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Synthesis and characterization of barium ferrite–silica nanocomposites

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ABSTRACT

In this work, we prepared barium ferrite-silica (BaM-SiO_2) nanocomposites of different molar ratios by high-energy ball milling, followed by heat-treatment at different temperatures. The microstructure, morphology and magnetic properties were characterized for different synthesis conditions by using X-ray diffraction (XRD), scanning electron microscopy (SEM) and vibrating sample magnetometry (VSM). The results indicate that 15 h of milling was enough to avoid the generation of hematite phase and to get a good dispersion of barium ferrite particles in the ceramic matrix. For milling periods beyond 15 h and heat treatment above 900 °C, the XRD patterns showed the presence of hematite phase caused by the decomposition of BaM. The agglomerate size observed through SEM analysis was around 150 nm with a good BaM dispersion into the SiO_2 matrix. The highest saturation magnetization (Ms) value obtained was 43 emu/g and the corresponding coercivity (Hc) value of 3.4 kOe for the composition 60BaM-40SiO_2 milled for 15 h and heat treated at 900 °C. This coercivity value is acceptable for the application in magnetic recording media.

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1. Introduction

In recent years, a large number of studies have been reported on magnetic materials, which are widely used as components in various technological applications [1]. Ferrites are an established group of magnetic materials which are used in many advanced technology applications. They can be classified into three different classes; spinel, garnets and hexagonal ferrites. Among these, hexagonal or M-type ferrites are the ones used for permanent-magnet applications [2]. These ferrites are hexagonal compounds of the general formula $MeFe_{12}O_{19}$, where Me = Ba, Sr, or Pb. Among these, $BaFe_{12}O_{19}$ often denoted as M-type BaM, have the lowest price per unit of magnetic energy and are used for numerous applications such as permanent magnets, particulate media for magnetic recording and microwave devices due to its excellent chemical and physical properties such as large magnetocrystalline anisotropy, high curie temperature, acceptable mechanical hardness, excellent chemical stability, relatively large saturation magnetization and high-corrosion resistance [3,4]. The magnetic properties of these materials arise from the interactions between metallic ions occupying particular positions relative to the oxygen ions inside the crystalline structure. Their magnetic properties depend mostly on their grain size, heat treatment temperature and phase purity, which very much depends on the preparation methods. There are several methods to synthesize barium ferrite and its composites including the traditional ceramic sintering route, sol-gel method, hydrothermal, chemical co-precipitation, sputtering techniques, etc [5–13]. Mechanical milling is a solid state technique used extensively for the synthesis of a wide range of nanostructured materials and powder particle refinement through mechanical assisted interactions [14] and has also been used in the synthesis of magnetic powders [15–17]. Milling induces rigorous plastic deformation due to the gradual distortion of the internal structure of the powders to the nanometer level, which affects the magnetic properties of magnetic materials. The magnetic properties of BaM can be

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modified by elemental substitutions on the Ba^{2+} or Fe^{3+} sites or both, or by making nanocomposites, which are comprised of nano-sized magnetic material embedded in an amorphous matrix [18,19].

Usually the magnetic nanocrystals obtained by different procedures have a strong tendency to aggregate and it is difficult to take full advantage of their unique physical properties, but the dispersion of these nanocrystals in an organic or inorganic matrix helps in reducing particle agglomeration [20]. It has been proposed earlier that a ceramic host can be an effective medium to incorporate particles with uniform size and homogeneous distribution of the metal oxides [21]. The magnetic and electromagnetic absorption properties can be modified by the incorporation of BaM nanocrystals into SiO_2, Al_2O_3, and ZrO_2 ceramic matrixes to make magnetic nanocomposites [4,21]. One of the challenges of BaM is to reduce the grain size for applications in high density magnetic recording media. In this work, we report the synthesis of $BaFe_{12}O_{19}$-SiO_2 nanocomposites with different ratios processed by high-energy ball milling and its characterization studies. To our knowledge this study presents first time the synthesis of BaM-SiO_2 nanocomposites by high-energy mechanical milling. The effect of milling time and heat treatment conditions on the microstructure and magnetic properties of ceramic BaM-SiO_2 nanocomposites is discussed.

2. Experimental

BaM-SiO_2 nanocomposites were synthesized by mechanical milling in a planetary ball mill RESTCH, model PM400. The raw materials consisted of BaM and SiO_2 powders of analytical grade reagent of particle size ~5 μm and 10 μm, respectively. The BaM/SiO_2 ratios used were 40:60, 50:50, 60:40 and 70:30 by volume percent. Stoichiometric mixtures of the above chemicals were placed in a silica containers together with 20 mm diameter quartz balls as milling media (ball to powder ratio = 10:1). Dry mechanical milling was carried out in air in a planetary ball mill by using a rotating speed of 350 rpm for 5, 15 and 30 h and the obtained powder after every milling time was heat treated at 500, 900, 1000 °C. Phase evolution on milling and the milled sample after heat treatment were analyzed by using X-ray powder diffraction in Philips X'pert Diffractometer using Ni-filtered CuK_α radiation (λ = 1.5418 Å) in the range of 2θ = 20-80°. The morphological features of the particles were observed by using a scanning electron microscope (SEM) Philips XL 30ESEM. The purpose of the heat treatment was to homogenize the grain size of the resulting nanocomposites and create mono-domains in the barium ferrite particles to modify the magnetic properties. The magnetic properties of the samples were measured at room temperature by using a vibrating sample magnetometer (Lakeshore model 7300/9300 VSM) with an applied field up to 1.3 T and calibrated with pure Ni standard.

3. Results and Discussion

The structural evolution and phase formation of the composite after milling and heat treatment was examined by X-ray diffraction. Fig. 1 shows the X-ray diffraction patterns of (a) pure SiO_2, (b) pure BaM, and 50BaM-50SiO_2 composites milled for (c) 5 h, (d) 15 h and (e) 30 h. The samples milled for 5 h indicated only the presence of barium ferrite and silica phases. There is no evidence of decomposition of either of the two phases after short milling times; however, BaM suffered a minor amorphization in the composite system as seen by comparing the diffraction patterns of pure BaM and nanocomposite. As the milling time increases from 5 h to 15 h, the diffraction peaks of BaM became broad and the peak height decreases due to the increase in defects and disorder in the crystal lattice with increasing milling time. For a milling time of 30 h, the diffraction pattern showed a change in the crystallinity as a result of the partial amorphization or structural disorder. The diffraction pattern shows the decomposition of BaM and also the intensities of the diffraction pattern diminish and the peaks become broadened due to the reduction of the particles and distortion of the lattice. In addition to that, the samples milled for 30 h show the presence of hematite (α-Fe_2O_3) as a secondary phase, with the most intense reflection (104) of hematite phase appearing at 33.15°.

Fig. 2 shows the XRD pattern of samples of different BaM/SiO_2 ratios milled at different times and heat-treated at various temperatures. It can be seen that samples of composition 60BaM-40SiO_2 milled for 15 h and heat-treated at 1000 °C (Fig. 2d) show the main peaks of the hematite phase. In addition, the samples milled for 30 h show an increase in the number of diffraction peaks of the hematite. From Fig. 2, it can be seen that samples of BaM-SiO_2 with a 60:40 ratio milled for 15 h and heat treated at 900 °C showed the best structural properties, though it also exhibits a small quantity of hematite. The result shows that for BaM-SiO_2 samples with ratios of 50:50 and 60:40, a heat-treatment at 900 °C is sufficient to promote structural rearrangement of Ba and Fe ions in the composites. As the temperature increases to 1000 °C, partial decomposition of BaM into hematite takes place inside the silica matrix.

Fig. 3 shows the SEM micrographs of (a) pure BaM, (b) silica, (c) nanocomposite milled for 15 h, and (d) 15 h milled sample heat-treated at 1200 °C. The particle size distribution of the BaM sample was found in the range of 1 μm and that corresponding to the silica varied from 10 to 50 μm. Milling greatly altered the size and shape of the particles. The sample 60BaM-40SiO_2 milled for 15 h showed agglomerates of size ranging between 0.5 and 1 μm (Fig. 3c). To observe the distribution of phases and the dispersion of the magnetic phase in the ceramic matrix, samples were heat-treated at 1200 °C and analyzed by SEM in backscattering electron mode, which is shown in Fig. 3d. The photograph shows a biphasic microstructure and also a good dispersion of the BaM phase into the ceramic matrix. In this SEM mode, the phase with lower atomic weight will appear darker, silica in the present case and BaM appears as the clear phase. The agglomerate size observed through scanning electron microscopy analysis is around 150 nm with a good BaM dispersion into the SiO_2 matrix.

The values of the saturation magnetization and coercivity as a function of milling time for different compositions, heat-treated at 500 °C is shown in Fig. 4. It is noted that the system 70BaM:30SiO_2 has the highest value of magnetization

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