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Abstract. Two-phase nano-powders based on Ba hexaferrite with addition of Mn have been obtained by a sol-gel auto-combustion method. Composition dependence of magnetic and structural properties has been studied using magnetic measurements, X-ray diffraction (XRD) and scanning electron microscopy (SEM). The observed decrease of saturation magnetization and coercivity with increase of Mn content is mainly attributed to the presence of Fe oxide as a result of Ba depletion. SEM images show chemically homogeneous, non-regular shaped agglomerated nano-particles (50-200 nm).

Keywords: Barium Ferrite, XRD, Magnetic measurements, SEM.

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INTRODUCTION AND EXPERIMENTS

M-type hexaferrites have been the subject of intensive studies due to an appealing combination of good hard magnetic properties and low cost [1]. Attempts have been made to improve their magnetic properties by substituting Fe3+ with a different ion, e.g. Mn3+ [2]. Another possible way is to make a spring magnet comprising two exchange-coupled (hard and soft) magnetic phases [3]. For the system involved, this can be a nano-composite of hexagonal ferrite BaFe12O19 and spinel ferrite MnFe2O4. In the present work we report on the effect of Mn addition combined with Ba depletion on magnetic and structural properties of Ba hexaferrite.

Powders with nominal composition Ba1-xMnxFe12O19 (x = 0, 0.2, 0.4, 0.6, 0.8) were prepared by a sol-gel auto-combustion technique [4], using carbonate and nitrates as sources of metals and citric acid as fuel. Final annealing, necessary for hexaferrite phase synthesis, was done at 800 °C for 4 hours (relatively low temperature to limit particle coarsening). Combustion method offers a quick hands-on route to produce fine hexaferrite precursors without any special setup or complex chemical reactions. X-ray diffraction measurements were done using a Bruker D8 Advance diffractometer with Cu-Kα radiation. Low-vacuum field emission scanning electron microscopy (FE-SEM) imaging of the surfaces and energy dispersive X-ray spectroscopy (EDS) were performed with an FEI Quanta 400FEG. Room temperature magnetic hysteresis measurements were done by SQUID Magnetometer (Quantum Design MPMS-5S), with a maximum applied field of ± 5 T, and were used to obtain coercivity (Hc) and saturation magnetization (Ms).

RESULTS AND DISCUSSIONS

Figure 1 depicts the magnetization curves for Ba1-xMnxFe12O19 samples. Perusal of figure 1 reveals substantial decrease of saturation magnetization with increase of x: obtained Ms values for x = 0, 0.2, 0.4, 0.6, 0.8 are respectively 60.12, 61.85, 49.76, 30.08, 25.02.5 T

FIGURE 1. Hysteresis loop of the samples with different Mn content. Inset: Hc dependence.
19.32 emu/g. Mn ions partially demolish the collinear ferrimagnetic structure of the hexaferrite, which results in the reduction of magnetization [2]. In our case, however, the drop of $M_s$ is mainly due to the presence of $\alpha$-Fe$_2$O$_3$ (hematite) phase confirmed by XRD. Inset of figure 1 shows the behavior of coercivity as a function of $x$: $H_c$ becomes lower with introduction of Mn. In a single-phase material, on the contrary, an increase of coercivity with increase of Mn concentration was observed [2].

Table 1 summarizes the lattice parameters $a$ and $c$, average grain size $D$ and weight fraction of the phases present in the sample, obtained by XRD Rietveld refinement. Perusal of table 1 shows that the sample without Mn contains only pure BaFe$_{12}$O$_{19}$ phase, with $a$ and $c$ matching well the reported values [1]. With introduction of Mn, new $\alpha$-Fe$_2$O$_3$ phase appears while the fraction of BaFe$_{12}$O$_{19}$ phase gradually decreases. Since no MnFe$_2$O$_4$ phase is detected, Mn partially substitutes Fe in Ba hexaferrite and/or Fe oxide. Figure 2 (a)-(c) shows the morphology of the produced powders, exhibiting agglomerated nano-particles with non-regular shapes, ranging from 50 to 200 nm. All samples appear chemically homogeneous, thus it is not possible to distinguish between BaFe$_{12}$O$_{19}$ and $\alpha$-Fe$_2$O$_3$ grains (both can be Mn-doped). In Figure 2 (d) the EDS spectra, obtained from the nanoparticles, reveal the presence of C (due to the carbon glue used to fix the samples), O, Fe, Mn and Ba. The insets of figure 2 (d) clearly show the decrease of the MnK band and the increase of the BaL band, with the increase of Mn content, whereas the Fe bands present constant intensities.

To summarize, composition dependence of magnetic and structural properties of Ba$_{1-x}$Mn$_x$Fe$_{12}$O$_{19}$ powders ($x = 0, 0.2, 0.4, 0.6, 0.8$) is reported. With increase of Mn content, linear decrease of magnetization is observed. Responsible for inferior magnetic properties is $\alpha$-Fe$_2$O$_3$ phase formed apart from BaFe$_{12}$O$_{19}$ phase due to Ba depletion. SEM shows agglomerated nano-particles with non-regular shapes, ranging from 50 to 200 nm.

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