CHAPTER - IV

HYSTERESIS BEHAVIOUR

Seek simplicity and distrust it

- Alfred North Whitehead

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HYSTERESIS BEHAVIOUR

INTRODUCTION

Magnetization is one of the important and fundamental parameters of oxide magnetic materials that decides upon their applications. Ceramic magnets are fabricated artificially to the required degree of magnetization by changing the concentration of constituents.

The studies on hysteresis provides a valuable information about saturation magnetization, permeability, coercivity and remanance.

The materials with low Hc are called 'soft' magnetic materials and are widely used in high frequency applications. For making permanent magnets, 'hard' magnetic materials with large Hc are used. The coercive force of ferrites is found to vary from 10^{-1} to 10^{+3} Oe [1]. The magnetic properties are very much sensitive to their chemical composition, crystal structure and microstructure. These properties are also affected by heat treatment, sintering atmosphere and rate of cooling. The porosity and grain size are found to be the influencing factors in controlling the magnetic properties of ferrites [2-4]. The basic magnetic properties of ferrites can be characterized by hysteresis technique, A.C. susceptibility and permeability.

4.1 MAGNETIZATION IN FERRITES

The magnetization in ferrites results from the distribution and alignment of magnetic ions on the tetrahedral and octahedral sites. If magnetic ions are sufficiently close to each other, the spontaneous magnetization (Ms) occurs even in the absence of external field. The electron can undergo an exchange interaction between neighbouring magnetic atoms or ions. This may be direct or may take place via an interatomic interaction. It may be either +ve or –ve and it is based on parallel and antiparallel alignment of the spins of neighbouring atoms respectively. When the exchange interaction is +ve it is ferromagnetic and the alignment is parallel and when it is –ve, then it is either anti-ferromagnetic or ferrimagnetic depending upon complete or incomplete cancellation of magnetic moments within the crystals. Gorter arrived at a scheme of spin ordering on the basis of Anderson's concept of super exchange coupling to arrive at a reasonably good value of saturation magnetization.

The saturation magnetization in ferrites is determined by its physico-chemical constitution and also be the cation distribution and other aspects of thermo-physical history. In case of ferrite the solid solubility affords to prepare mixed ferrites, crystallizing in the spinel structure either of normal or of the inverse type. The normal spinels are generally non-magnetic while inverse spinels are ferrimagnetic. For

the ferrite with inverse spinel structure, cation distribution is expressed as

$$[Fe^{3+}]_A\,[M^{2+}\,Fe^{3+}]_B O_4^{2-}$$

The Fe^{3+} ions on A site are coupled with their spin antiparallel to those Fe^{3+} ions on B site so that, net moment is due to divalent M^{2+} metallic ions

$$[Fe^{3+}\,A\uparrow]\;\;[Fe^{3+}\,B\downarrow 5\mu_B\,M_B^{2+}\downarrow]$$

The indirect exchange coupling between metallic ions acting through the angle AOB is close to 180^0. For perfect spinel lattice the major angles between the ions are ABO ($125^0 9'$ and $154^0 34'$), BOB (90^0 and $125^0 9'$) and AOA ($79^0 38$) as shown in fig. 4.1. The nearest to perfect alignment are Fe_A and Fe_B ions making an angle of $154^0\,34'$, thus giving the strong antiferomagnetic coupling [5].

There are two kinds of magnetic properties i.e. intrinsic and structure sensitive. The intrinsic properties of the ferrites are magnetic anisotropy (k), magnetostriction (λ), Curie temperature (Tc) and saturation magnetization (Ms) while the properties like permeability (μ) hystersis and magnetic loss (tans δ) and susceptibility are structure sensitive.

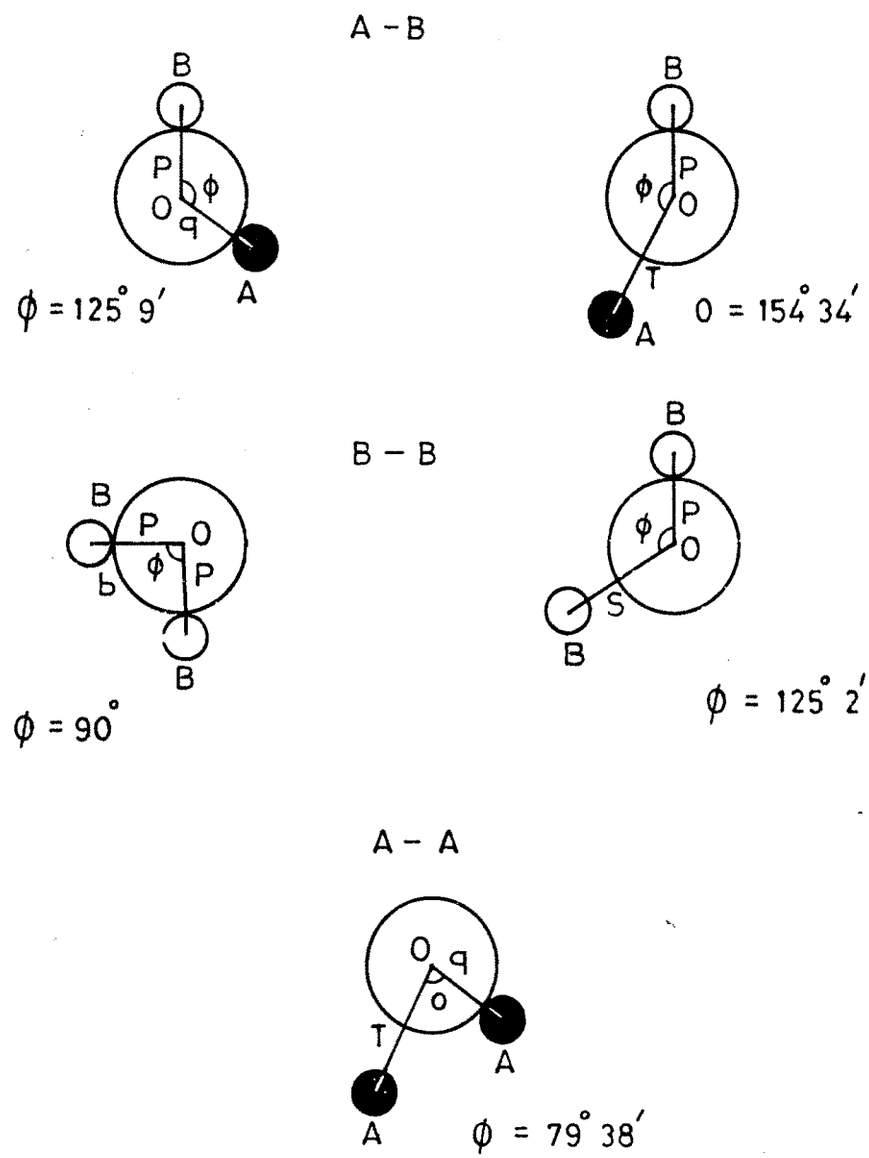


FIG.4- -1 - ANGLE BETWEEN A-A B-B & A-B CATION IN A SPINEL STRUCTURE.

4.2 HYSTERESIS

Hysteresis is the phenomenon in which magnetic induction lags behind the applied magnetic field. Hysteresis study on ferrites provides a valuable information on permeability, saturation magnetization, coercive force and remanence ratio. These are helpful in deciding the nature of application of ferrites. With the help of modern hystersis setup, it is possible to observe directly the shape of the hysteresis loop on the oscilloscope screen. The hysteresis properties mainly depend on the chemical composition, crystal structure, cation distribution and microstructure [3,4].

In ferrites, the oxygen on bonding gives rise to two sublattices, characterised by different oxygen environment for metal atoms. Magnetic interaction within the sublattice give rise to sublattice magnetization. Magnetization of two sublattices are opposite but are unequal, so a net magnetization results in case of ferrimagnetic materials.

A demagnetized ferromagnetic material exhibits a state of zero magnetization. This was first explained by Weiss [5]. In ferromagnetic materials, there are atoms with the magnetic moment arising from the unpaired spins tending to interact strongly with one other to cause parallel alignment of nearby moments. Weiss postulated that the existence of molecular field is responsible for spontaneous alignment of

these magnetic dipoles. He further suggested that a ferrimagnetic or a ferromagnetic material is divided into small regions called domains which are spontaneously magnetized in the block of unmagnetised material. These Weiss domains in the absence of external magnetic field are randomly oriented resulting in zero magnetization. Moreover these magnetic domains are separated by a thin layer in which the direction of magnetization changes from one orientation to another, characteristic of the domain concerned. This transition boundary is known as domain wall or Bloch wall [6]. Thus when the specimen is placed in the external magnetic field magnetization develops in the specimen. This takes place in two ways viz.-

(1) domain wall motion
(2) rotation of magnetization

When the applied magnetic field is small, magnetization is achieved by domain wall movement which is reversible. With further increase in field magnetization increases with irreversible domain wall movement and finally at high field saturation magnetization is reached by rotation of domain magnetization as shown by the curve OABC of fig. 4.2. In fact the rotation of domains has been observed by Barkhahausen [7] as early as 1919. Now if the magnetizing field is reduced to zero and an increasing magnetic field in the reverse direction is applied, the original magnetization curve OABC is not retraced and the magnetization lags behind the magnetic field. Increase in the reverse

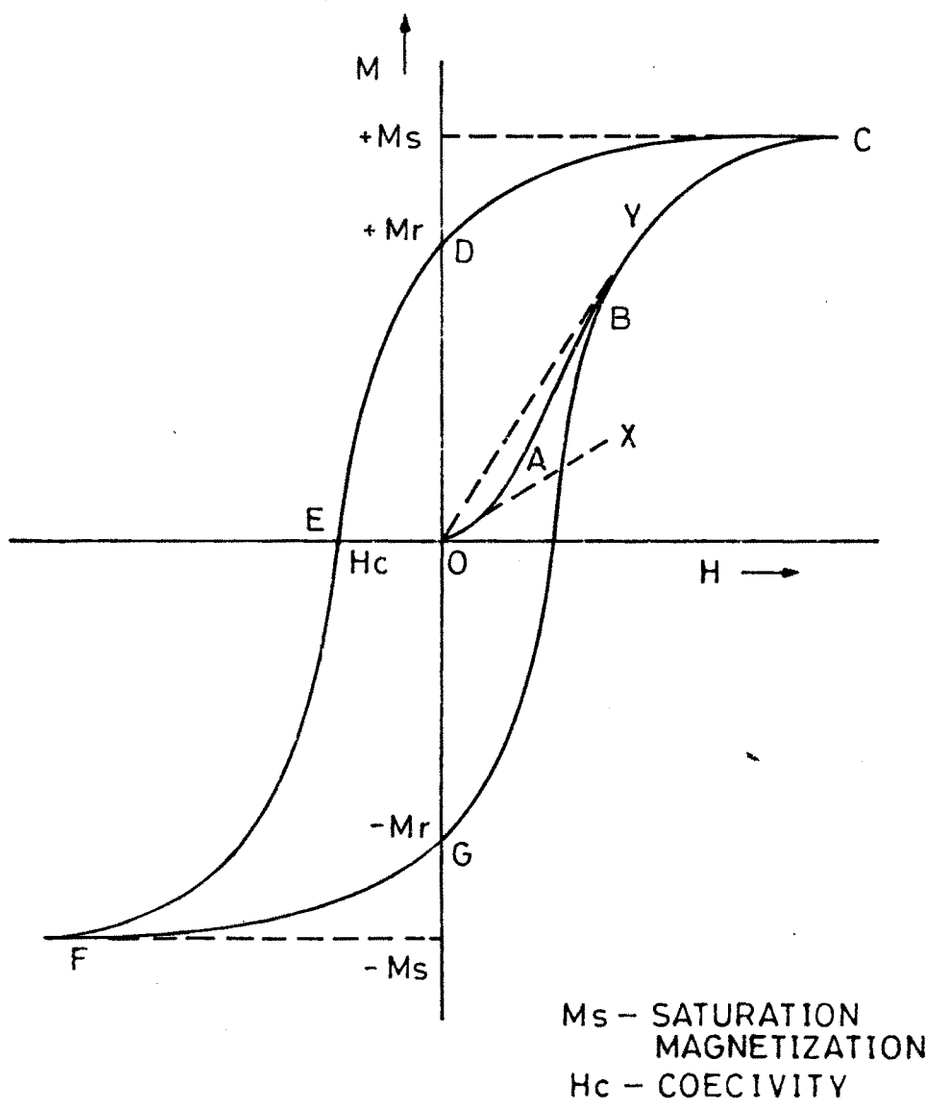


FIG. 4·2 – MAGNETIZATION CURVE (OABC) AND HYSTERESIS LOOP (CDEFGC)

field again leads to saturation but in the reverse direction and if the field is again returned to the original direction, the complete cyclic loop CDEFGC (hysteresis loop) is obtained as shown in fig 4.2. This phenomenon of lagging behind of magnetization to the magnetizing field is termed as hysteresis. The residual magnetization (Mr) at zero field is known as remanence and the phenomenon by the virtue of which a ferromagnetic/ferrimagnetic substance retains its magnetization even if the magnetizing field is reduced to zero is called as retentivity.

The coercive field (Hc) is the required magnetizing field to demagnetize the specimen i.e. to reduce the residual magnetization to zero. Initial susceptibility (χ_o) can be obtained from the slope of the tangent to the magnetization curve at the origin. Initial permeability (μ) can be obtained from the hysteresis loop of the magnetic induction is plotted against the magnetizing field.

4.3 EXPERIMENTAL TECHNIQUES

The saturation magnetization is measured using high field loop tracer manufactured by Magneta, Mumbai.

4.4 CALCULATION OF Ms and η_B

First CRO is calibrated using standard nickel sample whose saturation magnetization per unit mass is known and the calibration factor (CF) is determined. To measure the magnetization of the sample under observation vertical reading (R) on CRO corresponding to

saturation magnetization was noted and the magnetization per gram of the sample was determined using the following relation.

$$\sigma_s = (R \times C.F)/m \quad \ldots\ldots\ldots\ldots \quad (5.1)$$

where m = mass of pellet

C.F. = ratio of magnetization of Ni sample to its corresponding vertical reading on the oscilloscope screen. Saturation Magnetization (Ms) was determined using the relation

$$Ms = (1 - P) X \sigma_s X d_x$$

d_x – X ray density

P – porosity

The magnetic moment per formula unit (η_B) in Bohr magnetons, normally termed as magneton number is calculated using the following relation

$$\eta_B = (M \ X \sigma_s) / 5585$$

M = molecular weight of the sample

5585 – product of Avagadro's number (N) with Bohr magneton (μ_B- fundamental unit of magnetic moment).

Table 4.1 Values of saturation magnetization and magnetic moment for the samples

X	η_B, Bohr magnetons	Ms, emu/cc
0.1	2.07	126.5
0.2	1.92	135.2
0.3	2.10	168.4
0.4	1.91	150.8
0.5	1.63	85.2

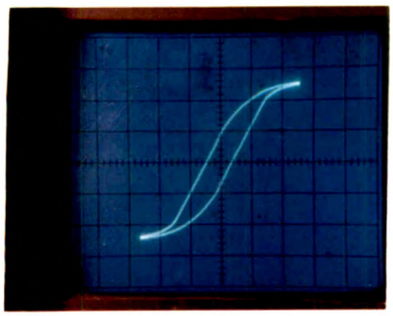
4.5 RESULTS AND DISCUSSION

Hysteresis loops for samples in the series $Ni_{0.7-x}\ Mg_x\ Zn_{0.3}\ Fe_2O_4$ are shown in the fig. 4.3.

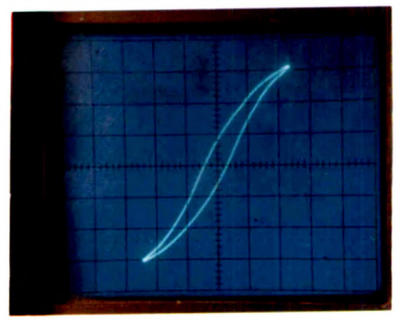
The values of saturation magnetization (Ms) and magnetic moment (η_B) were calculated from the hysteresis data and are given in Table 4.1. The variation of magnetic moment and saturation magnetization with content of Ni is shown in the figures 4.4 and 4.5 respectively. It can be observed that both of them increase with Ni content and have a maximum value at x = 0.3.

Magnetization in ferrites results from the distribution and alignment of magnetic ions on tetrahedral and octahedral sites. Nickel ferrite cooled slowly from sintering temperature has a structure of a completely inverse spinel. For nickel ferrite the cation distribution is expressed as [8].

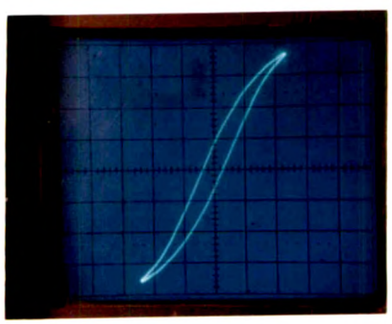
$$[Fe^{3+}]_A\ [Ni^{2+}\ Fe^{3+}]_B O^{2-}{}_4$$



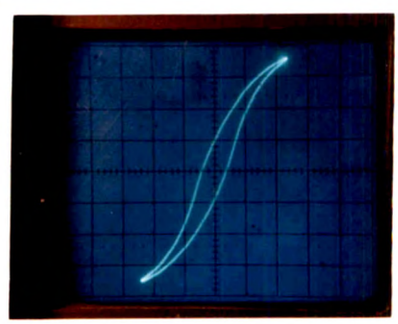
Ni Sample



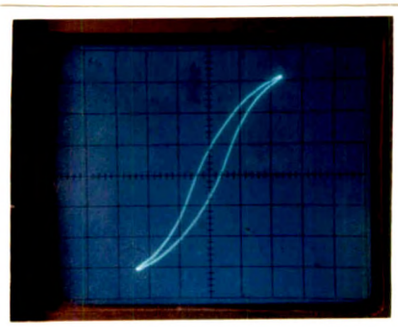
x = 0·1



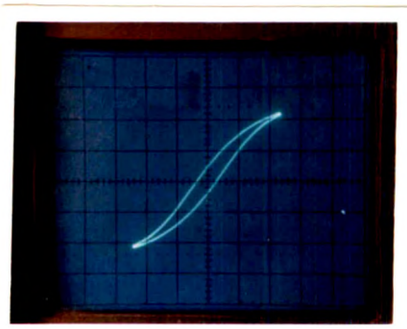
x = 0·2



x = 0·3



x = 0·4



x = 0·5

FIG. 4·3 – HYSTERSIS LOOP FOR $Ni_{0.7-x}Mg_xZn_{0.3}Fe_2O_4$

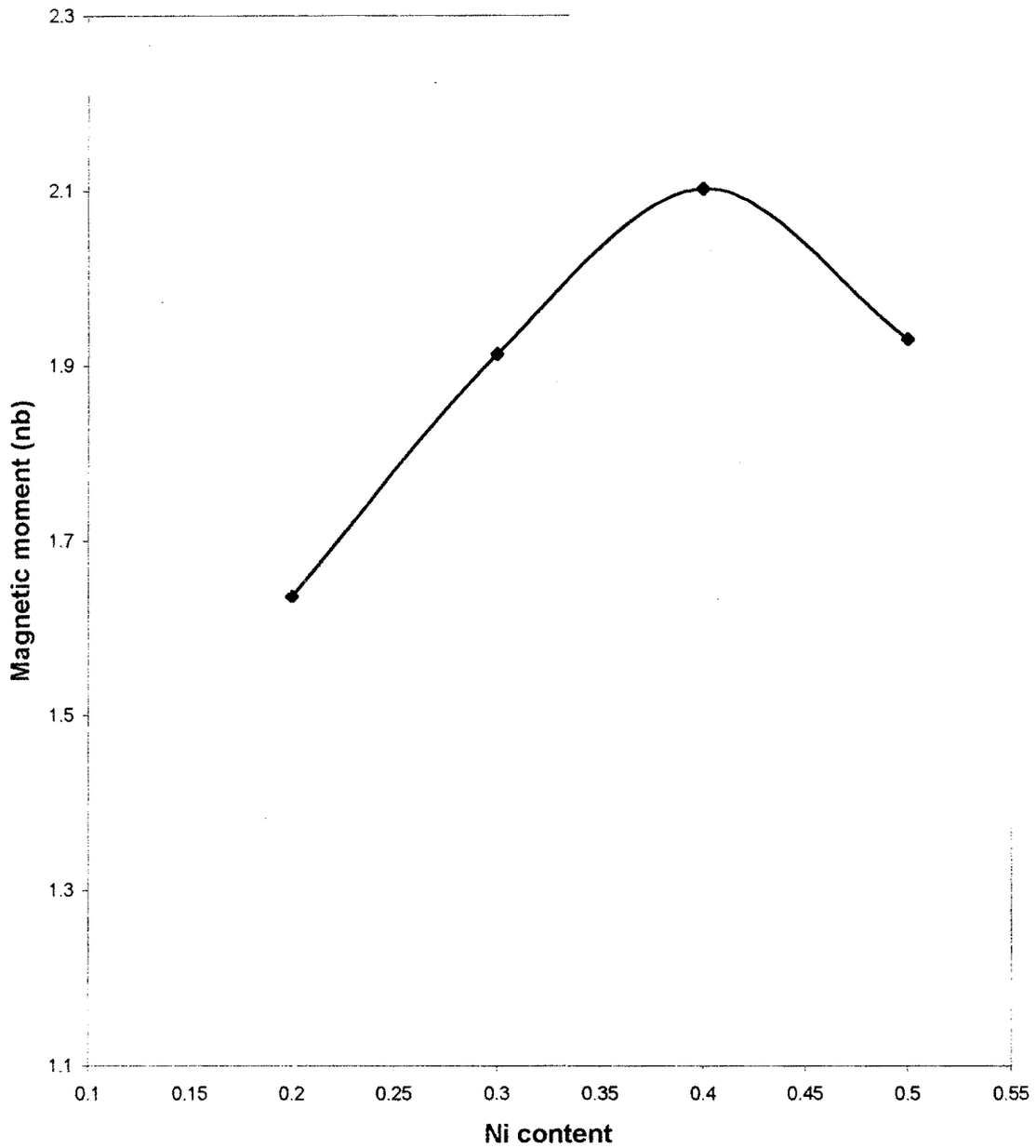


Fig.4.4 Varation of magnetic moment (n_b) with Ni content.

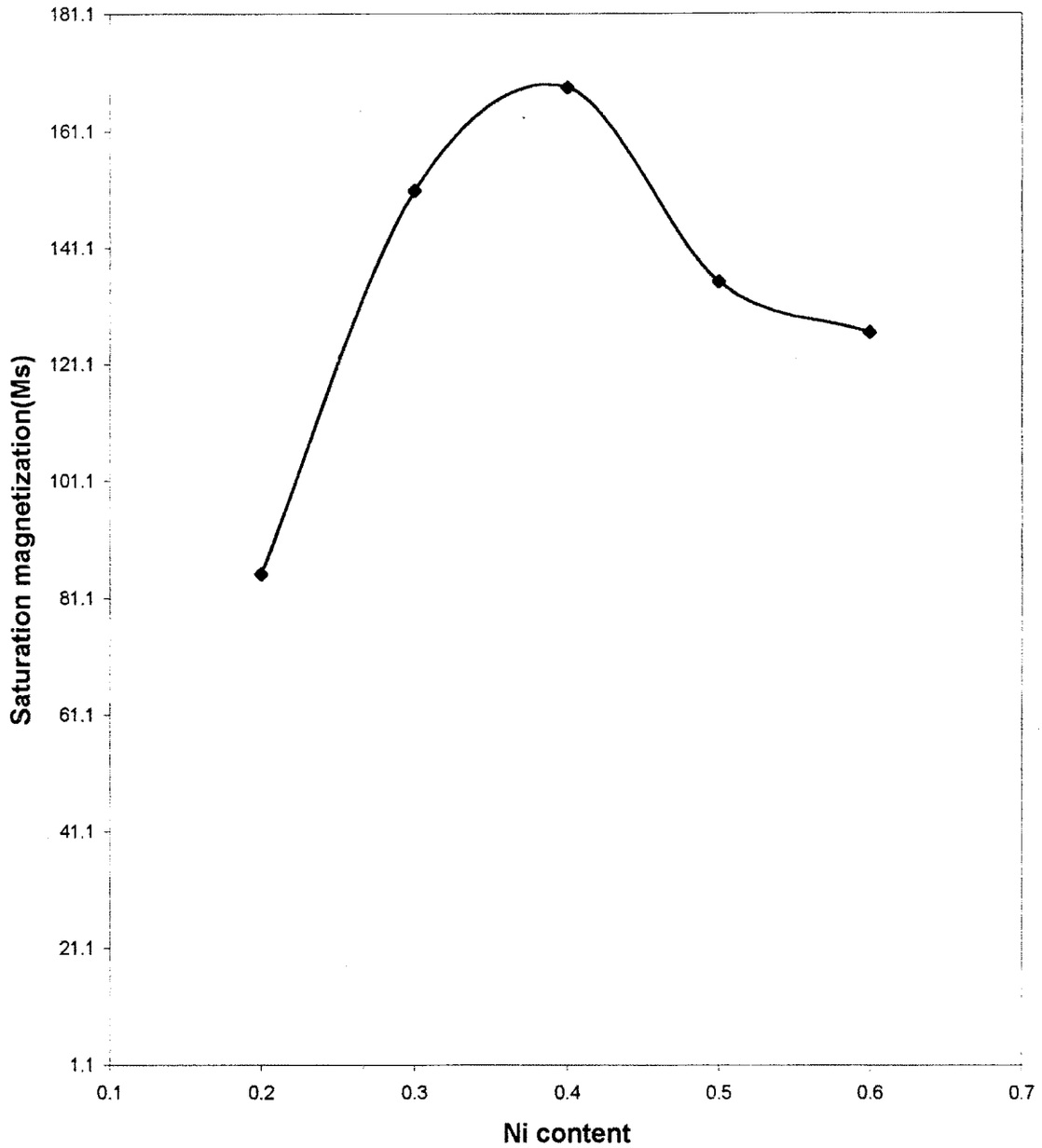


Fig.4.5 Varation of saturation magnetization (Ms) with Ni content.

The magnetic moment for Ni^{2+} is 2.2 μ_B and for Fe^{3+} it is 5 μ_B [9]. The Fe^{3+} ions on A sites are coupled with their spins antiparallel to the Fe^{3+} ions on B sites, so that the net magnetic moment is due to Ni^{2+} metallic ions. The magnetic moment is thus a function of Ni content.

The variation of Ms and η_B in the present case can be explained as follows. For the composition with x = 0.3 Zn occupies the A site and Mg a non magnetic on occupies the B site forcing some Fe^{3+} ions from B site to A site, affecting the M_A and M_B values. This results in the increase of magnetization. The decrease in saturation magnetization in the samples can be explained on the basis of change in the magnetization M_A and M_B on tetrahedral and octahedral sub lattices respectively. Mg^{2+} ions (with magnetic moment zero) besides preferring B sites have also a small tendency to occupy the A site. The partial replacement of Fe^{3+} ions by non magnetic Mg^{2+} ions results in lowering the value of both M_A and M_B. The decrease in $|M_A - M_B|$ there by decreases the net magnetization and hence the magnetic moment η_B.

Another important factor that influences the magnetization of ferrites is the microstructure. Each grain has certain amount of resultant magnetic moment. The presence of pores breaks the magnetic circuits among the grains, resulting in the net reduction of magnetic moment with increased pore concentration. [16]

Higher sintering temperature gives small number of large grains and consequently reduced porosity resulting in an increased magnetization.

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