

Structural Analysis Of Ba Y-Type Hexaferrite Samples Prepared By Chemical Co-Precipitation Technic

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Abstract- Y-type hexaferrites BaM₂Fe₁₂O₂₂ (where M₂ = Cr₂, In₂, Ti₂ selected as trivalent impurity) powders are synthesized by chemical co-precipitation method and subsequent heat treatment at 1000°C and 1100°C for 4 hrs & 6 hrs. The X-ray diffraction measurements results showed that the Y-type hexagonal structure. The X-ray diffraction patterns of the samples annealed at 1000°C & 1100°C for 4 hrs & 6 hrs which manifest that all the species have hexagonal crystal structure with lattice constants of $a = 4.31 - 4.56 \text{ \AA}$ and $c = 43.98 - 44.59 \text{ \AA}$ which is followed from standard values of $a \sim 5 \text{ \AA}$ and $c \sim 45 \text{ \AA}$. The SEM observation of powder BaM₂Fe₁₂O₂₂ sample shows that the microstructures of the compounds are hexagonal. The structural analysis and lattice parameters have been observed from the XRD patterns of the studied all six samples. There are six types of hexagonal compounds M, Y, W, Z, X and U. The preparation of the compounds has been done for the Y-type hexagonal ferrites. The SEM photograph shows clear hexagonal structure of the studied compounds. The lattice parameters have been calculated from the Miller indices which are common for some common planes by using crystallographic formula. The particle size, X-ray density, bulk density and porosity have been calculated from the studied structural data.

Keywords- XRD, Structural Properties, SEM, Lattice Constant, Particle Size, Porosity

I. INTRODUCTION

The chemical compounds consisting of ceramic materials with iron oxide (Fe₂O₃) as their principal component are Ferrites [1]. Ferrites have continued to attract attention over the years. As magnetic materials, ferrites cannot be replaced by any other magnetic materials because they are relatively inexpensive, stable and have a wide range of technological applications in transformer core, high quality filters, very high frequency circuits and operating devices [2]. Ferrites are also used as memory core devices in computers, cassettes and video tapes. The main application is in the d. c. motors in the automotive industry. Hexagonal ferrites are broad subset which is of great practical importance as well as scientific interest of iron oxides. These systems are ferrimagnets as dominant interaction between magnetic ions which is oxygen mediated antiferromagnetic superexchange. They offer a variety of magnetic structure and properties, these qualities are determined by structure and particular composition [3]. Hexagonal ferrites follow the category of hard magnetic materials. Hard ferrites play vital role in electronic industry, electronic information industry, car industry, motor cycle industry etc. They are also widely used in medical treatment, mining and metallurgy, industrial automation, oil industry and civil industry [4].

II. PROPOSED WORK

The family of hexagonal ferrites can be classified on the basis of their chemical composition and crystal structure. They are subdivided into six fundamental structure types: M, Y, W, Z, X and U [5, 6]. The structure of these different types of hexagonal ferrites are very complex and can be considered as a superposition of T, R and S blocks among the hexagonal c-axis, such as RST SR* S* T* S* for Z type, RS SR* S*S* for W type, RS R*S* for M-type and (TS)'' (TS) (TS)' (TS)'' for Y type, where T is a block of layers of oxygen ions(O₄-BaO₃-BaO₃-O₄) with composition Ba₂Fe₈O₁₄ is a three oxygen-layer block (O₄-BaO₃-O₄) with composition Fe₆O₈ and the asterisk means that the corresponding block has been turned 120 around the hexagonal axis [7]. Among these hexaferrite family, in 1956 Jonker has first discovered Y-type hexaferrite [8].

Barium hexaferrite is a well-known permanent magnet which has attracted extensive attentions due to their great technical importance [9]. Hexagonal ferrites which are divided into six different types M (AM₂Fe₁₂O₁₉), Y (AM₂Fe₁₂ O₂₂), W (AM₂Fe₁₆ O₂₇), Z(A₃ AM₂Fe₂₄ O₄₁), X (AM₂Fe₂₈ O₄₆), U (A₄M₂Fe₃₆ O₆₀), where A = Ba, Sr, La and M = divalent transition metal ions [10,11] are prepared by various synthesis routes such as co-precipitation [12,13], low temperature combustion [14], sol-gel [15], mechanical alloying [16,17], mechanical activation [18], solid-state diffusion reaction [19] and microemulsion & reverse microemulsion [20].

III. EXPERIMENT AND RESULT

All the samples have been prepared by the chemical co-precipitation technique. A series of polycrystalline mixed ferrites having chemical composition as BaM₂Fe₁₂O₂₂ (M = Cr, In, Ti) was prepared by chemical co-precipitation technique [21]. The analytical reagent (A. R.) grade nitrates were used as starting material. Stoichiometric amount of barium nitrate, strontium nitrate, indium nitrate, chromium nitrate (99.99%) etc. were dissolved one by one in 100 ml of de-ionized water. Ammonia solution (30%) was then added slowly in the mixture to adjust the pH of 8. The mixed solution was stirred for 2 hours and kept at room temperature for 24 hours for aging. The barium and strontium hexaferrite precipitates were separated in a centrifuge machine at 2500 rpm for 20 minutes. The prepared precipitate was then washed in 1:1 mixture of methanol and acetone followed by 100% de-ionized water to remove impurities. The precipitate was then kept for drying at 100°C for 24 hours. The efficiency of the method was refined by lowering the reaction temperature and shortening the required reaction time. Finally, it was calcinated at 1000°C and 1100°C for 4 hours and 6 hours to obtain different compositions of barium and strontium hexaferrites.

The series of polycrystalline mixed ferrites having chemical compositions BaCr₂Fe₁₂O₂₂, BaIn₂Fe₁₂O₂₂, BaTi₂Fe₁₂O₂₂, were prepared by chemical co-precipitation method at 1000°C and 1100°C for 4 and 6 hrs [21]. The efficiency of the method was refined by lowering the reaction temperature and shortening the required reaction time, due to which crystallinity improved and the value of saturated magnetization increased as well. The prepared samples were used for carrying out various studies and used accordingly.

3.1. Xrd Analysis-

The XRD patterns of Ba doped Cr, In, Ti (Fig. 1) of all six compounds (1- 6) taken after final sintering at 1000°C & 1100°C for 4 hr & 6 hrs showed the formation of hexaferrite along with some individual common planes, viz. (106), (009), (109), (0012), (200), (2 1 9), (1 1 17), (3 0 11) etc., which carries almost equal intensity. The existence of these common planes in all the compounds is an indication of hexagonal Y-ferrite, which belongs to the space group P6₃/mmc. The lattice parameter 'a' and 'c' for different compositions have been calculated using the values of d-spacing and from Bragg's law by using formula

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

And compared to the values reported in JCPDC (Joint Committee on Powder Diffraction Standards) cards [22]. The structural analysis have been carried out the lattice constants 'a' and 'c' found to be a = 4.31 – 4.56Å and c = 44.07-44.59 Å. This is in agreement with the fact that all hexagonal Y-type ferrite exhibit constant lattice parameters [23].

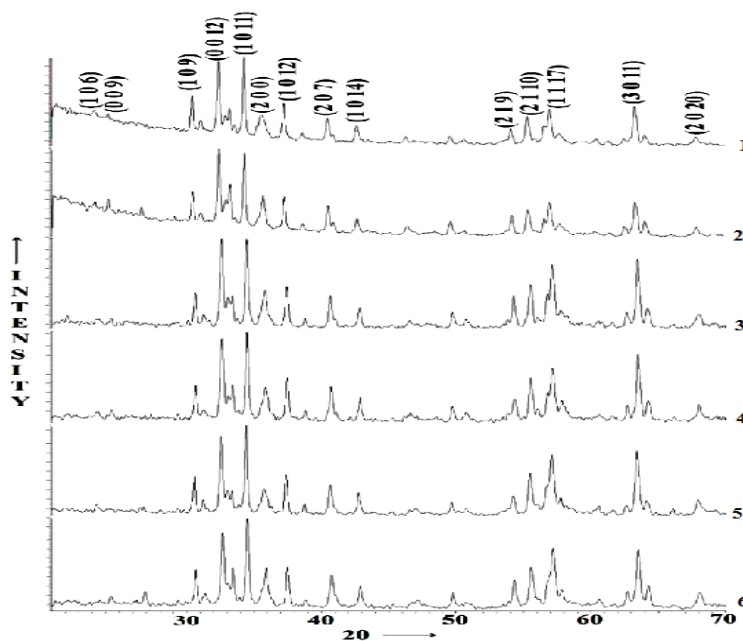


Figure 1. XRD of Ba doped Cr, In, Ti compounds.

3.2. Sem Analysis-

Scanning Electron Microscopy of the above six compounds (Fig. 2: 1-6) have been carried out which shows remarkable changes in the microstructure regarding grain size etc. One can also notice the presence of the large crystallite agglomerations composed of very small discrete crystallites in the samples treated at 1000°C & 1100°C for 4 hr & 6 hrs. This is because of doping impurity ions in Ba and calcinated at 1000°C & 1100°C for 4 hr & 6 hrs. The SEM analysis reveals that the morphological behaviour of the studied compounds shows the hexagonal structure of the samples. This analysis carried out for the different voltages.

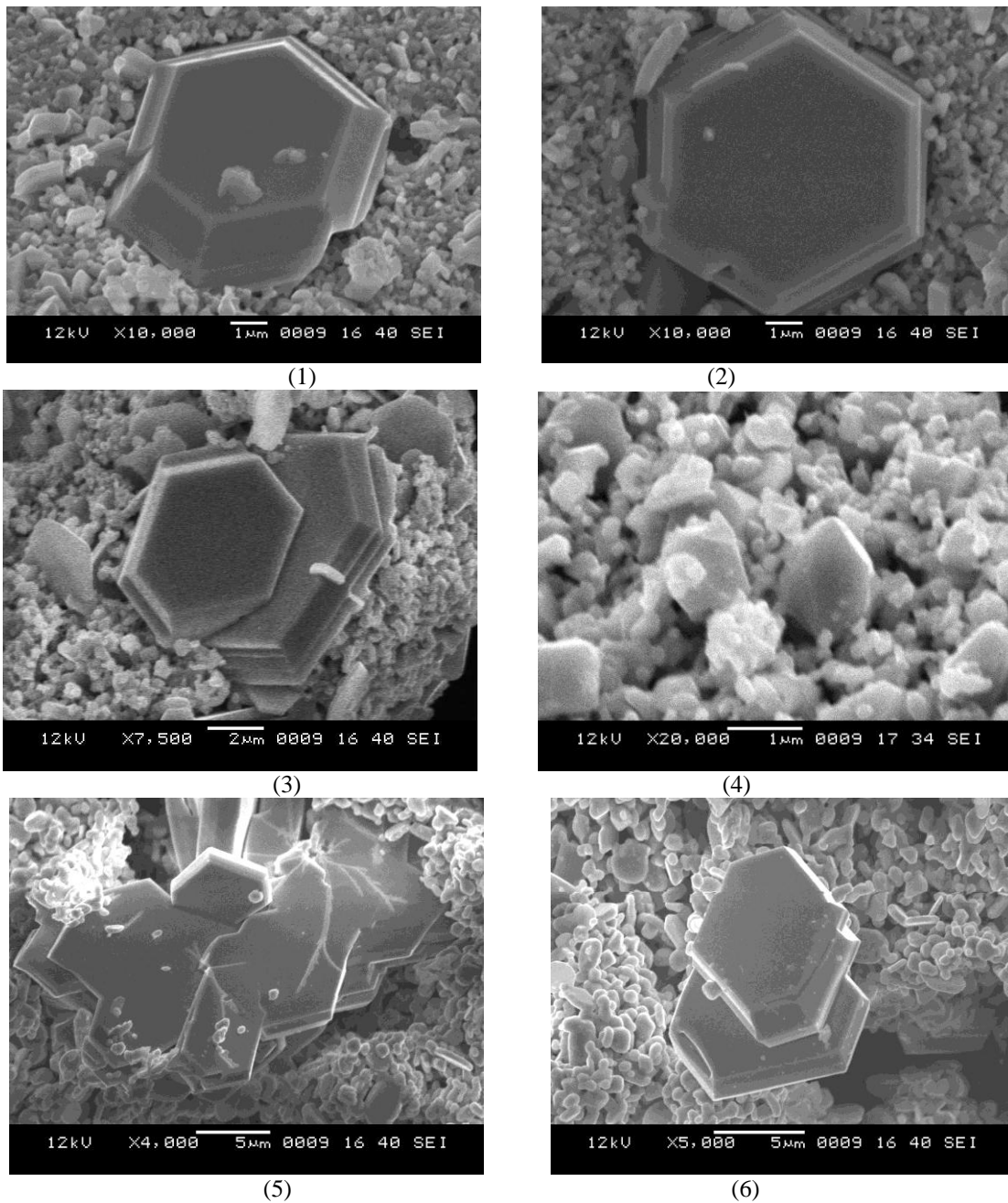


Figure 2. SEM of Ba doped Cr, In,Ti compounds.

3.3. Particle Size And Porosity Analysis-

The particle size of the studied compounds have been calculated by using the following relation

$$d = \frac{0.9 \lambda}{b \cos \theta} \quad (2)$$

where, d is the particle size, $\lambda = 1.54439 \text{ \AA}$ wavelength of X-ray, b is the width of peak at half of its maximum intensity of X-ray and θ is the location of the peak [24]. The particle size of the Ba hexaferrites at 1000°C and 1100°C for 4 hrs and 6 hrs with different doping ions (Cr, In and Ti) have been calculated and it varies from 33.83 nm to 33.87 nm. The comparative study of these compounds are shown in Table-(1, 2, 3) of Ba hexaferrites at 1000°C and 1100°C for 4 hrs and 6 hrs

The porosity of the studied compounds have been calculated by finding the X-ray density (dx) and bulk density (db). The X-ray density has been calculated by using the following relation for hexagonal materials [25].

$$dx = \frac{2M}{N \cdot 0.866 \cdot a^2 \cdot c} \quad (3)$$

where, M represents the molecular weight of the sample, 'a' and 'c' are the lattice parameters and N is the Avogadro's number. The X-ray density depends on the molecular weight and lattice constants of the compounds. The X-ray density varies in the range from 11.9889 g/cm³ to 13.5703 g/cm³. The comparative studies of Ba hexaferrites at 1000°C and 1100°C for 4 hrs and 6 hrs are shown in Table-(1, 2, 3).

The bulk density (db) can be calculated from the geometry of the crystal and mass of the samples. The bulk density of the studied compounds varies in the range from 2.5431 g/cm³ to 3.29637 g/cm³. The comparative studies of Ba hexaferrites at 1000°C and 1100°C for 4 hrs and 6 hrs are shown in Table-(1, 2, 3).

The percentage porosity (p) of the samples has been calculated by using the follow relation

$$p = 1 - \left[\frac{\text{bulk density}}{\text{X-ray density}} \right] \quad (4)$$

the porosity of the studied compounds varies in the range 0.7344 to 0.7952. The comparative studies of Ba hexaferrites at 1000°C and 1100°C for 4 hrs and 6 hrs are shown in Table-(1, 2, 3).

Table-1 Experiment Result

Compound	Calcinated Temp. (°C)	Time (hrs)	Lattice Constant		Particle size d (nm)	X-ray density dx (g/cm ³)	Bulk density db(g/cm ³)	% Porosity (p)
			a (Å)	c (Å)				
BaCr ₂ Fe ₁₂ O ₂₂	1000	4	4.3959	44.4720	33.85	13.0299	3.29637	0.7470
BaCr ₂ Fe ₁₂ O ₂₂	1100	6	4.3555	44.0713	33.87	13.5518	2.7754	0.7952

Table-2 Experiment Result

Compound	Calcinated Temp. (°C)	Time (hrs)	Lattice Constant		Particle size d (nm)	X-ray density dx (g/cm ³)	Bulk density db(g/cm ³)	% Porosity (p)
			a (Å)	c (Å)				
BaIn ₂ Fe ₁₂ O ₂₂	1000	4	4.3198	43.9881	33.88	13.5538	2.8599	0.7889
BaIn ₂ Fe ₁₂ O ₂₂	1100	6	4.5620	44.5905	33.83	11.9889	3.1835	0.7344

Table-3 Experiment Result

Compound	Calcinated Temp. (°C)	Time (hrs)	Lattice Constant		Particle size d (nm)	X-ray density dx (g/cm ³)	Bulk density db(g/cm ³)	% Porosity (p)
			a (Å)	c (Å)				
BaTi ₂ Fe ₁₂ O ₂₂	1000	4	4.5024	44.5082	33.83	13.5703	2.5431	0.7768
BaTi ₂ Fe ₁₂ O ₂₂	1100	6	4.3420	44.5021	33.84	11.7012	2.6754	0.7456

IV. CONCLUSION

Y-type hexaferrites BaM₂Fe₁₂O₂₂ (where M₂ = Cr₂, In₂, Ti₂ trivalent impurities) powders are synthesized by chemical co-precipitation method and subsequent heat treatment at 1000°C & 1100°C for 4 hrs & 6 hrs.

The X-ray diffraction measurements results showed that the Y-type hexagonal structure. The X-ray diffraction patterns of the samples annealed at 1000°C & 1100°C for 4 hrs & 6 hrs which manifest that all the species have hexagonal crystal structure with lattice constants of $a = 4.31 - 4.56 \text{ \AA}$ and $c = 43.98 - 44.59 \text{ \AA}$

The SEM observation of powder BaM₂Fe₁₂O₂₂ sample shows that the microstructure of the compounds are hexagonal.

The particle size analysis shows that the Ba Y-type hexaferrites varies in the range of 33.83 nm to 33.87 nm. The percentage porosity varies in the range of 0.7344 to 0.7952. Hence, the studied compound belongs to the hard ferrite.

V. REFERENCES

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