



Study of Structural Properties of Gd-substituted BaCO₃ M-type Hexagonal Ferrites

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ABSTRACT : A series of hexagonal ferrite samples with the composition of Ba_{1-x}Gd_xFe₁₂O₁₉ (x = 0.0, 0.10, 0.20 and 0.30) have been prepared by employing the ceramic technique. The structural properties of the calcined samples were studied using X-ray diffraction (XRD), SEM technique and FTIR spectrum. The samples were sintered at 1150°C for 8 hours. The X-ray diffraction patterns shows that the prepared samples have a single phase and the effect of composition on the unit cell parameters, density and porosity has been studied. Spectrum of Ba_{1-x}Gd_xFe₁₂O₁₉ ferrites have been studied. The lattice parameters 'c' and 'a' was found to increase by increasing Gd-content whereas the X-ray density decreases and porosity increase by increasing Gd-content. Microstructural analysis by scanning electron microscopy (SEM) suggest that the compound have small grains distributed uniformly and non-uniformly on the surface of the sample and also shows that the grain size has been decreases by increasing the Gd-content in the composition Ba_{1-x}Gd_xFe₁₂O₁₉.

Keywords: Hexagonal ferrites, X-ray diffraction, SEM, IR spectrum.

I. INTRODUCTION

The hexagonal M-ferrite are the most commercially important magnetic materials in use globally [1]. Hexagonal barium ferrites have been intensively investigated during the last few decades due to their considerable importance to the electronic material industry. The hexagonal Barium ferrite (BaM) is considered to be an excellent candidate for magnetic recording media and characterized with high magnetocrystalline anisotropy, moderate hard magnetic properties and high chemical stability, compared with other magnetic materials [2-5]. The common processing methods of hexagonal ferrites are conventional ceramic process of solid-state reaction [6], co-precipitation method [7], sol-gel process [8] and molten salt method [9,10] etc. The conventional ceramic process, which includes the mixing the raw materials, calculation, milling, pressing and sintering at 1200- 1350°C [11]. In a fine particle form, barium ferrite is suitable for high -density recording media. Ultrafine barium ferrite powder with narrow particle size distribution is desirable to increase the capacity of information storage as well as to reduce the medium noise [12].

Infrared (IR) spectroscopy is largely complementary to X-ray diffraction measurements. It points directly to the general nature of unknown substances, where it is able to detect and characterized non- crystalline compounds, such information being obtainable from the presence or absence of characteristic absorption bands. The frequencies at which there are absorption of IR radiation can be correlated directly to bonds with the compound [13-14]. The paper aims at synthesizing Gd Substituted BaM hexaferrite by

conventional ceramic technique. Effect of Gd -rare earth ion on the structure and particle size of hexaferrite were investigated by XRD, SEM and FTIR.

II. EXPERIMENTAL

The hexagonal barium ferrites of nominal composition (x = 0.0, 0.10, 0.20 and 0.30) were synthesized starting from ball-milling mixtures of BaCO₃, Fe₂O₃ and Gd-rare earth ion for 12h. After drying at 60°C for 6h, the powder mixture was heated at temperature of 800-1150°C for 4 h in a lid -covered alumina crucible with a heating rate of 5°C/min in air. Then after cooling to room temperature in furnace.

In order to make the sintered magnet , the barium ferrite powder were wet mixed in acetone medium with addition of 4% polyvinyl alcohol (PVA) binder solution by using a ball mill. After drying, pellets were prepared by pressing at 600MPa in a 10kOe magnetic field applied along the pressing direction. Then the pellets were sintered in a resistance heated furnace for 3h at beach specified level of sintering temperature from 950 to 1250°C. The crystal structure of the samples was examined by using a X-ray diffractometer (XPRT-PRO) with CuKα radiation. The microstructure was investigated using scanning electron microscope (SEM, JEOL-JSM 6100). The absorption bands of the samples was measured by using FTIR- Spectrometer (Thermo, Model no. IS10- Nicolet).

III. RESULT AND DISCUSSION

A. Phase Identification

Fig. 1 shows the XRD pattern obtained for different molar concentration in the prepared samples of

$Ba_{1-x}Gd_xFe_{12}O_{19}$ ferrites sintered at $1250^\circ C$ for 3h. This analysis reveals that the prepared samples were almost single hexagonal M-type Phase. There are no peaks of Fe_2O_3 and Gd_2O_3 phases in the graph which suggest that Ba ion is substituted by Gd ions. The respective peaks show that a magnetoplumbite structure has been formed. The variation in relative intensities of peaks may be related to the occupation of lattice sites by substituted ions. The lattice constants 'a' and 'c' with composition (x) for $Ba_{1-x}Gd_xFe_{12}O_{19}$ has been increases continuously with increasing substituted amount of Gd ions. The peaks for the doped barium ferrites appears at the same position as for undoped ferrite, with different intensities. The result indicates that the formation of temperature of $Ba_{1-x}Gd_xFe_{12}O_{19}$ is about $1250^\circ C$. It is about $50^\circ C$ higher than that of classical ceramic method for undoped barium ferrite as indicated in the literatures. In the doped ferrites cases, the dopant of Gd^{2+} seem to dissolve / arrange in the hexagonal structure to fulfill the formation of single hexagonal phase [15].

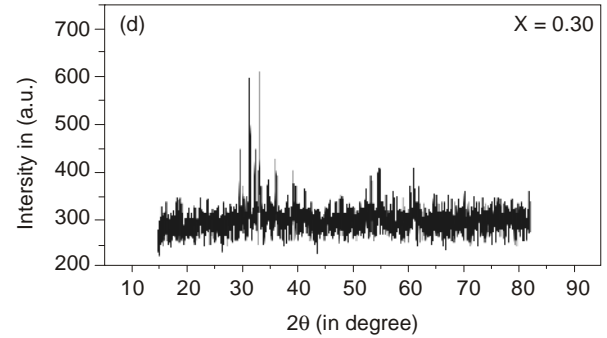
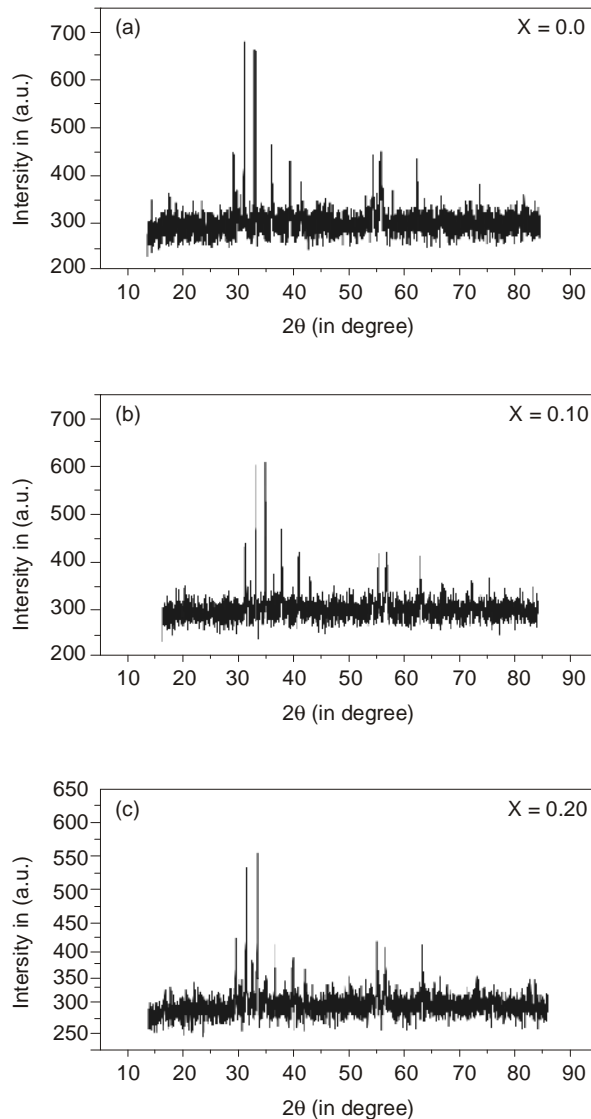


Fig. 1. XRD Patterns of $Ba_{1-x}Gd_xFe_{12}O_{19}$ for compositions (a) $X = 0$, (b) $X = 0.10$, (c) $X = 0.20$ and (d) $X = 0.30$.

B. SEM Microstructures

Fig. 2. shows the microstructures of prepared samples. It indicates that the M-type ferrite grains are homogeneous hexagonal shaped crystals [15]. It was found that the average grain size estimated from SEM was approximately $1\mu m$ and it was almost dependent on the composition X ($x = 0.0, 0.10, 0.20$ and 0.30). The complex permeability spectra of polycrystalline ferrite depends not only on the chemical composition of the ferrite but also on the post-sintering density and the microstructures such as grain size and porosity.

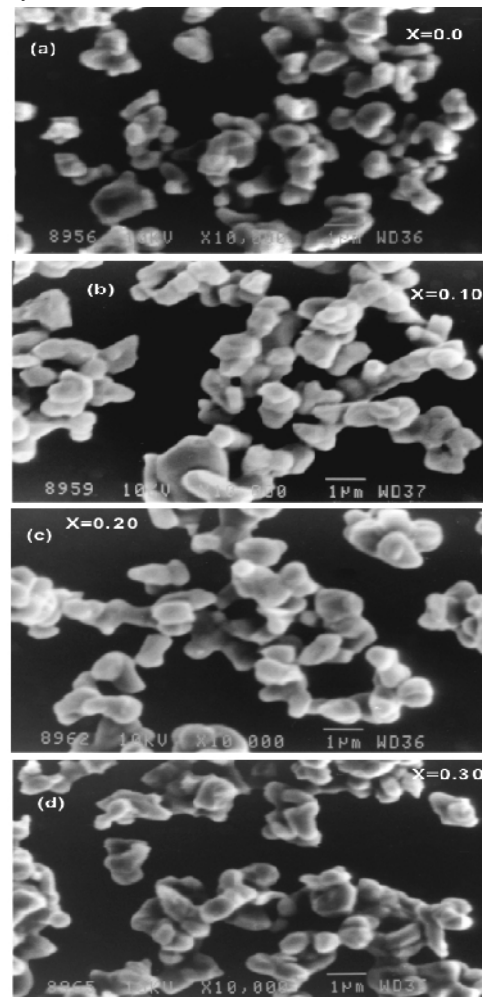


Fig. 2. SEM Photographs of $Ba_{1-x}Gd_xFe_{12}O_{19}$ for compositions (a) $X = 0$, (b) $X = 0.10$, (c) $X = 0.20$ and (d) $X = 0.30$.

C. IR Measurement Results

Fig. 3. shows the IR spectra of the four prepared compositions of $Ba_{1-x}Gd_xFe_{12}O_{19}$ ($X = 0, X = 0.10, X = 0.20$ and $X = 0.30$). For the sample of $X = 0.0$, two absorption bands can easily observed. By introducing Gd ion to the compositions a third band and appears and bands becomes sharper and often shifted towards higher frequencies. The band ν_3 shows the maximum absorptions as compared to the bands ν_1 and ν_2 . The frequencies at which there are absorption of IR radiation can be correlated directly to bonds within the compound [14].

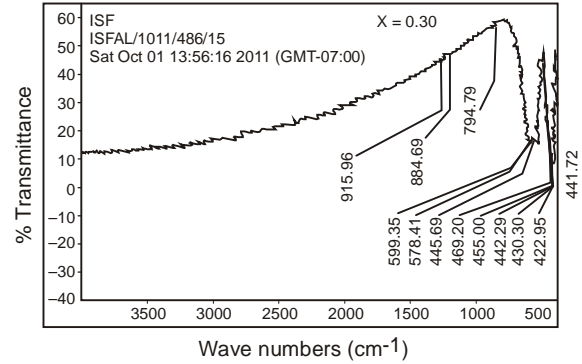
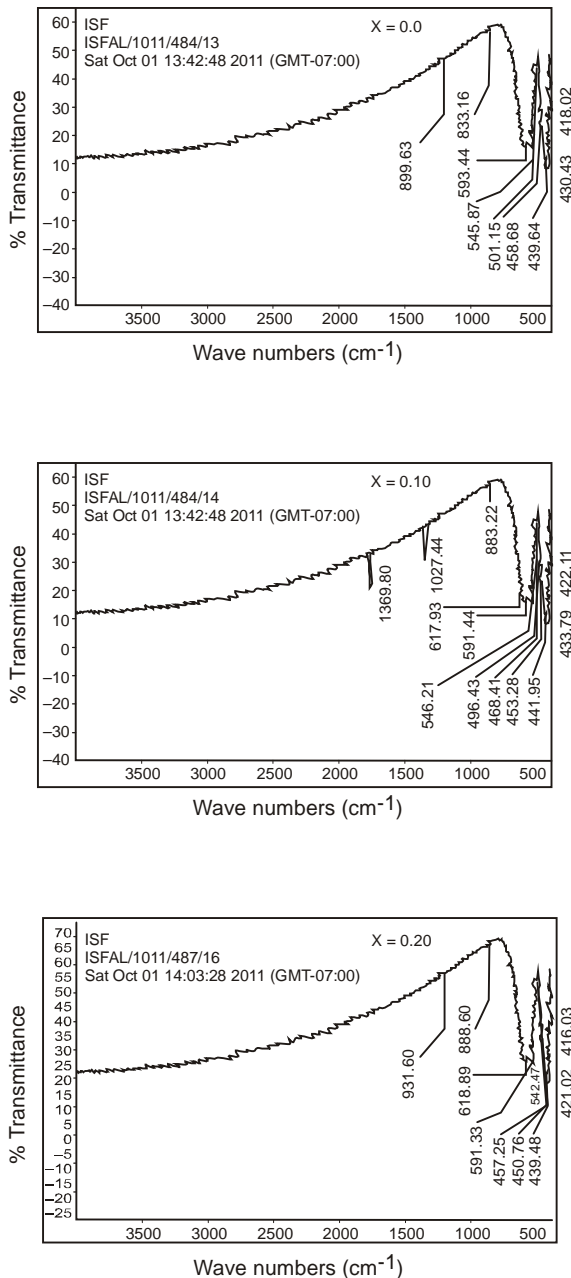


Fig. 3. FTIR of $Ba_{1-x}Gd_xFe_{12}O_{19}$ for compositions (a) $X = 0$, (b) $X = 0.10$, (c) $X = 0.20$ and (d) $X = 0.30$

IV. CONCLUSION

$Ba_{1-x}Gd_xFe_{12}O_{19}$ hexaferrite are prepared by the usual ceramic technique. From the above study, we conclude that the X-ray diffraction pattern shows that the prepared samples have a single phase structure. The lattice parameters 'c' and 'a' was found to increase by increasing Gd-content whereas the X-ray density decreases and porosity increases by increasing Gd-content. SEM suggested that the compound have small grains distributed on the surface of the sample and also shows the grain size has been decreases by increasing the Gd-content in the composition $Ba_{1-x}Gd_xFe_{12}O_{19}$. FTIR suggested that the frequency at which there are absorption of IR radiation can be correlated directly to the bonds within the compound.

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