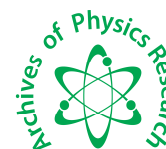




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Critical studies on effect of temperature on structural morphology of Ni-Zn substituted on Barium and Cobalt Ferrites

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ABSTRACT

Two new series of W type hexaferrites with the composition $Ba^{2+} Sr^{2+}_2 (Ni^{2+}_{x/2} Zn^{2+}_x) Fe^{3+}_{16-x} O^{-2}_{27}$ ($x=0.1$ to 0.4) and $Co^{2+} Sr^{2+}_2 (Ni^{2+}_{x/2} Zn^{2+}_x) Fe^{3+}_{16-x} O^{-2}_{27}$ ($x=0.2$ to 0.8) were prepared using co precipitation technique. The precipitate was calcinated at $1100^\circ C$ for 4 hours and characterization was carried out using X-Ray technique. The values of "a" for the samples investigated lies in the range 5.8347 \AA to 5.8755 \AA and $c = 33.1327 \text{ \AA}$ to 33.3587 \AA . They match well with the values reported by Sawadh and Kulkarni [1]. The effect of calcinations temperature and parameters involved during production on the structure and average particle size of the grown samples has been studied. It is conjectured that these do not have any predominant effect on the average particle size and its structure. However, the composite formed shows an increase in the grain/ particle size with the increase in the doping concentration. From the X-RD studies it is established that a W-type hexagonal phase is formed when the mixtures of $[Ba^{2+} Sr^{2+}_2 (Ni^{2+}_{x/2} Zn^{2+}_x) Fe^{3+}_{16-x} O^{-2}_{27}]$ ($x=0.1$ to 0.4) and $[Co^{2+} Sr^{2+}_2 (Ni^{2+}_{x/2} Zn^{2+}_x) Fe^{3+}_{16-x} O^{-2}_{27}]$ ($x=0.2$ to 0.8) are sintered at $1100^\circ C$ and diphas composite ferrite that consists of Co and Ba spinal ferrite and a composite W type hexaferrite is finally obtained.

Keywords: Calcination of hexaferrites, calcinations temperature, structure of W type hexaferrites, W type hexaferrites.

INTRODUCTION

Ferrites have continued to attract attention over last many years. In the fifties a large class of ferrimagnetic oxides with hexagonal crystal structure has been synthesized at the Philips laboratory [2]. They were called hexagonal ferrites in order to distinguish them from the ferrimagnetic oxides with spinel and garnet structure. The basic crystallographic and magnetic properties of the main hexagonal ferrites have been reviewed by J. Smit and H. P. J. Wijn in their classical book Ferrites [3].

As magnetic materials, ferrites cannot be replaced by any other magnetic material because they are relatively inexpensive, stable and have a wide range of technological applications in transformer core, high quality filters, high and very high frequency circuits and operating devices [4]. The physical properties of ferrites are controlled by the preparation conditions, chemical composition, sintering temperature and time, type and amount of substitutions [5].

The increasing exploitation of microwave (MW) frequencies for telecommunications has increased electromagnetic (EM) interference and pollution and stimulated the development of MW absorbers.

Hexaferrites have always been the subject of intensive studies as they possess an appealing combination of good magnetic properties and low cost. They belong to a large family of oxides with hexagonal crystal structure and contains ferrimagnetic compounds with easy axis of magnetization (M-type ferrites) and easy plane of magnetization (Y-type ferrites). As a consequence of that hexaferrites have been widely adopted in two distinct fields: permanent magnets and microwave technology components [6]. On the other hand, W-type ferrites $BaM_2Fe_{16}O_{27}$ ($M = Mg, Mn, Fe, Co, Ni, Cu, Zn$) can undergo spin reorientation transitions (SRT) between different anisotropy configurations (easy plane \leftrightarrow easy cone \leftrightarrow easy axis) induced by change of temperature or applied magnetic field [7-14]. The transition temperatures can be tuned by modifying the chemical composition (substitution of bivalent metal M). Moreover, some SRT are expected to be of the first order, which suggests a potential application of W-type ferrites in room temperature magnetic refrigeration [15].

Ferrite magnets are sintered permanent magnets composed of Barium or Strontium Ferrite. This class of magnets, aside from good resistance to demagnetization, has the popular advantage of

low cost. Ferrite magnets are very hard and brittle, and require specialized machining techniques. Moreover, they should be machined in an unmagnetized state. We are equipped to machine these materials to specifications. Anisotropic grades are oriented in the manufacturing direction, and must be magnetized in the direction of orientation. Isotropic grades are not oriented and can be magnetized in any direction, although some degree of greater magnetic strength will be found in the pressing dimension, usually the shortest dimension. Due to their low cost, Ferrite magnets enjoy a very wide range of applications, from motors and loudspeakers to toys and crafts, and are the most widely used permanent magnets today.

MATERIALS AND METHODS

$Ba^{2+} Sr^{2+}_2 (Ni^{2+}_{x/2} Zn^{2+}_x) Fe^{3+}_{16-x} O^{-2}_{27}$ with $x = 0.1, 0.2, 0.3, 0.4$ and $Co^{2+} Sr^{2+}_2 (Ni^{2+}_{x/2} Zn^{2+}_x) Fe^{3+}_{16-x} O^{-2}_{27}$ with $x = 0.2, 0.4, 0.6, 0.8$ were prepared. Synthesis and characterization was carried out using chemical coprecipitation techniques. A.R. Grade nitrates were used as starting materials. Stoichiometric mixture of Barium nitrate [$Ba(NO_3)_2 \cdot 6H_2O$], Strontium nitrate [$Sr(NO_3)_2$], Nickel nitrate [$Ni(NO_3)_2 \cdot 6H_2O$], Zinc nitrate [$Zn(NO_3)_2 \cdot 6H_2O$], Ferric nitrate [$Fe(NO_3)_3 \cdot 9H_2O$] and Cobalt nitrate [$Co(NO_3)_2 \cdot 6H_2O$], Strontium nitrate [$Sr(NO_3)_2$], Nickel nitrate [$Ni(NO_3)_2 \cdot 6H_2O$], Zinc nitrate [$Zn(NO_3)_2 \cdot 6H_2O$], Ferric nitrate [$Fe(NO_3)_3 \cdot 9H_2O$] (>99.9%) were used as a starting materials. They were dissolved one by one in 100 ml of de-ionized water. Ammonia solution (30%) was added slowly in the mixture so as to adjust Ph of 8. The mixed solution was stirred for two hours and was kept at room temperature for 24 hours so as to achieve aging. Barium strontium and Cobalt strontium hexaferrites precipitates was separated in a centrifuge machine at 2500 rpm for 20 minutes. The prepared precipitates were then washed in 1:1 mixture of methanol and acetone followed by 100% de-ionized water to remove impurities. The precipitate was dried at $100^\circ C$ for 24 hours and calcinated at $1100^\circ C$ for 4 hours to obtained $Ba^{2+} Sr^{2+}_2 (Ni^{2+}_{x/2} Zn^{2+}_x) Fe^{3+}_{16-x} O^{-2}_{27}$ and

$Co^{2+} Sr^{2+}_2 (Ni^{2+}_{x/2} Zn^{2+}_x) Fe^{3+}_{16-x} O^{-2}_{27}$ hexaferrites particles.

Lattice constant a and c of hexagonal Barium and Cobalt Ferrites were calculated using equation (1),

$$\frac{1}{d^2} = \frac{4}{3a^2} (h^2 + k^2 + l^2) + \frac{l^2}{c^2} \dots \dots \dots (1)$$

where h, k and l are Miller indices, d is interplaner distance. Lattice volume of all the samples can be obtained using equation (2),

$$V = \frac{\sqrt{3}}{2} a^2 c \dots \dots \dots (2)$$

X-ray diffraction pattern for the compound [$Ba^{2+} Sr^{2+}_2 (Ni^{2+}_{x/2} Zn^{2+}_x) Fe^{3+}_{16-x} O^{-2}_{27}$] with $x = 0.1, 0.2, 0.3$ and 0.4 and [$Co^{2+} Sr^{2+}_2 (Ni^{2+}_{x/2} Zn^{2+}_x) Fe^{3+}_{16-x} O^{-2}_{27}$] with $x = 0.2, 0.4, 0.6$ and 0.8 prepared using chemical Coprecipitation and were recorded on X-PERT PRO PA analytical X-ray diffractometer (PW 3050/60) using $CuK\alpha$

radiation source ($\lambda = 1.54443 \text{ \AA}$, 40 Kv, 30 mA). Figure 1 and 2 shows XRD pattern of $[\text{Ba}^{2+} \text{Sr}^{2+}_2 (\text{Ni}^{2+}_{x/2} \text{Zn}^{2+}_x) \text{Fe}^{3+}_{16-x} \text{O}^{-2}_{27}]$ and $[\text{Co}^{2+} \text{Sr}^{2+}_2 (\text{Ni}^{2+}_{x/2} \text{Zn}^{2+}_x) \text{Fe}^{3+}_{16-x} \text{O}^{-2}_{27}]$ hexaferrites calcinated at 1100°C for 4 hours. It is then compared with the characteristic diffraction peaks of the standard XRD (Sawadh P.S. and Kulkarni) for W – type phase ferrites, the crystal structure of $[\text{Ba}^{2+} \text{Sr}^{2+}_2 (\text{Ni}^{2+}_{x/2} \text{Zn}^{2+}_x) \text{Fe}^{3+}_{16-x} \text{O}^{-2}_{27}]$ and $[\text{Co}^{2+} \text{Sr}^{2+}_2 (\text{Ni}^{2+}_{x/2} \text{Zn}^{2+}_x) \text{Fe}^{3+}_{16-x} \text{O}^{-2}_{27}]$ samples were found to be a single W-type phase structure. Nearly no variation of the crystal structure were observed for all Mn Zn doped $[\text{Ba}^{2+} \text{Sr}^{2+}_2 (\text{Ni}^{2+}_{x/2} \text{Zn}^{2+}_x) \text{Fe}^{3+}_{16-x} \text{O}^{-2}_{27}]$ sample when x equals to 0.1, 0.2, 0.3 & 0.4 and $[\text{Co}^{2+} \text{Sr}^{2+}_2 (\text{Ni}^{2+}_{x/2} \text{Zn}^{2+}_x) \text{Fe}^{3+}_{16-x} \text{O}^{-2}_{27}]$ sample when x equals to 0.2, 0.4, 0.6 & 0.8 the XRD pattern shows three phases (W,M and α -Fe₂O₃ phase). It was reported that the unit cell of W-type phase is closely related to M-phase, the only difference is that the successive R blocks are inter placed by two S-blocks instead of one as in case of M-phase. It was also mentioned by Lotgering [16] that W-type hexagonal ferrites is chemically constant.

Table I :-Lattice constant a & c and volume of unit cell (V) For $[\text{Ba}^{2+} \text{Sr}^{2+}_2 (\text{Ni}^{2+}_{x/2} \text{Zn}^{2+}_x) \text{Fe}^{3+}_{16-x} \text{O}^{-2}_{27}]$ with x=0.1, 0.2, 0.3, 0.4

Sr.No.	Concentration (x)	Lattice parameter a (Å)	Lattice parameter c (Å)	c/a	Volume (nm ³)
1	0.1	5.8755	33.132	5.6390	0.9905
2	0.2	5.8231	33.172	5.6966	0.9740
3	0.3	5.8347	33.269	5.7019	0.9808
4	0.4	5.8337	33.297	5.7076	0.9813

Table II :-X-ray diffraction data For $[\text{Ba}^{2+} \text{Sr}^{2+}_2 (\text{Ni}^{2+}_{x/2} \text{Zn}^{2+}_x) \text{Fe}^{3+}_{16-x} \text{O}^{-2}_{27}]$ with x= (0.1 to 0.4)

d (obs.)	d (cal.)	I (obs.)	h k l	d (obs.)	d (cal.)	I (obs.)	h k l
3.68582	3.68582	27.75	0 0 9	1.6982	1.694104	46.83	1 1 16
2.95324	2.983444	45.57	1 0 9	1.66699	1.666792	24.61	2 0 15
2.77688	2.776538	91.13	1 0 10	1.62344	1.620972	37.44	2 1 11
2.52649	2.526489	94.05	2 0 0	1.48638	1.487867	23.12	2 0 18
2.42413	2.426809	47.85	1 0 12	1.45398	1.45776	23.48	2 2 0
2.21166	2.217936	22.14	0 0 15	1.45673	1.453093	20.84	2 2 2
1.84554	1.843284	32.72	1 1 14				

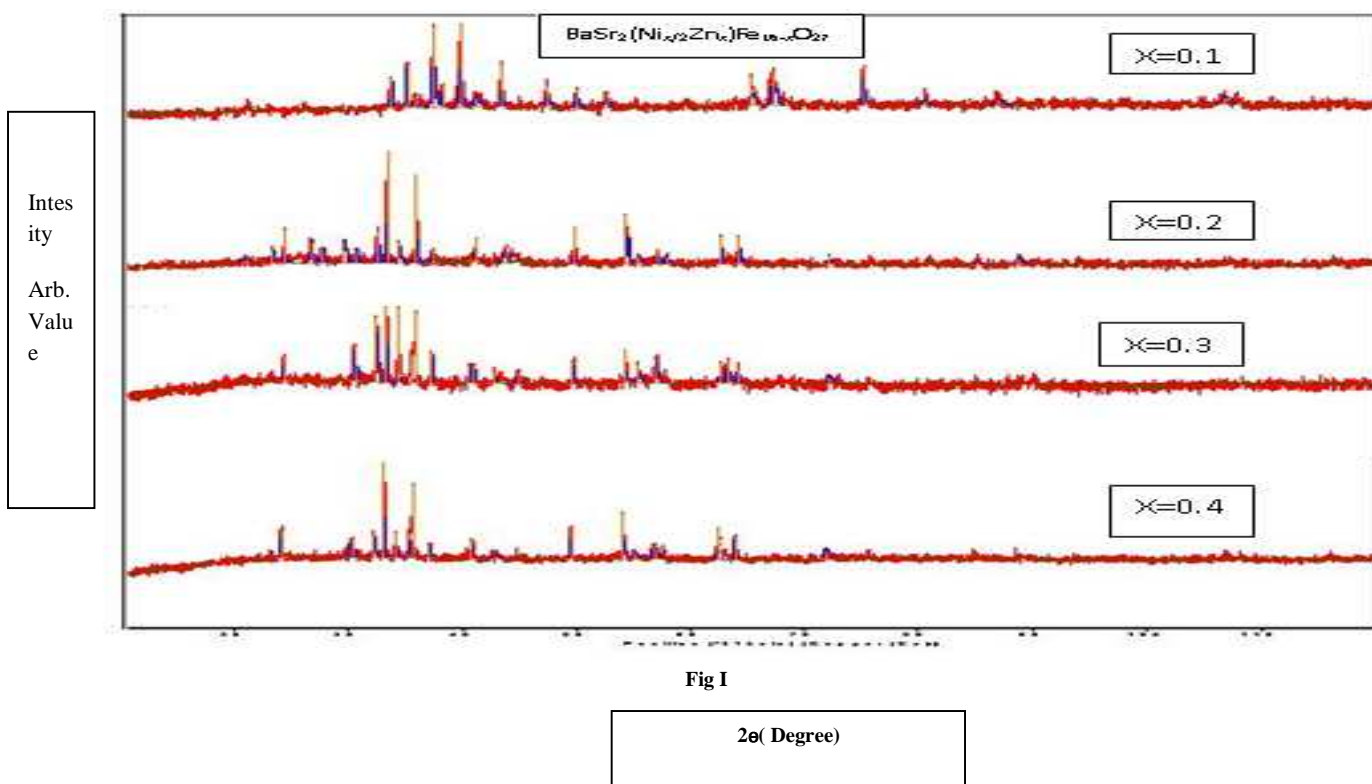


Fig I

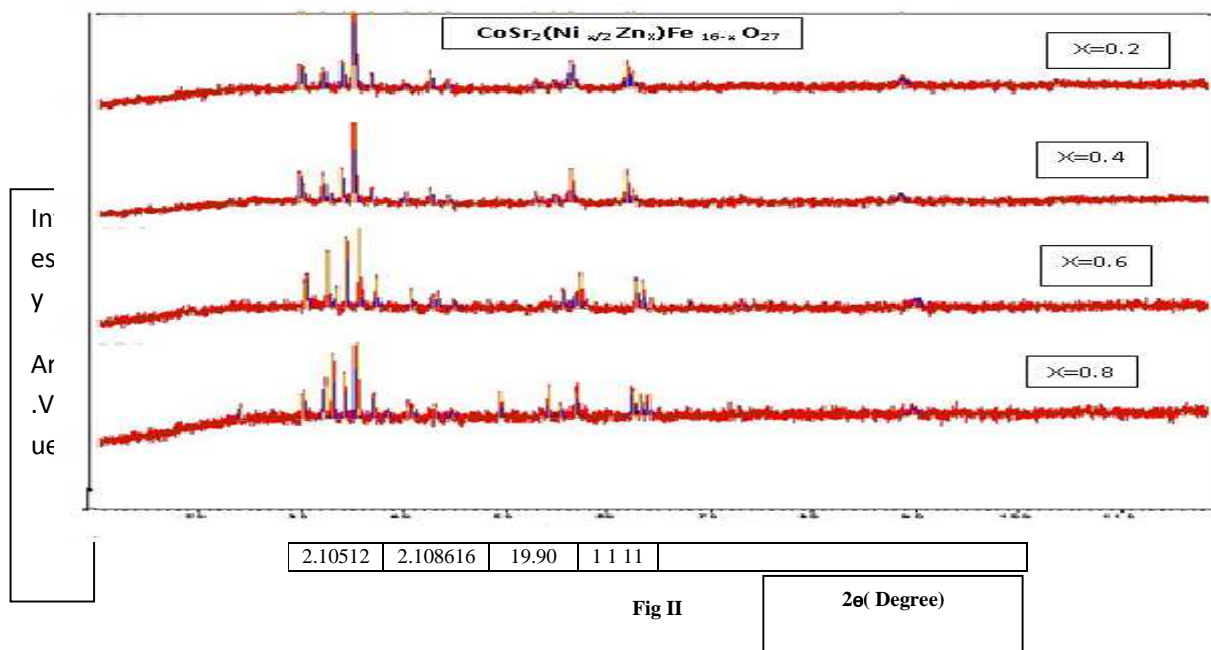
2θ(Degree)

Table III:-Lattice constant a & c and volume of unit cell (V)
 $\text{Co}^{2+} \text{Sr}^{2+}_2 (\text{Ni}^{2+}_{x/2} \text{Zn}^{2+}_x) \text{Fe}^{3+}_{16-x} \text{O}^{-2}_{27}$ with $x=0.2, 0.4, 0.6, 0.8$

Sr.No.	Concentration (x)	Lattice parameter a (Å)	Lattice parameter c (Å)	c/a	Volume V (nm ³)
1	0.2	5.8615	33.274	5.6767	0.9900
2	0.4	5.8489	33.216	5.6790	0.9840
3	0.6	5.8678	33.358	5.6849	0.9946
4	0.8	5.8713	33.358	5.6815	0.9958

Table IV:-X-ray diffraction data For [$\text{Co}^{2+} \text{Sr}^{2+}_2 (\text{Ni}^{2+}_{x/2} \text{Zn}^{2+}_x) \text{Fe}^{3+}_{16-x} \text{O}^{-2}_{27}$] with $x=(0.2 \text{ to } 0.8)$

d (obs.)	d (cal.)	I (obs.)	h k l	d (obs.)	d (cal.)	I (obs.)	h k l
3.70652	3.70652	27.12	0 0 9	2.02977	2.022055	8.23	2 0 10
2.97854	2.975735	36.35	1 0 9	1.69818	1.688848	41.27	1 1 16
2.94954	2.935663	22.05	1 1 0	1.66489	1.662618	14.90	2 0 15
2.77589	2.771403	44.08	1 0 10	1.62032	1.622628	34.75	2 1 11
2.7648	2.758331	65.83	1 1 4	1.48767	1.487867	34.20	2 0 18
2.61934	2.60414	35.18	1 0 11	1.47294	1.462181	16.15	2 2 2
2.54236	2.542359	100	2 0 0	1.45661	1.455776	30.02	2 2 0
2.42325	2.424058	31.54	1 0 12	1.09288	1.086255	6.52	0 1 30
2.23064	2.234316	21.71	2 0 7	1.84593	1.837787	32.81	1 1 14



RESULTS AND DISCUSSION

Two new series of W type hexaferrites with the composition $\text{Ba}^{2+} \text{Sr}^{2+}_2 (\text{Ni}^{2+}_{x/2} \text{Zn}^{2+}_x) \text{Fe}^{3+}_{16-x} \text{O}^{-2}_{27}$ ($x=0.1$ to 0.4) and $\text{Co}^{2+} \text{Sr}^{2+}_2 (\text{Ni}^{2+}_{x/2} \text{Zn}^{2+}_x) \text{Fe}^{3+}_{16-x} \text{O}^{-2}_{27}$ ($x=0.2$ to 0.8) were prepared using co-precipitation technique. The precipitate was calcinated at 1100°C for 4 hours and characterization was carried out using x ray technique. The values of "a" for the samples investigated lies in the range 5.8231 \AA to 5.8755 \AA and $c = 33.1327 \text{ \AA}$ to 33.3587 \AA . They match well with the values reported by Sawadh and Kulkarni [1].

CONCLUSION

The effect of calcinations temperature and parameters involved during production on the structure and average particle size of the grown samples has been studied. Addition of Ba and Co into the structure does not result in any change in the structure and the morphology of the sample remains hexagonal flaky with an average particle size of

about 2 nm. However, the composite formed shows an increase in the grain/ particle size with the increase in the doping concentration. From the X-RD studies it is established that a W-type hexagonal phase is formed when the mixtures of $\text{Ba}^{2+} \text{Sr}^{2+}_2 (\text{Ni}^{2+}_{x/2}, \text{Zn}^{2+}_x) \text{Fe}^{3+}_{16-x} \text{O}^{-2}_{27}$ ($x=0.1$ to 0.4) and $\text{Co}^{2+} \text{Sr}^{2+}_2 (\text{Ni}^{2+}_{x/2}, \text{Zn}^{2+}_x) \text{Fe}^{3+}_{16-x} \text{O}^{-2}_{27}$ ($x=0.2$ to 0.8) are sintered at 1100°C and diphasic composite ferrite that consists of Co and Ba spinal ferrite and a composite W type hexaferrite is finally obtained. It is conjectured that suitable amount of doping of Ba and Co will strongly enhance electromagnetic loss of the ferrite. The unit cell volume decreased from 0.9905 nm^3 to 0.974 nm^3 & from 0.9958 nm^3 to 0.984 nm^3 with the increase in the doping concentration of Ba and Co. This is attributed to the decrease in the lattice parameters "a" and "c". The crystalline size decreased with the increase in the doping concentration. This might be due to the fact that Ba and Co ions substitution inhibits the grain growth. Also the decreasing trend in crystalline size indicates that the dopants are entering into the hexaferrite lattice, which is again confirmed due to the variation in unit cell volume and lattice parameters. It is a well known fact that to obtain the suitable signal to noise ratio the crystalline size should be less than 50 nm. For the samples grown by us the crystalline size has been found to be about 1-2 nm. Thus, the crystals grown by us may be useful in the high density recording media to obtain suitable signal to noise ratio [17].

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