
Chapter I
Introduction to ferrites

1.1 INTRODUCTION

Mixed metal oxides with iron (Fe^{3+}) oxide as one of the main components are known as ferrites. The ferrites have aroused considerable interest during the last two-three decades. Ferrites exhibit interesting physical properties which make them useful in electronic devices and computers. The academic interest in the study of ferrite systems stems from the fact that they are the most important magnetic ceramics. They have attracted the attention of physicists, technologists, chemists and ceramists alike. There are many parameters which play an important role in determining the applications of the ferrite materials in technology [1]. The systematic studies on the various aspects of behaviour of ferrite have been carried out by Snoek (2), Smit and Wijn (3), Gorter (4), Verway (5), Neel (6), Yafet and Kittel (7) and Smart (8) etc. With these studies came forth a tide of multiferrous magnetic applications of ferrites in diverse areas. Ferrites have been opened a new era in the field of magnetic materials.

1.2 HISTORICAL

The load stone [Fe_3O_4] was the first magnetic material known to man. It is also called magnetite, ~~ox~~

ferrous ferrite. It is only in the 19th century that quantitative ideas of saturation magnetization could be put forth by Du Boise (9). In 19th century, physicists began to understand why substances behave magnetically. Hilpert (10) laid the foundation stone for modern ceramic magnets at Bell Telephone Laboratories.

The formation of ferrites proceeds by solid state reaction as explained by Hedvall (11), Jander (12) and Tammann (13), Kato and Takei (14), Forestier and Vetter (15) and Gibb (16) were instrumental in the development of ferrite technology. In 1947 Verway (5) explained the electrical conductivity on the basis of exchange of electrons between trivalent and divalent iron ions. He further proved that NiFe_2O_4 has the inverse spinel structure and it is ferrimagnetic, on the other hand ZnFe_2O_4 is normal spinel and it is nonmagnetic.

Neel (6) introduced the concept of magnetic spin-sublattices to explain the basic theory of spin-spin interaction in ferrites. Further work on exchange interaction was done by Kramers (17), Anderson (18), Van Vleck (19) etc. Yafet and Kittel (7) introduced the idea of triangular spin arrangement. The experimental evidence for Neel's theory was given by Gorter (20) and Guillaud (21). The dielectric behaviour of Ni-Zn

ferrites was studied by Koops (22) who gave the formula for a.c. conductivity.

1.3 STRUCTURE OF FERRITES

Ferrites are classified in three different crystal types -

a) Spinel structure

Spinel has a general formula $A^{2+}Fe_2^{3+}O_4^{2-}$ where A^{2+} is a divalent metal ion. In this structure, there are 8 tetrahedral and 16 octahedral sites for the cations. Tetrahedral and octahedral sites are surrounded by four and six oxygen ions respectively.

b) Garnet structure

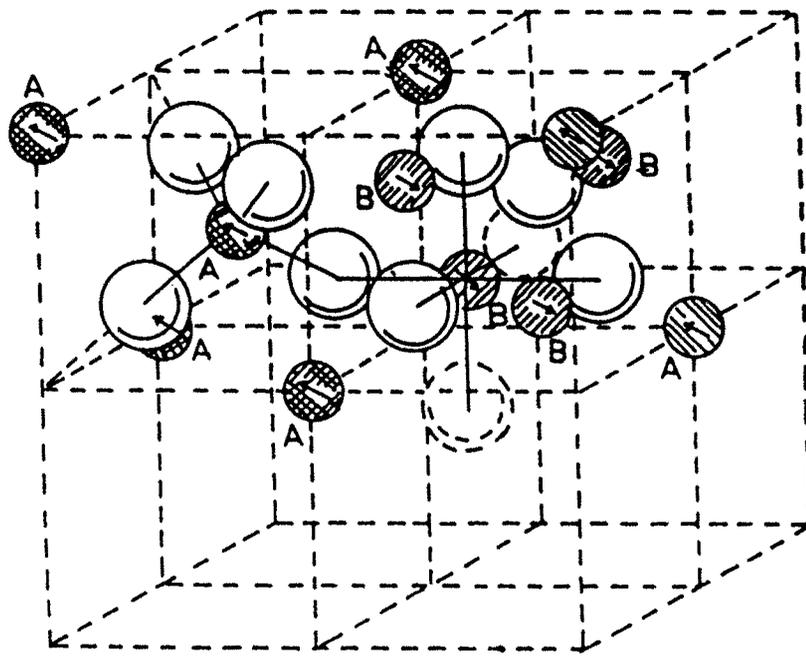
With general formula $A_3^{3+}Fe_5^{3+}O_{12}^{2-}$ where A^{3+} is a rare earth element or yttrium ion, garnets are used in microwave and bubble domain applications.

c) Hexagonal structure

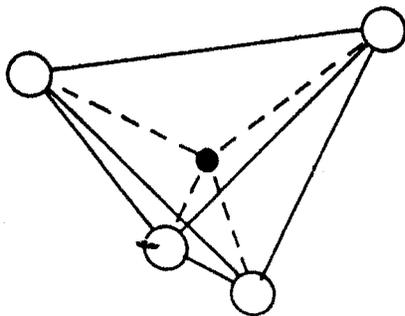
With general formula $A^{2+}Fe_{12}^{3+}O_{19}^{2-}$ where A^{2+} is Barium, these are used as permanent magnets.

1.4 SPINEL STRUCTURE

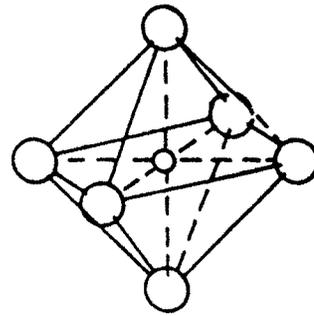
Spinel has a cubic, face centered crystal structure with space group $Fd_{3m} - O_h^h$ as shown in



THE SPINEL STRUCTURE



(a) Tetrahedral A site



(b) Octahedral B site

Fig. 1.1 — CRYSTAL STRUCTURE OF SPINEL FERRITES
SHOWING TETRAHEDRAL AND OCTAHEDRAL
CO-ORDINATION .

Figure 1.1. The structure is derived from that of the mineral spinel $MgAl_2O_4$. The unit cell contains 8 formula units of AFe_2O_3 . Hence the unit cell formula is $A_{8^{2+}}Fe_{16^{3+}}O_{32}^{2-}$. The 32 oxygen ions form two kinds of interstitial sites tetrahedral (or A) sites and octahedral (or B) sites. In all there are 96 interstitial sites per unit cell, of which 64 are tetrahedral and 32 octahedral. In the structure of ferrite only 8 tetrahedral and 16 octahedral sites are occupied and remaining ones are unoccupied.

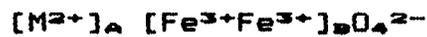
In an ideal close packed structure of oxygen anions lattice can incorporate in tetrahedral sites, the metal ions with a radii $r_{tetra} < 0.30 A^\circ$ and in octahedral sites ions with radii $r_{octa} < 0.55 A^\circ$. To accommodate cations such as Mg^{2+} , Co^{2+} etc., the lattice needs to be expanded. The tetrahedral and octahedral interstices are enlarged in the same ratio and distance between tetrahedral site $(0,0,0)$ and oxygen site is $3/8$ and $U_{ideal} = 3/8$. The incorporation of divalent metal ion in tetrahedral sites induces a larger expansion of tetrahedral sites. Therefore U_{obs} is always larger than U_{ideal} .

1.5 CLASSIFICATION OF SPINEL FERRITES

Spinel ferrites are classified on the basis of their cation distribution.

1.5.1 Normal Spinel Ferrites

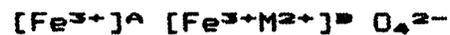
In normal spinel ferrites, all the divalent metal ion occupy A sites and all the trivalent iron ions occupy B sites. The structural formula for such a ferrites is



ex. $CdFe_2O_4$ and $ZnFe_2O_4$ have this type of structure and they are nonmagnetic.

1.5.2 Inverse Spinel Ferrites

In inverse spinel ferrites, all the divalent metal ions occupy B sites and trivalent Fe^{3+} ions are equally distributed among A and B sites. They are represented as

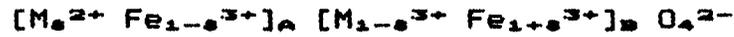


e.g. $NiFe_2O_4$ and $CoFe_2O_4$ have this type of structure and they are ferrimagnetic.

1.5.3 Random Spinel Ferrites

When the divalent (M^{2+}) and trivalent (Fe^{3+}) ions are randomly distributed over tetrahedral and octahedral sites, it is known as random spinels.

Ferrites like $MgFe_2O_4$ and $CuFe_2O_4$ have this type of structure and they are ferrimagnetic. The cation distribution among A and B sites can be represented as



where $\delta = 1$ for normal spinel

$\delta = 0$ for inverse spinel

$\delta = 1/3$ for random spinel

any value

between 0 to 1

1.6 TYPES OF FERRITES

Depending on the chemical composition, ferrites are classified into three groups.

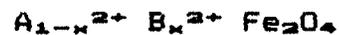
1.6.1 Simple Ferrites

When a divalent ferrous ion in Fe_3O_4 is replaced by other divalent metal ions like Cu, Co, Ni, Mn etc. the resulting ferrites are called simple ferrites.

1.6.2 Mixed Ferrites

When a ferrous ion in Fe_3O_4 is replaced by two other divalent metal ions like Cu, Co, Ni, Mn, Zn etc.

maintaining the stoichiometry of the system as it is, it constitutes a mixed ferrites. The general formula for such a ferrite system is



1.6.3 Substitutional Ferrites

When divalent metal ions or trivalent iron ions are replaced by other magnetic or non magnetic ions in the spinel structure the resulting ferrites are called substitutional ferrites



ferrite.

1.7 ELECTRICAL PROPERTIES OF FERRITES

Spinel has a wide range of resistivity from metallic or semiconducting to insulating type. Basically ferrite are semiconducting in nature with their resistivity varying from $10^{-3} \Omega \text{ cm}$ in case of Fe_3O_4 to $10^{11} \Omega \text{ cm}$ in certain magnesium and nickel ferrites at room temperature [23,24]. The physical and chemical properties of ferrites depend on the mechanism of charge transport. The conduction mechanism can be studied from the measurement of electrical

conductivity, thermoelectric power, magneto-resistance and Hall coefficient. The charge transport can vary with the composition and the method of preparation of ferrites.

1.7.1 D.C. Resistivity

The temperature dependence of electrical resistivity is given by the relation

$$\rho = \rho_0 \exp(\Delta E / kT) \quad \dots 1.1$$

where

k = Boltzmann constant

ΔE = activation energy required to cause an electron jump

T = absolute temperature.

The graph of $\log \rho$ vs $1/T$ shows a straight line behaviour and discontinuous break occurs closely at the ferrimagnetic Curie temperature [25]. The high value of ΔE is associated with high resistivity at room temperature [26]. During the course of ferrite preparation oxygen dissociation sets up above 1200 °C [27] giving rise to Fe^{2+} ions. The Fe^{2+} ions are responsible for the electron conduction. ←

The resistivity is also caused by the factors like porosity, grain size of individual crystallites which influences the conduction part due to number of grain

to grain contacts [28] and chemical inhomogeneity caused during preparation or heat treatment. In general, cation of low valence state give rise to p - type conduction and high valence state give rise to n - type conduction [2]. The presence of Fe^{2+} ion is sometime desirable [29] as it reduces magnetostriction and resistivity which is a hindrance in some cases and is unsuitable for microwave application [30].

Electrical conduction in ferrites can be associated with the presence of ions of a given element in more than one valence states distributed randomly over equivalent lattice sites. The conductivity is attributed to the occurrence of Fe^{2+} and Fe^{3+} ions on the identical lattice sites in the spinel structure. In this situation, the electron can move from trivalent iron ion to divalent iron ion within the octahedral positions. The transition does not cause a change in the energy state of the crystal.

1.8 MAGNETIC PROPERTIES OF FERRITES

Magnetic materials exhibits different kinds of magnetic ordering depending upon the spin orientation. Ferromagnetic materials, with the moments of all the ions oriented in the same direction give rise to high

values of magnetic moments, while antiferromagnetic substances, with spins aligned in an antiparallel fashion results into zero magnetic moment. In ferrimagnetic materials, the magnitude of magnetic moments oriented in either way differ, so that, there always exists the resultant uncompensated magnetic moments. The magnetic moments of the ferrimagnets are in general less than those of ferromagnets. Ferrites exhibit ferrimagnetic behaviour. The ferrites exhibit almost all the properties of ferromagnetic materials.

The magnetic properties of ferrites are intrinsic as well as structure sensitive. The saturation magnetization, anisotropy, magnetostriction and Curie temperature are intrinsic properties while permeability, resistivity and hysteresis loop are structure sensitive. The structural aspects like grain size, porosity, impurities and inclusions of non-magnetic ions, size distribution of inclusion etc affect the structure sensitive properties [31].

In 1946, Snoek developed ferrites with strong magnetic properties, high electrical resistivity and low relaxation losses. The detailed study of the magnetic behaviour of ferrites was carried out by Neel.

1.9 THEORIES OF FERRIMAGNETISM

To explain the spontaneous magnetization in ferromagnetic materials, Weiss proposed the existence of strong interactions between the neighboring atomic dipoles to keep them parallel. Each magnetic ion is influenced by its neighbor due to internal molecular field called as Weiss molecular field. This field is proportional to the intensity of magnetization and is given by

$$H_m = \gamma M \quad \dots \quad 1.2$$

where γ = molecular field constant.

M = magnetization.

In the presence of external magnetic field H, the effective field H_e is the vector sum of external and internal fields and is given by,

$$H_e = H + \gamma H_m \quad \dots \quad 1.3$$

1.9.1 Neel's Theory of Ferrimagnetism

Neel proposed the theory of ferrimagnetism by assuming two sublattices formed due to location of magnetic ions at crystallographically different sites. The computation of magnetization and susceptibility in terms of molecular field coefficients can be done with

the help of Neel's theory. The theory also characterizes A-A, B-B and A-B interactions.

Neel put forward the concept of negative interactions between the ions on A and B-sites to promote an antiparallel alignment of moments. Besides A-B interactions, A-A interactions and B-B interactions are taken into account though they are negative interactions. Thus the molecular field acting on an atom of A-site and B-site is given by,

$$\begin{aligned} \text{and} \quad H_{m_a} &= -\psi_{aa} M_a - \psi_{ab} M_b \quad \dots \quad 1.4 \\ M_{m_b} &= -\psi_{bb} M_b - \psi_{ba} M_a \end{aligned}$$

where ψ 's are appropriate molecular field constants, M_a = magnetization of A-lattice and M_b = magnetization of B-lattice. At equilibrium $\psi_{ab} = \psi_{ba}$. But in the ferrimagnetics the sublattices are crystallographically equivalent making $\psi_{aa} = \psi_{bb}$. Unless two sublattices are identical, $\psi_{ab} > 0$ favoring antiparallel alignment of M_a and M_b giving rise to net magnetization for ferrimagnets. In the presence of external field (H), total magnetic field acting on both the sides is given by

$$\begin{aligned} \text{and} \quad H_a &= H - \psi_{aa} M_a - \psi_{ab} M_b \quad \dots \quad 1.5 \\ H_b &= H - \psi_{bb} M_b - \psi_{ba} M_a \end{aligned}$$

1.9.2 Paramagnetic Region

In an assembly of N atoms per unit volume, each with angular momentum quantum number J the sublattice magnetization are represented by,

$$\begin{aligned} M_A &= (C_A/T) H_A \\ \text{and} \quad M_B &= (C_B/T) H_B \end{aligned} \quad \dots \quad 1.6$$

$$\begin{aligned} \text{where} \quad C_A &= \sum_i N_i g^2 \mu_B^2 S_i (S_i + 1) / 3K \\ C_B &= \sum_j N_j g^2 \mu_B^2 S_j (S_j + 1) / 3K \end{aligned}$$

Here N_i and N_j are number of atoms per unit volume with spin quantum numbers S_i and S_j respectively.

g = Lande's splitting factor.

μ_B = Bohr magneton.

Now the volume susceptibility can be defined as

$$X = (M_A + M_B) / H \quad \dots \quad 1.7$$

By substituting the values of M_A and M_B from equation (1.6) in equation (1.7) and simplifying further the expression for inverse susceptibility becomes,

$$1/X = T/C - 1/X_0 - \sigma / (T - \theta) \quad \dots \quad 1.8$$

$$\text{where} \quad C = C_A + C_B$$

$$1/X_0 = -1/C^2 [C_A^2 \chi_{AA} + C_B^2 \chi_{BB} + 2C_A C_B \chi_{AB}]$$

$$\begin{aligned} \sigma = & C_A C_B / C^3 [C_A^2 (\chi_{AA} - \chi_{BB})^2 \\ & + C_B^2 (\chi_{BB} - \chi_{AB})^2 - 2C_A C_B \end{aligned}$$

$$\begin{aligned} & (\gamma_{ab}^2 - \gamma_{ab}(\gamma_{aa} + \gamma_{bb}) \\ & + \gamma_{aa}\gamma_{bb}) \\ \Theta' = & -\{ C_A C_B / C \} [\gamma_{aa} + \gamma_{bb} + \gamma_{ab}] \end{aligned}$$

Equation (1.8) represent the hyperbola which cuts the temperature axis at

$$\Theta' = -C / X'_0 \quad (\text{from Figure 1.2a})$$

where " Θ " is asymptotic Curie point.

A sketch showing the variation of inverse susceptibility of a ferrimagnetic substance as a function of absolute temperature is given in Fig. 1.2a.

The anomalous shapes of spontaneous magnetization vs temperature curves for ferrimagnetic substances according to Neel's theory.

$$1/X = T/C + 1/X_0 \quad \dots \quad 1.9$$

Therefore, the expression for volume susceptibility becomes

$$X = C/(T + \Theta) \quad \dots \quad 1.10$$

where $\Theta > 0$.

The quantity $1/X$ becomes zero at where X becomes theoretically infinite or X is practically very large and substance passes from the paramagnetic to ferrimagnetic state with decrease in temperature. In other words, the substance obeys the Curie-Weiss law with asymptotic Curie point. Θ .

If the temperature is greater than T_{FN} (Neel temperature), the $1/\chi$ vs T plot is essentially linear as predicated by the molecular field theory for antiferromagnetic materials. For $H = 0$ the ferrimagnetic Neel temperature comes out to be

$$T_{FN} = -1/2 (C_A \gamma_{aa} + C_B \gamma_{bb}) + 1/2 [(C_A \gamma_{aa} - C_B \gamma_{bb})^2 + 4 C_A C_B \gamma_{ab}^2]^{1/2}$$

Ferrimagnetic materials show spontaneous magnetization in the region $0 < T < T_{FN}$. Above the Neel temperature ferrimagnetic materials turn into paramagnetic.

1.9.3 Spontaneous Magnetization

The magnetizations of A & B sites are antiparallel to each other and we know that $\gamma_{ab} > \gamma_{aa}, \gamma_{bb}$ and γ_{ab} is non zero. At low temperature the magnetizations of the sublattices of ferrimagnetic substance form the spontaneously magnetized systems. The expressions for individual spontaneous magnetizations of A & B sites are given by,

$$\begin{aligned} \text{and } M_{ASP} &= N_a g \mu_B S_a B_s(X_a) \\ M_{BSP} &= N_b g \mu_B S_b B_s(X_b) \end{aligned} \quad \dots \quad 1.11$$

where B 's are Brillouin's functions, N 's are number of atoms per unit volume of appropriate lattices

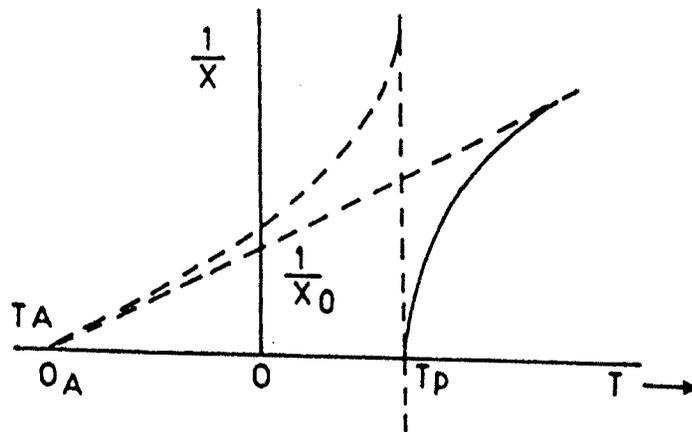


Fig. 1.4(a) - A SKETCH OF THE INVERSE SUSCEPTIBILITY OF A FERRIMAGNETIC SUBSTANCE AS A FUNCTION OF ABSOLUTE TEMPERATURE .

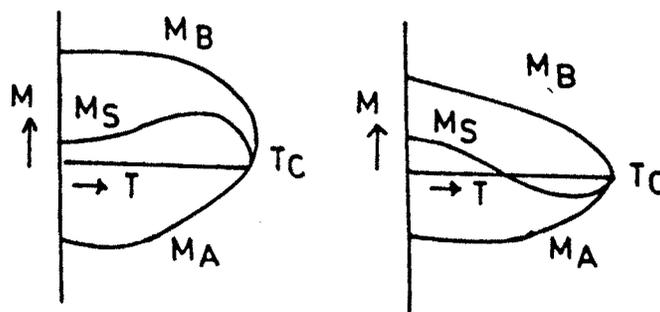


Fig. 1.4(b) - THE ANAMOLUS SHAPES OF THE SPONTANEOUS MAGNETIZATION VERSUS TEMPERATURE CURVES FOR FERRIMAGNETIC SUBSTANCES ACCORDING TO NEELS THEORY $M_S - M_B - M_A$ IS THE RESULTANT MAGNETIZATION .

and S 's are spin quantum numbers of atoms at appropriate lattices.

Therefore, the net spontaneous magnetization can be written as,

$$M_{sp} = |M_{BSP} - M_{ASP}| \text{ or } |M_{ASP} - M_{BSP}| \dots 1.12$$

The saturation magnetization at two sites is written as

$$\begin{aligned} M_{Asat} &= N_A g S_A \mu_B \\ \text{and} \\ M_{Bsat} &= N_B g S_B \mu_B \end{aligned}$$

Therefore

$$\begin{aligned} M_{ASP} / M_{Asat} &= B_e(X_A) \\ \text{and} \\ M_{BSP} / M_{Bsat} &= B_e(X_B) \end{aligned}$$

The graph of M vs T is shown in Figure 1.2b. Such curves are of different variety. They provide information about spontaneous magnetization and exchange energy at different temperatures.

1.10 YAFET - KITTEL THEORY

The Neel's theory fails to explain the reduction in magnetization with the help of antiparallel spin arrangement, as well as to explain the behaviour of mixed ferrites. Yafet and Kittel (7) proposed a new model based on triangular spin arrangement. They concluded that when a strong negative interaction

exists within the B sublattice the two equivalent B_1 and B_2 get spontaneously magnetized. The magnetizations are not exactly antiparallel, but are aligned at an angle [Figure 1.3]. This triangular arrangement within the lattice results in values of reduced magnetization of same order and magnitude as those stated by Neel's model.

The interaction energy E for this case is given by

$$E = 8N [6J_{ab} S_a S_b \cos\phi - J_{bb} S_b^2 (2 \cos\phi - 1)] \dots 1.13$$

where ϕ = Angle between A and subdivision of B

J = Exchange integrals

Interaction energy is minimum for J_{ab} negative and J_{bb} positive. If $\phi = 0$, Neel state will be obtained. If J_{bb} is also Negative, the ratio of exchange energies is given by

$$\gamma_{ex} = J_{bb} S_b / J_{ab} S_a > 3/4 \dots 1.14$$

Under this condition, Neel's state will not be minimum and magnetization of sublattice B_1 and B_2 will be inclined to sublattice A at an angle given by

$$\cos\phi = 3/4 J_{ab} S_a / J_{bb} S_b \dots 1.15$$

Yafet-Kittle stated that the Neel's model is stable for $\gamma_{ex} > 3/4$, if the total number of sublattices is restricted to six.

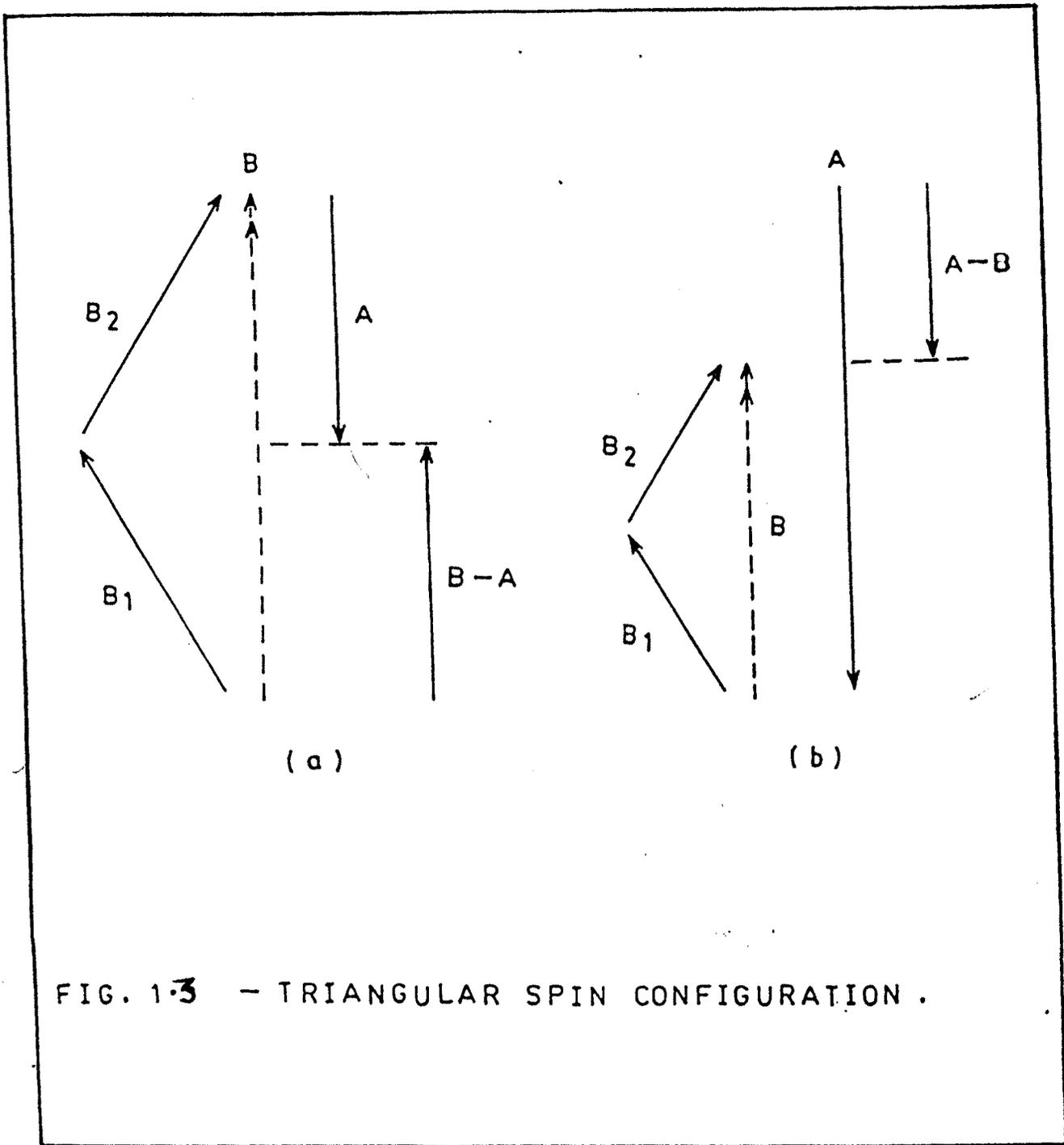


FIG. 1.3 - TRIANGULAR SPIN CONFIGURATION .

The theory of triangular spin arrangement has been further developed by Lotgering [32] and approved by many workers [33-38].

1.11 APPLICATIONS OF FERRITE

Among the magnetic ceramics, magnetic materials are most important and relevant materials from the point of view of their applications, such as home appliances, electronic products, automobiles, communication equipments and data processing devices. These materials have now become a vital part of everyday life in modern times.

The low frequency applications of soft ferrites ($H_c < 100$ Oe) include magnetic heads, inductors and transformer cores, filter cores and many other applications. For high frequency applications of soft ferrites, high resistivity and variation of R.F. permeability are important factors. High frequency applications include a large number of microwave components such as circulators, insulators, phase-shifters, YIG-tuned filters, gyrators, switches and substrates for microwave integrated circuits.

The most of families of the microwave applications are based on Mg and Ni ferrites. The microwave

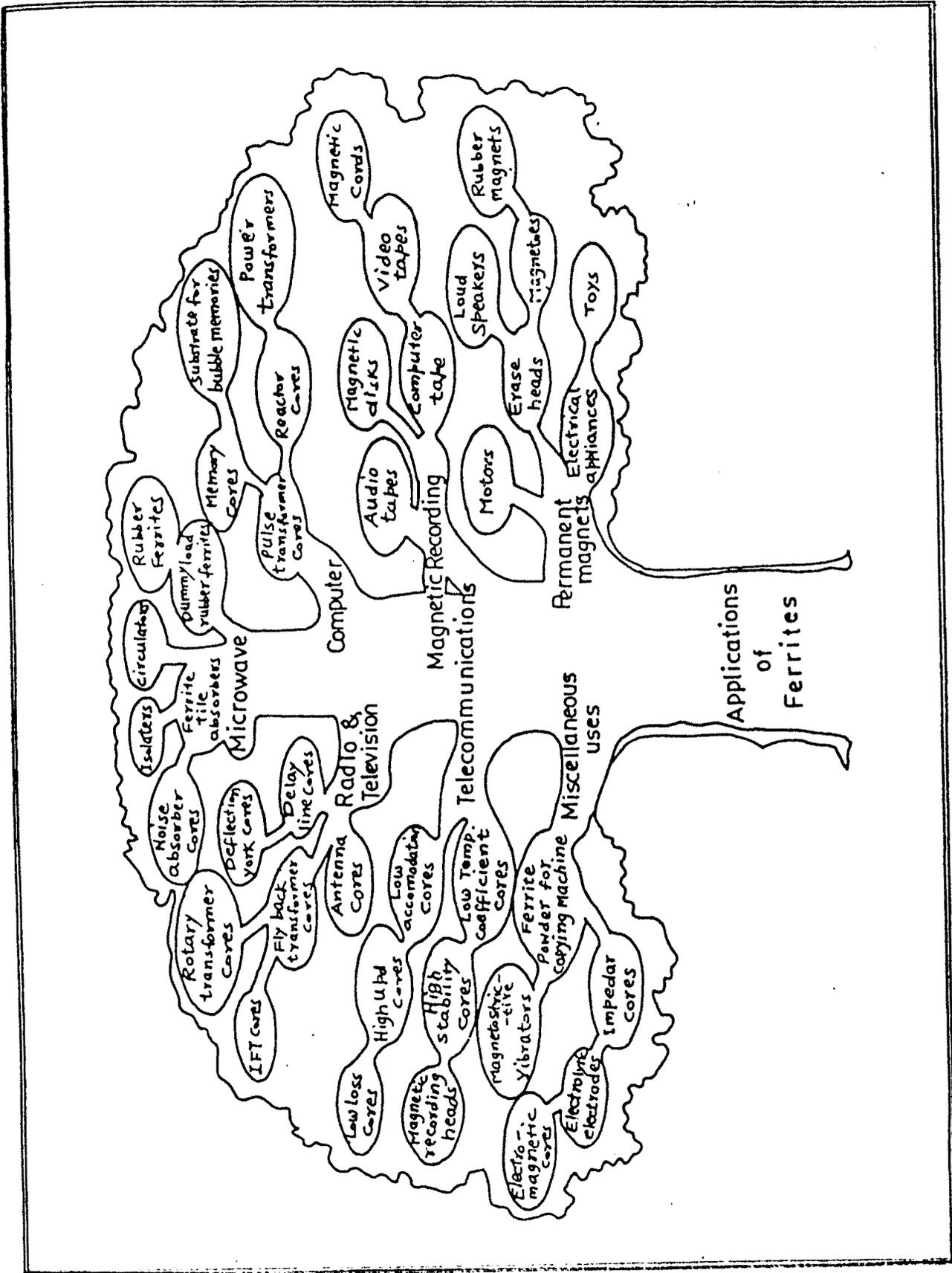


Fig. 1.4

technology is widely used in radar system for air traffic control, communication network and microwave heating. It finds wide applications in motors, generators, loudspeakers and telephones.

Today, ferrites are used in radio, television, bubble devices, audio, video and digital recording. Some ferrite exhibit a typical rectangular hysteresis loop. This triangularity of hysteresis loop and coercive force are important factors in computer and digital application. There are other applications of ferrites which are illustrated by Bahadur [39] in the form of tree shown in Figure 1.4.

1.12 ORIENTATION OF WORK

Since the last two or three decades the field of electronics has seen a tremendous expansion and breath taking developments in the fields of consumer electronics, computers, TV and radio communications information technology, robotics and automation in general. In electronic applications a ferrite becomes an essential component. However, the utility and the applicability of the ferrite depends critically on its physical properties like saturation magnetization, susceptibility and permeability, coercive field etc. Many workers have extensively studied the soft ferrites

with a view to determine their electrical and magnetic properties and to decide their suitability for important application. The soft ferrites have aroused considerable interest due to their interesting electrical, magnetic, electronic, microwave properties and electronic and computer applications.

$MgFe_2O_4$ is a partially inverse ferrite like $CuFe_2O_4$. It is also n-type semiconductor. $ZnFe_2O_4$ is also a n-type semiconductor [40]. Hence addition of zinc in Mg ferrite is expected to retain n-type conduction. The magnetization in ferrites is enhanced by addition of zinc, however the Curie temperature decreases. Hence it was proposed to carry out electrical and magnetic studies on Mg-Zn ferrites with Ti^{4+} ion substitution, to investigate the role of Ti^{4+} in influencing the bulk magnetic and electrical properties in view of the reports. Recent data reports an increase of resistivity of the ferrite with the tetravalent ion substitution (like Ti^{4+} , Sn^{4+} and Ge^{4+}) [41] Ti^{4+} substituted Mg-Zn ferrite. Suryavanshi et al [42] and Khan et al [43] have studied Sn^{4+} substituted ferrites. The trivalent ion Cr^{3+} substituted Mg-Zn ferrites system was studied earlier by Patil et al [44].

The following experimental studies were undertaken in the present investigation -

1) Preparation of Ti^{4+} substituted Mg-Zn ferrites with the general formula $Zn_xMg_{1-x+t}Ti_tFe_{2-2t}O_4$ (where $x = 0.3$, $t = 0, 0.05, 0.1, 0.2$ and $x = 0.2, 0.3, 0.4, 0.6$) by ceramic method.

2) X-ray diffraction studies to confirm homogeneity and single phase formation along with determination of lattice parameters, bond lengths, site radii and impurity phases and the infra red absorption studies to investigate the positions of absorption bands

3) D.C. resistivity measurements to understand the conduction mechanism in these compounds.

4) Magnetization studies to observe the variation in the magnetic moments, magnetization and hysteresis with composition. The a.c. susceptibility studies are conducted to observe the domain structure and to determine the Curie temperature. Initial permeability studies were undertaken to understand the variation of initial permeability with temperature.

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