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CHAPTER - VI

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### SUMMARY AND CONCLUSIONS

There has been a considerable advance in the understanding of the relationship between microstructural characteristics and physical properties of ferrites in the last few years(1). The increased knowledge of the relation between microstructure and physical properties has to a large extent been brought about by increased knowledge of the development of microstructure in ceramic processing and by new developments in ceramic technology such as use of very pure raw materials, very fine ferrite powders and hot pressing. The aspects of microstructure are very broad and as such this field of science is rapidly developing. The applications of materials have led to the development of several new materials during the last few years. The recent developments in ferrites have emerged under these considerations.

Copper-manganese ferrites have been studied in the present case. They were chosen particularly because they show electrical switching (2), lattice distortion(3), structural changes with temperature( 4). Electrical conduction (5) of these ferrites is sensitivity to most of the conditions of preparation. In order to unreveal the role of composition, heat treatment and the microstructure the fallowing studies were undertaken.

1. Preparation of copper-manganese ferrites by the ceramic technique.
2. Detection and confirmation of the spinel structure by XRD.
3. Measurement of D.C. electrical conductivity.
4. Measurement of saturation magnetization, Curie temperature and I-V characteristics, and
5. Observation of microstructure by scanning electron microscopy and its correlation with properties.

In chapter I, a short survey of magnetic materials, the ferrites is given along with their crystal structure and properties. The role of various factors in controlling their electrical properties is also briefly explained. The applications of ferrites and the orientation of the present work are at the end.

The standard ceramic method is employed to prepare the ferrite samples. A flow chart involving all the steps in preparation of these materials is given in chapter II. The actual steps followed in preparing these samples, giving a specimen calculation, are also presented. The different methods of preparation of ferrites are discussed in detail. In the same chapter, the work carried out on the crystal structure calculation in order to confirm the single phase formation in these samples is given. In the same chapter the work carried out on x ray crystal structure calculation for confirm the single phase formation, in these samples. The details of observed and

calculated 'd' values the Miller indices of the reflecting planes and the lattice parameters have been given here along with the x ray diffractograms of the samples. It is found that all the compositions of mixed ferrites crystallize in cubic structure except for the pure  $\text{CuFe}_2\text{O}_4$  which is tetragonal. The physical and x ray densities are also presented.

In chapter III, studies on D.C. resistivity measurements for samples sintered at  $800^\circ\text{C}$  and  $900^\circ\text{C}$  for 20 hours are reported. The D.C. conductivity measurements were carried out by usual two probe method in the temperature range  $28^\circ\text{C}$  to  $600^\circ\text{C}$ . The  $\log \rho$  vs. inverse absolute temperature show a change in the slope at a particular temperature which co-relates well with Curie temperature for that compositions. The slope is more in the paramagnetic region indicating that the activation energy is more in that region than in ferromagnetic region. In all the cases curve at  $900^\circ\text{C}$  lie necessarily below than the curves at  $800^\circ\text{C}$ . The fact clearly reveals that the resistivity decreases when the samples are sintered at higher temperatures. The reduction of activation energy at higher sintering temperature may be attributed to the reduction of porosity and increased grain size at higher sintering temperature. The grain size of individual crystallites influence the conduction due to increased number of grain to grain contacts(6). The observed SEM photographs are in support/<sup>of</sup> such a explanation.

In chapter IV the details of measurement of Curie temperature, magnetization and I-V characteristics are given along with the details of experimental set up used. The observed Curie temperatures which give a magnetic transformation from ferri to paramagnetic state are discussed. The compositional dependence of Curie temperatures ( $T_c$ ) is found to be almost linear. Secondly, the saturation magnetization ( $M_s$ ) values have been obtained with the help of high field loop tracer. The high field loop tracer which was used in the present case was supplied by M/S Arun Electronics, Bombay. The magnetization for the sample sintered at 900°C and 800°C for 20 hours is measured. At high sintering temperature the porosity of each sample is reduced and there by saturation magnetization is increased markedly. All the details of experimental set up, method of calibration and together with a specimen calculation are presented. At the end of this chapter the experimental set up for I-V characteristics and theory of switching behaviour have been discussed. The I-V characteristics of  $Cu_x Mn_{1-x} Fe_2 O_4$  ferrite samples ( $x = 0.6, 0.8$  and  $1.0$ ) quenched from temperature 900°C are given. The addition of  $Mn^{2+}$  in  $Cu_x Mn_{1-x} Fe_2 O_4$  system affect the switching property at room temperature, the results on increased concentration of  $Mn^{2+}$  indicate that the distribution of the variation of the switching parameter like switching field slowly become unimportant as the presence of Cu becomes less and less switching behaviour is not shown by manganese ferrite.

The increasing demand for ferrite materials with specific properties to operate in more diversified environments has brought about the need to know how the microstructure of such materials dictates their behaviour. It is therefore evident that the significance of ferrite microstructure with regard to their behaviour should be an important topic.

The chapter V deals with this area. The scanning electron micrographs of these ferrites are presented at the end of this chapter. Relevant theory for ceramic microstructure, control of grain size, porosity hot pressing and microstructure and magnetic property are given here. A true structure property relationship study in ceramic involves a deliberate change in microstructure by giving separate heat treatments and then analysing the properties in the light of developed microstructure. Though such an approach has not been followed strictly, here the observed microstructure is clear enough to show the grain structure and porosity distribution of pores, etc. The decrease in porosity is clearly observed from the micrographs for the samples sintered at higher temperatures. The recent theories confirm that the porosity of copper manganese ferrites increases with an increase of copper content in ferrite (7), same can be observed from the micrographs. The mechanism of pore growth combined with grain growth gives rise to the formation of a microstructure in which the residual porosity is only present in intergranular pores. The figs. 5.1a and 5.1b give a representative picture for copper and copper manganese ferrites.

R E F E R E N C E S

1. Stuijts A.L., Proc. Am. Ceram. Soc. Symp. Pittsburgh, National Bureau of standards miscellaneous publication No. 257 P. 73 (April, 1963).
2. Yamashiro T., Jap. J. Appl. Phys. 12, 148 (1973).
3. Ohinishi H., Teranishi T., J. Phys. Soc. Jap. 16, 35 (1961)
4. Miyadai T., Seino D., Miyashara S., Ferrites Proc. Int. Conf. Japan P. 54, (1970).
5. Naik B.N., Sinha A.P. B., Indian J. Pure Appl. Phys. Vol. 7, No. 3 P. 170-4 (March 1969).
6. Standey K.T., Oxide magnetic materials, 2nd Ed. Clarendon Press. Oxford P. 139 (1972).
7. Anrievsky A.I., Uspekhi Fiz. Nauk (USSR) Vol. 12 No. 4, 661-4 (April, 1967).