

**CHAPTER I**  
**AN INTRODUCTION TO FERRITES**

## CHAPTER - I

### AN INTRODUCTION TO FERRITES

#### 1.1 THE PAST, PRESENT AND FUTURE OF FERRITE

Magnetic materials have played a prominent role in the development of modern technology. Ferrites, ceramic ferromagnetic materials, have been considered as highly important electronic materials for more than half a century.

Magnetoceramics, which are mainly composed of ferric oxide ( $\alpha$ - $\text{Fe}_2\text{O}_3$ ) and certain divalent metal oxides, are called "Ferrites". Hilpert<sup>1</sup> was first in preparing  $\text{Fe}_3\text{O}_4$ . Magnetite,  $\text{Fe}_3\text{O}_4$  a natural mineral, is a genuine ferrite and it is said that ancient people had recognized its magnetism and that it was used as a mariner's compass in China more than two thousand years ago. However, the first attempt to prepare ferrites was not made until the beginning of this century. Actual commercialization of ferrites started about 60 years ago. However, the commercial ferrites did not attract much attention because of the fact that their magnetic properties were considerably inferior to those of ferromagnetic alloys. Around the same time, physicists and electronics engineers worldwide became greatly interested in the unique magnetism and the expanded high frequency applications of ferrites.

Recently researchers in chemistry, ceramics and metallurgy have been engaged in the development of new ferrites, enhancement of existing ferrite properties and improvement of ferrite manufacturing processes. The ferrite production has increased enormously in the recent years.

## **1.2 FERRITE INDUSTRY IN INDIA**

Present Indian ferrite industry has come a long way. Commercial production of ferrites started in sixties.<sup>2</sup> The production of ferrites was about 7000 MT in 1990 and about 15000 MT in 1995. Indian ferrite industry is now poised for big jump and getting ready to compete in the world market. The growth pattern of ferrite production for soft and hard categories for the last 10 years is shown in Fig. 1.1.

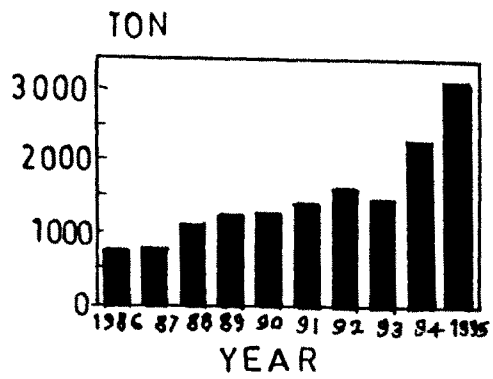
## **1.3 HISTORICAL DEVELOPMENTS**

The history of Asia Minor, another cradle of civilization, claims that the loadstone was discovered as a natural magnet in magnesïa more than 3500 years ago. The stone was called magnes lapis, which means magnesian stone. Magnet and magnetism were derived from the word magnesian.

Many reports are available on the historical development of the science and technology of ferrites. The first systematic study on

### PRODUCTION GROWTH

#### SOFT FERRITE



#### HARD FERRITE

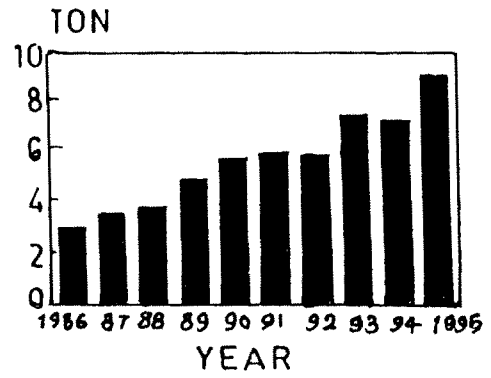


Fig. 1.1 GROWTH PATTERN OF INDIAN FERRITE INDUSTRY .

the relationship between chemical composition and magnetic properties of ferrites was reported by Hilpert in 1909. Hilpert successfully prepared copper, cobalt, magnesium and zinc ferrites. Forestier, in 1928, prepared ferrites by basic precipitation from chloride solutions and subsequent heat treatment. Several Japanese investigators took active part in ferrite preparation in the early thirties. Neel<sup>3</sup> was the first to explain the basic theory of spin-spin interaction. Anderson<sup>4</sup> and Van Vleck<sup>5</sup> developed the superexchange theory for the extensive study of basic interactions. Yafet and Kittel<sup>6</sup> introduced the triangular spin arrangements to extend superexchange theory. Gilleo<sup>7</sup> proposed the method of determining cation distribution from Curie temperature.

In 1932, Japanese chemists, Kato and Takei<sup>8</sup> discovered that solid solutions of magnetite and cobalt ferrite were strongly magnetized at  $\cong 300^{\circ}\text{C}$  and that the solid solution had practical applications. The early achievements in ferrites are concentrated in the period 1935-1970.

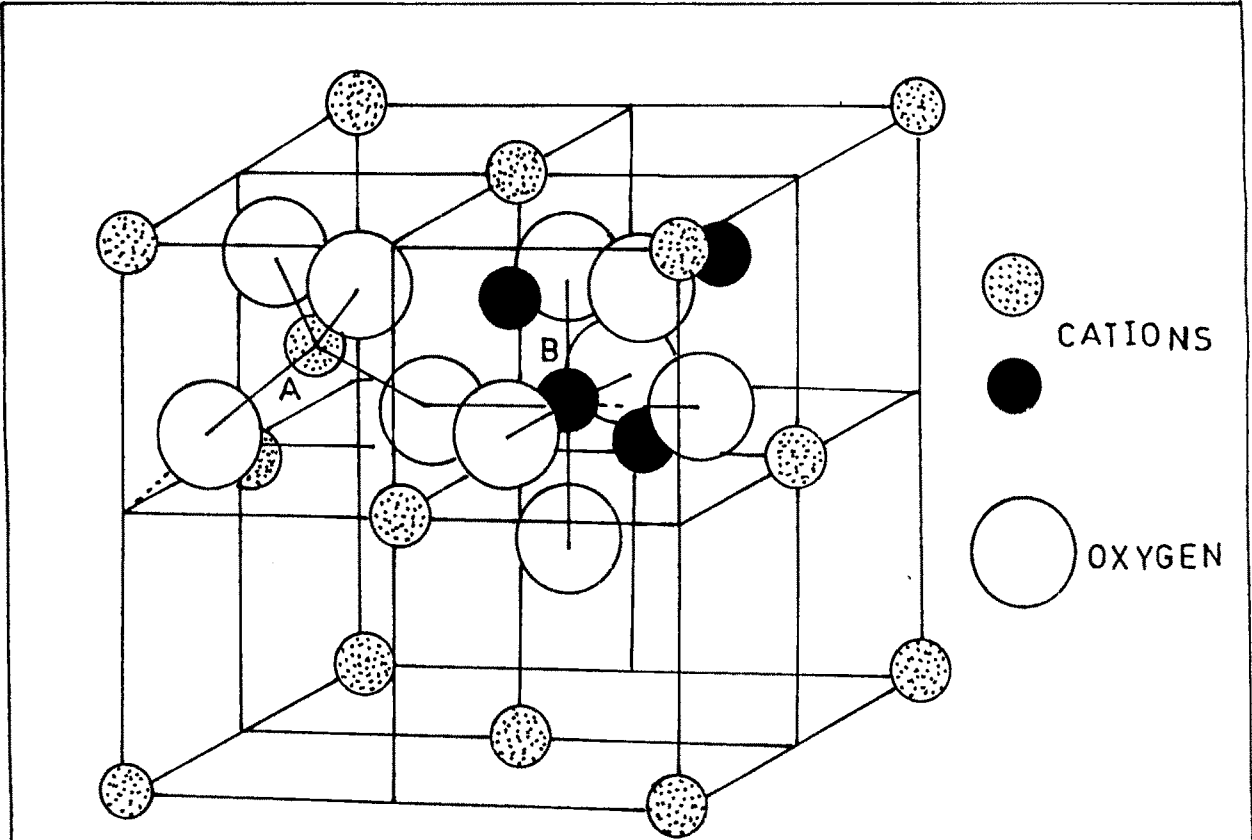
#### 1.4 STRUCTURE OF SPINEL FERRITES

The structure of spinel ferrite was determined by Bragg<sup>9</sup> and Nishikawa.<sup>10</sup> The compounds with the general formula  $\text{AB}_2\text{O}_4$  crystallize with the same structure as the mineral spinel,  $\text{MgAl}_2\text{O}_4$  (Fig. 1.2a). The three important parameters associated with atomic

arrangement of spinel are, the lattice parameter 'a', oxygen parameter 'u' and cation inversion parameter 'i'. Oxide spinels are used as examples to explore the interrelationship between these parameters.

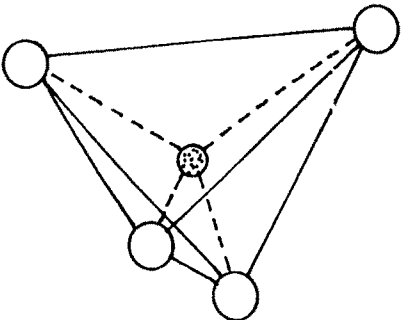
The general chemical formula for spinel ferrite is  $M^{2+}Fe_2^{3+}O_4^{2-}$  (where M is a divalent metal ion and Fe is a trivalent (ferric) iron ion. In the formula  $AB_2O_4$ , A and B refer to tetrahedral and octahedral cation sites respectively. The oxygen anions are arranged in a face centered cubic lattice. Each unit cell contains 8 formula units with  $O^{2-}$  anions in the 32(b) sites and  $Fe^{3+}$  and  $M^{2+}$  cations occupying the 16(d) and 8(a) sites. The space group of ferrites is  $Fd_3m O_h^7$  and lattice parameters are typically of the order of  $8.3 \text{ \AA}$ . The relative positions of the A and B cations with respect to neighbouring oxygen anions is quite important for strong superexchange, which is dependent on both the cation-anion-cation bond angle and the interatomic distance between the cations and anions.

The metal ions occupy the interstitial positions of four fold (a) and six fold (d) oxygen co-ordination. In one, magnetic ion is surrounded by four oxygen ions at the vertices of tetrahedron and is called tetrahedral or A site, (Fig.1.2b) whereas in the other it is surrounded by six oxygen ions at the vertices of an octahedron and is called octahedral site or B site (Fig. 1.2c).



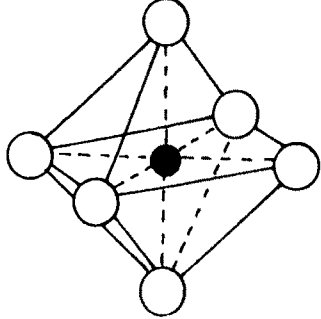
( a )

THE SPINEL STRUCTURE (SCHEMATIC )



( b )

TETRAHEDRAL SITE



( c )

OCTAHEDRAL SITE

FIG 1-2

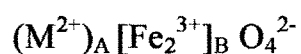
There are 96 interstices between the anions in the unit cell of  $AB_2O_4$  compound. Of which only eight tetrahedral and sixteen octahedral sites are occupied by the cations. The equilibrium distribution of cations in the spinel structure depends on ionic radii, electronic configuration, electrostatic energies and polarization effects.

## 1.5 CLASSIFICATION OF FERRITES

The ferrites are classified on the basis of cation distribution. Barth and Posjank<sup>11</sup> have observed different types of cation distribution in ferrites.

### a. Normal Spinel Ferrites

In normal spinel ferrites all the  $M^{2+}$  ions occupy the tetrahedral sites, while  $Fe^{3+}$  ions occupy the octahedral sites. In these ferrites there is no A-B interaction as A sites do not contain any magnetic ion. The B-B interaction lines up with half the magnetic ions in one direction so that alternate planes of B sublattice are magnetised in opposite direction. This results in the nonmagnetic character of the ferrite.  $ZnFe_2O_4$  and  $CdFe_2O_4$  are the ferrites with the normal spinel structure. The cation distribution for normal spinel ferrite is given by

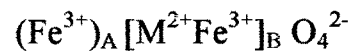


For normal spinel inversion parameter  $i = 0$



### b. Inverse Spinel Ferrites

When the divalent cations are on B sites and the trivalent ions are equally divided between A and a B site, the structure is called an inverse spinel structure. The cation distribution for inverse spinel ferrite is



The inverse spinel ferrites show ferrimagnetic behaviour.

e.g.  $\text{MgFe}_2\text{O}_4$ ,  $\text{CoFe}_2\text{O}_4$  show inverse spinel structure.

For inverse spinel structure inversion parameter  $i = 1$ .

### c. Random Spinel Ferrites

When all the divalent and trivalent iron ions are distributed randomly over A and B sites then resulting ferrite is called random spinel ferrite.

The cation distribution of random spinel ferrite is expressed as-



where  $x$  = coefficient of normalcy

$1-x$  = coefficient of inversion

Inversion parameter  $i = 2/3$  corresponds to a random distribution of cations.

## 1.6 TYPES OF FERRITES

The spinel ferrites are classified depending upon their chemical compositions as follows.

### a. Simple Ferrites

When a divalent ferrous ion in  $\text{Fe}_3\text{O}_4$  is replaced by another divalent metal ion like Mn, Co, Ni, Cu, Zn, Cd or Mg the resulting ferrite is called simple ferrite e.g.  $\text{MnFe}_2\text{O}_4$ ,  $\text{CuFe}_2\text{O}_4$ ,  $\text{ZnFe}_2\text{O}_4$ .

### b. Mixed Ferrites

When a ferrous ion in  $\text{Fe}_3\text{O}_4$  is replaced by two other divalent metal ions like Cu, Co, Cd, Ni etc. without altering the stoichiometry of the system, the resulting ferrite is called mixed ferrite.

e.g.  $\text{Zn}_x \text{Co}_{1-x} \text{Fe}_2\text{O}_4$ ,  $\text{Cd}_x \text{Mg}_{1-x} \text{Fe}_2\text{O}_4$

### c. Substitutional Ferrites

When the divalent metal ions as well as trivalent iron ions are replaced by other magnetic or non-magnetic ions in the spinel structure, the resulting ferrite is called substitutional ferrite.

e.g.  $\text{Ni}_{1-x} \text{Zn}_x \text{Fe}_2\text{O}_4$ ,  $\text{Ni Fe}_{2-x} \text{Al}_x\text{O}_4$ .

The net magnetization in such a ferrite depends on the site occupancy of the substitution.

## 1.7 PROPERTIES OF FERRITES

### 1.7.1 Electrical Properties

Spinels have a wide range of resistivity<sup>12</sup> from  $10^{-5}$  to  $10^{12} \Omega \cdot \text{cm}$ . 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 effect. The charge transport can vary with the composition and the method of preparation of ferrites. The conductivity in ferrite depends upon purity of compound, stoichiometry, sintering temperature, time, atmosphere, physico-chemical history, density, porosity, grain size and chemical homogeneity<sup>13</sup>.

The temperature dependence of resistivity is given by

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

where  $k$  = Boltzmann constant

$\Delta E$  = activation energy

$T$  = absolute temperature

The plots of  $\log \rho$  vs  $1/T$  show a straight line and break occurs closely at the ferrimagnetic Curie temperature.<sup>14</sup> The high value of  $\Delta E$  is associated with high resistivity at room temperature. During the preparation of polycrystalline ferrites, oxygen dissociation sets

up above 1200<sup>0</sup> giving rise to Fe<sup>2+</sup> ions. The Fe<sup>2+</sup> ions are responsible for the electron conduction.

The ions having two valance states get distributed randomly over the crystallographically equivalent sites. The high conductivity of ferrites is due to the presence of Fe<sup>2+</sup> and Fe<sup>3+</sup> 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 and back within the octahedral position. The transition does not cause a charge in the energy state of the crystal.

### 1.7.2 Dielectric Properties

In ferrites eddy currents may flow due to dielectric displacement. Ferrite show abnormally high dielectric constant and dispersion of dielectric constant and resistivity in the frequency range from a few Hz to MHz. Koops<sup>15</sup> described the phenomenological theory of dielectric dispersion in ferrites at low frequencies. The theory of dielectric dispersion is based on the assumption of parallel resistance and capacitance of material. The theoretical and observed frequency variations of dielectric constant and resistivity are in good agreement for some samples. Activation energy observed by Kamiyoshi<sup>16</sup> and Fairweather<sup>17</sup> from temperature variation with relaxation time agrees will with those obtained with temperature variation of

resistivity. The high dielectric constant arising at low frequency may be due to oxygen ions, which have three B-site, and one A site neighbours. This is justified in view of the fact that oxygen possesses the maximum polarizability. Abnormally high dielectric constant have been reported by several workers as a result of lower frequency.<sup>18</sup>

When a dielectric material is subjected to an a.c. electric field, there is a relative displacement of positive and negative ions resulting in an electric dipole moment. The dipole moment per unit volume is called polarization. The total polarization of dielectrics can be represented as

$$P = P_e + P_i + P_o + P_s \quad \dots \quad (1.2)$$

where

- $P_e$  = electric polarization
- $P_i$  = ionic polarization
- $P_o$  = orientational polarization
- $P_s$  = interfacial or space charge polarization

These polarizations are shown in Fig. (1.3a). The frequency range of each of these polarization is as shown in Fig. (1.3b).

### 1.7.3 Magnetic Properties

#### a. Magnetisation in Ferrites

The magnetisation in ferrites results from the distribution and alignment of magnetic ions on the octahedral and tetrahedral sites.

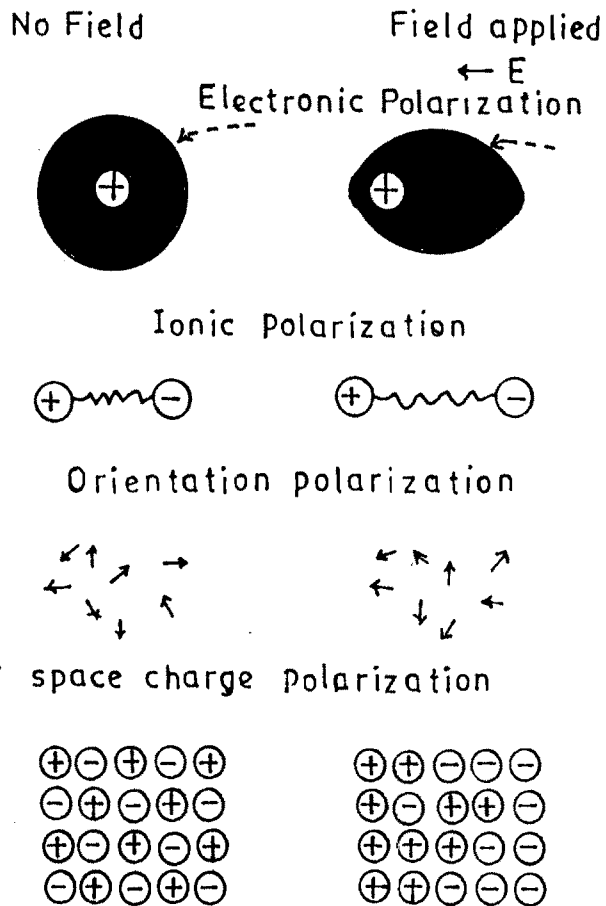


FIG. 1-3 a - SCHEMATIC REPRESENTATION OF DIFFERENT MECHANISMS OF POLARIZATION .

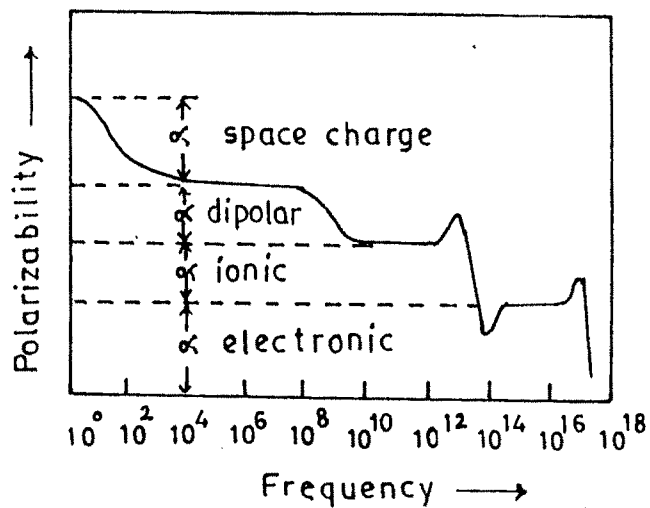


FIG. 1-3 b - FREQUENCY DEPENDENCE OF SEVERAL CONTRIBUTION TO THE POLARIZABILITY .

Certain characteristic features of the ferrite can be explained on the basis of their magnetic properties. The susceptibility, permeability, saturation magnetisation, Curie temperature depends upon the concentration of divalent ions on both the cation sites and these properties are called intrinsic properties. The properties, which depend upon the structure of ferrite, are called structure sensitive properties e.g. coercive force. The intrinsic properties of polycrystalline ferrite are influenced by pinned wall domains due to porosity at the grain boundaries.

The exchange interaction between the magnetic moments of neighbouring ions is responsible for the magnetization in ferrites. The interaction is positive when the moments are parallel and negative when the moments are antiparallel. The interaction arises from an indirect exchange mechanism in which magnetic ions are coupled through electron transfer with intermediate anions. This concept of indirect exchange has been forwarded by Kramers<sup>18</sup> and then developed by Anderson<sup>4</sup> in the form of superexchange interaction. The idea of superexchange interaction is used to account for the magnetization in normal and inverse spinel ferrites.

#### **b. Magnetic Interaction in Ferrites**

The various possible magnetic interaction were studied by Gorter.<sup>19</sup> As metal ions occupy two different sites viz. tetrahedral

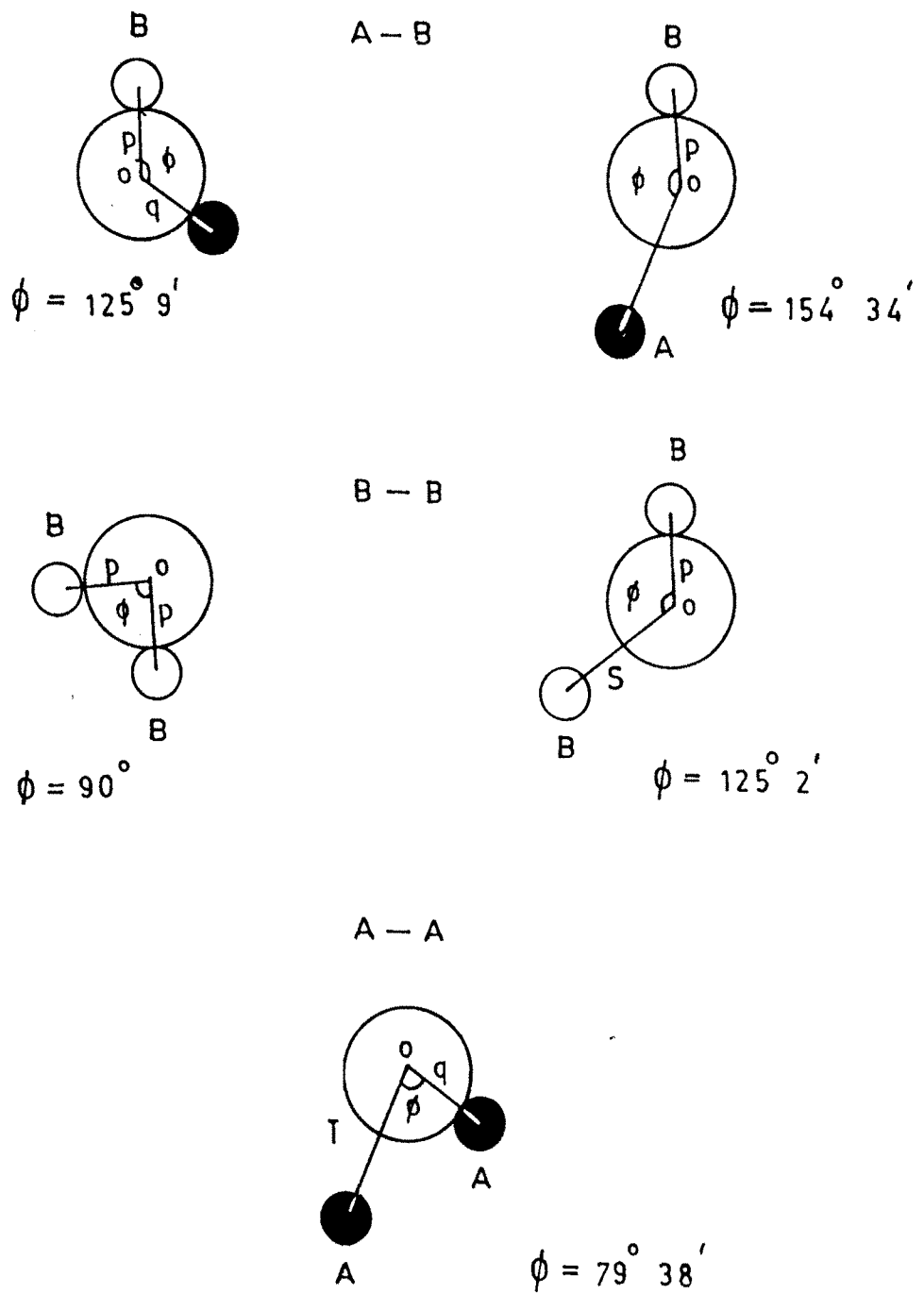


FIG. 1-4 - ANGLE BETWEEN A-A, B-B & A-B  
 CATIONS IN A SPINEL STRUCTURE.



and octahedral sites, three kinds of magnetic interactions between metal ions are possible via intermediate  $O^{2-}$  ions by superexchange mechanism, namely A-A, A-B and BB interactions (Fig. 1.4). In the superexchange interaction, there is an excitation of oxygen ion from its ground state.<sup>20</sup> The superexchange interaction increases rapidly with the decrease in the distance between the ions. The A-B interaction is the only predominant interaction in ferrites. It is observed that the interaction energies between two magnetic ions A and B depend on the distance of these ions from the oxygen ion through which interaction occurs and the angle between A-O-B. The angle of  $180^\circ$  gives maximum interaction energy. The spinels of A and B site ions, will oppositely magnetize the sublattices A and B giving resultant magnetic moment equal to the difference between those of A and B site ions. In general, value of saturation magnetic moment for B site ( $M_B$ ) is greater than that of the A site ( $M_A$ ). The resultant saturation magnetization ( $M_s$ ) can be written as-

$$M_s = | M_B - M_A | \quad \dots \quad (1.3)$$

## 1.8 APPLICATIONS OF FERRITES

Ferrites have vast applications from microwave to radio frequencies (Fig. 1.5a, 1.5b). The low frequency applications of soft ferrites include magnetic heads, inductors, transformer cores, filter cores etc. High frequency applications include a large number of

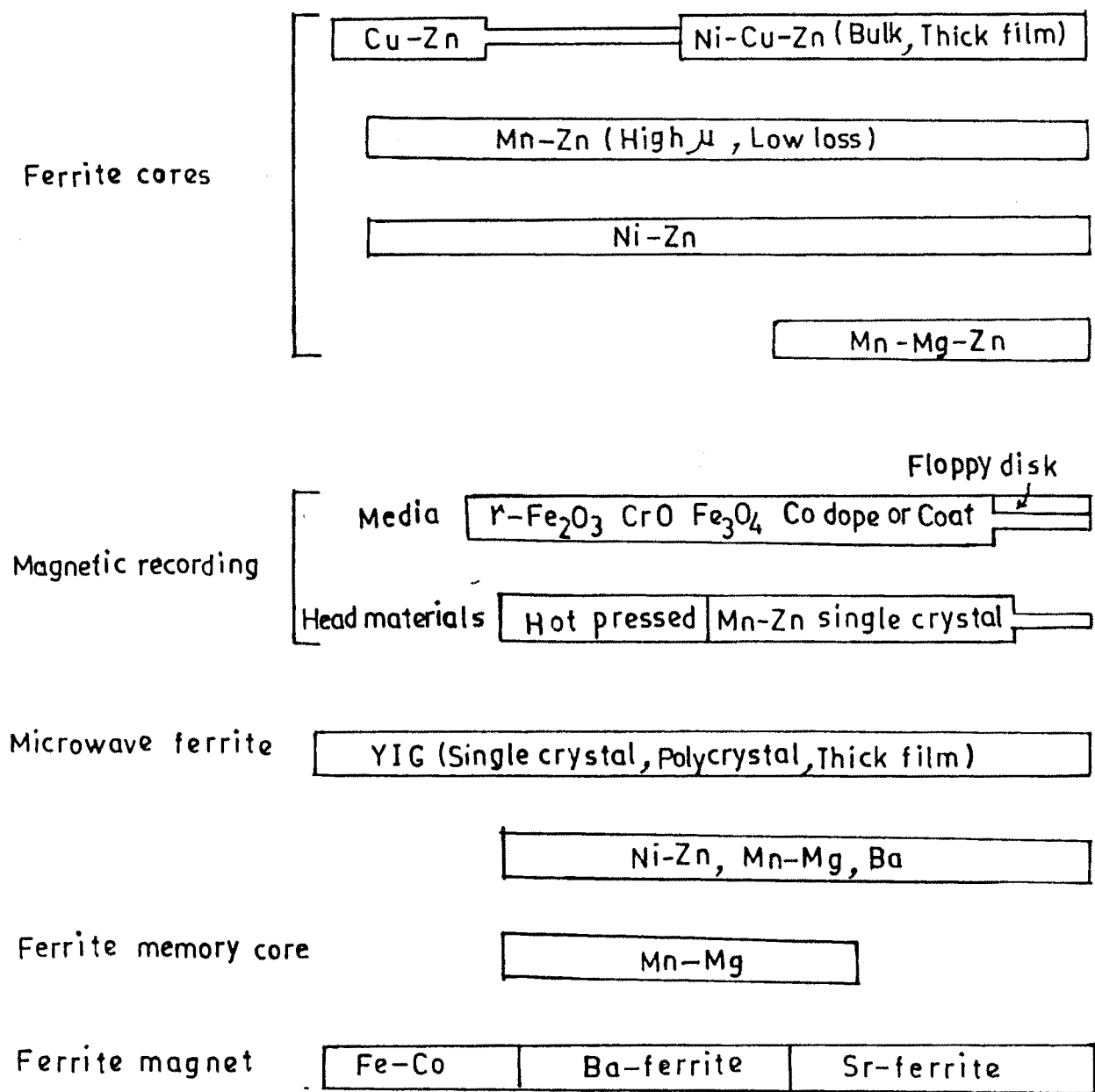


Fig. 1.5 (a) — APPLICATIONS OF FERRITES .

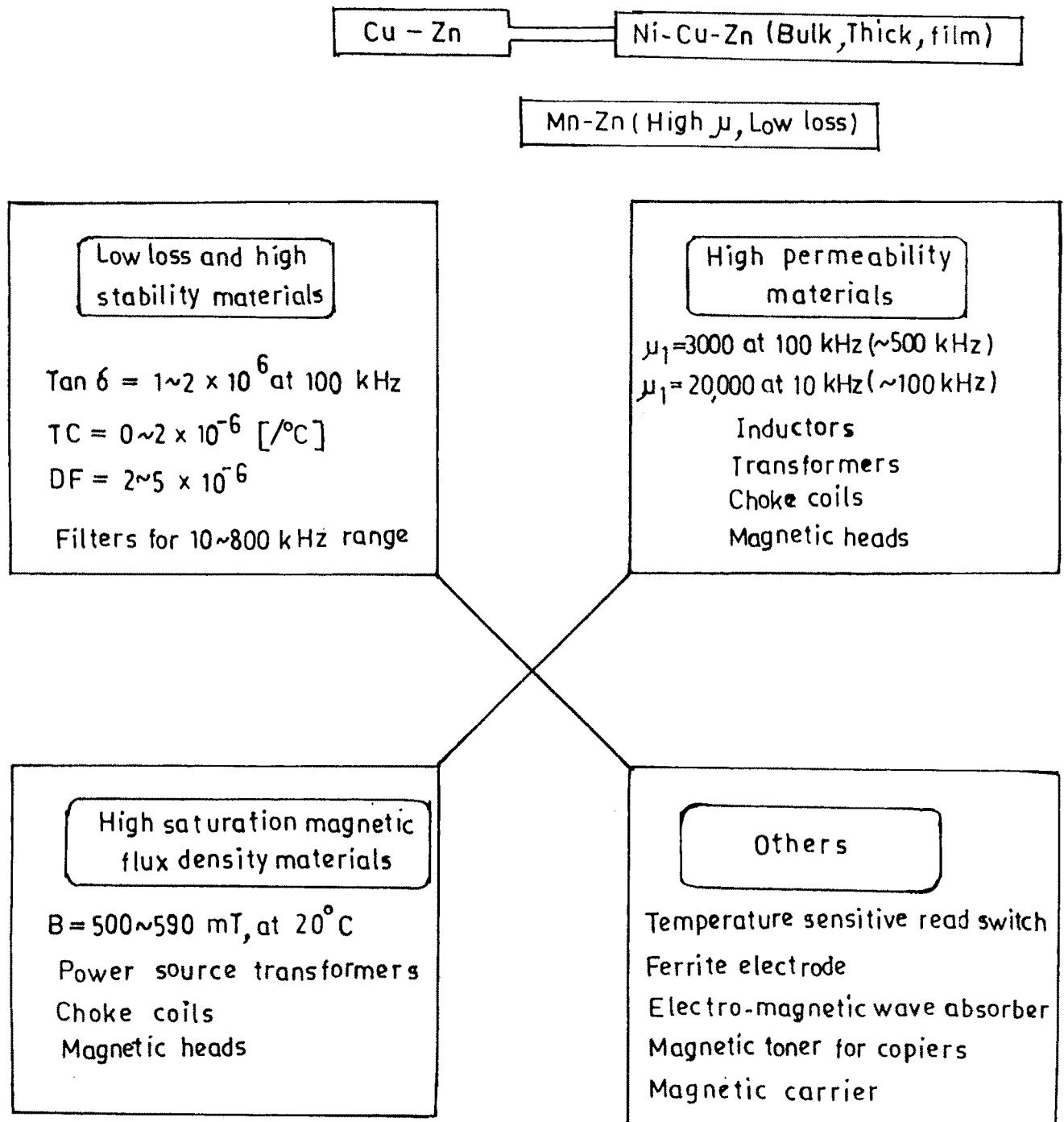


Fig. 1.5 (b) — RELATION BETWEEN THE CHARACTERISTICS OF SOFT FERRITIES AND THEIR APPLICATIONS .

microwave components such as circulators, insulators, gyrators; phase shifters, YIG-tuned filters, switches and substrate for microwave integrated circuits etc.

The microwave ferrites are widely used in radar systems for air traffic control, communication network and microwave heating. Hexaferrites find wide applications in motors, generators, loudspeakers, telephones, motor switches, magnetic latches and magnetic separators.

The spinel ferrites are extensively used for miniaturization. Some ferrites exhibit a typical rectangular hysteresis loop. Due to this square loop characterization they are suitable for flip-flops in computer and memory devices. The square loop ferrites typically utilize hysteresis information.<sup>21</sup> Other applications are loading MW antenna in radio receivers, broad band transformers, mechanical filters, directional couplers etc.

A wide variety of ferrite cores are available now-a-days which find extensive applications in TV, audio, tape recorders, telephone exchanges, computers, control equipments, transmission equipment etc. The paints containing ferrites when applied over tall buildings are effective in suppressing the ghost images.<sup>22</sup> Some ferrites are finding use as catalysts in chemical reactions and environmental protection.

Power ferrites for high frequency applications have been developed in order to suppress gyromagnetic and dielectric losses by high a.c. resistivity, low magnetic flux density and small core sizes. The piezomagnetic ferrites have been developed for use in ultrasonics and radio frequency electronics.

Ferrites have entered new areas like atmospheric pollution control<sup>23</sup>, hydrogen production<sup>24</sup> sensors, bioceramics and cancer treatment.<sup>25</sup> Applications of ferrite as humidity sensors<sup>26</sup> and thermojunctions<sup>27</sup> are being developed.

Research on the application of ferrites for protecting the natural environment is being currently investigated. These include -

- i. Washer disposal method for factory drains<sup>28</sup>
- ii. Heat decomposition of  $\text{CO}_2$  using a mixture of carbon and ferrite powders heated at  $300^\circ\text{C} - 700^\circ\text{C}$ .<sup>29</sup>
- iii. Transformation of solar energy to hydrogen energy using ferrites as catalysts.<sup>30</sup>
- iv. Heat decomposition of  $\text{NO}_x$  gas using spinel ferrite.<sup>31</sup>

## 1.9 REVIEW OF LITERATURE

The polycrystalline Ni-Zn ferrites are suitable for magnetic heads, radio frequency coils, transformer cores and rod antennas. Ni-Zn ferrite is the most suitable material for device applications in the supermicrowave and lower millimeter wave range.<sup>32,33</sup> The Ni-

Zn ferrites possess a very high electrical resistivity, excellent magnetic properties and low eddy current losses than Mn-Zn and Mg-Zn ferrites.

Many workers have studied the electrical<sup>34</sup> and magnetic properties<sup>35</sup> of non-stoichiometric Ni ferrites and the mixed stoichiometric Ni ferrites.<sup>36-42</sup> Bagdanovich<sup>43</sup> has reported the influence of porosity on the initial permeability, Q factor and parameters of hysteresis loop of Ni-Zn ferrite. Developments in the preparation of Mn-Zn and Ni-Zn ferrites, with reference to technically important properties such as  $M_s$ ,  $H_c$ ,  $\mu_i$ , loss factor, dependence of  $\mu_i$  on temperature, time and applied magnetic field etc. are reported by Das.<sup>44</sup> The magnetic phase transition for  $Fe_3O_4$  and structurally disordered  $Zn_x Ni_{1-x} Fe_2O_4$  are reported by Hang et.al.<sup>45</sup> Mossbauer and magnetization studies of  $Ti^{4+}$  substituted Ni-Zn ferrite are carried out by Srivastav et.al.<sup>46</sup>

Rezlescu et.al.<sup>47</sup> have studied the effect of addition of CaO,  $Na_2O$ ,  $ZrO_2$ ,  $Sb_2O_3$  and  $K_2O$  on the densification of Li-Zn and Ni-Zn ferrites. They have observed that such additives affect the porosity. Das et.al.<sup>48</sup> have studied the microstructure of Ni-Zn ferrites obtained from hydrothermal process. Patil et.al. have studied the lattice parameter of Mn Ti and Mn Sn substituted Ni-Zn ferrite and it is found to increase with Mn addition.<sup>49</sup> Seeback coefficient and

electrical conductivity of mixed Ni-Zn ferrites at room temperature are measured by Ravinder et.al.<sup>50</sup> The effect of substitution of a Jahn Teller ion like  $\text{Cu}^{2+}$  on the properties of Ni-Zn ferrites has shown some interesting results.<sup>51</sup> Ni-Cu-Zn ferrite give high initial permeability and have tremendous potential for applications as SMO, MICI, in computer memory devices and in logic devices. Joshi et.al.<sup>52</sup> have reported their results on magnetization, Curie temperature and Y-K angles of Cu substituted Ni-Zn ferrites. They have observed that on addition of copper the Curie temperature and magnetisation are lowered. This is attributed to lower magnetic moments of  $\text{Cu}^{2+}$ .

The low loss Ni-Cu-Zn ferrites for deflection yoke used in display monitors with a high resolution have been developed earlier.<sup>53</sup> Narn et.al.<sup>54</sup> have studied the effect of Cu substitution on the electrical and magnetic properties on Ni-Zn ferrites. Miggue et.al.<sup>55</sup> and Nakamura et.al.<sup>56</sup> have studied the electromagnetic properties and microstructure of low temperature fired Ni-Cu-Zn ferrite. Deshmukh<sup>57</sup> has studied effect of Cu substitution on the properties of Ni-Zn ferrites prepared by chemical route. They have reported the change in magnetization on copper substitution.

Nakamura et.al.<sup>58</sup> have studied the low temperature sintering of Ni-Cu-Zn ferrite and clarified the relation between preparation

conditions (calcination temperature particle size of starting materials) and some properties of the sintered ferrite. They observed that the sintering density and the complex permeability of Ni-Cu-Zn ferrite ceramics can be controlled by particle size, starting oxide materials and calcination temperature.

Jau-Ho Jean et.al.<sup>59</sup> have studied the effect of PbO addition on the densification, microstructure and properties of low temperature Co-firable Ni-Cu-Zn ferrite. They have observed that the densification kinetics are greatly enhanced with a small amount of PbO which is present as a dopant in the NiCuZn ferrite. The grain size becomes larger when PbO is added.

Zhang et.al.<sup>60</sup> obtained the ultra-fine and highly reactive Ni-Cu-Zn ferrite powder by gel self propagating method. They observed that densification of ferrites and homogeneous grain growth were the main factors affecting high initial permeability. Lin et.al.<sup>61</sup> have applied microwave-sintering technique for densifying Ni-Cu-Zn ferrite materials and multilayer chip (MC) inductors. They have reported that high impedance multilayer chip inductors can be achieved by microwave sintering of the materials at 800-850<sup>0</sup>C for 20 minutes.



## 1.10 ORIENTATION OF THE PROBLEM

The production of high quality ferrites will continue to increase every year. It will experience a steady growth with further advancement in science and technology. In the last few decades the soft ferrites have aroused considerable interest due to their electrical, magnetic, electronic, microwave and computer applications.

Snoek has explained that the Ni-Zn ferrites have quite excellent magnetic properties. Ni-Zn ferrites exhibit significantly low eddy current losses than Mn-Zn ferrites. Ni-Cu-Zn ferrite has many applications including rotary transformers, noise filters, multilayer ferrite chip components etc. The new power ferrites have two new attributes not previously needed in other applications. (i) They have to possess higher saturation. (ii) they require relatively low core losses at higher flux densities and at fairly high frequencies.

At present Ni-Cu-Zn ferrites have been used extensively for the production of the MLCI so develop Ni-Cu-Zn ferrites with excellent magnetic properties especially with high initial permeability is needed to be done. From the literature survey, it is observed that the nickel ferrites seem to be interesting.

In view of the above consideration, it was proposed to undertake work on preparation, characterization and measurement of

electrical magnetic properties of Ni-Cu-Zn ferrites. The work involved -

- i. The preparation of  $\text{Ni}_x\text{Cu}_{0.2}\text{Zn}_{0.8-x}\text{Fe}_2\text{O}_4$  ferrites with  $x = 0.2, 0.3, 0.4, 0.5, 0.6, 0.7$  and  $0.8$ .
- ii. X-ray diffraction studies for determination of crystal structure and lattice parameters.
- iii. Determination of Curie temperatures.
- iv. D.C. and A.C. electrical conductivity measurements to understand the conduction mechanism.
- v. Magnetisation studies to observe the variations in the magnetic moments, magnetization and hysteresis with composition at room temperature and
- vi. The variation of permeability with temperature to understand the role of substitution.

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