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Influence of Mn Addition On Magnetic And Structural Properties of Barium Hexaferrite

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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.

PACS: R61.05.cp, 75.47.Lx, 75.50.Gg

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 Fe^{3+} with a different ion, e. g. Mn^{3+} [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 $\text{BaFe}_{12}\text{O}_{19}$ and spinel ferrite MnFe_2O_4 . 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 $\text{Ba}_{1-x}\text{Mn}_x\text{Fe}_{12}\text{O}_{19}$ ($x = 0, 0.2, 0.4, 0.6, 0.8$) 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-K_α 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 (H_c) and saturation magnetization (M_s).

RESULTS AND DISCUSSIONS

Figure 1 depicts the magnetization curves for $\text{Ba}_{1-x}\text{Mn}_x\text{Fe}_{12}\text{O}_{19}$ samples. Perusal of figure 1 reveals substantial decrease of saturation magnetization with increase of x : obtained M_s values for $x = 0, 0.2, 0.4, 0.6, 0.8$ are respectively 60.12, 61.85, 49.76, 30.08,

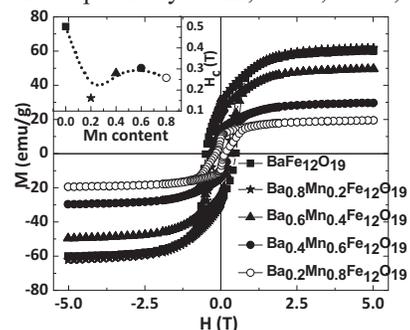


FIGURE 1. Hysteresis loop of the samples with different Mn concentration. Inset: H_c dependence.

TABLE 1. Parameters of XRD analysis, obtained using Rietveld refinement, where a, c – Lattice parameters, D – Average grain diameter, wt. % of the phase present in the sample.

Mn	M-ferrite				Hematite			
	a (nm)	c (nm)	D (nm)	wt. %	a (nm)	c (nm)	D (nm)	wt. %
0	0.5589	2.3215	78	98	–	–	–	–
0.2	0.5892	2.3214	131	89	0.5042	1.3754	129	11
0.4	0.5891	2.3202	124	73	0.5039	1.3744	131	27
0.6	0.5891	2.3202	86	45	0.5038	1.3748	183	55
0.8	0.5889	2.3190	51	28	0.5036	1.3744	218	72

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 α -Fe₂O₃ (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 BaFe₁₂O₁₉ phase, with a and c matching well the reported values [1]. With introduction of Mn, new α -Fe₂O₃ phase appears while the fraction of BaFe₁₂O₁₉ phase gradually decreases. Since no MnFe₂O₄ 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 BaFe₁₂O₁₉ and α -Fe₂O₃ grains (both can be Mn-doped). In Figure 2 (d) the EDS spectra, obtained from the nano-powders, 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 Mn_K band and the increase of the Ba_L 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_xFe₁₂O₁₉ 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 α -Fe₂O₃ phase formed apart from BaFe₁₂O₁₉ phase due to Ba depletion. SEM shows agglomerated nano-particles with non-regular shapes, ranging from 50 to 200 nm.

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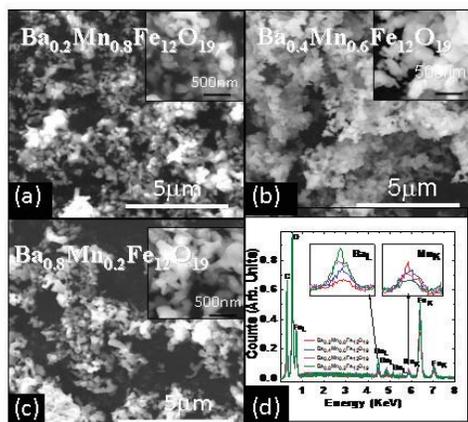


Figure 2. SEM images of Mn doped Barium ferrite powders: (a) $x = 0.8$, (b) $x = 0.6$; (c) $x = 0.2$; (d) EDS spectra of the sample with insets of Ba_L and Mn_K peaks.