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The effect sintering temperature on structural and magnetic properties of Nano-crystalline Al^{3+} doped Magnesium-copper ferrites synthesized by Sol-gel method

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ABSTRACT

Aluminium doped Mg-Cu ferrite ($Mg_{0.5}Cu_{0.5}Fe_{1.7}Al_{0.3}O_4$) nanoparticles were synthesized by sol gel auto combustion techniques using respective metal nitrates with citric acid as fuel for combustion process. The resulting sample annealed at 400^0C and 700^0C temperature. The formation of ferrite nanoparticles with single phase spinel structure is evident from X-ray diffraction analysis of the as-burnt, sintered sample at 400^0C and 700^0C temperature. An intense XRD peak indicates the formation of nanoparticles. Average crystallite size calculated from most intense reflection peak (311) using Debye–Scherrer's formula is found to be nanometer in range and that increases with increase in sintering temperature. The lattice constant and other structural parameters evaluated from XRD data are in reported range. EDS data confirms that the reactant have under gone complete reaction. Magnetic study of the prepared samples is carried out using vibrating sample magnetometer (VSM). VSM data shows that the prepared samples are soft ferromagnetic materials.

Key words: Ferrite, nanoparticles, magnetic, X-ray diffraction, SEM, VSM.

INTRODUCTION

Ferrite materials are special class among the numerous magnetic materials as they are widely used magnetic materials as they have excellent electrical and magnetic properties [1, 2]. The interesting physical properties of ferrites arise from their ability to distribution of the cations among the available sites. Now a days nano-magnetic materials have attained great interest because of their potential applications in many fields like high-density data storage, ferro-fluid, magneto-optical recording, magnetic resonance imaging, drug delivery, magneto-caloric refrigeration etc [3].

Among the different spinel type ferrite material Mg-Cu ferrites are great importance because of their excellent chemical stability, high electrical resistivity, low eddy current and dielectric losses, low coercivity, and moderate saturation magnetization [4]. Due to these important properties they are widely used as magnetic materials at high frequency applications.

The number of methods has been used to prepare nanosized ferrite particles. Fine particle nature of nanosize can be achieved through number of methods which include chemical precipitation [5], micro-emulsion [6], sol-gel [7], hydrothermal process [8] etc. The role of synthesis variables such as pH, fuel, fuel ratio, annealing temperature and time are also important in governing the size of the particles and hence the physical properties. Among the various fuels citric acid is commonly used for sol-gel synthesis. No more work reported about synthesis and magnetic properties of Aluminium doped magnesium-copper ferrite synthesized by various methods.

In this study, we report the synthesis of aluminium substituted for iron in magnesium- copper ferrite nanoparticles using sol-gel auto combustion technique and sintered at 400°Cand 700°C. The characterizations of X-ray diffraction, scanning electron microscopy for structural and magnetic properties of ferrite were investigated. The structural, morphological and magnetic properties are presented.

METHODOLOGY

(A) Preparation method: A. R. grade precursors viz. aluminium nitrate [Al $(NO_3)_3$.9H20], magnesium nitrate [$Mg(NO_3)_2$.6H20], copper nitrate [$Cu(NO_3)_2$. 3H20] and ferric nitrate [$Fe(NO_3)_3$.9H20] were used to synthesize the nanoparticles. Citric acid [$C_6H_8O_7.H_2O$] was used as fuel. The stoichiometric amounts of nitrates were dissolved in de-ionized water with continuous magnetic stirring. Then the citric acid solution prepared separately was mixed in the metal nitrate solution. The moar ratio of acid to nitrates was maintained 1:1. The nitrate–citrate solution was neutralized by adding liquid ammonia to it and then allowed to evaporate till the formation of viscous gel by heating at 80°C on hot plate with continuous stirring. The gel is then burnt in to ashes of Al doped Mg-Cu ferrite nanoparticles by raising its temperature to 100°C.

The thermo-gravimetric analysis of the as prepared sample suggests the formation of spinel phase decomposition of all the precursors at about 400°C. Accordingly the as burnt powder was annealed at temperature 400°C and 700°C for 3 hour to obtain the spinel phase of the Al doped Mg-Cu ferrite nanoparticles.

(B) Measurements and Characterization: The sintered samples were analyzed by X-ray diffractometer (BRUKER AXS MODEL-D8 advance) using Cu-Kα radiation at room temperature in 2θ range from 20° to 80°. The lattice constant was determined from X-ray data analysis using formula.

$$\frac{Sin\ 2\ \theta}{H^2 + k + l^2} \quad \ldots 1$$

Were, 'a' is lattice constant, (hkl) represents Miller indices and 'λ' is the wavelength of X-ray used and 'θ' is the glancing angle.

The crystallite size 'D' was determined from the full width half maximum (β) of the strongest reflection (311) and by using the Scherrer's formula [9],

$$D = \frac{0.9\lambda}{B\cos\theta} \quad \ldots\ldots 2$$

Where, λ' is the wavelength of X-ray used and 'θ' is the Bragg's angle.

The X-ray density was calculated from lattice parameter (a) using formula,

$$d_x = \frac{8M}{N_A a^3} \quad \ldots\ldots 3$$

Where, M is molecular weight, N_A is the Avogadro's number and a is lattice constant.

The bulk density (d_b) samples were calculated by measuring mass and volume of pellet as

$$\text{Bulkdensity}(d_b) = \frac{\text{Mass of Pellet}}{\text{Thickness of pallet (h) X Cross section area } (\pi r^2)}$$

$$d_b = \frac{M}{h \pi r^2} \quad \ldots\ldots 4$$

Where, r is radius of pellet and h is thickness of pellet. From the x-ray density (d_x) and bulk density (d_b) values, porosity was calculated using the relation.

$$Porosity(P) = 1 - \left(\frac{d_b}{d_x}\right) \quad \ldots\ldots\ldots 5$$

Morphological study is carried out using scanning electron microscope (SEM). The magnetic properties were measured (Model- LAKESHORE Model-7307) at room temperature using vibrating sample magnetometer (VSM) technique**.**

RESULTS AND DISCUSSION

A) Structure properties: The structural analysis of $Mg_{0.5}Cu_{0.5}Fe_{1.7}Al_{0.3}O_4$ ferrite samples of as-burnt, sintered at 400°C and 700°C temperature was performed by powder XRD method. As-burnt, sintered at 400°C and 700°C temperature samples with sample code of ($Mg_{0.5}Cu_{0.5}Fe_{1.7}Al_{0.3}O_4$) is given in table 1. The X-ray diffraction (XRD) patterns of all above samples are as shown in figure 1.

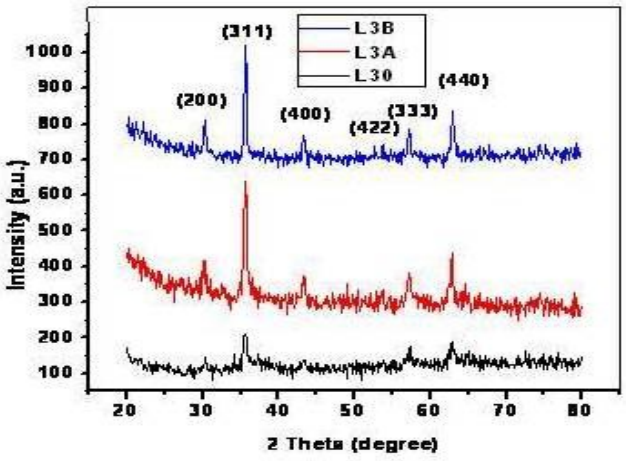


Figure 1: Powder X-ray diffraction (XRD) pattern of L30, L3A and L3B samples

Table1: Sample code of $Mg_{0.5}Cu_{0.5}Fe_{1.7}Al_{0.3}O_4$ sample as-burnt, sintered at 400°C and 700°C temperature

Temperature of sample	Sample code
As-burnt sample (27°C)	L30
400°C	L3A
700°C	L3B

Table 2: Various parameters of Aluminium doped Mg-Cu ferrites nanoparticle

Parameters	Unit	L30	L3A	L3B
Crystallite size(D) nm	Nm	20.4	32.53	48.62
Lattice constant (a)	A°	8.3532	8.3656	8.3949
Unit cell volume	$(A°)^3$	583	585	592
X-ray density (d_x)	g/cm^3	4.8072	4.7857	--
Bulk density (d_b)	g/cm^3	1.6457	1.8694	--
Porosity (P)	(%)	65.76	60.94	--

All observed peaks of XRD correspond to standard spinel diffraction patterns with no extra peak which confirmed single phase cubic structure. The prominent (hkl) planes (220), (311), (400) (422), (333) and (440) were indexed. The broader peaks observed in XRD pattern suggest the nano-crystalline nature of sample. The values of structural parameters obtained from XRD analysis are given in table 2. It's observed from table 2 that the crystallite size **(**D) as well as lattice constant (a) increases with sintering temperature of sample as shown in figure 2**.** X-ray density and porosity were decreases with sintering temperature. These structural parameters obtained for the present sample are good agreement with those reported in the literature [10]. Morphology study of the samples carried out by using SEM reveals the spherical shape of the particles and size of particle increases with increasing sintering temperature as shown in figure 3.

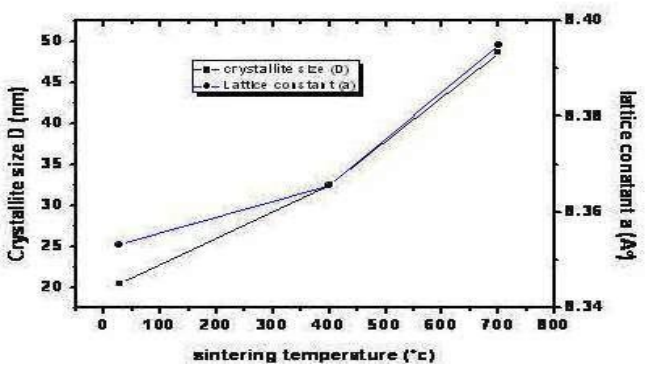


Figure 2: Variation of crystallite size with sintering temperature

In order to confirm the stoichiometry of the prepared nanocrystalline Al substituted Mg-Cu ferrites, energy dispersive spectroscopic (EDS) analysis carried out. Figure 4 shows EDS pattern nanocrystalline Al substituted Mg-Cu ferrite samples. It is clear that Mg^{2+}, Cu^{2+}, Al^{3+}, Fe^{3+} and $O^{2-}$are present in proper proportions. This suggests that the expected stoichiometry is maintained in the prepared samples. The EDS results also confirm that precursors used for the synthesis have fully undergone the chemical reaction to form the single phase aluminium doped Mg-Cu ferrite samples in Nano crystalline nature.
Some of the samples showed impurities of Zn of about less than 1%, which might have crept into the samples due to contents of sample surface and porcelain boat and get loss due to sintering temperature. The important point is that EDS result pointed no loss of ingradients after high temperature sintering and results matched the expected values within the experimental error.

B) Magnetic properties: The room temperature measurement of magnetic properties of L30, L3A and L3B ($Mg_{0.5}Cu_{0.5}Fe_{1.7}Al_{0.3}O_4$) samples was carried out using vibrating sample magnetometer. Figure 5 shows the hysteresis loop of aluminium doped Mg-Cu ferrite nanoparticles at room temperature.

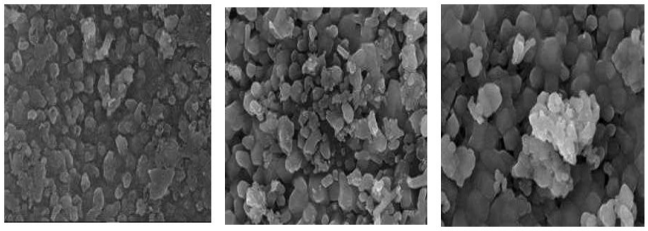
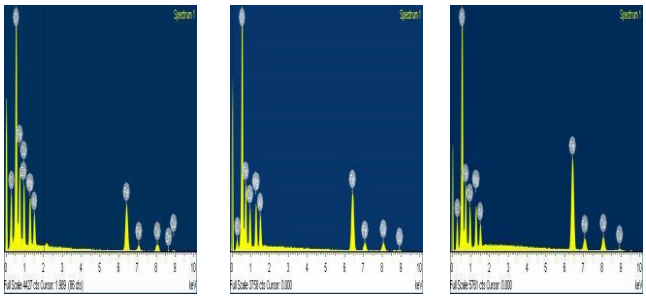


Figure: 3 SEM image of L30, L3A and L3B samples



L30 **L3A** **L3B**

Figure 4: EDS pattern of L30, L3A and L3B samples

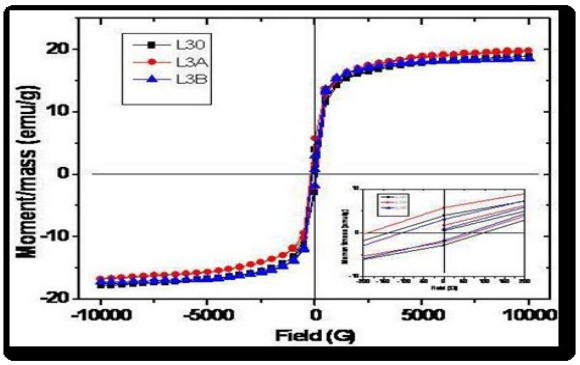


Figure 5: Hysteresis plot of L30, L3A and L3B samples measured at room temperature.

Table 3: Magnetic properties of $Mg_{0.5}Cu_{0.5}Fe_{1.7}Al_{0.3}O_4$

Parameter	Unit	L30	L3A	L3B
Saturation Magnetization (M_s)	emu/g	18.42	19.86	17.91
Coercivity (H_{ci})	G	119.15	130.25	80.35
Retentivity (M_r)	emu/g	3.99	5.76	5.05
Squareness(M_r/M_s)	--	0.1862	0.2147	0.1348

The saturation magnetization (Ms), coercivity (Hci) and remanance magnetization (Mr) measured from hysteresis loop are listed in table 3. The saturation magnetization (M_s) initially increases with sintering temperature due to increase in particle size [11] and then decreases as given in table 3. Similar behavior for coercivity (H_{ci}) and retentivity (M_r) of samples. Compared with values of coercivity (H_{ci}) and retentivity (M_r) of all samples, sample L3B having least these values as given in table 3. This shows that sample is soft ferrite material.

CONCLUSIONS

The nano-crystalline aluminium doped Mg-Cu ferrite was successfully synthesized by sol-gel method. The X-ray diffraction results showed the formation of single phase cubic spinel structure with particle size in nanometer range. The EDS data shows that all elements present in aluminium doped Mg-Cu ferrite sample have maintained their stoichiometry proportions. The hysteresis pattern exhibits soft ferromagnetic nature of the samples.

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