International Journal of Knowledge Engineering
ISSN: 0976-5816 & E-ISSN: 0976-5824, Volume 3, Issue 1, 2012, pp.-140-142.
Available online at http://www.bioinfo.in/contents.php?id=40

STUDIES OF STRUCTURAL AND ELECTRICAL PROPERTIES OF ZrCo SUBSTITUTED Ca HEXAFERRITES

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Received: February 28, 2012; Accepted: March 06, 2012

Abstract- A series of ZrCo substituted M-type nanohexafrrites with generic formula $CaFe_{12-2x}(Zr_xCo_x)O_{19}$ were prepared by a novel microwave assisted auto combustion technique. The structural analysis of the prepared samples has been studied by X-ray diffraction patterns technique, which confirmed the presence of M-type hexaferrite phase structure. Average grain size calculated is in the range of nm . The variation in lattice parameters and density with substitution of ZrCo were studied, showed increasing trend. Electrical conductivity is measured by two probe method using LCR-Q Meter. The electrical conduction in ferrite can be explained by Verwey hoping mechanism of electron, room temperature resistivity of samples is of the order of 10^7 ohm- cm. Curie temperature shows decreasing trend with substitution.
Keywords- ZrCo nanoferrites, auto combustion, XRD, electrical conductivity etc

Citation: Asmita Deshpande, et al (2012) Studies of Structural and Electrical Properties of ZrCo Substituted Ca Hexaferrites. International Journal of Knowledge Engineering, ISSN: 0976-5816 & E-ISSN: 0976-5824, Volume 3, Issue 1, pp.-140-142.

Introduction

In modern techno scientific era, the industrial application of nano materials has grabbed a principal importance owing to their improved electric and magnetic characteristics. Hexagonal ferrites especially M-type ferrites have been proved to be the promising candidates for nano materials due to their ease of applicability in high density recording media, microwave absorption devices, magneto-optic recording media,[1] etc. The present module of research aims to develop a systematic method which facilitates the production of ultra fine hexagonal ferrite particles especially in nano scale and to find possible ways of improving their behavioral characteristics. The Microwave Induced sol-gel combustion route used in this research assures higher homogeneity, nonosized particles and more sustained electric and magnetic properties [2,13]. In this synthesis, a simultaneous or coupled divalent and tetravalent substitution of Transition Metal Ions Co^{2+} and Zr^{4+} for Fe^{3+} ions helps to improve the characteristics mentioned earlier [3]. Substituted magnetoplumbites with Ca^{2+} for Ba^{2+}, trivalent metallic ion for Fe^{3+} or a combination of divalent and tetravalent ions for Fe^{3+} have been reported[4]. The calcium hexaferrits[5] attracted the attention as they have been less studied and have magnetic properties comparable to BaM or SrM.

There are many procedures for synthesis. The known sol-gel technique is reported to have many advantages for structural, electrical and magnetic parameters over old techniques like solid state method, co-precipitation, Hydro-thermal synthesis, Glass crystallization, etc. [6]. This technique is improved by replacing conventional heating furnace by scientific microwave source of specific frequency, hence the name 'The Microwave Induced sol-gel combustion route' [7,8].

In the present paper, an attempt has been made to synthesized Zr-Co substituted Ca Hexaferrites from metal nitrates and urea as a precursor by microwave assisted auto combustion method. The prepared samples were characterized by XRD and Lattice parameters of the synthesized samples are calculated [11]. Also the Electrical Behavior of the samples is studied.

Experimental procedure

The large number of preparation methods have been reported earlier for the synthesis of ferrites. The known sol-gel combustion

technique has many advantages to synthesis substituted magnetoplumbites by virtue of simplicity in operation, low anneal or calcine temperature, short reaction rate [12]. In addition sol-gel combustion route gives ultra fine powder of nanoparticles with better particle size distribution, excellent chemical homogenetty and probability of of forming single domain structure[13].

The samples of M-type substituted hexaferrites with formula $CaFe_{12-2x}(Zr_xCo_x)O_{19}$ are synthesized for x=0.5 and x=1. The reactive oxidants such as $Ca(NO_3)2$, $Fe(NO_3)2.9H_2O$, $Co(NO_3)2.6H_2O$, $ZrO(NO_3)2.H_2O$ were dissolved into an unionized distilled water at the temperature of 50°C for 15-20min, urea is used as fuel which gives requisite energy to initiate exothermic reaction. The gel produced is then kept for an hour in the room temperature and then it is heated in the digitally controlled microwave oven of 2.54GHz for 15-20 min, The gel get burnt and finally gets converted in homogeneous powder. The sample is then sintered by giving sporadic moderate heat treatments for further few minutes with further grinding. The samples so produced are then kept in humidity free atmosphere in air tight compartment.

The structural characteristics of the samples are studied by X-ray diffraction, scanning electron spectroscopy.

Material Characterizations

The x-ray diffraction pattern of the samples were obtained at room temperature by Philips Expert Diffractometer with Cu Kα radiation (λ=1.5443 °A), at a scan speed of 2°/min., with an accuracy of 0.01 at 35kV and 20mA. The pallet of the samples for electrical conductivity measurement were made by using KBr dye punch of 1.5 cm diameter and applying pressure of 6 tons in hydrolic press (SES Roorky). Electrical conductivity of the samples were measured by two probe method using LCRQ Meter (Aplab).

Result and discussion

Structural Properties

The diffraction patterns of samples are taken with Philips X'pert Diffractometer and Cu Ka radiation with wavelength l=1.5443 A.U. Fig 1 shows the intensity graphs for samples x= 0.5 and x=1 respectively. Lattice parameters *a* and *c* of the samples are shown in table 1. The experimental density D (bulk density) values were found to be in general less than those of D_x (theoretical density) which are expected due to the presence of unavoidable pores created during firing. It is observed that the porous nature decreases with increasing ZrCo content; in agreement with observations made by previous workers[13] [14]. The crystallite size was determined using the well known Scherrer formula [15] from the strongest peak.

Table 1- Structural data

x	Lattice Parameters a (Å)	c (Å)	Cell Volume $(Å)^3$	Bulk Density (D) gm / cm^3	X-Ray Density (Dx) gm/ cm^3	Porosity	Grain Size (nm) XRD
0	5.807	21.582	630.2752	1.8042	2.6786	0.3265	14.5031
0.5	5.825	22.124	650.1017	2.0096	2.5239	0.2038	14.501
1	5.823	22.128	649.761	1.9875	2.4522	0.1896	20.714

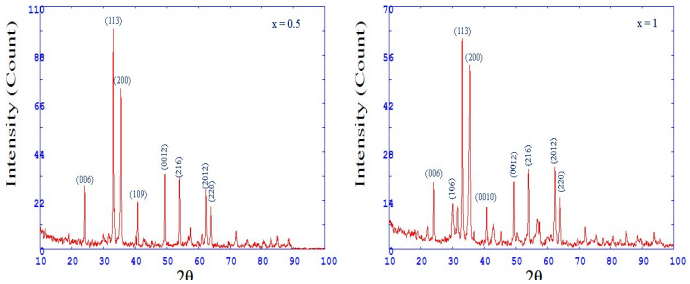

Fig. 1- XRD patterns

Electrical Properties

The variation of D.C. electrical conductivity with inverse absolute temperature of samples is shown in Fig 2. The variation of log σ with reciprocal temperature indicates that the synthesized hexaferrite samples are semiconducting in nature. The slope of the conductivity graph breaks near the Curie temperature T_C. This change at transition temperature (Tc) may be assigned to the magnetic order phase Transition from ferri to paramagnetic state. Activation energy calculated in paramagnetic region is more than in ferrimagnetic region, shows increasing trend with substitution . The electrical conduction in ferrite can be explained by Verwey hoping mechanism of electron[9]. Accordingly, electronic conduction in ferrites occurs due to hopping of electron between ions of same element present in more than one valence state, distributed randomly over crystallographically equivalent lattice sites. The room temperature resistivity and curie temperature of samples are shown in table 2, indicates that with substitution of ZrCo the resistivity is increased. This is due to the fact that, when $Zr^{4+}Co^{2+}$ ions substituted for Fe^{3+} ions, Zr^{4+} and Co^{2+} occupies the octahedral B sites, thereby decreasing the Fe3+ ions at B Sites. The number of Fe^{2+} ions formed also decreases as this depends on the content of Fe^{3+} ions. This reduces the hopping electrons between Fe^{3+} and Fe^{2+} ions, resulting in there is increase in resistance [13].

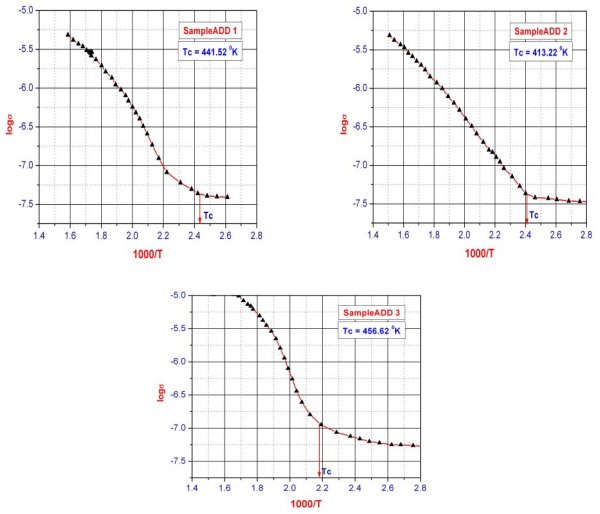

Fig. 2- Variation of electrical conductivity with inverse of temperature

Table 2- Electrical resistivity and Curie Temp.

x	Activation Energy Eg (eV)		Resistivity at room Temp W-cm.	Conductivity at room temp. in S/cm	Curie Temp. T_c (K)
	Ferri magnetic	Para magnetic			
0	0.3256	0.6861	2.5241×10^{7}	2.7032×10^{-8}	441.52
0.5	0.3612	0.7534	3.1243×10^{7}	3.1826×10^{-8}	449.73
1	0.5239	0.7817	3.7103×10^{7}	3.9618×10^{-8}	456.62

Result and Discussion

The substitution of Co^{2+} and Zr^{4+} in calcium hexaferrites by replacing Fe^{3+} is found to be successful by *'The Microwave Induced sol-gel combustion route'*..

The X-ray diffraction studies confirm the formation of hexaferrites and the a & c values of the sample supports this confirmation.

The route used in this research assures higher homogeneity, non-osized particles and more sustained electric and magnetic properties.

Activation energy calculated in paramagnetic region is more than in ferrimagnetic region. The electrical conduction in ferrite can be explained by Verwey hoping mechanism of electron. The room temperature resistivity increases with substitution of ZrCo, due to decreasing Fe^{+3} ions at octahedral B sites.

Further magnetic and electrical studies will help to know the detailed magnetic character of the sample.

It is known that the nanohexaferrites with such special magnetic properties are highly useful in various aerospace and military applications.[16,17]

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