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Comparison between Powders of Strontium Hexaferrite Processed by Dynamic Gas Heat Treatment and Re-Calcination

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The utilization of classical ceramic technology for ferrite preparation permits to obtain strontium hexaferrite with big crystals, of micrometer size, with a smaller coercive force than the hexaferrite with submicron crystals. In this work the strontium hexaferrite was obtained with submicron crystals, by hot reduction of the hexaferrite obtained through ceramic technology in CO atmosphere, followed by the thermal re-oxidization in air. The effects of treatment in CO atmosphere and of the re-calcination treatment on the magnetic and electric properties of the strontium hexaferrite were investigated.

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1. Introduction

The hexaferrites present a real interest from the standpoint of their applications in various domains, such as permanent magnets, magnetic storage mediums, microwaves, given their special properties: high coercivity and remanence, low manufacturing cost, chemical and thermal stability [1–4].

The highest coercivity value can be obtained when the crystals have the maximum size at which they remain monodomains. One of the methods used to obtain submicron powder of strontium hexaferrite with big coercivity is the method of reduction and re-oxidization of hexaferrite powder [5].

In this work a strontium hexaferrite powder was obtained, calcined at the temperature of 1100 °C for 1 h, to which a hot reduction treatment in CO flux was applied for 1 h at the temperature of 900 °C, followed by re-calcination for 1 h at the temperature of 1000 °C.

2. Experimental methods

The strontium hexaferrite powder was prepared using the classical ceramic technology. The quantities of Fe_2O_3 and SrO with 99% purity (Merck) were weighted according to the stoichiometric formula, and they were homogenized in a ball mill for 14 h, after which they were treated in air for 12 h at the temperature of 1100 °C.

The powder was dynamically treated in a horizontal furnace in CO flow for 1 h at the temperature of 900 °C. The resulted powder was re-calcined in air for 1 h at 1000 °C.

The samples were made from the hexaferrite initially obtained at 1100 °C, as well as from the material resulting after the dynamic treatment at 900 °C in CO atmosphere, and from the material resulting after re-calcination in air at 1000 °C. The samples were investigated from microstructural, magnetic and electric standpoint.

In order to use the hexaferrite in the magnetic memory applications, it needs to have the highest coercivity possible, and this can be obtained for a crystal size of 0.5–0.6 μm.

Three samples of strontium hexaferrite were obtained and their microstructure was investigated by X-ray diffraction, using a Cu K_α anticathode, at a 4 degree/min scanning rate (Shimadzu LabX XRD-6000), and by electron microscopy.

For the measurements of remanent magnetization and coercivity, spherical samples were prepared with the diameter of 5–6 mm. The measurements were carried out with a vibrating sample magnetometer at the temperature of 20 °C and an applied field of 800 kA/m.

In order to carry out electric investigations, the strontium ferrite powder was uniaxially pressed in shape of discs with the diameter of 10 mm and thickness of 2–3 mm. Silver electrodes were deposited on the both plane sample surfaces. The electric properties were investigated with Solartron 1260 A Impedance/Gain Analyser, in the frequency range $1 \div 10^6$ Hz.

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3. Results and discussion

The analysis of the X-ray diffraction presented in Fig. 1 shows that the sample initially prepared at the temperature of 1100 °C mainly contains $SrFe_{12}O_{19}$, and small amounts of hematite (Fe_2O_3) and magnetite (Fe_3O_4). After carrying out the dynamic treatment in CO flux for 1 h at 900 °C, most part of strontium hexaferrite had disappeared, being converted in Fe_2O_3, SrO, FeO and α-Fe. After re-calcination in air for 1 h at 1000 °C, the strontium hexaferrite has completely regenerated, only small amounts of hematite and magnetite remained, similar to the initial sample.

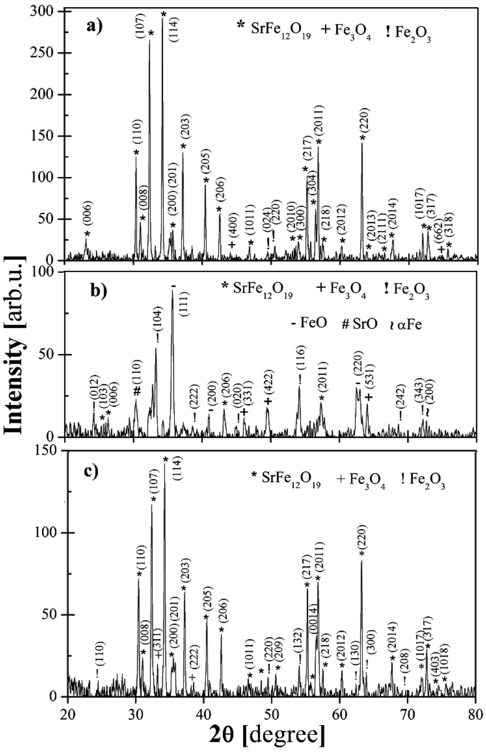

Fig. 1. X-ray diffraction patterns of the powders annealed in (a) air at 1100 °C, (b) CO dynamic atmospheres, (c) air at 1000 °C.

The initially obtained hexaferrite powder contains crystallites with size as big as 5–10 μm, Fig. 2a. After the treatment in CO atmosphere, spherical crystallites resulted with the mean size of 0.2 μm, Fig. 2b. After re-calcination at 1000 °C, plate crystals with the mean size of 0.5 μm were formed (Fig. 2c).

The sample initially calcined at 1100 °C is typically magnetic hard, with H_c = 127 kA/m and M_R/M_S = 0.54 (see Table I). The sample dynamically treated in CO atmosphere at 900 °C is typically magnetic soft and presents a diminution of M_R and M_S, as well as a drastical decrease of H_c down to 23.8 kA/m. After re-calcination in air at 1000 °C, the strontium hexaferrite has higher M_R and M_S, and coercivity almost twice as big as the initial sample.

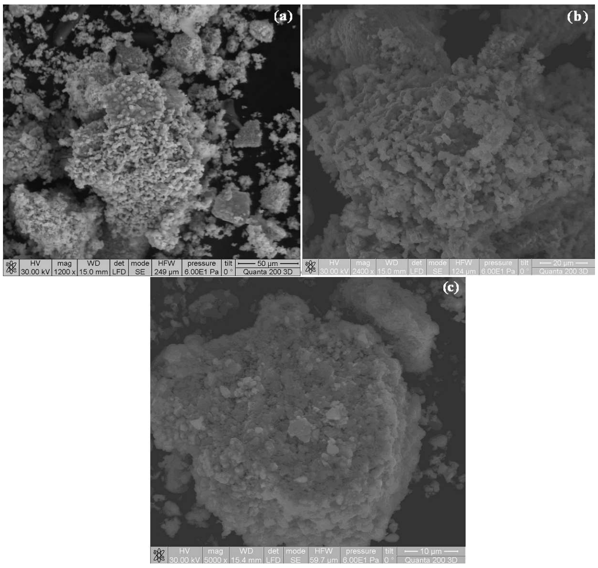

Fig. 2. SEM micrographs of (a) initial powder, (b) the powder after dynamic CO treatment, (c) the powder after re-calcination.

As the result of the electric investigation, it was found out that the sample initially treated at 1100 °C and the sample re-calcined at 1000 °C have similar electric properties, as can be seen from Fig. 3 and Table II. The sample dynamically treated in CO flow has its conductivity and dielectric losses much higher than the samples treated in air.

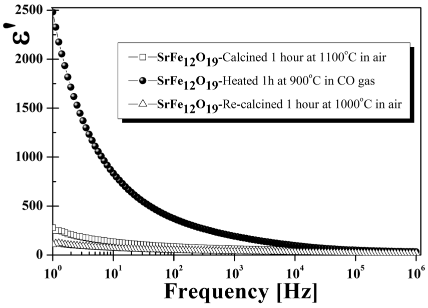

Fig. 3. Real part of permittivity vs. frequency at room temperature in $SrFe_{12}O_{19}$ hexaferrites.

TABLE II

Summary of the dielectrics characteristics of $SrFe_{12}O_{19}$ hexaferrites.

Sample	Dielectric loss low frequency [10^3 Hz]	Conductivity [S/m]
calcined at 1100 °C in air	0.18	1.12×10^{-8}
heated at 900 °C in CO	0.55	6.27×10^{-7}
re-calcined at 1000 °C in air	0.17	2.87×10^{-9}

TABLE I

Summary of the magnetic properties of strontium hexaferrites.

Sample	Coercivity H_c [kA/m]	Saturation magnetization M_S [emu/g]	Remanence M_R [emu/g]
calcined at 1100 °C in air	127	42.2	25.7
heated at 900 °C in CO	23.8	39.9	15.4
re-calcined at 1000 °C in air	214	48.1	28.1

4. Conclusions

By reduction in CO flow and re-oxidization, the strontium hexaferrite preserves its composition and electric properties, a controlled diminution of its crystal size and an important improvement of its magnetic properties take place.

The treatment of $SrFe_{12}O_{19}$ in CO atmosphere for 1 h at 900 °C resulted in:

— decomposition of $SrFe_{12}O_{19}$ in α-Fe, FeO and SrO; increase of conductivity and dielectric loss;

— crystallite diminution and loss of hard magnetic character, through the noticeable decrease of H_c and of M_R.

The re-calcination in air for 1 h at 1000 °C resulted in:

— complete regeneration of $SrFe_{12}O_{19}$, noticeable decrease of conductivity and dielectric loss;

— crystallite growth at optimum size and improvement of hard magnetic properties, as compared to the initial hexaferrite.

The presented method permits to obtain submicron hexaferrite powder, with applications in magnetic memory media.

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