for a surprisingly new class of magnetic materials called " Ferrites ". The number of research workers in Bell laboratories and elsewhere contributed significantly to the development of ferrite technology. The technical importance of ferrites lies primarily in their high resistivity. The recent achievements in sophisticated applications for cores of transformers in radio and video frequencies, antenna rods in miniaturizing transistor designs and computer science are more impressive and important than ever. Most widely used low frequency ferrites are from spinel group.

The microstructure plays a dominent role in determining high power ability as well as low power loss in saturated microwave devices. Fine grained ferrites possess higher low power loss and are able to handle higher γf power. In the recent years much progress has been made in the control of ferrite material properties through chemical composition and preparation technique.

In the present case copper-cobalt ferrites have been selected particularly because these ferrites are frequently used to reduce magnetostriction by promoting grain growth in switching and memory devices. For application at frequencies above 50 Mzs cobalt is preferred to preclude magnetic losses[1]. Also in the control of spin wave line width (ΔH_k) cobalt is invariably doped to various ferrites for the rapid relaxation to the lattice[2]. In order to understand the effect of composition and heat treatment on the characteristics of microstructure and properties the following studies are undertaken in the present case.

1) Preparation of copper-cobalt ferrite,
2) Characterization of spinel structure by x-ray diffraction,
3) Measurement of d.c. electrical resistivity,
4) Measurement of saturation magnetization,
5) Far Infrared absorption spectroscopic studies and
6) Scanning electron microscopy of ferrites.

Chapter I deals with the historical background of ferrites along with the structure of spinel ferrites. The general properties and applications are also explained briefly. The role of various characteristic elements of microstructure on electrical and magnetic properties and orientation of the present work are discussed at length at the end of the Chapter.

Details of the preparation of $Cu_x Co_{1-x} Fe_2O_4$ ferrites by ceramic method are dealt within the II Chapter. The possible stages of the preparation techniques are presented in flow chart. Also it includes the characterization of samples by X-ray diffraction. The lattice parameter calculations using X-ray diffractograms confirm the spinel structure of ferrites. The single phase nature is represented by diffractograms. It is observed that cobalt ferrite shows cubic structure while copper and mixed copper-cobalt ferrites show tetragonal structure. There is a increase in lattice parameter with the rise in cobalt concentration. It is due to the fact that the increased cobalt concentration causes the cobalt ions to occupy B-sites replacing Fe^{3+} ions which in turn replace Cu^{2+} ions in the A-sites. This results in the increase of the total length of the cube edge.

Measurements of d.c. resistivity of the sample sintered at 800°C and 900°C for 14, 20 and 30 hrs are reported in Chapter III. D.C. conductivity is measured by conventional two probe method from room temperature to 750°C. The plots of log ρ vs $\frac{10^3}{T}$ show a bend at a particular temperature which co-relates well with the Curie temperature of that composition. Paramagnetic region is associated with higher activation energy than that of ferromagnetic region. It is due to the magnetic disorder during conduction process[3]. In general, the curve for a sample sintered at higher temperature for a longer period shifts towards lower resistance side. Curie temperature decreases with increase in sintering temperature and time. This has been attributed to the decreasing number of A.B. interactions or due to the large crystallites favouring easy transition at higher sintering temperature and time. This is the clear evidence for the decrease in resistivity and transition temperature. Due to reduction of sources of current barriers in the form of grain boundries at higher temperature, grain size increases with consequent decrease in grain boundary area. The conduction increases as a effect[4]. The reduction in activation energy at elevated sintering temperature and time is attributed to the enhancement of grain growth and reduction in porosity[5]. Electron micrographs of the samples support this view strongly.

Chapter IV has two parts. Part A deals with the hysteresis studies of ferrites. The saturation magnetization is measured by High Field Loop Tracer, supplied by M/s. Arun Electronics, Bombay. All the details of experimental set up, method of calibration along with specimen calculations are presented. It is observed that there is a marked increase in saturation magnetization of a sample sintered at higher temperature and time. This is attributed to the effect of porosity, grain growth and cation distribution. The pores and voids between the grains break the magnetization circuit and cause for decrease in magnetization at low sintering temperature and time. Also the degree of inversion decreases with low temperature and causes low magnetization.

The magnetic moment of cobalt ferrite is higher than that of copper and mixed copper-cobalt ferrites. This indicates that the internal molecular fields are higher in case of cobalt ferrite compared to other ferrites. Such behaviour has been observed in cobalt-zinc ferrites[6].

Part B of Chapter IV is devoted for far infrared absorption studies. First highest frequency γ_1 band is assigned to tetrahedral sites while the next frequency γ_2 band is assigned to octahedral sites. The spectra represent the third band γ_3 around 375 cm^{-1} whose intensity increases with cobalt concentration. It is concluded that ν_3 band is due to $Co^{2+} - O^{2-}$ octahedral complexes. In otherwords the band can be assigned to the divalent metal ion-oxygen complexes in the octahedral sites. This is in good agreement with the earlier reports in case of cobalt-zinc and Nickel-zinc ferrites[6,7].

Electron microscopy results are put in the last Chapter. The micrographs of all the samples are obtained using JEOL Scanning electron microscope at the Minerological Institute, University of Mysore, Mysore. Microstructure-property correlation and the aspects of ferrite microstructure are explained in detail. Mechanism of neck growth as a result of migration of vacancies from pore or neck to grain boundries is clearly indicated in the micrographs. Due to unequal diffusion porosity develops at the base of the neck[8]. The mechanism of grain growth combined with pore growth results in the characteristic microstructure of ferrites in which residual porosity appears at the intra-granular space. This is clearly indicated in the figures 5.1 to 5.5. Micrographs also reveal two other aspects of microstructure. One is having large grain with closed pores (Fig 5.3). Such a structure is attributed to oxygen vacancies or dopents. Another is having small and pore free crystallites (Fig 5.1 to 5.6). Usuallylarge pores appear at the grain boundries. Such a structure is attributed to cation vacancies or dopings. Similar explanations support electron microscope observations in case of Ni-Zn ferrites[9].

From the present work it is evident that porosity and grain growth are the predominent factors in deciding the conduction mechanism, activation energy and magnetization. Higher the sintering temperature and time, more will be the conductivity, magnetization and less will be the activation energy for conduction in both para and ferromagnetic regions. It is due to the increased grain size and decreased porosity at higher sintering temperature and timings. For more detail understanding, microstructural analysis of the samples is required to be carried out over a wide range of sintering temperatures.

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