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-Shikshanmaharshi Dr. Bapuji Salunkhe

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Department of Physics

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Research Project Title

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1621	Adake Girish Suresh	Dr. M. M. Karanjkar	Preparation of $Ni_{0.8}Cd_{0.2}Fe_2O_4$ by Sintering method
1622	Bhosale Komal Jotiram	Prof. S. V. Malgaonkar	Preparation of $ZnFe_2O_4$ by microwave auto combustion method
1623	Burud Priyarani Ravindra	Prof. S. V. Malgaonkar	Preparation of $Co_{0.1}Zn_{0.9}Fe_2O_4$ by microwave auto combustion method
1624	Chougale Pratiksha Pandit	Prof. S. V. Malgaonkar	Preparation of $Co_{0.2}Zn_{0.8}Fe_2O_4$ by microwave auto combustion method
1625	Chougale Sunil Ratnakar	Prof. S. V. Malgaonkar	Synthesis and characterization of CuO thin film by SILAR
1626	Devtale Rishikesh Tukaram	Prof. S. V. Malgaonkar	Synthesis and Characterization of MnO_2 thin film by CBD
1627	Ghosalkar Prnav Shankar	Prof. S. V. Malgaonkar	Synthesis and Characterization of CuO thin film by Hydrothermal Method
1628	Jadhav Gauri Govind	Prof. S. V. Malgaonkar	Preparation of $Co_{0.3}Zn_{0.7}Fe_2O_4$ by microwave auto combustion method
1629	Jagdale Manasi Khanderao	Dr. G. J. Navathe	MnO_2 thin film synthesis by electrodeposition method
1630	Kamble Prasad Vilas	Dr. G. J. Navathe	Preparation of Fe_2O_3 thin film by SILAR method
1631	Mane Snehal Narayan	Dr. G. J. Navathe	Synthesis and Characterization of $ZnFe_2O_4$ thin film by CBD
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1635	Pednekar Bhagyashri Mahadev	Dr. S. I. Inamdar	Preparation of MgZnFe ₂ O ₄ by Combustion Method
1636	Pradnyasagar Prajyot Sunilkumar	Dr. S. I. Inamdar	Preparation of MgZnFe ₂ O ₄ by Combustion Method
1637	Sawant Manisha Shivram	Dr. M. M. Karanjkar	Preparation of Ni _{0.6} Cd _{0.4} Fe ₂ O ₄ by Sintering Method
1638	Shaikh Sadiya Mustafa	Dr. M. M. Karanjkar	Preparation of Ni _{0.5} Cd _{0.5} Fe ₂ O ₄ by Sintering Method
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1640	Shinde Swapnil Sakharam	Dr. M. M. Karanjkar	Preparation of Ni _{0.6} Cd _{0.4} Fe ₂ O ₄ by Sintering Method
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Preparation of Nickel Cadmium Ferrite by sintering method

A Dissertation Report Submitted to

**Vivekanand College Kolhapur,
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**For the Partial Fulfilment of
Degree of Master of Science**

In

PHYSICS

Under the Faculty of Science

by

Miss. **Rutuja Shubhash shetti.**
B.Sc.

**Under the Guidance of
Dr. M. M. Karanjkar
M.Sc., Ph.D.**

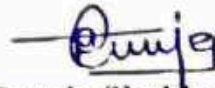
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2021-2022**

DECLARATION

I hereby declare that, the project entitled "preparation of Nickel Cadmium Ferrite by sintering method" completed and written by me has not previously formed the basis for the award of any Degree or Diploma or other similar title of this or any other University or examining body.

Place: Kolhapur

Date: 25/06/2022



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2021-2022

Handwritten signature and date: 25/06/22




CERTIFICATE

This is to certify that the project entitled "**preparation of Nickel Cadmium Ferrite by sintering method**" which is being submitted herewith for the award of the Degree of **Master of Science in Physics of Vivekanand College (Autonomous), KOLHAPUR**, is the result of the original project work completed by **Miss. Rutuja Shubhash Shetti** . under our supervision and guidance and to the best of our knowledge and belief the work embodied in this project has not formed earlier the basis for the award of any Degree or similar title of this or any other University or examining body.


Place: Kolhapur

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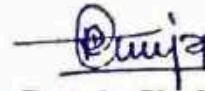
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1 Introduction

2 Synthesis

3 Literature Survey

4 Characterization

5 Result & conclusion

6 Reference



Chapter 1

Introduction

scientists & technology as difference in properties. In 1930 the first ferrite compounds are manufactured by yogoro kato & takeshi takei of the Tokyo institute of technology . The finding was somewhat accidental as a result of assistant had a mistake who was work for a group as preparing the sample of hexagonal lanthanum ferrite & use as a semiconductor material. On finding that it was a magnetic material & verifying its X-ray crystallography. They passed it to research group of magnetism. Barium hexaferrite both have low raw material price & high coercivity. In permanent magnets increasing the use of low cost & good performance .

A ferrite made by firing & mixing large proportion of iron oxide & combine with one or more additional metallic elements with a small proportions, for ex. Barium manganese, nickel & zinc they are insulators meaning that they are electrically non- conductive & it can simply be magnetized or attracted to magnet it is ferrimagnetic. Ferrites are the part of ceramic magnetic oxides materials in which containing electrical insulator and magnetic conductor with numerous field in large number of applications. Ferrimagnetic material proposed by L. Neel & ferrites are one of the class of ferrimagnetic material that is metal oxide (MO) & iron oxide(Fe_2O_3) of nickel, cobalt ferrite material are add up to by divalent metal ions. The subject of interest in the synthesis methods & synthesis parameters for the scientists and technology as differences in properties.



The study of ferrite materials being since last seven to eight decades & its starts in 1949. To scatter energy the eddy currents are generated in ferrimagnetic material when it engross in alternating magnetic field. glaze of the ferromagnetic core which can be keep down the losses to keep under control the eddy current path. Since, eddy current losses in ferromagnetic material are inversely proportional to the resistivity they can be keep down by using high resistivity of magnetic material. Cubic spinel structure, cubic garnet structure & hexagonal structure are all these types of ferrites & they are occupy distinct chemical formula & they are equally important for their applications. Ferrites are classified into two types that is soft ferrites & hard ferrites & they are classified as they are controlled by their properties. That is high permeability, dielectric losses, high saturation magnetization & low eddy current, high electrical resistivity. These properties are useful various applications for example memory chips, automobile etc because of their eddy current are very low & dielectric losses they are used at high frequency applications that is RF transformers , inductors. For many factors the properties of ferrites are sensitive such as preparative conditions, method of preparation & nature of substituents ferrites are usually constructed by ceramic technology using high purity metal oxide.

Composition & properties

Many ferrites are spinel & its formula is AB_2O_4 where A & B are various metal ions, usually iron Fe. Spinel ferrites usually made up of cubic close packed oxides in which A cations possess one eighth of tetrahedral holes & B actions possess half of octahedral holes. The inverse spinel structure can able to be done.



In which one eighth of the tetrahedral holes are occupied by B cation then one fourth of the octahedral sites are occupied by A cation & the another one fourth occupied by B cation. This is called the inverse spinel structure. Its also possible to have mixed structure spinel ferrite & its formula is

represents degree of inversion. $ZnFe_2O_4$ is a magnetic material with the formula $ZnFe_2O_4$ & Fe^{3+} occupying the octahedral sites & Zn occupy the tetrahedral sites & this is the example of normal structure spinel ferrite.

Crystal chemistry of spinel ferrites -

From the mineral spinel $MgAl_2O_4$ derived the structure of spinel ferrites there are 8 molecules per unit cell in that the structure is complex & where A is divalent metal ion. From a face centered cubic lattice the 32 large oxygen ions in which two kinds of interstitial sites are present. The smaller metal ions occupying the interstitial spaces. These spaces are of two types. One is tetrahedral A site whose corners are occupied by oxygen ions at the center of tetrahedron. There are 64 tetrahedral sites which is surrounded by 4 oxygen ions. & the other is octahedral site. It occupy the corners of an octahedral in which oxygen ions around it. There are 32 octahedral sites in which surrounded by 6 oxygen ions. A & B sites are the crystallographic environments therefore they are distinctly different. In metal ions only one eighth of the A sites & one half of the B sites are occupied. $MgFe_2O_4$ in this chemical formula. This structure is normally referred to as the normal spinel structure only if M^{2+} are in A sites & Fe^{3+} in B sites. When the M^{2+} divalent metallic ion occupies the B site & the Fe^{3+} ions are equally divided into A & B sites then this configuration is called inverse spinel. $NiFe_2O_4$ is inverse spinel & $ZnFe_2O_4$ is normal spinel.

Intermediate structures can exist had shown by X- ray neutron diffraction therefore the normal & inverse structure is to be regarded as extreme cases. In some ferrites the distribution of the divalent ions on A & B sites can be altered by heat treatment. It depends on whether material is quenched from annealed or a high temperature.



The cation distribution there is various factors are determining including valency of the cation size of the cation & the oxygen parameters of the anions. Tetrahedral sites as this is favored by polarized effect occupies by large divalent ions. Intermediate O^{2-} ion will become a polarized towards B sites when the A site ions have a lower valency & B sites ions have a higher valency. Thus polarization has normal spinel configuration. It has been proved that the normal structure has the lowest energy when $u > 0.379$, whereas, the inverse structure has the lowest lattice energy for u parameter $u < 0.379$.

In spinel ferrites these factors alone cannot give consistent picture of the observed cation distribution. $ZnFe_2O_4$ crystallizes in normal spinel structure while $CoFe_2O_4$ is an inverse spinel, although Co^{2+} & Zn^{2+} have almost same ionic radii. In order to explain the site favour of transition metal ions in oxides, two theories have been proposed, which depend on concept of chemical bonding in oxides. The first theory is a molecular orbital theory which is based on the covalent bonding between oxygen & transition metal atoms & second one is based on crystal field theory which is purely ionic type of bonding.



Magnetic properties of ferrites

Ferrites are known that in ferrimagnetic material. To governing the magnetic properties of these ferrites the various features that are the nature of cations, preparative procedure of heat treatment, site preference energy of cations & Madelung energy. According to Neel in 1948, prior recognized structure in distinctly different from ferrites display a magnetic structure. He also described that the spontaneous magnetisation of ferrites are on the basis of Heisenberg's exchange forces. As stated by this theory, spin angular momentum of the exchange energy between two adjacent atoms is given by $E_{ex} = -2J_{ex}S_i \cdot S_j$ where E_{ex} is the exchange energy S_i & S_j are the total spins of adjacent atoms & J_{ex} is the exchange integral which exhibit the possibility of exchange of electron. There are three kinds of magnetic influence on each other are possible in ferrites between the magnetic ions which are hold in two crystallographically different lattice sites. Through the in between O^{2-} ions these interactions are possible by super exchange mechanism. A-A interactions, B-B interactions & A-B interactions These are three possible interactions. On A site & B site the exchange force acting between these ions & is governed by equation. From the value of J_{ex} finding the interactions is ferri, ferri, antiferro, these interactions energies are negative for ferrites & it has been promoted experimentally & hence produce an anti parallel orientation. However, the magnitude of A site & B sites magnetization are not similar & this will induce a resultant net magnetization in ferrites. In general, the magnitude of interaction energy between two magnetic ions are controlled by the distance from these ions to the oxygen ions. An angle of 180° will give increase to the highest exchange energy & energy decrease rapidly with increasing distance. It is Based on the values of distance & the angle θ & it concluded that of the three interactions that is greatest magnitude in A-B interaction, weakest is the A-A interaction, thus forcible is only A-B interaction. In ferrites the spin of the A & B site ions in ferrites will be oppositely magnetized in A-B sub lattices with distinct between those of a & B site ions equal to the resultant magnetic moment. In general, the value of saturation magnetic moment for A lattice M_A is less than B lattice. M_B So that the resultant saturation magnetization M_S

Is written as $M_S = M_B - M_A$



Classification of magnetic materials

The classification of magnetic materials is as follows-

Diamagnetic materials-

These materials do not contain any unpaired electrons. These are repelled by magnetic field and hence do not get magnetized. They produce magnetic field which opposes the external magnetic field. In this process of magnetization, the atoms of the diamagnetic materials behave as if magnetic moments. The magnetic spinning electrons produce magnetic field in opposite direction of applied field. For such materials the value of susceptibility is independent of temperature and is negative. Examples of diamagnetic materials are copper, water, vacuum, salt, etc.

Paramagnetic materials-

In paramagnetic materials there are unpaired electrons. The unpaired electrons are free to align with magnetic moment. In such materials, field in magnetic moments aligns in the direction of the applied field. Examples of the paramagnetic materials are magnesium, aluminum, etc.

Magnetic substances are those which are capable of retaining the magnetic field even after removal of the external field. In such materials, the magnetic moments align in the direction of the applied field. Examples of the magnetic materials are iron, nickel, cobalt, etc. These materials are called ferromagnetic. They are characterized by high magnetic susceptibility and are dependent on temperature. Examples of ferromagnetic materials are iron, nickel, cobalt, etc.



Ferrimagnetic material -

It is one that has population of atoms with distinct magnetic moments as in antiferromagnetic however in Ferrimagnetic materials the opposing moments are not equal & a spontaneous magnetization remains. This happens when the populations consist of different materials or ions. Ferrimagnetism is shown by ferrites & magnetic garnets. The oldest known substance magnetite is a Ferrimagnet. It was originally distributed as a ferromagnetic before Neel's discovery of ferrimagnetism & anti ferromagnetic in 1948.

Ferromagnetic material -

In this certain material forms permanent magnets or is attracted to magnets. Ferrimagnetism is the strongest type & is responsible for the common aspect of magnetism. But the forces are usually so not strong that by sensitive instruments they can be identified only in a laboratory. An everyday example of ferromagnetism is refrigerator magnets used to grip notes on a refrigerator door. By an external magnetic field & remain magnetized permanent magnets that can be magnetized later the external field is removed. Only rare substances are ferromagnetic. The usual ones are iron, nickel, cobalt & most of their alloys. It is mainly important in industries & modern technology.

Anti ferromagnetic material-

Anti ferromagnetic materials display a minimum in the graph of susceptibility versus temperature. On the curve the maximum is called the Curie point. The susceptibility is temperature dependent for antiferromagnetic materials. Anti ferromagnetic materials follow Curie-Weiss law & become paramagnetic above Neel temperature. By the application of a sufficiently high magnetic field parallel to the spin axis, some anti ferromagnetic materials can be made ferromagnetic.



Neels theory

Neels theory is based on-

- 1) tetrahedral A site & octahedral B site are sub lattices of the Ferrimagnetic crystal lattice in a spinel structure.
- 2) A-B, A-A, & B-B are three magnetic interactions A-B interaction & B-A interaction are identical & the predominant over B-B interaction & A-A interaction. Therefore, the magnetic moment is difference between two average sub- lattice magnetic moment.
- 3) there exist magnetic ion in material in which fraction λ appears on tetrahedral A site & remaining fraction μ appears on B site & formula is given by

$$\lambda + \mu = 1.$$

- 4) due to magnetic interactions from Weiss molecular point of view with in the material. The magnetic fields is written as.

$$H = H_0 + H_M$$

H_0 is externally applied fie

H_M Is internal or molecular field

Neels theory explain the magnetic properties of several spinel. Neels determine the distribution among tetrahedral A site & octahedral B site from knowledge of resultant saturation magnetization of $0 k$ & it is given by

To a ferromagnetic material applying internal Weiss molecular field then we get

$$H_A = H_{AA} + H_{AB}$$

$$H_B = H_{BB} + H_{BA}$$



Electrical properties of spinel ferrites

Spinel ferrites are important on conventional magnetic materials therefore their wide variety of applications. These materials have low electrical conductivities than other magnetic materials hence they find wide used at microwave frequency. In general, spinel ferrites are semiconductors with their conductivity lying in between 10^2 & 10^{-11} $\text{ohm}^{-1} \text{cm}^{-1}$ presence of Fe^{2+} & metal ions (M^{3+}) in conductivity shows Fe^{2+} results in n- type behavior & of M^{3+} in p- type behavior. The conductivity arises causes the mobility of the extra electron or the positive hole through the crystal lattice.

Classification of ferrites

Ferrites are classified according to magnetic properties. Ferrites classified as soft & hard which based on their low or high coercivity of their magnetism respective.

Soft ferrites-

it is in a cubic crystal structure and ferromagnetic materials. Ferrites are used in electromagnetic cores contains nickel, zinc or manganese compound. Soft magnetic material can be easily magnetized or demagnetized. These ferrites have a low coercivity therefore it is called as soft ferrites. The low coercivity means easily reverse direction of materials magnetization without dissipating much energy while, high resistance prevent eddy current, energy losses of another source because at high frequencies comparatively low losses of them.



They are used in cores of RF transformers & switched mode power supplies & inductors in used & loop stick antennas used in AM radios. And it is also used in telephone signals transmitters and also receivers. To permit miniaturization for such type of applications the driving force to rise frequency.

Soft ferrites that commonly used are 1)

Manganese zinc ferrites -

its formula is given by. $Mn_a(Zn_{1-a})Fe_2O_4$ has higher saturation induction & permeability than $NiZn$.

2) Nickel - zinc ferrites-

Its formula is given by. $Ni_aZn_{1-a}Fe_2O_4$ $NiZn$ have higher resistivity than $MnZn$ & they are suitable for frequencies above 1 MHz.

Hard ferrites -

Which have a high coercivity therefore it has a permanent magnet that's why it is made of hard ferrites. using iron oxide & barium or strontium carbonate in the producing of hard ferrites. & the high coercivity of hard ferrites that means the material are hardly becoming demagnetized & it is a necessary characteristic for permanent magnet & it is also have high permeability. These used in refrigerator magnet. The maximum magnetic strength H is 30 to 160 kiloampere & magnetic field is 0.35 Tesla.



Types of ferrites

Ferrites are classified into 4 groups namely spinel, Garnet, ortho, hexagonal ferrites.

1) Spinel ferrites -

Spinel ferrites has a chemical formula which is MFe_2O_4 & where M is a divalent metal ions. Tetrahedral (A) & octahedral (B) these two interstitial sites are the crystal structure of spinel ferrites. In tetrahedral A site & octahedral B site can be accommodate a variety of cations & enabling wide variation in the properties of ferrites. M is a divalent metal ions & it is replace by another divalent metal ions & similarly Fe^{3+} ions can replace by another trivalent ions such as Ga^{3+} , Al^{3+} , Cr^{3+} ions & we can also combine the divalent & trivalent ions & replace it.

2) Garnet -

Ferrimagnetic garnet has a chemical formula which is $Me_3Fe_5O_{12}$ where Me stands for trivalent ions & it contains eight molecules of $Me_3Fe_5O_{12}$ & cubic unit cell. In ratio 3:2 the Fe^{3+} ions are distributed over two sites that is tetrahedral & octahedral sites.

3) Ortho - ferrites

Ortho ferrites have chemical formula $MeFe_5O_3$ & Me stands for a large trivalent metal ions. It shows a weak ferromagnetism. In the Fe ion the direction of spin orientation in $HOFeO_3$ & $ErFeO_3$ has been determined experimentally at room temperature & it is parallel to the (100) axis on decreasing the temperature, the spin axis rotates, the direction at 1.25 k is (001) for $HOFeO_3$ & (110) for $ErFeO_3$.



4) Hexagonal ferrites -

There are number of ferrites which crystallize in hexagonal structure. These ferrites are sub classified into 5 compounds namely M, W, Y, Z & U compounds. These compounds have

chemical formula MeFe_2O_4 . Where Me stands for divalent ion of large ionic radius. Some compounds are trivalent Me (e.g. Al, Ga, Cr, Fe) are also known. In these for charge compensation one iron per formula unit is present as Fe^{3+} to allow.

5) Super paramagnetism-

It is unique class of magnetic materials. Their large magnetic susceptibility & are saturated at moderate magnetic fields thus super paramagnetism is exhibited by single domain particles that behaves like ordinary ferromagnetic materials below Curie temperature. They display no hysteresis above Curie temperature super paramagnetic materials behave like ordinary paramagnetic materials. Single domain having all atomic moments ordered represented by the whole particle. The particle shape detected the magnetization of the particles is no larger fixed in the direction. Application of magnetic field to ensembles of such thermally demagnetized particles. In results larger magnetic response than the case of paramagnet.

6) Spin canting-

In deciding the magnetic properties the large surface area of magnetic nano particles is important. In the interior normal co- ordination breaks down at the surface to have very different form (10). As compared to bulk spin structure of such nano particles is very complicated. For investigate spin canting at high magnetic field mossbauer spectroscopic technique can be used. The exchange bonds for the surface atom become dominant this is the main effect of finite size on magnetic nano particle. Mostly anti ferromagnetic are the exchange interactions. It depends on bond angle & bond length between intervening anions & the metal cations. In case of magnetic nano particles, some of the exchange bonds are made from the surface, there can be frustration & canting spin order.

Classification of spinel ferrite

The cations are distributed in tetrahedral (A) & octahedral (B) site depending on their electronic configuration, their ionic radii & the electrostatic energy in the lattice. Spinel ferrites classified as normal, random & inverse spinel ferrites.



Normal spinel ferrites-

The divalent metal ions are totally occupied at tetrahedral site & trivalent ferric ions are totally occupied at octahedral B site in this type of spinel ferrite. For spinel ferrite the distribution of cations represented by MFe_2O_4 can be written as. $[M]^A[Fe]^B O_4$

The best example of spinel ferrite is zinc ferrite & cadmium ferrite because zinc ions & cadmium ions occupy tetrahedral A site

Inverse spinel ferrite-

The divalent metal ions are totally occupied at tetrahedral A site & the trivalent ferric ions are totally occupied at octahedral B site in this type of spinel ferrite. For spinel ferrite the distribution of cations can be represented by MFe_2O_4 can be written as. $[Fe]^A[MFe]^B O_4$
Cobalt ferrite & nickel ferrite are the example of inverse spinel ferrite.

Random spinel ferrite-

In this type of spinel ferrite, the trivalent ferric ions & divalent metal ions occupy both tetrahedral & octahedral site. In this type of spinel ferrite the distribution of cations can be represented by $[M_{1-x}Fe_{2-x}M_x]O_4$

Where x is distribution parameters vary between 0 & 1. The example of random spinel ferrite are magnesium ferrite & copper ferrite.

At $X=0$, we get normal spinel, structure

At $X=1$, we get inverse spinel structure

At $X=0 < X < 1$ we get random spinel structure.



As field decreases the magnetization also decreases & brought to negative value of magnetization. If the field is increasing then the magnetization start increasing in reverse direction. The permeability of magnetic material is given by the slope of hysteresis curve which near to origin μ_0 & it is depends on the state of magnetization of the sample. μ_m Is maximum value of permeability. The differential permeability is known as the slope of magnetization curve at the point of intersect.

Domains-

The concept of domain was introduced by Weiss. On the basis of magnetic domains. The magnetic behavior can be explained they exist several magnetic domains which are separated by domain walls, inside a magnetic material. The magnetization depends on the type of magnetic material & temperature, each domain is spontaneously magnetized. Neighbouring domains magnetization is not parallel. The overall magnetization curve given by vector sum of the magnetization of all domains. This is zero in case of a material is demagnetized. Domain structure is a natural consequence of the various contribution to the energy which is 1) anisotropy 2) exchange 3) magnetism. If this material consisting a single domain it will shows result of free poles formed on the surface because of a high value of a magnetostatic energy. When the domains magnetized in opposite direction then the magnetostatic energy is reduced roughly by half. It creates addition wall energy because of each subdivision lowers the magnetostatic energy. The wall energy increases from that on the opposite side of the wall & the magnetization is in anti parallel directions.

Which requirement of exchange forces in case of ferromagnetic materials prefer parallel orientation therefore the subdivision process continues only as long lowering of magnetostatic energy is larger than increase in the wall energy.



Single domain particles-

A function of the volume of domain which is the total magnetization energy, while the total wall energy is directly proportional to the domain wall area. The energy of a wall dividing the particles into two parts of Domain may be larger than the reduction in magneto static energy, if the volume is small enough. In such a case single domain has stable. Magnetization changes can be brought about by rotation of magnetic vector against the magneto- crystalline anisotropy & demagnetizing fields, in a single- domain - particles.

Uses-

Ferrite cores are used in transformers, electronic inductors & electromagnet in which high electrical resistance of ferrites lead to low eddy current losses. They are commonly used in lump in a computer cable which is called as ferrite bead. Which helps to prevent the electrical noise in high frequency from entering & exiting the equipment. In the residual magnetic fields of hard ferrite cores early computer memories stored data, which are manufactured into arrays of core memory. Ferrite powders are also used in coating of the recording tapes in magnetic & they are used in stealth aircraft used as components of radar absorbing materials or coating by using ferrite particles. & it is used for electromagnetic measurement in the absorption tiles lining the rooms. Ferrite magnet are used in most common radio magnet & also used in loudspeakers. Ferrite nano particles are exhibit super paramagnetic particles.



Structure

Cubic symmetry contains eight molecules of MeFe_2O_4 where ($\text{Me} = \text{Cd}^{2+}, \text{Ni}^{2+}, \text{Cu}^{2+}, \text{Zn}^{2+}, \text{Co}^{2+}$) it has the smallest cell of the spinel lattice. Form an Fcc lattice by the relatively large oxygen ions. Two kinds of interstitial sites occur in the cubic close packed structure which is tetrahedral A & octahedral B site that are surrounded by 4 & 6 oxygen ions respectively. 64 tetrahedral & 32 octahedral site are present of which only 8 & 16 respectively are occupied by metal ions as above mentioned cubic unit cell.

The interstitial is in the center of a tetrahedron formed by four lattice atoms in a tetrahedral site. Three atoms are touching each other in the plane & the fourth atom sits in the symmetrical position on top. Tetrahedral site offers space for an interstitial atom & again tetrahedral site has defined geometry. Octahedron formed by an octahedral position for an atom is the spaces in interstices between six regular atoms. In these four atoms are positioned in a plane & other two are in symmetrical position either above or below. All sphere can be considered as hard & touching each other.

The primitive tetrahedral unit cell consists of two molecular MeFe_2O_4 units of spinel ferrite & it is represented by two octants. Four primitive units cells combining then form the conventional cubic unit cell of spinel.

Cubic unit cell consisting 32 anions & 24 cations & total is 56 atoms & their are $Z=8$ formula units per cubic unit cell. In the cubic unit cell there are 96 interstices between the anion. 64 tetrahedral interstices (8a, 8b, 48f) only 24 are occupied by cations that exist between the anions, only 8 are occupied by cations in spinel ferrites. Half of the 32 octahedral interstices from the remaining 16 cations. Tetrahedral & octahedral sites locations is always same & do not depend on nature of the constituent cations & the position of anions is depends on the size of A & B cations. & the anions are arranged in pseudo cubic close packed spatial arrangements. But some spinel possess ideal cubic close packed anion sub lattices. As the parameter a of the spinel lattice is large.



Chapter 2

Synthesis

Different methods of preparation of ferrites:

In this chapter, we are going through a variety of synthesis techniques for the ferrites or magnetic materials. The synthesis technique plays a very important role. They are very important for controlling the size and surface area of materials. The synthesis of variety and different ferrites or nanoparticles of magnetic material has been reported using many chemical methods which are sol-gel method, co-precipitation, microwave method, ceramic method, citrate precursor method, hydrothermal method, ball milling method, solid-state reaction method, etc. these are the methods for synthesis which will be discussed in the following subsections.

Ferrites are produced by heating a mixture of finely powdered precursors & calcination of carbonates occurs during these heating process is MCO_3

The oxide of barium & strontium are supplied their carbonates $BaCO_3$ or $CoCO_3$ then the resulting mixture of oxides are undergoes method of sintering. This method is high temperature process as similar to firing of ceramic ware. The product is cooled & milled to particles smaller than. Small enough each particles consisting single magnetic domain then the powder mould into a shape, dried & resintered. In external magnetic field the shaping may be performed, in order to achieve a preferred orientation of the particles that is anisotropy with dry pressing small & geometrically easy shapes may be produced. However, in a such a process Small particles may together & lead to comparison between poorer magnetic properties & wet pressing process without remilling direct sintering & calcination as possible as well milled & pressed of electromagnets. The vary strongly between the sintered product & precursor. During sintering to allow efficient staking product in the furnace & to prevent parts sticking together .by using ceramic powder separator sheets many manufactur separate it & these sheets are available in many materials such as zirconia, alumina, magnesia. particle size to the ware being sintered. By matching the material, contamination & surface



Damage can be reduced while maximizing furnace loading. There are various methods for preparation of spinel forms thin & thick films the oldest method is ceramic method of preparation of spinels. Oxides & carbonate are precursor compounds of cations in the desired spinel. But this method produce distribution in particle size become extremely wide & can not produce fine particles & extended milling introduces significant quantities of undesired impurities. The lack of homogeneity of material prepared this is major drawback found in this method.

To complete solid state reaction leads to drastic decrease in surface area of the resulting material by sintering again the high temperature (1200k) is required.

For the creation of homogeneous catalyst components or for the moulding of precursors with a definite stoichiometry, co- precipitation is a very suitable method for the creation of homogenous catalyst components.. And it is easily converted into active catalyst. This method is based on stoichiometric mixture of aqueous solution such as nitrates , chloride, sulphate of trivalent & divalent ions in the concentration required for the spinel composition. In the form of hydroxides their forming simultaneously precipitation by. NaOH Or NH₄OH. To form the oxides ,This is followed by wading , filtering & calcination of the product. By altering the PH of solution ,temperature & the nature of the reagents, morphology, texture, structure & size of the particles can be accurately controlled. At high impurity spinel particles with the narrow size distribution in the ranges 5-500 nm may be obtained by this method. Co- precipitation method can be prepared by **bronsted acid** sites in different cataionic environment with also lewis sites, it makes catalyst active & effective for many organic transformations. Under hydrothermal condition the method of precipitation of solution is of current interest & during the reaction at relatively low temperature attractive for the direct synthesis of crystalline ceramic particles. It has been reported that by controlling the hydrothermal conditions , uniform ferrite particles with controlled size , shape & stoichiometry can be produced. At temperature between the boiling & critical points of water (100-374°C) & at eleva te pressure up to (15Mpa),in general hydrothermal reaction are carried out. By this method the powder synthesized has excellent homogeneity & particle uniformity.



In the hydrothermal treatment crystalline powders are directly prepared. In this solution the metal sulphate are dissolved in to distilled water & to control the PH the aqueous amount of ammonia. Was poured in to this solution. This mixed solution at desired concentration has placed in a stainless steel vessel after sealing itplacrd in to thermostatted oven heated for 550 h at 150 - 240°C with constant rotation

This precipitated solid product has separated by washing, centrifuging & then dried in an oven. Synthesis of fine oxide powders with well controlled Particle size & morphology can be possible by hydrothermal method.

Sol gel method is receiving much attention because of it can be applied wide variety of materials they offers possibility of controlling not only the size but also their shape and distribution of particles. By using this technique ,A broad range of spinel with any desired shape can be prepared. For preparation of Sol, there is a dispersed phase in liquid ,dispersion of solid. Mixing concentrated solutions containing the cations of interest with organic solvent as dispersion medium, by this method Sol is prepared. Leading to the formation of a gel, the Sol is destabilized by adding water. By high pressure heating this is transferred to the solid phase and by the liquid containing in the gel is transformed into supercritical vapours. For the preparation of fine spinel particles combustion synthesis is Novel method making use of exothermic redox reaction between tetraformal triazine or oxalic acid dihydrazine metal nitrate. In this process in a pyrex dish stoichiometric ratio of nitrates is dissolved in minimum amount of water. In a muffle furnace it is heated at 350°C. To obtain good combustion a heating rate of 75°C/min is used. This method can be used for NiZn & co- spinels. In a spray drying technique of preparation of spinel involves precipitation by solvent evaporation from a concentrated solutions .



Sintering method:

In many areas sintering technology is an old and extensive in ceramic fabrication it has been used. In recent years the development of different sintering technologies such as flash sintering microwave sintering and so on...

The material is forced to equilibrium concentration & developed the microstructure in this sintering process & these method have two steps-

Pre- sintering & final sintering-

Swallow & Jordan was explained the purpose of pre- sintering.

In the final sintering process reducing the evolution gas to decompose carbonates and higher oxides

To help the homogenizing the material

Effect of variation in the compositions of the raw materials has reduced.

During the final firing, to control shrinkage of the material

During sintering grain growth & densification occurs at the same time and increase the variety of microstructures. For densification sintering reactivity is important and the reactivity means for sintering the amount of energy is available. For the process to be proceeding it must be sufficiently high that is particle size must be small of particles. It is equally important that material must be good sinterability.

At high pressure concentrated solution is atomized into a fine droplets of 100- 500 μm diameter, to ensure that the particles remains small. By an upward stream of hot gas the solvent is evaporated. Some methods are currently in development such as efficient way to control the Composition, texture, homogeneity, & structural properties of spinel particles. The aqueous concentrated solutions is atomized into fine droplets & it is frozen rapidly by blowing into low temperature bath such as liquid nitrogen & ice- acetone. In vacuum droplets are dried to produce fine powders anhydrous salts are calcined.

Sonochemical method can also applied for the spinel.



Ceramic method-

This method is used in the commercial production of ferrites, which has chemical knowledge. When all compounds are solid then it is known as ceramic method. By the final product are mixed together & ground to fine powder in an mortar, the high purity oxides material in the proportions required. In a stainless steel balls, the mixing is often performed by wet milling for a long period. To bring about partial reaction, after milling the mixture is dried & presintered for an extended period at 850°C for 24 hours. by applying the pressure of 5 - 10 tones then formation of pellets using binder if necessary. At elevated temperature pellets are sintered depending on the compositions. To get desired phase the sample is cooled at room temperature.

Precursor method-

There is possibility of losing elements due to volatile nature of elements, at the time of sintering, at elevated temperature for extended hours by using precursor method this can be controlled. For single phase spinel this method is successfully used by number of workers. At relatively low temperature this method involves heat treatment & short duration. In this method during the initial stage of preparation that gives rise to homogeneous mixture. In required stoichiometry proportions the starting material weighed accurately. Then mixture is mixed with distilled water and solution is heated at 40- 50 ° c for half an hour to obtain homogeneous, brown colour transparent glassy material to obtain spinel ferrites the dried citrate mixture is calcinated for at about 1000°C for 1-2 hours.

Sol - gel method -

It can be prepared pure, nano crystalline and homogeneous ferrites by using this method. At relatively low temperature as compared to other the powder preparations are as following step

- 1) starting material are dissolved in water with constant stirring and are weighed accurately.
- 2) to bring about gel formation the solution is refluxed at 100°C for long time and it is dried in furnace.
- 3) pelletization of sample is done and pellets are sintered at certain temperature.



Synthesis of NiCd-Ferrite by Sintering Method:

A Ni Cd-Ferrite material were obtained by the sintering method which is a ceramic method. One specimen of the composition $Ni_xCd_{1-x}Fe_2O_3$ with $X=0.4$ where,

Molecular weight are $(NiCo_3)_{0.4} = 1.1869$ gm

$(CdCo_3)_{0.6} = 2.5857$ gm

$Fe_2O_3 = 3.9925$ gm

NiCdfe2o4 nanoparticles were prepared by sintering method from a mixture of $NiCo_3$, $CdCo_3$ & Fe_2O_3 powders. High purity reagent powder Ni, Cd & Fe_2O_3 were mixed. According to their molecular weight & mixed for 6 hours by using mortar & pestle. Then the mixture decomposed by heat . we use muffle furnace . the given sample was presintered at $450^\circ C$ for 6 hours to form ferrite through chemical reaction. The pre sintered material was ball - milled again for 6 hours while, adding few drops of a acetone as a binder to making a fine powder. Then for final sintering heated at $600^\circ C$ for 6 hours.



Chapter 3

Review of literature

- 1) Structural and magnetic properties of NiCd ferrites :
R. Suresh , P. Moganavally , M . Deepa .

Ferrite compositions of NiCdFe₂O₄ were synthesized at low temperature through the chemical co-precipitation method .In presence of spinel structure of ferrites the size of particle is 15-23 nm. Magnetic measurements of the samples carried out by means of vibrating sample magnetometer and these studies reveal addition of Cd concentration affects magnetic properties.

- 2) Characterization of Cadmium substituted Nickel ferrites prepared using auto-combustion method :

Manojit De , Aniruddha Mukherjee , Hari S. Tiwari

Here, We have investigated synthesis of (NiFe₂O₄) and cadmium substituted nickel ferrite powder with particle size in nanometer range using auto-combustion technique and sinterability. Also observed that estimated bulk densities of materials decreases with increase in Cd concentration while they increase with the rise of sintering temperature . XRD patterns shows information of single phase compound of cubic spinel structure , no other reflections are present all composition sintered .

- 3) Synthesis and characterization of Cadmium doped Nickel ferrite nanoparticles and it's optical properties :

Sandhya Singh , Gurav Hitkari , G. Pandey

Cd- Ni ferrites nanoparticles with a composition of NiCdFe₂O₄ have been successfully prepared via simple coprecipitation technique using NaOH solution is used as precipitating agent . They found to be of almost spherical shape moderate particle size with a narrow size distribution of nickel ferrites particles by Sol gel method. & irregular shape & very small



particles size with the wide size distribution by co - precipitation method. Sol gel method exhibits more impurity of nickel ferrite particles. Found to be high saturation magnetization & hysteresis. With small saturation magnetization co - precipitation derived nickel ferrite particles annealed at 400°C exhibited super paramagnetic nature. In both methods saturation magnetization increased with annealing temperature. Co - precipitation derived particles became Ferrimagnetic at the annealing temperature of 600°C.

4) Physical , Elastic and thermal characterization of Ni-Cd Nanoferrites :

Asghari Maqsood ,M. Arshad

By using combination of co-precipitation and mechanical alloying has been prepared nickel cadmium ferrite mechanical milling of hydroxide precursor this work has shown that nickel ferrite phase can be formed directly.during the washing with deionized water the XRD study showed that sodium chloride can be efficiently removed.after a simple washing process ultrafine NiCdFe₂O₄ particles were obtained. The ultrafine powder possessed super paramagnetic behavior & good soft magnetic properties

5) Effects of Ni²⁺ substitution on Structural , Magnetic and Electrical properties of Cd spinel ferrite Nanoparticle :

A. K. Nandanwar , N. S. Masharam , V.B. Korde , D . S. Choudhary

By sol-gel auto combustion method the magnetic Ni²⁺ nano particles are make ready. In order to make ready ferrofluid distilled water was useful as a based fluid. Construct the structure & morphology of the nano particles by using x- ray diffraction (XRD), Scanning Electron microscopy (SEM) these characteristics the single phase spinel structure with crystallite of 27 nm these results display by XRD. Electrical conductivity of ferrofluids was measured by aqua Pro Electrical Conductivity (EC) meter by spevific gravity bottle density of ferrofluids was measured. It is measured as a function of volume fraction it is notice that, density of ferrofluids rises with rising volume fraction and electrical conductivity.

6) Chemical synthesis and studies on Structural and magnetic properties of fine grained Ni-Cd Ferrite :

M.B. Shelar , P. A. Jadhav , D. R. Patil , B. K. Chougule .

Successfully synthesized by self propagating auto combustion method using stable ferric & nickel salts as precursors & glycine as fuel of magnetic nano particles of Nickel Cadmium ferrites (NiCdFe₂O₄) & Found the structure & morphology of the nano particles by using x-



ray diffraction (XRD), Scanning Electron microscopy (SEM), vibrating sample magnetometer (VSM). This study revealed that by using higher dose of fuel, it was possible to produced larger size of nano particles with lower saturation magnetization.

7) Synthesis of NiFe₂O₄ nanoparticles for energy and environment applications Ying Zhang¹, Gaurab Rimal², Jinke Tang² and Qilin Dai¹

By the organic passivating ligand ratios, reaction temperatures, NFO nano particles growth is managed. NFO nanoparticles size are 25 nm to 160 nm. By collecton related mechanism, leading to tunnable magnetic properties & concentration cell device experiment has the NFO growth mechanism is managed.

8) Preparation & characterization of nickel ferrite nano particles via co - precipitation method Suresh sagadevan, zaira zaman chawdhury, Rahman R. Rafique

Using co - precipitation method nickel ferrite nano particles were synthesized to determine the structure of NiCdFe₂O₄ nano particles the X- ray diffraction (XRD) pattern was used. Scanning Electron microscopy (SEM), vibrating sample magnetometer (VSM).

9) Soft magnetic material(NiCdFe₂O₄) particles synthesized by solvent coprecipitation method

From the comparison of the characteristic=cs of NiCdFe₂O₄ nanoparticles prepared by crystallization properties of magnetic nickel ferrite nanoparticles have been investigated by using the solvent coprecipitation method at different temperatures. The results indicated that crystallite size changed increasingly with demonstating the high saturation magnetization moment ,calcined temperatures ,near to the well crystallized NiCdFe₂O₄ particles, and suitable soft magnetic properties.



Chapter 4 Characterization

1 | XRD -

It is used for phase identification of a crystalline material and it is a rapid analytical technique. Unit cell dimensions are provided by X-ray Diffraction. The analysed material is homogenized and average bulk composition is determined. It is finely ground. Fundamental Principle Of X- ray Powder Diffraction – for the learning of crystal structure and atomic spacing of x ray diffraction is now a common technique. It is a based on a crystalline sample and constructive intrusion of monochromatic x rays. By cathode ray tube are generated x rays and magnetic radiations are manufactured them in the direction of the sample and together to concentrate. when conditions regale braggs law then the interaction of the incident rays with the sample produces constructive interference. this law relates To the diffraction angle the wavelength of electromagnetic radiation and in a crystalline sample lattice spacing. by a range of two theta angles by scanning the sample. Because of the random orientation of the powdered material, all possible directions of the lattice should be accomplished. Each mineral has a set of unique d spacings causes the conversion of the diffraction peaks to d spacing allows identification of the mineral. With standard reference patterns and comparison of d spacing this is achieved angle between the incident and diffracted ray are a key component of all diffraction. 36 x ray tube, a sample holder and an x ray detector these three basic elements of the x ray diffractometers. In a cathode ray tube by heating a filament x ray are generated by applying a voltage and bombarding the target material with electrons, to produce electrons accelerating the electrons towards a target material. Of the target material to remove inner shell electrons when electrons have adequate energy. at angle theta the sample rotates in the path of the collimated x ray beam these is a geometry of an x ray diffractometer. To collect the diffracted x rays and rotates at an angle of two theta the x ray detector is mounted on an arm. To maintain the angle and rotate the sample is termed a goniometer these instrument is used. At two theta angle from 5° to 70° angles data is collected for typical powder patterns that are presented in the x ray scan. It is generally used for the identification of undisclosed crystalline materials. In geology, environmental science, material science, engineering and biology is critical to in these determination of undisclosed solids. characterization of crystalline material identification of clays and mixed layer clays of fine grained that are difficult to determine optically. using



Rietveld refinement determine the crystal structure determining the quality of the film by rocking curve measurements and dislocation density. Using glancing incidence x ray reflectivity measurements determining the thickness, roughness and density of the film. 37

2 | Scanning electron microscopy -

it is type of electron microscope which produces by scanning the surface with a focused beam of electrons images of sample produce. Producing various signals that contain information about the surface composition of the sample and surface topography when the electrons interact with atoms in the sample in a raster scan pattern the electron beam is scanned, and with the intensity of the detected signal to produce an image, the position of the beam is combined. Using a secondary electron detector, secondary electrons by atoms excited by the electron beam are detected in the most common SEM mode. The number of secondary electrons can be detected cause the depends, signal intensity, other things on specimen topography. in high vacuum in conventional SEM, or in low vacuum environmental SEM, wet condition in variable pressure, at a wide of cryogenic or elevated temperature with specialized instruments, specimen are observed. from an electron gun fitted with a tungsten filament cathode in a typical SEM an electron beam is thermionically emitted. Tungsten has the highest melting point and lowest vapour pressure of all metals therefore it is normally used in thermionic electron gun. by repeated random scattering and absorption within a teardrop shaped volume of the specimen the electrons lose energy when the primary electron beam interacts with the sample. Is called as interaction volume, which is approximately $5\ \mu\text{m}$ and less than $100\ \text{nm}$ into the surface. by the specimen can detected when the beam current absorbed which is used to create images of the distribution of specimen current. Magnification over a range of about 6 orders of magnitude from about 10 to 3000000 times magnification can be controlled With adequate small diameter the electron gun can generate a beam. Magnification results from the ratio of dimensions of the raster on the specimen in an SEM as in scanning probe microscopy and the raster on the display device. By the current supplied to the x, y, scanning coils or the voltage supplied to the x, y, detector plates then magnification is controlled.



Chapter 5

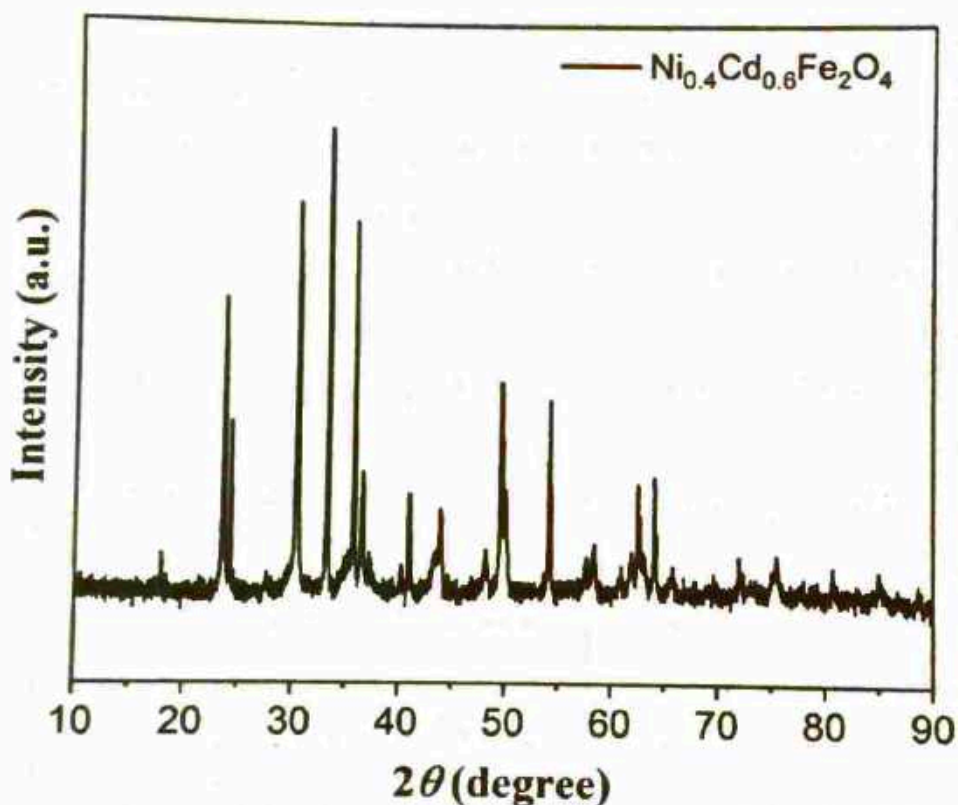
Result & Conclusion

XRD –

The sharp peaks indicate well-crystallized grains. All peaks are identified as the cubicspinel structure . The intensity of planes depends on the cation distribution in the tetrahedral site (A) & octahedral site (B) .

XRD patterns of nickel ferrite ceramic conventionally sintered at several tempratures, for 6 hours it is based on the Jcpds File.

following fig. shows the X-Ray powder diffraction pattern of nickel ferrite particles



The most torridity XRD peak at $2\theta = 33.685^\circ$ & plane spacing $d = 2.5130 \text{ \AA}$ correspond to the (311) plane of the $\text{NiCd-Fe}_2\text{O}_4$ nanoparticles. The considered diffraction peaks with 2θ values of $30.294^\circ, 35.685^\circ, 37.329^\circ, 43.363^\circ, 47.464^\circ, 53.819^\circ, 57.374^\circ$ correspond to crystal planes. (220), (311), (222), (400), (331), (422), (511) respectively. According to the standard of JCPDS.

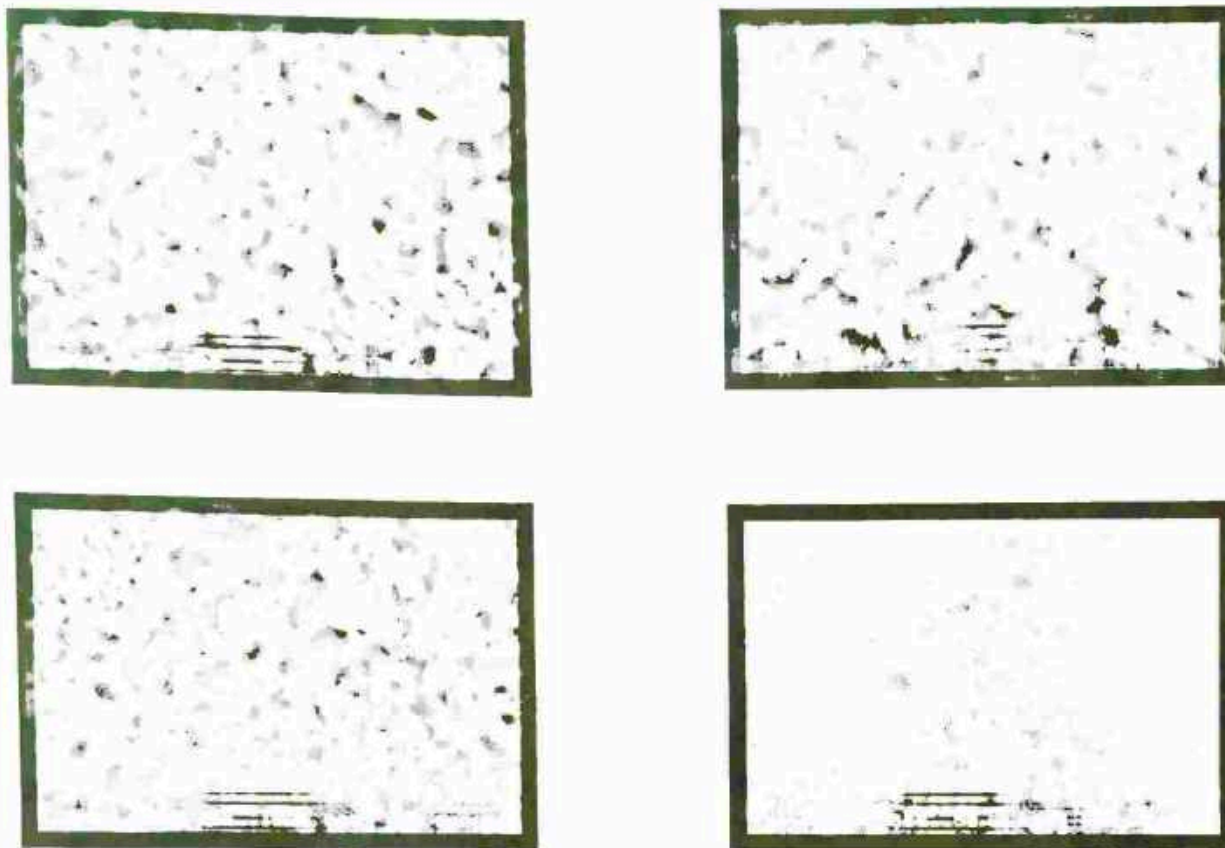
Observation Table –

hkl	2θ	θ	$\sin \theta$	d	a	D
(111)	18.419	9.2095	0.1600	4.8125	8.3354	0.0156
(220)	30.294	15.147	0.2613	2.9468	8.3348	0.0160
(311)	35.685	17.8425	0.3064	2.5130	8.3346	0.0161
(222)	37.329	18.6645	0.3200	2.4062	8.3354	0.0162
(400)	43.363	21.6815	0.3694	2.0844	8.3376	0.0165
(331)	47.464	23.732	0.4025	1.9130	8.3385	0.0168
(422)	53.819	26.9095	0.4526	1.7012	8.3341	0.0172
(511)	57.374	28.687	0.4800	1.6041	8.3351	0.0175
(440)	63.003	31.5015	0.5225	1.4736	8.3359	0.0180



SEM-

The size distribution & morphology of $\text{NiCdFe}_2\text{O}_4$ nanoparticles are determined using SEM synthesized particles are shown in fig.



Grain size = 512 nm

SEM is a type of electron microscope that produce images of a sample by scanning it with with a focused beam of electrons. The electron interacts with atoms in samples and produce signals that can be detected and that contain information about the samples surface topography and composition. SEM image shows that the nanoparticles have uniform almost spherical structural morphology with cramped size distribution but with aggolomerate to some extent



CONCLUSION

Ni-cd ferrite samples have been prepared by sintering method. The effect of the sintering temperature on its microstructural & electric & magnetic properties has been investigated. High values of relative density were obtained for samples is sintered at 1200°C. as reported in the literature , the dc electric resistivity of samples is influenced by both microstructural properties & different oxidation state of ions that support the electronic hopping conduction process. To complete solid state reaction leads to drastic decrease in surface area of the resulting material by sintering again the high temperature (1200) required.



Chapter 6

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FERITE (NiCdFe₂O₄) NANOPARTICLES

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11) Influence of preparation method on structural and magnetic properties of nickel cadmium
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14) History and applications of important ferrites

Leena Jaswal, 1 Brijesh Singh²

15) Preparation of nanocrystalline nickel cadmium ferrite thin films and investigations on their
gas sensing capability



PREPARATION OF MAGNESIUM ZINC FERRITE BY
SINTERING METHOD

A Dissertation Report Submitted to

Vivekanand College Kolhapur,
(Autonomous)

For the Partial Fulfillment of

Degree of Master of Science

In

PHYSICS

Under the Faculty of Science

By

Miss. Neha Sunil Thorat
B.Sc. (Physics)

Under the Guidance of

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Department of Physics,
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(Autonomous)
2021-2022.



DECLARATION

I hereby declare that, the project entitled "**PREPARATION OF MAGNESIUM ZINC FERRITE BY SINTERING METHOD**" completed and written by me has not previously formed the basis for the award of any Degree or Diploma or other similar title of this or any other University or examining body.

Place: Kolhapur

Date: 27-6-2022



Miss. Neha Sunil Thorat

B.Sc.(Physics)



CERTIFICATE

This is to certify that the project entitled “**Preparation of Magnesium Zinc Ferrite By Sintering Method**” which is being submitted herewith for the award of the Degree of Master of Science in Physics of Vivekanand College Kolhapur, (Autonomous). is the result of the original project work completed by Neha Sunil Thorat under the supervision and guidance and to the best of our knowledge and belief the work embodied in this project has not formed earlier the basis for the award of any Degree or similar title of this or any other University or examining body.

Place: Kolhapur

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25/6/22

(Examiner)



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:: Chapter 1::

Introduction to Ferrite

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1.1 History of Ferrites:-

Ferrite, a ceramic-like material with magnetic properties that are useful in many types of electronic devices. Ferrites are hard, brittle, iron containing, and generally grey or black and are polycrystalline that is made up of large number of small crystals. The importance of ferrite material has been known to mankind for many centuries. In early 12 centuries the Chinese were known to use lodestones (Fe_3O_4) in compasses for Navigation.

The William Gilbert work done a milestone in the history of magnetism in 1600. His work "De Magnate, Magnetic Isque Corporibus, et de Magno Tellure" observed the magnetic properties of lodestone up to that point in time. It was not until two hundred years later that main developments started to occur. These developments included work done by Hans Christian Orsted, Andre Marie Ampere, Wilhelm Eduard Weber, and Pierre Curie. Their work added the basis of electromagnetic theory in general and for crystal structures.

In 1819 Hans Christian Oersted searched that an electric current in a wire affected a magnetic compass needle. With further contributions by Michael Faraday, James Clerk Maxwell, Hertz and many others, the new science of electromagnetism developed. At about the same time, in 1948, L. Neel announced his celebrated theoretical contribution on ferrimagnetism. This dealt with the main phenomenon of "spin-spin interaction" taking place in the magnetic sub lattices in ferrites. This stage was now set for the development of microwave ferrite devices.

In 1952, C. L. Hogan from Bell Labs created the first non-reciprocal microwave device at 9 GHz that was based on the Faraday rotation effect. Research was finished to better the properties of the spinel ferrite materials by various cation substitutions. This updated the magnetic properties for different frequency ranges, power requirements, and phase shift applications.



In 1956, Neel, Bertaut, Forrat and Pauthenet observed the garnet ferrite class of materials. This type of ferrite material has three sub lattices, and it is also noticed to as rare-earth iron garnets. These materials, although having a magnetization lower than spinel ferrite, possess extremely low ferromagnetic line width. Another class of ferrite material that was created during this time is the hexagonal ferrite. These materials have three main sub lattices combined in different numbers in a hexagonal structure. The high anisotropy fields have been utilized in microwave ferrite devices in the millimeter range.

In 1959, J.Smit and H.P.J. Wijn published an encyclopedic book on ferrite materials entitled Ferrite. Developments have been made on the magnetic characteristics of ferrite materials since the 1950s that have bettered microwave device performances. These involve both compositional and processing modifications. New applications of ferrite materials continue to be grasped, such as in the cellular phone, medical, and automotive markets.



1.1.1. Ferrites:-

In usual language the ferrites has the materials which are formed when the Iron oxide is mixed with the one or more other compounds here we are making the ferrite material in which the Iron oxide is mixed with the Magnesium and the Zinc such ferrites also called as the compound ferrite. let us know the basic definition of ferrite, according to Wikipedia A ferrite is a ceramic material made by mixing and firing large proportions of iron(III) oxide (Fe_2O_3 , rust) mixed with small proportions of one or more metallic elements, such as magnesium, barium, nickel, and zinc.

And according to the Oxford a ceramic compound consisting of a mixed oxide of iron & one or more other metals which has ferrimagnetic properties & is used in high frequency electrical compounds such as aerials. Also according to the science direct, a ferrite is a type of ceramic compound composed of iron oxide (Fe_2O_3) combined chemically with one or more metallic elements. They are ferrimagnetic, meaning they can be magnetized or attracted to a magnet, and are electrically nonconductive, one of the little substances that combine these two properties.

Ferrite is a Ceramic compound consisting of a mixed oxides of iron and one or more other metals which appears ferrimagnetic property. The ferrites are mainly used due to their high resistivity, low Eddy currents, low dielectric losses and high Curie temperature. Now, we will see the meaning of the terms which are included in the definition above, the word ceramic came from ancient Greek language means the potter's clay. The ceramic materials has the solid material comprising an inorganic compound of metal, non-metal or ionic & covalent bond.



1.2 Types of ferrites :-

Ferrites are mainly classified into three types according to their structures which are,

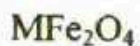
1. Spinel Ferrites [MFe_2O_4]
2. Garnet Ferrites [$M_3Fe_5O_{12}$]
3. Magneto Plum bite Ferrite [$MFe_{12}O_{19}$]

Also these ferrites are classified in terms of their magnetic behavior, they can be again divided into three types as follows,

1. Soft Ferrites.
2. Semi-Soft Ferrites.
3. Hard Ferrites.

1.2.1 Spinel Ferrites:-

The name of spinel is given to these types of ferrites after the mineral called 'spinel' which have the formula $MgAl_2O_3$. In this type of ferrites the structures shows molecular formula like Spinel. The spinel ferrite consist the general formula as,



Where, M- divalent metal ion. (Co, Ni, Mg, Zn, Cd etc.)

Fe- Iron ion.

O- Oxygen ion.

Spinel ferrites consist of Eight (8) unit cells of FCC structures. The FCC structures consists of two types of voids (empty space where the substitution takes place) Octahedral Voids and the tetrahedral voids. Let's study these voids in detail,



The spinel ferrites consists of eight unit cells of FCC, now consider one unit cell of FCC, here the oxygen ion takes place at the lattice points of FCC that is at the corners and at the faces. We know numbers of atoms present in one unit cell of FCC are Four (4). Then the voids generated in FCC in such ideal cases are calculated by in simple way given below,

The tetrahedral voids in FCC are double the no. of atoms present in FCC structure. So,

$$4 \times 2 = 8 \text{ tetrahedral voids.}$$

Similarly, the octahedral voids present in FCC are equal to the number of atoms present in FCC structures.

$$4 \times 1 = 4 \text{ Octahedral Voids.}$$

Also these voids takes place at particular conditions lets see which are that conditions or situations.

1. Tetrahedral Voids:-

The tetrahedral voids took place where the three atoms are present in one plane and the one atom is placed above these three atoms, then the void is created known as tetrahedral void. Shown in fig below. Such voids take place at the each corner; also they took place inside the atom so their contribution is also one. And these tetrahedral voids are smaller than the octahedral voids.

Generally, the divalent metal atoms occupy the tetrahedral voids. Each FCC structure consists of 8 corners hence, the tetrahedral voids in FCC are 8. But we know the no. of unit cells of FCC in Ferrites are 8. So, total tetrahedral voids in ferrite are,

$$\text{Total Tetrahedral voids} = 8 \text{ voids in each corner} \times 8 \text{ unit cells of FCC}$$

$$\text{Total Tetrahedral voids in spinel ferrites} = 64.$$



But not all these voids are filled by the divalent atom, only one/eight ($\frac{1}{8}$) of these voids are occupied by the divalent atom. So, only eight voids are occupied by the divalent atom.

2. Octahedral voids:-

In one unit cell of FCC four octahedral voids take place, again these voids are divided in two types according to where they are present which are Body centered octahedral and the edge centered octahedral void. The trivalent Fe atom occupy these voids.

1. Body Centered octahedral void:-

The body centered octahedral void is generated where the four atoms took place in one plane and the two atoms are placed above and below from the plane then the void is created in between this arrangement, known as Body centered octahedral void. The name body centered is given to this void because this void is present at the center of FCC unit cell. As shown in following fig.

So, only one void is takes place at the center hence,

$$\text{Total Body centered Octahedral voids} = 1$$

2. Edge centered octahedral voids:-

The edge centered octahedral void also take place where the four atoms took place in one plane and the two atoms are placed above and the below the plane of four atoms, then the void is generated known as Edge centered octahedral void.



These voids are present at each edge, the total numbers of edges are 12, also each edge is divided into four unit cells that is each edge is shared into four neighboring cells. So, in calculation of total voids in one unit cell we have to take contribution of edge to one unit cell which is $(\frac{1}{4})$. Hence,

$$\text{Total Edge Centered Octahedral voids} = 12 \text{ total edges} \times \frac{1}{4}$$

$$\text{Total Edge Centered Octahedral voids} = 3.$$

Hence,

$$\text{Total Octahedral Voids} = 1 \text{ Body Centered oct. void} + 3 \text{ Edge centered oct. voids}$$

$$\text{Total Octahedral Voids} = 4.$$

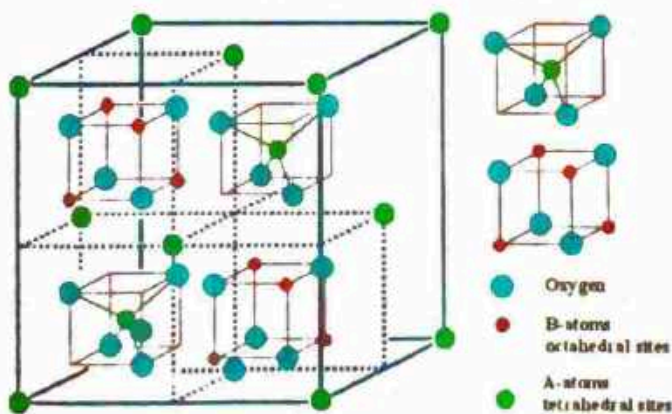
Similarly,

$$\text{Total Octahedral voids in spinel ferrite} = 4 \text{ voids in one cell} \times 8$$

$$\text{Total Octahedral voids in spinel ferrite} = 32.$$

Similar to the tetrahedral voids all the octahedral voids are not occupied by the trivalent atom only half of octahedral voids that is 2. (one is body centered and other is edge centered).

Hence, in spinel ferrites total voids are 64 tetrahedral voids and 32 octahedral voids in which only 8 tetrahedral and the 16 octahedral voids can be occupied by the atoms.



AB_2O_4 spinel The red cubes are also contained in the back half of the unit cell

Now, going further these spinel ferrites are again divided into two types as,

1. Normal Spinel ferrites.
2. Inverse Spinel Ferrites.

1) Normal Spinel Ferrites:-

The normal spinels ferrites are made up of the 8 FCC cells. The anions (usually the Oxide O^{2-}) occupy the FCC lattice points. The divalent A^{+2} cations (metal divalent atoms) occupies the $1/8^{\text{th}}$ of tetrahedral voids; And the trivalent B^{+3} cations occupy octahedral half ($1/2$) of the octahedral voids. Generally they are represented as,



That means, in a normal spinel, there are $8 \times 4 = 32$ anions occupying the lattice points of 8 FCC unit cells. Whereas, the number of divalent A^{II} cations occupying tetrahedral voids is $8 \times 1/8 \times 8 = 8$ and the number of trivalent B^{III} ions occupying the octahedral voids = $8 \times 1/2 \times 4 = 16$.

i.e. The ratio of $A^{II} : B^{III} : O^{2-} = 8 : 16 : 32 = 1:2:4$ which confirms with the formula of normal spinel.

2) Inverse Spinel Ferrites:-

Here, the lattice points are occupied by the anions (O^{2-}) and the divalent A^{+2} cations occupy octahedral voids and half of B^{+3} occupy the half of tetrahedral voids and remaining half occupies the octahedral voids.

Generally they are presented as, $B(AB)O_4$

The some examples of Spinals are given in table below,

Normal Spinal	Inverse Spinal
$MgAl_2O_4$	$Pb_3O_4 = Pb^{II}(Pb^{II}Pb^{IV})O_4$
Na_2WO_4	$Fe_3O_4 = Fe^{III}(Fe^{II}Fe^{III})O_4$
$CuFe_2O_4$	$CoFe_2O_4 = Fe^{III}(Co^{II}Fe^{III})O_4$

Table 1.1: Examples of spinals



The difficulty arises that how to know the type of spinels externally, it is not possible to decide the type of the spinel, there are some rules using which we can decide the type of spinel ferrites.

3) Random spinel ferrite:

The divalent metal ions have M^{2+} and trivalent Fe^{3+} ions are distributed at both tetrahedral (A) site and octahedral [B] site then the ferrite is termed as random spinel ferrite. The very best known example of random spinel ferrite is copper ferrite.

The distribution of ions between two types of sites is determined by a delicate balance of contributions, such as the magnitude of ionic radii, their electronic configuration and the electrostatic energy of the lattice. The cation distribution of random spinel ferrite is given by;



Examples- $CuFe_2O_4$, $MgFe_2O_4$

4) Rules to decide Normal or Inverse Spinel Ferrites:-

Rule 1:- If normal spinel structure is assumed then both the divalent and the trivalent metals are non transition metals since no CFSE is involved.

There is a tendency of creation of inverse spinel structure in some cases (not all the cases) which contain transition metal ions. This is because, the transition metal ion may receive extra stability (LFSE) in octahedral geometry, prefers octahedral voids over tetrahedral ones.

The d^0 ; high spin d^5 , d^{10} ions have no choice between tetrahedral and octahedral coordination since the LFSE is zero.

Rule 2:- Usually d^3 & d^8 ions have strongest preference for octahedral geometry. Other ions with d^1 , d^2 , d^4 , d^6 , d^7 , d^9 too have slightly more choice for octahedral symmetry. That means, if A^{II} has d^3 or d^8 configuration and

the B^{III} ion has been configuration other than these, then the spinel is inverted.

Rule 3:- If A^{+2} is d^0 or d^5 or d^{10} configuration & B^{+3} has d^1 or d^2 or d^3 or d^4 or d^6 or d^7 or d^8 or d^9 configuration then structure is always spinel.

Rule 4:- If A^{+2} has high CFSE and the B^{+3} has the low CFSE then in such cases the structures is inverse spinel

1.2.2. Garnet Ferrites:-

The chemical formula of garnet is $Me_3Fe_5O_{12}$ where, Me may be a trivalent ion like rare-earth element or yttrium. The unit cell is cubic and it contains eight molecules of $Me_3Fe_5O_{12}$ i.e. (160 atoms). The metal ions are distributed over three sorts of sites. The Me ions cover the dodecahedral sites (called c sites), where they're surrounded by eight oxygen ions, the Fe^{3+} ions distributed over the tetrahedral and octahedral sites in the ratio 3:2. Thus, the cation distribution of $Me_3Fe_5O_{12}$ can be written as $Me^c_3Fe^a_2Fe^d_3O_{12}$.

1.2.3. Magneto plum bite ferrites:-

Although the spinel and the garnet ferrites consists of cubic structure as shown in fig above (page no. 11), there are another types of ferrites which shows the hexagonal structure, they are known as hexagonal ferrites or magneto plum bite ferrite.

Generally they are represented as,



Where, the M is the any divalent cation with high ionic radii, Fe is the trivalent ion and the oxygen plays the role of anion in this structure.

So, these are the three types of ferrites which can be summarize using following table,



Type of ferrite	Structure	General Formula	Example
Spinel	Cubic	MFe_2O_4	$MgFe_2O_4$
Garnet	Cubic	$R_3Fe_5O_{12}$	$Y_3Fe_5O_{12}$
Magneto plum bite	Hexagonal	$MFe_{12}O_{19}$	$BaFe_{12}O_{19}$

1.3. Magnetism and types of magnetic materials:-

Magnetism has combined electromagnetic force. Also it refers to physical phenomena arising from the force caused by magnets, objects that produce fields that attract or repel other objects. To get to know about the ferrites one should have a basic knowledge of magnetism. Since all the materials on the earth contains magnetism in it in small or large proportion. Origin of magnetism in any material arises because of the motion of electrons inside the atoms of a particular material. If each orbit of electron considered as a tiny loop of current when it has certain magnetic moment called orbital magnetic moment.

Electron also spins around an axis which produces spin magnetic moment. The resultant magnetic moment of an electron is a vector sum of these two magnetic moments. In an atom the magnetic moments of core electrons cancel out and magnetic moment of atom is vector sum of magnetic moment of unpaired electrons only.

On the basis of magnetic properties of material and the behavior of magnetic moments of the material, substances can be classified into following types



1) Diamagnetic material:-

This type of materials does not contain any unpaired electrons that is why they do not show any magnetic property. When external magnetic field is applied to it, it gets magnetized but in non cooperative way that is it repels external magnetic field. It shows magnetization is in the exact opposite direction of the external magnetic field. Susceptibility of diamagnetic material is negative and small. After the removal of external magnetic field it gets demagnetized.

Examples- CuO, Bi etc.

2) Paramagnetic material:-

This type of materials has some unpaired electrons and the magnetic moments of all the atoms are oriented randomly and neutralize each other magnetic moments. This type of materials is also called magnetically dilute system. Paramagnetic materials obey Curie Weiss law. When external magnetic field applied to it, it gets magnetized.

It shows magnetization in the direction of external magnetic field. After the removal of external magnetic field, it gets demagnetized.

Susceptibility of a paramagnetic material is positive and greater than 1.

3) Ferromagnetic material:-

Each atom of ferromagnetic materials has certain magnetic moment and there are domains where all the atomic magnets oriented in the same direction. All the domains in a particular ferromagnetic material are randomly oriented and the resultant magnetic field is zero.

When external magnetic field is applied to it, all the domains will orient in the direction of the external magnetic field, and material gets magnetized. After the removal of external magnetic field, material still remains magnetized. That is a permanent magnet is produced. There is a temperature

criteria to ferromagnetic material. That is after particular temperature material shows no ferromagnetic nature it becomes paramagnetic material and that temperature is Curie temperature. Susceptibility of ferromagnetic material is very hard

Examples- Co, Ni etc.

4] Antiferromagnetic material:-

This type of materials has adjacent metal ions coupled with their spins antiparallel to each other. There are always equal and opposite number of alignments so that in the absence of magnetic field there is no magnetization. Susceptibility of antiferromagnetic material is high at Neel temperature. In reality, antiferromagnetic order exists at sufficiently low temperature, but pernicious at and above certain temperature that is Neel temperature.

5] Ferrimagnetic material:-

This type of materials have ions on two sets of lattice sites and they have opposite spin arrangement but this time the spin arrangement do not cancel each other because of the difference in the magnitude.

Therefore there is a resultant permanent magnetic moment in the direction of external magnetic field produced after it placed in the external magnetic field. Susceptibility of ferrimagnetic material is positive and high.

Examples- Fe_3O_4 , NiFe_2O_4 etc.

Basic terms used in magnetism

❖ External magnetic field strength (H):-

- It is the magnetic field which is used to study the magnetic nature of a given material. Magnetic field is measured in A/m or Oe.



❖ Magnetization (M):-

- Net magnetic dipole moment per unit volume is called magnetization. Magnetization is a vector quantity and measured in A/m.

❖ Susceptibility (χ):-

- Magnetization and external magnetic field intensity are related to each other by susceptibility
- Mathematically susceptibility is ratio of magnetization to external magnetic field strength.
- Susceptibility can be positive or negative.
- Sign of Susceptibility indicates whether a material is attracted into or repelled out of the magnetic field.
- Susceptibility is a dimensionless quantity hence it has no unit.

❖ Permeability (μ):-

- Permeability is ability of a material to permit the external magnetic lines of force to go through it.
- Permeability is also the measure of the ability of a material to support the formation of magnetic field within itself.
- Mathematically permeability is ratio of induced magnetic field to the external magnetic field strength.
- The unit of permeability is henry/m.

1.3 Application of ferrites :

1. Ferrites are mostly applicable in electronic products, automobiles, communication equipment's, data processing devices. Earlier, metallic magnets were used but for frequency greater than megahertz metals and alloys are not suitable as soft magnets as the Eddy current losses are generally very high. Another reason is resistivity of ferrites is million times greater than that of metals.



2. Soft ferrites or high frequency ferrites are used in radio, TV, microwave satellite communication, audio/video/digital recording as permanent magnets.

3. Hard ferrites are used as permanent magnets in speakers and micro motors.

4. Ferrites are used in telecommunications - ferrite cores are used on large scale and they are used to manufacture filter inductor, false and matching transformer.

5. Ferrite are used for power conversion generally in the form of switched mode power supply (SMPS)

6. Ferrites can be used to produce iron power ring cores, beads on wire, wideband chokes, cable shields, rod and tubes.

7. Magnetic sensors - ferrite can be used as temperature control sensors in which the sharp and definite Curie temperature is used in a temperature control sensors. Ferrites can also be used in position and rotational angle sensors or proximity sensors.

8. Magnetic shielding-

Radar absorbing paint containing ferrites has been developed to render submarines.

9. Pollution control-

The precipitate of ferrite precursor to scavenge pollutant materials such as Hg is used in various countries.

10. Ferrite electrodes-

Ferrites with high corrosion resistance and appropriate conductivities are used in chromium plating.

11. Entertainment-

Ferrites are used in radio and TV circuits as deflection yokes, flyback transformer and SMPS Transformers for power application.



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:: Chapter 2 ::

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2.1 Sol-Gel method:

In the Sol-Gel method, the formation of a gel provides a high degree of homogeneity and the need for atomic diffusion reduces during the solid state calcinations. A solution of appropriate precursors is formed. First, followed by conversion into a homogeneous oxide after the condensation and hydrolysis, oxide product is yielded at drying and subsequent calcinations of the gel.

2.2 Precipitation method:

In the precipitation condition method for the employed the precipitation of substances normally soluble under these conditions. An inclusion occurs when in the lattice site the impurity occupies as in the crystal structure of carrier, resulting in the crystallographic defect, which can occur when the charge of impurity and the ionic radius are similar to those of carrier, when an adsorbed impurity is physically trapped inside the crystal as it grows as the occlusion occurs.

2.3 Microwave-Assisted method:

The new technique of microwave-assisted method which mergeability for the synthesis of ferrites. The advantage of this method are generally fast, lower reaction time, simple, good yield, high energy efficiency, cost effective, and the appropriate dielectric loss of synthesized ferrite. Moreover, the synthesized spinel ferrite nanoparticle on commercial scale but the low yield can be obtained when compared to other methods such as the hydrothermal, coprecipitation thermal decomposition method. In this method as the internal rather than external by heating the energy as the case of conventional combustion reaction

and utilized the microwave energy for the combustion of precursor to form ferrite, as the conversion of microwave energy to thermal energy due to heat generation, and the temperature usually ranges from 100°C to 200° C for the short period of time.

2.4 Molten salt low temperature method:

This method is also the chemical reaction method, for an inhibiting the grain growth sodium chloride plays a vital role. as the effective environment friendly route in low cost, high yielding, convenient and the inexpensive method. As the metal oxide can be obtained by grinding solid metallic salts with sodium hydroxide in solution. in presence of sodium chloride the grain growth is inhibited as it leads to the formation of coating surrounding the nanoparticle and from aggregating the large particle to preventing the nuclei. The reagent are to be taken in the agate mortar and pestle specified molar ratio and grounded together, followed by the calcinations and filtering.

2.5 Ceramic method:

Sintering process

Sintering is defined as, the thermal treatment of compact at a temperature below the melting point of the main constituent, for the purpose of increasing its strength by bonding together of the particles. Solid state sintering involves only solids and pores.

Products that have been dried and surface finished are called, "green products". The finished product requires firing treatment known as sintering in



order to develop the desired microstructure and properties. This process called firing proceeds in three stages.

1. Reactions prior to sintering which include organic burnout and the elimination of decomposed gas product.

2. Sintering

3. Cooling

Prior to sintering, the binder and the other additives in the powder compact have to be removed. Binder removal is accomplished thermally by heating the green body in high temperature. Thermal debonding of binder in ceramics is a complex process, which takes place in three different stages.

In the first stage of sintering, the binder present in the green body gets soft when the temperature reaches the range of 150-200°C. At this stage, chemical decomposition and binder removal are negligible. In the second stage of 200-400°C, most of the binder is removed by chemical decomposition. In the final stage, the small amount of binder still remaining in the powder compact is removed when the temperature reaches slightly above 400°C.

Sintering is the term used to describe the consolidation of the product during firing. Sintering gives strength to the shaped powders by joining the particles into an aggregate. During sintering, interparticle bond reduces the surface area, which in turn lowers the surface energy. The bonds grow by various mechanisms that occur at the atomic level. For many metals and ceramics, the bonding occurs by solid-state diffusion. Several reviews (Yan 1982, Handwerker 1988, Shaw 1989) give the details of solids state sintering theory. Solid state sintering is usually employed in the case of single-phase material. Many of the sintering systems involve multiphases. The term liquid phase sintering describes the sintering process, in which a portion of the material, which is being sintered, is in liquid state. The formation of liquid phase during sintering usually increases the sintering rate and enhances the



densification by particle rearrangement under the influence of capillary force and filling of pores.

Microstructural change during sintering takes place normally in three stages. Outlines the classic stages of sintering that represents the geometric progression involved in transforming the powder compact into a strong and dense component. Sintering usually occurs at temperature in excess, approximately one-half of the absolute melting temperature.

In the first stage of the sintering, the formation of grain boundaries causes the particles to join together. The degree of bonding in the initial stage ranges from point contact to highly deformed interface. The intermediate stage is the most important stage for densification and hence determines the properties of the sintered compact. The growing of neck is the focus of the initial stage of sintering. It loses its identity in the intermediate stage and the attention shifts to the pore volume, size, shape and the distribution. During this stage, the grain size increases. The pores attached to the grains coalesce (increase pore size) and the porosity is eliminated. Normally, the pores located on the grain boundaries shrink substantially faster than the isolated pores or the pores located in the interior of the grain.

When the grain growth occurs in the intermediate stage, pore migration and pore rounding occur due to active surface transport. As the pores shrink, the final stage of sintering occurs when the pore size/grain size ratio is small.

If the grain growth is rapid in comparison with pore mobility, the pores are isolated from the grain boundaries, resulting in slow densification. Alternatively, if pore mobility is high, it disappears as it touches the grain boundary; it moves to new position and makes contact with the next layer of pores. Thus, the boundary begins to move slowly throughout the sample to sweep the region through which it moves free of pores which results in high densification. Microstructural changes in the final stages of sintering may



proceed in a desirable or an undesirable manner. Many of the ferrite properties are highly dependent on processing parameters such as sintering temperature, sintering time, and heating atmosphere etc., as these parameters greatly influence crystal composition and size and also the size and volume of the pores. Many researchers have reported the electrical and magnetic properties of magnesium-zinc ferrites with respect to sintering conditions.

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:: Chapter 3 ::
Literature survey

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3.1 Literature survey of $MgZnFe_2O_4$

In recent years, the researchers strongly focused on the synthesis of nanosize (nanocrystalline) Magnesium Ferrite by the solution combustion method and microwave-assisted combustion method. Using this method, the heating and evaporation of metal nitrate solution with an organic compound like fuels (such as glycine, urea, carbohydrazide, citric acid etc.) results in self-firing and generates intense heat by exothermic reaction. This intense heat is used to synthesize the nano powder. This novel approach has the advantages of inexpensive raw materials, a relatively simple preparation process and a fine resulting powder with high homogeneity.

Microwaves have been used for applying microwave irradiation to chemical reactions. Microwaves act as a high frequency electric field generator and will generally heat anything with a mobile electric charge. Polar solvents are heated as their component molecules are forced to rotate with the field and lose energy in collisions. Semiconducting and conducting samples heat when ions or electrons within them form an electric current and energy is lost due to the electrical resistance of the material. Heating a reaction or chemical reactor by microwave radiation (as seen in a domestic microwave oven) has a number of advantages over conventional heating. Ferrites are mostly prepared via conventional ceramic processing involves commercially both long and high temperature treatments for the oxides used in their preparation. Magnesium ferrites having spinel crystal structure are extensively used in a number of electronic devices because of their high permeability at high frequency, remarkably high electrical resistivity, mechanical hardness, chemical stability, and reasonable cost.

The studies on magnetic and dielectric properties in Magnesium ferrites synthesized by conventional technique have been reported by so many



workers. The conventional method is the most common method for preparing ferrites for so decades. They investigated the frequency, temperature and composition dependence of Magnesiumferrites. They suggested the variation of dielectric constant depends linearly on the variation of available ferrous ions on octahedral sites. Dielectric constant declined with increasing frequency. The maximum dielectric dispersion was seen which be explained on the basis of available ferrous ions on octahedral sites.

In conventional methods, there are some inherent drawbacks such as poor compositional control, chemical in homogeneity, coarser particle and introduction of some impurities during milling. Thus, the coarser and non-uniform particles cause the formation of some voids and low-density areas in the green compact. However, the variety wet methods such as ball milling, hydrothermal, sol-gel, microemulsions, and co-precipitation have been used to synthesize ferrite materials. Each method has unique advantages. But trying to improve the physical properties of ferrites by new designing in synthesize is still a matter of primary interest. Recently microwave assisted methods have been used to prepare ultrafine, homogenous and reproducible powder. In the recent past, the microwave assisted method has been found to have distinct advantages over the conventional dry processing. The final goal is to fabricate the ferrites with better physical and magnetic properties that are useful in a variety of industrial applications.



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:: Chapter 4 ::

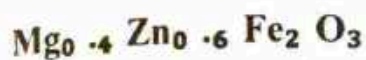
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4.1 Synthesis of material :

Magnesium zinc ferrite are a series of synthesis inorganic compounds of zinc magnesium and iron with general formula $MgZnFe_2 O_4$. The zinc ferrite can based pigment, because of their high opacity, especially in applications requiring heat stability. it is one of the widely studied materials because the high quality of magnesium zinc ferrite nanostructure has generated a lot of interest owing to their potential application in magnetic behavior, gas sensing, electrical sites. Among the various sites zinc ferrite find the special position due to high electromagnetic performance, mechanical hardness, excellent chemical stability, low coercivity.

Here we see how we prepare the $MgZnFe_2 O_4$



Chemical weights:

a) 0.8431 gm of magnesium carbonate.

b) 1.8807 gm of zinc carbonate.

c) 3.9925 gm of iron oxide

1. Magnesium Carbonate ($MgCO_3$)_{0.4} :

Molecular weight: 33.724 gm

2. Zinc Carbonate ($ZnCO_3$)_{0.6} : 75.228 gm

3. Ferric Carbonate ($Fe_2 O_3$) : 159.69 gm

Common factor = 40

$$MgCO_3 = 33.724 \div 40 = 0.8431 \text{ gm}$$

$$ZnCO_3 = 75.228 \div 40 = 1.8807 \text{ gm}$$

$$Fe_2 O_3 = 159.69 \div 40 = 3.9925 \text{ gm}$$



4.2 Method Of Preparation :

1. first clean the crucible with the help of water, then the mixture of above precursors material of zinc carbonate, magnesium carbonate and iron oxide added into it.
2. We added acetone slowly in this mixture to sensing characteristic of wet chemically prepared magnesium zinc ferrite nano - particles have been investigated.
3. After that we milling this mixture up to 4 to 5 hours to form a powder of nanoparticles.
4. After milling this mixture powder of nanoparticles is formed and this powder is heated into muffle furnace at 600 degrees Celsius for 4-5 hours.

X- Ray Diffraction :

The X-ray diffraction is an important technique for characterization of bulk and thin film samples. It is used for investigation of crystal structure and phase of material. The structural identification and determination of lattice parameters are based on the interpretation of XRD pattern.

The phenomenon of X-Ray Diffraction consists of reflection of the X rays from the different crystallographic planes of material and Bragg's law governs it,

$$2d\sin\theta = n\lambda(1)$$

Where,

d = interplanar spacing

θ = Bragg's Angle / Diffraction angle

λ = X- Ray wavelength (1.5406 Å)

n = Order of diffraction

In Powder diffraction method the crystal to be examined is reduced to a fine powder and placed in a beam of monochromatic X-Rays. Each particle of the powder is tiny crystal or assemblage of smaller crystals, oriented at random with respect to incident beam of X-Rays. For example, some of the crystals are correctly oriented so that their (1 1 1) plane, can be reflects the incident beam. Other also correctly oriented for (2 2 0), (3 1 1) reflections and so on.



The principal of X-Ray powder diffractometer is that every set of lattice planes will be capable of reflections. Ideally according to Bragg's law for particular d values, the constructive interference of X-rays should occur only at a particular theta value i.e. Bragg's angle and for all other angles there should be destructive interference and intensity of diffracted beam will be minimum for destructive interference.

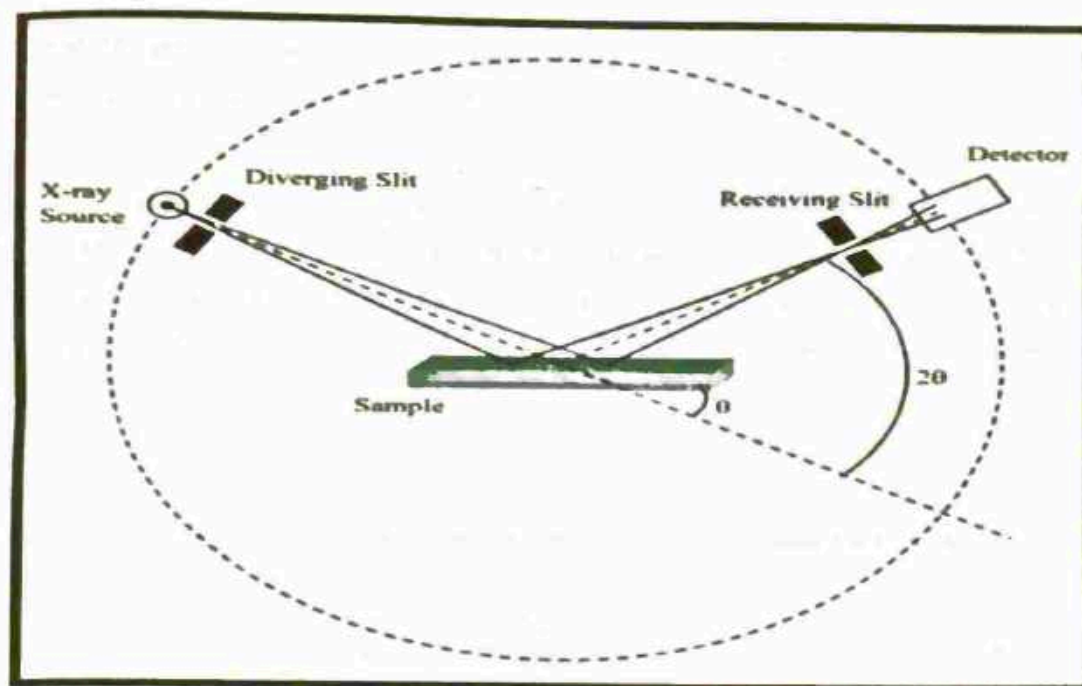


Fig. Schematic diagram of X-Ray Diffractometer

Identification Of Phases :

Phases of different materials can be identified by the following way : The observed d values are compared with standard d values ASTM (International American Standard for Testing of materials) standard data file or JCPDS (Joint committee for Powder diffraction Standards) data file for the same material synthesized by same standard chemical method. This analysis reveals the different phases present in the sample and Miller indices ($h k l$) of the atomic planes. The lattice Parameters for the unit cell of the present phase can be calculated by using Cullity's equation. Absence of reflection peaks indicates amorphous nature of the sample. A single peak indicates epitaxial growth while many reflection peaks indicate Polycrystalline (heteroepitaxial) growth. However if size of the diffracting tiny crystal is small, then there is no more complete destructive interference which broadens the peak corresponding to diffracted beam in proportion to the size of the tiny crystal. This can be used to



calculate the crystal size. The crystallite size was calculate by Scherer relation, and formulated as,

$$D = \frac{0.9 \times \lambda}{\beta \cos \theta}$$

Where,

D = Crystal size

Lambda = X ray wavelength

β = Line broadening at Full Width at Half Maxima (FWHM),

θ = Bragg's Angle / Diffraction Angle

Scanning Electron Microscopy (SEM) : A scanning electron microscope (SEM) projects and scans a focused stream of electrons over a surface to create an image. The electron beam interacts with the sample, thereby producing various signals that can be used to obtain information about the surface topography and composition.

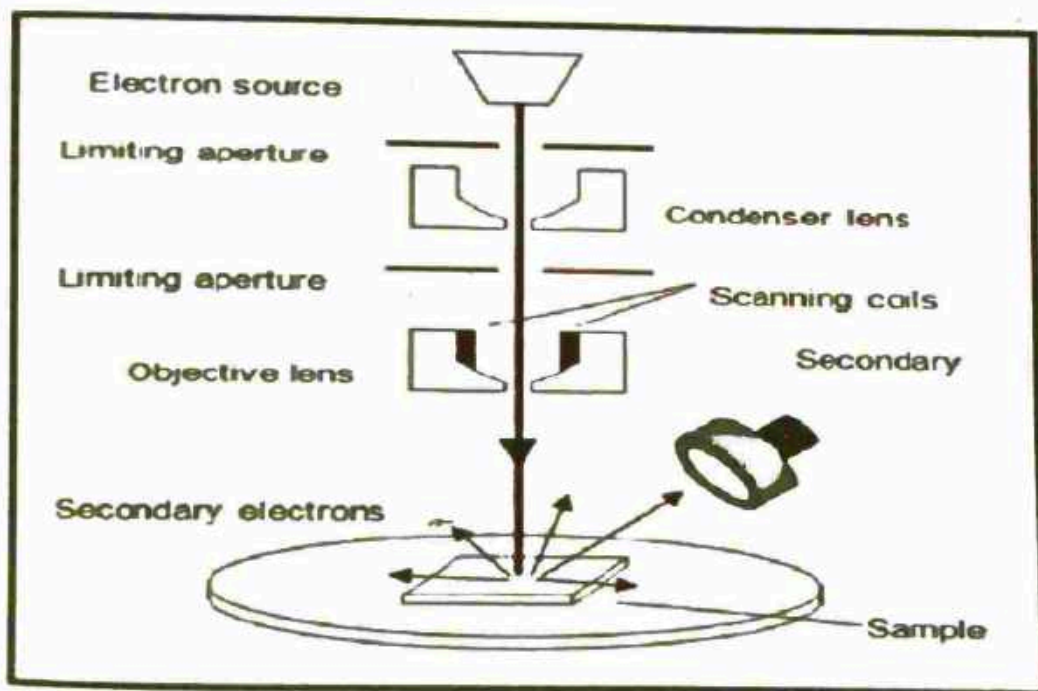


Fig. Block Diagram Of Scanning Electron Electroscopy



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5.1 X-Ray diffraction Analysis :

After sintering process, the magnesium zinc ferrite powder is formed. Then we analyse the XRD pattern and calculate the crystalline size of particles.

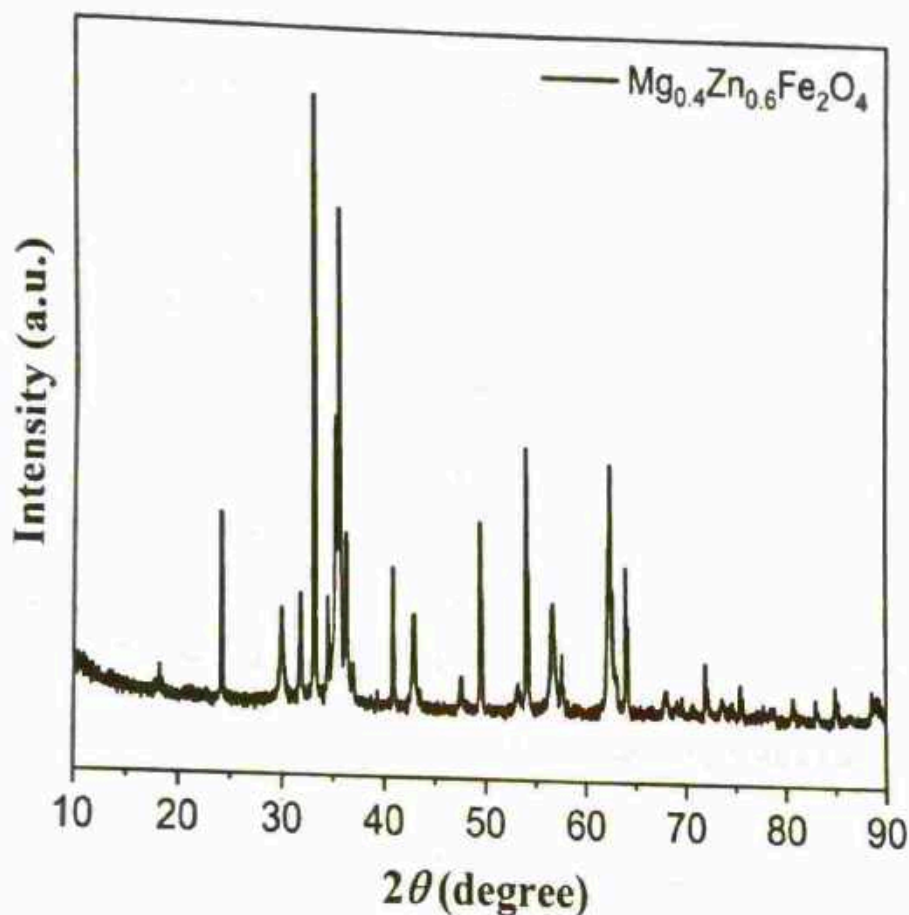


Fig. XRD pattern of prepared material

The X-ray diffraction pattern obtained for the sample $Mg_{0.4}Zn_{0.6}Fe_2O_4$ using Cu (K- alpha) radiation [wavelength = 1.5406 AU] are shown in above fig. The values which diffracts in X-ray spinel's are (1 1 1), (2 2 0), (3 1 1), (2 2 2), (4 0 0), (4 2 2), (3 3 3), (4 4 0). Graph shows maximum (h k l) value (3 1 1) at angle 35.652° .

Observation Table :

1) For Lattice parameter (a) :

S. No.	hkl	d_{obs}	d_{calc}	$\frac{d_{obs}}{d_{calc}}$	$\frac{1}{d_{obs}^2}$	$\frac{1}{d_{calc}^2}$	$\frac{1}{d_{obs}^2} - \frac{1}{d_{calc}^2}$
1	(1 1 1)	18.260	9.1300	0.15867	0.31735	4.85457	8.4083
2	(2 2 0)	29.99	14.9950	0.25873	0.517469	2.9771	8.4208
3	(3 1 1)	35.652	17.8260	0.30612	0.612254	2.516275	8.3458
4	(2 2 2)	36.5217	18.2608	0.31334	0.62668	2.45835	8.5159
5	(4 0 0)	42.82607	21.4130	0.36508	0.73017	2.10990	8.4396
6	(4 2 2)	53.47818	26.7390	0.44992	0.89985	1.71206	8.3873
7	(3 3 3)	56.5217	28.2608	0.47348	0.94697	1.62687	8.4534
8	(4 4 0)	62.6080	31.3043	0.51958	1.03916	1.48253	8.3864

Mean lattice constant $a = 8.42 \text{ \AA}$

2) For Crystallite size (D) :

S. No.	hkl	d_{obs}	d_{calc}	$\frac{d_{obs}}{d_{calc}}$	$\frac{1}{d_{obs}^2}$	$\frac{1}{d_{calc}^2}$
1.	(1 1 1)	18.260	9.130	0.9873	0.9336	
2.	(2 2 0)	29.99	14.995	0.9659	0.9543	
3.	(3 1 1)	35.652	17.826	0.9519	0.9683	
4.	(2 2 2)	36.5217	18.260	0.9496	0.9707	
5.	(4 0 0)	42.8260	21.413	0.9309	0.9902	
6.	(4 2 2)	53.4781	26.739	0.8930	1.0321	
7.	(3 3 3)	56.5217	28.260	0.8808	1.0465	
8.	(4 4 0)	62.6086	31.304	0.8544	1.0788	

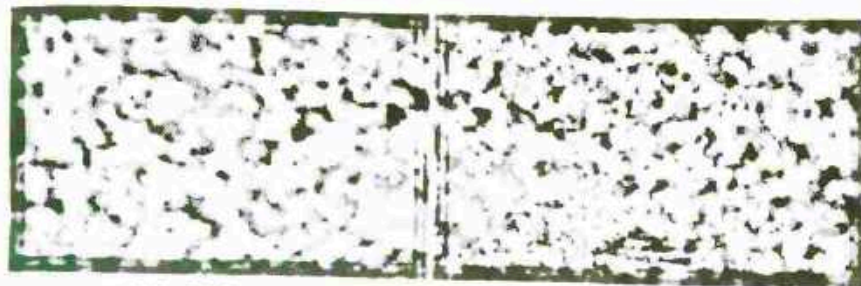
Mean Crystallite Size = 0.99 nm



5.2 SCANNING ELECTRON MICROSCOPY (SEM)

ANALYSIS :

Here we calculate the grain size of particles by SEM. The morphological characteristics of the obtained $Mg_{0.4}Zn_{0.6}Fe_2O_4$ nano particles were investigated by the high resolution scanning electron microscopy and are shown in Fig. images of $Mg(0.4)Zn(0.6)Fe_2O_4$ samples reveal that all the samples exhibit a compact arrangement of homogeneous nanoparticles with nearly spherical shape.

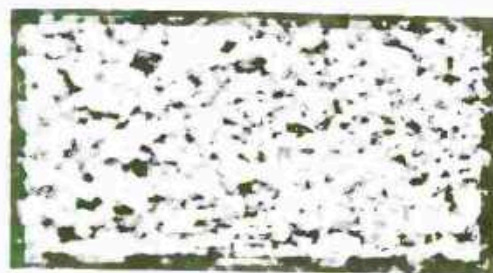


× 10,000

Grain Size = 250 nm.

× 5000

Grain Size = 270 nm



× 2,500

Grain Size = 270 nm

Mean Grain Size = 263.33 nm

Fig. SEM Images



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6.1 Summary :

The title of our project was to prepare the Mg doped $ZnFe_2O_4$ powder by sintering method and to analyses the XRD pattern and characteristics of this nanoparticles. Nanoparticles of $Mg_{0.4}Zn_{0.6}Fe_2O_4$ Ferrite were Synthesized using sintering process. X-Ray diffraction data reveal the formation of cubic spinel phase for all composition. An appropriate ratio of magnesium Carbonate, Zinc carbonate and Ferrite oxide is taken into crucible which was milled with adding slowly acetone. This mixture is milled up to 4 to 5 hrs. to form a powder of this material. After that the material was heated at $600^{\circ}c$ for 5 to 6 hrs. in Muffle furnace. The brownish colored Powder is obtained.

This powder is used for XRD and SEM for finding structure and characterization of this material.

6.2 Conclusion :

After working for last 8 months on this project finally this time to conclude here, as our aim to prepare the Mg doped Zinc Ferrite by using the sintering method and to do so we choose the above procedure of sintering process. Then we took the characterization test to confirm our material nanoparticles are made or not, from peaks in the XRD graph we calculate the crystallite size from (h k l) and 2 theta values. And also we confirm the structure of material by calculating grain size of the prepared nanoparticles. Hence we Succeeded to prepare the magnesium doped Zinc Ferrite powder by using Sintering method.

