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Executive summary of the minor research project:

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- 3. Title of Research Project:

Studies on humidity and gas sensing properties of rare earth Metal oxide added magnesium cadmium ferrites

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1. INTRODUCTION

Ferrites belong to the class of ferrimagnetic materials having spinel structure (after mineral spinel Mg Al₂O₄). Ferri magnetism is a special case of ferromagnetic materials having partially compensated anti parallel spins, resulting in to non – zero magnetic moment. Hence spontaneous magnetization occurs. Ferrimagnetic substance consists of self saturated domain and exhibit magnetic, hysterics, saturation etc. Ferrites are compounds of mixed oxide which contain iron oxide as the main constituent. They have wide range of electrical resistivity of 10^2 to $10^{11} \Omega$ cm. Hence they can be considered as a magnetic semiconductor.

During last few decades the extensive development was done in the field of ferrite by main contributors and they are found to be technically useful magnetic materials. The basic properties of the ferrites are dependent on the kinds of metal atoms and their geometric arrangement among the interstices of closed packed cubic array of oxygen atom in the spinel structure. They are ceramic material with high permeability combined with high resistivity. The technical applications of ferrites depend on saturation magnetization, Curie temperature, gram size etc. Commercially important ferrites require high purity chemical homogeneity, fine grain size, and high density.

The ferrites are used in electric & magnetic devices used in industry, consumer electronics, computers, low power transformers, cable T.V. system television receives, switch mode power supply, aerospace navigational system and specialized commercial and military communication systems. Ferrites are used in the high frequency range due to high resistivity, high permeability, mechanical hardness, Curie temperature, low porosity, chemical stability. Mixed ferrites have been commercially used in radio frequency circuits, high quality filters, and rod antennas, read / heads for high speed digital tapes and operating devices.

The substitution of magnetic or non-magnetic magnet ions for ferrimagnetic ions in ferrites enhanced their magnetic properties and modifies their microstructure as well as electrical & magnetic properties. The ferrites like cadium, zinc, magnesium, nickel etc are found useful in the field of sensors like temperature sensor, gas sensor, humidity sensor, pressure sensor etc. Many researchers have reported the ferrites to be gas sensor for LPG, Cl₂, Co, CH₄, C₂H₅OH, H₂S & NH₃ and also humidity sensor.

2. EXPERIMENTAL DETAILS

Polycrystalline ferrite with general formula $Mg_{1-x}Cd_x$ Fe₂O₄ (0.0, 0.2, 0.4, 0.6, 0.8 and 1.0) with 5% addition of R ions were prepared by the oxalate co-precipitation method using sulphates of analytical reagent of (AR) grade. The high purity starting materials $MgSO_47H_2O$ (purity 99.99%, Sd fine), $3CdSO_48H_2O$ (purity 99.99%, Sd fine) FeSO_47H_2O (purity 99.5%, Thomas Baker) and Sm_2 (SO₄)₃8H₂O (purity 99.9%, Alafa Aesar), La₂(SO₄) ₃8H₂O (Alfa Aesar) were used in desired stoichiometric proportion and dissolved in distilled water. The pH of the solution was maintained at 4.8 by adding drop wise concentrated H₂SO₄. The resulting solution was heated at 80^oC for 1 h in order to complete the ionization of metal sulphate. The solution of ammonium oxalate (AR) was added in the solution by stirring until the process of precipitation is completed.

The resultant precipitation containing the solid solution of cadmium oxalate, magnesium oxalate, ferrous oxalates and R ions (R = Sm, Y and La) oxalates, was digested on sand bath for 1h in order to settle down the precipitate at bottom of the beaker. Further it was filtered by using Whatman filter paper no. 41 with the help of suction flask operating on the vacuum pump and finally washed with distilled water in order to remove sulphate ions. The absence of sulphate ion in the filtrate was confirmed with barium chloride test. The coprecipitate product was dried and presintered at 700^oC for 6 h in air. The presintered powder was milled in an agate motar with AR grade acetone as a base and pressed in the form of pellets at pressure of 7 tones/cm² by using hydraulic pressure machine. Polyvinyl alcohol 2 % wt was used as a binder. The size of pellets was 1.3 cm diameter. The pellets were finally sintered at 1050^oC for 5 h in air followed by slow cooling in the furnace at room temperature.

The ferrite phase formation of sintered powder was confirmed by powder X-ray diffraction (XRD) technique using a Philips PW-3710 X-ray diffractometer with CuK_{α} radiation (λ = 1.5424 A°). The crystallite size is calculated by Scherrer formula. The morphological feature of fractured surface of the sintered pellet at 1050^oC was examined with a scanning electron microscope (model JEOL – JSM 6360). FTIR absorption spectra of

powdered sample were recorded on a Perkin-Elmer spectrum one spectrometer by KBr pellet technique between 350 cm⁻¹ to 800 cm⁻¹. The physical density of palletized samples was measured by using Archimedes principle.

Dc resistivity was measured from two probe method. Magnetization measurement of the pelletized samples was measured at room temperature using high field loop tracer. Curie temperature of sintered pellet was measured by Loria-Sinha technique. The gas sensing of ferrite pellet were performed in temperature range of 30^oC to 400^oC. The sensor resistance was measured using a conventional circuit in which the element was connected with an external resistor in series at a circuit voltage of 10 V. Relative humidity (%RH) is measured by using standard hygrometer (test chamber) along with a thermometer. The optimization of preparative parameter is presented in table I.

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Sr.No.	Preparative parameter	Range
1	Presintering temperature	700 ⁰ C
2	Sintering temperature	1050 ⁰ C
3	Sintering time	5 hour
4	Rate of heating and cooling	80 ⁰ C/h
5	pH of solution	4.8

3. RESULT AND DISCUSSION

3.1 XRD analysis

The typical XRD patterns of 5% R ions (R = Sm, Y and La) added $Mg_{1-x}Cd_x Fe_2O_4$ (x = 0.0, 0.2, 0.4, 0.6, 0.8 and 1.0) presented in figure. The allowed planes in the diffraction patterns confirm the formation of cubic spinel structure in pure ferrite.



The planes corresponding to peaks (220), (311), (222), (400), (422), (333)/ (511), (440) and (533) have been observed. It further shows that, R ions ion induces second phase indexed by planes (210), (310), (321) and (432) which corresponds to orthoferrite (RFeO₃). The appearance of second phase is mainly explained due to high reactivity of Fe³⁺ ions with R^{3+} ions near or on the grain boundaries. Although the radius of R^{3+} ions is larger than Fe³⁺ the phase contains a small quantity of R ions in the interstitial position of spinel lattice.

Using Bragg's equation the lattice constant 'a' is calculated for prominent peak (311). The calculated and observed values of inter planer distances are in good agreement with each other.



The lattice constant of CdFe₂O₄ and MgFe₂O₄ prepared by oxalate co-precipitation method at low sintering temperature are very close to those reported by ceramic method. The lattice constant increases with increase in Cd²⁺content and shows non linear behavior. The increase in lattice constant with Cd²⁺ content can be explained on the basis of difference in ionic radii of constituent ions. This behavior is appeared clearly in the physical properties such as density, porosity, ionic radii and bond length. Due to addition of R³⁺ions in the Mg-Cd ferrite the lattice constant decreases slightly. Such reduction in the lattice constant in rare-earth substituted ferrites suggests the occupancy of rare earth ion on B-sites.

The average crystallite size of all the samples is determined from the full width half maxima of the (311) reflection in the XRD patterns by using the Debye Scherrer formula. The average crystallite size 'D' of the samples prepared under investigation is lies in the range of 28.86 nm to 32.06 nm (Table 2). This shows that the synthesized powder consists of nano crystallites. The average crystallite size of samples prepared by oxalate co-precipitation method is smaller than that reported by ceramic method. Crystallite size decreases in La³⁺ added samples while it slightly increases in case of Sm³⁺ and Y³⁺ samples. This deviation is due to method of preparation.

TABLE II

Sr.	Parameter	Pure	R ions added Mg-Cd		
No.		Mg-Cd	Mg-Cd + 5%	Mg-Cd +	Mg-Cd +
			Sm ³⁺	5% Y ³⁺	5% La ³⁺
1	LATTICE CONSTANT	8.37 - 8.71	8.37 - 8.64	8.37 - 8.70	8.36 - 8.70
	(A ⁰)				
2	CRYSTALLITE SIZE	27.79 - 30.40	28.69 - 32.66	28.86 - 32.06	25.65 - 30.26
	(nm)				
3	GRAIN SIZE (µm)	1.16 - 2.5	0.69 - 0.99	0.71 - 0.99	0.36 - 0.66
4	X-RAY DENSITY	4.52 - 5.78	4.19 - 6.27	4.70 - 6.02	4.89 - 6.12
	(gm/cm ³)				
5	PHYSICAL DENSITY	4.37 - 4.79	4.008 - 5.64	4.57 - 5.10	4.61 - 4.95
	(gm/cm^3)				



The values of X-ray density (ρ_x) and physical density (ρ_p) of the sample are given in Table II. It revealed that, the X-ray and physical density for all compositions of Mg-Cd

ferrite increases with increase in cadmium content. The X-ray densities (ρ_x) are higher than physical densities (ρ_p). The physical densities of the samples are 86.96% to that of X-ray densities. In this case the increase in X-ray density with Cd²⁺ content suggests that the increase in mass overtakes the increase in volume of the unit cell. The X-ray and physical density of R ions added Mg-Cd ferrite is higher than pure Mg-Cd ferrite.



The tetrahedral and octahedral ionic radii (r_A , r_B) sites, the bond lengths on tetrahedral (A-O) and octahedral (B-O) sites of cubic spinel structure by using Standely's equations. It is observed that, the tetrahedral and octahedral site radii increases with increase in cadmium content. This is due to increase in lattice constant with increase in cadmium content. The ionic radii in A-sites are less than those in B-sites for all the Cd containing samples. This may be due to the migration of Fe³⁺ ions (ionic radii 0.67A°) from A-site to B-site. The bond length on A-site is less than that of B-site. It is also clear that the tetrahedral and octahedral bond length increases with increase in Cd²⁺ content. The increase of bond length is associated with the increase in the lattice constant. This is because the smaller ionic radius of Fe³⁺ (0.67A°) ion is replacing the larger ionic radius of Cd²⁺ ion in spinel lattice

from B-site to A-site. In R^{3+} ions added sample both bond length and site radii on B-site increases.

3.2. Scanning electron microscopy

SEM micrographs of fractured surface of pellet sintered at 1050° C for all the composition prepared under investigation are shown in figure. It is evident that morphology of R ions added sample deviate from fine structure of the pure ferrite. The addition R ions shows formation RFeO₃ crystalline phase as confirmed by XRD patterns. The formation of crystalline secondary phase fills intergranular voids and shows good densification of rare earth containing ferrite.



The average grain size for all the samples is is tabulated in Table 2. It is observed that the average grain size increases with increase in Cd^{2+} content, which can be attributed to the higher mobility of Cd ions induced in liquid phase sintering. The average grain size of R^{3+} ions added samples are lower than that of pure Mg-Cd ferrites. It is an established fact

that the grain growth takes by grain boundary migration. The addition of R^{3+} reduces the grain growth probably due to the fact that R^{3+} deposites on the grain boundaries and forms thin layer, which in terms hambers its motion or migration.

3.3. FT IR Study

The Infrared spectra of ferrite system 5 % R ions added $Mg_{1-x}Cd_xFe_2O_4$ (x = 0.0, 0.2, 0.4, 0.6, 0.8 and 1.0) with shows two major bands in the range 350 cm⁻¹ to 800 cm⁻¹. The high frequency band (v₁) is in the range 554 cm⁻¹ to 579 cm⁻¹ and the lower frequency band (v₂) is in the range 427 cm⁻¹ to 472 cm⁻¹. These bands are common characteristics of spinel structure. The absorption band (v₁) is caused by scratching vibrations of the tetrahedral metal -oxygen bond and absorption band (v₂) is caused by metal-oxygen vibrations in octahedral sites.



These band positions are expected because of the difference in the Fe ³⁺- O ²⁻ distances for the tetrahedral and octahedral complexes. The shifting and splitting is absent in pure Mg-Cd ferrites. By adding R ions, the absorption band v_2 shifts to higher frequency side and also band broadens which indicates that the occupancy of R³⁺ on octahedral B site.

From figure, it is also observed that the intensity of v_2 bands decreases while broadening increases. It is reported that the system in more disorder state gives broader and less intense band in IR spectra.

4. DC RESISTIVITY

The dc electrical resistivity (ρ) was measured in the range 300-800 ⁰K. It is observed that electrical resistivity of all the samples decreases with increase in temperature, suggesting the semiconducting nature of the samples. It is also observed that resistivity of the samples increases with increase in Cd²⁺ content. Conduction in ferrites is due to hopping of electrons between Fe²⁺ to Fe³⁺ ions. It is reported that Cd²⁺ ions prefer the occupation of tetrahedral A- sites, Mg²⁺ and Fe³⁺ ions partially occupy the A and B-sites.



The increase in Cd^{2+} content decreases the Fe^{3+} ion concentration at A-site. This will force Fe^{3+} ions to B-sites. Due to this, the number of Fe^{2+} and Fe^{3+} ions responsible for electric conduction in ferrites decreases at A-site. This will results the increase in resistivity with increase in Cd^{2+} content. The activation energy ΔE in paramagnetic region is greater than in ferromagnetic region. This can be attributed to effect of the magnetic ordering in the conduction process. The activation energy decreases with increase in cadmium content.

The resistivity of R ions added samples increases. It is well known that the conduction mechanism in ferrite is mainly due to hopping of charge carriers between Fe^{2+} and Fe^{3+} ions inside the grain inhibits the motion of electronics between the Fe^{2+} and Fe^{3+} ions, which leads to an increase of electrical for all R ions added samples.

5. MAGNETIZATION AND CURIE TEMPERATURE MEASUREMENT

It is observed that the magnetic properties are changed by the substitution of Cd^{2+} content and also method of preparation. The parameter Ms, Mr, Mr/Ms, 4π Ms and n_B increase from 16.90, 2.68, 0.158, 920.746, 0.605 to 30.44, 8.71, 0.286, 1705.17, 1.282, for x = 0 to x = 0.4 and thereafter decrease with increase in x. The decrease in these values results from the existence of spin canting, which has been reported in several nanometer-sized ferrites. The low values of Mr/Ms ratio indicate an appreciable fraction of superparamagnetic particles. Mr/Ms increases to 0.286 for x =0.4 and then decreases to 0.13 indicating a fraction of the particles is in the blocked state.



Saturation magnetization shows a maximum of 35.4 emu/g at x=0.4 and decreases to 2.46 at x = 1. These values are lower than the bulk value for the magnetization of the Mg-Cd ferrites prepared by ceramic method at 1000 ^oC for 48 h (50.12 emu/g) and it is also lower than the reported value (44.81 emu/g). The change of coercive force may be due to the low crystalline anisotropy, which arises from crystal imperfection and the high degree of aggregation.

The saturation magnetization increases with Cd^{2+} content up to x = 0.4 and then goes on deceasing. It suggests the existence of canted spins giving rise to Y-K angles comparing the strength of A-B and B-B super exchange interaction. Neel's two sublattice model is applicable to the system for $x \le 0.4$ and the Niessen's three sub lattice models are predominant for $x \ge 0.4$. The calculated Y-K angles are found to increases with increasing Cd^{2+} content. The observed variation of saturation magnetization and magnetic moment with Cd^{2+} content can be explained on the basis of Neel's two sublattice model. According this model, three kinds of exchange interaction viz. A-A, B-B and A-B are possible. A-B interaction is effective and stronger than the other two. The occupancy of Cd^{2+} at A-site successively reduces the Fe³⁺ ions on A- site and equal number of Fe³⁺ ions are transferred to the B-site. This slowly builds up the strength of A-B interaction and hence the magnetization and magnetic moment of the sample increase with Cd^{2+} content. Beyond certain limit of Cd^{2+} . A- site moment is weak to affect B site moments. Therefore B-B interaction become stronger and A-B interaction decreases. The value of Y-K angles goes on increasing there by reducing the net magnetization.

Curie temperature (T_c) is measured by Loria Sinha method and that observed in DC resistivity are good aggrement with each other. From this study it can be seen that the Curie temperature decreases with increasing cadmium content. It is attributed to decrease in

A-B intraction between Cd^{2+} and Fe^{3+} ions. The Curie temperature decreases with addition of rare earth ions and confirms the fact that R-R ions interactions are very weak.

6. HUMIDITY AND GAS SENSING PROPERTY

The progress in information processing techniques, development of microprocessors, electronics and computer technologies are necessary in development of sensors. Microprocessors are used in measuring and control systems. This increases the role of sensors acting as the primary unit of information perception and demand for their growth. Sensors become important factors in automation, an air quality monitoring and robot technology and its gain an increasing greater significance as structural elements of systems. The sensors are device which converts physical or chemical quantity into electrical signals convenient to use.



Sensors can be classified according to operating principle, type of out put signal, their purpose, the materials and their technology of production. According to principle the sensor can be classified as physical and chemical ones. Physical sensors employ physical effect like piezoelectric, magnetostrictional, ionization, thermoelectric, photoelectric, magneto electric etc. Chemical sensors include sensors in which as a result of chemical adsorption electrochemical reaction etc.

The sensitivity is a degree of the influence of certain gas on the resistance of sensor. It is generally determined by ratio of the resistance Ra in the air to the resistance Rg in a gas with given concentration Cg,

$$S_{g(\%)} = \frac{\text{Ra} - \text{Rg}}{\text{Ra}} \ge 100$$

It is a function of concentration of gas and nature of sensing material. In oxidizing gas medium the resistance of sensor rises and decreases in reducing gas atmosphere.

The effect of the partial substitution of rare earth (R) ions (R = Sm, Y and La) in MgFe₂O₄ and CdFe₂O₄ on the humidity characteristics was also investigated. The measurements of relative humidity show an increase in ρ addition of Cd²⁺ in MgFe₂O₄ ferrites. The partial substitution of octahedral Fe³⁺ by rare earth (R) ions localizes the octahedral Fe²⁺ ions and thus, the number of the electronic jumps between Fe²⁺ and Fe³⁺ ions (hopping conduction mechanism in ferrites) decreases. Moreover, microstructure (porosity, grain size, structural defects) has a great role on the electrical resistivity. A smaller grain implies an increase of the grain boundary surface which normally account for high resistivity of a polycrystalline material.

The humidity sensitivity depends upon the microstructure. The larger the specific surface area and porosity of the specimens the more water vapors can be physically adsorbed, resulting in a larger decrease of the resistivity. The R ions added ferrite is more sensitive to humidity change. Its electrical resistivity decreases then the relative humidity increases from 10% to 75%. In comparison with R ions added ferrite, the unadded ferrite is less sensitive to relative humidity. It is thought that R ions have preferable effects on the sensitivity to water vapors (humidity). The humidity response varies from 30 to 75 % and 75 to 30 % for MgFe₂O₄ and lower values for CdFe₂O₄. The response decreases for rare earth ions. These results may suggest that the adsorption or desorption rate of water vapors is controlled by the diffusion rate of these through the microspores which in its turn is dependent on the pore size distribution. The R ions added MgFe₂O₄ ferrites have greater pores than CdFe₂O₄ ferrites, which shows a longer response time to humidity changes.



The changes in particle size and surface area suggest that the addition of R ions (R = Sm, Y and La) in solid solution occurs during co-precipitation preparation which enables a better homogeneity in the powders and hence, a more controlled microstructure. The more porous structure of this sample impedes the motion of charge carriers that will lead to an increase of the resistivity. Moreover, the tetravalent ions are known to localize the Fe²⁺ ions if they are present on the same sublattice and reduce Fe²⁺ \leftrightarrow Fe³⁺ hopping conduction.

Presence of R ions on B sublattice is the most effective in this regard because Fe^{2+} ion has a preference for this site.

The gas sensing property of Mg-Cd for LPG and ethanol saturated vapors in air was investigated. The gas sensitivity is strongly related to the working temperature, material composition, mean particle size and porosity. The pure magnesium and Cadmium ferrite are less sensitive to both the gases. The rare earth ions added ferrites are more sensitive to LPG and ethanol. It can be concluding that the gas sensitivity is thermally activated and depends on gas type and ferrite composition. The gas sensitivity increases with increasing the operating temperature and reached a maximum value at an optimum temperature of about 300°C, except pure samples. It is evident that the sensitivity to LPG is higher than that to ethanol for all the R ions added samples. This is attributed due to the smaller grain size and higher surface area. Also, the porous structure promotes the increase of the sensitivity. These results suggest that the role of R ions in MgFe₂O₄ ferrite is to facilitate the oxidation of reducing gases. The time taken by this sensor element at the optimized temperature of 300°C to reach about 90% of the maximum sensitivity was found to be 5 minutes and the time taken by the sensor to come back once the gas was removed is found to be longer, of about 9 minutes.

7. Conclusions

The R ions added Mg-Cd ferrites have been prepared successfully using oxalate coprecipitation method at a sintering temperature 1050° C for 5 h. The average crystallite size for all the samples lies in nano-particle range of 25.65 nm to 32.66 nm. The lattice constant increases with increase in Cd²⁺ content. The grain size of the sample increases with increase in Cd²⁺ content. An addition of R ions decreases the grain size of the samples. The addition of R³⁺ ions shifts the v₂ band towards the higher frequency side. The broadening of band is also observed, which suggests the occupancy of R^{3+} ions on octahedral B- site. DC resistivity decreases with temperature and increases with increase in Cd²⁺ content. The saturation magnetization of rare earth ions added Mg-Cd ferrite is higher. The gas and humidity sensitivity is improved by rare earth ion addition.

The R ions added ferrites exhibit better humidity sensitivity in comparison with pure ferrite samples. Also, this ferrite shows a shorter response time. These results show that the promoting effects of R ions addition on humidity sensitivity of Mg and Cd ferrites can be related with the changes in porosity and grain size (specific surface area). The influence of R ions (R = Sm, Y and La) on gas sensitivity of MgFe₂O₄ and CdFe₂O₄ ferrites, was studied. It was evidenced the presence of a secondary phase in the R ions containing ferrite induce structural changes regarding grain sizes and porosity. The sensitivity measurements to LPG and ethanol vapors evidenced that the gas sensitivity largely depends on the ferrite composition, working temperature and gas composition. But further investigations are necessary to clarify the exact role of R ions which is responsible for improving the sensitivity ferrite.

Publication:

^{1.} Structural analysis of Y ³⁺ doped Mg-Cd ferrites prepared by oxalate co-precipitation method, *A. B. Gadkari, T. J. Shinde and P. N. Vasambekar,* Material Chemistry and Physics, 114 (2-3) (15 Apr. 2009) 505-510.

^{2.} Structural and magnetic properties of nano-Crystalline Mg-Cd ferrites by oxalate coprecipitation method, *A. B. Gadkari, T. J. Shinde and P. N. Vasambekar, Journal of* Material Science:Material in Electronics, (online April 2009)

^{3.} Structural analysis of Sm³⁺ added nanocrystalline Mg-Cd ferrites prepared by oxalate coprecipitation method, *A. B. Gadkari, T. J. Shinde and P. N. Vasambekar*, Materials Characterization, (on line 22 June 2009)

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