STRUCTURAL AND MAGNETIC PROPERTIES OF Pb-DOPED $BaFe_{12}O_{19}$ by Co-PRECIPITATED METHOD

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ABSTRACT: *$BaFe_{12-x}Pb_{xO19}$ (x=0.0, 0.2, 0.6, 0.8 and 1.0) was synthesized by co-precipitation method. All samples were sintered at 965±5°C for three hours. For structural analysis, X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) were used. Dopant owns physical properties like higher ionic radii mobility and preference occupancy were the reasons for generation impurity phases. So 63% phase purity achieved. Analysis of XRD data showed that dopant was responsible of variations of different structural parameters because of which properties changed. SEM micrographs showed that lead substitution was responsible of nonuniform variations in crystallite size. Magnetic properties were investigated by magnetometer at room temperature for all samples. All magnetic properties except coercivity modified. Obtained range of coercivity and crystallite size are useful parameters for high density recording media applications.*

Keywords: $BaFe_{12-x}Pb_x O_{19}$, Co-precipitation method, Pb-dopant, Magnetic properties

1. INTRODUCTION

Ferrites because of its doping abilities and applications are one of the best materials for almost all type of applications. This material was first reported by Kato and Takei in 1930. This material consists of iron and oxide as major elements along with divalent and trivalent dopant. Ferrites gained interest due to their usability in high frequency applications like radio and TV communications, computers, microwave devices and other high technological devices. Ferrites have two classes depend upon structure and composition i.e. soft ferrites and hard ferrites [1, 2]. Crystal structure of ferrites can be explained in terms of lattice sites A octahedral and B tetrahedral sites. For antiferromagnetic, magnetic moment of these two sites have equal magnetic moment and for ferrites it is unequal. For ferrites this magnetic moment is the difference between A and B sites. This phenomenon is known as uncompensated anti-ferromagnetism or ferrimagnetism [3, 4].

Hard ferrites itself is a big family and M –type is one of its attractive and fascinating member. M-type structure consists of SRS*R* blocks. Symmetry between S and R blocks symmetry breaks due to trigonal bipyramidal 2b site. This site contains one big divalent Ba or Sr or Pb ion. Presence of Fe^{3+} ion is another factor responsible of uniaxial or planar anisotropy of M-type materials [5]. The whole compound exhibits hexagonal close-packed structure with magnetic moments of cations aligned along c-axis [6,7]. By controlling synthesis methodology and its related parameters its properties can be modified. In presented composition Fe ions are replaced by Pb ions from x=0.0 to 1.0, modification in properties were obtained. Low temperature - synthesis method i.e. Co-precipitation method was used. This method has some advantages like simple techniques, easy to manage and user friendly. Structural and magnetic properties were studied and presented here.

2. Experimental Procedures

To synthesize $BaFe_{12-x}Pb_xO_{19}$, analytical graded 99.99% chemicals were used. They were from Sigma Aldrich, Mark and Fluka. $BaCO_3$, Fe $(NO_3)_3.9H_2O$ and PbO were dissolved in HNO3, DI-H2O and HCl respectively at room temperature along with fast magnetic stirring. For ferrtization, M=5 solution of NaOH was used. Washing was completed in order to minimize the impurities thus homogeneity improved. By removing water contents paste like material obtained. It was dried in an oven at 110±2ºC for overnight. The dried material was crushed into powder form through grinding. This powder then transformed into pellets by applying suitable hydraulic pressure for five minutes. These pellets were sintered at 965±5°C for three hours in a furnace. For identification, synthesized samples were given name Ko, K_1, K_2, K_3, K_4 and K_5 for x =0.0, 0.2, 0.4, 0.6, 0.8 and 1.0 respectively.

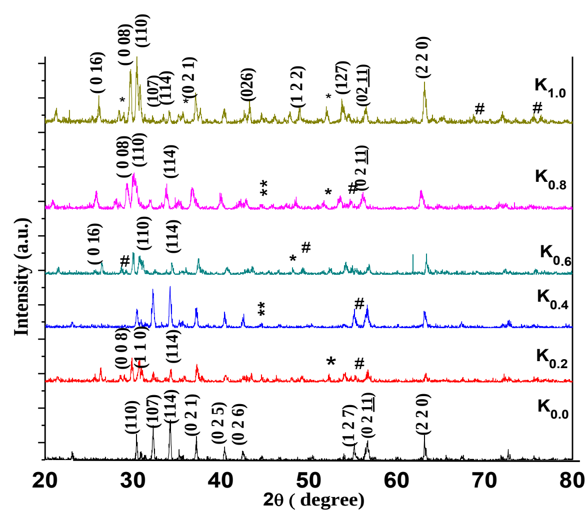

Figure 3.1: Indexed XRD of the Pb-doped $BaFe_{12}O_{19}$ synthesized by co-precipitation method.

3 RESULTS AND DISCUSSION

3.1 Structural analysis

XRD and SEM were used for structural analysis. All samples were exposed to CuKα have λ= 1.5406Å in the range of 20° to 80°. Indexed XRD graph figure 3.1 shows that almost 63% phase purity achieved. Indexed XRD graph of $BaFe_{12-x}Pb_xO_{19}$ has explained that lead in place of iron not only increased the crystallization process but also increased the magnitude of stresses and distortions in successive compositions. Difference in ionic radii (Pb^{2+} =1.76Å and Fe^{3+} = 0.64Å) caused the main peaks (107) and (114) in x=0.0 to decrease and weak in incoming compositions.

Newly developed /generated peaks of same phase as (008) and (110) which are strong showed that phase developed process changed. In each successive composition, slight shift in peaks towards smaller 2θ along with broadening also observed. Lead being low melts point, higher mobility and radii not only generated new phases but also distorted their development pace. Because of which variations in crystallization mechanism observed. So distortions and stresses of different magnitude in different lattice sites of structure not only decreased the phase purity, but also restricted the crystallite size growth as compared to initial enhanced values as shown in graph 3.1. Almost same trend as reported by other researchers when they used oxide dopant like Al_2O_3, SiO_2 and PbO [8, 9].

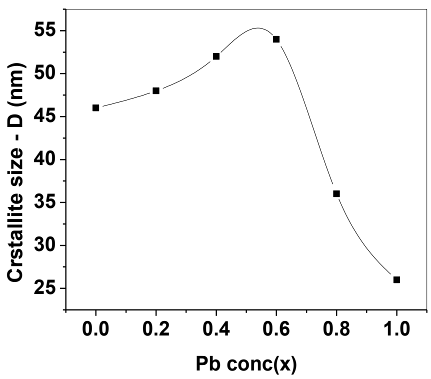

Figure 3.2: Crystallite size (*D*) as function of Pb conc(x)

Because of higher density of lead (11.34g/cm^3) than iron (6.98 g/cm^3) and greater ionic radii magnetic properties modified. The range of crystallite size obtained in synthesized composition is a useful addition towards magnetic media and storage devices applications.

Since growth mechanism has non-uniform trends so lattice parameters "*a*" and "*c*" also have non-uniform behavior. Graph in figure 3.3 confirmed it. So this variation and diffusion on different lattice sites of structure caused the volume of unit cell to squeeze. Following graphs represent these variations.

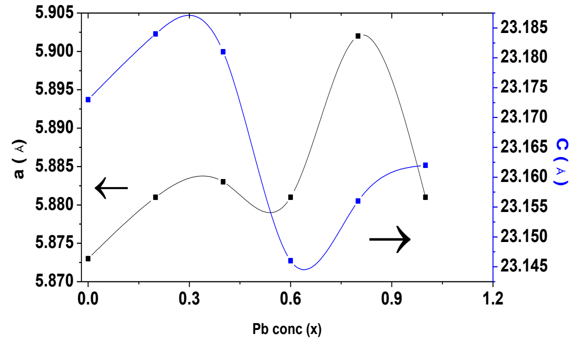

Figure 3.3: Variations in lattice parameters as a function of Pb conc(x)

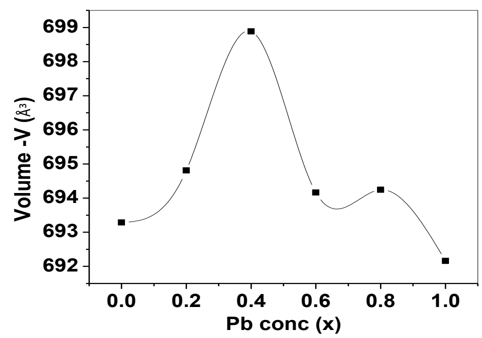

Figure 3.4: Volume as function of Pb conc(x)

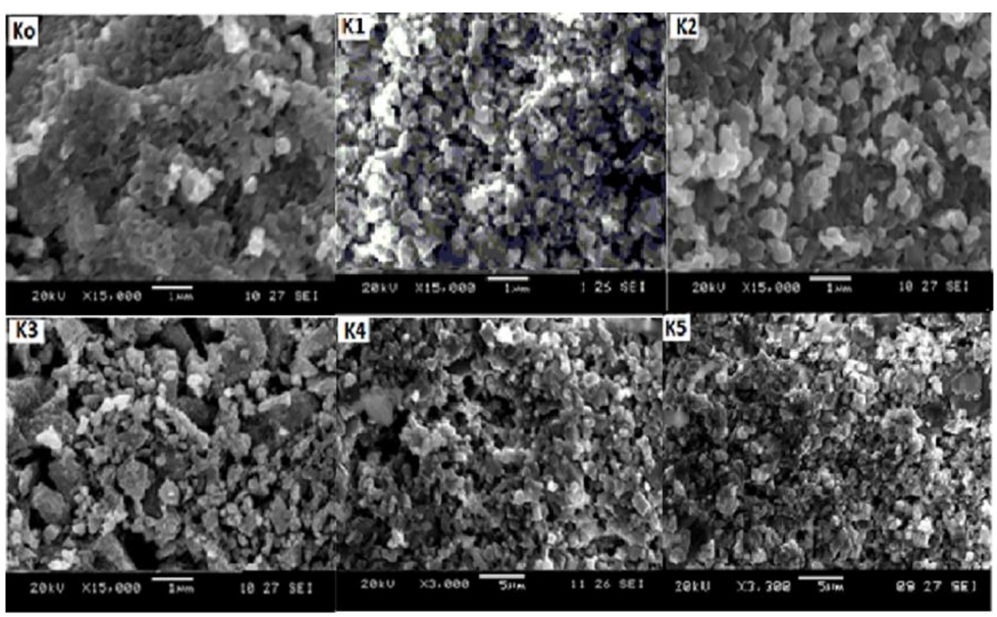

Figure 3.5: SEM micrographs of samples sintered at 965±5°C for three hours

3.2- SEM Analysis

To investigate the surface morphology and particles growth of $BaPb_xFe_{12-x}O_{19}$, SEM was used. Figure 3.5 has explained these features in detail.

By observing K_o and K_1 samples growth variations their orientation and distribution has changed. K_3 confirmed slight restriction started. XRD data also supported it. Further substitution of dopant not only restricted the grain growth but also distorted. As a result grain size/crystallite size decreased. Similar phenomena was also observed and reported by Mingquan Liu *et al* [10]

4-Magnetic Properties

Magnetic properties $BaFe_{12-x}Pb_xO_{19}$ were investigated by using magnetometer at room temperature. Hysteresis loops in the form of *MH* and *BH* has shown in Figure 4. Its further analysis has discussed below.

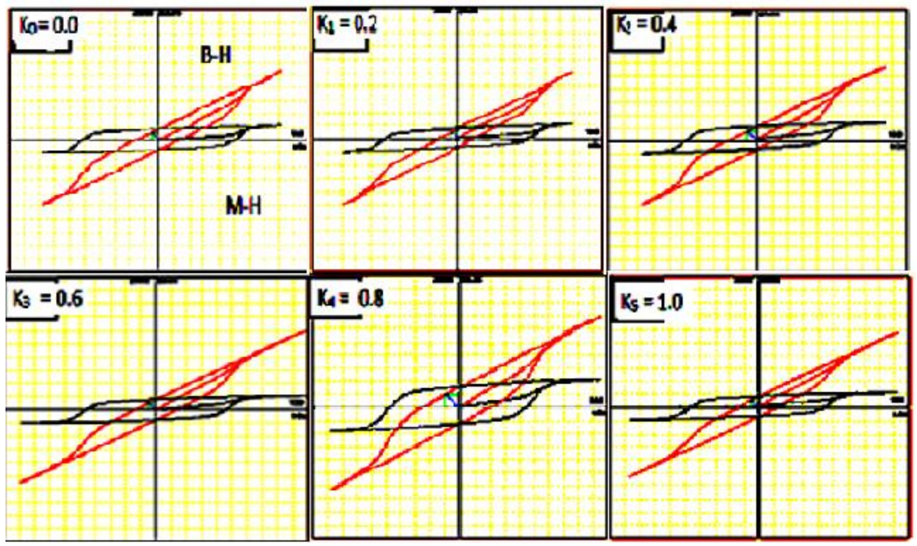

Figure 4.1: MH and BH loop obtained for $BaPb_xFe_{12-x}O_{19}$ composition
[Scale: X-axis H(kOe) : (+10 to -10kOe) and Y-axis B(kG) : (+20 to -20kG)]

4.1- Magnetic analysis

It has reported [11] that grains dimensions and anisotropy energy influence the magnetic properties like coercivity. Coercivity decreases with increase of grain size and with decrease of anisotropy energy. According to Zheng *et al* [12] when non-magnetic and higher ionic radii dopant like PbO diffused into $4f_{vi}$ and 4e or 2b site of iron. Anisotropic energy will decrease so coercivity decreased. SEM micrographs for K=0.6 to K=1.0 showed that due to grain growth restriction, grains boundaries increased thus coercivity increased. Graph below confirms it. But maximum energy product $(BH)_{max}$ increased after "x"=0.2, slight fall at 0.2 was because of coercivity. Increasing trend shows that smart range obtained lies between 0.7291MGOe to 2.013MGOe [13].

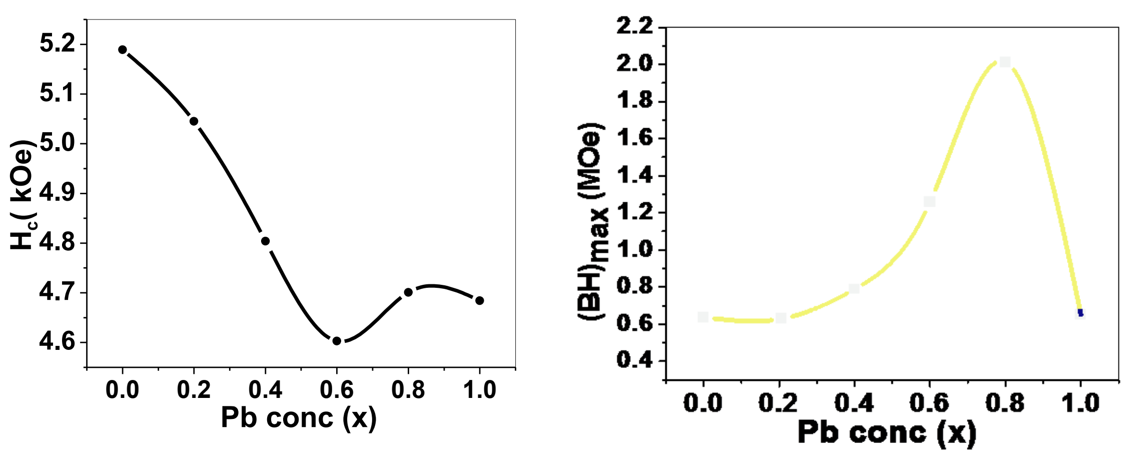

Figure 4.2: *Hc* and (*BH*) $_{max}$ as function of Pb conc (x).

This trend is because of strong inter-grains interactions. Increase in remanance was the reason for increase of maximum energy product. It is a useful achievement which defines the merit of permanent magnet i.e. energy stored by the magnet. This stored energy is to bias or control different devices like phase shifter, filters and various tunable devices [14]. This parameter i.e. $(BH)_{max}$ is very sensitive to microstructure which developed during annealing. Stoner Walfarth and Henkle has described these features well in their discussions[15]

5- CONCLUSIONS

$BaFe_{12-x}Pb_{xO19}$ (x=0.0 to 1.0) was synthesized with co-precipitation. All samples were sintered at 965±5°C for three hours. Dopant low melting point, higher ionic radii and mobility were the reasons for initiating impurity phases because of which 63% phase purity achieved. Lead higher mobility developed non-uniform variations in different structural parameters which were responsible for properties to change. Coercivity and crystallite size decreased useful for magnetic recording media applications. Maximum energy product also increased which was useful for smart storage devices like ATM cards.

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