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Study of the structural and magnetic properties of metallic iron-hematite particles for use in magnetorheological fluids

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Abstract Magnetorheological (MR) fluids are new iron-based materials, whose applications include brakes, dampers, clutches, shock absorbers systems and polishing of optical surfaces (lens and mirrors). They are dependent on the size and shape of particles as the magnetic properties. Interested in the possibility of using iron-rich powders, commonly used in nondestructive testing, ranging in size from a few μm to about 200 μm and lower cost than those commercially used for MR fluids, a study of the structural and magnetic properties of iron-rich metallic particles by X-ray diffraction (XRD) and Mössbauer spectroscopy (MS) at room temperature has been done. Powders, as received, were separated into particle sizes smaller than 20 μm (sample A) and in the range of 20–38 μm (sample B) because these are the sizes generally required for applications in MR fluids. The particles whose sizes exceed the above values were ground in a high energy planetary mill for 3 h, using different values of rotational speed/time: 200 rpm for one hour, a pause of 10 s, 140 rpm for one hour, pause 10 s and then 175 rpm during the last hour. These powders were sieved to obtain particles smaller than 20 μm (sample C). According XRD results, in all samples, only α-Fe (lattice parameter a = 2,867(2) Å) and Fe_2O_3 (lattice parameter a = 5,037(1) Å and c = 13,755(8) Å) were present. The Mössbauer spectra were fitted with two sextets. The hyperfine parameters values allowed us to assign the highest relative area spectrum (sextet) corresponding to α-Fe and the second one to Fe_2O_3 in accord to the XRD results. Thus, the preparation method using mechanical milling for diminishing the size of the metallic particles allowed us to get particles with size and magnetic properties that could lead to potentially MR fluids applications.

Keywords X ray diffraction · Mössbauer spectroscopy · Magnetorheological fluid

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1 Introduction

Magnetorheological (MR) fluids are iron based new materials. MR fluids are considered smart materials because the rheological property (viscosity) can be altered or changed by applying a magnetic field in order to perform a controlled function. According Kciuka and Turczyn [1], MR fluids consist of suspensions of micron sized (3 to 5 μm) magnetizable particles suspended in an adequate carrier liquid (mineral oil, synthetic oil, water or ethylene glycol). Additional stabilizers and surfactants are included to prevent gravitational settling, enhance lubricity and change initial viscosity. O'Grady et al. [2] state the feasibility of the use of particles with sizes in a wider range (1–100 μm) without appreciable loss of magnetic response, because in micrometric magnetic particles there are multidomains that under the action of a magnetic field can increase the net magnetic moment of the particles. Thus, a deep knowledge of particle sizes and their magnetic properties is mandatory when some kind of magnetic particles is intend to be used in MR fluid projects. In this work, a study of structural and magnetic properties of metallic iron-hematite particles, commonly used in non-destructive testing, whose sizes vary from a few microns to 200 μm, was performed by X-ray diffraction (XRD) and Mössbauer Spectroscopy (MS) at room temperature.

2 Experimental

Magnetic particles (Magnavis 8A Red, main sizes 180 μm) which are coloured by red pigments [2], commonly used in non-destructive testing, were selected to be used in this study in order to establish their viability for the preparation of MR fluids. Powders, as received, were separated by size particles scale less than 20 μm (sample A) and in the range from 20 μm to 38 μm (sample B), because these sizes are generally referred for their applications [3]. The particles that exceeded these were ground in a high energy planetary mill for around 3 h, using different rotation speed/time values: 200 rpm and one hour, 10 s pause, 140 rpm one hour, 10 s pause and 175 rpm in the last hour. The jars and balls are made of hardened stainless steel with a relation sample mass to balls mass of 1 to 15 using 30 g of powders. In the milling process atmospheric air was used. This powder was sieved in order to separate it in the same previous sizes ranges. The sample of ground and sieved powders with sizes less than 20 μm, was named sample C. The samples A and B present a red coloration but C sample exhibits a gray-brown colour. All Mössbauer spectra were obtained with the aid of a homemade autonomous Mössbauer spectrometer [4] operating in constant acceleration mode using a LND proportional counter (Xe-CO_2, 1520 Torr) with a 57Co (Rh) source at RT. Spectra were fitted with MOSFIT program and the isomer shifts are referred to α-Fe. The X-ray patterns of the samples were performed at RT using the Cu/Kα radiation (PanAnalytical Diffractmeter). To obtain the lattice parameters and phase proportion it was used the GSAS program, which consider Rietveld analysis to refine de XRD patterns [5]. The main crystallite size in the parallel ($\phi||$) and perpendicular ($\phi\perp$) directions to the incident X-rays were calculated too.

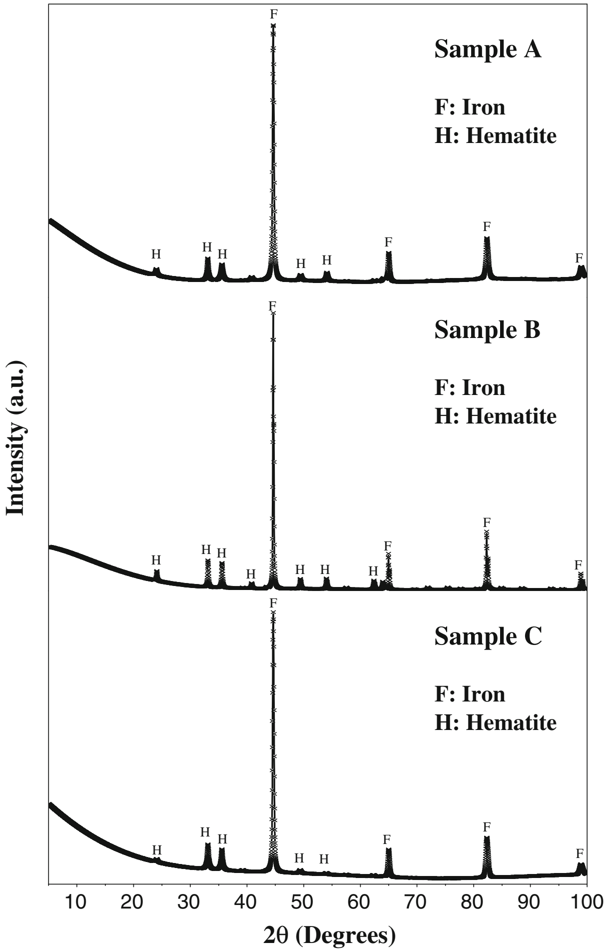

Fig. 1 XRD patterns of samples A, B, C. Note the peaks corresponding to metallic iron (F) and hematite (H) are the only ones present

3 Results and discussion

3.1 XRD

Figure 1 shows the X ray patterns of the samples according powder particles sizes: A (less than 20 μm), B (20 to 38 μm), and C (less than 20 μm after grinding and sieving). It is apparent that only the α-Fe (F) and Fe_2O_3 (H) peaks are present. According Rietveld fitting results for the mentioned samples, the lattice parameters correspond to α-Fe (a = 2,867(2) Å) and Fe_2O_3 (a = 5,037(1) Å and c = 13,755(8) Å). The weight fractions were 76.0% and 24.0%, (sample A), 68.7% and 31.3% (sample B), 68.5% and 31.5% (sample C) for α-Fe and Fe_2O_3, respectively. This clearly shows that the grinding process of particles with sizes larger than 38 μm can produce particles (sample C) with the same phases as those found in A and B, although apparently

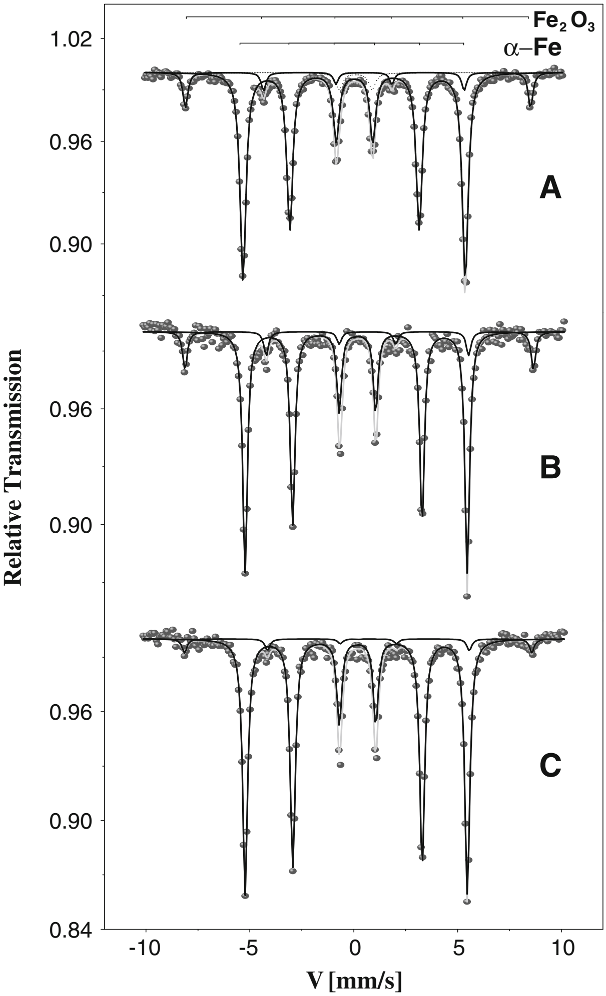

Fig. 2 Room temperature Mössbauer spectra. The hyperfine parameters correspond to metallic iron (α-Fe) and hematite (Fe_2O_3)

favors the increase of the Fe_2O_3 component compared with the same phase in sample A, where A and C are samples with the same range in particle size. This difference in the proportion of Fe_2O_3 is probably due to its recognized high fragility (an oxide of iron) compared to α-Fe [6]. There is a significant reduction in the crystallite size of the sample (C) compared with sample (A): a) from $\phi\perp = 77{,}7$ to 55,1 nm and $\phi|| = 71{,}0$ to 56,2 nm, for α-Fe, and b) from $\phi\perp = 79{,}3$ to 22,1 nm and $\phi|| = 84{,}9$ to 22,3 nm for Fe_2O_3. According the ratio $\phi_{(C)}/\phi_{(A)}$, there is a crystallite size reduction to ∼2/3 in α-Fe and to ∼1/3 in Fe_2O_3. It is characteristic of the grinding process that there is a decrease in particle size due to material fracture which is accompanied by a reduction in crystal size and a distortion in the crystalline lattice [7].

3.2 Mössbauer

Figure 2 shows the Mössbauer spectra obtained for the different samples A, B and C. All spectra were fitted with two sextets. The values of their Mössbauer parameters correspond to the metallic iron (α-Fe) and hematite (Fe_2O_3). More precisely, in samples A and B, the relative areas are very similar, 90% and 10% for α-Fe and Fe_2O_3, respectively. In sample C the relative area of α-Fe increases to 96% with the corresponding decreasing in Fe_2O_3 relative area (4%). This effect could be related to removing of Fe_2O_3 of the surface of particles by milling, that is expected because the high fragility of iron oxides. This is consistent with the color variation experienced by the sample C compared to the original samples A and B. Thus, it is clear the Fe_2O_3 is on the surface of particles. It is not clear why is observed a decreasing in the relative area of Fe_2O_3 meanwhile in XRD results, the weight fraction (%) varies from 24.0% (A) to 32.4% (C). In summary, the results reveal that our milling process reduces the size of particles but do not induce changes in the structure or magnetic properties (hyperfine field) of the constituent particles [8] and occurs the removing of Fe_2O_3 from the particles surface.

4 Conclusions

XRD allowed us to determine two phases present in each sample, corresponding to α-Fe and Fe_2O_3, and quantify the weight fraction of them and the variation of the crystallite sizes. There were also identified by MS both α-Fe and Fe_2O_3, magnetic phases and their relative area determined, showing an increase of the α-Fe relative area in the ground sample, because removing of pigment, associated to Fe_2O_3 from the particles surface after grinding. Thus, the preparation method using mechanical milling for diminishing the size of the metallic particles allowed us to get particles with size and magnetic properties that could lead to potentially MR applications.

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