## INDEX

### CHAPTER II

Crystal Structure, Properties and Applications of Ferrite, Ferroelectric and ME Composites

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2.1 GENERAL INTRODUCTION TO FERRITE

Ferrites are chemical compounds consisting of ceramic materials with iron oxide (Fe₂O₃) as their principal component. Ferrite crystallizes in spinel, garnet, ortho ferrite and hexagonal structures. By virtue of their crystal structure they can accommodate variety of cations at available sites bringing wide variation in the properties. Ferrite possesses both electrical and magnetic properties, which makes them useful in much technological applications such as antenna rod, transformer core, memory chips, etc. [1]. Ferrite possesses high electrical resistivity, low eddy current and dielectric loss, high saturation magnetization, high permeability, high Curie temperature, etc. These remarkable properties of ferrites depends on many factors such as method of preparation, preparative parameters, cation distribution, nature and type of dopant etc. [2]. The most common method of preparation of ferrite is conventional ceramic method. In the recent years wet chemical methods have become popular because of emerge of nano science and nanotechnology. The wet chemical methods yield particles of nanometer dimension and their properties are found to be superior and useful in recent applications like medical diagnostics, gas sensors, catalyst etc. [3,4].

2.2 SPINEL FERRITE

Spinel ferrite crystallizes in the cubic structure. The spinel lattice is composed of
a close-packed oxygen anions arrangement in which 32 oxygen ions form the unit cell. These anions are packed in a face centered cubic (FCC) arrangement leaving two kinds of spaces between anions tetrahedrally coordinated sites (A), surrounded by four nearest oxygen atoms and octahedrally coordinated sites [B], surrounded by six nearest neighbour oxygen atoms (Fig. 2.1). There are total 64 tetrahedral sites and 32 octahedral sites in the unit cell, of which only 8 tetrahedral sites and 16 octahedral sites are occupied, resulting in a structure that is electrically neutral [5]. Normal spinel structures are usually cubic closed-packed oxides with one octahedral and two tetrahedral sites per oxide.

![Fig. 2.1 Crystal structures of spinel](image1.png)

![Fig. 2.2 Inverse spinel structures](image2.png)
The tetrahedral points are smaller than the octahedral points. B\textsuperscript{3+} ions occupy the octahedral holes because of a charge factor, but can only occupy half of the octahedral holes. A\textsuperscript{2+} ions occupy 1/8 of the tetrahedral holes. A common example of a normal spinel is MgAl\textsubscript{2}O\textsubscript{4}. Inverse spinel structures however are different in the cation distribution, in that the entire A cations and half of the B cations occupy octahedral sites, while the other half of the B cations occupy tetrahedral sites. A common example of an inverse spinel is Fe\textsubscript{3}O\textsubscript{4}, if the Fe\textsuperscript{2+} (A\textsuperscript{2+}) ions are d\textsuperscript{6} high-spin and the Fe\textsuperscript{3+} (B\textsuperscript{3+}) ions are d\textsuperscript{5} high-spin. As shown in Fig. 2.2.

2.3 GARNET

The garnets have orthorhombic crystal structure. (Oxygen polyhedral, surrounding the cations) but with trivalent cations (including rare earth and Fe\textsuperscript{3+}) occupying tetrahedral (d), octahedral (a) or dodecahedral (a), 12-sided distorted polyhedral (c) sites. Specifically, the interaction between tetrahedral and octahedral sites is antiparallel, and the net magnetic moment is antiparallel to the rare earth ions on the c sites. The garnet structure is one of the most complicated crystal structures and it is difficult to draw a two dimensional representation that shows clearly all the ions (160) in the unit cell [6]. The chemical formula for garnets is 3Me\textsubscript{2}O\textsubscript{3} · 5Fe\textsubscript{2}O\textsubscript{3} where ‘Me’ represents the trivalent rare earth ions like non magnetic yttrium or a magnetic rare earth.

2.4 ORTHO FERRITE

Rare earth orthoferrites are classified as ferrites, although they are antiferromagnets. The magnetic oxides with perovskite structure, which are an exception
in the group of oxides. The perovskite structure is shown schematically in figure 2.3. Large divalent or trivalent ions (A) occupy the corners of a cube and small trivalent or tetravalent metal ions (B) occupy the centre of the cube. The oxygen ions are situated centrally on the faces of the cube.

The general chemical formula is $\text{ABO}_3$, where A represents yttrium or a rare earth. The magnetic structure inside of a sub lattice is usually collinear ferromagnetic, but the different sub lattices are coupled antiferromagnetical to the different number of magnetic ions in different magnetic moment, giving rise to ferrimagnetisms. The nature of the super exchange interaction depends not only on the type of the magnetic ion, but rather strongly on the bond length and bonding angle [7].

2.5 HEXAGONAL FERRITES

Hexagonal ferrite possesses magneto plumbite structure. The symmetry of magneto plumbite structure is hexagonal. Thus, it has a major preferred axis called the ‘c’-axis and a minor axis called the ‘a’-axis. The preferred direction is used to good advantage as a permanent magnetic material. The oxygen ions are closely packed as they are in the spinel structure but there are oxygen layers which now include the $\text{Ba}^{2+}$, $\text{Sr}^{2+}$ or
\( \text{Pb}^{2+} \) ions which have about the same ionic radii as the oxygen ions and therefore can replace them in the lattice. Several related ferromagnetic oxides are also available, all of which can be derived by combining the ferrite spinel \((\text{MeO.fe}_3\text{O}_5)\) and ferroxdure \((\text{BaO} \cdot 6\text{Fe}_2\text{O}_3)\).

2.6 PROPERTIES OF FERRITES

The magnetic properties of ferrites, like saturation magnetization \((\text{Ms})\), initial permeability \((\mu)\), Curie temperature \((T_c)\) and coercivity \((H_c)\) depend on the concentration of divalent metal ions on both tetrahedral and octahedral sites. Hence the properties are known as “intrinsic properties”. On the other hand, the hysteresis loop, resistivity, a. c. conductivity and dielectric constant depend on the structure of ferrites and hence are called as “structure sensitive proportion”. The magnetic properties are strongly dependent on the chemical composition, sintering atmosphere, sintering temperature, sintering time, grain size, crystal structure, cation distribution and porosity of the materials.

2.6.1 Magnetic anisotropy

The magnetic material can be magnetized in any direction. In these materials, very small field is required for magnetization. This dependence of strength of magnetization on the magnetization direction is known as magnetic anisotropy and the energy required to turn the magnetization from the preferred direction to the desired direction is known as anisotropy energy [8]. Magnetic anisotropy depends upon Dipolar instruction energy in crystal lattices and nature of magnetic ion involved. The anisotropy of the materials not only depends on the nature of the magnetic moment but also on the physical shape of the magnetic moment. When magnetic field is applied, the functional change in the length of the material occurs and gives positive or negative anisotropy.
constant. Magnetic anisotropy is the important property of magnetic materials, from both technological and fundamental point of view.

2.6.2 Magnetostriction

Magnetic materials experience change in the dimensions during magnetization. The major effect in a change in a shape, with the volume remains approximately constant. This phenomenon is known as magnetostriction. These changes occur, when the material is cubic but the magnetic order is non-cubic. If material shrinks in the direction of applied magnetic field, the magnetostriction is considered to be negative and it expands, then termed as positive magnetostriction.

The saturation magnetization of a material decreases on heating towards the Curie temperature due to the decrease in the degree of alignment of the atomic magnetic moments. As this alignments and the saturation magnetization decreases so does the volume expansion caused by the spontaneous magnetostriction and hence the materials contracts. In the case of inverse this contraction due to loss of spontaneous magnetostriction is equal to the expansion caused by normal thermal vibration processes and hence the material shows no change in dimensions [9].

However, above the Curie temperature there is no longer any magnetic ordering and thermal expansion occurs normally. Field induced magnetostriction occur when the magnetic domains align and the dimension change caused by the ordering of the atomic magnetic moments all combine to give a dimensional change to the block of material.

2.6.3 Magnetic hysteresis of ferrite

When ferromagnetic material, consisting of magnetic domain with spins oriented at random, is subjected to an external magnetic field, the domain start orienting in the
direction of the magnetic field and the net magnetization may vary between zero and saturation value.

Fig. 2.4 Magnetic Hysteresis loop of ferrite

Under the influence of an external field, the domain wall experiences a pressure and grows at the expense of unfavorably oriented domains. Due to the irreversible domain wall movements, the magnetization will always lag behind the field and an open loop will be traced. This phenomenon is known as magnetic hysteresis and the loop is called a hysteresis loop as shown in Fig. 2.4.

At certain magnetic field the material attains a saturation value (i.e. saturation magnetization-M_s) and after reduction of the magnetic field, certain magnetization is retained which is termed as residual or remanent magnetization (M_r). When the direction of magnetic field is reversed, the remanent magnetization reduces and finally becomes zero at certain magnetic field, called as coercive field (H_c). The loop provides information about the work done in cycle of magnetization, coercivity and saturation magnetization. Extensive work has been done on the magnetization of ferrites through
hysteresis measurements. The shape of hysteresis loop provides the information about the domain state of grains inside the sample. Based on the hysteresis phenomenon, Bean has classified the magnetic particles into single domain, multi domain and super magnetic domain [10].

### 2.7 APPLICATIONS OF FERRITES

Ferrites have importance in engineering and technology because they possess spontaneous magnetic moment below the Curie temperature, just as iron, cobalt, nickel. Due to very low eddy current losses, ferrites are used as a core of coils in microwave frequency devices and computer memory core elements. Due to relatively low permeability and flux density compared to iron, ferrites are not suitable for the use in high field and high power applications, such as motors, generators and power transformers, but they can be used in low field and low power applications. Ferrites are used as ferromagnetic insulators in electrical circuits. Ferrites like ZnO find low frequency applications in timers. They are also used as switches in refrigerators, air conditioners, etc. Ferrites are used as magnetic head transducer in recording. Ferrites have many applications in society on the basis of electronic power, communication and information storage. Ferrites have become established in many branches of telecommunication and electronic engineering and they now embrace very wide diversity of compositions, properties and application. The Mn-Zn ferrite and Ni-Zn ferrites were developed for a wide range of applications where high permeability and low losses were the main requirements. The applications started in the field of telephone transmission where carrier systems being essentially analogue required large number of high performance inductors, transformers mainly operating in frequency range from about 40
KHz to 500 KHz. Later on other ferrite compositions such as Li-Zn or Mg-Zn ferrites were introduced for deflection yokes requiring high resistivity to facilitate the winding of the coils directly to the ferrite. In domestic radio receivers ferrite rods or plates enabled compact amplitude modulation antennas to be made. The most important application of the ferrite is in recording heads for audio, data and video recorders, their use in inductive elements for audio interference suppression and their application as cores for transformers in switch mode power supplies [11].

**Ferrites for Telecommunications**

Telecommunication is the first important branch of technology where ferrites have been used on a large scale. Today, against many predictions, it still is an important market for ferrite cores. Most important applications are in filter inductors and Pulse as well as matching transformers.

**Ferrites for Power conversion**

Power conversion is a major application area for modern ferrites. Largest application of suppression ferrites are industrial, computer, telecom, medical, aerospace applications, shield beads, snap-on cores and flat cable beads.

### 2.8 CRYSTAL STRUCTURE OF FERROELECTRIC

Barium titanate (BaTiO$_3$) is one of the first simple structures to exhibit ferroelectric properties. It belongs to the family of compounds with general formula ABO$_3$, A is di or monovalent metal ion B is tetra or pentavalent metal ion, whose representative in nature is the mineral CaTiO$_3$, called perovskite. It is widely used because of:

1) Its coupling factor is higher than that of any previously known material.
2) It is chemically more stable than Rochelle salt

3) It has the advantage of easy manufacture by ceramic technique and

4) It has a wider temperature range of operation [12].

It is a ferroelectric oxide that undergoes a transition from a ferroelectric tetragonal phase to a paraelectric cubic phase upon heating above 130°C. The structure of cubic perovskite BaTiO$_3$ is shown Fig. 2.5 (a), titanium atoms are octahedrally coordinated by six oxygen atoms. In Perovskite structure BaTiO$_3$, barium are located at the corners, the titanium ion is located at the center of the cubic cell. Each barium ion is surrounded by 6 oxygen ions and each oxygen is surrounded by 4 barium and 2 titanium ions. The tetragonal unit cell is shown in Fig. 2.5 (b).

![Unit cell of BaTiO3](image)

**Fig.2.5.** Unit cell of BaTiO3 in both the (a) cubic Pm-3m structure and (b) tetragonal P4 mm structure.
The elongation of the unit cell along the c-axis and consequently the deviation of the \( c/a \) ratio from unity are used as an indication of the presence of the ferroelectric phase [13, 14].

In the tetragonal unit cell, atoms are displaced in the \( z \)-direction and the cell is elongated along the \( c \)-axis. The Curie temperature of \( \text{BaTiO}_3 \) is 120\(^0\)C. The space group is ‘pm3m’ with one formula unit per unit cell. The co-ordinates of the atoms in this structure are given as follows-

Ba at (0, 0, 0);

Ti at (1/2, 1/2, 1/2) and

3O at (1/2, 1/2, 0); (1/2, 0, 1/2); (0, 1/2, 1/2)

At high temperature it has the cubic perovskite structure in which Ba\(^{2+}\) ion are at the cube corner, Ti\(^{4+}\) ions at the body centre and O\(^{2-}\) ions are at the face center of the cube.

2.9 PROPERTIES OF FERROELECTRICS

Ferroelectric materials have a spontaneous electric polarization, they have been studied since over a century ago when large piezoelectric constant were observed. The ferroelectric have received a great amount of interests because of their various use in a range of applications, including transducers, actuators, capacitors and memory applications. A ferroelectric material possesses at least two equilibrium orientations of the spontaneous polarization vector in the absences of an external electric field, and the spontaneous polarization can be switched by an electric field. The polar character of the orientation state should represent an absolutely stable configuration in null field.
2.9.1 Hysteresis behavior of ferroelectrics

The polarization as a function of the applied field for a ferroelectric crystal is a hysteresis loop. At small values of the A. C. Electric field, the polarization increases linearly with the field amplitude. This corresponds to segment AB in this region; the field is not strong enough to switch domains with the unfavorable direction of polarization. As the field is increased to polarization of domains with an unfavorable direction of polarization will start to switch in the direction of the field, rapidly increasing the measured charge density (segment BC). Once all the domains are aligned (point C) the ferroelectricity again behaves linearly (segment CD). If the field strength starts to decrease some domains will switch back, but at zero field the polarization is non-zero (point E) as shown Fig 2.6. To reach a zero polarization state the field must be reversed (point F). Further increase of the field in the negative direction will cause a new alignment of dipoles and saturation (point G). The field strength is then reduced to zero and reversed to complete the cycle.

![Ferroelectric Hysteresis loop](image-url)
The value of polarization at zero fields (point E) is called the remanent polarization. The field necessary to bring the polarization to zero is called the coercive field. The spontaneous polarization is usually taken as the intercept of the polarization axis with the extrapolated linear segment CD [15]. If a low electric field is applied over a very long time period the polarization will eventually switch [16]. The ferroelectricity of a crystal disappears as it is heated up to Curie temperature Tc, above Tc the crystal is said to be paraelectric.

2.9.2 Piezoelectric effect

The piezoelectricity is the property of crystal to exhibit electric polarity when subjected to the stress. It is termed as a flow of charges in certain direction after application of pressure to the piezoelectric crystal. Basically the word ‘piezo’ means ‘pressure’ hence the original meaning of the word piezoelectricity implies ‘pressure electricity’. If the pressure is replaced by tension, the charge will flow in the opposite direction. Also if we apply an electric field to the crystal plate, it will get stretched and if we reverse the field direction the crystal will get compressed i.e. under an applied electric field, the cations are drawn in the direction of electric field, and the anions in the opposite direction, leading to the relative change in the inter-ionic distance and depending on the applied electric field contraction and expansion occurs. This is termed as the converse piezoelectric effect. Certain materials produce electric charges on the surface after application of mechanical stress. The charges induced are proportional to the mechanical stress. This is called the direct piezoelectric effect. The three characteristics possessed by ferroelectrics, namely the switchable spontaneous polarization, the polarization-structure relationship and the high dielectric permittivity these classes are technological important
class of materials. These materials are very promising for a variety of applications such as high permittivity capacitors, ferroelectric memories, pyroelectric sensors, piezoelectric sensors, piezoelectric transducers, electro-optic devices and positive temperature coefficient (PTC) and thermistor [17].

BaTiO$_3$ is one of the most studied and widely used piezoelectric materials. The reasons for this are - (1) It is chemically more stable than Rochelle salt. (2) It has a wider temperature range of operation. (3) Its coupling factor is higher than that of any previously known material, and (4) It has the obviously advantage of easy manufacture by ceramic technique [18].

The piezoelectric properties of ferroelectric ceramic BaTiO$_3$ can be affected by its own stoichiometry, microstructure and dopants entering into the A to B site in solid solution. The modified BaTiO$_3$ with dopant have been developed to stabilize the tetragonal phase over a wider temperature range and used as commercial piezoelectric material.

**2.10 APPLICATIONS OF FERROELECTRIC MATERIALS**

Ferroelectrics are the material that shows strong anomalies in many of their physical properties such as piezoelectricity, dielectric permittivity etc. Piezoelectricity is a phenomenon that allows ferroelectric materials to convert mechanical energy into electrical energy and vice-versa. The characteristics can be made use of in micro machines such as accelerometers, displacement transducers (sensors and actuators) of different kinds including those required for inkjet printers, VCR head positioning and
medical ultrasonic imaging probes. The other remarkable property of ferroelectric is their very high dielectric permittivity.

The high dielectric constant also makes them very important dynamic random access memory (DRAM) strong capacitors. Pressure and acceleration sensors are now commercially available. Electro-optic materials will become key components in the displays and optical communication systems in the future, for thermistor applications, semi conducting ferroelectric ceramics with a positive temperature coefficient (PTC) of resistivity due to junction effect has been developed from barium titanate based materials. In an addition, ferroelectric show electro-optic activity and nonlinear optical effects, making them useful in lasers, computer display, and image storage system and optical switches for integrated optical system.

**Ferroelectric Materials are also used in—**

i. **Capacitors** – A capacitor is an electronic component used for a number of electronic circuits. It consists of two plates with a dielectric material. Ferroelectric material is preferred for the manufacturing of a capacitor. Capacitors are generally formed by the Barium Titanate (BaTiO$_3$) material.

ii. **Storage memories** – SRAM and DRAM are volatile. EEPROM and CMOS require a battery backup and are very costly. Ferroelectric Random Access Memory is non-volatile as well as cheaper.

iii. **Waveguides** – A waveguide is a device which controls the propagation of light within the device. It carries the electromagnetic wave. The material used inside a waveguide is ferroelectric material.
iv. **Optical Memory Display** – Latest optical memory display technologies are derived using the ferroelectric material. PLZT is most preferred ferroelectric material for this purpose.

v. **Displacement Transducers** – The ferroelectric material is used in pressure sensors. These pressure sensors are also termed as displacement transducers.

### 2.11 MAGNETOELECTRIC (ME) COMPOSITES

Composite materials containing piezoelectric (ferroelectric) and piezomagnetic (ferrite) phases exhibit magneto electric effect (ME) [19]. These composites have ME property which is absent in their constituent phases. The ME effect couples two field effects: (1) magnetization due to application of electric field and (2) electric polarization due to the application of magnetic field. The magnetoelectric effect is due to the mechanical coupling between piezomagnetic (ferrite) and piezoelectric (ferroelectric) phases [20].

### 2.12 PROPERTIES OF ME COMPOSITES

The physical properties of a material that is formed two or more single phase compounds are determined by the properties of the constituents as well as by the interaction between them. The composite electro ceramics can be classified in to three categories. 1) Sum property 2) Combination property 3) Product Property

i. **Sum property** - Sum property is a weighted sum of the contribution from the constituent phases, the weight being determined by the volume fraction of these
phases in composites. Physical properties like density and resistivity are examples of sum properties.

\[ \text{phase 1: } A \rightarrow B_1 \cup A \rightarrow B^* \]

ii. **Combination Property** - Combination Property denote effects in which the amplitude is higher for the composite than for the end compounds

\[ \text{phase 1: } A \rightarrow B_1 / C_1 \cup A \rightarrow (B / C)^* \]

iii. **Product Property** - Product Property is a more interesting result of a composite structure; It refers to effects, which are present in the composite but in none of its end compounds

\[ \text{phase 1: } A \rightarrow B \cup \text{phase 2: } A \rightarrow C \]

The concept of “product property” in magnetoelectric composites was first proposed by the Philips Laboratory using BaTiO$_3$ - CoFe$_2$O$_4$ as a model system. The magnetoelectric coefficient in such composite material (dE/dH)$_H = 130$ mV/cm/Oe was reported to be superior to single-phase materials such as Cr$_2$O$_3$. There have been considerable studies on magnetoelectric composites thereafter. In addition to the above “one-step process” of unidirectional solidification, different methods have been used to synthesize the composite materials, i.e. sol gel, ceramic etc. [21].

**2.13 APPLICATION OF ME COMPOSITES**

During the last decades, piezoelectric materials made large progress in entering commercial mass markets throughout all branches, where piezoelectric are used as the
basic materials for actuators as well as sensors. Especially in transportation industry, they found numerous fields of applications. The development of piezoelectric actuators for fuel injection systems is a popular application of the near past, active noise and vibration reduction is a current activity, and the use of piezoelectric materials for energy harvesting in vibrating structures is one possible future trend. Recent developments in ceramic processing and possibilities in tailoring of special properties will be shown, as well as latest developments in manufacturing of piezoelectric actuator and sensor components.

Applications of piezoelectric materials have expanded into many fields since the discovery of the effect by the Curie brothers in 1880-1881. An overview on the manifold applications is given in [22, 23]. Lead-Zirconate –Titanate (PZT) ceramics, which were discovered in 1954 and replaced barium titanate ceramics in all fields of piezoelectric applications, became the material dominating the world market till today [24]. Piezoelectric devices fit into four general categories, depending of what type of physical effect is used:

i. **Generators and sensors**- Generators and sensors make use of the direct piezoelectric effect, means that mechanical energy is transformed into a dielectric displacement. This, in turn, is measurable as a charge or voltage signal between the metalized surfaces of the piezoelectric material.

ii. **Actuators**- Actuators work vice-versa when transforming electrical energy into mechanical by means of the inverse piezoelectric effect.

iii. **Transducers**- In transducers both effects are used within one and the same device. For all of these basic functionalities, different designs are available. A good overview on the different design, types and possibilities for applications are found [25].
In a ME effect energy of one form conversed into another form i.e. conversation of energy from magnetic to electric form and vice versa. The linear ME effect has positive or negative sign depending on the ME annealing conditions in the case of polycrystalline material [26]. If the magnetic and electric fields during annealing are parallel to each other the ME effect is positive. If the magnetic and electric fields are antiparallel then the effect is negative. This shows that material can be used in binary data storage devices. Such a memory will be an effective read only memory (ROM) [27]. The composite of piezoelectric and magnetostrictive materials, the magnetic field input is required to have two components d. c. bias and a. c. signals, either of the two can be detected by providing the other components of fixed magnitude in the input. Thus, such a composite can be used as a magnetic probe for detecting d. c. as well as a. c. fields. These composite can be used as a ME transducers in recording heads and electromagnetic pick-ups. The possible application of the ME material have been reviewed by Wood and Austin [28]. For such applications ME composite material are suitable because of their large ME coefficient compared to single phase ME materials, they are used for devices such as

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