INFLUENCE OF AL SUBSTITUTION ON STRUCTURAL PROPERTIES OF W-TYPE SR-MG FERRITES

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A B S T R A C T

Strontium W-type Hexa-ferrites with composition $SrMg_2Al_xFe_{16-x}O_{27}$ (x = 0.0, 0.2, 0.4, 0.6, 0.8 and 1.0) has been prepared by sol gel auto combustion method at 850 °C for 10 hrs. X - Ray diffraction (XRD) pattern confirm single phase W-type hexa-ferrites and various factor as lattice parameter, cell volume, X-ray density, bulk density, porosity and grain size calculated from XRD data. Microstructure was studied by scanning electron microscope. All the ferrites having grain size between 19 nm to 26 nm are suitable for low signal to noise ratio in the high density recording media.

Keywords: Hexa-ferrite; sol gel; lattice parameter; XRD.

1. Introduction

Nanotechnology and nanomaterial have achieved tremendous progress in past three four decades, nanomaterial's which are the materials with basic structural unit, grains, particles or other constituents component smaller than 100 nm in at least one dimension have evoked a great attention for the different applications [1] due to their very small size and large surface area. Nanomaterial are said to have interesting physical, electrical and magnetic properties that are different from that of their bulk counterpart [2].

Ferrites nano materials are considered to be very important class of magnetic materials from the application point of view. Hexa-ferrites are widely used as permanent magnets and have high corecivity. They are also used at very high frequency. The Hexa-ferrites (Hexagonal) are 6 types M, W, X, Y, Z and U [3-4] amongst the different ferrites na-nomaterials. W -type hexaferrites having formula of $AMe_2Fe_{16}O_{27}$ (A^{2+} = Ba, Sr, La; Me^{2+} = Zn, Ni, Co, Mg, Mn etc) and have potential for application in magnetic and microwave devices and also have many applications from Microwave to radio frequency range. Several studies of substitutions of divalent and rare earth elements in W- type ferrites on the Morphology, Electric and Magnetic prop-erties have been reported. Few studies are available on the trivalent element substations in W type hexagonal ferrites. The property of these nanomaterial depends upon the composition and microstructure of the surface which are influenced by their synthesis route. Researchers attemptedto substitute divalent and trivalent cations such as Cr, Co,La, Cu, Mg, Al [5-10]. The aim of present work is the in-vestigation the role of Al substitution in the Sr-Mg W-type ferrites on its microstructure and to optimse a smaller grain of such combination so that further divalent substitution in such combination possibly give changes in their electric and magnetic behavior. It has been reported that less crystal size less than 50nm is required to obtain the suitable signal to noise ratio in the high density recordingmedia [11]

2. Experimental

Sol gel method is used for the synthesis of Al substituted SrMg–Wtypeferrites.Forthesynthesis of$SrMg_2Al_xFe_{16-x}O_{27}$ where, x = 0.0, 0.2, 0.4, 0.6, 0.8 and1.0, the A.R. grade nitrates were used as starting material.Stoichiometricamountof Strontium Nitrate $Sr(NO_3)_2$ (+99% Fishers Scientific),Magnesium Nitrate, $Mg(NO_3)_2.6H_2O$(+99%Merk)Aluminium Nitrate,$Al(NO_3)_3$. $9H_2O$ (+98.0Fishers Scientific), Ferric Nitrate, $Fe(NO_3)_3$. $9H_2O$

(+98% Fishers Scientific)were dissolved one by one in 100 ml of de-ionized water. Citric Acid (99.9%, SD fine Mumbai) of 1:1 ratio was added as chelating agent and as a fuel .The mixed solution was kept at room temperature for 20-24 hrs. Ammonia solution (30%) was then added slowly in the mixture to adjust pH value between 7-8. The mixed solution were stirred using magnetic stirrer while beingheated at 800C to transform sol into a gel ,after 7-8 hrs. the dried gel underwent auto combustion to produce fluffy powdered sample. The powders were grinded and filtered. The grinded and filtered sample is further sintered at 850^0C for 10 hrs. at heating rate of about 30C/min and kept at room temperature for 24 hours for aging. The powder sample pressed in to pellets of 10mm diameter at a pressure up to ~4 tones. The pellets were then annealed at a temperature of 850^0C for 2 hrs. The X ray diffraction pattern were recorded using Bruker AXS D8 Advance at 40kV/35mA. The radiation used was CμKα (λ=1.5406Å).SEM JEOL JSM 2300 was used for SEM images of prepared samples.

3.Results and Discussion

3.1 Morphological study

3.1.1 XRD

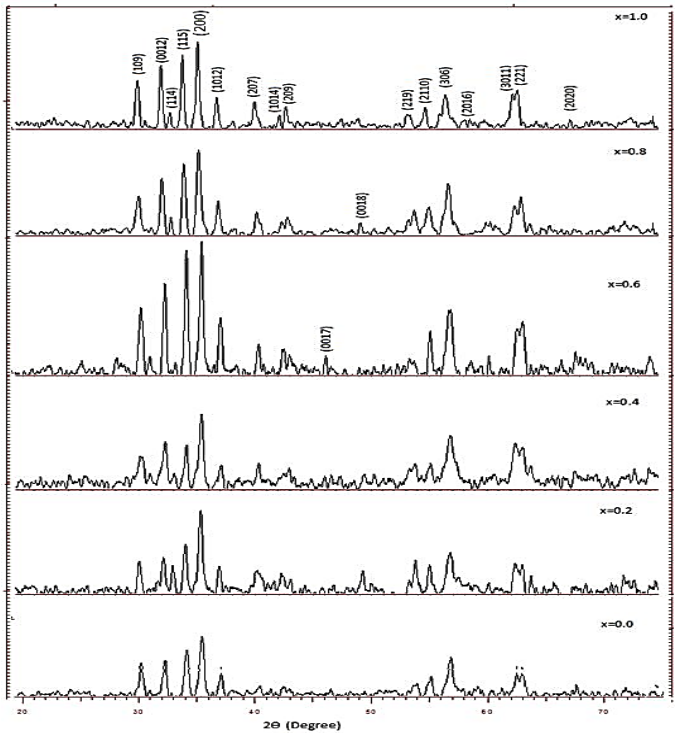

Figure 1- X-ray diffraction patterns for $SrMg_2Al_xFe_{16-x}O_{27}$

Figure 1 shows the XRD patterns for all the investigated samples $SrMg_2Al_xFe_{16-x}O_{27}$ for x = 0.0, 0.2, 0.4, 0.6, 0.8 and 1.0. No extra peak is observed, which indicates that the formation of W type hexa-ferrite, a homogeneous single phase formation of these compounds is confirmed by X-ray diffraction technique through the actual available JCPDS (PDF #781551) of $BaMg_2Fe_{16}O_{27}$ and (PDF #410029) of $SrMn_2Fe_{16}O_{27}$. The lattice parameters were calculated from the Bragg's relation. It is observed that the value of lattice parameters (a and c) for all the samples are closely related with each other as reported earlier studies of W-type hexa-ferrite. As the concentration of Al increases the lattice constant c decreases gradually because of the radius of Al^{3+}(0.53Å) is less as compared to the radius of Fe^{3+}(0.64Å).The structure ofWtype hexa-ferrite is RSSR*S*S* and as the iron ions exist on seven different site known as 4fvi, 2d, 12k, 6g, 4f, 4fiv and 4e [12]. The lower value of c site may be replacement of some Fe^{3+} ions due to substitution of Al^{3+} ions. The value of lattice parameter "a" is remain almost unchanged as it decreases but show very small variation. Where as parameter 'c' decreases. The variation in lattice parameter "c" and ratio "c/a" as a function of Al^{3+} content.

3.1.2 SEM

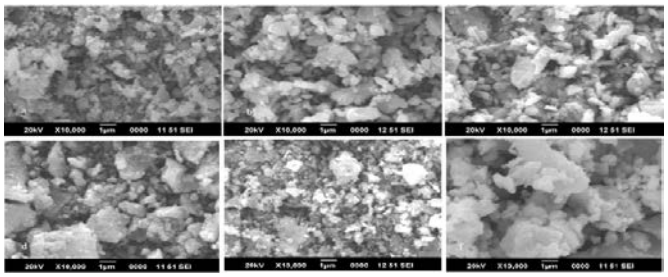

Figure 2- SEM micrograph for $SrMg_2Al_xFe_{16-x}O_{27}$ (x=0.0,0.2,0.4,0.6,0.8,and 1.0)

The two dimensional high magnification surface morpho-logical studies of $SrMg_2Al_xFe_{16-x}O_{27}$ for x = 0.0, 0.2, 0.4, 0.6, 0.8 and 1.0 has been carried out using Scanning Electron Microscope (SEM). The images are shown in Figure 2. From the micrographs, one can see that samples a, b, c, d, e and f all are consisting micro grained particles with the high porous microstructure. The grain size and porosity of prepared samples are presented in Table 1. The sizes of samples is less than 30 nm with high values of porosity,the sintering temperature 850^0C may be possible for high porosity of samples [13] also bulk density is found to be lessthan the X-ray density due to presence of pores created during sintering process. The porosity slightly decreases with substitution, it causes densification of the compound [14].

The less variations may be due to same sintering temperature for all samples. The grain size (D) for each composition was calculated from XRD patterns by using well known Debye-Scherrer formula. The particle size calculated using peak broadening of most intense peak is tabulated in Table 1.the particle size found to be 19 nm to 26 nm with the variation of the substitution. So these materials can be used for application in high density recording media in obtaining suitable signal to noise ratio. Also higher porosity samples contains smaller crystal size which leads to high coercive fields[15].Our results show that the porosity increases with de-creasing grain size measured by Debye-Scherrer formula for each sample.In the present study of $SrMg_2Al_xFe_{16-x}O_{27}$ (x = 0.0, 0.2, 0.4, 0.6, 0.8 and 1.0), equation 1 - 3 were used to calculate X-ray density, Bulk density and Porosity of all the sample respectively and the calculated values are listed in Table 1.

The X-ray density (dx) is determined from the relation-

$$d_x = 2M/NA0.866a^2c \quad (1)$$

where, M is the molecular weight, NA is Avogadro's number, "a" and "c" are lattice constants.

The Bulk Density is calculated by using the formula

$$d_B = m/\pi r^2 h \quad (2)$$

where, m, r and h are the mass, the radius and thickness of the pellet respectively.

The Porosity for the present study is calculated by using the formula

$$P = 1 - d_B/d_x \quad (3)$$

where, d_B is bulk density and dx is X-ray density respectively.

The values for X-ray Density dx, Bulk Density d_B, and porosity P, as a function of Al^{3+} content is tabulated in the Table 1.The variation in porosity and grain size of synthesized samples shown in Figure 4 and Figure 5 respectively which indicates the higher porosity contain smaller particle size of hexa-ferrite.

Table 1 - Parameters as estimated for different concentration of Al^{3+}

X	a (Å)	c (Å)	c/a	Volume ($Å^3$)	Dx	dB	P	Grain Size(nm)
0.000	5.865	33.526	5.716	998.711	4.863	2.350	0.516	19.480
0.200	5.864	33.481	5.710	997.013	4.852	2.482	0.488	23.538
0.400	5.862	33.466	5.709	995.883	4.837	2.383	0.507	19.939
0.600	5.861	33.402	5.699	993.637	4.830	2.493	0.483	24.595
0.800	5.872	33.391	5.686	997.058	4.793	2.408	0.497	20.617
1.000	5.860	33.374	5.695	992.477	4.797	2.590	0.460	25.566

4. Conclusion

The Sol-gel method is an effective method to prepare single phase Sr Mg –W type ferrites with excellent chemical homogeneity and also useful for production of Nano size crystal ferrites. The lattice constants are found to be in range of a=5.86 Å and c=33.5 Å to 33.2 Å. The average size of synthesized W-type hexaferrite is about 22nm which is suitable signal to noise ratio in the high densityrecording media. Hence it can be conclude that Substitution of Al^{3+} content in the composition replaces Fe^{3+} ions of ferrites. Lower nano size grains of ferrite powder contain high porosity. It is known that ferrites need generally high temperature for formation of single phase crystal, however by employing sol gel method it can be synthesized at relatively low temperature and low cost arrangement.

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