Development of Mn-Zn Ferrite by controlling its microstructure.

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF BACHELOR OF TECHNOLOGY

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CERTIFICATE

NATIONAL INSTITUTE OF TECHNOLOGY, 2013

This is to certify that the thesis entitled, **"Development of Mn-Zn Ferrite by controlling its microstructure"** submitted by **Yengkhom Hollender Singh** in partial fulfilment of the requirements of the award of Bachelor of Technology Degree in Ceramic Engineering at the National Institute of Technology, Rourkela is an authentic work carried out by him under my supervision and guidance.

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ABSTRACT

Manganese-Zinc Ferrite is a very high permeability magnetic material. The permeability is depended on the amount of manganese and zinc used in the synthesis of ferrite material. In different literatures, there are several works upon the varying manganese and zinc content Manganese-Zinc Ferrite. The present work of project is mainly focused on the differences in the magnetization by analysing the amount of manganese, zinc and ferrite used, firing temperature of the pressed sample, the amount of unreacted compound present and the microstructure of the sample. First, powder of MnO_2, ZnO and Fe_2O_3 were mixed by pot milling. After calcination the powder at 950°C, the powders were pressed into pellet. For the phase formation, the powder were characterized by using XRD and for the microstructure analysis, the powder were characterized by using SEM. The pellet of different composition were sintered at different temperature and characterized for apparent porosity, bulk density, phase formation, microstructure and B-H loop.

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CHAPTER 1

INTRODUCTION

1.1 Objective of the present study:

Soft-Ferrites are usually the kind of magnetic material of high magnetic initial permeability. Chemical compounds of ferrites are usually consisting with iron oxide. In general ferrites are used in transformer cores, permanent magnets and in many other applications. According to their crystal structure, ferrites have many types. They are:

1. Spinel - It has cubic structure and their general formula is AFe_2O_4.

   (A: Mn, Zn, Ni, Mg, Cu.)

2. Garnet - It has cubic structure and their general formula is $A_3Fe_5O_{12}$.

   (A: Y, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, and Lu.)

3. Ortho Ferrite - It has perovskite structure and their general formula is $AFeO_3$.

   (A: Y, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, and Lu.)

4. Magnetoplumbite- It has hexagonal crystal structure and their general formula is $AFe_{12}O_{19}$. (A: Ba, Sr, Pb)

Permeability is the measurement of the material's ability to support the formation of a magnetic field within itself. If a material is more conductive to a magnetic field then their permeability will be higher. Most of the materials including metals have permeability of 1 or near 1 including gold and copper. But iron and nickel are exceptions, they have very high permeability as compare to others metal as high as 100 or more. The metals having notable

exceptions are manganese, cobalt, nickel, iron and chromium and they are called ferromagnetic material.

Mn-Zn Ferrite has a spinel ferrite structure and they have a very high initial permeability. They have cubic structure and spinel lattice is arranged in a closed packed oxygen anions in which the unit cell is formed by 32-oxygen ions. The anions are arranged in a face centred cubic (FCC) arrangement and between the anions, two kinds of spaced are left and they are:

1. Tetrahedrally coordinated sites (A Sites) – Sites were surrounded by four nearest oxygen atom.
2. Octahedrally coordinated sites (B Sites) - Sites were surrounded by six nearest oxygen atom.

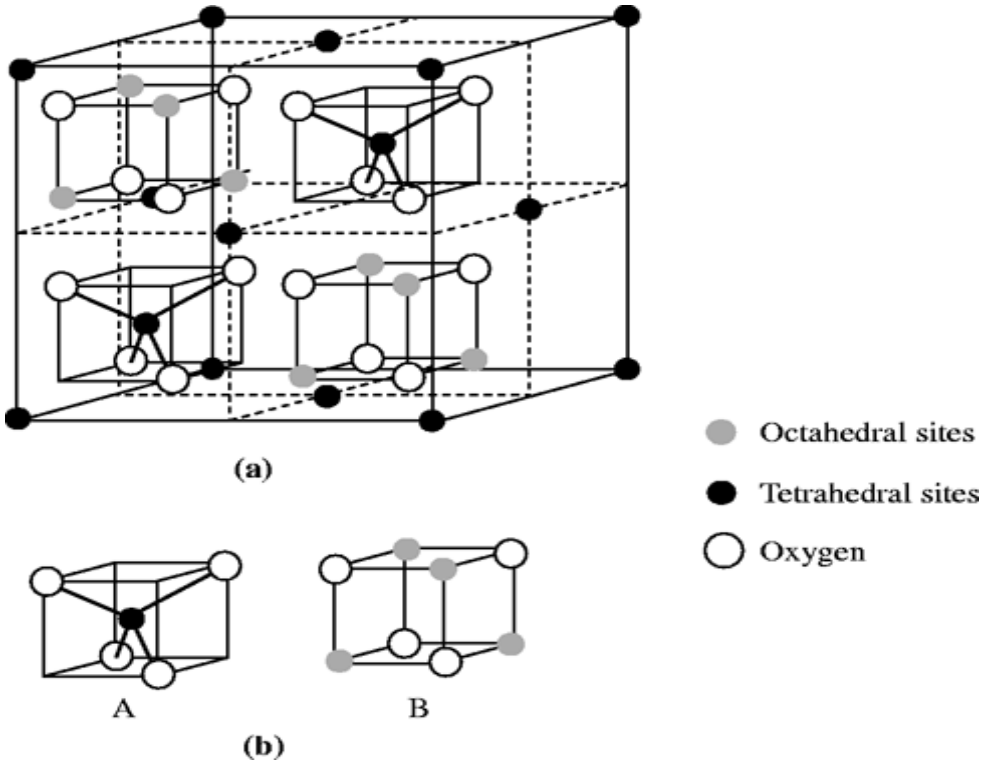

Fig 1.1: Unit Cell of the Spinel Structure (FCC) – A.

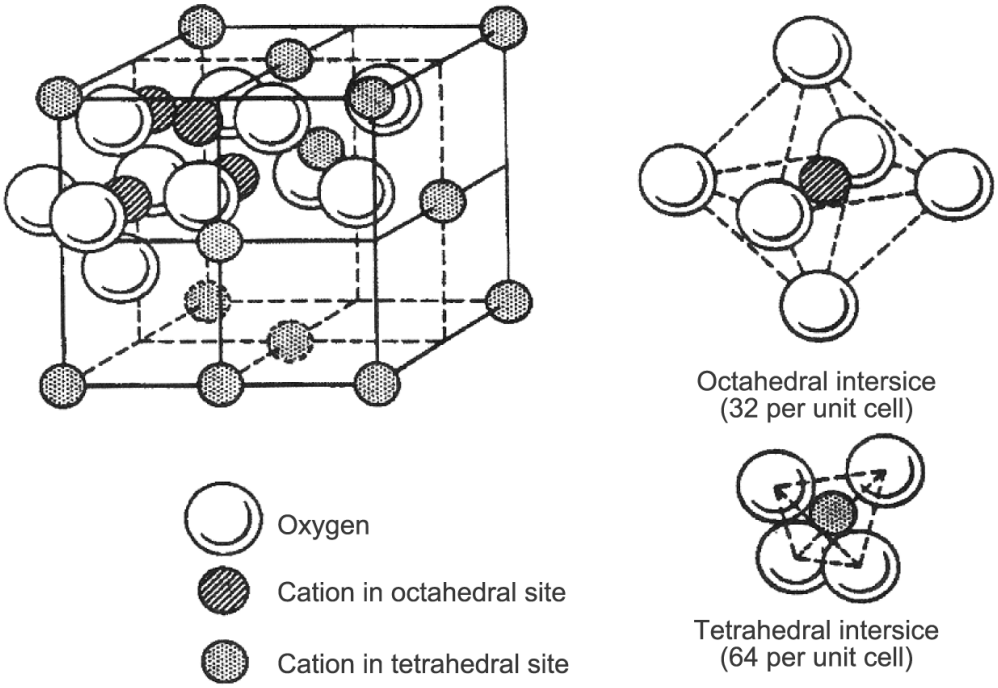

Fig 1.2: Unit Cell of the Spinel Structure (FCC) - B

In the unit cell, there are total 32 octahedral sites and 64 tetrahedral sites and out of which only 16 octahedral sites and 8 tetrahedral sites are occupied. Because of this structure, they are electrically neutral.

In spinel's, they have cubic structure and have general formula of $A(Fe_2O_4)$, where A is usually a divalent cation like zinc, manganese, magnesium, copper, cobalt or nickel. But in this project, A has two composition i.e. manganese and zinc where A is Mn_x and Zn_{1-x}. In general we used MnO_2, ZnO and Fe_2O_3 powder for the composition. MnO and Fe_2O_3 both have high magnetic permeability and low loss but it differ when changes in temperature. ZnO has high conductivity and high heat capacity, ability to sustain a large electric fields etc. The permeability of the material is depending on the sintering temperature, bulk porosity, porosity, microstructure etc.

CHAPTER 2
LITERATURE REVIEW

2.1 Introduction:

In this chapter, review of the works carried out by many researchers on the synthesis of Mn-Zn Ferrite by many different methods, influence of other composition addition on the magnetic properties and grain growth of Mn-Zn high permeability Ferrite.

C. Guillaud, Ph.D. et al found that Fe_2O_3 content in the composition should be between 49.7 and 50.6 mol % to acquire a high initial permeability and low loss in ferrite [1]. The average grain diameter size has been limited at maximum upto 20 microns otherwise pores will appear in its grain boundary as well as in the grain itself and this will lower the initial permeability by locking of the wall.

K. Janghorban, et al found that there is a noticeable on the development of microstructure by the influence of V_2O_5 and two mechanisms were proposed for the grain growth promotion of V_2O_5 [2]. The concentration of FeO is also increased by the addition of V_2O_5. About 0.02 wt. % of V_2O_5, there is about 42% of magnetic permeability increased. Upto 1 wt. % of V_2O_5 increases the Curie temperature by 50 %. V_2O_5 beyond about 0.02 %, there is decreases in the linear shrinkage.

Kaigi Jiang et al found that influence of $CaCO_3/SiO_2$ improve the initial permeability's frequency stability at wider frequency but decreases the initial permeability [3]. Suitable amount of B_2O_3 can increase the activity of the grain growth but it decrease the porosity of ferrite. When concentration of B_2O_3 is 0.03 wt. %, then the Mn-Zn Ferrite has large homogeneous grain size and it also have a lowest porosity so they have a very highest initial porosity.

H. Shokrollahi et al found that electrical and magnetic properties of ferrite depend on microstructures control [4]. As compare to porosity of microstructure, grain size is more important which affect the magnetic properties of high permeability ferrite. For the grain growth promotion in Mn-Zn Ferrite, one important mechanism is to reduce the pore drag and impurity on the grain boundary motion. Another possible mechanism is the increased pore mobility due to the excess cation vacancies creation.

K. Mandal et al found that spinel ferrite $Mn_{0.5}Zn_{0.5}Fe_2O_4$ in SiO_2 matrix with less than 20nm dimension can be prepared by sol gel method [5]. This ferrite shows super paramagnetic behaviour when the particle size is less than 20nm.

CHAPTER 3
EXPERIMENTAL

PROCEDURE

3.1 Batch Calculation of Ferrite ($Mn_{0.5}$ $Zn_{0.5}$ Fe_2O_4)

1. ZnO = 81.37

   NICE Chemical – Laboratory reagent

   Assay = 99.5 %

2. MnO_2 = 86.94

   LOBA CHEMIE PVT. LTD

   Minimum assay = 70%

3. Fe_2O_3 = 159.69

   s.d. fine – CHEM Ltd

   Minimum assay = 95.0 %

Reagent Name	Molecular Weight	Assay
Fe_2O_3	159.69	95%
ZnO	81.37	99.50%
MnO_2	86.94	70%

Table 3.1: Reagent name, molecular weight and assay of the initial composition

Sample is $Mn_{0.5}$ $Zn_{0.5}$ Fe_2O_4 {MnO_2 (0.5 mol) + ZnO (0.5 mol) + Fe_2O_3 (1 mol) }.

1 mol of Fe_2O_3 of 95 % = 15.969 gm.

0.5 mol of ZnO of 99.5 % = 4.0685 gm.

0.5 mol of MnO_2 of 70 % = 4.347 gm.

If Fe_2O_3, ZnO and MnO_2 are 100 % pure then,

Fe_2O_3 = (15.969/95) × 100 = 16.809 gm.

ZnO = (4.0685/99.5) × 100 = 4.0889 gm.

MnO_2 = (4.347/70) × 100 = 6.21 gm.

Therefore, the final weight I took is,

Fe_2O_3 = 16.809 gm.

ZnO = 4.0889 gm. and

MnO_2 = 6.21 gm.

First, the composition Fe_2O_3, ZnO and MnO_2 having weight of 16.809 gm. 4.0889 gm. and 6.21 gm. are taken in a container. The three compositions are mixed in a container by adding iso-propanol because iso-propanol does not react with the composition. If water or any other solution were used instead of iso-propanol, it may react with the compositions and may change the chemical properties of the compositions. After adding iso-propanol to the compositions, shacked it well to wet all the composition powder by iso-propanol. After that around 10 balls were put inside a pot and do mixing by pot milling for 6-8 hours and then the composition pasted is poured in a Petridis and it is covered by aluminium sheet to prevent from impurity.

3.2 Synthesis of Ferrite Powder and pellet:

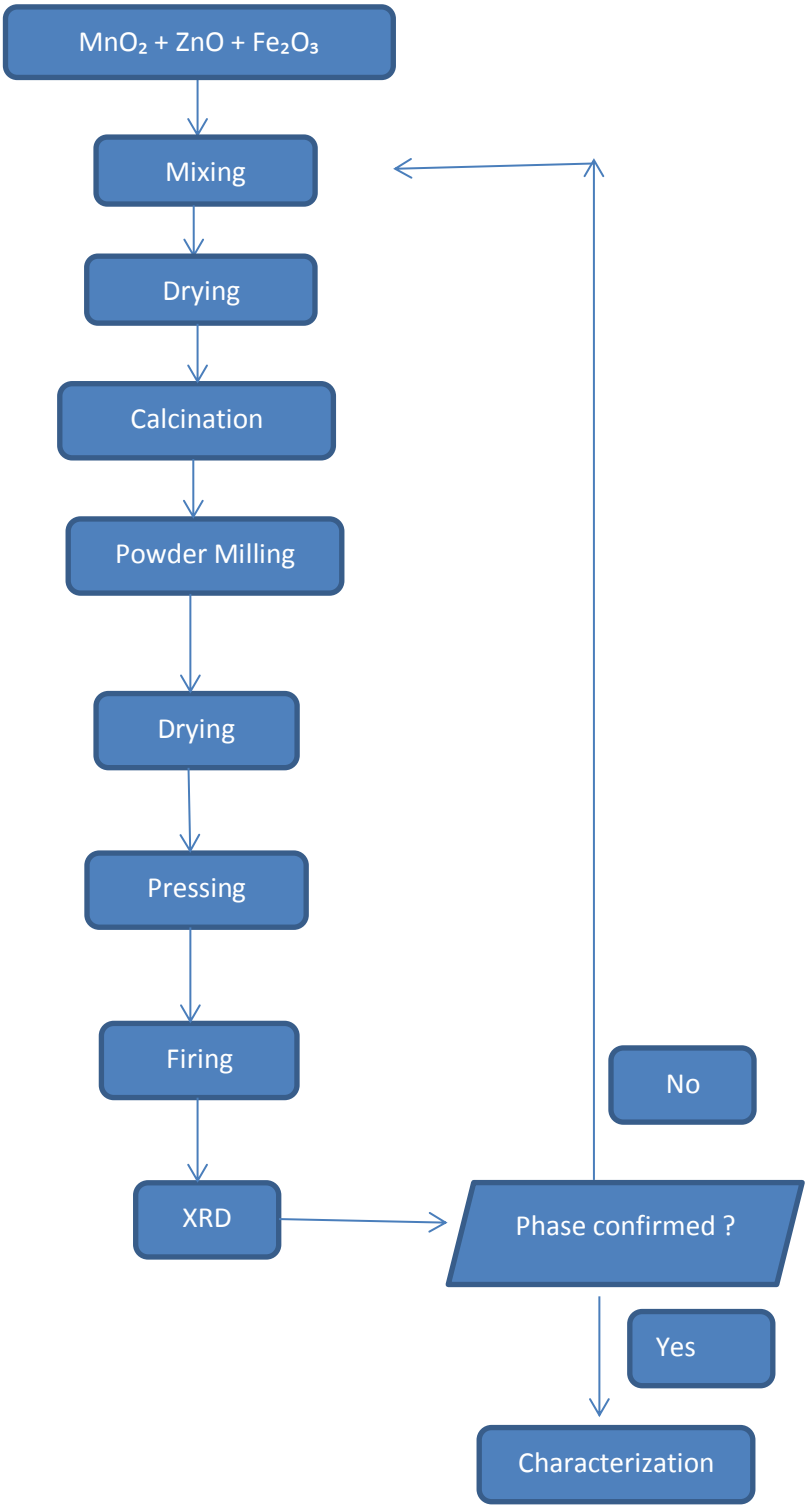

Fig 3.1: Flow Chart for Synthesis of Mn-Zn ferrite.

For comparison of the properties, a composition was also made with different amount of same composition. So, a total of two compositions were made i.e. $Mn_{0.5}Zn_{0.5}Fe_2O_4$ and $Mn_{0.4}Zn_{0.6}\ Fe_2O_4$.

Amount of MnO_2, ZnO and Fe_2O_3 taken in the composition of $Mn_{0.5}Zn_{0.5}Fe_2O_4$ and $Mn_{0.4}Zn_{0.6}\ Fe_2O_4$ are given in the following chart:

Composition	MnO_2	ZnO	Fe_2O_3
$Mn_{0.5}Zn_{0.5}Fe_2O_4$	6.21 gm.	4.0889 gm.	16.809 gm.
$Mn_{0.4}Zn_{0.6}\ Fe_2O_4$	4.968 gm.	4.906 gm.	16.809 gm.

Table 3.2: Batch calculation of the Mn Zn-Ferrite compositions.

After pot milling by adding iso-propanol in the three different composition of powder for one whole day, the powder is dried in an electrical drier for 2 days. Then calcination of powders was done at 950 °C for 2 hours. After calcination, milling of powder is done and then again dried the powder.

3.3 Powder Characterization:

Phase Analysis

By performing X-ray diffraction with a Philip's Diffractometer (model: PW-1830, Philips, Netherlands) the phase formation in the burnt and calcined powder were studied. After performing X-ray diffraction with a Philip's Diffractometer, a graph was plotted X-rays intensity against the angle theta (2θ). By using the Bragg's law i.e. $n\lambda = 2d \sin\theta$, angle of each diffraction was converted to d-spacing.(where 'n' is order of diffraction and 'λ' is the wavelength of x-ray. By using Philips X-pert high score software, different phases present in the calcined powder were identified.

3.4 Fabrication and Sintering of Ferrite Samples

After calcination, calcined powder of each composition is mixed thoroughly with 2 wt. % of binder i.e. polyvinyl alcohol.

Firstly, calcined powders of each composition were mixed with the PVA binder separately for 1 hour. The powders mixed with PVA binder were dried. It was observed that nice granules of ferrite powder were produced where that exhibited good flow ability and compressibility. Then the granulated powders were uniaxially pressed at a load of 4 TON for 90 sec by using a hydraulic press to form a pellet of 12.5 mm diameter. Green density of the green pellet was calculated from weight/volume. From the specimen dimension, volume was calculated and weight was measured by using electronic balance. Then sintering of the green samples were carried out by firing the sample in an electric furnace at 1300 °C for 4 hours at the rate of 3°C per minute.

3.5 Characterization of sintered specimen

Various physical properties of the sintered pellet such as bulk density, apparent porosity, phase analysis, microstructural analysis and BH-loop measurement were carried out.

3.5.1 Apparent Porosity and Bulk Density:

By using Archimedes principle, bulk density and apparent porosity of sinter pellet were determined. By using electronic balance, dry weights of the sintered pellet were noted down. Then the soaked weight and suspended weight of the samples were also measured after immersed the sintered sample in a beaker that containing water and kept on chamber to make vacuum the beaker so as to ensure all the open pores were filled up with water completely. Then, the apparent porosity and bulk density were calculated by using the following formula:

$$Apparent\ Porosity\ = \frac{W - D}{W - S}$$

$$Bulk\ Density\ = \frac{D}{W - S}$$

Where, Dry weight of the sample = D, Soaked weight of the sample = W, Suspended weight of the sample = S.

3.5.2 Phase Analysis:

By performing X-ray diffraction with a Philip's Diffractometer (model: PW-1830, Philips, Netherlands) the phase formation in the sintered samples were studied and different phases present in the calcined powder were identified by using Philips X-pert high score software.

3.5.3 Microstructural Analysis:

By using Scanning Electron Microscopy (SEM), the microstructures of the sintered samples were analysed. Firstly, under vacuum condition the samples were thinly coated by palladium to make the surface conducting for viewing through SEM. The mounted specimens were studied by SEM (JEOL-JSM 6480 LV).

3.5.4 BH-loop Measurement:

For magnetic property measurement one ‘Magneta’ Pulse field magnetic loop tracer was used where the inserting the samples of tablet form was very easy. The applied magnetic fields were varied from -6000 to +6000 Oe. Before putting the sample dry weight of the samples were taken and calibration was accomplished with that instrument.

Chapter 4

Results and Discussion

4.1 Bulk Density:

The following tables show the apparent porosity, bulk density and % of theoretical density of Mn-Zn Ferrite having different composition and sintered at two different temperatures. First group of pellets were sintered at 1350^{o}C and the second group were sintered at 1300^{o}C. The soaking time of isothermal sintering was given as 4 hour for each group.

Composition	Temperature (°C)	Bulk Density	Apparent Porosity (%)	% of Theoretical Density
$Mn_{0.5}Zn_{0.5}Fe_2O_4$	1300	4.36	3.44	87.04
$Mn_{0.5}Zn_{0.5}Fe_2O_4$	1350	4.43	4.2	88.44

Table4.1: Bulk density and apparent porosity value with % theoretical density of the ferrite having composition as $Mn_{0.5}Zn_{0.5}Fe_2O_4$.

Composition	Temperature (°C)	Bulk Density (B.D.)	Apparent Porosity (%) (A.P.)	% of Theoretical Density
$Mn_{0.4}Zn_{0.6}Fe_2O_4$	1300	4.1	7.1	81.85
$Mn_{0.4}Zn_{0.6}Fe_2O_4$	1350	4.23	7.23	84.44

Table4.2: Bulk density and apparent porosity value with % theoretical density of the ferrite having composition as $Mn_{0.4}Zn_{0.6}Fe_2O_4$.

As significant impurity phases were found in both calcined powder and sintered product, another three new compositions of ferrite were made by grinding the calcined powder very well taking in agate mortar first and then recalcined the powder at 950 ^{o}C. The sintered temperature was chosen as 1300 °C with same isothermal holding time.

Composition	Bulk Density	Apparent Porosity (%)	% of Theoretical Density
$Mn_{0.5}Zn_{0.5}Fe_2O_4$	4.8	3.2	95.8
$Mn_{0.4}Zn_{0.6}Fe_2O_4$	4.6	7.3	91.8

Table4.3: Two different composition of ferrite sintered at 1300 °C.

When the temperature is increased, the apparent porosity is decreased and the bulk density is increased. So theoretical densities were increased accordingly. But the increased B.D. values were not found to be increased significantly.

4.2 Structural Characterization:

4.2.1 X-Ray Diffraction Analysis:

4.2.1.1 Calcined Powder Characterization:

Firstly phase characterizations of two different composition of calcined powder at 950^{o}C for 2 hours were given below:

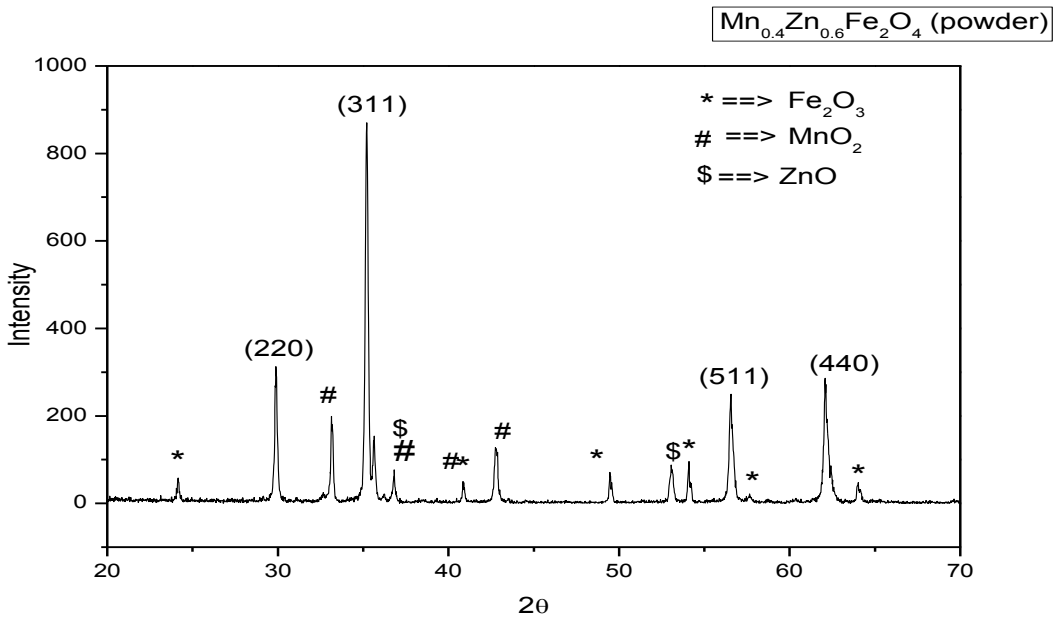

Fig4.1: XRD plot of $Mn_{0.4}Zn_{0.6}Fe_2O_4$ calcined powder.

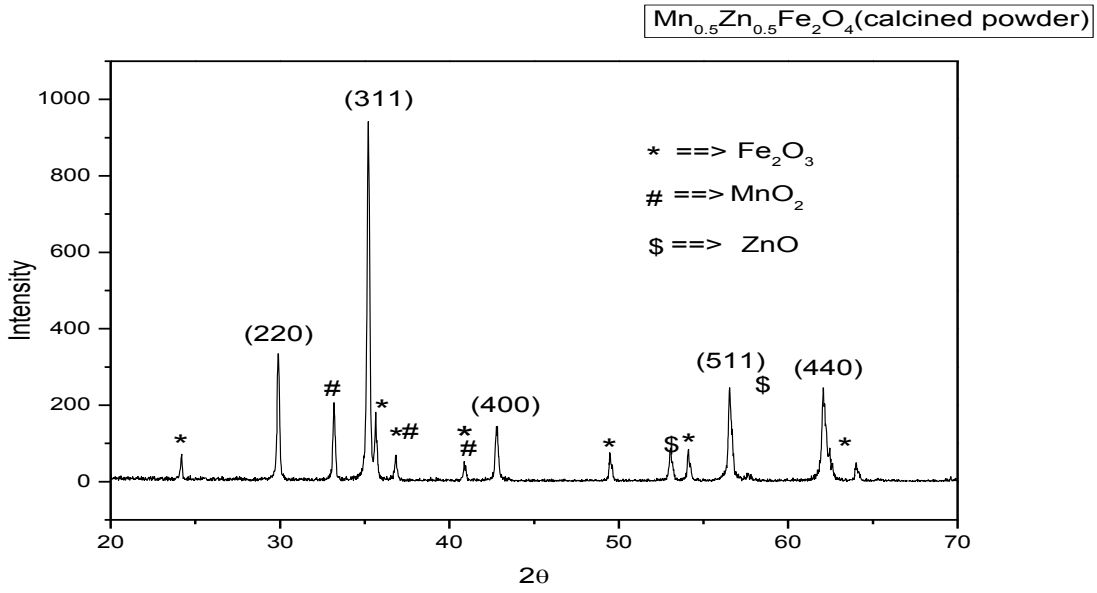

Fig4.2: XRD plot of $Mn_{0.5}Zn_{0.5}Fe_2O_4$ calcined powder.

In both the powder composition, there were lots of unreacted initial compounds were present i.e. MnO_2, ZnO and Fe_2O_3 but the main compound is Fe_2O_3.

4.2.1.2 Sintered Pellet Characterization:

Pellets were made from the calcined powder and sintered at two different temperatures i.e. 1300°C and 1350°C. Phase characterizations of the pellet were given below:

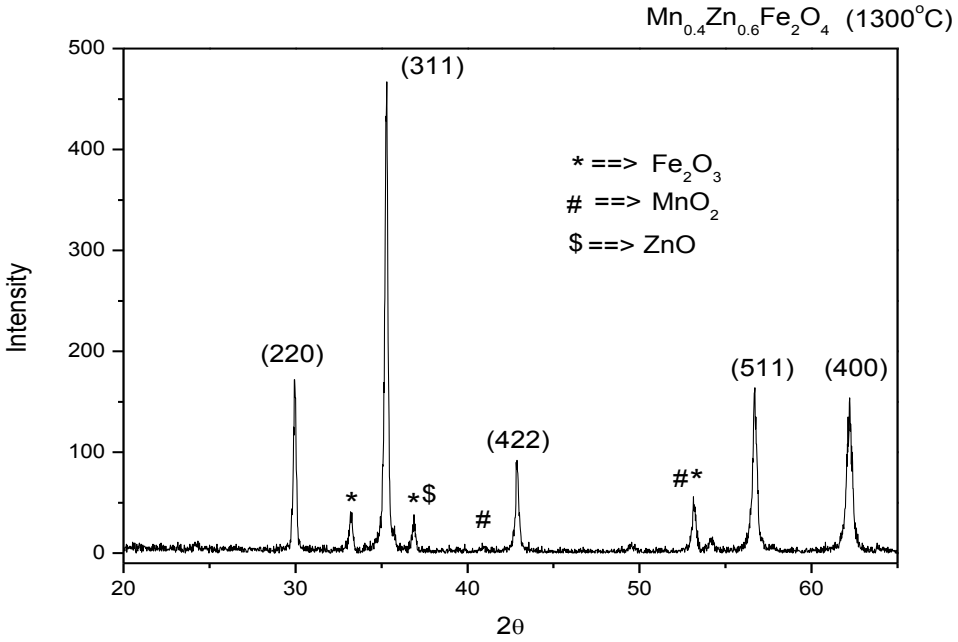

Fig4.3: $Mn_{0.4}Zn_{0.6}Fe_2O_4$ composition pellet sintered at 1300 °C.

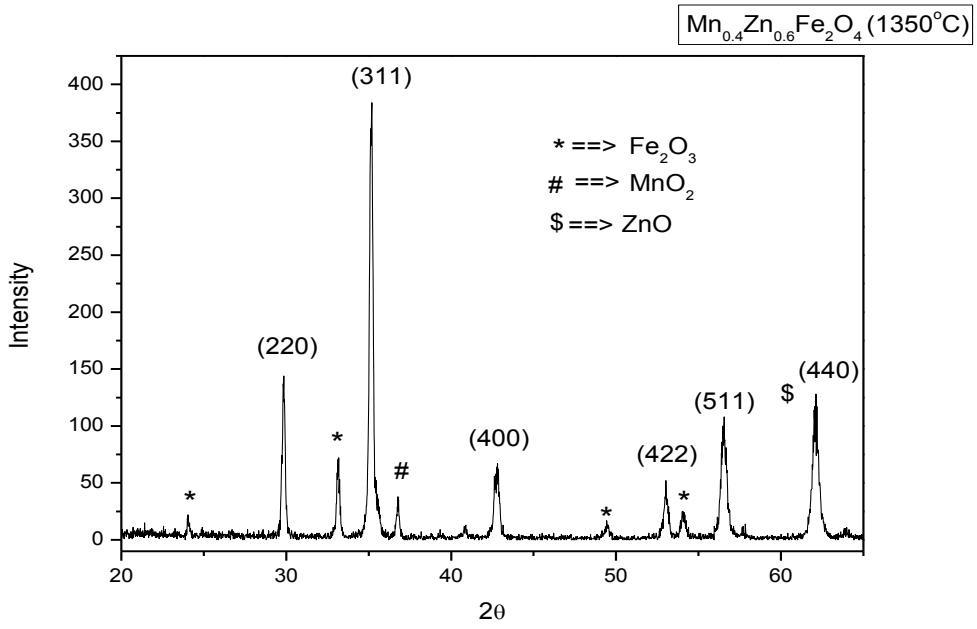

Fig4.4: $Mn_{0.4}Zn_{0.6}Fe_2O_4$ composition pellet sintered at 1350 °C.

In these two XRD plot, the composition sintered at 1300ºC had more spinel formation than the others. So, pellet will be sintered only at 1300°C.

The following two graphs is the comparison of phase formation and unreacted compound present in two different composition sintered at 1300°C:

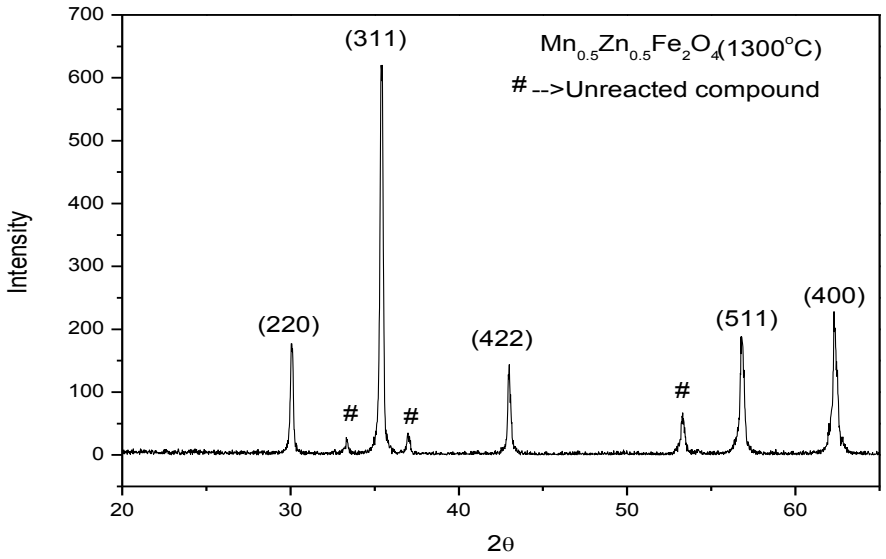

Fig4.5: $Mn_{0.5}Zn_{0.5}Fe_2O_4$ composition pellet sintered at 1300 °C.

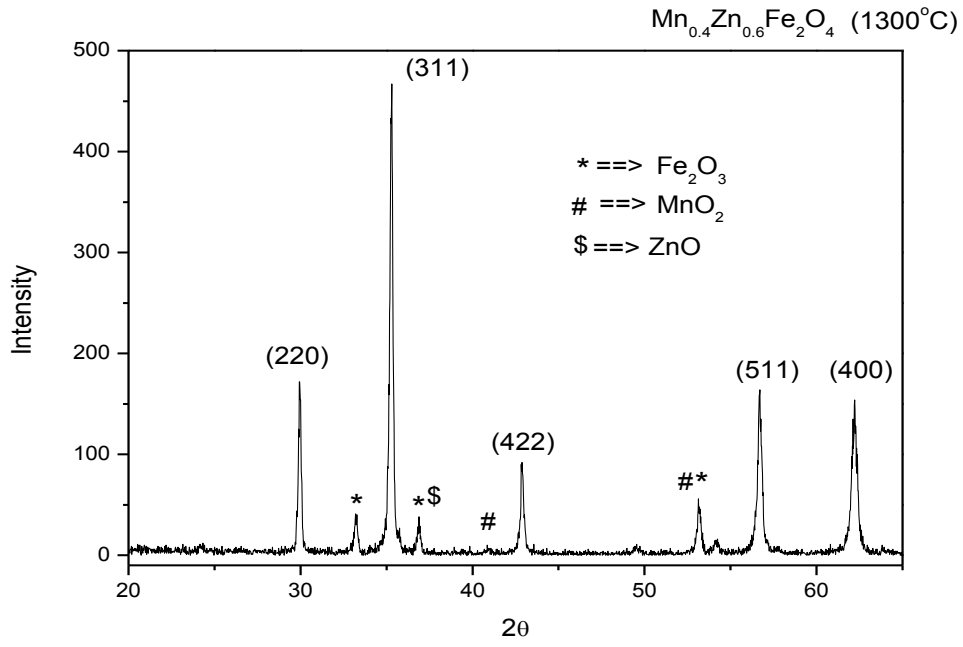

Fig4.6: $Mn_{0.4}Zn_{0.6}Fe_2O_4$ composition pellet sintered at 1300 °C.

In these two compositions, there is less unreacted compound present in the $Mn_{0.5}Zn_{0.5}Fe_2O_4$ composition sintered pellet and also spinel formation is also high as they have high peak value than $Mn_{0.4}Zn_{0.6}Fe_2O_4$ composition sintered pellet.

In order to decrease unreacted compound present in the sintered composition, another three compositions were made. Two compositions were having same percentage of MnO_2 and ZnO but differ in the contained of Fe_2O_3 i.e. $Mn_{0.4}Zn_{0.6}Fe_2O_4$ (95% Fe2O3), $Mn_{0.4}Zn_{0.6}Fe_2O_4$ (100% Fe2O3) and $Mn_{0.5}Zn_{0.5}Fe_2O_4$.

Comparisons of XRD plot for these three calcined powder compositions were given below and it is found that composition $Mn_{0.4}Zn_{0.6}Fe_2O_4$ (95% Fe_2O_3) has more spinel formation and less unreacted compound present than the other two composition i.e. $Mn_{0.4}Zn_{0.6}Fe_2O_4$ (100% Fe_2O_3) and $Mn_{0.5}Zn_{0.5}Fe_2O_4$.

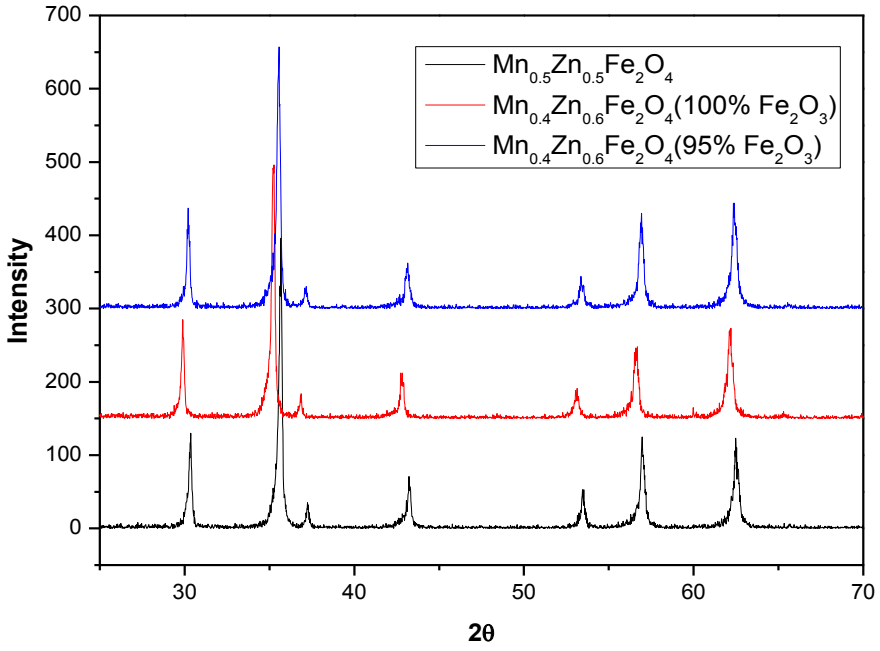

Fig4.7: XRD plot comparison of three new compositions.

The following XRD plots were the comparisons of these two compositions having same MnO_2 and ZnO contained but differ in the contained of Fe_2O_3 i.e. 95% Fe_2O_3 and 100% Fe_2O_3 contained.

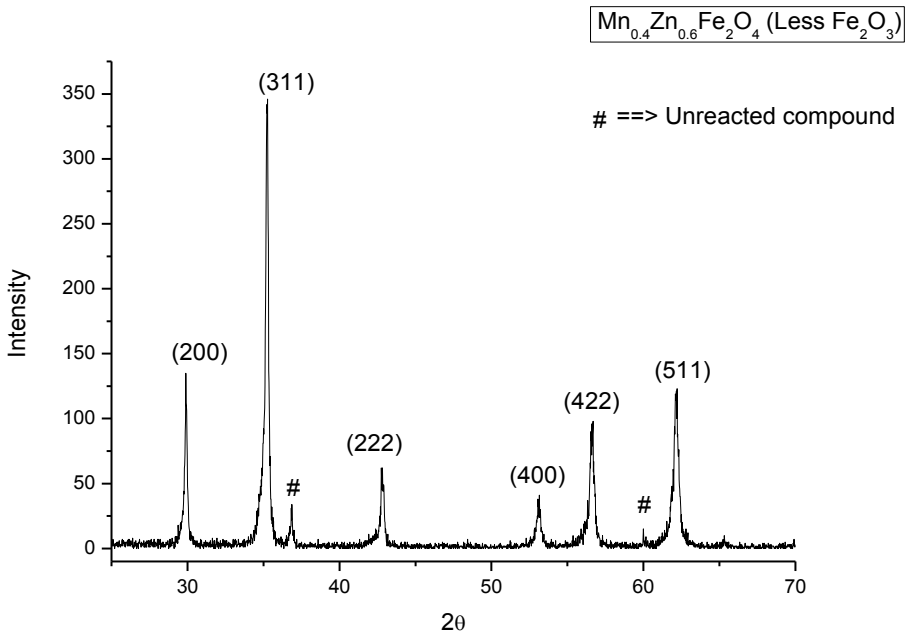

Fig4.8: Composition having 95% Fe_2O_3.

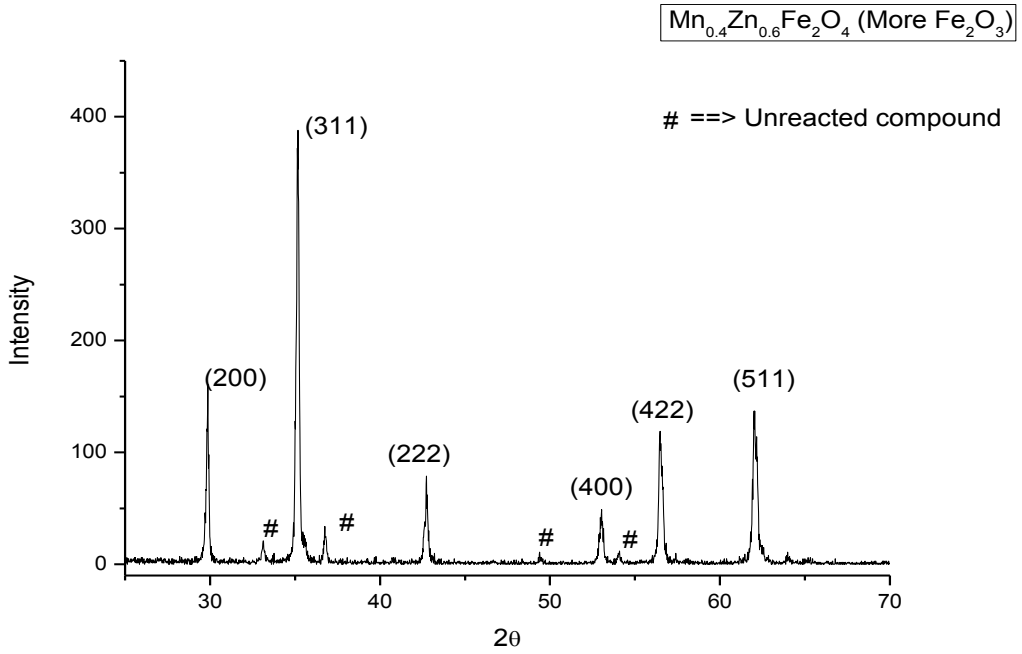

Fig4.9: Composition having 100% Fe_2O_3.

In these two XRD plot, there was decreased in the contained of unreacted Fe_2O_3 in the composition having 95% Fe_2O_3 contained.

4.2.2 Microstructural Characterization:

Microstructures were studied by placing as sintered sample under electron microscope. The following figures show the microstructure of different ferrite composition sintered at 1300 ℃.

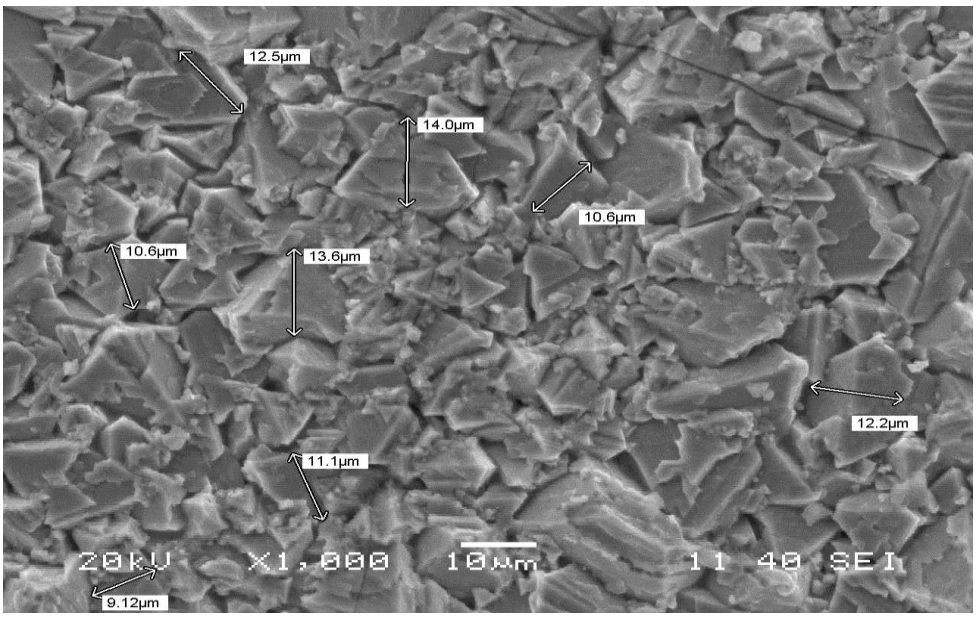

Fig4.10: Microstructure of $Mn_{0.5}Zn_{0.5}Fe_2O_4$ composition sintered at 1300 ℃.

The average grain size of $Mn_{0.5}Zn_{0.5}Fe_2O_4$ composition sintered at 1300 ℃ is 11.715µm.

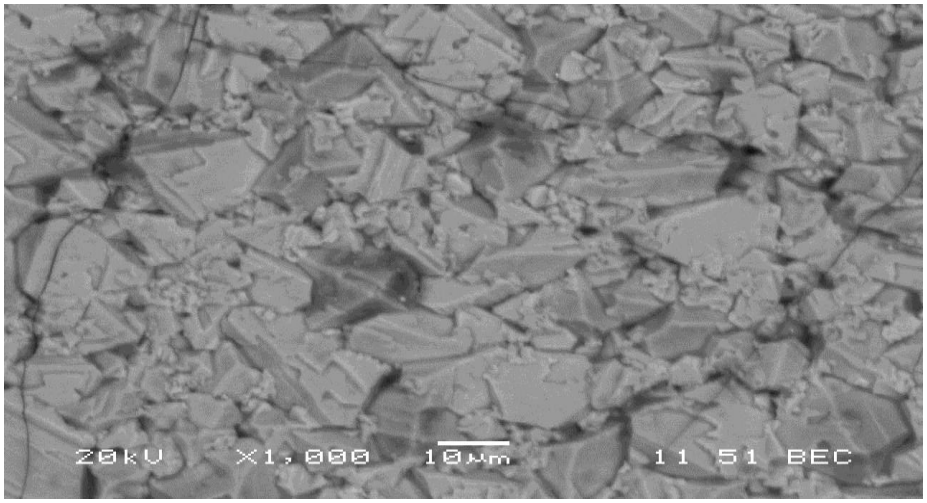

Fig4.11: Back scattering microstructure of $Mn_{0.5}Zn_{0.5}Fe_2O_4$ composition sintered at 1300℃.

It was suspected that the dark portions over the grains may be the unreacted Fe_2O_3 along with white portions which are clearly the spinel phase.

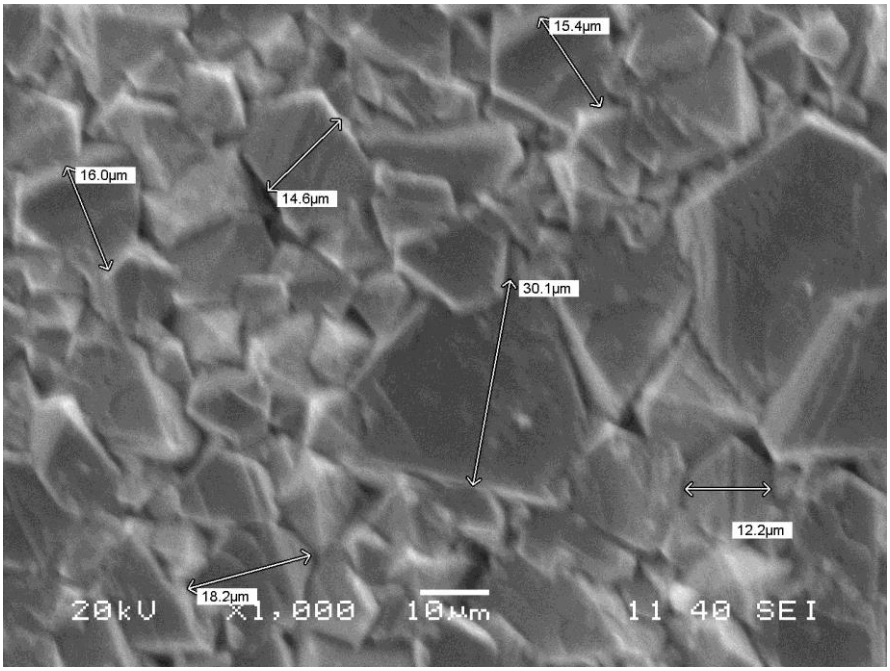

Fig4.12: Microstructure $Mn_{0.4}Zn_{0.6}Fe_2O_4$ of composition sintered at 1300 ℃.

The average grain size of $Mn_{0.4}Zn_{0.6}Fe_2O_4$ composition sintered at 1300 ℃ is 17.75 µm.

The following table shows the different in grain size for different two ferrite compositions sintered at 1300 ℃:

Composition	Average Grain Size
$Mn_{0.5}Zn_{0.5}Fe_2O_4$	11.715 µm.
$Mn_{0.4}Zn_{0.6}Fe_2O_4$	17.75 µm.

Table4.4: Comparison of grain size between the two compositions i.e. $Mn_{0.5}Zn_{0.5}Fe_2O_4$ and $Mn_{0.4}Zn_{0.6}Fe_2O_4$.

The composition $Mn_{0.5}Zn_{0.5}Fe_2O_4$ had the lower and homogeneous grain size as compare to the other composition.

4.3 BH - loop:

The BH-loop of the three compositions was as followed:

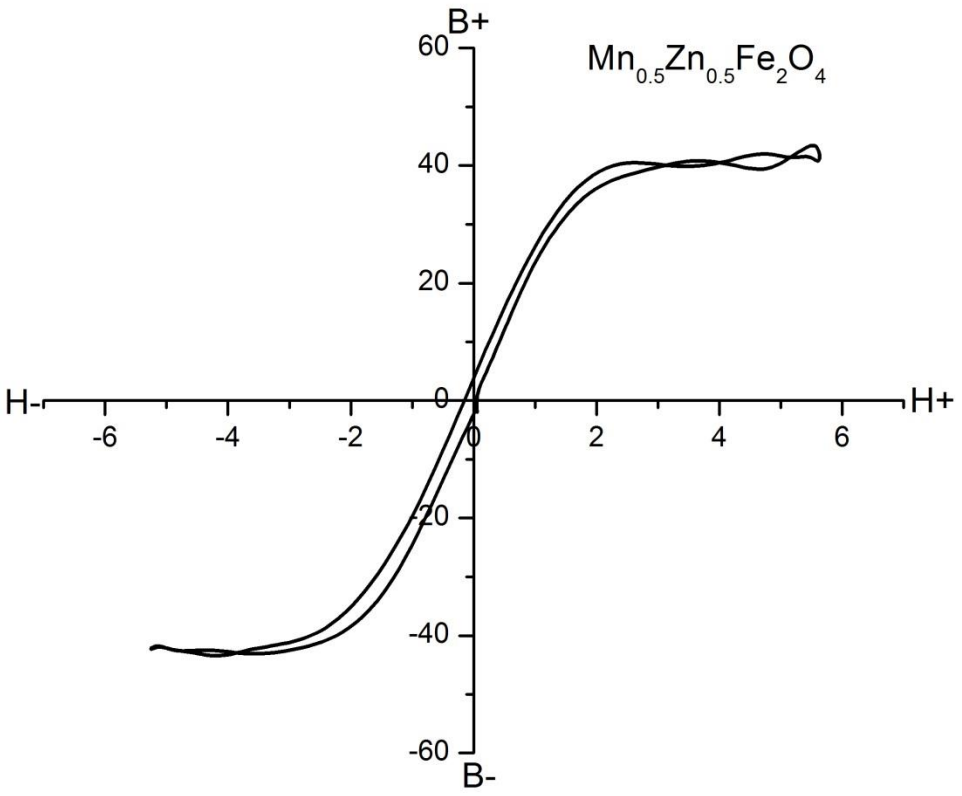

Fig4.13: BH-loop of $Mn_{0.5}Zn_{0.5}Fe_2O_4$.

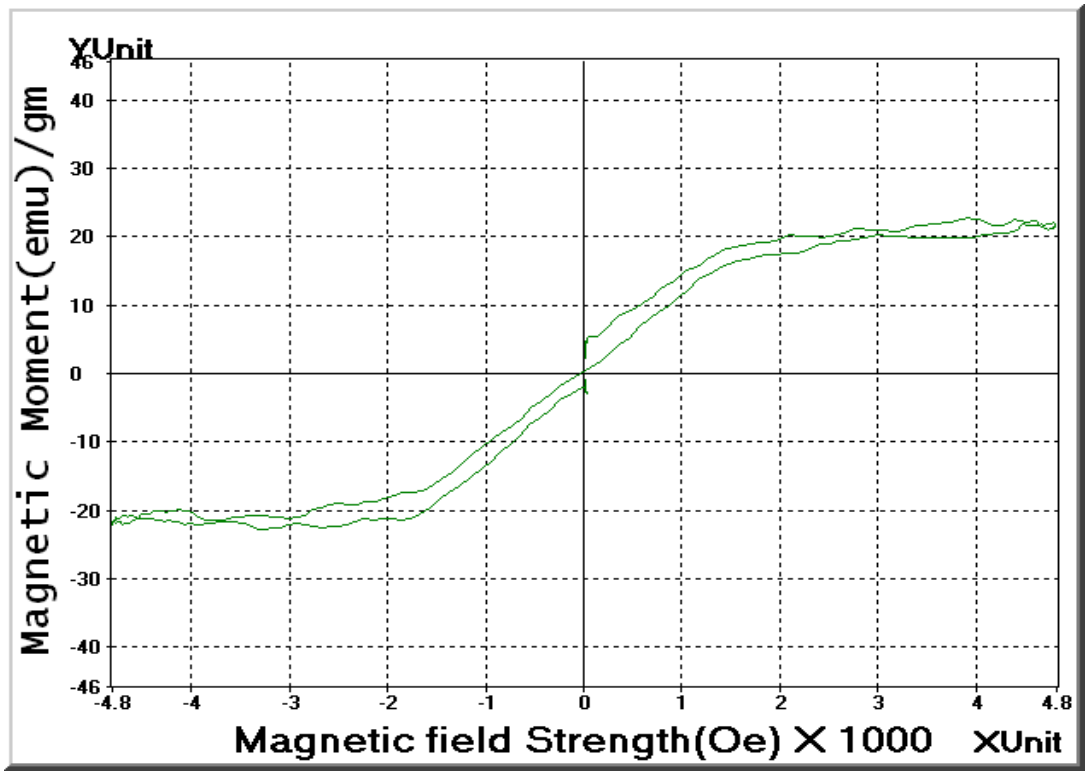

Fig4.14: BH-loop of $Mn_{0.4}Zn_{0.6}Fe_2O_4$ (100% Fe_2O_3).

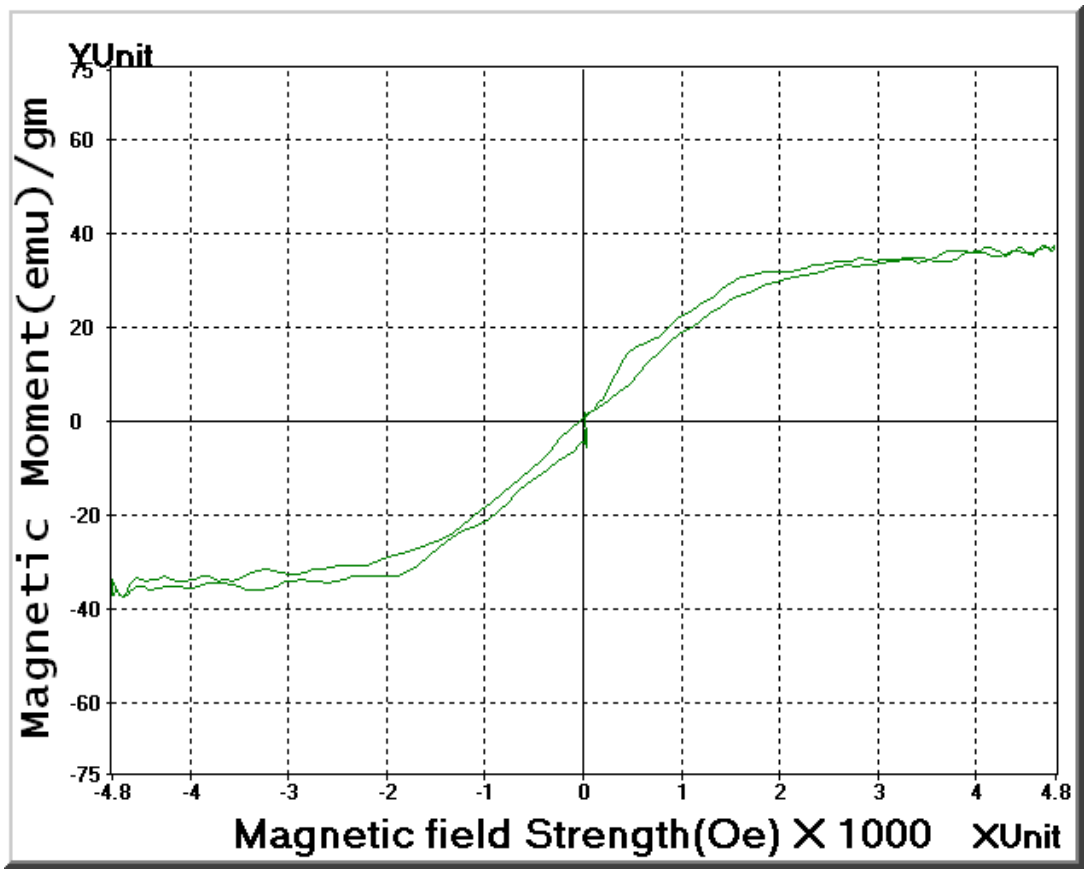

Fig4.15: BH-loop of $Mn_{0.4}Zn_{0.6}Fe_2O_4$ (95% Fe_2O_3).

Composition	M_S (emu/gm.)	M_r (emu/gm.)	H_C (Oe)
$Mn_{0.5}Zn_{0.5}Fe_2O_4$	40.04	4.24	0.07
$Mn_{0.4}Zn_{0.6}Fe_2O_4$ (100%)	22.77	0.99	40.61
$Mn_{0.4}Zn_{0.6}Fe_2O_4$ (95%)	37.5	1.04	14.3

Table4.5: Saturation magnetization (M_S), Remnant magnetization (M_r) and Coercive Field values of three different compositions.

Comparing to these three compositions, $Mn_{0.5}Zn_{0.5}Fe_2O_4$ has a high saturation magnetization then $Mn_{0.4}Zn_{0.6}Fe_2O_4$ (100%) and $Mn_{0.4}Zn_{0.6}Fe_2O_4$ (95%) and the $Mn_{0.4}Zn_{0.6}Fe_2O_4$ composition having less Fe_2O_3 $Mn_{0.4}Zn_{0.6}Fe_2O_4$ (95%) have more saturation magnetization than the other one i.e. $Mn_{0.4}Zn_{0.6}Fe_2O_4$ (100%).

Chapter 5

Conclusions

1. The sintering temperature of two ferrite composition is 1300 ºC at normal atmosphere.
2. First, the Fe_2O_3 impurity in product was minimized by recalcination of powder at 950 ºC for 4hour. The unreacted F_2O_3 was again minimized by modifying the batch calculation.
3. $Mn_{0.5}Zn_{0.5}Fe_2O_4$ had the highest bulk density and low porosity than the other composition.
4. For achieving a high permeability, grain homogeneous and not size with be not so high otherwise there will be decrease in permeability.
5. Ferrite composition having $Mn_{0.5}Zn_{0.5}Fe_2O_3$ shows more saturation magnetization and also when decreased a little percentage of Fe_2O_3 (large unreacted compound present in the sintered pellet is Fe_2O_3) in the composition, there will be increased in the saturation magnetization.

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