

Synthesis of Nano sized Sm-Gd doped Magnesium ferrite and their Permittivity and Hysteresis Studies

Vasant Naidu
Professor/ECE
Sethu Inst. of Tech.,
Pulloor – 626115,
Tamilnadu, India

S. Vijayaragavan
Scholar./ECE
Sethu Inst. of Tech.,
Pulloor – 626115,
Tamilnadu, India

R. Legadevi
Research
Assistant./ECE, Sethu
Inst. of Tech. Pulloor
626-115, Tamilnadu,
India,

A. Senthil Kumar
Professor/ Sethu
Inst. of Tech. Pulloor
626-115, Tamilnadu,
India

ABSTRACT

The compositions of magnesium ferrite having general formula $Mg Sm_x Gd_y Fe_{2-x-y} O_4$ (where $x = 0.012, 0.013, 0.014, 0.015$ and $y=0.001$) were prepared by sol-gel method. The samples were characterized by XRD, EDAX, FTIR and SEM techniques. The nano size was confirmed by the XRD and SEM micro graphs. In the Hysteresis studies, it was found that the saturation magnetization of Gd^{3+} , Sm^{3+} substituted Mg ferrites was higher than the unsubstituted ferrite and these studies were useful to understand the electromagnetic properties of these nano materials. The electrical studies of the nano Sm-Gd doped nano Mg-ferrite it was seen that the dielectric constant (ϵ_r), dielectric loss (ϵ_r') was obtained and the permittivity studies show the decrease in relative permittivity of the material.

Key words: Nano materials, saturation magnetization, VSM, SEM, EDAX, Permittivity studies

1. INTRODUCTION

Spinel ferrite of chemical formula ($MgFe_2O_4$) play a significant role in microwave control components such as circulators, isolators and phase shifters[1]. Among different ferrites, magnesium ferrite ($MgFe_2O_4$) enjoys a special attention because of its vast applications in high

density recording media, heterogeneous catalysis, adsorption, sensors and magnetic technologies. Nano particles of $MgFe_2O_4$ have good photo electrical properties[2-4]. The physical and chemical methods widely used in the synthesis of nano ferrites are ball-milling, sol-gel, co-precipitation and hydro thermal methods[5-6]. Among the various routes, the sol-gel method [7-9] yields more promising results in the synthesis of ultrafine particles at a fairly low temperature. This work describes the study of $MgFe_2O_4$ ferrite synthesized using sol-gel (Auto combustion) method and the impact of nano regime on its structural and magnetic properties.

2. EXPERIMENTAL PROCEDURE

2.1 Synthesis Technique

Analytical grade Citric acid [$C_6H_8O_7 \cdot H_2O$] Merck, Ferric Nitrate [$Fe(NO_3)_3 \cdot 9H_2O$] Merck, Magnesium nitrate [$Mg(NO_3)_2 \cdot H_2O$] Lobo Chem., Samarium Nitrate [$Sm(NO_3)_2 \cdot H_2O$] Alfa Aesar and Gadolinium nitrate [$Gd(NO_3)_3 \cdot XH_2O$] Alfa Aesar were used to prepare $Mg Sm_x Gd_y Fe_{2-x-y} O_4$ with $x = 0.012, 0.013, 0.014, 0.015$ and $y = 0.001$ by sol-gel auto-combustion method[9]

The required amount of metal nitrates and citric acid are dissolved in deionized water, Sm and Gd

atoms are substituted for Fe atoms at x and y ratio respectively and were mixed in a molar ratio of 1:1 [9]. The pH of the solution was adjusted to 7 using ammonia solution. Then the solution was heated at 80°C to transform into gel. When ignited at any point of the gel, the dried gel get burnt in a self-propagating combustion manner until all gels were completely burnt. The auto ignition was completed within a minute, yielding into a brown-colored ash. The s-prepared powders of all the sample were sintered in a microwave furnace VBCC/MF to a temperature of 850°C for 3 hours and the sintered ferrites were characterized with respect to crystallite size and lattice parameter determination using XRD. The FWHM value of the peak corresponding to plane was considered after correction for instrumental broadening.

The experimental magnetic moment was calculated from the following formula [10].

$$\eta = \frac{[M_W * M_S]}{5585}$$

Where MW is the molecular weight of the sample and Ms is the saturation magnetization in emu/g obtained from the VSM data.

2.2 XRD and EDAX Studies

The phase composition of fine ferrite powder was carried out using PAN analytical X'pert PRO diffraction meter with Cu K_α radiation (λ=1.54Å) at 40KV and 30mA with a scanning rate of 0.01°/s and scan speed of 1°/min in a 2θ range of 10-80°A. The crystallite size was calculated by the Scherrer formula. The energy dispersive studies (EDAX) were done on Genesis EDAX to confirm the presence of chemical composition of the powder.

2.3 FTIR Analysis

Infrared absorption spectra in the range of 362-572 cm⁻¹ were recorded at room temperature by using SHIMADZU FTIR spectrum one spectrometer using KBr pellet method. The spectrum transmittance (%) against wave number (m⁻¹) is used for interpretation of the results.

2.4 SEM Studies

The micro structure and the morphological studies of the nano ferrite powder were carried out using a scanning electron microscope (HITACHI model S-3000H). The grain size was determined from the SEM microphotographs.

2.5 Magnetic Measurements

Commercial vibrating sample magnetometer (VSM) Lakeshore (Model 73009) was used for analyzing the Magnetic measurements of the samples. With the maximal applied magnetic fields up to 0.95T magnetic hysteresis loops were measured at room temperature. The Magnetic field sweep rate was kept at 5 Oe/s for all measurements, so that the measurement of hysteresis loops with maximum field of 0.989T was taken at an interval of 3 hrs. The saturation magnetization (MS), coercive force (HC) and remanent magnetization (MR) and magnetic moment (η) has been determined from magnetization study.[9].

2.6 Electrical Measurements

N4L LCR meter was used for the Electrical measurements such as capacitance and permittivity. The experimental set up for measuring the dielectric properties in the microwave region consisted of a pallet holder connected to the N4L LCR meter interfacing the computer [11]. The microwave properties of the four samples Sm(x) - Gd(y) doped magnesium ferrite x = 0.012, 0.014, 0.015, 0.016 and y=0.001, were investigated at the frequency range from 10 MHz to 20 GHz

3 RESULTS AND DISCUSSION

3.1 X-ray diffraction

Fig 1 shows the morphology and X-ray diffraction pattern of the 1.2, 1.3, 1.4, 1.5% of Sm and 0.1 % Gd of the magnesium ferrite powder. The single phase formation of the composition of the ferrite has been conformed from X-ray diffraction pattern. The broader peaks of XRD indicated the fine crystalline size of the ferrite powder and the

phase formation behaviour of Mg Sm_x Gd_y Fe_{2-x-y} O₄ in the XRD fig 1.

As the Fig.1 shows the X-ray diffraction patterns of as burnt ferrite powder of different composition of MgSm_xGd_yFe_{2-x-y}O₄. The patterns also revealed the spinel ferrite phase, these XRD patterns match with the JCPDS card number 89-3084 for magnesium ferrite powder and peaks of Sm and Gd are marked in the XRD, which match with the JCPDS card number 75-0161. The figure also confirm the absence of metal oxide phase in the as burnt powder. The X-ray pattern shows reflection plane (110), (220), (311), (331), (222), (400), (422). It was observed that the appearance of plane (311) is there in all the sample patterns, which clearly indicates the presence of MgFe₂O₄ in single-phase cubic spinel structure. The secondary phase (orthorhombic) was also observed due to the rare-earth ortho-ferrite SmFeO₃. The presence of secondary phase also suggest the solubility limit of Sm₃₊ ions in the spinel lattice due to its crystalline size (42.67 nm, 44.58 nm ,49.43 nm and 51.78nm) which is similar to the results of Wilhelm Van Aulock [11]. It was well known that the degree of replacement of the host cations by the other ions in the host lattice depending on the cations radius of the substituent [12]. The lattice constant *a* (Å) in the spinel structure are mentioned in Table 1 had been calculated from the prominent peak (311) by using Bragg's equation.

$$a = d_{hkl} \sqrt{h^2 + k^2 + l^2}$$

h , *k* , *l* are the indices of the mentioned planes. Lattice constants of all samples prepared in investigation are listed in Table 1. The lattice constant is smaller than pure MgFe₂O₄ and increases with the increase in radius of Sm and Gd ions. This is attributed to the large difference between cation radii of R³⁺ and Fe³⁺ owing to the removal of rare-earth ions from the spinel lattice. The size of crystallite was evaluated by measuring the FWHM of the most intense peak (311) mentioned in Table 1 using the Debye Scherrer's formula [13]. The mean crystalline size of the sample lies in the range of 42.67 nm to 51.78 nm

$$D = \frac{0.94\lambda}{\beta \cos\theta}$$

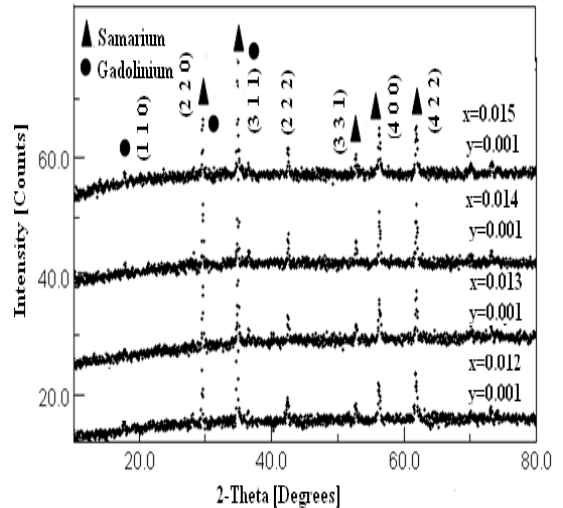


Fig .1 XRD patterns of sintered Mg Sm_x Gd_y Fe_{2-x-y} O₄ ferrite with different Sm-Gd content x=0.012, 0.013, 0.014, 0.015 and y=0.001.

Table.1. XRD, Cation distribution of Mg Sm_x Gd_y Fe_{2-x-y} O₄.

3.2 EDAX

| Mg Sm _x Gd _y Fe _{2-x-y} O ₄ | | Lattice constant (Å°) | Crystalline size(nm) |
|--|-------|-----------------------------|-------------------------|
| x | y | | |
| 0.012 | 0.001 | 9.873 | 42.67 |
| 0.013 | 0.001 | 9.743 | 44.58 |
| 0.014 | 0.001 | 9.454 | 49.43 |
| 0.015 | 0.001 | 9.256 | 51.78 |

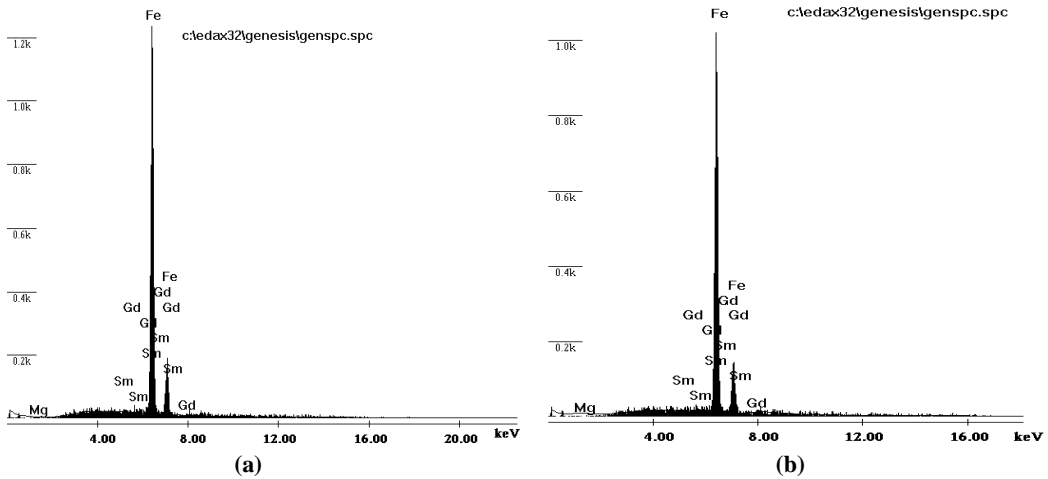


Fig.2(a)EDAX pattern for Mg Sm_xGd_y Fe_{2-x-y}O₄ x=0.012 y=0.001
(b) EDAX pattern for Mg Sm_xGd_y Fe_{2-x-y}O₄ x=0.014 y=0.001

The EDAX spectra (Fig.2) obtained from the center of grain boundary MgFeO₃ phase indicated the presence of mainly Mg, Fe and oxygen along with small amount of Sm, and Gd. The grain size of ferrite matrix phase as well as the MgFeO₃ phase increased with increasing the La₃₊ substitutions. The EDAX spectra (Fig.2) obtained from the center of Mg substituted Gd-Sm ferrite grains indicated the presence of small concentration of Mg inside the grains at 2.2 keV, where as Sm grains are seen in between the energy range of 5keV and 7keV, where as Gd grains are seen in between the energy range of 5.5keV and 8keV where as the Fe peaks are seen at 6.2keV and 6.7keV

3.3 SEM analysis

The visualized micro structure of the samples taken by scanning electron micrograph are shown in Fig 3 The unsubstituted specimen Fig 3 (a) showed the presence of a monophasic homogeneous microstructure with an average

grain size 0.42 nm [1] .Whereas Sm- Gd doped specimens shown in Fig 3 (b, c and d) had a bi-phasic microstructure constituting of dark ferrite matrix grains and small whitish grain at the grain junction/boundary (in the encircled region) it is in

accordance to Sattar et, al [2] that the rare earth ions occupy either the iron positions or go to the grain boundaries. However we have to exclude the probability that the rare earth ions occupy the A- site of Fe³⁺ions .

This is due to the fact that the tetrahedral sites are small to be occupied by the large rare earth ions which have large ionic radius. Of course the probability of occupancy in the octahedral site (B-site) by the rare earth ions will increase with in decrease in ionic radius R. The amount of Sm-Gd FeO₃ was maximum in x¼ 0.015 composition. The grain size of matrix phase was maximum in x¼ 0.015 composition Fig 3(d).Here the relatively lower grain size of ferrite matrix in x¼ 0.012 compositions may be due to the grain growth inhibition caused by Sm-Gd FeO₃ Fig. 3(a) The grains in the unsubstituted sample are inhomogeneous Fig 3(a), as these grains are affected by certain stress. Where as in Fig 3(b-d) the grains for the Sm-Gd substituted Mg Ferrite samples are nearly homogeneous due to the decrease of stress as shown in the encircled regions in Fig 3(b-d) The SEM monographs confirm these results that the stability has increased for the substituted samples.

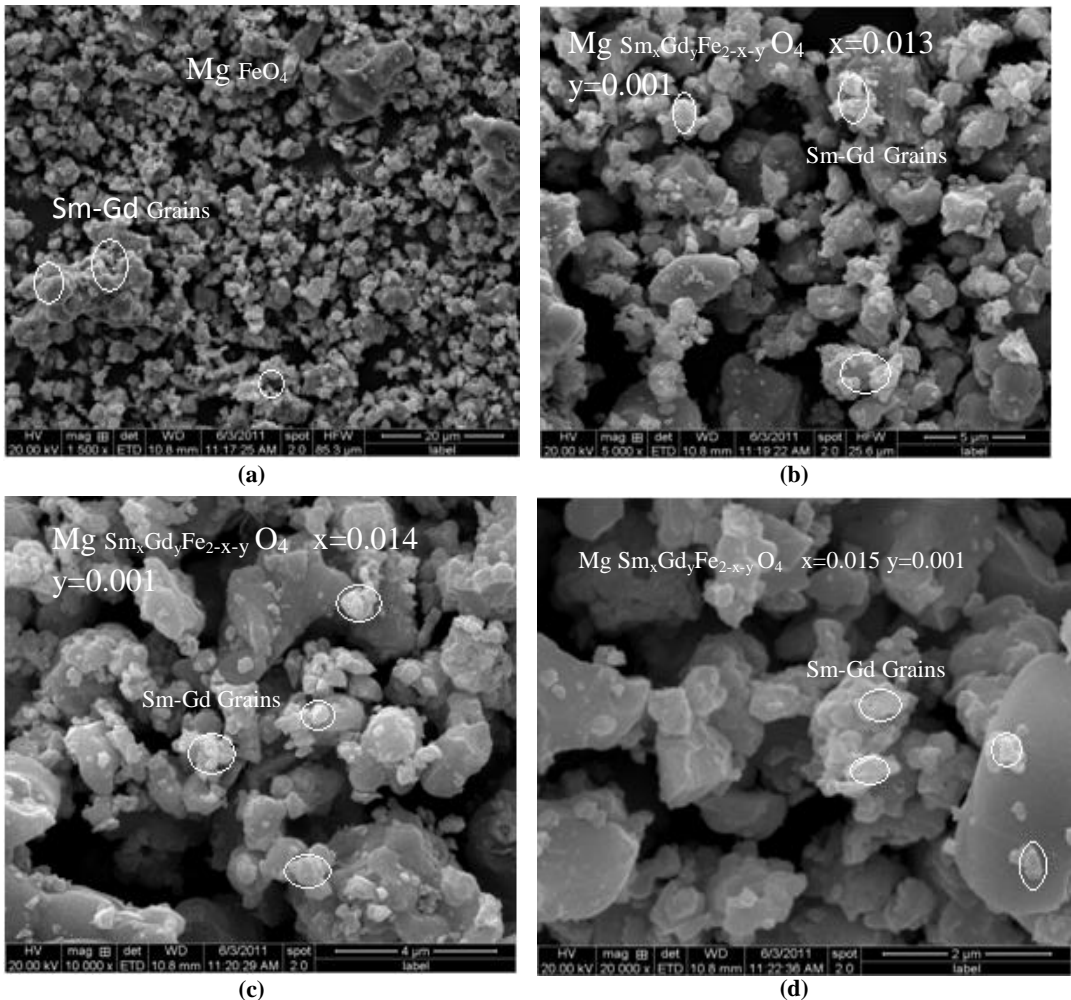


Fig.3 (a)SEM images for MgFeO_4 (b) $\text{MgSm}_x\text{Gd}_y\text{Fe}_{2-x-y}\text{O}_4$ $x=0.013,y=0.001$ (c) $\text{MgSm}_x\text{Gd}_y\text{Fe}_{2-x-y}\text{O}_4$ $x=0.014,y=0.001$ (d) $\text{MgSm}_x\text{Gd}_y\text{Fe}_{2-x-y}\text{O}_4$ $x=0.015,y=0.001$

3.4. FTIR study

The study of far-infrared spectra is an important tool to get the information about the position of ions in the crystal. FTIR absorption spectra of the samples in the range of $362 - 572 \text{ cm}^{-1}$ are given in Fig.4. The spectra show two major absorption bands in the given frequency range. The high and

low frequency absorption bands (ν_1, ν_2) are observed in a frequency range of $3.62 \times 10^4 \text{ m}^{-1}$ to $4.06 \times 10^4 \text{ m}^{-1}$ and $4.28 \times 10^4 \text{ m}^{-1}$ to $5.72 \times 10^4 \text{ m}^{-1}$, which is attributed to tetrahedral and octahedral complexes $\text{Fe}^{3+}\text{O}^{2-}$. These two bands have been reported by Waldron [14] in spinel structure of ferrite. No shift of absorption band ν_1 is observed. The absorption band ν_2 is slightly shifted to a higher frequency side with addition of

R ions and is attributed to increase in bond length on the B-site [15]. This suggests that the rare-earth ions occupy the B-site. The difference in frequencies between ν_1 and ν_2 is due to changes in bond length ($\text{Fe}^{3+}\text{O}^{2-}$) at tetrahedral and octahedral sites [16]. The broadening of the ν_2 band is observed in rare-earth added MgFe_2O_4 , which suggests the occupancy of rare-earth ions on the B-sites [17].

In nano particles, the inter-ionic separation may face an extension due to less number of structural matters in the surrounding of each particle. Thus the nano size of the ferrite particles and ultimate change of the nature of ions in the respective size

could have caused reduction of magnetization in MgFe_2O_4 . Thus in nano regime, the magnetization is said to be dependent on grain size and cation distribution. Fig 4 show that the change in ν_1 and ν_2 , the value of change in the difference of frequency decrease with the increase in the values of Sm-Gd. The change in retentivity and change of coercive force was seen with the change in concentration. Table.2 gives the absorption frequencies for different doping values of Samarium and Gadolinium, which leads into the change in retentivity and change in coercive force.

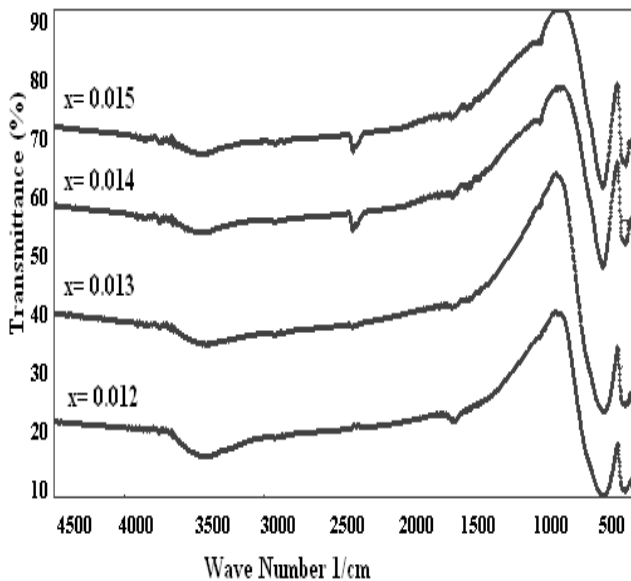


Fig.4 FTIR absorption spectra of the as synthesized powders.

| Nano Composite $\text{Mg Sm}_x\text{Gd}_y\text{Fe}_{2-x-y}\text{O}_4$ | | Absorption/ cm^{-1} | |
|--|---------|------------------------------|---------|
| | | ν_1 | ν_2 |
| x=0.012 | y=0.001 | 362 | 568 |
| x=0.013 | y=0.001 | 387 | 428 |
| x=0.014 | y=0.001 | 406 | 572 |
| x=0.015 | y=0.001 | 403 | 563 |

Table 2. FTIR to show the rare-earth ions occupation in the B-site

3.5 Hysteresis studies

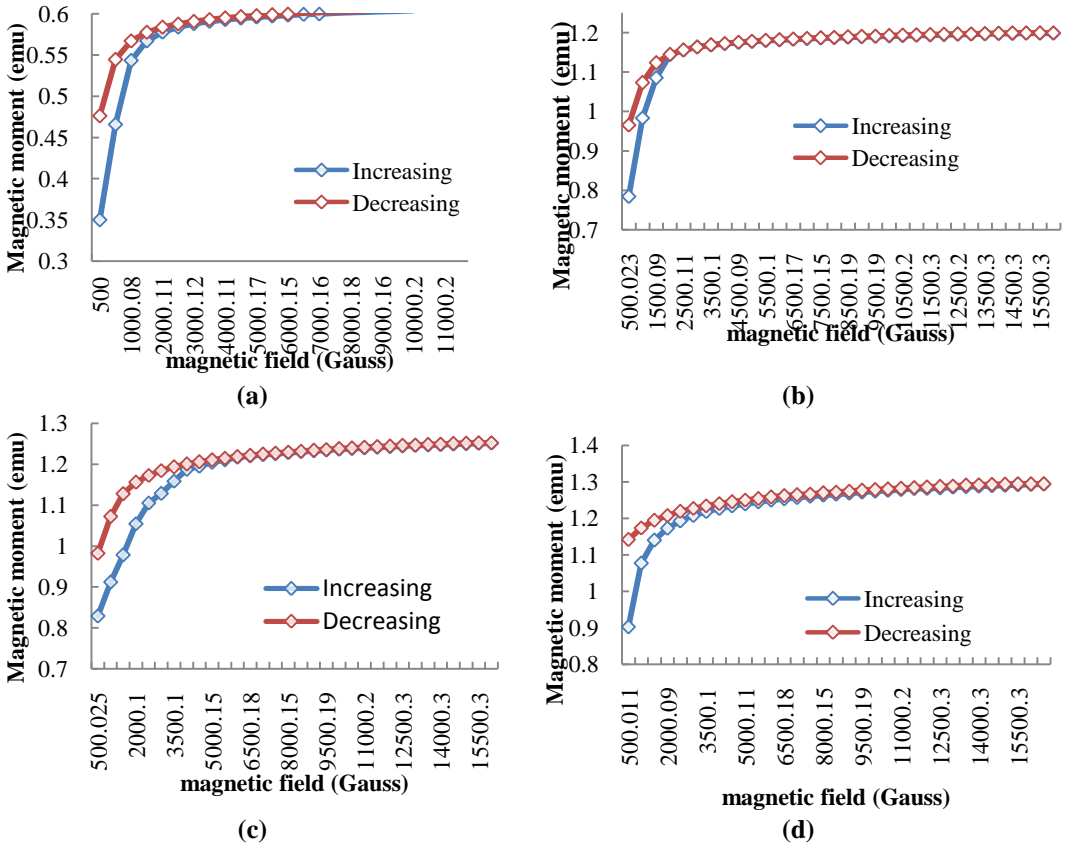


Fig.5 (a) Saturation magnetization (Ms) for Mg Sm_xGd_yFe_{2-x-y}O₄ x=0.012 y=0.001 (b) x=0.013 y=0.001 (c) x=0.014 y=0.001 (d) x=0.015 y=0.001

Table 3 Coercivity, Magnetic saturation, Magnetic moment and Retentivity of Mg Sm_x Gd_y Fe_{2-x-y} O₄

| Zn _{1-x-y} Ni _y Sm _x Fe ₂ O ₄ | | | Magnetic Saturation (emu) | Magnetic moment (η) | Coercivity (G) | Reten tivity (emu) | Dielectric constant (ε _r) | Dielec tric loss (ε _r ') |
|---|-------|-------|---------------------------|---------------------|----------------|--------------------|---------------------------------------|-------------------------------------|
| Samples | x | y | | | | | | |
| MSGF-1 | 0.012 | 0.001 | 0.8115 | 0.02479 | 40.562 | 0.1546 | 1.5685 | 1.5968 |
| MSGF-2 | 0.013 | 0.001 | 1.2148 | 0.04758 | 66.584 | 0.1894 | 1.5055 | 1.5303 |
| MSGF-3 | 0.014 | 0.001 | 1.2679 | 0.04537 | 85.41 | 0.2056 | 1.4025 | 1.4630 |
| MSGF-4 | 0.015 | 0.001 | 1.3159 | 0.04794 | 104.89 | 0.2468 | 2.2563 | 2.4718 |

Fig. 5 shows the variation in saturation magnetization (M_s) for the different x values of $MgSm_xGd_yFe_{2-x-y}O_4$ ferrite, the saturation magnetization (M_s) value increases with increase in the value of x. The increasing Samarium and Gadolinium content induced a polar-to-non polar phase transition. Within the polar region, a rhombohedral and two orthorhombic modifications of $MgSm_xGd_yFe_{2-x-y}O_4$ were found. It was shown that Sm-Gd substitution resulted in the appearance of spontaneous magnetization, which was significantly enhanced upon the composition-driven transition from a rhombohedral to an orthorhombic phase. Table 3 shows that the change in retentivity and change of coercive force with change in concentration respectively. The coercive force shows linear increase with the increase in the doping concentration table 3. The values of magnetic parameters such as M_s, H_c, M_R of nano particles of $MgSm_xGd_yFe_{2-x-y}O_4$ obtained from the VSM data are 0.6119 emu, 64.582 G, 0.068 emu respectively. As reported [18,19] M_s value for bulk particle of $MgFe_2O_4$ as 27 emu/g. Therefore the increase in saturation magnetization can be attributed to the effect of nano regime on it. The difference in the value of M_s can be explained in the light of cation distribution. Any change in the concentration and nature of the ions in A and B site should cause resultant magnetization to be different from reported one [20].

3.6 Electrical Properties

The dielectric constant ϵ_r and dielectric loss ϵ_r' of the sintered samples $MgSm_{0.012}Gd_{0.001}Fe_{2-0.012-0.001}O_4$, $MgSm_{0.013}Gd_{0.001}Fe_{2-0.013-0.001}O_4$, $MgSm_{0.014}Gd_{0.001}Fe_{2-0.014-0.001}O_4$, $MgSm_{0.015}Gd_{0.001}Fe_{2-0.015-0.001}O_4$ over the microwave frequency range from 10M–20GHz are shown in figure 6. The plots (a, b) show that the ϵ_r and ϵ_r' values tend to decrease exponentially, it is also seen that the value of ϵ_r and ϵ_r' increases with the addition of Samarium and Gadolinium atom concentration, while the capacitance value remains fairly constant over the frequency range of study. The maximum value of dielectric constant 2.2563 is observed for the sample $MgSm_{0.015}Gd_{0.001}Fe_{2-0.015-0.001}O_4$ and minimum is 1.4025 for the sample $MgSm_{0.014}Gd_{0.001}Fe_{2-0.014-0.001}O_4$.

The high value of dielectric constant of the sample MSGF -4 may be explained on the basis of the structural changes associated with the magnesium ferrite when the grain size is reduced to nanometer order. Magnesium ferrite crystallizes into a cubic close-packed arrangement of oxygen ions. It belongs to the class of ferrites with an inverse spinel structure, having structural formula, $Fe^{3+}[Mg^{2+}Fe^{3+}Sm^{3+}Gd^{3+}]O_4$ [21].

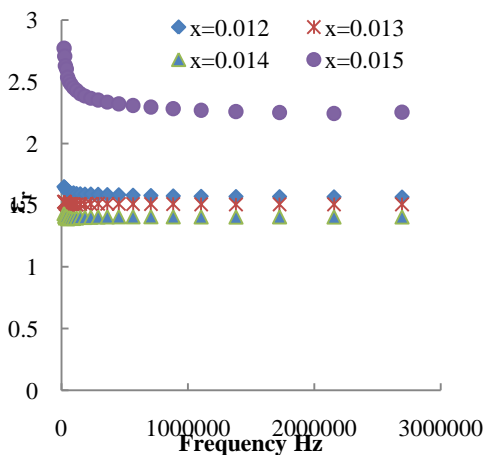
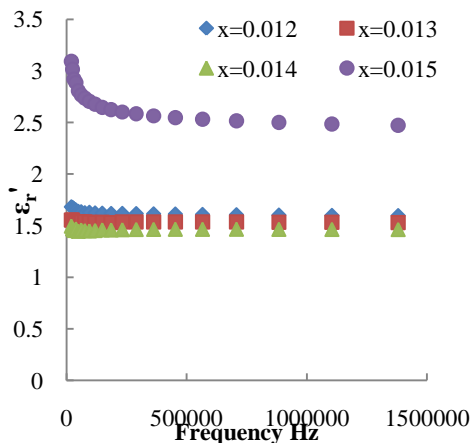


Fig.6(a) Dielectric constant(ϵ_r) for $MgSm_xGd_yFe_{2-x-y}O_4$



(b) Dielectric loss(ϵ_r') for $MgSm_xGd_yFe_{2-x-y}O_4$

The metal ions given in the square bracket are called octahedral (B site) ions and that outside the square bracket are called tetrahedral (A site) ions. The magnesium ions ($\text{Sm}^{3+}\text{Gd}^{3+}$) together with the presence of Sm^{3+} and Gd^{3+} ions gives rise to p-type carriers (holes) where as Fe^{2+} and Fe^{3+} ions produce n-type carriers (electrons). Therefore, both electrons and holes that are present in the B sites are due to the presence of Ni and Fe ions. Since only iron ions are present in A sites, electrons are the carriers in A sites. The distance between the ions in A sites (0.357 nm) is larger than the distance between the ions in B site (0.292 nm) [22]. Also, the degree of covalence for the A site ions is higher than that of the B site ions. All the above factors result in a high activation energy for the A sites compared to the B sites. Hence, in ordinary magnesium ferrite with an inverse spinel structure the electron movement in B sites dominates compared to that in A sites.

From the Fig. 6(a, b) it is observed that the dielectric constant as well as the dielectric loss decreases with increasing frequency, thus exhibiting the normal ferromagnetic behaviour. A more dielectric dispersion was also observed at lower frequency region and it remains almost independent of applied external field at high frequency. Similar behaviour of dielectric constant with frequency was also observed by many researchers [22]

The dielectric dispersion observed at lower frequency region is due to Maxwell-Wagner [23]. Interfacial type of polarization, which is in agreement with Koops phenomenological theory [24]. The decrease in dielectric constant with frequency can be explained by the supposition that the mechanism of polarization process in ferrite is similar to that of conductivity process [25]. By the electronic exchange, $\text{Fe}^{+2} \rightarrow \text{Fe}^{3+} + e^{-1}$ one obtains local displacement of electrons in the direction of electric field. These displacements determine the polarization in ferrites. It is known that effect of polarization is to reduce the field inside the media. The decrease of polarization with increase of frequency may be due to the fact that, beyond a certain frequency of the electric field, the electronic exchange

half of the iron ions (Fe^{3+}) occupy the B site and the remaining half of the iron ions reside in A site.

between ferrous and ferric ions cannot follow the alternating field. Hence dielectric constant may decrease substantially as frequency is increased as in Fig. 6(a, b).

4. CONCLUSION

From the above experimental results, it is clearly evident that the nano size of the ferrite particles has caused increase in magnetization in Sm-Gd doped MgFe_2O_4 . Since Saturation magnetization and coercive force increases with the increase in Sm-Gd. For the sintered powder of magnesium samarium ferrites produced by sol gel route and for this grounded powder the following correlations were observed between microstructure and magnetic properties and between microstructure and electrical properties:

a) The as prepared powder still required finer milling and an increase in the sintering temperature in order to yield fully dense ceramics.

b) The X-ray diffraction studies show that the Sm-Gd addition is not soluble in the spinel phase but instead forms another ortho ferrite phase, which exerts pressure on the spinel phase and prevents its expansion due to Sm addition. The lattice parameter did not change with Sm addition, but the oxygen positional parameter values showed a deviation from the ideal spinel case which indicates the presence of some distortion in the unit cell due to Sm addition.

c) The saturation magnetization of this powder was determined using a Vibrating Sample Magnetometer (measured at LAKESHORE VSM model 73009). Hysteresis losses, maximum magnetization and magnetic remanence showed a minimal change.

d) There was no substantial change in the coercive force as expected, therefore doping percentage has to be increased.

e) The effect of substituting magnesium with samarium in magnesium samarium ferrites on the magnetization of magnesium ferrite differs significantly from that promoted by replacing iron with samarium, probably due to differences in the ratio of the cationic radii of the coupling nonferrous elements. As these studies will be very useful for the application of the ferrite materials in the antenna construction. This change will be also suitable for reducing the size of the antenna. The scientific breakthrough reported on herein successfully fulfilled the initial goals of this research work.

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