Structural and magnetic properties of barium hexaferrite nanostructured particles prepared by the combustion method

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Received 21 March 1995; revised 8 June 1995

Abstract

The combustion method, a fast and simple way of preparing sub-micrometer sized particles from a solution of the corresponding metal nitrates and a reducing agent (ODH, TFTA) which is used as a fuel, was adapted to the synthesis of barium hexaferrite particles. Structural and magnetic properties were investigated by X-ray diffraction, transmission electronic microscopy, magnetic measurements and Mössbauer spectrometry on nanostructured as well as on microstructured particles resulting from annealing treatments under different conditions. High values of the coercive field (5.3 kOe) and of the magnetization (57.8 emu/g), at 13.5 kOe, were obtained on well crystallized BaFe_{12}O_{19} particles annealed at 850°C.

1. Introduction

Hexagonal magnetic hard ferrites such as BaFe_{12}O_{19} are currently magnetic materials with great scientific and technological interest, because of their relatively strong anisotropy and moderated, but still interesting magnetization. The production of barium hexaferrite permanent magnets, their application in microwave devices, or the use of hexaferrite fine particles in perpendicular magnetic recording, represent areas in which barium hexaferrite plays an important role.

In the case of fine particles (~ 0.1 μm), one can expect peculiar magnetic properties compared with those observed for sintered materials [1]. On the one hand, the value of the coercive field increases whereas the magnetic losses decrease, and on the other, the values of the saturation magnetization and coercive fields are observed to be lower than those expected for this type of particles. Sometimes this behaviour can be explained through surface effects [2-4], particle size and morphology [3,5,6], impurities [7], or crystallographic defects [4,7,8]. In any case, the magnetic properties of these materials depend largely on the microstructure, and the microstructure depends on the preparation method. Recently, a wide range of chemical methods have been used to obtain ultrafine mixed oxide particles, all of which require a low-temperature process in order to control the growth of the particles as a first synthesis stage. Some of these methods are: the organometallic precursor method [9], the pyrosol method [4], chemical coprecipitation [7], sol–gel [10], mechanical milling [11], etc. However, a number of difficulties in obtaining particles of small and homogeneous sizes with high chemical purity as well as in a crystallized state, have been pointed out in most
papers reporting the synthetic methods developed up
to now.

In the present paper we report the structural and
magnetic properties of BaFe₁₂O₁₉ nanostructured
particles synthesized using the combustion method
[12]. This method was successfully applied to the
production of mixed oxides with spinel, garnet and
orthoferrite structures [13–15] as well as high-tem-
perature superconductors [16]. In this process, we
make use of the great exothermicity of the redox
reaction between metal nitrates oxidizing agents and
tetraformal trisazine (TFTA, C₄H₁₂N₆), or oxalic
acid dihydrazide (ODH, C₂H₆N₄O₂) , reducing
agents, to generate, in a short time, sub-micrometer
sized particles (<< 0.1 μm), which after an anneal-
ing process result in BaFe₁₂O₁₉ microstructured par-
ticles.

In order to study the structural and magnetic
properties of these particles, we have used X-ray
powder diffraction and Mössbauer spectrometry
 techniques which provide information on the lattice
parameters, the presence of impurity phases, the
different iron phases, the distribution of the iron ions
in the lattices, as well as the hyperfine parameters.

2. Sample preparation

The corresponding metal nitrates and adequate
quantities of reducing agents were dissolved in the
minimum amount of water required for their solu-
tion. Tetraformal trisazine (TFTA, C₄H₁₂N₆), was
prepared by means of the reaction of formaldehyde
and hydrazine hydrate at 0°C [17]. The stoichio-
metric compositions of the reducing agent—metal
nitrate mixtures were calculated taking into account
the concepts of propellant chemistry [18], the equiva-
ence ratio φₑ, and the reducing and oxidizing ele-
ment valences. Let us remark that in this context, C,
H, Ba and Fe are the reducing species with valences
of + 4, + 1, + 2 and + 3, respectively, whereas O is
the oxidizing species with a valence of −2; the
valence of nitrogen is taken as zero.

Series of samples were prepared with each reduc-
ing agent. The proportion of reducing agent was
varied along each series in order to check its influ-
ence on the size and purity of the particles. The ratio
of metal nitrates to ODH (or TFTA) reducing agent
was: Ba(NO₃)₂:Fe(NO₃)₃·9H₂O: ODH (TFTA)=
1:12:19 (6.8) and 1:12:38 (13.6) and the quantities
used are shown in Table 1.

The mixture containing the metal nitrates and the
reducing agent was heated to boiling with frothing
and foaming. At the point of complete dehydration,
the foam ignites (sometimes it even flames) at a
temperature of approximately 300°C for 5 min or
less. The combustion residue is ash-like, foamy and
brown. It is concluded that the porosity of the ash
increases with the quantity of reducing agent, whereas
the density decreases. After burning, the ash was
annealed at temperatures of 700–1000°C to obtain
barium hexaferrite particles. The results obtained on
the samples are presented in Table 2. They corre-
spend to ash samples annealed at 700, 750, 850 and
1000°C for 100 h in cumulative or independent
treatments.

The combustion method has the advantage of
permitting the production of barium hexaferrite pre-
cursor materials in a quick and simple manner, with-
out any special setup or complicated chemical reac-

<table>
<thead>
<tr>
<th>Sample</th>
<th>Amount of reducing agent (g)</th>
<th>Molar proportion nitrates: reducing agent</th>
<th>φₑ</th>
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<td>ODH</td>
<td></td>
<td></td>
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<tr>
<td>A</td>
<td>3.466</td>
<td>1:4.146</td>
<td>1</td>
</tr>
<tr>
<td>B</td>
<td>6.932</td>
<td>2:2.92</td>
<td>0.68</td>
</tr>
<tr>
<td>C</td>
<td>6.932</td>
<td>2:2.92</td>
<td>0.68</td>
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<td>TFTA</td>
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<tr>
<td>X</td>
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<td>0:52</td>
<td>1</td>
</tr>
<tr>
<td>Y</td>
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<td>1:1.05</td>
<td>0.55</td>
</tr>
<tr>
<td>Z</td>
<td>3.088</td>
<td>1:1.05</td>
<td>0.55</td>
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Table 2

<table>
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<th>Sample</th>
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<td>X1, Y1, A1, B1</td>
<td>700°C/100 h</td>
</tr>
<tr>
<td>A2, B2</td>
<td>700°C/4 h + 750°C/100 h</td>
</tr>
<tr>
<td>X3, Y3, A3, B3</td>
<td>700°C/4 h + 1000°C/4 h</td>
</tr>
<tr>
<td>C1, Z1</td>
<td>700°C/100 h</td>
</tr>
<tr>
<td>C2, Z2</td>
<td>700°C/100 h + 750°C/100 h</td>
</tr>
<tr>
<td>C3, Z3</td>
<td>700°C/100 h + 750°C/100 h + 850°C/100 h</td>
</tr>
<tr>
<td>C4, Z4</td>
<td>700°C/100 h + 1000°C/100 h</td>
</tr>
</tbody>
</table>
tions, in contrast with some of the other methods mentioned above.

3. Experimental results

The samples were characterized by X-ray powder diffraction on a Philips PW1710 diffractometer using Cu Kα radiation. All patterns were taken at room temperature, in the step-scan mode spanning an angular range of 110° in 2θ (from 10° to 120°) in steps of 0.04° 2θ, and a counting time of 16–29 s per step. The sizes and shapes of the particles were analyzed by transmission electron microscopy (TEM) on a Philips CM12 microscope operated at 100 kV.

57Fe absorption Mössbauer spectroscopy was carried out using a conventional spectrometer operating in the constant acceleration mode and a 57Co source diffused in a Rh matrix. Previous Mössbauer studies on crystalline BaFe12O19 have revealed rather complex spectra, because the hyperfine structure results from the presence of five kinds of iron sites. As the highest resolution is observed at room temperature, the Mössbauer spectra were systematically performed on all the samples at room temperature. The hyperfine data were refined with the Mosfit program.

The values of the chemical isomer shift are reported relative to metallic α-Fe at room temperature.

The magnetic properties were measured with a vibrating sample magnetometer VSM operating in magnetic fields between 0 ≤ H ≤ 13.5 kOe in the temperature range 77 K ≤ T ≤ 1100 K.

3.1. X-ray and TEM results

The ash resulting from the combustion, which was studied by X-ray diffraction, is a mixture of iron oxides and barium nitrate particles. Broad Bragg peaks were evidenced and the broadening was attributed to the small particle size and to the lack of crystallinity of the samples. When the ash is annealed at 700°C for 2 h, an important amount of BaFe12O19 phase occurs: the presence of very well-defined Bragg peaks indicates a good crystalline state of the samples (Fig. 1). We then performed a Rietveld refinement [19] of the X-ray powder diffraction patterns of the samples using the Fullprof [20] and ARIT4 [21] programs. This refinement using the P63/mmc space group confirms the well established hexagonal phase of BaFe12O19 (Fig. 2). The lattice parameters, which are in good agreement with the more common published data [22], remain indepen-

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![Fig. 1. X-ray diffractograms of sample C after different thermal treatments.](image-url)
dent of the annealing treatment of the samples. Also, for the as-prepared sample, we note the presence of two additional compounds, identified as hematite \( \alpha-\text{Fe}_2\text{O}_3 \) and barium monoferrite \( \text{BaFe}_2\text{O}_4 \) phase impurities induced during the synthesis. These impurities represent less than 10% in all the samples, except for the sample prepared with the smallest amount of reducing agent \( (X1) \), which has 50% of these impurities. After annealing at 750°C, the minor phase was reduced to less than 5% in all samples, and after annealing at 850°C it was reduced to less than 1%.

The electron transmission micrographs in Fig. 3 (sample C3) clearly show nanostructured particles for the low-temperature annealed samples (the average diameter is estimated at 70 nm). The size of the particles increases slightly with annealing temperature: for treatments at 750, 850 and 1000°C, the average diameters are 0.1, 0.3 and 0.5 \( \mu \text{m} \), respectively (see Table 3).

The as-prepared particles are agglomerated due to sintering. They are platelet-shaped, more spherical for low annealing temperatures and with a tendency towards a hexagonal shape for higher annealing temperatures.

### 3.2. Magnetic results and discussion

The Mössbauer spectra (see Fig. 4) were fitted assuming the presence of five iron components \( (12k, 4f_1, 4f_2, 2a \text{ and } 2b) \), in agreement with the assignment proposed by Evans et al. [23]. Two additive components corresponding to \( \alpha-\text{Fe}_2\text{O}_3 \), hematite, and \( \text{BaFe}_2\text{O}_4 \), barium monoferrite, were also introduced during the fitting procedure. The contribution of the
hematite phase is not easy to estimate because the values of its hyperfine parameters are very close to those of the 4f2 iron site.

Due to small discrepancies in the relative population of iron in the 12k position, regardless of the sample and of the annealing treatment conditions, we assumed the theoretical value of the population in all samples to be 50% of the amount of iron in BaFe12O19. According to this assumption, we have estimated the relative populations of the other barium hexaferrite iron positions. Thus we have obtained an estimate of the proportion of α-Fe2O3 that agrees quite well with the results provided by X-ray diffraction. The second impurity, BaFe2O4, was not observed (the weight in iron atoms was estimated at less than 0.5%).

The final values of the hyperfine parameters, except for those of the quadrupole shift, were very similar to those obtained on well-characterized and well-crystallized BaFe12O19 samples (prepared from high-purity initial materials) obtained after longer annealing (see Table 4).

The fluctuations observed in the isomer shift and hyperfine field values, corresponding to the 2b iron positions, are explained by the rather small contribution of this position; however, in the cases of the positions with larger populations (4f1 and 12k), the final values of the hyperfine parameters are very similar to those obtained on well-characterized and

<table>
<thead>
<tr>
<th>Sample</th>
<th>Annealing temperature (°C)</th>
<th>BaFe2O4 (%)</th>
<th>α-Fe2O3 (%)</th>
<th>D (μm)</th>
<th>Hc (Oe)</th>
<th>Ms (emu/g)</th>
<th>μB</th>
</tr>
</thead>
<tbody>
<tr>
<td>C1</td>
<td>700</td>
<td>0</td>
<td>2.5</td>
<td>&lt;0.1</td>
<td>5208</td>
<td>54.3</td>
<td>25</td>
</tr>
<tr>
<td>C2</td>
<td>750</td>
<td>0</td>
<td>1.4</td>
<td>0.05–0.15</td>
<td>5256</td>
<td>55.7</td>
<td>27</td>
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<tr>
<td>C3</td>
<td>850</td>
<td>0</td>
<td>0</td>
<td>0.15–0.20</td>
<td>5285</td>
<td>57.8</td>
<td>29</td>
</tr>
<tr>
<td>C4</td>
<td>1000</td>
<td>0</td>
<td>0</td>
<td>0.15–0.50</td>
<td>5145</td>
<td>59.0</td>
<td>34</td>
</tr>
</tbody>
</table>

| Z1     | 700                       | 3.8         | 13.0        | <0.1   | 4709   | 46.8      | 31 |
| Z2     | 750                       | 2.1         | 7.9         | 0.05–0.15 | 4800   | 51.0      | 32 |
| Z3     | 850                       | 0           | 1.6         | 0.15–0.20 | 4893   | 55.6      | 35 |
| Z4     | 1000                      | 0           | 0           | 0.15–0.50 | 4597   | 57.4      | 35 |

| A1     | 700                       | 5.4         | 1.2         | <0.1   | 5379   | 52.8      | 28.7 |
| A2     | 750                       | 5.2         | 1.6         | –      | –      | –         | –   |
| A3     | 1000                      | 0           | 0           | 0.2–0.5 | 5209   | 58.0      | 29.4 |

| B1     | 700                       | 5.0         | 1.1         | <0.1   | 4792   | 47.7      | 22.6 |
| B2     | 750                       | 3.3         | 0.7         | –      | –      | –         | –   |
| B3     | 1000                      | 0           | 0           | 0.2–0.5 | 4895   | 52.6      | 38.2 |

| X1     | 700                       | 25.2        | 10.4        | <0.1   | 4192   | 29.8      | 13  |
| X3     | 1000                      | 0           | 0           | 0.1–0.3 | 3980   | 55.5      | 22.6 |

| Y1     | 700                       | 3.5         | 0.4         | <0.1   | 4692   | 48.6      | 42.4 |
| Y3     | 1000                      | 0           | 0           | 0.1–0.5 | 4679   | 55.4      | 40.3 |

Sintered ceramic | 1100 | 0 | 0 | >2 | 1925 | 57.5 | 29 |
well-crystallized BaFe$_{12}$O$_{19}$ samples synthesized according to the usual ceramic method.

In addition, a small discrepancy in the relative populations for the iron sites can be observed as compared to the expected values: the relative population for the 4f$_1$ and 2a positions is higher than expected, in contrast with the case of the 2b and 12k positions.

First, the relatively large population of the 4f$_1$ position, and secondly, the small populations of the 2b and 12k positions, whose spins are aligned antiparallel and parallel, respectively, to the resulting direction of the magnetization, lead to a lowering of the number of the Bohr magnetons, $\mu_B$, for BaFe$_{12}$O$_{19}$. Even though the high population of the 2b position leads to an increase in the number of Bohr magnetons, it is not sufficient to compensate for this reduction. These features explain (i) the discrepancy with the theoretical Bohr magnetons estimated on the well-crystallized BaFe$_{12}$O$_{19}$, and (ii) the increase in the number of Bohr magnetons for the better crystallized samples, resulting from either larger annealing times or higher annealing temperatures.

Hysteresis loops were recorded between $-13.5 \leq H \leq 13.5$ kOe for the different samples and at different temperatures (see Fig. 5), and the corresponding values of the coercive field, $H_c$, and the magnetization, $M_s$, for 13.5 kOe are given in Table 3 and Fig. 6.

The increase in the magnetization with annealing temperature is attributed to the improved purity and crystallinity of the BaFe$_{12}$O$_{19}$ phase, as defined by X-ray diffraction and Mössbauer spectrometry. The presence of small quantities of $\alpha$-Fe$_2$O$_3$, which is a weak ferromagnet at temperatures below $T_N = 634^\circ$C, or of BaFe$_2$O$_4$, which is antiferromagnetic at temperatures below $T_N = 607^\circ$C, in the hexaferrite samples annealed at low temperatures, gives a slightly reduced magnetization of the samples compared with the magnetization values for the samples without this impurity (Table 3).

Furthermore, the relative populations of the different iron positions, which determine $\mu_B$, will also...
Table 4
Hyperfine parameters at the different iron sites for BaFe$_{12}$O$_{19}$ samples: isomer shift IS (mm/s) given relative to metallic iron at 300 K, quadrupole shift 2\(e\) (mm/s), hyperfine field \(H_M\) (T) and relative area (%) of the subspectra

<table>
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<tr>
<th>Position</th>
<th>Parameter</th>
<th>12k (\pm 01)</th>
<th>(2e) (\pm 01)</th>
<th>(H_M) (\pm 4) (\pm 2)</th>
<th>4f$_1$ (\pm 01)</th>
<th>(2e) (\pm 01)</th>
<th>(H_M) (\pm 4) (\pm 2)</th>
<th>4f$_2$ (\pm 02)</th>
<th>(2e) (\pm 02)</th>
<th>(H_M) (\pm 4) (\pm 3)</th>
<th>2a (\pm 05)</th>
<th>(2e) (\pm 05)</th>
<th>(H_M) (\pm 4) (\pm 2)</th>
<th>2b (\pm 05)</th>
<th>(2e) (\pm 05)</th>
<th>(H_M) (\pm 4) (\pm 2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C1</td>
<td>0.35</td>
<td>0.40</td>
<td>41.5</td>
<td>50.3</td>
<td>0.27</td>
<td>0.26</td>
<td>48.9</td>
<td>21.0</td>
<td>0.43</td>
<td>0.11</td>
<td>51.3</td>
<td>16.3</td>
<td>0.27</td>
<td>-0.02</td>
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<tr>
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<td>0.36</td>
<td>0.40</td>
<td>41.4</td>
<td>50.8</td>
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<td>0.18</td>
<td>48.9</td>
<td>18.3</td>
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<td>-0.03</td>
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determine the resulting magnetization for each sample. If we take a series of samples annealed at different temperatures we can observe how the sign of the variation of the resultant magnetization agrees with that of the variation of $\mu_B$ calculated from Mössbauer spectrometry. Increased annealing time and temperature gives an increase in the magnetization, which leads to an increase of $\mu_B$. This is because an increased diffusion of $\text{Fe}^{3+}$ ions in the lattice is favoured, and the relative population values approach the theoretical ones corresponding to pure barium hexaferrite. This explains why the magnetization values of the small particles are smaller than expected. This has been observed before [8].

The difference between the theoretical and the experimental values of the iron site populations, which is larger in the case of the samples annealed at 700 and 750°C than in those annealed at 850 and 1000°C, can be also attributed to stacking faults of the RSR*S* blocks.

At room temperature, the values of the coercive field $H_c$ remain high, regardless of the annealing treatment conditions ($4.8 < H_c < 5.3$ kOe). These values are similar to those expected for single-domain hexaferrite particles [24], with a tendency to increase with annealing temperature up to 850°C, because the crystallinity and purity of the samples is better. However, at higher temperatures, $H_c$ drops due to the increase in grain size, i.e. to a smaller nucleation field [25–29].

Fig. 7 shows the evolution of the magnetization $M_s$ and that of the coercive field $H_c$ at 13.5 kOe with temperature (between room temperature and the Curie temperature, $T_C$). One can observe the decreasing values of $M_s$ when the temperature increases, up to $T_C$. However, $H_c$ increases with temperature up to 200–300°C and then decreases at higher temperatures. These results can be explained by assuming a single-domain structure around 200–300°C. Kools [28,29] proposed the following expression for the coercivity:

$$H_c = a \cdot H_A - b \cdot M_s,$$

where the first term represents the average internal field needed to nucleate a reversed domain, and the second term corresponds to the effect of internal demagnetization fields. In our case, $H_A$ is nearly independent of $T$ (up to 300°C) and $M_s$ decreases with $T$, implying a positive temperature coefficient for the coercivity, $H_c$.
4. Conclusions

The present results give clear evidence of the potential the combustion method: under optimal conditions and adequate annealing treatment, this process leads to the synthesis of barium hexaferrite nanostructured particles. Furthermore, it confirms the compromise that must be made when obtaining crystalline pure single-domain BaFe$_{12}$O$_{19}$ particles. The magnetic performance of the BaFe$_{12}$O$_{19}$ particles resulting from annealing at approximately 850°C is promising: the main magnetic characteristics (magnetization value of 57.8 emu/g and coercive fields of 5285 Oe, at 13.5 kOe), are attributed to the high purity (< 1% of impurities), good crystalline state, and the small homogeneous size of the particles (the dimensions of platelet-shaped particles are in the range 0.1–0.2 μm).

Acknowledgements

The authors would like to thank Professor J.L. Fourquet and G. Courbion for helpful discussions. One of us (S.C.) thanks the Xunta de Galicia for scholarships (1990–94) and for providing financial support for a stay at the University of Le Mans (France).

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