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ORIGINAL ARTICLE

EFFECT OF Al^{3+} ON STRUCTURAL AND MAGNETIC PROPERTIES OF NANOCRYSTALINE COPPER FERRITE

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Abstract:

Nano-crystaline Alluminum substituted copper ferrite samples $CuFe_{2-2y}Al_{2y}O_4$ (where y=0.0,0.05,0.15 & 0.25) have been prepared by mill scale fines via conventional oxide ceramic process. The effect of Al^{3+} on both structural and magnetic properties is studied. Archimedes's method as well as the universal testing machine was used for determining the structural properties of the samples. Meanwhile the types of phase formed and the magnetic properties of the produced samples are investigated using X-ray diffraction, Infra red absorption and vibrating sample magnetometer. Cation distribution is estimated on the basis of magnetic moment per unit cell in Bohr magneton calculations. Occupancy of Cu^{2+} ion on octahedral (B) site of the tetragonal spinel of $CuFe_2O_4$ produces tetragonal prolate type distortions in the lattice. The addition of Al^{3+} ion in the host lattice suffers the prolate type distortion which reduces tetragonality (c/a) ratio. The lattice constant, X-Ray density, physical density, porosity, particle size, site radii (rA and rB), bond lengths (A-O and B-O) are calculated for the samples. Two prominent infrared absorption bands for all ferrite samples are observed; one at 600 cm^{-1} due to tetrahedral (A) interstitial voids and other at 400 cm-1 due to octahedral (B) interstitial voids is observed. All Al^{3+} substituted copper ferrite samples exhibits the single domain to supper paramagnetic (SD – SP) transition near Curie temperature.

KEYWORDS:

Nano-crystalline ferrite, substituted copper ferrite, prolate type distortions, Tetragonal distortions, Tetragonality ratio, single domain to super-paramagnetic (SD-SP) transition.

INTRODUCTION:

Copper ferrite exhibits inverse spinel tetragonal structure (1-3). Huheey (4) reported that Cu^{2+} (d^9) ion is the John-Teller ion and its unpaired electron in dz^2 orbit causes the elongation of Tetrahedron to the tetragonal shape. Degree of inversion in copper ferrite depends upon heat treatment during the preparation(5).When the concentration of Cu^{2+} ion is larger on octahedral (B) site than tetrahedral (A) site, it produces the square bond SP^2d orbital (6), that would give rise the macroscopic tetrahedral observable crystal structure. Tetragonality ratio for slow cooled copper ferrite is reported (7-9) in the range of 1.03 to 1.07 when Cu^{2+}, Mn^{3+}, Cr^{3+} occupies (B) site and produces prolate distortion (c/a) >1 and when Cr^{3+}, Mn^{4+},

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Ni^{2+} occupied on (A) site produces oblate distortions (c/a) < 1, (10, 11).When critical factor of these elements occupy either sites then they only distort the lattice. Cu^{2+} produces tetragonal distortions in the cubic spinel. 70% of copper occupies on (B) site (12). 10 to 40% occupancy of Cu^{2+} at (A) site in $CuFe_2O_4$ is reported (13). It is interesting to study the nature of distorted inverse spinel tetragonal structure of copper ferrite by substituting Al3+ ion in the lattice of copper ferrite.

EXPERIMENTAL PROCEDURE:-

Stichiometric compositions of $CuFe_{2-2y}Al_{2y}O_4$ (where y=0.0,0.05,0.15 & 0.25) nano-particle size polycrystalline ferrites are prepared by standard ceramic technique by using AR grade Fe_2O_3, Al_2O_3 and CuO. The sintering process was carried out at 1000^0C for 48 hours. The completion of the solid state reaction was confirmed by X-ray powder diffractometry. The sample was characterized by Infrared absorption spectroscopic method. Saturation magnetization of each composition was carried out using high field hysteresis loop tracer.AC susceptibility of slowly cooled samples was measured in the temperature range 300-800 0K using Helmholtz's double coil set-up operating at 263 Hz with constant field of 7 Oersted.

RESULT AND DISCUSSION:-

The X-ray diffraction patterns of the samples are presented in (fig.1). Powder X-ray diffractometry of the ferrite samples reveals the single phase spinel structure, as well defined reflection is observed without any ambiguity. The diffraction peaks are corresponding to (200), (311), (400), (422), (333/511), (440) and (533) planes. The lattice

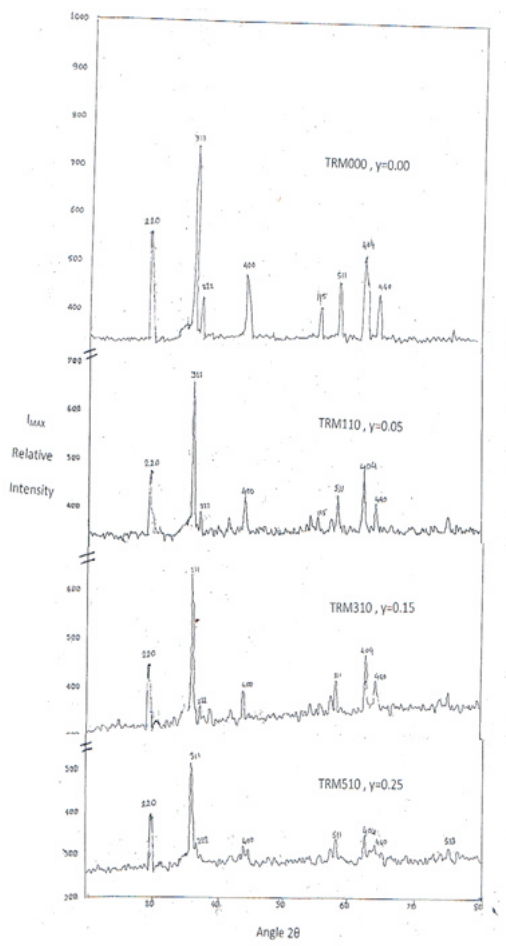

Fig.1 XRD pattern of ferrite system Cu Fe_{2-2y} Al_{2y} O_4 (y=0.0, 0.05, 0.15 & 0.25)

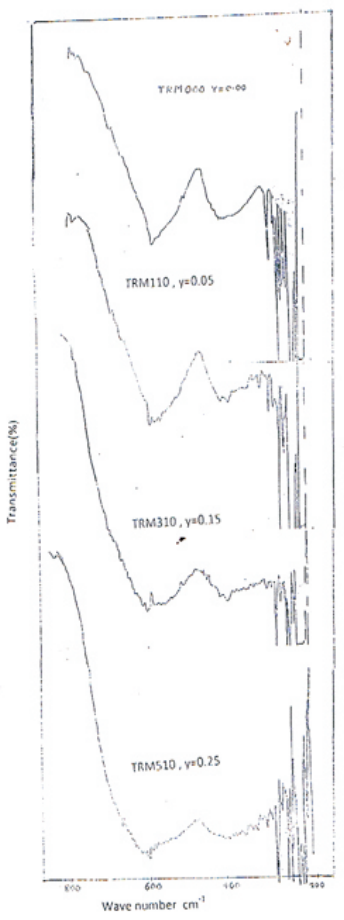

Fig.2 Infrared absorption spectra of Cu Fe_{2-2y} Al_{2y} O_4

constants 'a' and 'c' for all prepared samples are calculated by using prominent (311) XRD peak. The calculated and observed values of inter planer distance (d) are found in good agreement with each other for all reflections. The particle size (D) for all the ferrite samples is calculated by Debye Scherer formula , physical density (a), x-ray density (x), porosity (p), ionic site radii (rA, rB) and ionic bond lengths (A-O, B-O) are calculated from the formulae given by Gadkari et.al (14) and presented in (table 1). From the calculations of lattice constants 'a' and 'c' for all the prepared ferrites it is observed that c>a and tetragonality ratio (c/a) is found in the range of 1.03 to 1.07.This result is in good agreement with previous report (7-9).In this present report tetragonality ratio for copper ferrite is 1.06. It means 70% copper resides on B site and it exhibits prolate type distortions in the crystal lattice. The previous report (12) well supports the present results reported this communication. Both Fe^{3+} and Cu^{2+} are John-Teller ion which produces prolate type distortions on (B) site and hence c > a and (c/a) = 1.06.Therefore copper ferrite exhibits tetragonal spinel structure in host crystal lattice of copper ferrite. In addition of Al^{3+} content in tetragonality ratio is found decreasing. It means that Al^{3+} suppresses the tetragonal prolate type distortions on B site and hence (c/a) ratio decreases and automatically crystal lattice turned from tetragonal spinel to cubic spinel.

Figure 2 depicts that the infrared absorption spectra showing two distinct absorption band 1 due to tetrahedral (A) site interstitial voids near 600 cm^{-1} and other 2 due to octahedral (B)site interstitials voids near 400 cm^{-1}. Our results in this present communication are well supported by previous reports (15-16).

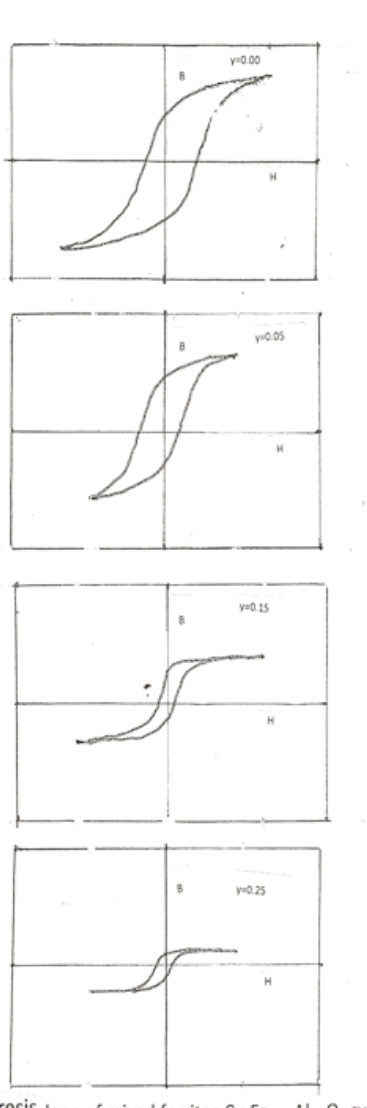

Fig. 3 Hysteresis loop of mixed ferrites Cu Fe_{2-2y} Al_{2y} O_4 system

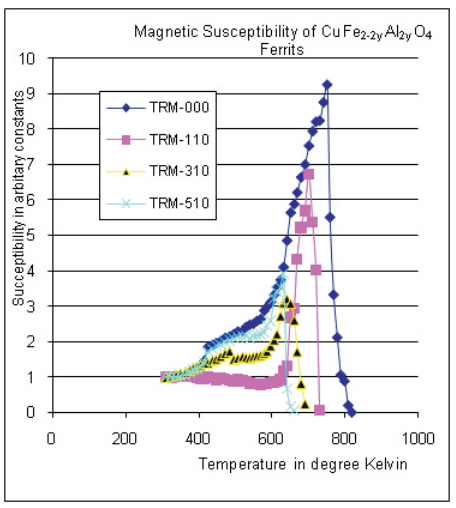

Fig.4 Magnetic Susceptibility of mixed ferrites Cu Fe_{2-2y} Al_{2y} O_4 system

Figure 3 depicts the variation of saturation magnetization (s), magnetic moments per unit cell in Bohr magnetron (nB), remnant magnetization (Br), coercive force (Hc) with Al3+ content which is presented in (table 3). All (s), (nB), (Br) and (Hc) decreases with Al^{3+} ion and hence it reduces all above values which are correlated with each other.

Close inspection of (fig.4) depicts the normalized AC susceptibility (/RT) as a function of temperature. For all prepared ferrite samples; normalized AC susceptibility slowly increases with increasing temperature up to certain point, beyond which it drops off sharply but goes on decreasing slowly with increase in temperature. Increase of normalized AC susceptibility up to crystallographic phase transition temperature (Tp) suggests that the prepared ferrite samples exhibit single domain structure, while the exponential decrease in the normalized AC susceptibility (paramagnetic tail) beyond (Tp) indicates the single domain to super paramagnetic (SD-SP) transition. The sharp drop in normalized susceptibility near phase transition suggests that impurity phases are not formed in the present ferrite samples. This fact is also confirmed by X-ray diffraction analysis. The paramagnetic tail indicates the existence of super paramagnetic cluster in the sample by addition of Al3+ in the host lattice of copper ferrite. The Curie temperature of such samples can be determined by drawing a tangent to the paramagnetic tail on the temperature axis. The TP and TC for all ferrite samples prepared are determined and presented in (table 4).Similar type behavior is observed by Karche et.al,(17) in $Cd_xMg_{1-x}Fe_2O_4$ ferrite sample for x = 0.4.The Curie temperatures estimated from normalized AC susceptibility variation with temperature experiment are in excellent agreement with their values measured by Loria Sinha method.

CONCLUSION:-

Addition of Al^{3+} content in the host lattice of the tetragonal copper spinel ferrite suppresses the tetragonal prolate type distortions and hence crystal structure turned into cubic spinel. All prepared ferrite samples exhibits single domain to super paramagnetic transition. Super paramagnetic cluster is increased due to addition of Al^{3+} in the host lattice of copper ferrite. Al^{3+} affect the structural properties and magnetic properties. Particle size of all prepared samples is found within the nano range.

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Table 1 X-ray characterization data of Ferrite $CuFe_{2-2y}$ Al_{2y} O_4 system

y	Lattice constant			Physical density gm/cc	X-ray density gm/cc	Porosity (P)	Particle size A.U.	Ionic Radii A.U. Tetrahedral (R_A)	Ionic Radii A.U. Octahedral (R_B)	Ionic Bond length In A.U. (A-O)	Ionic Bond length in A.U. (B-O)
	a	c	c/a								
0.00	8.169	8.677	1.0622	3.798	5.437	0.3015	10.55	0.528	0.695	1.901	2.043
0.05	8.205	8.666	1.0562	3.748	5.400	12.163	12.163	0.532	0.698	1.897	2.048
0.15	8.250	8.655	1.0491	3.718	5.224	12.154	14.154	0.541	0.701	1.895	2.051
0.25	8.399	8.650	1.0249	3.657	5.102	18.253	18.253	0.537	0.703	1.887	2.053

Table2 Infrared absorption data of Ferrite $CuFe_{2-2y}$ Al_{2y} O_4 system

Sample Id	Wave No. for Tetrahedral side γ_1 cm^{-1}	Wave No. for octahedral side γ_2 cm^{-1}
TRM000	600	400
TRM110	600	405
TRM310	600	410
TRM510	600	415

Table3 Magnetization data of ferrite $CuFe_{2-2y}$ Al_{2y} O_4 system

Sample Id	Y	Curie Temp. in ^{0}k		Saturation Magnetization (σ_s) in emu/gm	Magnetic moment per unit cell ($n_{B)}$ in Bohr magnetron
		from Loria sinha method	From magnetic susceptibility Expt.		
TRM000	0.00	748	768	30.35	1.300
TRM110	0.05	690	730	24.75	1.026
TRM310	0.15	651	700.5	14.17	0.5852
TRM510	0.25	631	668.5	11.21	0.4514

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