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Mechanosynthesis, crystal structure and magnetic characterization of M-type $SrFe_{12}O_{19}$

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Abstract

M-type strontium hexaferrite was prepared by mechanosynthesis using high-energy ball milling. The influence of milling parameters, hematite excess and annealing temperature on magnetic properties of $SrFe_{12}O_{19}$ were investigated. Commercial iron and strontium oxides were used as starting materials. It was found that mechanical milling followed by an annealing treatment at low temperature (700 °C) promotes the complete structural transformation to Sr-hexaferrite phase. For samples annealed at temperatures from 700 to 1000 °C, saturation magnetization values (M_s) are more sensitive to annealing temperature than coercivity values (H_c). The maximum M_s of 60 emu/g and H_c of 5.2 kOe were obtained in mixtures of powders milled for 5 h and subsequently annealed at 700 °C. An increase in the annealing temperature produces negligible changes in magnetic saturation and coercivity. An excess of hematite as a second phase produces a slight decrease in the saturation magnetization but leads to a significant increase in coercive field, reaching 6.6 kOe.

Keywords: Mechanochemical processing; Oxide materials; Hexaferrite; $SrFe_{12}O_{19}$; Magnetization

1. Introduction

M-type hexagonal ferrites are important permanent magnet materials widely produced since their discovery in the 1950s. Although many other magnetic materials have been synthesized after these years, the performance/cost ratio is still extremely favorable to M-type ferrites. Among these ferrites, strontium hexagonal ferrite, $SrFe_{12}O_{19}$, possesses a special place due to its magnetic properties [1].

Hexaferrites can be synthesized by several processes. The conventional and oldest one is by calcination and sintering of a mixture of oxides or carbonates in a furnace at 1300 °C [2]. This process produces large particles and consumes extensive energy [3]. Nanostructured hexaferrites can be produced by wet chemical methods such as sol–gel [4], hydrothermal [5], coprecipitation [6,7], sol–gel-autocombustion [8], spray-drying and microemulsion [9], among others [10,11]. A particular method is mechanosynthesis; typically this method promotes formation of ferrites by mechanical activation of carbonate strontium and iron oxide [12,13]. In comparison with the traditional method (solid state reaction), the mechanochemical method has been demonstrated to achieve high coercivity and magnetic saturation in these materials [13–15].

Luo et al. [13] prepared strontium hexaferrite by mechanosynthesis of a mixture of $SrCO_3$ and Fe_2O_3 and post-annealing. They obtained an amorphous material after ball-milling for 30 h, and a $SrFe_{12}O_{19}$ single phase after annealing at 900 °C for 2 h. The saturation magnetization was 58.2 A m^2/kg (58 emu/g), and the coercivity was 281.2 kA/m (3500 Oe) at room temperature. A similar study by mechanosynthesis was reported by Sharma et al. [14] to obtain barium hexaferrite; using the same experimental parameters they found an improvement in some magnetic properties such as saturation magnetization and coercivity. Tiwary et al. [15] showed that the mechanochemical process can produce a partial transformation into the hexaferrite phase using a planetary mill for 50 h followed by annealing at

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900 °C for 1 h, using ores as precursors (celestite and blue dust). Other authors like Ketov et al. [16] studied the effect of mechanochemical treatment and subsequent annealing of $SrFe_{12}O_{19}$ powders. They concluded that milling caused a deterioration of the magnetic properties but the annealing process led to a sharp improvement in them due to crystallization and formation of the $SrFe_{12}O_{19}$ phase with fine crystallites. The best properties achieved for the milled hexaferrite ($\mu_0 H_{ci} \approx 0.42$ T, $B_r \approx 0.24$ T and $(BH)_{max} \approx 9.6$ kJ/m^3) were reached after milling and annealing the powder at 950–1000 °C for 1 h. In this case, the average particle size was about 1 μm and the average crystallite size was 100–200 nm. Sort et al. [17] prepared hexaferrites with high coercivity by using pure $SrFe_{12}O_{19}$ as a precursor milled with FeS, which promoted the presence of Fe_2O_3 in the final product. All the hexaferrites obtained by means of mechanical activation showed coercivities in the 3100–5700 Oe range, magnetic remanences about 2350 G and BH_{max} values from 1.2 to 1.85 MGOe, higher than those of the phases obtained by other methods [3–13].

Although there are a great number of interesting works in this subject, in all of them, the milling times used to synthesize hexaferrite are higher than 30 h, promoting iron contamination from the milling media which affects to the magnetics properties, as will be demonstrated in this paper. Besides, the starting materials are subjected to calcination as pretreatment and the annealing temperatures to assist the synthesis are higher than 900 °C, increasing the processing cost.

In this work, it is shown that mechanosynthesis for only 5 h of milling time of a mixture of commercial oxides containing hydroxides and carbonates, without a pretreatment of calcination, followed by annealing at temperatures as low as 700 °C, induces the complete formation of nanostructured strontium hexaferrites without iron contamination. The obtained hexaferrite has adequate magnetic properties to be applied as a permanent magnetic material. Also, the effects of synthesis parameters (milling time and temperature of annealing) on the magnetic properties are reported, finding an interesting improvement in the coercivity of the material. We use an extrapolation of magnetization data, based on the Stoner–Wohlfarth model, to estimate the saturation magnetization of samples.

2. Experimental procedure

Fe_2O_3 (Sigma Aldrich, 99% purity) and SrO (Sigma Aldrich, 99.9% purity) powders were used as precursor materials. These powders were mixed in a stoichiometric ratio according to the following equation:

$$SrO + 6Fe_2O_3 \rightarrow SrFe_{12}O_{19} \quad (1)$$

For some samples, as we detail below, a SrO excess up to 22% in weight. was introduced in the starting mixture of reagents, in order to compensate the partial hydration or carbonation of SrO in the air atmosphere.

A total of 5 g of the starting mixtures was loaded with steel balls of 1.27 cm diameter into a steel cylindrical vial (50 cm^3 steel/steel, S/S) at room temperature in air and milled for different times from 0 to 12 h. The ball to powder weight ratio was 10:1. To prevent excessive heating of the vials, the experiments were carried out by alternating 90 min of milling followed by 30 min in standby. All experiments were performed in air at room temperature.

The milled powders for different times, also called as-milled, were characterized by X-ray diffraction (XRD) using a Siemens D5000 diffractometer with CoKα$_1$ (λ=1.79003 Å) radiation. Patterns were collected in a 2θ interval of 20–100° with increments of 0.02° (2θ). Rietveld refinement was performed on the X-ray patterns in order to obtain cell parameters. This method takes into account all of the information collected in a pattern, and uses a least squares approach method to refine the theoretical line profile until it matches the measured profile [18].

The annealing temperature of the milled powder was estimated by studying the thermal behavior in a differential scanning calorimeter (TGA/SDTA 851e Mettler-Toledo). The temperatures of the phase transformation were obtained from SDTA curves. The experiments were performed at a heating rate of 10 K min^{-1} using a pure argon flow of 70×10^{-2} m^3 s^{-1}. Magnetic susceptibility and magnetization studies were carried out at room temperature using a Microsense EV7 vibrating sample magnetometer with a maximum field of ± 18 kOe. The as milled powders were annealed in a tube furnace at temperatures from 700 to 1000 °C for 2 h in air atmosphere. These powders were also characterized by XRD and VSM.

3. Results and discussion

Fig. 1 shows the X-ray diffraction (XRD) patterns of stoichiometric mixtures of precursors after different milling times, from 0 to 12 h. XRD pattern, corresponding to the mixture of powders at 0 h of milling time (precursors), shows peaks of starting materials: Fe_2O_3 (ICSD 22505 *R3cH*), $SrCO_3$ (ICSD 202793, *Pmcn*), and $Sr(OH)_2H_2O$ (ICSD 63016

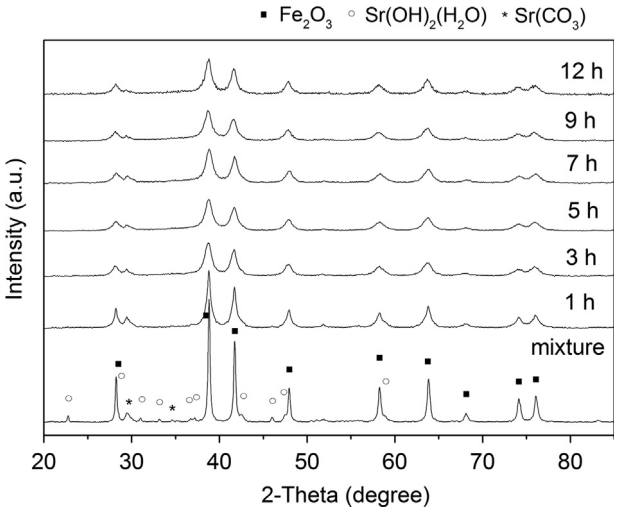

Fig. 1. X-ray powder diffraction patterns of precursor's mixtures mixture milled for different times, from 0 to 9 h.

Pmc21). Although pure SrO was used as the starting material, an unexpected mixture of strontium carbonate and hydroxide was detected. It can be attributed to the high reactivity of SrO; this material reacts with the oxygen and humidity of the atmosphere to produce strontium carbonate and hydroxide [19]. Therefore, the amount of oxides calculated was not stoichiometric and an excess of strontium oxide (SrO) is necessary to complete the reaction.

As can be observed in Fig. 1, after milling all the X-ray diffraction patterns present a general peak profile broadening and a diminution in the peak's intensity as compared to the precursors before milling, probably as a consequence of crystallite size reduction and lattice strain promoted by the milling process, generating a distortion and accumulation of defects in the crystal structure and an increase in internal stress (energy). The formation of strontium hexaferrite, $SrFe_{12}O_{19}$ (ICSD 16158 P63mmc), does not occur during the milling process, as shown by the absence of the corresponding $SrFe_{12}O_{19}$ diffraction peaks in the X-ray diffraction pattern at the end of the process. On the other hand, the λ-Fe_2O_3 diffraction peaks remained unchanged during the whole milling process, which indicates that it did not promote the synthesis of the hexaferrite. After 1 h of milling, the reflection peaks corresponding to $Sr(OH)_2H_2O$ have vanished while other peaks remain constant. For periods larger than 3 h of milling, no difference was observed in the intensity, linewidth, position or number of peaks.

At this point we can conclude that these experimental conditions and the mechanical process do not provide enough energy to complete the reaction (Eq. 1) corresponding to the formation of strontium hexaferrite, so an annealing process is needed to achieve the reaction. We decided to use the lowest temperature to avoid particle growth and at the same time, to apply the treatment for a short milling time to prevent iron contamination [20]. In order to know the adequate annealing temperature for the sample milled for 5 h, differential thermal analysis (TG/DTA) was carried out. Results are shown in Fig. 2. In the curve corresponding to DTA of the powders milled for 5 h (Fig. 2), it can be appreciated that a broad exothermic peak appears after 710 °C. This event can be associated to the formation of hexaferrite from the precursors. Sharma et al. [14] observed this DTA peak in the mechanosynthesis of barium hexaferrite at temperatures higher than 834 °C. Based on the DTA results, we decided to perform an annealing of the milled powders at temperatures in the range of 700– 1000 °C. Besides, in the TGA section of the same Fig. 2, a weight loss occurs between 600 °C and ~675 °C, due to the $SrCO_3$ and $Sr(OH)_2(H_2O)$ decomposition into SrO, H_2O and CO_2. This result is in good agreement with the XRD patterns shown in Fig. 1.

The XRD patterns of samples milled for 5 h and subsequently annealed at 700, 900 and 1000 °C are shown in Fig. 3. As can be observed, in all the annealed samples a mixture of

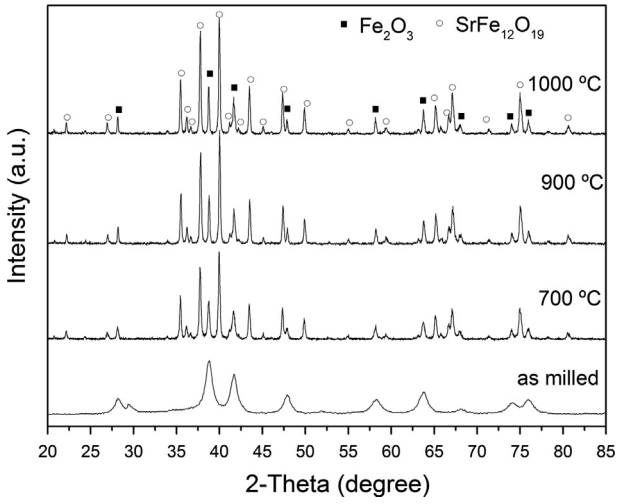

Fig. 3. X-ray diffraction patterns of precursor's mixtures milled for 5 h and annealed at different temperatures.

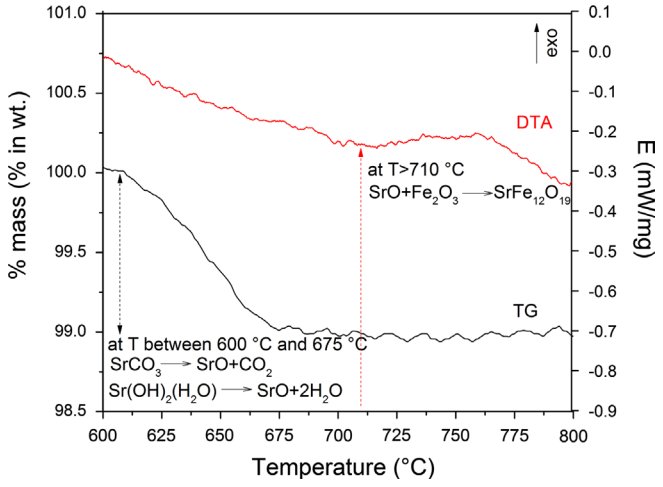

Fig. 2. TGA/SDTA of a mixture of elemental powder: Fe_2O_3+SrO milled for 5 h.

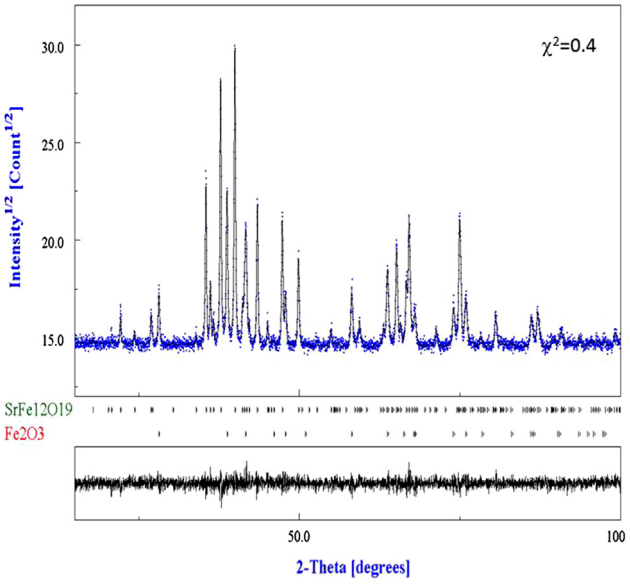

Fig. 4. Rietveld refinement from X-ray diffraction patterns of precursor's mixtures milled for 5 h and annealed at 700 °C.

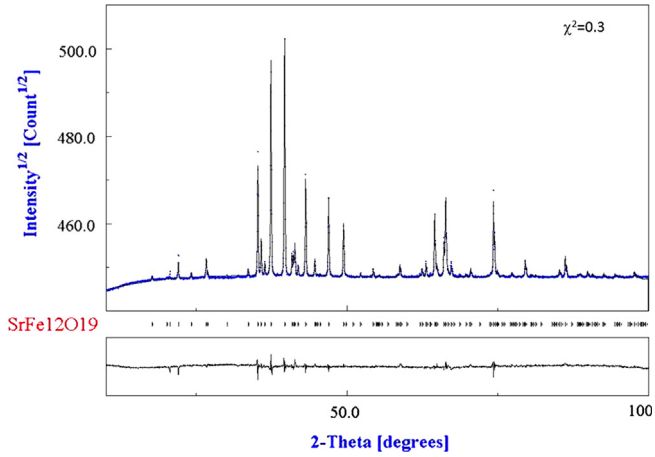

Fig. 5. Rietveld refinement of the X-ray diffraction patterns of precursor's mixtures, with SrO in excess, milled for 5 h and annealed at 700 °C.

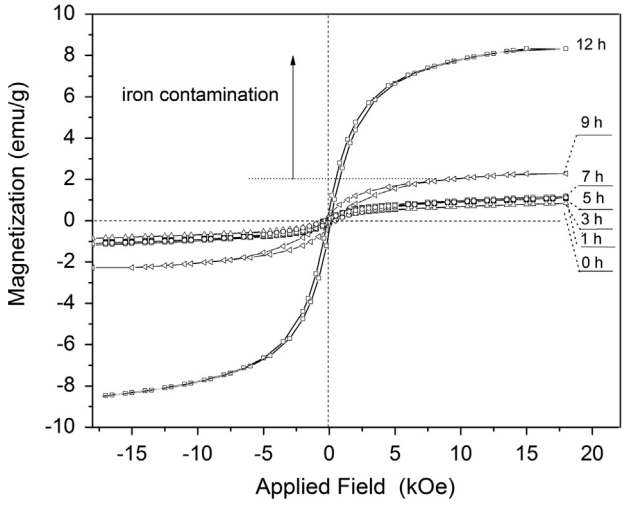

Fig. 6. Magnetic hysteresis loops for precursor's mixtures milled for different times, from 0 to 12 h.

diffraction peaks belonging to the strontium hexaferrite ($SrFe_{12}O_{19}$) and hematite (Fe_2O_3) is present. Comparison with the as-milled mixture of oxides shows a remarkable narrowing of the peaks, due to the crystallization of the milled powders. For the milled sample annealed at 700 °C, a mixture of Fe_2O_3 (PDF2 #99-101-2092) and $SrFe_{12}O_{19}$ (PDF2 #43260) was found. The amount (% in weight) of these oxides was 22% and 78% correspondingly, in accordance with a Rietveld refinement shown in Fig. 4. In Fig. 3 it can also be observed that when increasing the temperature of the heat treatment the relative amounts of Fe_2O_3 and $SrFe_{12}O_{19}$ remain constant, confirming a complete reaction at 700 °C; however, changes in crystallite size, crystallite, defects, etc., are expected [21].

According to the results of Rietveld refinement shown in Fig. 4 about the presence of hematite in the synthesized powder, in order to compensate the partial hydration or carbonation of SrO in the air atmosphere, a mixture of starting oxides plus an excess of SrO (22% in weight. to complete the reaction with the remaining Fe_2O_3) was milled for 5 h and subsequently annealed at 700 °C. The XRD pattern and the Rietveld refinement corresponding to this mixture are shown in Fig. 5. As can be observed in Fig. 5, in these experimental conditions the diffraction peaks belonging to starting oxides, Fe_2O_3 and compounds of strontium, have vanished; only peaks corresponding to the strontium hexaferrite are present, showing that using these experimental conditions a pure strontium hexaferrite without Fe_2O_3 is obtained. The amount of hexaferrite obtained through the Rietveld refinement was 100% in weight.

Fig. 6 shows hysteresis loops for precursors' mixtures as a function of milling time (with hematite as the second phase). The initial mixture (milling time $=0$ h) shows a very small value of magnetization ($\sim$1 emu/g) associated with the magnetic behavior of α-Fe_2O_3 which is a weak ferromagnetic material at room temperature, with $M\sim0.8$ emu/g at 18 kOe [22]. The effects of SrO can be neglected, as it is diamagnetic. Increasing the milling time up to 7 h has a negligible effect on the magnetization, since the hexaferrite phase has not been produced. The value of magnetization of the sample increases from $\sim$1.8 emu/g to $\sim$8 emu/g after 9 h and 12 h of milling time, respectively. As XRD results had shown (Fig. 1), this increase in magnetization is not related with any hexaferrite formation; it is associated with the presence of metallic iron into the mixture as a contaminant generated by the mechanical process. The saturation magnetization of pure iron is 218 emu/g [23], therefore the contamination by iron for these samples can be estimated (by assuming a simple additive effect of magnetization) as 0.5% and 3.5% in weight for 9 h and 12 h of milling time, respectively. Magnetization measurements afford a simple way to estimate iron contamination in this case. On the other hand, these results are in very good agreement with XRD patterns, where only the α-Fe_2O_3 phase is observed for samples milled for times higher than 9 h.

In Fig. 7 is shows the hysteresis loops for the sample milled with an excess of SrO, in order to achieve a stoichiometric ratio, milled for 5 h and subsequently annealed at a temperature of 700 °C (square spots curve). This sample presents only hexaferrite, as was confirmed by XRD and Rietveld refinement shown in Fig. 5. This material exhibits a radically different magnetic behavior as compared with those in Fig. 6. It shows a strong magnetization ($M_s\sim60$ emu/g at 18 kOe) with a coercivity of $\sim$5.2 kOe, very close to typical values reported for nanostructured Sr ferrites [23]. The hysteresis loops showed a considerable slope at high fields, indicating that the saturation value of magnetization could be reached only by means of higher applied fields.

In the same Fig. 7, the hysteresis loops obtained for the sample of a mixture in non-stoichiometric ratio, milled for 5 h and annealed at 700 °C, is also shown (black spots curve). As expected, the magnetization decreases by about 12 emu/g (60 emu/g), when compared with the stoichiometric sample shown in the same figure. This is due to the fact that the hexaferrite is diluted by hematite (Fe_2O_3). An additional effect of the dilution is that the coercive field exhibits an increment, reaching 6.6 kOe. As hexaferrite grains are separated by a non-ferromagnetic or weakly ferromagnetic phase, the possibility of magnetic flux continuity decreases and isolated grains

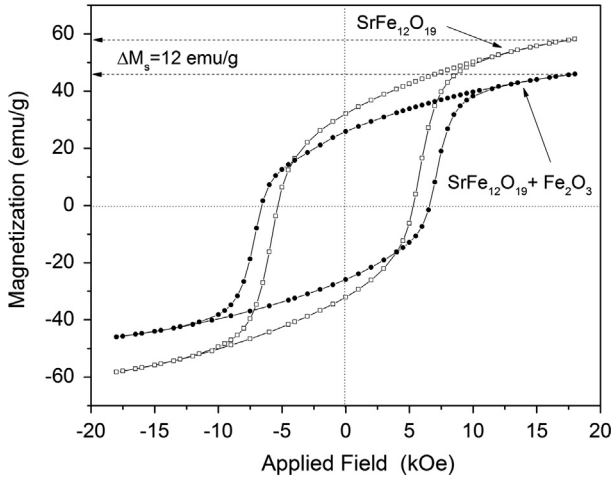

Fig. 7. Hysteresis loops for precursor's mixtures with and without SrO in excess milled for 5 h and annealed at 700 °C.

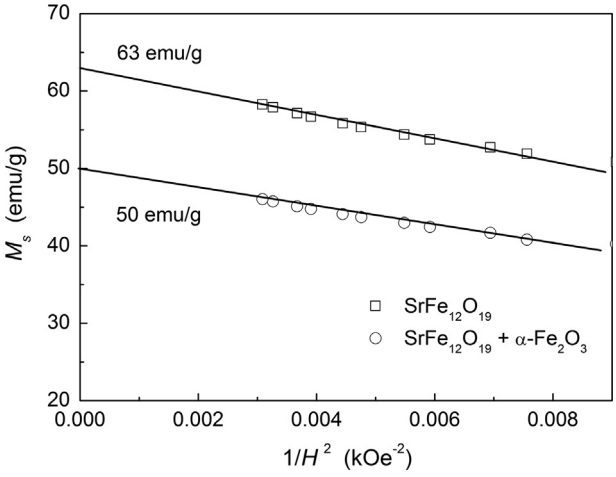

Fig. 8. Plot of magnetization as a function of $1/H^2$ corresponding to precursor's mixtures with and without SrO in excess milled for 5 h and annealed at 700 °C, showing a linear relationship.

approach the single domain behavior. This effect has also been observed in composite ferrite–titanate materials [24] (cobalt ferrite and barium titanate), where cobalt ferrite exhibited a higher coercive field in the composite than as a single phase.

By using an approximation to saturation derived from the Stoner–Wohlfarth (SW) model, it is possible to estimate the value M_s. This approximation leads to the expression [25]

$$M(H) = M_s\left[1 - H_a{}^2 \sin{}^2 2\theta_0 / 8H^2\right] \quad (2)$$

where H_a is the anisotropy field, and θ_0 is the angle between the applied field and the anisotropy axis. The SW model is intended for non-interacting, single domain particles with uniaxial anisotropy and should lead to a linear relationship on plotting the measured magnetization at high fields as a function of $1/H^2$. This is shown in Fig. 8, for both the stoichiometric and the Fe_2O_3 excess samples, with quite an acceptable agreement. The M_s observed values are ~63 and 50 emu/g for the stoichiometric and the Fe_2O_3 excess samples, respectively. This agreement could also point to the single domain nature of the obtained material, following the Stoner–Wohlfarth model. As a consequence, an excess of hematite remains in the final product. In this work it was demonstrated that the presence of Fe_2O_3 together with hexaferrite in the final product decreases the magnetization slightly but increases the coercivity.

These results confirm that mechanosynthesis is a convenient method to prepare nanostructured strontium hexaferrite, by lowering the sintering temperatures as compared to those usually needed in the solid state technique.

As discussed in the previous section, a common problem in the mechanosynthesis of hexaferrites, when starting from SrO, is that this reagent is extremely hygroscopic and can acquire a high content of water, carbon and other compounds during weighting. We measured the hysteresis loops of these samples, milled for 5 h in the high energy ball mill and subjected to several annealing temperatures, from 700 to 1000 °C, without pretreatment of the precursors (calcination); the results are presented in Fig. 9. As was demonstrated in XRD pattern of these samples showed previously in Fig. 3, in these experimental conditions (non-stoichiometric ratio), the presence of hematite is confirmed. For this reason, as can be observed in Fig. 9, the magnetization shows a lower value as compared with hematite-free sample, simply because of a dilution effect, since hematite is a weak ferromagnetic material. On the other hand, an increment in coercive field appears which can be explained by the same dilution effect. In spite of the presence of hematite as a second phase together with the hexaferrite, the annealing treatments have only a small effect on the hysteresis loops. Magnetization increases slightly with annealing temperature, owing to a more complete progression of the reaction. The hematite content, estimated from Rietveld calculations, is about 22%. The hysteresis loop for the sample

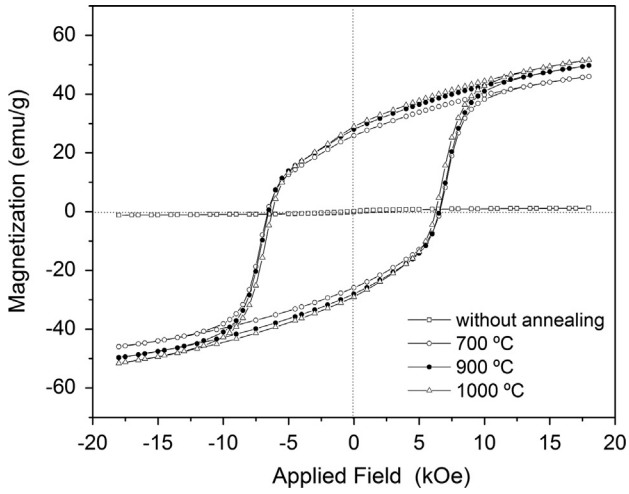

Fig. 9. Hysteresis loops of precursor's mixtures annealed at temperatures in the range 700–1000 °C. The hysteresis loop of the sample after milling (5 h) and before annealing is also shown.

after milling 5 h without thermal treatment is also shown in Fig. 9, for comparison purposes only.

4. Conclusions

Strontium hexaferrite has been successfully prepared by short time milling (5 h) using untreated commercial oxides by mechanosynthesis followed by an annealing at a low temperature (700 °C). The magnetic properties obtained are in agreement with permanent magnetic values of nanostructured $SrFe_{12}O_{19}$ synthesized by different methods. It has been shown that a Fe_2O_3 excess in the initial mixture of reagents, due to the high reactivity of SrO, leads to an enhancement of the coercive field and a slight decrease of magnetization. An estimation of the saturation magnetization based on the Stoner–Wohlfarth model exhibited a good agreement with the model. The combination of XRD analyses and magnetic hysteresis loops allowed distinguishing between an increment in magnetization due to the formation of the hexaferrite and the effect in magnetization as a consequence of iron contamination due to extended times of milling. The magnetic results show that if milling times equal or lower than 5 h are used for the mechanical activation of the precursor's mixtures, iron is not present in the sample, therefore the mechanical process does not affect the magnetic properties.

Acknowledgments

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