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Synthesis of $Y_3Fe_5O_{12}$ (YIG) assisted by high-energy ball milling

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Abstract

Yttrium iron garnet, $Y_3Fe_5O_{12}$ (YIG) powders were synthesized by mechanochemical processing (MCP) from Fe_2O_3 and Y_2O_3, followed by an annealing. The aim of this work was to demonstrate that MCP followed by annealing at relative low temperatures can induce the formation of nanostructured YIG. The effect of synthesis process on the final magnetic properties was also studied. The precursors mixed in a stoichiometric ratio to obtain YIG were milled at room temperature in a shaker mixer mill with a ball:powder weight ratio of 10:1. In order to achieve a single-phase of nanostructured YIG a short thermal annealing at temperatures from 700 to 1100 °C was done. X-ray diffraction (XRD) and scanning electron microscopy (SEM) were used to characterize the synthesized powders. The milling process promotes the formation of a perovskite phase (orthoferrite $YFeO_3$) independent from the milling time; garnet could only be obtained after an annealing process. The partial formation of the garnet phase was observed in mixtures milled for 5 h. In order to obtain a pure YIG, it is necessary to do a post-treatment of an annealing at temperatures of 900 °C, around 400 °C lower than those used to prepare the material by solid state reaction. Also, the effect of synthesis method into the magnetic behavior of the garnet was shown.

Keywords: Mechanochemistry; YIG; Annealing; Iron garnet; Mechanochemical processing

1. Introduction

Ferromagnetic garnets have attracted much attention as microwave device materials and as a magneto-optical recording medium as well as for their unique magnetic and magneto-optical properties [1–4]. In order to improve their magnetic properties, some authors are trying to dope the YIG with different cations such as bismuth [5,6], lanthanum [7,8], cerium [9], titanium [10], gadolinium [9,11] and other rare earths.

Garnets are characterized by a compact oxygen array [7] and are assigned to space group Ia3d $8(O_h^{10})$ where the cations are located at the center of corresponding oxygen polyhedra [5,12]. Due to the ability to exchange positions of the cations in the cell, these materials are the basis for many high-technology devices in telecommunications. They are also the basis for many devices working in the microwave range, as they show an extremely small linewidth [13] in ferromagnetic resonance processes. The fundamental magnetic properties of YIG originate from the magnetic ions and their relationship to the surrounding oxygen ions.

Yttrium ferrite garnet can be synthesized by several methods. The most conventional and oldest one is the sintering of the corresponding oxides in a furnace at 1400 °C (solid state reaction) [6]. This process generates large particles and consumes a great amount of energy [14]. It also produces an intermediate phase $YFeO_3$ undesirable for the same magnetic applications. Iron garnet powders can be produced by wet chemical methods such as sol–gel [1,9,14], coprecipitation [15], pulsed laser ablation [16], plasma spraying [17], citrate gel process [3] etc. A particular method is mechanochemical synthesis which promotes the formation of a new oxide by mechanical activation of a precursor's oxides or salts [18] and can lead to a change in the distribution of cations in interstitial sites so magnetic properties are affected [19]. Mechanosynthesis of yttrium iron garnet with subsequent heat treatments

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using temperatures of annealing higher than 900 °C have been reported in the literature. Paesano et al. [20] reported the use of mechanosynthesis to obtain YIG from a mixture of Y_2O_3 and Fe_2O_3 with a subsequent annealing at 1000 °C. Widatallah et al. [21] presented a detailed study of the influence of the milling process on the formation of single phase YIG by mechanochemical synthesis, using similar experimental conditions to Paesano [20]. Niyaifar Ramani et al. [5,6] also used high energy milling to mechanically activate the mixture BiO–Y_2O_3–Fe_2O_3 and then obtained doped Bi-YIG by a subsequent annealing. All these authors magnetically characterized the obtained powders.

In mechanochemical synthesis experiments involving metallic oxides in air, a reduction of iron oxides phase has been observed to occur in closed stainless steel containers after prolonged milling time. The reaction occurs in a steady-state manner during milling [19–25]. High-energy ball milling has demonstrated to be a technique useful to promote the synthesis of nanostructured ferrites by mechanical activation of oxide compounds and it has shown excellent results [25–28]; however according to some authors [21,29,30], this technique does not lead to the direct synthesis of YIG, and it is necessary to apply an annealing at high temperatures in order to promote the diffusion and complete the reaction. Opposed to these results, there are other authors [31] who state that MCP can promote the complete synthesis of YIG, in contrast with others who [29,30] showed that mechanical milling leads to the decomposition of YIG into $YFeO_3$, orthoferrite, Fe_2O_3 and Y_2O_3, the opposite process. The reason for the instability of the garnet phase is not yet understood and more work is being done in this direction. In this work, it is shown that MCP followed by annealing at relatively low temperatures (400 °C lower than the reported) can induce the complete formation of nanostructured YIG. Also, the effect of synthesis parameters on the magnetic properties of the garnet is reported and compared with the same materials synthesized by other methods.

2. Experimental procedure

Fe_2O_3 (Sigma Aldrich, 99% purity) and Y_2O_3 (Sigma–Aldrich, 99.9% purity) powders were used as precursor materials. These powders were mixed in a stoichiometric ratio according to the following equation:

$$3Y_2O_3 + 5Fe_2O_3 + 0.0547O_2 \rightarrow 2Y_3Fe_5O_{12+\delta} \tag{1}$$

The oxygen excess (0.0547 mol) was calculated from the air contained in the milling vial that was used in the process. A total of 5 g of the starting mixtures were loaded with steel balls of 1.27 cm in diameter into a cylindrical steel vial (50 cm^3) (steel/steel, S/S) in air at room temperature and milled for 9 h. The ball to powder weight ratio was 10:1 according to previous studies [25]. 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.

The milled powders were characterized by X-ray diffraction (XRD) using a Siemens D5000 diffractometer with $CoK\alpha_1$ ($\lambda = 1.7889$ Å) radiation. Patterns were collected in a 2θ interval of 20–120° with increments of 0.02 (2θ). Rietveld refinement was performed on the X-ray patterns. This method takes into account all of the information collected in a pattern, and it uses a least squares approach method to refine the theoretical line profile until it matches the measured profile. The lattice parameters of the powder were obtained from the XRD line positions using a refinement method [32].

The morphologies of the milling powders were analyzed using a JEOL JSM-6300 scanning electron microscope, working at 15 kV. The stability of the synthesized powder was measured by studying the thermal behavior in a differential scanning calorimeter (TGA/SDTA 851^e Mettler-Toledo). The temperatures of the phase transformation were estimated from SDTA curves. The experiments were performed under a heating rate of 10 K min^{-1} using a pure argon flow of 666×10^{-3} m^3 s^{-1}. Magnetic susceptibility and magnetization studies were performed at room temperature using a Microsense EV7 vibrating sample magnetometer with a maximum field of 18 kOe.

3. Results and discussion

Fig. 1 shows the X-ray diffraction (XRD) patterns of mixtures after different milling times, from 0 to 9 h. As expected, the XRD pattern corresponding to 0 h shows the peaks of the starting oxides: Fe_2O_3 (ICDS # 43465) and Y_2O_3 (ICDS # 78581). After 5 h of milling, the reflection peaks of Y_2O_3 have disappeared and those of α-Fe_2O_3 broaden and their intensities decrease implying a crystallite size reduction.

It can be observed that the formation of yttrium ferrite with perovskite structure ($YFeO_3$ ICDS 43260, orthoferrite) starts after 1 h of milling, and it is completed after 5 h. This is shown by the presence of the peaks corresponding to this phase at this time together with the absence of Y_2O_3 peaks. The presence of Fe_2O_3 (ICDS # 00-033-0664) is due to the excess of reactant introduced into the vials to obtain garnet structure YIG. The disappearance of peaks attributable to Y_2O_3 in the early stages of the milling process implies that it has a faster fragmentation rate relative to Fe_2O_3. This is suggestive that the un-reacted

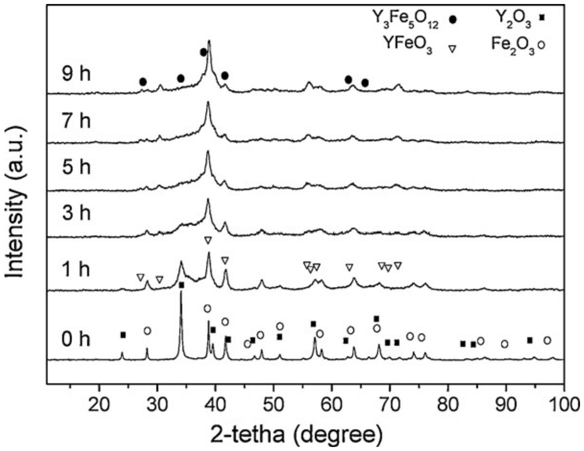

Fig. 1. X-ray powder diffraction patterns of $Fe_2O_3 + Y_2O_3$ mixture milled at different times, from 0 to 9 h.

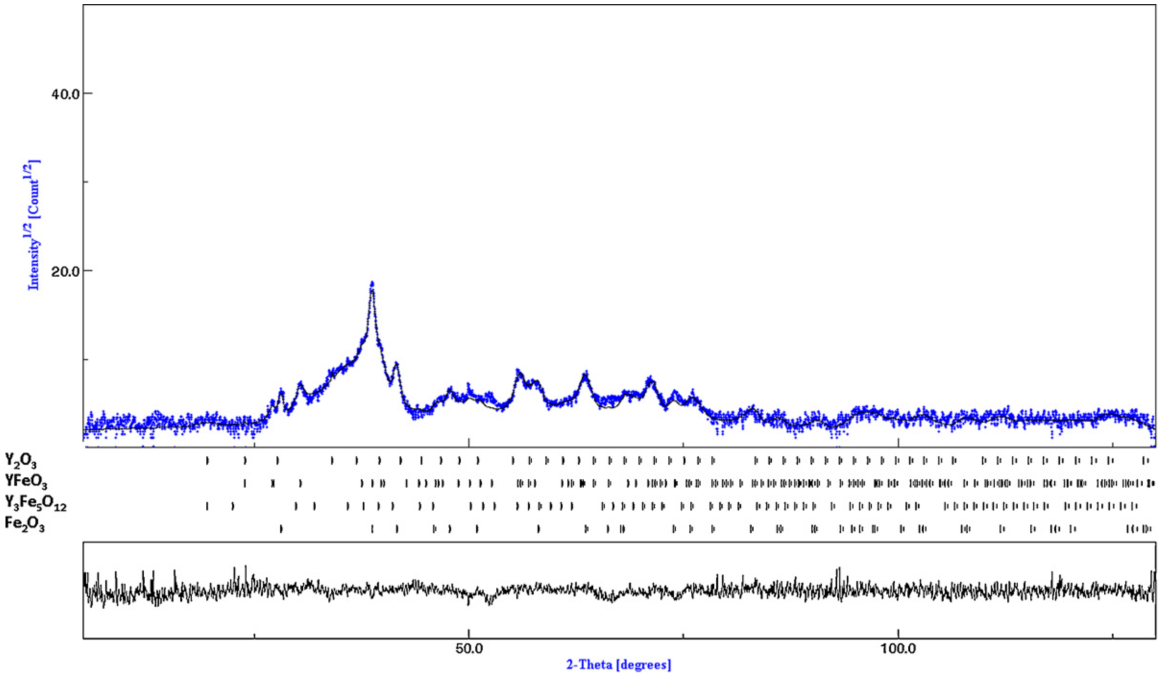

Fig. 2. Rietveld refinement of the mixture of oxides ($Y_2O_3 + Fe_2O_3$) after 5 h of milling.

Y_2O_3 in the mixture forms thin coating layers around the α-Fe_2O_3 nanoparticles that are undetectable by XRD. This is consistent with the work of Widatallah et al. [21]

For milling times longer than 5 h, a remarkable peak profile broadening was noticed probably as a consequence of crystallite size reduction and lattice strain promoted by the milling process. It is possible to confirm that these peaks belong to YIG (ICDS # 2012) by means of a Rietveld refinement, showed in Fig. 2. These results suggest that the perovskite structure is easier to form than the garnet structure using mechanosynthesis which is presented as a transient phase before the garnet phase is finally formed [20]. These results are opposite to the conclusions for studies conducted at equilibrium conditions [28]. It is confirmed therefore that the structural factor is the most important variable in the mechanosynthesis process [29] as observed in these results.

Besides, the quantitative accumulated microstrain (rms) into $YFeO_3$ crystalline structure during the mechanosynthesis process was calculated using Rietveld refinement; the results are presented in Fig. 3. As expected, the microstrain increases with the milling time due to the effect of mechanical milling on the internal energy of the unit cell, showing an increase from 0.00 at 0 h to 0.04 at 3 h of milling time, when microstrain reaches its maximum value. After this time, microstrain remains in an equilibrium value. This increase in microstrain in the early stages, as result of an accumulation of internal energy, allows the complete formation of orthoferrite. The observed behavior confirms the X-ray diffraction patterns showed in Fig. 1, in which only orthoferrite diffraction peaks can be observed after 5 h of milling. After this time, microstrain in $YFeO_3$ crystal is held, possibly because mechanical energy of the process is used to crystallite size reduction and at the same time, to promote the formation of YIG, which partially appears after 5 h of milling. According with these results, after 3 h of milling, the internal energy of the system increases, either as surface energy or strain, so this energy could promote a diminution in the temperature necessary to synthesize YIG assisted by thermal treatment.

The presented results demonstrate that it is possible to partially obtain YIG by mechanochemical process. However, it seems that the impact between the powders and the milled medium for 9 h (maximum time tested) does not provide enough energy to complete the reaction to obtain yttrium iron garnet. This is probably due to the fact that the YIG crystal structure has a larger unit cell and is denser than $YFeO_3$.

Fig. 4 shows the Gibbs free energy results for garnet and perovskite structures, starting from the same precursor oxides: $Y_2O_3 + Fe_2O_3$. In this figure the chemical feasibility for the

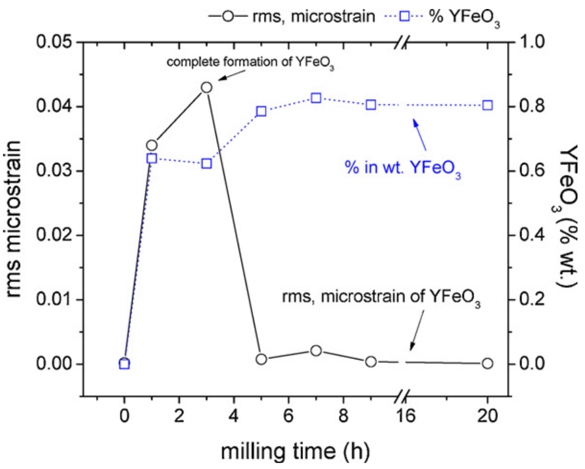

Fig. 3. Microstrain (rms) and % in wt. of $YFeO_3$ during the mechanosynthesis process.

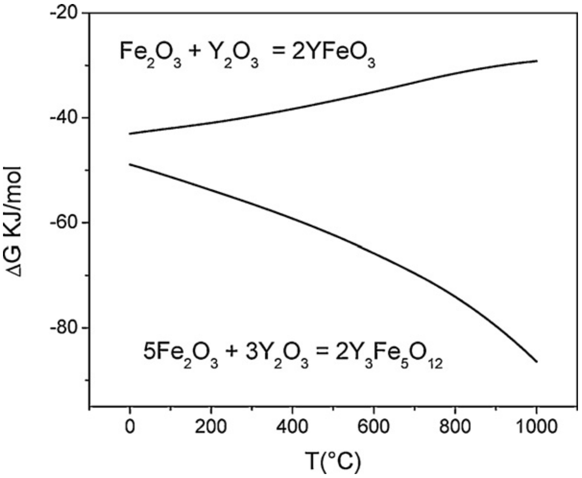

Fig. 4. Gibbs free energy calculation for orthoferrite and garnet formation from iron and yttrium oxides mixtures with the YIG stoichiometry.

garnet formation is notable; its ΔG decreases with temperature in equilibrium conditions. These results are in clear contrast with the X-ray diffraction results of Fig. 1. This fact can be explained by considering the high energy mechanical activation where powders are impacted between them and the deformation of the crystallites tending to form smaller structures. The unit cells for both structures are shown in Fig. 5; while the unit cell for $YFeO_3$ has 16 atoms, the unit cell for garnet $Y_3Fe_5O_{12}$ contains 160 atoms.

MCP is a method which promotes the synthesis of structures out of equilibrium conditions in ceramic materials but the experimentally obtained results demonstrate that MCP cannot induce the complete formation of YIG, possibly due to structural aspects. According to other authors [20,21,29,30], YIG cannot be obtained by mechanosynthesis: they suggest that it is necessary to promote the complete synthesis by means of an annealing process. In order to determine the adequate temperature of annealing, a differential thermal analysis (DTA) of the mixture milled for 5 h was carried out. The obtained results are presented in Fig. 6.

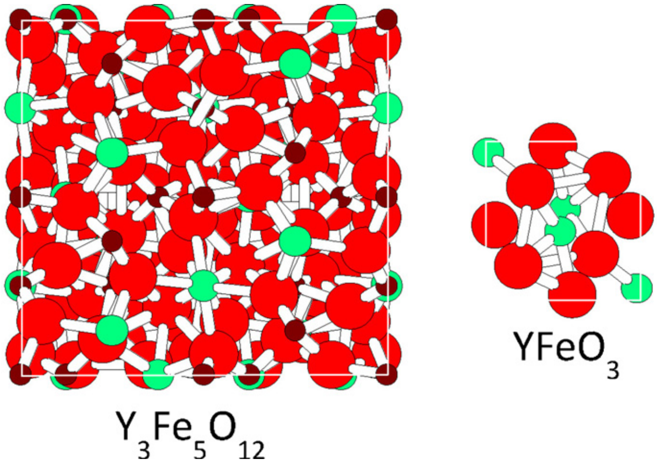

Fig. 5. Unit cell of garnet ($Y_3Fe_5O_{12}$) and perovskite ($YFeO_3$) phases.

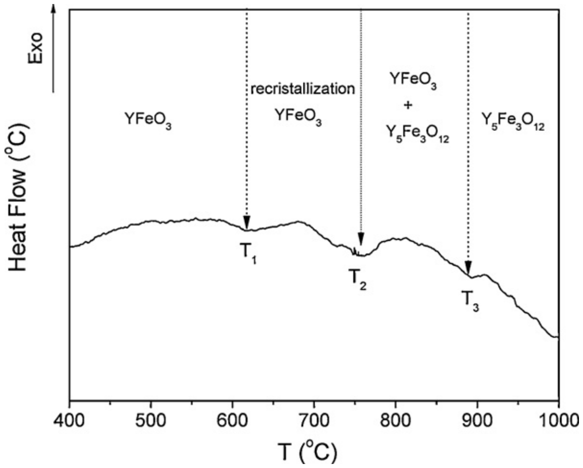

Fig. 6. DTA of mixture of elemental powder: $Fe_2O_3 + Y_2O_3$ milled for 5 h.

As can be observed in Fig. 6, three broad exothermal peaks appears within the temperature range from 400 °C to 1000 °C, which can be associated with structural changes of the orthoferrite structure (obtained as milled, Fig. 1). The first broad peak (at 615 °C) can be associated to a recrystallization of orthoferrite obtained by mechanosynthesis. The second peak (at 752 °C) defines the beginning of a garnet structure formation and the last (around 890 °C) is related to the complete disappearance of the orthoferrite and complete formation of garnet.

In order to confirm the structural changes of the powder milled 5 h, it was annealed at temperatures where structural changes were found according with DTA studies (Fig. 6): 700, 800, 900 and 1000 °C; the results are showed in Fig. 7.

The X-ray diffraction patterns for sample as milled and annealed at different temperatures confirm the assumptions made from thermal analysis. It can be observed in Fig. 7 that at 800 °C a mixture of perovskite and garnet structures persists, and at 900 °C a complete formation of garnet structure is achieved. After this temperature, the garnet structure is the

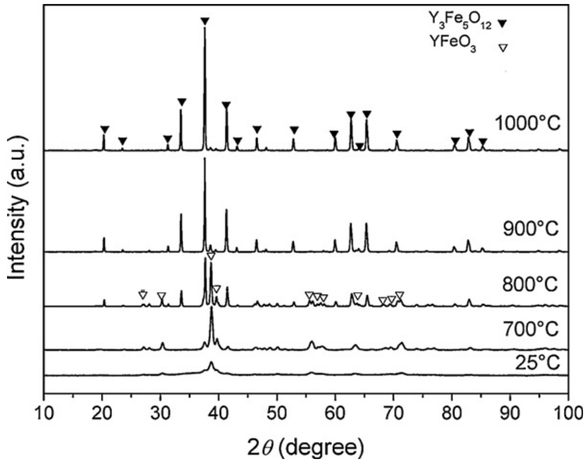

Fig. 7. X-ray diffraction patterns of milled powder ($Fe_2O_3 + Y_2O_3$) for 5 h and annealed at different temperatures (from 700 to 1000 °C) for 3 h.

only existing phase. This temperature represents a considerable reduction in comparison with sintering temperatures of the samples used in the conventional ceramic method ($T > 1400$ °C) [1]. This statement can be confirmed by Rietveld refinement, as is shown in Fig. 8, where it appears a refinement of the samples heat treated at 800 °C and 900 °C respectively, for 3 h. In Fig. 8a, it can be observed the presence of a mixture of YIG and precursors when the sample is heat treated at 800 °C while in Fig. 8b, corresponding to the sample heat treated at 900 °C, only YIG can be detected. This confirms the complete reaction at 900 °C of heat treatment.

Representative SEM micrographs of the powder mixtures as milled and annealed at different temperatures are shown in Fig. 9. The micrographs of the power as milled (Fig. 9a and b) showed that the particles had less than 1000 nm in diameter with nearly uniform size distribution and also contain some agglomeration. An irregular and rounded morphology is observed. The powder becomes rounder and bigger as

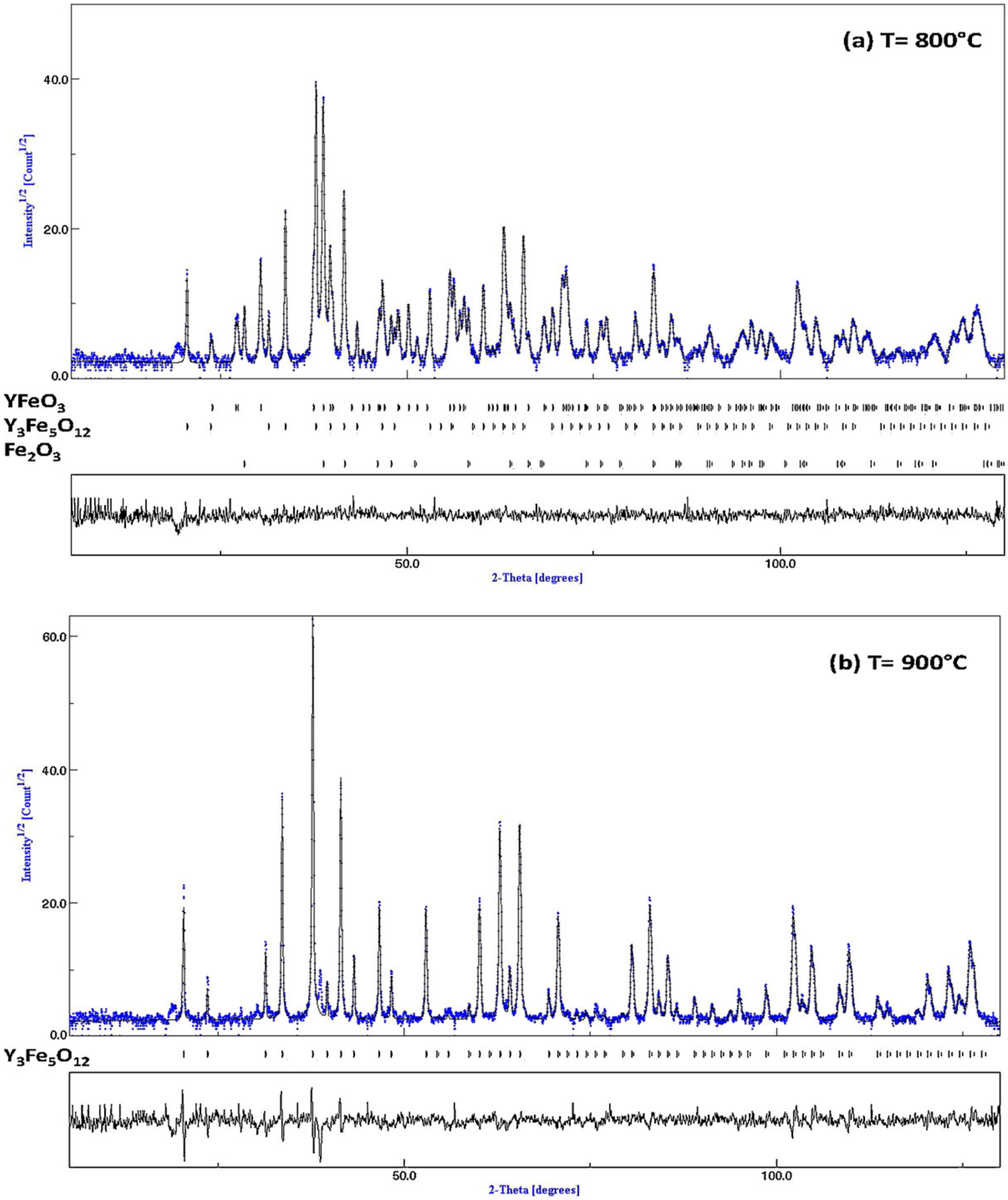

Fig. 8. Rietveld refinement of the mixture of oxides ($Y_2O_3 + Fe_2O_3$) after 5 h of milling and annealed at (a) 800 °C and (b) 900 °C for 3 h. The figure shows the experimental (points) and refined pattern (continuous line) and the difference curve (lower curve). Ticks indicate the Bragg reflexions.

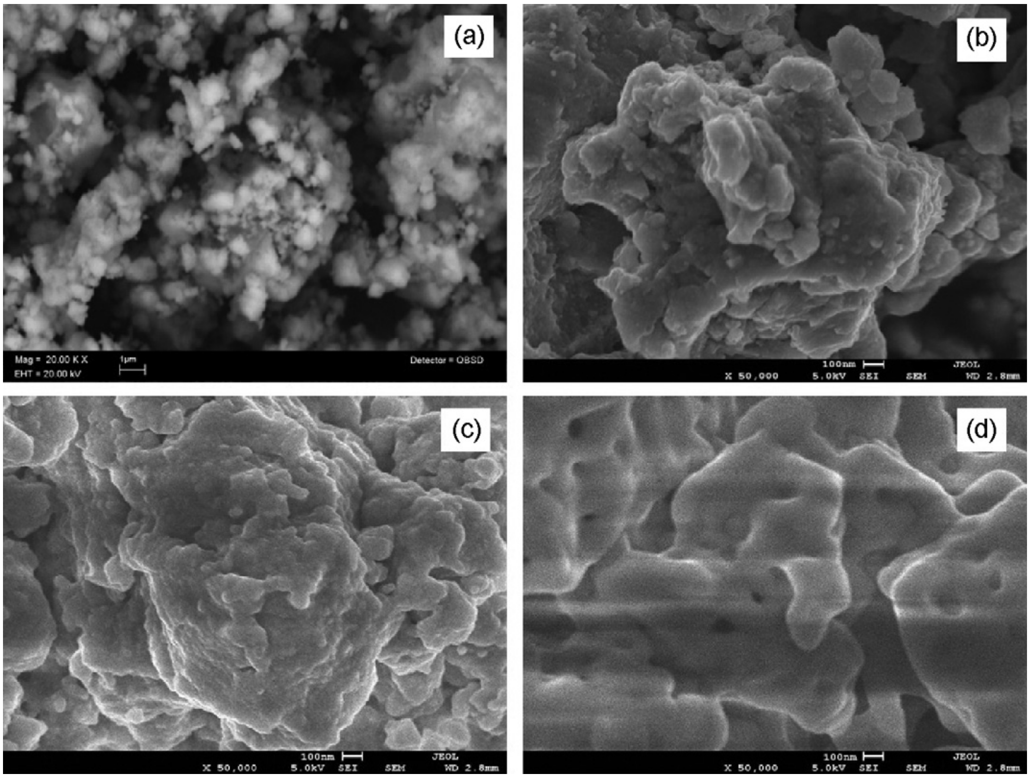

Fig. 9. SEM micrographs of milled powder ($Fe_2O_3 + Y_2O_3$) for 5 h and annealed at different temperatures: (a and b) without annealing, (c) at 800 °C and (d) at 900 °C.

temperature of annealing increases as can be observed in Fig. 9c, and finally close to roughly spherical particles with a smooth surface at 900 °C were obtained (Fig. 9c), as a consequence of an enhanced diffusion process.

Finally, Fig. 10 shows the hysteresis loops for powder mixture milled for 5 h and then annealed at the same temperatures. The milled sample shows an antiferromagnetic order in good agreement with X-ray diffraction results, where the dominant phase is $YFeO_3$ with a perovskite structure (ABO_3-type). In this structure, the B magnetic sites are occupied by the same element leading to a superexchange interaction, and therefore to an antiparallel arrangement of spins. As the transformation from orthoferrite ($YFeO_3$) to YIG progresses, the hysteresis loops exhibit an increasing value of saturation magnetization (M_s) and a coercive field in the 100 Oe range. For annealing temperatures of 900 and 1000 °C, M_s attains the reported value for YIG single phase (27.4 emu/g), in agreement with similar materials synthesized by other methods [3,8,15,25].

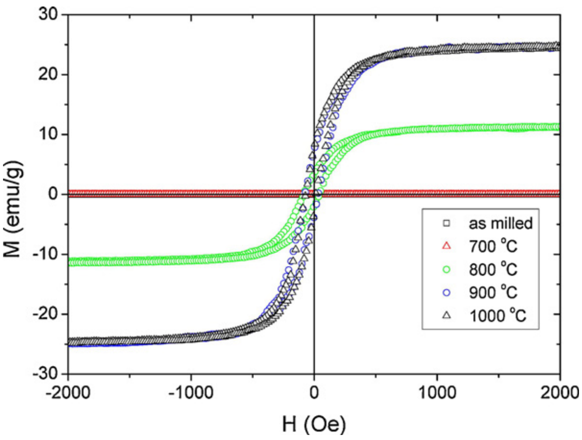

Fig. 10. Magnetization hysteresis loops for different annealing temperatures.

4. Conclusions

$Y_3Fe_5O_{12}$ (YIG) was successfully obtained by high energy ball milling (5 h) and subsequent annealing at 900 °C for 3 h. Results of ball milling set out the feasibility to obtain yttrium orthoferrite instead of yttrium iron garnet, opposite to thermodynamic expectations. Milled powders can be subsequently annealed at relatively low temperature (900 °C, which involves a relatively low energy) to complete the structural transformation from orthoferrite to YIG, as confirmed by structural characterization. Finally, the highest saturation magnetization was obtained for YIG, the lowest for orthoferrite and mixtures of YIG and orthoferrite phases showed an intermediate value.

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