

# MICROSTRUCTURE AND MAGNETIC PROPERTIES – RELATIONSHIPS IN $\text{Co}_x\text{Mg}_{1-x}\text{Fe}_2\text{O}_4$ FERRITE SYSTEM

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## ABSTRACT:

The relationships between microstructure and magnetic properties in  $\text{Co}_x\text{Mg}_{1-x}\text{Fe}_2\text{O}_4$  ( $x=0.0, 0.2, 0.4, 0.6, 0.8$  and  $1.0$ ) ferrite system are presented. The porosity and average grain size are found to show decreasing trend as cobalt content is increased. The magnetic parameters such as saturation magnetization ( $\sigma_s$ ), coercive force ( $H_c$ ) and remanance ratio ( $\sigma_r/\sigma_s$ ) are found to show increasing trend as cobalt content is increased. The magnetic parameters such as coercive force strongly dependant on the microstructural parameters like average grain size. The observed microstructural parameters, variation in the magnetic parameters and relationship between them are explained on the basis of magnetocrystalline anisotropy and domain behavior in the  $\text{Co}_x\text{Mg}_{1-x}\text{Fe}_2\text{O}_4$  ferrite system.

**Keywords:** coercive force, grain size, porosity, saturation magnetization.

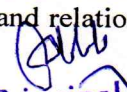
## INTRODUCTION:

Recent trends in ferrite technology have shifted towards the study of ferrites in relation to their microstructure and modifications of their properties through microstructure control (Yuijie Yong et.al.,2018). It has been well established that the properties of ferrites are strongly dependent on the intrinsic parameters such as grain size, porosity etc(Rodrigue G.A. ,1977). The relation between microstructure and magnetic parameters like coercive force has been studied by many investigators( Riqukawa,1982 and Patil S.H.et.al, 1991).

Of the numerous ferrites that have been known so far, the Mg-ferrite is unique in the sense that it exhibit interesting magnetic and crystallo- chemical properties mainly because of the different possible cationic arrangements. It does not form either completely inverse or completely normal spinel structure as shown by X-ray diffraction ( Bertan f, 1951) and neutron diffraction( Satyamurthy N.S. et.at., 1970).The Mg-ferrite has negative magnetocrystalline anisotophy and its degree of inversion can be varied by quenching temperature. The Mg-ferrite is one of the most widely used component of microwave ferrite family (Van Hook A, 1972). The proper selection of substituents and appropriate firing temperature make them suitable for wide range of applications.

The Co-ferrite, on the other hand, is promising material for application like perpendicular magnto optical information storage ( Ahrenkiei A, 1975), magneto elastic stress sensors ( Paulsen J et. al.,2015), medical applications (Amiri S et. al , 2013)etc. The Co-ferrite has not so much prone to heat treatment like the Mg-ferrite under similar conditions ( Rao V,1975).

Thus in the  $\text{Co}_x\text{Mg}_{1-x}\text{Fe}_2\text{O}_4$  ferrite system, the competition between  $\text{Mg}^{2+}$  and  $\text{Co}^{2+}$  ions for a particular site in the spinel lattice is expected to give interesting physico- chemical properties. Not much data on magnetization of  $\text{Co}_x\text{Mg}_{1-x}\text{Fe}_2\text{O}_4$  ferrites is available in the literature in relation to their microstructure. So in this research paper, the microstructure and magnetic behavior and relationships in the  $\text{Co}_x\text{Mg}_{1-x}\text{Fe}_2\text{O}_4$  ferrite system are reported.

  
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**EXPERIMENTAL:**

The ferrite system  $\text{Co}_x\text{Mg}_{1-x}\text{Fe}_2\text{O}_4$  (with  $x=0.0, 0.2, 0.4, 0.6, 0.8$  and  $1.0$ ) were synthesized by the standard ceramic technique. High purity ferric oxide ( $\text{Fe}_2\text{O}_3$ ), Cobalt oxide ( $\text{CoO}$ ) and magnesium oxide ( $\text{MgO}$ ) were taken in calculated molar proportions and mixed thoroughly in the acetone medium in agate mortar. The pre-sintering was carried out at  $800^\circ\text{C}$  for 10 hours and samples were cooled at the rate of  $80^\circ\text{C}$  per hour.

To prepare pellets, approximately 1gm of pre-sintered powder was taken and subjected to a pressure of about 5 tons per square inch for two minutes by keeping it in a die of 1 cm diameter. The pellets as well as pre-sintered powder were again sintered at  $1250^\circ\text{C}$  for 40 hours and cooled at the rate of  $80^\circ\text{C}$  per hour.

For characterization of the samples, technique of X-ray diffraction (XRD) performed on a diffractometer (Philips pw 1820) using  $\text{Fe-K}_\alpha$  radiation ( $\lambda=1.936 \text{ \AA}$ ) was used. The microstructure was studied by using SEM (Cambridge STEROSCAN 250 MKIII).

The magnetic parameters were measured by hysteresis loops taken at  $27^\circ\text{C}$  (Room temperature) with the help of high field loop tracer (TIFR make).

**RESULTS AND DISCUSSION:**

The magnetic parameters like saturation magnetization ( $\sigma_s$ ), Remanance ratio ( $\sigma_r/\sigma_s$ ), Coercive force ( $H_c$ ) etc of  $\text{Co}_x\text{Mg}_{1-x}\text{Fe}_2\text{O}_4$  ferrite system are already reported by us (Kadam S.M. et.al., 1992) and are given in following table 1.

Table 1: Magnetic parameters of  $\text{Co}_x\text{Mg}_{1-x}\text{Fe}_2\text{O}_4$  ferrite system

| x   | $\sigma_s$<br>emu/gm | $\sigma_r/\sigma_s$ | $H_c$<br>Gauss | $\mu_B$<br>Bohr magneton |
|-----|----------------------|---------------------|----------------|--------------------------|
| 0.0 | 27.9                 | 0.32                | 50             | 1.00                     |
| 0.2 | 28.3                 | 0.38                | 200            | 1.04                     |
| 0.4 | 36.8                 | 0.44                | 600            | 1.41                     |
| 0.6 | 52.4                 | 0.50                | 750            | 2.07                     |
| 0.8 | 69.9                 | 0.52                | 800            | 2.07                     |
| 1.0 | 25.5                 | 0.53                | 850            | 3.59                     |

Table 1 shows that saturation magnetization ( $\sigma_s$ ), Magneton number ( $\mu_B$ ), Remanance ratio ( $\sigma_r/\sigma_s$ ) and Coercive force ( $H_c$ ) are increased as cobalt content is increased. These increase in magnetic parameters are not contrary to the expectation because the presence of  $\text{Co}^{2+}$  ions gives rise to large induced anisotropy due to relatively high orbital contribution to the magnetic momentum. The variation of  $\sigma_s$  and  $H_c$  with cobalt content is shown in Fig. 1.

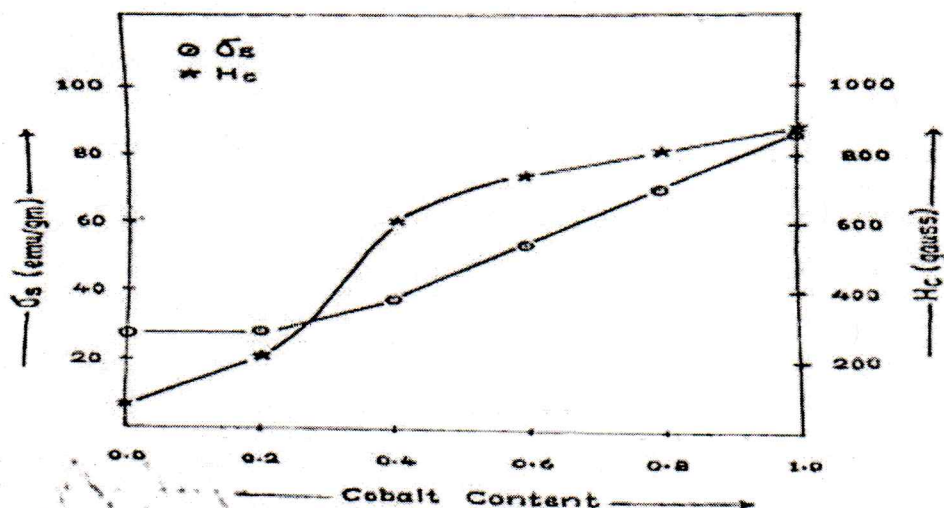


Fig.1. Variation of Saturation magnetization ( $\sigma_s$ ) and Coercive force/field ( $H_c$ ) with cobalt content of  $\text{Co}_x\text{Mg}_{1-x}\text{Fe}_2\text{O}_4$  ferrite system.



The average grain size is determined by counting the number of grain boundaries intercepted by measured lengths of random straight lines drawn on the SEM micrographs of the samples.

The densities ( $d_{\text{exp}}$ ) are measured by hydrostatic method. The X-ray densities ( $d_{\text{X-ray}}$ ) are calculated from the data of lattice parameters by the XRD analysis. The porosity (P) is calculated by the following formula (Kurtz S.K. et.al, 1980),

$$P = \frac{d_{\text{X-ray}} - d_{\text{exp}}}{d_{\text{X-ray}}} \times 100 \% \quad (1)$$

For a material having very high anisotropy constant like cobalt ferrite, the expression for critical grain size ( $D_{\text{cr}}$ ) is given by (Radhakrishnamurthy C et. al, 1979),

$$D_{\text{cr}} = \frac{188}{4\pi\sigma_s^2} \quad (2)$$

Where  $\sigma_s$  is saturation magnetization.

The microstructural parameters like average grain size (D), porosity (P%) as determined by equation (1) and critical grain size ( $D_{\text{cr}}$ ) as determined by equation (2) of  $\text{Co}_x\text{Mg}_{1-x}\text{Fe}_2\text{O}_4$  ferrite system are listed in Table 2.

Table 2: Microstructural parameters of  $\text{Co}_x\text{Mg}_{1-x}\text{Fe}_2\text{O}_4$  ferrite system.

| x   | Lattice constant<br>a<br>Å | Average grain<br>size<br>D<br>µm | Critical grain<br>size<br>( $D_{\text{cr}}$ ) | Porosity<br>P% |
|-----|----------------------------|----------------------------------|---|----------------|
| 0.0 | 8.377                      | 0.650                            | 0.0192  | 20.4           |
| 0.2 | 8.376                      | 0.397                            | 0.0186  | 19.6           |
| 0.4 | 8.373                      | 0.378                            | 0.0105  | 19.0           |
| 0.6 | 8.370                      | 0.300                            | 0.0055  | 18.8           |
| 0.8 | 8.368                      | 0.294                            | 0.0031  | 17.9           |
| 1.0 | 8.366                      | 0.292                            | 0.0021  | 13.8           |



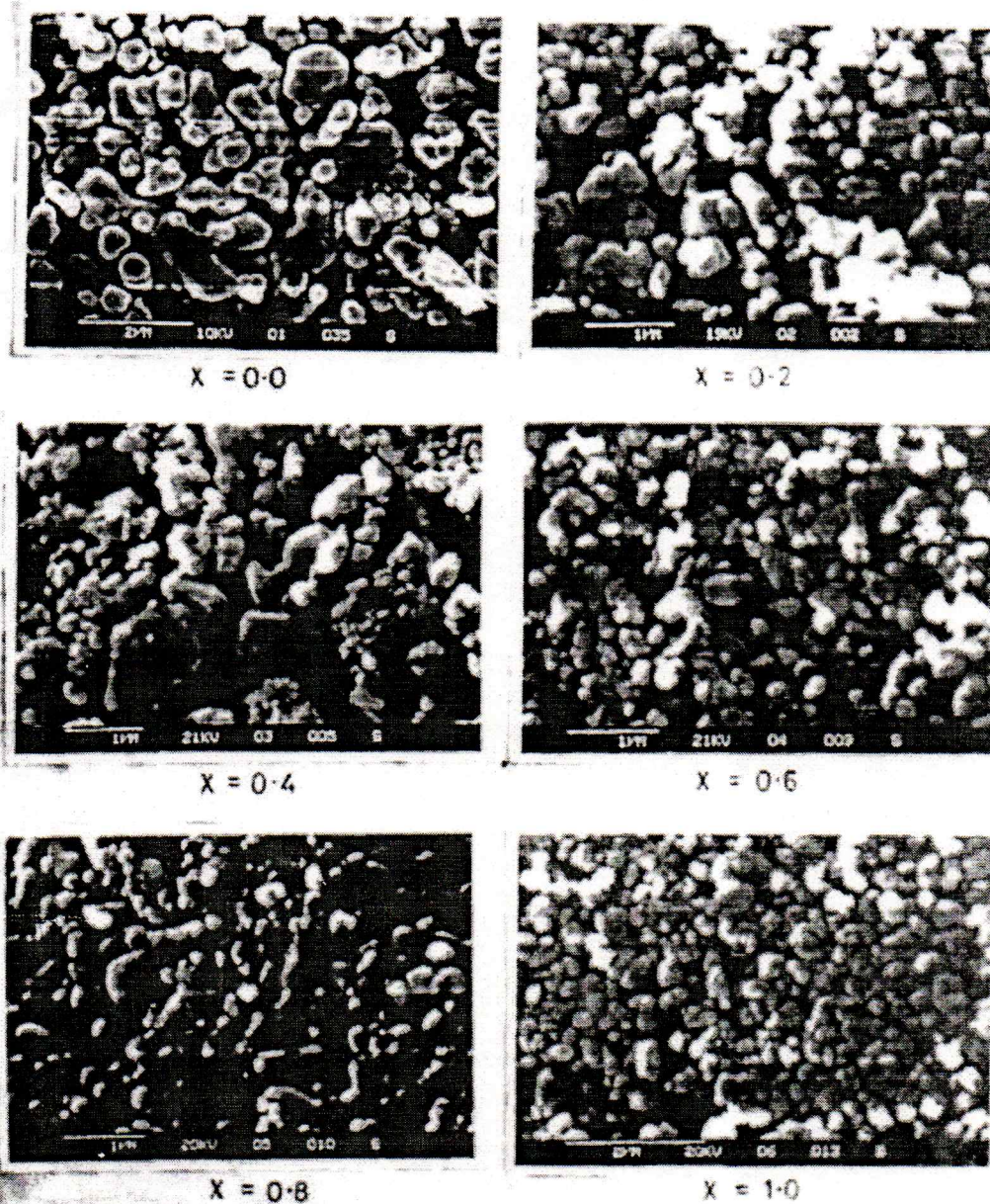


Fig.2 : SEM micrographs of  $\text{Co}_x\text{Mg}_{1-x}\text{Fe}_2\text{O}_4$  ferrite system.

Table 2 shows that porosity (P) and grain size (D) are increased as cobalt content is increased. From the micrographs ( Fig. 2), it is observed that as cobalt content is increased, pores disappear causing the small second phase solid inclusions on the boundaries of grains. Due to this, grain growth become inhibited as cobalt content is increased. Under such a conditions, decrease in grain size as well as porosity is expected and is observed in present ferrite system. The variation of porosity and grain size with cobalt content is shown in Fig.3



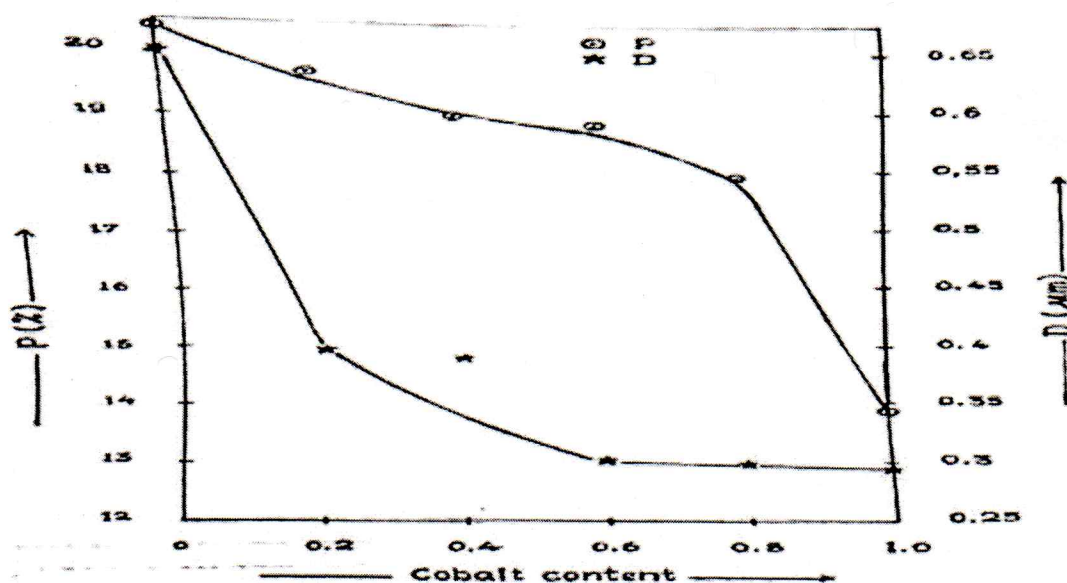


Fig.3: Variation of Porosity (P) and Grain size (D) of  $\text{Co}_x\text{Mg}_{1-x}\text{Fe}_2\text{O}_4$  ferrite system.

The cobalt ferrite is characterized by high values of magneto-crystalline anisotropy. This property is due to the triplet orbital states of  $\text{Co}^{2+}$  ( $3d^7$ ) ions at octahedral sites of crystal lattice. Compared with other 3d ions,  $\text{Co}^{2+}$  ions have a large unquenched orbital momentum and hence a large spin-orbit interaction. The diffusion of electrons between  $\text{Co}^{2+}$  and  $\text{Co}^{3+}$  ions give rise to an induced anisotropy in the domain walls. Thus stabilizing a particular structure and inhibiting domain wall motion. Hence decrease in grain size is expected as cobalt content is increased and is observed in present ferrite system.

The coercive force ( $H_c$ ) is a structure sensitive property of ferrites and depends on the factors such as anisotropy, particle size and porosity. The coercive force can be increased by increasing the anisotropy or reducing the particle size of the samples (Sato H. et.al,1990). In the  $\text{Co}_x\text{Mg}_{1-x}\text{Fe}_2\text{O}_4$  ferrite system it is observed that coercive force ( $H_c$ ) is increased as cobalt content is increased (Table 1 and Fig. 1).

This is attributed to

- (1) Since cobalt ferrite has positive magneto-crystalline anisotropy as against the negative magneto-crystalline anisotropy of magnesium ferrite and as the substitution of small amount of cobalt in the negative magneto crystalline anisotropy materials changes into the positive magneto-crystalline anisotropy, the solid solutions of  $\text{Co}_x\text{Mg}_{1-x}\text{Fe}_2\text{O}_4$  ferrites should show an increase in coercive force with increasing cobalt content and is observed.
- (2) Average grain size shows decreasing trend with increasing cobalt content as evident from microstructure data causing increase in coercive force ( $H_c$ ) with increasing cobalt content.
- (3) Addition of cobalt in magnesium ferrite aids the formation of single domain (SD) fraction in the mixed ferrite system and increases coercive force ( $H_c$ ). The Mg- ferrite has multi domain (MD) structure while the Co-ferrite has single domain (SD) structure. Thus in the solid solution of  $\text{Co}_x\text{Mg}_{1-x}\text{Fe}_2\text{O}_4$  ferrite system MD structure slowly changes to SD structure as cobalt content is increased causing increase in coercive force ( $H_c$ ) with increasing cobalt content.

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