
CHAPTER - I

INTRODUCTION TO FERRITES

4.

INTRODUCTION

1.1 Historical

A ferrite may be defined as a magnetizable insulator. More specifically, it is metal oxide which contains magnetic ions arranged in a manner which produce magnetization. Magnetic materials and compounds have attracted a great deal attention due to their structural and magnetic properties. These have wide spread applications and the field of ferrites has become the subject of tremendous interest to physicists and chemists. These materials continue to attract the attention of research workers because of their potential applications as electronic and magnetic ceramics. The technological importance of magnetic materials can be gauged from the vast spectrum of their applications.

The first known magnetic material was the natural spinel 'magnetite' or ferrous ferrite (Fe_3O_4). Its saturation magnetization was measured by Du-Bois [1]. A variety of synthetic ferrites were prepared by Hilpert [2] who also suggested basic formula for the ferrites to be $\text{MeO} \cdot \text{Fe}_2\text{O}_3$ where Me is a divalent metal ion. Ferrites posses the structure of spinel MgAl_2O_4 .

The preparation of ferrites by solid state reaction has been explained by Hedvall [3], Tammann [4] and Jonkar [5]. The strong foundation for improved

properties of ferrites at high frequencies was laid down by Snoek [6] by establishing the importance of accurate oxygen content and homogeneous product. Kato and Takai [7], Forestier [8], Barth and Posjank [9] have studied the ferrites and reported the structure of ferrite to be spinel type. Verway [10] reported investigations on the ordering of the divalent and trivalent iron ions in magnetite and suggested that, the existence of neighbouring Fe^{2+} and Fe^{3+} ions can give rise to higher conductivity because of the exchange of electrons between adjacent ions. He proved that, inverse spinels are ferrimagnetic and normal spinels are non-magnetic [11].

The basic theory of spin-spin interaction in ferrites was developed by Neel [12] who introduced the idea of magnetic sublattice. A more detailed examination of the basic interaction of the spin system was made by Anderson [13] and Van Vleck [14].

Yafet and Kittle [15] extended the theory of magnetic sublattice by postulating a triangular arrangement of the three sublattices, when antiferromagnetic exchange interactions between sublattices is comparable to that among the spin moments within the sublattices, formed by Fe^{3+} ions on tetrahedral and octahedral sites. The direct proof of Neel's mode of ferromagnetism came up only with neutron

diffraction studies on magnetite [16] and zinc ferrite [17].

1.2 Structure of Ferrite

Ferrites are classified in three different crystal types:

i) Spinel structure

Spinel have general formula $A^{2+}Fe^{3+}_2O_4$ where A^{2+} is 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.

ii) Garnet Structure

With general formula $A^{3+}Fe^{3+}_3O_{12}$ where A^{3+} is rare earth ion or yttrium ion. Garnets are used in microwave and bubble domain applications.

iii) Hexagonal Structure

With the general formula $A^{2+}Fe^{3+}_{12}O_{19}$ where A^{2+} is Barium, these are used as permanent magnet materials.

1.2 Spinel Structure

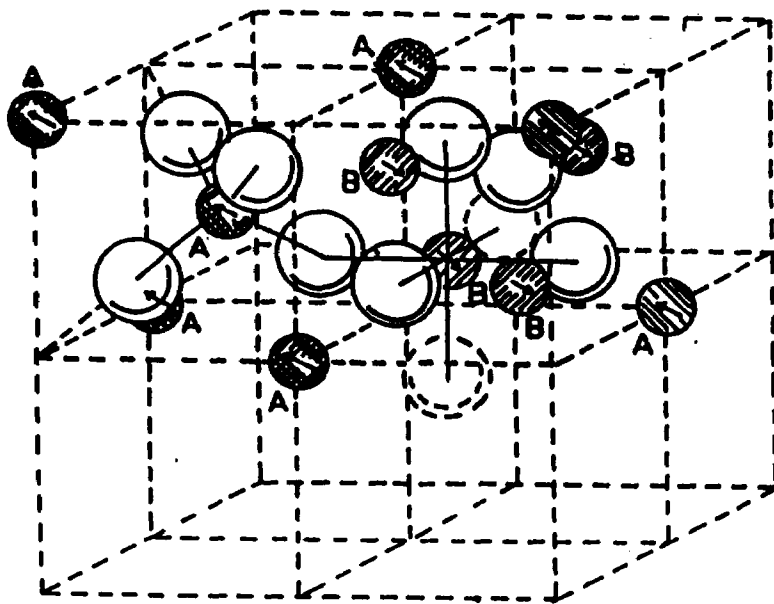
Spinel was originally the name of naturally occurring mineral, $MgAl_2O_4$. The spinel has a cubic face centered crystal structure with space group $O^7h-Fd\bar{3}m$.

It is shown in Fig.1.1. The unit cell contains 8 formula units of AFe_2O_4 . Hence unit cell formula is $A^{2+}_8Fe^{3+}_{16}O^{2-}_{32}$. The 32 oxygen ions form two kinds of interstitial sites - tetrahedral or A site (four oxygen neighbours) and octahedral or B sites (six oxygen neighbours) per unit cell. In all 64 being tetrahedral and 32 octahedral. Out of these, only 8 tetrahedral and 16 octahedral sites are occupied by cations.

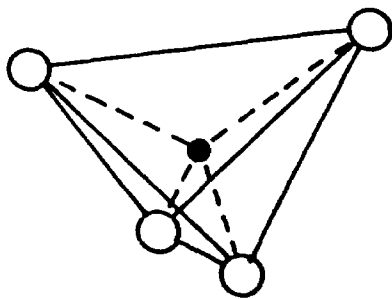
In an ideal close packed structure of oxygen anions, lattice can incorporate in tetrahedral sites the metal ions with a radius $r_{tetra} < 0.30 \text{ \AA}$ and in octahedral sites ions with radius $r_{octa} < 0.55 \text{ \AA}$. To accommodate cations such as Mg^{2+} , Co^{2+} etc. the lattice needs to be extended. The tetrahedral and octahedral sites 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 ions in tetrahedral sites induces a larger expansion of tetrahedral sites. U_{obs} is always larger than U_{ideal} .

1.4 Classification of Spinel Ferrites

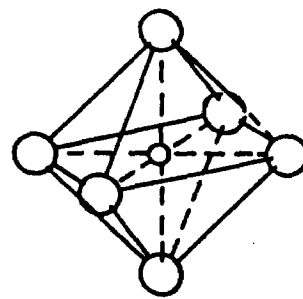
On the basis of cation distribution, spinel ferrites are classified in three groups -



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 .

1.4 1. *Normal spinel ferrites*

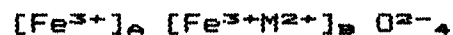
In normal spinel ferrites, all the divalent metal ion occupy A-site and all the trivalent ions occupy B-site. The structural formula for such ferrite is



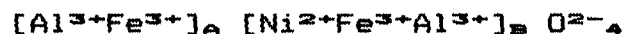
e.g. $CdFe_2O_4$ and $ZnFe_2O_4$ have this type of structure and they are non-magnetic.

1.4 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 the A and B sites. They are represented as

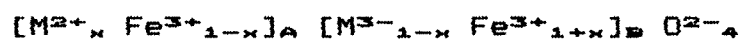


e.g. $NiFe_2O_4$ and $CoFe_2O_4$ have this type of structure and they are called ferrimagnetic. The present sample $NiAl_xFe_{2-x}O_4$ is of this type. Its cation distribution is,



3. *Random spinel ferrites*

In random spinel ferrites all the divalent metal ions and trivalent iron ions are randomly distributed on A and B-sites, depending on the sintering condition. The general formula for cation distribution is,



Where $x = 1$, for normal spinel

$x = 0$, for inverse spinel

$0 < x < 1$, for random spinel

e.g. CuFe_2O_4 and MgFe_2O_4 have this type of structure and they are ferrimagnetic.

1.5 Types of Ferrites

According to chemical composition, ferrites are classified into three groups :

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 ferrites are called simple ferrites.

2. Mixed ferrites

When a divalent ferrous ion in Fe_3O_4 is replaced by two other divalent metal like Cu, Co, Ni, Mn and Zn etc. maintaining stoichiometry of the system, it constitutes a mixed ferrite.

The general formula for this system can be written as $\text{A}^{2+}_{1-x} \text{B}^{2+}_x \text{Fe}^{3+}_2 \text{O}^{2-}_4$

3. Substitutional ferrites

When a 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.

e.g. $\text{NiFe}_{2-x}\text{Cr}_x\text{O}_4$, $\text{CuFe}_{2-x}\text{Al}_x\text{O}_4$ and $\text{NiAl}_x\text{Fe}_{2-x}\text{O}_4$ etc.

1.6 Electrical properties of ferrites

Ferrites are oxide semiconductors. Their electrical conductivity σ is low in comparison with that in magnetic metals. For the application at microwave frequencies, however, even a value of $\sigma = 10^{-4}\Omega^{-1}\text{cm}^{-1}$ may be prohibitively large because of current damping. It is therefore imperative to control the conductivity.

The conductivity becomes temperature dependent and increases with increase in temperature according to relation

$$\sigma = \sigma_0 \exp(-\Delta E/KT)$$

where, K - Boltzmann constant

ΔE - Activation energy for conduction

σ_0 - constant

and temperature dependence of electrical resistivity obey the law -

$$\rho = \rho_0 \exp(-\Delta E/KT)$$

Here, ΔE - activation energy required to cause an electron jump. K - Boltzmann constant and T - absolute temperature.

The graph of $\log \rho$ vs, $1/T$ is a straight line and breaks occur closely at the ferrimagnetic Curie temperature [18]. The value of ΔE is associated with high resistivity at room temperature [10]. During the course of ferrite preparation oxygen dissociation sets up above 1200 °C [19] give rise to Fe^{2+} ions. The Fe^{2+} ions are responsible for the electron conduction.

Electronic conduction in ferrites can be associated with the presence of ions of a given element in more than one valence state distributed randomly over crystallographically equivalent lattice points. Thus in ferrites, the conductivity is attributed to the occurrence of both Fe^{2+} and Fe^{3+} ions on the identical lattice sites in spinel structure. In this situation the electron can move from trivalent iron ion to divalent iron ion within the octahedral positions and transitions do not cause any change in energy state of the crystal. The classic example of conduction is $Fe^{2+}Fe^{3+}_2O_4$.

The resistivity will also be caused by following factors,

1. Porosity and pores filled with air.
2. The grain size of individual crystallites influences the conduction path due to number of grain to grain contacts [20].

3. Chemical inhomogeneity caused during preparation or heat treatment.

In general, the substitution of a cation of low valence state gives rise to p-type conduction while the substitution of cation of high valence state give rise to n-type conduction [6]. The presence of Fe^{2+} ion is desirable [21] as it reduces magnetostriction and resistivity which is hindrance in some cases and is unsuitable for microwave applications [22].

The prominent charge carriers in ferrites can be determined using thermoelectric power measurement. Thermoelectric properties and Hall effect are widely used in the interpretation of conduction mechanism in semiconductors. The sign of thermoemf gives the information about the type of conduction in semiconductor i.e. whether it is p-type or n-type. Another significance of thermoemf is that it enables one to calculate the values of Fermi energy, drift mobility and carrier concentration [23].

1.7 Magnetic Properties of Ferrites

The magnetic properties of ferrites like saturation magnetization, susceptibility, hysteresis loop, resistivity, a.c. conductivity and permeability are intrinsic as well as structure sensitive.

The intrinsic properties like susceptibility, saturation magnetization depend upon divalent metal ion. Whereas structure sensitive properties like hysteresis behaviour, resistivity, a.c. conductivity depends upon grain structure and porosity. The details of magnetic behaviour of ferrite were studied by Neel. Magnetic materials exhibit different kinds of magnetic ordering depending upon spin orientation. Ferrimagnetic materials with the movement of ions oriented in the same direction give rise to high values of magnetic moment while antiferromagnetic substances, with spins aligned in a antiparallel fashion, show a resultant magnetic moment of zero value.

In ferrites, the metallic ions occupy two different sites i.e. tetrahedral and octahedral and there are three kinds of magnetic interactions between metallic ions. The magnetic interaction is explained by considering the intermediate O^{2-} ions known as superexchange interaction. Hence the exchange interaction between the neighbouring atoms is indirect and takes place through non-magnetic oxygen ions. It has been established that the interaction energies are negative and hence induce the antiparallel orientation which gives lowest energy. An angle of 180° gives rise to the greatest exchange energy and energy decreases very rapidly with increasing distance between the ions.

The various possible configurations of ions pairs in spinel ferrite with favourable distances and angles for an effective magnetic interactions are given by Gorter [24]. Based on the values of the distances and angles these are prominent interactions.

The value of saturation magnetic moment of B lattice (M_B) is greater than that of A lattice (M_A) so that, resultant saturation magnetization (M_S) is written as

$$M_S = |M_B - M_A|$$

With the help of this theory Neel could explain the experimentally observed magnetic saturation data for ferrites.

Hysteresis study of ferrites gives valuable data on saturation magnetization, coercive force and remanance ratio. Ferrites are mainly classified in different categories based on their coercivity. Coercive force can range from 0.1 Oe to 3KOe. When the coercivity is greater than 1.5 KOe the ferrites are termed as 'hard ferrites'. The coercive force of soft ferrites lies in the range from 10 Oe to 1.5 KOe. Hysteresis properties are mainly dependent on crystal structure, cation distribution, chemical composition, sintering atmosphere, heat treatment and final fabrication.

A.C. susceptibility measurements are now commonly used to determine magnetic and superconducting properties of materials [25,26]. Magnetic materials contain three types of particles as multidomain (MD), single domain (SD) and superparamagnetic (SP). These domain structures change with chemical composition and thermal treatment. A.C. susceptibility and hysteresis studies are useful to explore these domain structure. The shape of thermal variation of a.c. susceptibility and hysteresis are correlated with domain structure. The Curie temperature can also be measured when the χ_{ac} is zero at certain temperature.

1.8 Theories of Ferrimagnetism

Spontaneous magnetization M_s is one of most striking property of magnetic materials. 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 following factors obviously determine the value of M_s .

- 1) Magnitude of the atomic magnetic moment and number of these ions per unit volume.

- 2) The magnetic ordering temperature (T_c).

- 3) The pattern in which the magnetic moment are ordered i.e. all parallel or partly antiparallel.

Neel has introduced the term ferrimagnetism by considering the properties of ferrites. Antiferromagnetism materials showing indirect exchange interaction. The intervening ions give rise to the two sublattices characterized by different oxygen environment for the magnetic atoms. Magnetic interactions within the sublattice give rise to a sublattice magnetization. At any temperature magnetization of two sublattices are not equal. However, they are opposite in nature and the net magnetization result as in the case of ferrimagnetic materials. Therefore, ferrites exhibit almost all the properties of ferrimagnetic materials.

1.8.a NEEL'S THEORY OF FERRIMAGNETISM

Neel assumed that the ferrimagnetic crystal lattice could be divided into two sublattices formed by A-site (tetrahedral) and B-site (octahedral) in a spinel structure. He defined the interaction within the material from the Weiss molecular field. The magnetic field acting upon an atom or ion is written as,

$$H = H_0 + H_m \quad \dots \quad 1$$

Where H_0 is external applied field and H_m is internal or molecular field. When the concept of molecular field is applied to ferrimagnetic material we have,

$$\begin{aligned} H_A &= H_{AA} + H_{AB} \\ H_B &= H_{BB} + H_{BA} \quad \dots \quad 2 \end{aligned}$$

The molecular field components may be written as,

$$\begin{aligned} H_{AA} &= \gamma_{AA}M_A; & H_{AB} &= \gamma_{AB}M_B \\ H_{BB} &= \gamma_{BB}M_B; & H_{BA} &= \gamma_{BA}M_A \end{aligned} \quad \dots \quad 3$$

Where γ 's are appropriate molecular field coefficients and M_A and M_B are magnetic moments of A and B sublattices.

a) **Paramagnetic susceptibility**

In the paramagnetic region, the magnetization is given by,

$$M = Ng J \mu_B B_J(x) \quad \dots \quad 4$$

Where N is number of free paramagnetic ions per unit volume and J is the angular momentum quantum number.

The Brillouin function $B_J(x)$ is given by

$$B_J(x) = \frac{2J+1}{2J} \coth \left[\frac{(2J+1)x}{2J} \right] - \frac{1}{2J} \coth \left(\frac{x}{2J} \right) \quad \dots \quad 5$$

Where $x = \mu_0 g J \mu_B H / KT \quad \dots \quad 6$

K = Boltzman's constant

T = absolute temperature

Writing $X = M/H_0$ and $M = M_A + M_B$

and after necessary simplification, Neel rewrites the equations of susceptibility such as,

$$\frac{1}{X} = \frac{T}{C} + \frac{1}{X_0} - \frac{\xi}{T-\theta} \quad \dots \quad 7$$

Where λ and μ are the fractions appearing on A-site and on B-site respectively such that $\lambda + \mu = 1$ equation (7) represents hyperbola Fig. 1.3a.

The asymptote to the hyperbola is given by,

$$\frac{1}{X} = \frac{T}{C} + \frac{1}{X_0} \quad \dots \quad 8$$

It cuts temperature axis at $T_A = -C/\chi_0$ and is called as asymptotic Curie point. The paramagnetic Curie point T_p is found by equating $1/\lambda$ to zero. which yields,

$$T_p = \sqrt{AB} \frac{C}{2} [\lambda\alpha + \mu\beta + \{(\lambda\alpha - \mu\beta)^2 + 4\lambda\mu\}^{1/2}] \dots 9$$

Where T_p is the Curie temperature when M_A and M_B are antiparallel.

a) Spontaneous Magnetization

The equation (9) may be written as

$$T^{\pm p} = \sqrt{ABC/2} [\lambda\alpha + \mu\beta - \{(\lambda\alpha - \mu\beta)^2 + 4\lambda\mu\}^{1/2}] \dots 10$$

Where $T^{\pm p}$ is the Curie temperature when M_A and M_B are parallel.

In ferromagnetic material, the spontaneous magnetization M_{sp} may depends on temperature and is given,

$$M_{sp} = NgJ \mu_B B_j(x) \dots 11$$

In the absence of an applied field, the Brillouin function parameter becomes

$$x = \frac{\mu_0 g J \mu_B \gamma M_{sp}}{KT} \dots 12$$

The magnetization M_A and M_B cannot be observed independently, so the net magnetization 'M' can be written as

$$M = M_A + M_B \dots 13$$

Spontaneous magnetization can be written by assuming the similar behaviour below and above Curie point.

Thus,

$$\frac{M_{A=sp}}{M_{B=st}} = BJ (\mu_0 g \mu_B J \cdot H_A / KT) \quad \dots 14$$

and $\frac{M_{A=sp}}{M_{B=st}} \approx BJ (\mu_0 g \mu_B J H_B / KT) \quad \dots 15$

The observed total spontaneous magnetization is given by,

$$M_{sp} = M_{B=sp} - M_{A=sp} \quad \dots 16$$

Since, analytical solution of the above equation is not possible, therefore, the graphical method is always used. The graphs are known as (σ, T) curves [Fig.1.3b]

Where, $\sigma = M_{sp} / M_{B=st}$ and $T = T / T_C \quad \dots 17$

The (σ, T) curves are of different types which depends on relative values of interactions and relative magnitudes of sub-lattice magnetizations.

1.8b Yafet Kittle Theory

The Neel's theory fails to explain the reduction in magnetization with antiparallel spin arrangement, as well as to explain the behaviour of mixed ferrites. Yafet and Kittle [15] proposed as new model based on triangular spin arrangements. They concluded that when a strong negative interaction exists within the B-sublattice the two equivalent B_1 and B_2 get

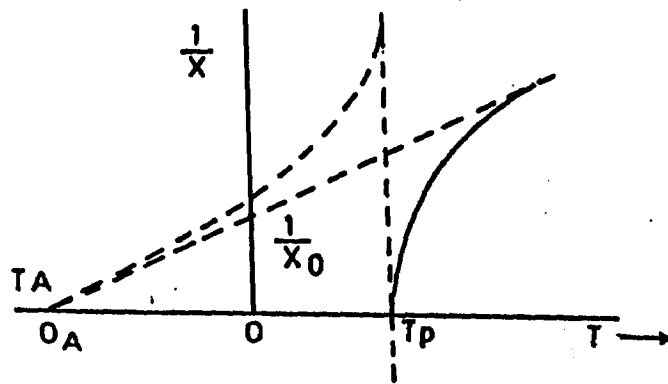


Fig. 1.3(a) - A SKETCH OF THE INVERSE SUSCEPTIBILITY OF A FERRIMAGNETIC SUBSTANCE AS A FUNCTION OF ABSOLUTE TEMPERATURE .

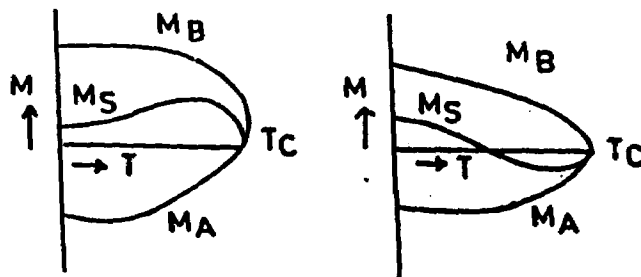


Fig. 1.3(b) - THE ANAMOLUS SHAPES OF THE SPONTANEOUS MAGNETIZATION VERSUS TEMPERATURE CURVES FOR FERRIMAGNETIC SUBSTANCES ACCORDING TO NEELS THEORY $M_S - M_B - M_A$ IS THE RESULTANT MAGNETIZATION .

spontaneously magnetized. The magnetization are not exactly antiparallel but are aligned at an angle (Fig. 1.4). 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
 $E = 8N [6 J_{ab} S_a S_b \cos\phi - J_{bb} S_b^2 (2 \cos\phi - 1)] \dots 18$
 Where ϕ - angle between A and subdivision of B

J is exchange integrals.

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

$$\gamma_{ex} = J_{bb} S_b / J_{ab} S_a > 3/4 \dots 19$$

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,

$$\cos \phi = 3/4 \cdot \frac{J_{ab} S_a}{J_{bb} S_b} \dots 20$$

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

This theory of triangular spin arrangement has been further developed by Lotgering [27] and approved by many workers [28-33].

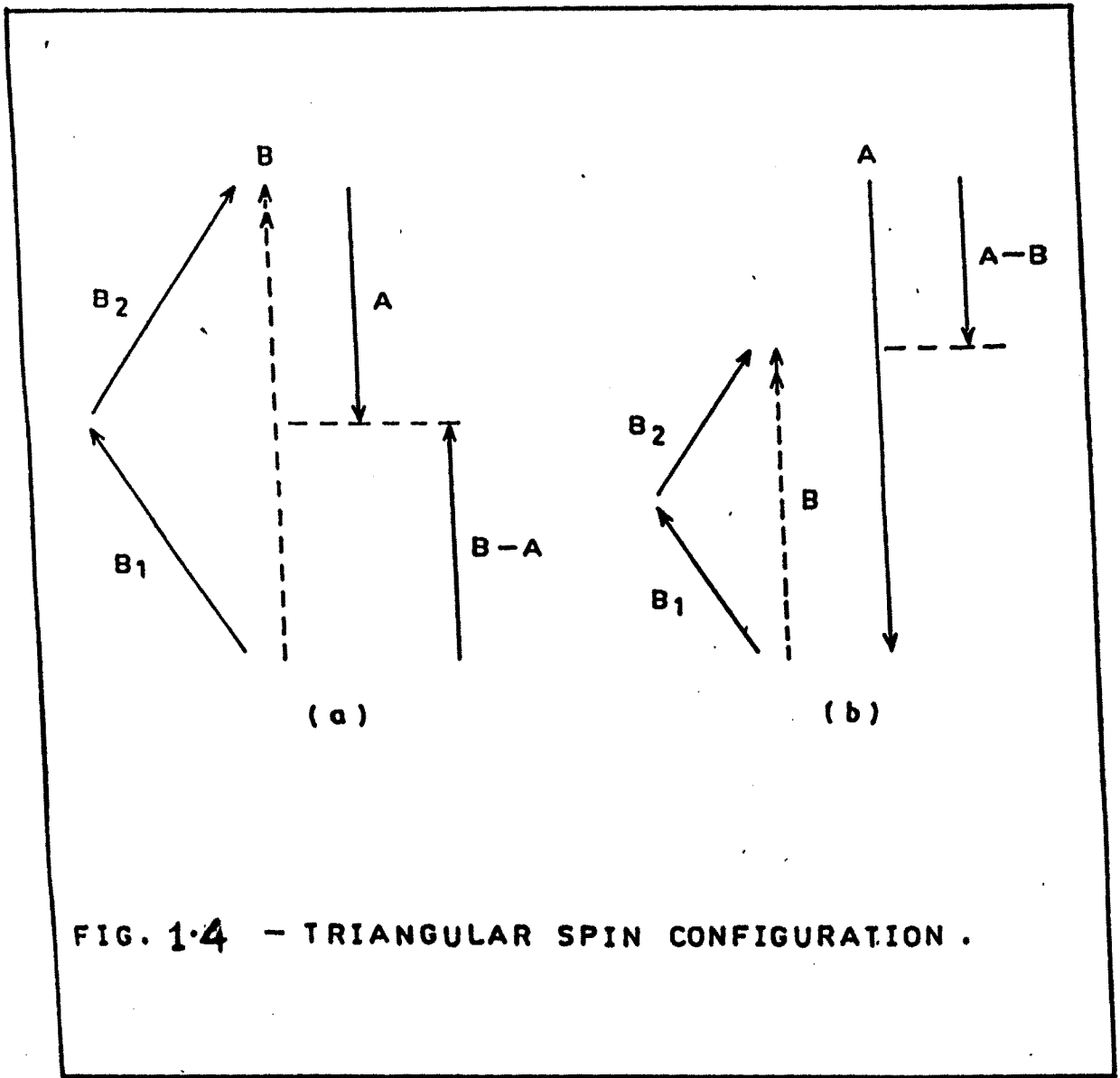


FIG. 1.4 - TRIANGULAR SPIN CONFIGURATION .

1.9 Applications of Ferrite

The magnetic materials have become a part of modern industrial society. They play a key role in power distribution, conversion between electrical and mechanical energy and in many other products such as home appliances, equipment and data processing devices etc.

The high permeability and high resistivity characteristics of ferrites are used for low and high frequency applications. The low frequency soft magnetic materials are used principally in motors, generators, magnetic recording heads, filter cores, transformer and related equipment. The high frequency applications of soft ferrites includes a large number of microwave components such as circulators, isolators and phase shifters.

In hard ferrites, a large crystalline anisotropy is a characteristics. Hence a large coercivity is an inherent property of hexa ferrites and due to suitable $(BH)_{max}$ of hexa ferrite. They find wide applications in motors, generators, loudspeakers and telephones.

The ferrites can be used in magnetic amplifiers as non-linear circuit elements. For this purpose ferrites with very small coercive force are to be used which give a sharp kink in the hysteresis curve. Storm [34]

and Attura [35] used in Mn-Zn ferrites for this purpose in order to get coercive force as low as $20 M_o$.

In large nuclear particle accelerators, the near circular path of charged particles can be obtained by the use of guiding magnetic fields. The design of such huge machine is of great importance and many hundreds of tons of commercial ferrite materials has been in the proton synchrotrons.

Today, ferrites are used in radio, television, microwave and satellite communications, bubble devices, audio, video and digital recording. There are other applications of ferrites which are given by Bahadur [36].

1.10 Orientation of the Problem

During the last forty years, the soft as well as hard ferrites have aroused considerable interest due to their interesting structural, electrical and magnetic properties along with applications in electronic, microwave and computer industry. In order to correlate the physical properties of ferrites and their applications in modern electronic devices many workers carried out tremendous work on this subject. The chief goal of modern day ferrite research is to study the relationship between structure of ferrite and their properties with the advent of developments in

microscopical devices such as scanning electron microscopy, transmission electron microscopy etc. It has been possible now a days to correlate the microstructure with characteristics properties of ferrites.

The substituted Ni ferrites find a wide a range of applications at radio and microwave frequencies where electrical and magnetic losses are required to be minimum [37,38,39]. The small substitution of Sn^{4+} ions in Ni ferrite has been found to increases the resistivity, decreases dielectric constant and eddy current losses [40]. The addition of Al^{3+} , on the other hand, increases resistivity thereby lowering the dielectric losses and decreases the saturation magnetization. These are the properties suitable for manufacturing of μ -wave devices operating at L, S and C bands [41]. Besides, Al addition inhibits grain growth and improve the mechanical strength of ferrites [42]. The work is aimed at the detailed studies on the electrical transport properties of Ni-Al ferrites. The effect of Al^{3+} addition on the magnetization behaviour of the ferrite will also be studied.

The samples with general formula $\text{NiAl}_x\text{Fe}_{2-x}\text{O}_4$ where $x = 0.0, 0.2, 0.4, 0.6, 0.8$ and 1.0 were prepared by the standard ceramic technique using pure oxide

constituents. The following steps were involved in the problem.

1) X-ray diffraction studies to confirm homogeneous and single phase formation, along with determination of lattice constant, bond lengths and site radii.

2) Infrared absorption studies to investigate the intrinsic vibration of cations at different sites.

3) The measurement of d.c. electrical conductivity and thermoemf properties to propose the mechanism of conduction and type of charge carriers.

4) Hysteresis and a.c. susceptibility studies to get the information regarding magnetic behaviour and domain structure.

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