International Journal on Science and Technology (IJSAT)
E-ISSN: 2229-7677 ● Website: www.ijsat.org ● Email: editor@ijsat.org

Synthesization of Gadolinium Doped Nano Magnesium Samarium Ferrite for Inorganic Gas Sensor Studies

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Abstract

The Magnetic Properties Of Mg-Ferrite Ceramics Mgsm$_x$gd$_y$fe$_{2\text{-X-Y}}$o$_4$, (Where X = 0.012, 0.013, 0.014, 0.015 & Y = 0.001) Were Synthesized By Sol Gel Auto Combustion Method. The Structure And Composition Gd Doped Mg-Ferrite Ceramics Was Analyzed And The Nano Size Was Confirmed By Sem Monographs. The Vsm Studies Confirm The Magnetic Behavior And Used To Understand The Electromagnetic Properties Of These Nano Materials.

Key Words: Ferromagnetic Materials, Nanomaterials, Magnetizations, Xrd, Sem, Ftir And Vsm

1. Introduction

In Advanced Technologies The Ferrites Are Used For Magnetic Or Electrical Applications, As They Are The Required High-Density Materials. Synthesis Of Nanometer Size Particles Proved To Be One Of The Interesting Fields Of Material Science In Material Processing And Technological Applications, As The Small Size Particles Have Some Of The Interesting Properties As Compared To Bulk Particles. These Particles Have Improved Catalytic, Dielectric And Magnetic, Properties, As They Possess High Resistivity And Negligible Eddy Current Losses. Magnetic Nano Particles Promise Some Interesting Applications, Such As In High Frequency Devices, Magnetic Fluids, High Density Magnetic Recording, Color Imaging Etc

The Various Processing Techniques, Which Are Used For The Synthesis Of Spinel Ferrite Powders Include Microwave Refluxing, Sol-Gel, Hydrothermal, Co-Precipitation, Spray Pyrolysis. In Fact There Are Numerous Papers On Synthesis Of A Gadolinium Ferrite By Various Methods. In The Present Investigation We Have Employed Sol-Gel Auto-Ignition Method To Synthesize Gd Doped Magnesium Ferrite Nano Particles. The Sol-Gel Auto Ignition Method Is Used To Speed Up The Synthesis Of Complex Materials. It Is A Simple Process, Which Offers A Significant Saving In Time And Energy Consumption Over The

Traditional Methods, And Requires Less Sintering Temperature. This Method Is Employed To Obtain Improved Powder Characteristics, More Homogeneity And Narrow Particle Size Distribution, Thereby Influencing Structural, Electrical And Magnetic Properties Of Spinel Ferrites. We Examined The Microstructures Of The End Products By X-Ray Diffraction And Sem To Obtain Quasi-Three Dimensional Information On The Grain Shape, Size And Pore Size. It Has Been Already Reported The Magnesium Ferrite Is An N Type Semiconductor With A Bandgap Of 2.01ev. Gadolinium Substituted Ferrite, In Particular, Has Been Found To Exhibit A Large Magneto-Optic Effect And Low Propagation Loss, Which Will Be Good Materials For The Devices With Higher Quality. A Low Coercivity, High- Remanence, Soft Magnetic Material, Having Hysteresis Loop, Is Required To Be Applied In Microwave Devices, The Most Important Static Magnetic Properties Are The Magnetization (Ms), Anisotropy Constants, Neel Temperature, Remanent Magnetization, Coercivity (Hc). In General, Ms And Hc Are Required For Applications.

2. Experimental Procedure

2.1 Synthesis Technique

Nanocrystalline Powders Of $Mgsm_xgd_yfe_{2-X-Y}o_4$(X = 0.012, 0.013, 0.014, 0. 015 And Y = 0.001) Were Prepared By Sol-Gel Auto-Ignition Method. The Metal Nitrates Were Dissolved Together In A Minimum Amount Of De- Ionized Water To Get A Clear Solution. An Aqueous Solution Of Citric Acid Was Mixed With Metal Nitrates Solution, Then Ammonia Solution Was Slowly Added To Adjust The Ph To 7. The Mixed Solution Was Moved On To A Hot Plate With Continuous Stirring At 94˚c. The Viscous Brown Gel Burnt With Glowing Flints. The Auto Ignition Was Completed Within A Minute, Yielding Into Brown-Colored Ash. The As-Prepared Powders Of All The Samples Were Sintered In A Microwave Furnace Vbcc/Mf To A Temperature Of 600˚c For 1.5 Hours. The Grain Size Of The Nano Ferrite Is Determined Using Scherrer's Equation. The Fwhm Value Of The Peak Corresponding To Plane Was Considered After Correction For Instrumental Broadening. Using The Knowledge Of Site Preference Of The Ions And The Ionic Size Data Of The Respective Ions, The Cation Distribution Has Been Estimated Theoretically Using The Following Formula Proposed.

$$\eta = \frac{M_W \times M_S}{5585}$$

Where Mw Is The Molecular Weight Of The Sample And Ms Is The Saturation Magnetization In Emu/G.

2.2 Ftir Study

Infrared Absorption Spectra In The Range Of 3.72 $X10^4$ M^{-1} To 5.43 X 10^4 M^{-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.

2.3 Sem Studies

Micrographs Of The Sensor Were Recorded Using A Scanning Electron Microscope (Hitachi Model S-3000h).

2.4 Magnetic Measurements

The Magnetic Measurements Were Performed Using The Commercial Vibrating Sample Magnetometer (Vsm) Lakeshore (Model73009). Magnetic Hysteresis Loops Were Measured At Room Temperature With Maximal Applied Magnetic Fields Up To 0.95t. Magnetic Field Sweep Rate Was 5 Oe/S For All Measurements, So That The Measurement Of Hysteresis Loops With Maximum Field Of 0.989 T Took About Three Hours.

3. Results And Discussion

3.1 Sem Analysis

Figure Shows The Microstructure Of Sintered Specimen Of Sm And Gd Doped Magnesium Ferrite Studies Are Made From The Sem Images. Unsubstituted Specimen The Presence Of A Monophasic Homogeneous Microstructure With An Average Grain Size 0.42nm.Whereas, Sm And Gd-Doped Specimens' Show A Bi-Phasic Microstructure Constituted Of Dark Ferrite Matrix Grains And Small Whitish Grain At The Grain Junction/Boundary. As Proposed By Sattar Et, Al. 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 Fe^{3+} 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 Of The Octahedral (B-Site) By The Rare Earth Ions Will Increase With Decrease In The R Ionic Radius. This Indicated Whitish Grains Were $Smfeo_3$.

The Grains In The Unsubstituted Sample Are Inhomogeneous I.E., The Grains Are Affected By Certain Stress, While The Grains For The Fe Substituted Sample Are Nearly Homogeneous Due To The Decrease Of Stress. The Photographs Confirm These Results That The Stability Increased For Substituted Samples.

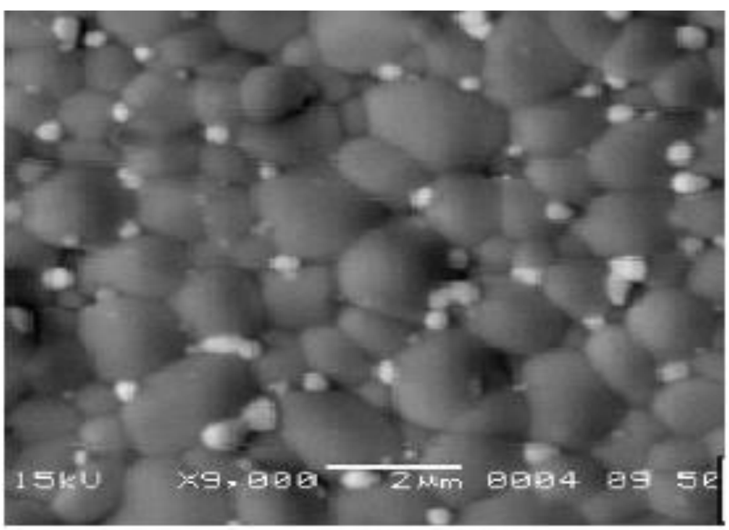

Fig.1 $Mgsmxgdyfe2_{-X-Y}o_4$ X = 0.012, Y = 0.001

3.2 Hysteresis Studies

Fig. Shows The Variation In Saturation Magnetization (Ms) For The Different X Values Of $Mgsm_xgd_yfe_{2-X-Y}o_4$ Ferrite, The Saturation Magnetization (Ms) Value Increases Within Increase In The Value Of X In Table 1. The Increasing Gadolinium Content Induced A Polar-To-Nonpolar 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 Gadolinium Substitution Resulted In The Appearance Of Spontaneous Magnetization, Which Was Significantly Enhanced Upon The Composition-Driven Transition From A Rhombohedral To An Orthorhombic Phase.

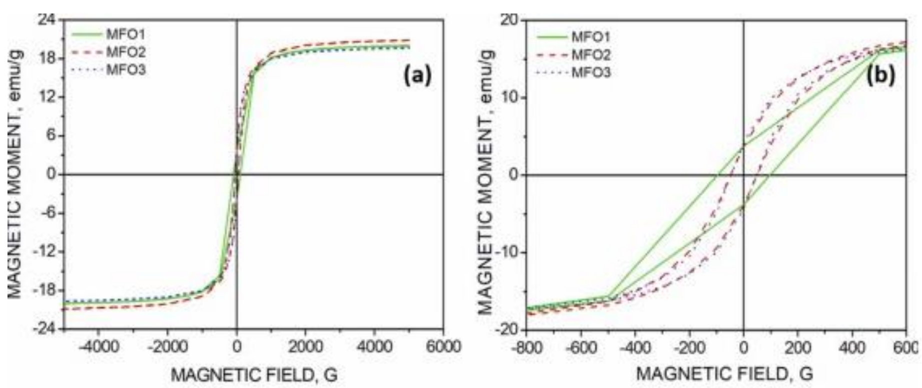

Fig.2 $Mgsmxgdyfe_{2-X-Y}o_4$ X = 0.012, Y = 0.001

Table 1 Show That The Change In Retentivity And Change Of Coercive Force With Change In Concentration Respectively. The Coercive Force Show Linear Increase Initially And Later Shows A Gradual Decrease With The Increase In The Dopant Concentration. The Values Of Magnetic Parameters Such As M_s, H_c, M_r Of Nano Particles Of $Mgsm_xgd_yfe_{2-X-Y}o_4$ Ferrite Obtained From The Vsm Data Are 25.0 Emu/Gm,723.24g, 11.507 Emu/Gm Respectively. As Reported M_s Value For Bulk Particle Of $Mgfe_{2-X-Y}o_4$ 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 .

3.3 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 3.5×10^4 M^{-1} To 5.8×10^4 M^{-1} Are Given In Fig. The Spectra Show Two Major Absorption Bands In The Given Frequency Range. The High And Low Frequency Absorption Bands (Y_1, Y_2) Are Observed In A Frequency Range Of 5.59×10^4 M^{-1} To 5.73×10^4 M^{-1} And 3.54×10^4 M^{-1} To 4.11×10^4 M^{-1}, Which Is Attributed To Tetrahedral And Octahedral Complexes Fe^{3+}–O^{2-} These Two Bands Have Been Reported By Waldron In Spinel Structure Of Ferrite. No Shift Of Absorption Band Y_1 Is Observed. The Absorption Band Y_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. This Suggests That The Rare-Earth Ions Occupy The B-Site. The Difference In Frequencies Between Y_1 And Y_2 Is Due To Changes In Bond Length (Fe^{3+}–O^{2-}) At Tetrahedral And Octahedral Sites. The Broadening Of The Y_2 Band Is Observed In Rare-Earth Added $Mgfe_{2-X-Y}O_4$. Which Suggests The Occupancy Of Rare-Earth Ions On The B-Sites .

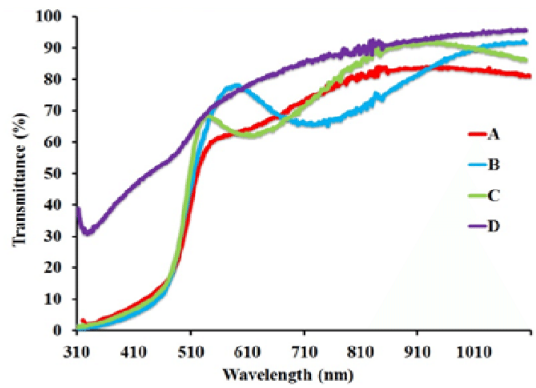

Fig.3 Ftir Absorption Spectra Of The Samples.

3.4 X - Ray Diffraction

Fig. Shows The X-Ray Diffraction Patterns Of Pure $Mgfe_{2-X-Y}O_4$ And X Of Samarium Ions Added To $Mgfe_{2-X-Y}O_4$. The X-Ray Pattern Shows Reflection Plane (220), (311), (222) , (400), (422), And (440). It Was Observed That The Appearance Of Plane (222) Is There In All The Sample Patterns Clearly Indicate That The Pure $Mgfe_{2-X-Y}O_4$ Shows The Presence Of Single-Phase Cubic Spinel Structure. The Secondary Phase (Orthorhombic) Is Observed In Rare-Earth Ions Added Samarium Ferrites. The Observed Secondary Phase Includes Rare Earth Orthoferrite $Smfeo_3$. It Is Well Known That The Degree Of Replacement Of The Host Cations By The Other Ions In The Host Lattice Depends On The Cations Radius Of The Substituent .The Lattice Constant *A* (Nm) Of Spinel Structure Could Be Calculated For Prominent Peak (311) Using Bragg's Equation

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

Where *Hkl* Are The Indices Of Mentioned Planes.

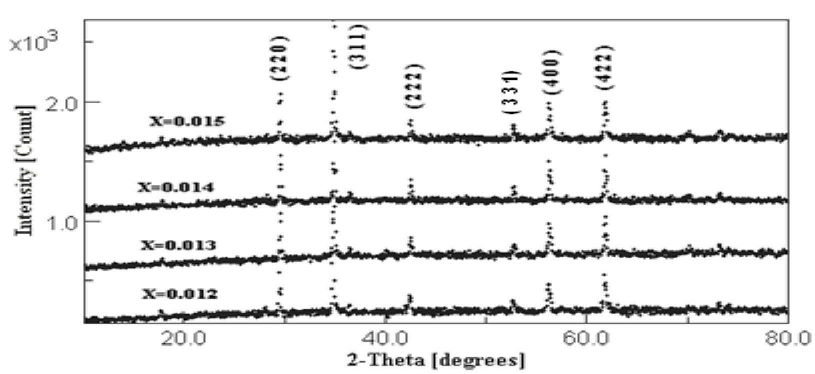

Fig.4 Xrd Patterns Of Sintered $Mgsm_xgd_yfe_{2-X-Y}O_4$, Ferrite With Different Sm And Gd Content

(Where X = 0.012, 0.013, 0.014, 0.015 & Y = 0.001)

Lattice Constants Of All Samples Prepared In Investigation Are Listed In Table 2. The Lattice Constant Is Smaller Than Pure Mgfe$_{2\text{-X-Y}}$o$_4$ And Increases With The Increase In Radius Of Sm 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) By The Debye Scherrer Formula .

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

Xrd Patterns Exhibit Narrow Reflection That Points To The Narrow Size Crystallites. The Results Are As Shown In Table. The Mean Crystallite Size Of The Sample Lies In The Range Of 43.88 Nm To 72.78 Nm. The Crystallite Size Of R Ions Added Sample Is Smaller Than Pure Magnesium Ferrite.

Sample	Lattice Constant(A□□	Particle Size(Nm)
X=0.012 ; Y=0.001	8.4235	72.78
X=0.013 ; Y=0.001	8.4208	70.37
X=0.014 ; Y=0.001	8.4167	64.36
X=0.015 ; Y=0.001	8.4156	43.88

Table.1 Xrd, Cation Distribution Of Mgsm$_x$gd$_y$fe$_{2\text{-X-Y}}$o$_4$

Material	Ra	Rb	Ro	.57735ra+Rb+2.0951ro =A	1.33 Ra*Ra+0.0675r0* R0-0.6rar0=B	A+√(A* A-1.866b)= C	A=C/1.10 6
Samarium	13.31164	94.75924	124	362.2371	283.1697	723.7441	654.3799
	31.90992	167.5227	124	445.7383	18.04513	891.4388	806.0025
	34.58824	162.9466	124	442.7085	55.65558	885.2997	800.451
	37.2665	155.018	12	436.3267	112.3473	872.4132	788.8004

	6	5	4				

Table.2 Lattice Parameters As A Function Of Sm Content

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 Doped $Mgfe_{2\text{-}X\text{-}Y}O_4$. Since Saturation Magnetization And Coercive Force Increases With The Increase In Sm, These Parameters Will Be Very Useful For The Application Of The Ferrite Materials Inorganic Gas Sensor Studies.

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