CHAPTER - IV

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PART - A : HYSTERESIS STUDIES

INTRODUCTION

The magnetic properties of ferrites are as important as their electrical properties. The magnetic properties of ferrites are identical with those of ferromagnetic materials. The physical properties exhibited by ferrites are hysteresis, magnetic anisotropy, magnetostriction, Curie temperature and magnetic susceptibility. The properties like saturation magnetization, Curie temperature are known as intrinsic properties, while permeability, hysteresis etc. are structure sensitive properties. The saturation magnetization, coercivity and remananee are studied with the help of hysteresis. The hysteresis properties of ferrites are highly sensitive to their chemical composition, porosity, grain size, crystal structure, cation distribution and microstructure (1). These properties are also influenced by condition of sintering.

On the basis of coercive field, ferrites have been classified into two categories.

1) Soft Ferrites

Ferrites with low Hc are called "soft ferrites". These are easy to magnetize and demagnetize. Such ferrites have high electrical resistivity and low eddy current losses as reviewed by Lee and Lynch (2). They are used in the manufacture of transformer cores, motors and generators where high frequency inductance and small hysteresis losses are essential requirement (3). These are also used in microwave devices.

2) Hard Ferrites

Ferrites with high value Hc are called hard ferrites and are used as permanent magnet materials for which high remanance is desirable (4). They are used in loud speakers, telephones, electrical motors, memory devices in computers etc.

The magnetic interaction in ferrites arises from an indirect exchange mechanism in which magnetic ions are coupled through electron transfer with intermediate anions. The concept of indirect exchange has been put forward by Kramers (5) and then developed by Anderson (6) in the form of superexchange interaction. The idea of superexchange interaction is used to account for the magnetization in normal and inverse spinel ferrites. The magnetization depends upon the distance of the metal ions

to the oxygen ions through which superexchange takes place and the angle between the metal ions as shown in Fig. 1.2. The angle of 180^0 gives the greatest interaction energy and it decreases very rapidly with increasing distance(7). Ferrimagnetism is the term coined by Neel (8) to describe the magnetism in ferrites. In ferrites, magnetic ions occupy the sublattices A and B. The magnitude of magnetic moments of A and B sublattices are opposite and unequal due to the difference in the number and nature of ions on sublattices. The resultant magnetization is the difference of magnetic moments of A and B sublattices.

4.A.1 MODELS FOR MAGNETIZATION

In order to explain the spontaneous magnetization in ferromagnetics, Weiss (9) postulated the existence of small magnetized regions called domain. The magnitude of spontaneous magnetization of the specimen is determined by the vector sum of the magnetic moments of the individual domains. The Weiss field is found to be proportional to the intensity of magnetization and is given by

$$H_m = \gamma M \qquad ...4.1$$

where γ = Molecular field constant

M = Intensity of magnetization

When the external field (H) is applied the effective field becomes the vector sum of internal and external fields and is given by

$$H_c = H + H_m = H + \gamma M \qquad ....4.2$$

4.A.1.1 Neel's Model

To explain magnetic properties of ferrites, Neel has proposed a theory by assuming two sublattices formed by the location of magnetic ions at crystallographically different sites. Making use of the idea of Weiss field, he has postulated the existence of negative interaction between the ions on A and B sites; which tends to promote an antiparallel spin arrangement of the ions over A and B sites. Besides the negative A-B interactions, one must take into account of A-A and B-B interactions. These turn out to be negative as well, but are considerably weaker than the A-B interactions.

In ferrimagnetics the sublattices are crystallographically equivalent making $\gamma_{aa} \neq \gamma_{bb}$, where ν_{aa} and ν_{bb} are respective molecular field constants. Unless the sublattices are identical, $\gamma_{ab} > 0$, favouring antiparallel alignment of magnetic moments of A and B sites, giving rise to net magnetization of ferrimagnets. Therefore, the net spontaneous magnetization can be written as,

$$M_{sp} = M_{Bsp} - M_{Asp} \quad ....4.3$$

The above theory is supported by experimental studies of Neel (8), Gorter (7) and Smart (10).

4.A.2 MAGNETIZATION IN FERRITES

Magnetization is one of the most fundamental properties of ferrites. Spontaneous magnetization (M_S) occurs even in the absence of external field. For many applications of magnetic materials a high value of M_S is advantageous. In some microwave applications a material with a low value of M_S is preferred.

The saturation magnetization in ferrites is determined by the cation distribution. The normal spinel are generally nonmagnetic while inverse spinels are ferrimagnetic. For the ferrite with inverse spinel structure cation distribution is expressed as,

$$[Fe^{3+}]_A\ [M^{2+}\ Fe^{3+}]_B\ O^{2-}{}_4 \quad ....4.4$$

The Fe^{3+} ions on the A site are coupled with their spins antiparallel to those Fe^{3+} ions on B sites, so that the net moment is due to Me^{2+} ions.

$$(Fe_A \uparrow 5\ \mu_B)\ (Fe \downarrow 5\ \mu_B\ M_B) \quad ....4.5$$

In case of partially inverse ferrites where the fraction of divalent metal ions are on B sites, the arrangement of moments becomes

$$[(1\text{-}d)\ m \uparrow d\ Fe \uparrow]_A\ [(1\text{-}d)\ Fe_B \downarrow dm \downarrow]_B$$

The net magnetic moment is given by the difference between moments on A site and B site.

$$\mu_B = M\ (1\text{-}2d) - 10\ (1\text{-}d) \qquad ....4.6$$

For normal spinel d = 0 and for inverse spinels it is equal to one.

4.A.3 MAGNETIZATION PROCESS

A demagnetized ferrimagnetic material exhibits a state of zero magnetization. When sufficiently large magnetic field is applied to a ferro or ferrimagnetic substance, the magnetization changes from zero to saturation value. This was explained first by Weiss on the basis of domain theory. These domains are arranged such that the magnetization is zero. Their existence is a consequence of a process which minimizes the free energy of the system. The magnetization is constrained to be homogeneous inside the domain and its direction to very within the domain wall.

A ferro or ferrimagnetic substance become magnetized due to domain boundary displacement and domain rotation. Both processes can take place irreversibility resulting in a hysteresis loop. Domain rotation are mostly associated with intrinsic properties viz. the chemical composition of the material. Domain boundary displacement depends not only on the intrinsic properties but also on factors concerned with the sintering process such as porosity, size and shape of pores and shape of crystal.

The process of magnetization of the specimen to saturation involves its conversion to a single domain state so that the magnetization vector lies parallel to applied field. According to domain theory this can happen in two ways.

1) Domain that are oriented with respect to the applied magnetic field may grow at the expense of the other domain that are unfavourably oriented. On reversing the field, however, the domain boundaries return to their original position. This process of magnetization is called reversible boundary displacement.

2) The magnetization vector within each domain may rotate in the direction of a filed when the applied field is large. This process is called as magnetization by domain rotation.

Here spin have to be turned from an easy direction to hard direction. The rotation of domains have been observed by Berkhausen (11). He has shown that, the discontinuous variation of the magnetization at low field takes place where the field is continuously increasing. These are known as Berkhausen jumps. Magnetization by domain rotation requires large energy.

4.A.4 MAGNETIC HYSTERESIS

When a ferromagnetic substance is subjected to an external magnetic field, the domains start orienting in the direction of applied field. Its magnetization increases as a function of applied field and reaches a saturation value at a certain field at which all the domains rotate in the direction of magnetic field. On decreasing the external field to zero, magnetization lags behind the applied field. Such a behaviour is called the phenomenon of hysteresis. The residual magnetization at zero external field is called retentivity (Mr). When the direction of the field is reversed, the retentivity goes on decreasing and becomes zero at a certain value of magnetic field called coercivity (Hc) or coercive force. The variation of magnetization over one complete cycle of external magnetizing

field is called hysteresis loop (Fig. 4.1). The loss of energy per unit volume per cycle due to hysteresis is equal to the area of loop.

The slope of the tangent to BH curve at the origin gives the magnitude of initial permeability. The maximum permeability occurs near knee of the magnetization curve. Permeability plays an important role in magnetic recording as it decides the distortion factor. The coercivity and retentivity are the important parameters in selecting the material for permanent magnet.

The shape of hysteresis loop provides the valuable information about the domain state of grains inside the sample. Bean (12), has classified the magnetic particles into single domain (SD), multidomain (MD) and superparamagnetic (SP) particles on the basis of hysteresis phemenon.

4.A.5 EXPERIMENTAL TECHNIQUES

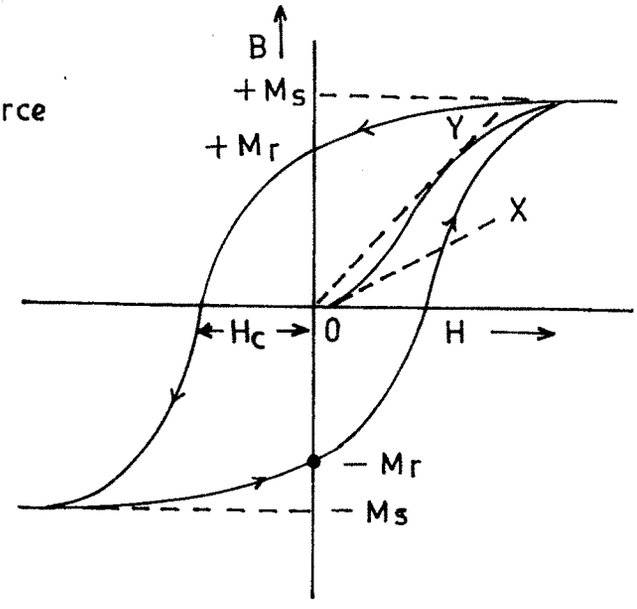
The magnetization measurements were made by using a high field hysteresis loop tracer designed by Tata Institute of Fundamental Research, Bombay (Fig. 4.2a, 4.2b). The essential parts of the hysteresis loop tracer (13) are -

1. Electromagnet
2. Pickup coil system

Ms – Saturation Magnetization

Mr – Retentivity

Hc – Coercive force



HYSTERESIS LOOP

OA – Reversible boundary displacements

AB – Irreversible boundary displacements

BC – Magnetization rotation

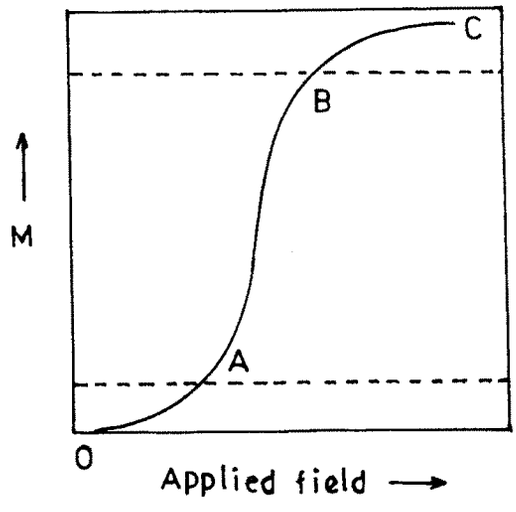


FIG. 4.1

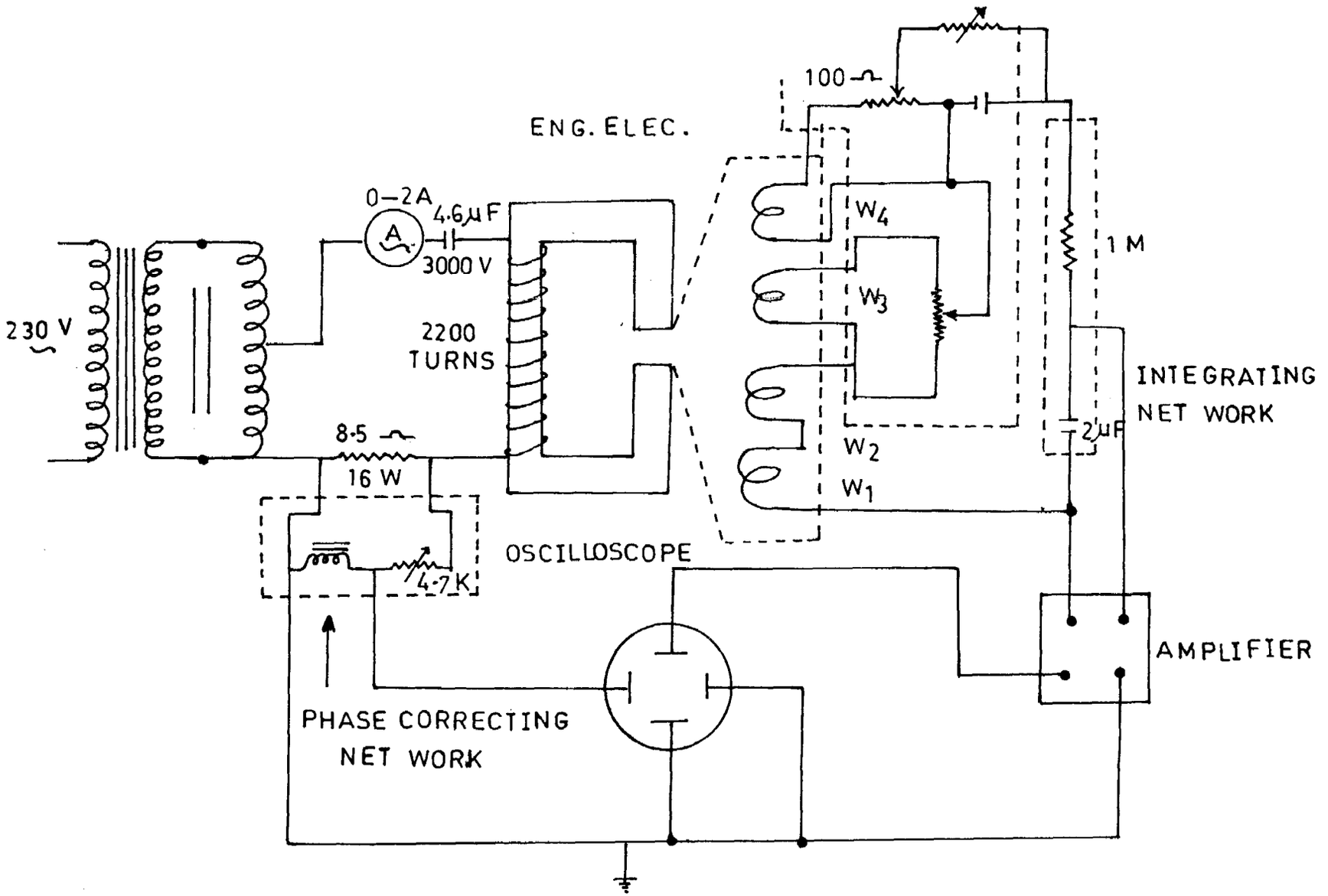


FIG.4·2(a)– SCHEMATIC DIAGRAM OF THE HYSTERESIS LOOP TRACER .

3. Balancing and integrating network

4. Preamplifier

1. Electromagnet

The two C. cores (English Electric Co-type WR/110/32/13) of laminated grain oriented silicon steel with cross section of the holes 5 x 2.5 cm^3 have been used for this purpose. From one of C cores a piece of 1.2 cm in thickness was cut to get a pole gap of 1.2 cm. The two C cores were joined together. The energizing coil for the magnet consist of 2200 turns of 20 swg super enameled copper wire (resistance 11.5 ohms) wound on a perspex former and held together on the wooden base.

2. Pick-up Coil System

A multicoil which can be introduced in the pole gap serves as a pick up coil system. It consists of different windings, one over the other, wound with 39 swg super enameled copper wire on a perspex former. A multicoil is connected to the balancing and integrating network to a amplifier and then to the y plates of the oscilloscope. The input of the x plate is taken from the emf developed across a resistance connected in series with proper inductance and variable resistance is also provided. The multiple

unit was slid into the pole gap and current in the energizing coil was increased to produce the required field. The potentiometer and variable resistance in the balancing network were adjusted to get a horizontal trace on the oscilloscope. After reducing the current to zero; the multicoil was pulled out and the ferrite pellet kept at the central gap in the multicoil spool and introduced into the pole gap. The current in the energizing coil was raised to a sufficient value till the sample saturates. The hysteresis loop produced on the oscilloscope was recorded for an accurate measurement of magnetization.

The vertical displacement on CRO was noted by placing pure Nickel sample of mass 1.0611 gm in the air gap between two pole pieces. The calibration factor (CF) was calculated by the relation

$$CF = \frac{x}{R_1} \text{ emu/div} \qquad ...4.7$$

Where, x = mass of nickel sample x magnetization of Ni per gm.

= 1.0611 gm x 53.34 emu/gm

= 56.599 emu

R_1 = Vertical reading for Ni on CRO

= 13 div.

$$CF = \frac{56.599}{13} = 4.67 \text{ emu/div.}$$

The vertical displacements for all samples were noted by introducing each pellet separately into air gap between the pole pieces of an electromagnet. The saturation magnetization was then calculated by the relation.

$$\sigma_s = \text{Vertical displacement x calibration factor}$$

$$= \text{div x CF emu.} \qquad ....4.8$$

and

$$\sigma^{-}_s = \frac{\sigma_s}{\text{mass of sample}} \quad \text{emu/gm} \qquad ...4.9$$

The saturation magnetization in emu/gm for a given porosity was calculated by the relation.

$$M_s = (1\text{-}P)\ dx\ \sigma^{-}_s \quad \text{emu/gm} \qquad ....4.10$$

where,

$$P = \text{porosity} = \frac{dx - da}{dx} \times 100 \qquad ...4.11$$

dx = x-ray density (gm/cc)

da = actual density (gm/cc)

The magnetic moment per molecular unit in Bohr magnetons was calculated by the relation.

$$\mu_B = \frac{\text{Molecular weight}}{5585} \times \sigma_s \qquad ...4.12$$

EXPERIMENTAL SET UP FOR HYSTERESIS LOOP STUDY .

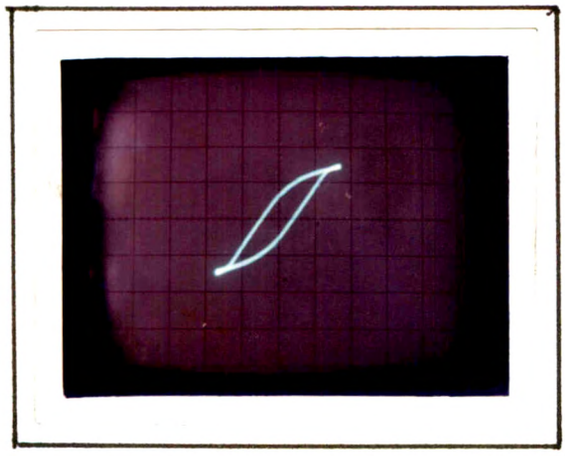
FIG. 4-2(b)

4.A.6 RESULTS AND DISCUSSION

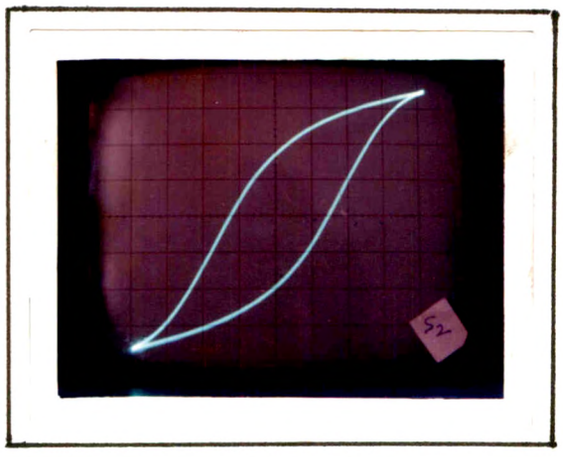
Hysteresis loops for the samples in the series $Ni_{(0.5-x)}$ $Mg_{(x-0.1)}$ $Zn_{0.5}$ $Co_{0.1}$ Fe_2O_4 are shown in Figs. 4.3 to 4.5. The data on magnetic moment, saturation magnetization and Mr/Ms is given in Table 4.1. The variation of magnetic moment μ_B and saturation magnetization Ms with x is shown in Figs. 4.6 & 4.7 respectively. From these figures it can be observed that the magnetic moment μ_B and saturation magnetization ($4\pi M_s$) go on decreasing with content of Mg(x). The value of M_r/M_s also shows a decreasing tend with content of Mg(x). The variation of saturation magnetization is attributed to the magnetic dilution of the samples by substitution of Mg^{2+} ions, having zero magnetic moment as compared to $5\mu_B$ for Fe^{3+} and $2\mu_B$ for Ni^{2+}(23).

Magnetization in ferrites results from the distribution and alignment of magnetic ions on the tetrahedral and octahedral cation sites. On the basis of site preference energy size of the cations and valency it has been concluded that Zn^{2+} ions have a preference of A site while Mg^{2+}, Ni^{2+} and Co^{2+} prefer B sites. The Fe^{3+} ions are randomly distributed over A and B sites. As the content of Mg^{2+} ions inceases in the samples the content of Ni^{2+} decreases. Secondly, Zn^{2+} which also has a zero magnetic moment and occupies the

Ni SAMPLE



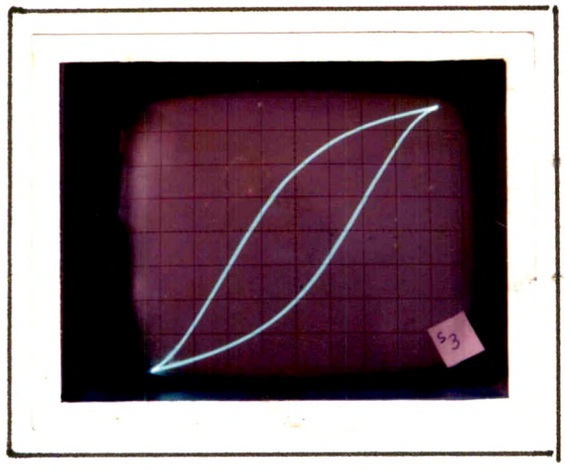
X = 0·1



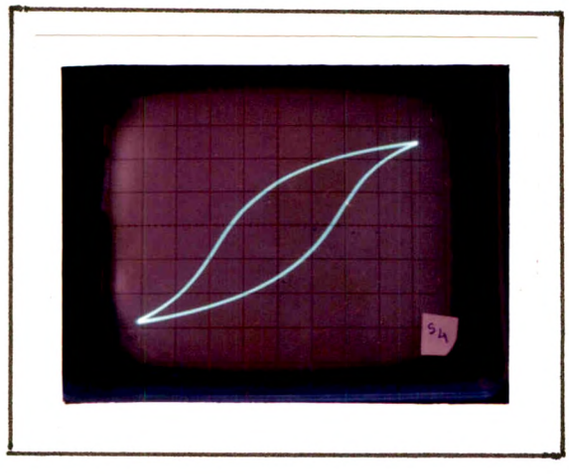
HYSTERESIS LOOPS FOR $Ni_{(0.5-x)}Mg_{(x-0.01)}Zn_{0.5}Co_{0.01}Fe_2O_4$.

FIG. 4·3

X = 0·2



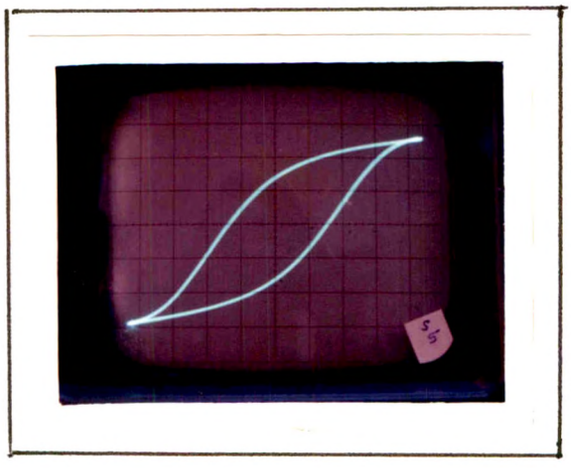
X = 0·3



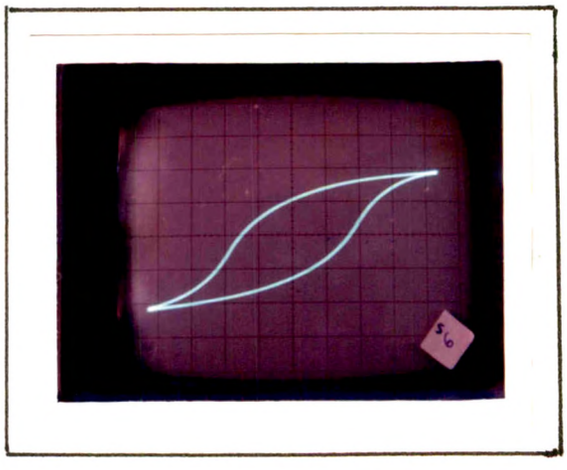
HYSTERESIS LOOPS FOR $Ni_{(0.5-x)}Mg_{(x-0.01)}Zn_{0.5}Co_{0.01}Fe_2O_4$.

FIG. 4·4

X = 0·4



X = 0·5



HYSTERESIS LOOPS FOR $Ni_{(0.5-x)}Mg_{(x-0.01)}Zn_{0.5}Co_{0.01}Fe_2O_4$.

FIG. 4·5

Table 4.1

Date on magnetic moment, saturation magnetization and M_r/M_s for $Ni_{0.5-x}$ $Mg_{x-0.01}$ $Zn_{0.5}Co_{0.01}$ Fe_2O_4 ferrites

x	Magnetic moment (μ_B)	$4\pi M_s$ Gauss/Cm^2	Mr/Ms
0.1	2.88	3726	0.45
0.2	2.56	3388	0.47
0.3	2.04	2638	0.46
0.4	1.65	2076	0.52
0.5	1.59	2053	0.55

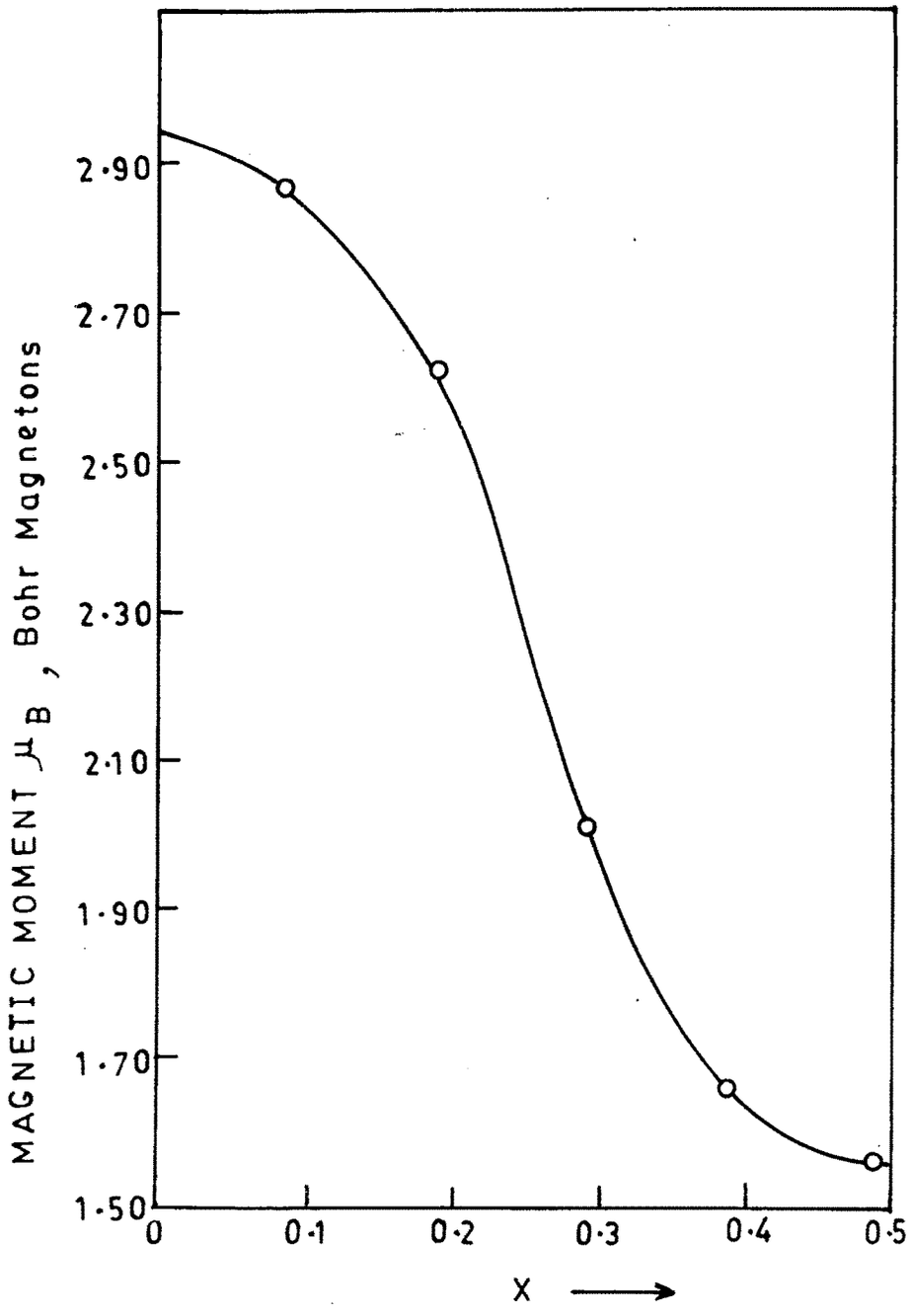


FIG. 4·6 – VARIATION OF MAGNETIC MOMENT WITH X .

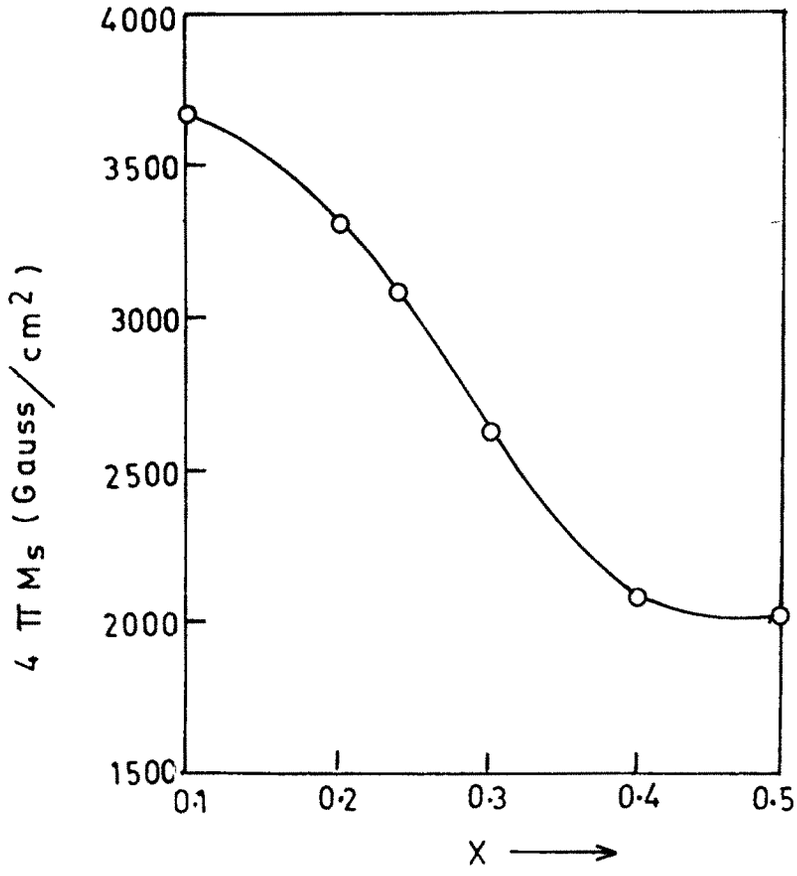


FIG.4·7 VARIATION OF SATURATION MAGNETISATION (4 π Ms) WITH X .

A site, considerably reduces the magnetic moment. The Co^{2+} ions which have large positive magnetocrystalline anisotropy greatly influences the magnetization.

The magnetization per formula unit (μ_B) of ferrites has been explained by Verwey and Heilman (14), Gorter (7), Dunitz and Orgal (15), Miller (16) on the basis of site preference energies, electronic configuration of cations, covalancy of bonds, crystal field theory and Medulung repulsion term.

Another important factor, which influences the magnetization in ferrite is the microstructure. The microstructure depends upon sintering conditions such as sintering temperature and rate of cooling. These factors determines the grain size. The larger grain have smaller porosity and higher magnetization. The presence of pores breaks the magnetic circuits among the grains which results in net reduction of magnetization

Many investigators have studied the magnetic properties of Ni ferrites and Ni containing ferrites. (17, 18, 19) Krishnan (20) has studied the magnetic properties of $NiFe_2O_4$ and has concluded that this ferrite if cooled rapidly from higher temperature, a certain amount of Ni^{2+} ions are frozen in the A site which also cause a small change in the magnetic moment.

Part - B : INITIAL PERMEABILITY

INTRODUCTION

Initial permeability (μi) is a structure sensitivity property which depends upon method of preparation, porosity and grain size. It plays an important role in the materials for various applications. High initial permeability at low frequency is the technological requirement of ferrites. Such ferrites are wildly used in low frequency inductor and transformer cores. Permeability is a structure sensitivity property which mainly depends upon the method of preparation and heat treatment. The origin of permeability has been a subject of research for many years. Basically two processes are responsible for permeability Viz.

1) rotation of domain

2) domain wall displacement

It is generally assumed that the initial permeability is caused by the reversible displacement of the domain walls, the contribution of rotation of spin inside each domain being negligibly small on account of relatively high anisotropy. In polycrystalline ferrites due to sintering a certain amount of our pores are present. In such a structure the domain walls will extent from pore so that they are

not free to move. These pores have a considerable demagnetizing effect, which leads to rather intrinsic pattern of Weiss domain. It is different to say which kind of magnetization process will give the predominant contribution to the initial permeability.

For reversible rotational processes, Chikazumi (21) has given the permeability as -

$$(\mu-1) = \text{constant } M_s^2 \, \text{Sin}^2\theta/K_1 \qquad ...4.13$$

where, θ is the angle between M_s and μ,

For reversible wall processes permeability is -

$$(\mu-1) = \text{constant } M_s^2 \, S/\propto \qquad ...4.14$$

where, S = wall surface area

$\propto$ = second derivative of wall energy with respect to wall displacement.

At low frequency domain wall motion is dominant mechanism. The second source of initial permeability viz. domain rotation is largly responsible for high frequency permeability. Both the mechanism contributing to the initial permeability domain wall motion and domain rotation are both related to μi, M_s and K_1.

The initial permeability is called simply the permeability of material and is defined as the derivative of induction B with respect to internal field H. in the magnetized state.

$$\mu = d_B / d_H, \qquad ...4.15$$

$$H \rightarrow 0, B \rightarrow 0$$

For polycrystalline material with large number of randomly oriented crystallites the permeability is scalar at least at low frequency.

$$B = H + 4\pi M$$

$$\frac{d_B}{dH} = 1 + 4\pi \frac{dM}{dH}$$

$$\mu = 1 + 4\pi\chi$$

$$\chi = (\mu-1)/4\pi \qquad ...4.16$$

is the susceptibility.

4.B.1 MODELS OF PERMEABILITY

1) Globus Model

Globus (22) has developed a model in which initial permeability μi is mainly due to the reversible motion of domain wall under a very small magnetic field and contribution of the spin rotation is rather negligible. Globus has considered a model where domain wall are pinned at the grain boundaries and they bulge under

the application of magnetic field until the critical field is reached when the wall gets unpinned. In such a model

$$\mu i - 1 = 3Ms^2D/16\,\gamma \qquad ...4.17$$

where, M_s is saturation magnetization

D = grain diameter

γ = domain wall energy per unit area since $\gamma/\sqrt{AK_1}$

$$\mu i = M_s^2\, D/|\, K_1\,| \qquad ...4.18$$

Such a model can be extended to the case where the domain walls are pinned at intergranular pores and D can be interpreted as the distance between the intergranular pores 1-e span of domain wall.

Globus and coworkers (23, 24) realized that in polycrystalline ferrites the magnetization mechanism might be different due to granular structure of ferrites. They developed a model in which domain wall bulging of 180^0, instead of domain wall displacement, is responsible for the initial permeability.

In another model they (25,26) used the existence of easy axis in each grain, the domain walls, under influence of flux forces tend to align themselves along the director circle a best a they can in order to minimize the demagnetizing field.

Globus developed a model, the main features of the model are -

1. In a grain model as spherical 180^0 Bloch wall exist in a diametrical position in the thermally demagnetized state.

2. The domain is pinned to the grain boundary and initial permeability is mainly due to bulging of domain walls when the sample is subjected to a weak applied field.

3. The domain walls act cooperatively from grain and consequently in a toroidal sample, because of existence of several easy axes in each grain.

2. Non Magnetic grain boundary Model

Various authors have proposed different models to account for the grain size dependence of initial permeability. In the grain size dependence of μi of ferrite studied particularly monodomain grain is consistent with this model and grain boundary may be hard magnetic and concluded that the Globus model is based on the presence of an intergranular wall that is one within each grains. Globus model cannot account for grain size dependence of initial

permeability when intergranular domain walls are absent. Then Globus model no longer applies.

Non magnetic grain boundary model can account for the entire grain size dependence of μi of low anisotropy, ferrite studied irrespective of domain state Jonson (27). This model can account for the entire grain size dependence of μi at low anisotropy. This model describes the grain size dependence of rotational permeability in polycrystalline materials (28). According to this model there is almost linear dependence of permeability on grain size for the fine grained polycrystals where $D << \mu i \delta$.

It is given by relation,

$$\mu_e = \mu i D/(\mu i\ \delta + D) \qquad ...4.19$$

where μ_e = effective permeability

μi = complex permeability

δ = grain boundary thickness

For large grain size $D >> \mu i \delta$, the model predicts constant rotational permeability equivalent to that in single crystal of the same material

$$\mu_e \sim \mu i_s$$

3. Two Pinning Mechanism

In this model it is assumed that a component that varies with spatial coordinates along the grain boundary is responsible for permeability (29-32). The microstress origin is due to disorientation of magnetic axis of the grains and their sources are located on the grain boundary. As a result of alternation of the region with best and worst coincidence on grain boundary. Thus domain wall is linked near grain boundary with the regions of the increasing anisotropy.

$$\Delta k = (|K| - |K_1|) \qquad ...4.20$$

The spatial pores of their sizes is Δr. The location of domain wall will be determined by the condition, that domain wall near GB contains the minimal spatial magnetistriction. This solution produces a condition of minimum strain when net magnetization is zero and offers the least resistance to the motion of domain walls.

Initial permeability depends strongly on the chemical composition and temperature. One can say that the initial permeability is governed by both intrinsic parameters like saturation magnetization, magnetocrystalline anisotropy constant etc. which depend on temperature and are specific to the given composition and by technological parameters such as microstructure, grain size, stoichiometry, internal stress and crystalline defects. All of which

depend on the sintering condition. These parameters are responsible for the diversity of thermal spectra of initial permeability for ferrite samples of a given composition. In many ferrite samples, the initial permeability increases with increase of temperature upto Curie temperature T_c. This is because the anisotropy field usually decreases with permeability much faster than M_s. In ideal homogenous ferrites, there will be only a temperature at which the crystal anisotropy is exactly zero. The crystal anisotropy constant K_1 when passes through zero, the permeability rises to a high peak at this point became initial permeability is inversely proportional to K_1.

4.B.2 DEPENDENCE OF INITIAL PERMEABILITY

1. Temperature Dependence

Permeability of spinel ferrites can often be increased by forming a solid solution of spinels possessing a negative magnetostriction with another spinel possessing a positive magnetostriction. This solution produces a condition of minimum strain when net magnetization is zero and offers the least resistance to the motion of domain walls.

The rapid increase in initial permeability just before Tc is known as Hopkhinson effect. He explained this behaviour as the

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rise of temperature diminishes the magnetic moment rapidly as critical temperature at which magnetism disappears is approached, but the facility with which molecules have their axes directed increases with temperature as critical temperature approached.

From the graph of variation of initial permeability with temperature, the slope at specific temperature can be expressed as a material parameter called the temperature factor which is define as -

$$T_f = \frac{\Delta T}{\Delta \mu i^2} \qquad ...4.21$$

where $\Delta\mu$ is difference in permeability at temperature T_1 and T_2 respectively and ΔT the change in temperature.

The temperature factor can be used to predict the variation in magnetic properties of a magnetic component. A thermal hysteresis is observed when temperature is cycled from higher temperature above T_C to lower temperature. This is explained by taking into account the domain wall pinning component between the intrinsic parameter M_s and K_1 around the transition point (33).

2. Microstructure dependence

The initial permeability is dependent on grain size, density and porosity. Porosity at grain boundaries and within the grains causes hindrance to domain wall motion and is very damaging to initial permeability (34). As the grain size increases, it is expected that the domain walls can move easily and the initial permeability will be larger. Microstructure plays a very important role because of fine grains where there is reversal of magnetization by rotation since the domain walls are energetically favourable in fine grains. Also the coercive force increases with porosity because of internal demagnetizing field where the decrease in permeability is caused by the same cause. The large grain causes reduction in initial permeability due to presence of voids that induce demagnetizing field and impede domain wall motion. Thus the grain size is a very important parameter in the permeability. A duplex structure is undesirable because it lowers the permeability. It is often due to particle impurity, which produces rapid growth locally while other impeded areas are unaffected. (24, 25). Igarashi studied the effect of grain size on initial permeability of Ni-Zn ferrites and showed that initial permeability is proportional to $(D_m)^{1/3}$ where D_m is average grain diameter. This relationship can be derived

theoretically from Globus using the assumption that wall spacing is determined in condition minimizing the total magnetostatic energy and that the domain wall is fixed by grain boundaries as pores located inside the grains.

3. Frequency dependence

At low frequencies the domain wall motion is dominant mechanism and permeability is almost independent of frequency. The domain structure is responsible for high frequency permeability. At low frequencies the applied field causes domain wall shift and this motion results in change of net magnetization. At high frequencies the domain wall inertia procludes any appreciable wall motion but the mechanism can rotate within each domain. This mechanism is same as ferrimagnetic resonance. Smit and Wijn (35) have explained the extension of the loss over a relatively broad frequency region in terms of additional effects upon the resonance condition due to magnetizing field in the domain structure.

4.B.3 EXPERIMENTAL TECHNIQUES

For the determination of permeability the samples in the form of torodis were made with the help die having inner diameter 1 cm and outer diameter of 2 cm. An insulated copper wire was wound on toroid so as to form the core. By using LCR bridge (Ablab Model

4910) the values of inductance and Q factor were noted with increasing temperature for the test frequency of 1 kHzS (Fig. 4.8). The readings were noted from room temperature to 500^0 C. The temperature of furnace was measured by cromel-alumel thermocouple. The permeability was calculated using the relation.

$$\mu i = \frac{L}{0.0046\, N^2 h \log\left({}^{OD}/_{ID}\right)} \qquad ...4.22$$

where L = inductance in μH

N = number of turns of copper wire on toroid

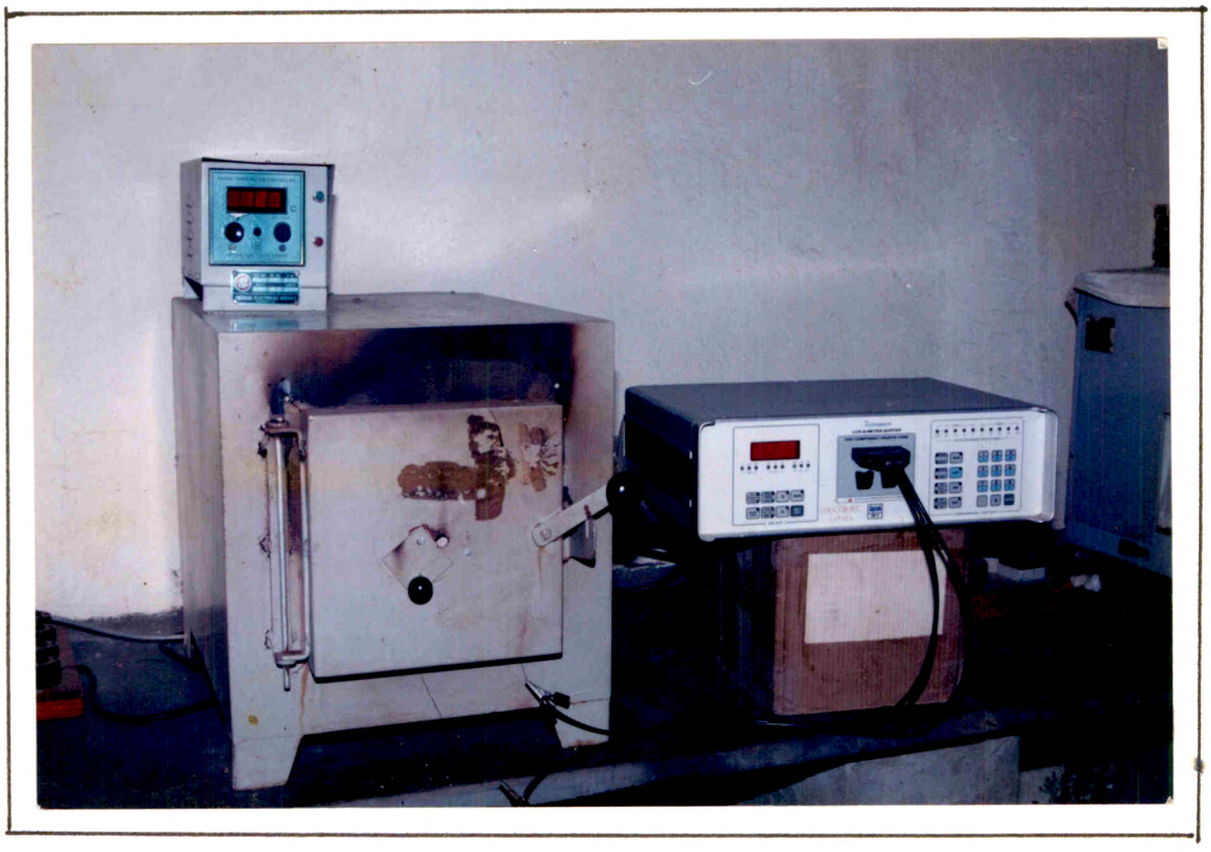
h = height of core 'in inches'

OD = Outer diameter of toroid 'in inches'

ID = Inner diameter of toroid 'in inches'

4.B.4 RESULTS AND DISCUSSION

The variation of initial permeability with temperature for different samples is shown in Fig. 4.9. From this figure it is observed that initial permeability increased gradually with temperature and then it drops sharply to zero near Curie temperature. The sharp decrease near Curie temperature suggests single phase formation of ferrites. This observation is supported by X-ray diffraction studies which do not show any impurity peaks. The Curie temperatures of the samples obtained from permeability



EXPERIMENTAL SET UP FOR PERMEABILITY STUDY .

FIG. 4.8

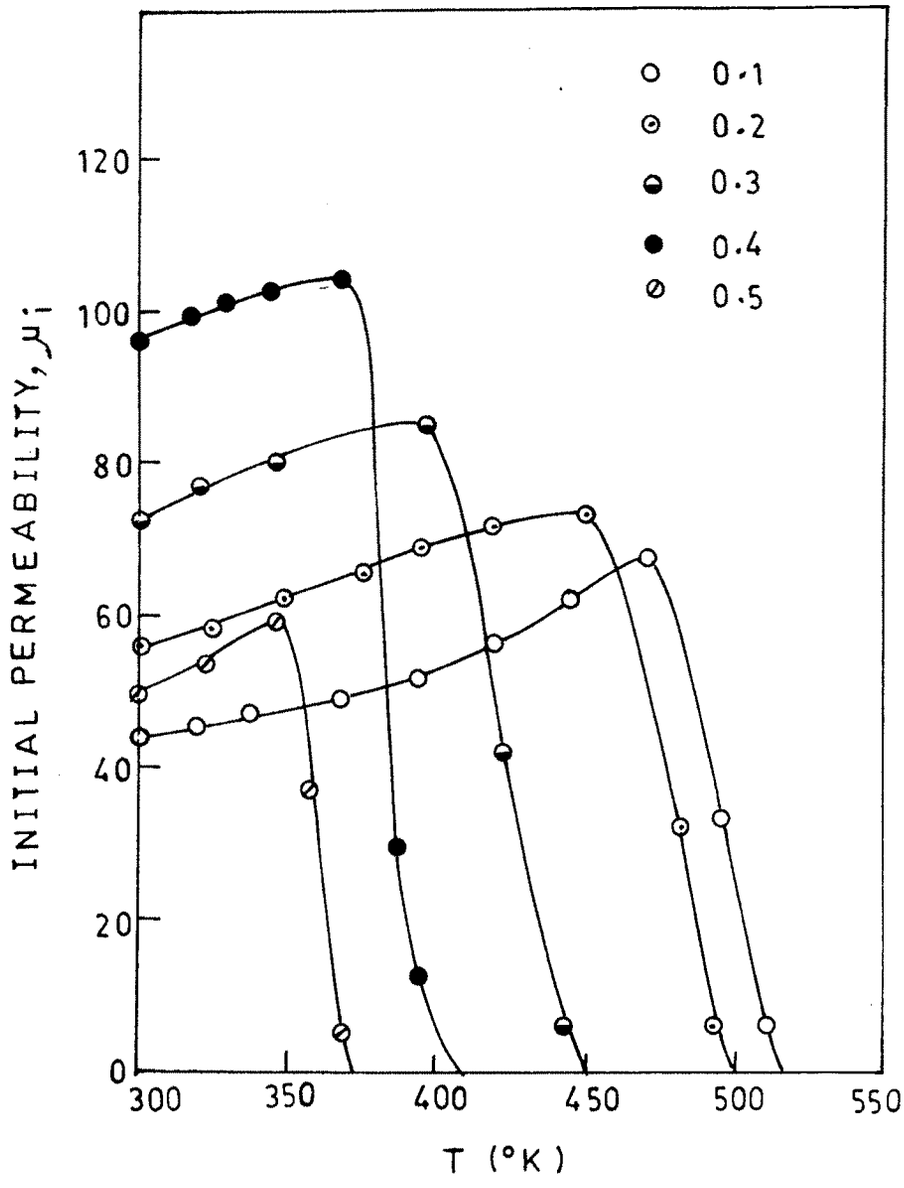


FIG.4·9 - VARIATION OF INITIAL PERMEABILITY WITH TEMPERATURE K.

data are given in Table 4.2 The Curie temperture goes on decreasing with x.

The increase of μi with temperature can be explained as follows.. The anisotropy field usually decreases with temperature much faster than the saturation magnetization Ms. The high value of μi corresponds to zero point in anisotropy caused by presence of Fe^{2+} ions in the sample. μi - T does not show a second maximum. Excess of iron leads to the formation of divalent ion in the ferrite and are homogeneous with only one temperature at which the crystal anisotropy passes through zero and the permeability then rises to peak value at this point. The share decrease of μi near Tc indicates single phase formation of the compounds.

The smaller values of initial permeability have been attributed to the polycrystalline system consisting of crystallites, grain boundaries and pores. Any disturbance caused by inclusions, pores inhomogeneities or excessively large grain boundary will decrease the permeability. These factors also seem to be responsible for lower values of permeability in the present case.

As the non magnetic character of the samples increases with substitution of Mg^{2+} in place of Ni^{2+} and the presence of Zn^{2+} in the samples, the decrease in Curie temperature is expected.

Table 4.2

Data on Curie temperatures obtained from electrical resistivity and permeability measurements

x	Curie (^{0}k) Temp. (permeability measurements)	Curie (^{0}k) Temp. (resistivity measurements)
0.1	518	516
0.2	500	500
0.3	476	485
0.4	423	410
0.5	416	400

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