FMR STUDY OF THE INFLUENCE OF CARBURIZATION LEVELS BY METHANE DECOMPOSITION ON NANOCRYSTALLINE IRON

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Abstract. The methane decomposition on nanocrystalline iron, promoted with small amounts of calcium, aluminum and potassium oxides, was studied. The iron nanocrystallites are bridged by the promoters. The carburization process was carried out in differential tubular reactor with thermogravimetric measurement of mass changes. The carburization was investigated for a reduced iron catalyst with pure methane at temperature 650 °C under atmospheric pressure during several different carburizing levels. The carburized samples contained nanocrystalline forms of iron carbide (Fe₃C), iron and graphite, what was evidenced by XRD measurements. Ferromagnetic resonance (FMR) spectra of Fe₃C and α-Fe nanoparticles have been recorded at room temperature in samples subjected to different carburization levels. The FMR spectra have been analyzed in terms of four components having Callen lineshape and revealed anisotropic magnetic interaction in two types of nanoparticles. The anisotropic resonance fields (parallel and perpendicular) were used to determine the magnitude of the anisotropy fields for both types of nanoparticles while the dipol-dipol interaction between nanoparticles was responsible for the linewidths of the spectral components.

1. INTRODUCTION

A growth of interest in nanomaterials all over the world has been observed in recent years, especially in the area of encapsulated magnetic materials inside an inert matrix like carbon [1]. Chemical properties of nanomaterials could differ substantially from the properties of the relevant bulk substances. Such materials have new, better mechanical properties compared with the traditional ones. One of nanomaterials group which evokes interest through particular magnetic properties are nanocrystalline iron carbides [2]. As a result of catalytic decomposition of hydrocarbons (methane) on nanocrystalline iron the nanocomposites including iron carbide, iron, graphite and carbon nanotubes are obtained [3,4]. The nanocomposites based on iron may find numerous applications, including fillers for polymer composites, magnetic materials for data storage, constructional and tool materials, magnetic inks, ferrofluids and in biotechnology [5-8].

Magnetic properties of strongly correlated spin system of Fe/Fe₂O₃ at high temperatures aroused great interest in both scientific and technological literature in a recent decade and still produces a very large amount of work, e.g. [9-13]. Magnetic nanoparticles embedded in various materials have been widely used in many technological applications including magneto-optical devices, magneto-sensor...
electronics, magneto-optics, catalysis, biomedical and building materials, etc. [14-21]. The study of the magnetic properties of nanoscale particles in different materials is very important because it would allow modification of their properties in such way to make them more functional. The magnetic nanoparticle agglomerates of iron and iron carbide placed in a non-magnetic matrix have shown already very interesting behavior in the ferromagnetic resonance (FMR) studies [21-31]. All three main parameters of the FMR absorption signals (intensity, linewidth and resonance field) were very significantly affected by the concentration and size of nanoparticle agglomerates. A strong magnetic interaction between agglomerates and inside agglomerates has been observed. It was deduced that the ferromagnetic interaction between agglomerates strongly decreased with carbon concentration.

In this paper the FMR study on six samples with different content of magnetic nanoparticle of α-iron and iron carbide in a (non-magnetic) carbon matrix has been presented. The samples have been synthesized by carburization of nanocrystalline iron with methane and characterized by XRD, and TGA methods. The aim of the study is to determine what kind of information about the magnetic interactions between α-iron and iron carbide nanoparticles and aggregates could be inferred from the FMR study of such complicated magnetic system.

2. EXPERIMENTAL

The iron catalyst contained nanocrystalline iron and aluminum, calcium and potassium oxides. Nanocrystalline iron was bridged by the promoters, which protected the iron structure from sintering at elevated temperatures and wetted the nanoparticle surface, forming a mechanically stable structure [31].

Elemental composition of the used catalyst, determined by an atomic emission spectrometer with plasma excitation AES-ICP was the following: Al₂O₃ - 3.3 wt.%, CaO - 2.8 wt.%, K₂O - 0.69 wt.%, oxygen in iron oxide-6.5 wt.%. Specific surface of the catalyst, after reduction of the passive layer, determined by thermal desorption was 9 m²/g. An average crystallite size of iron as determined by X-ray diffraction using the Sherrer’s equation was 32 nm.

Catalytic decomposition of methane on the iron catalyst was performed in a differential tubular reactor with thermogravimetric (TGA) mass measurements. On the platinum basket a monolayer catalyst fraction of 1.2-1.5 mm was placed. To remove the passive oxide layer, a catalyst sample was polythermally reduced with hydrogen under constant load at 15 °C/min up to 650 °C. After the mass of the sample levelled, reduced iron catalyst was carburized by methane at 650 °C to various carburizing degrees expressed by \( \frac{n_C}{n_{Fe}} \), which is the ratio of moles of carbon to moles of iron in the iron - carbon system. The resulting sample was cooled to room temperature in an atmosphere of methane. Six different samples, designated as sample A, B, C, D, E, and F have been prepared. They differ in carburization levels by methane decomposition on nanocrystalline iron. Specific compositional characteristics of these samples are presented in Table 1.
Table 1. Compositional characteristics of the investigated samples: \( n_C/n_{Fe} \) is the ratio of carbon to iron atoms, \( \alpha \) is the degree of conversion (see Eq. (1)), wt is weight percentage of a specific phase.

<table>
<thead>
<tr>
<th>Sample designation</th>
<th>( n_C/n_{Fe} )</th>
<th>( \alpha )</th>
<th>wt(( \alpha )-Fe)[wt.%]</th>
<th>wt(Fe(_3)C)[wt.%]</th>
<th>wt(C)[wt.%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>1.2</td>
<td>0.19</td>
<td>3.26</td>
<td>80.99</td>
<td>15.75</td>
</tr>
<tr>
<td>B</td>
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<td>0.23</td>
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<td>C</td>
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<td>0.27</td>
<td>4.75</td>
<td>51.70</td>
<td>43.55</td>
</tr>
<tr>
<td>D</td>
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<td>51.41</td>
</tr>
<tr>
<td>E</td>
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<td>0.50</td>
<td>8.91</td>
<td>29.52</td>
<td>61.56</td>
</tr>
<tr>
<td>F</td>
<td>29.0</td>
<td>0.84</td>
<td>7.12</td>
<td>7.21</td>
<td>85.66</td>
</tr>
</tbody>
</table>

The ferromagnetic resonance (FMR) measurements were carried out using Bruker E 500 spectrometer working in the X-band \((\nu = 9.45 \text{ GHz})\) of microwave absorption at 100 kHz magnetic field modulation. The investigated samples, containing nanoparticles of \( \alpha \)-iron and iron carbide dispersed in carbon matrix with mass of about 2 mg were placed into quartz tubes of 4 mm in diameter. The FMR studies were performed at room temperature.

3. RESULTS AND DISCUSSION

For obtained nanocrystalline carburized iron samples with different \( n_C/n_{Fe} \) ratio, X-ray diffraction patterns was obtained (Philips X’Pert PRO), which is shown in Fig. 1. It was found that the carburized samples contain nanocrystalline graphite, nanocrystalline iron carbide (Fe\(_3\)C-cementite) and nanocrystalline iron. With increasing carburization degree the intensity of reflections from graphite and iron increases, while from iron carbide decreases. Based on the intensity of the Fe(200) and Fe\(_3\)C(210) reflections the degree of conversion, designated as \( \alpha \), for Fe\(_3\)C to \( \alpha \)-Fe was calculated. The degree of conversion \( \alpha \) is a ratio of intensity of iron reflex divided by the sum of iron intensity and product of iron carbide intensity and coefficient \( \alpha \):  

\[
\alpha = \frac{I_{Fe}}{I_{Fe} + aI_{Fe,C}},
\]

(1)

where

\[
a = \frac{I_{Fe}}{I_{Fe,C}}.
\]

The subscript 0 indicates that the intensities are taken for pure iron to pure iron carbide obtained in the same conditions. Given the degree of carburization the diagram of the phase composition (wt.% mass) for different samples was constructed (see Fig. 2). With increasing carburization degree the increase of the degree of conversion \( \alpha \) is observed.

Strong magnetic materials like \( \alpha \)-Fe and Fe\(_3\)C should be subjected to a special preparatory procedure to increase their magnetic rigidity. Before the FMR measurements have been done samples have been magnetized and demagnetized many times in magnetic fields up to 1.7 T. This process has allowed recording of the same FMR spectrum independent of the direction of the magnetic sweep (downfield or upfield). This is especially important for the low magnetic field part of the FMR spectrum that is rather sensitive to this treatment (for magnetically untreated sample the spectrum shifts to higher magnetic fields in the case of downfield sweep).

Fig. 3 presents the registered FMR spectra of all investigated samples. A very intense, broad and strongly asymmetric absorption line has been recorded. A very similar resonance line, but less intense, has been recorded for small amounts of \( \alpha \)-iron and iron carbide nanoparticles embedded in different diamagnetic matrices [21-30]. As there are two kinds of nanoparticles present in our samples an attempt to fit the registered FMR spectra with two component lines was carried out. It was found that with two components the fitting was very unsatisfactory and more components are needed. The presence of additional two components could be justified by a large magnetic anisotropy of the \( \alpha \)-Fe and Fe\(_3\)C nanoparticle systems.

In case of magnetic nanoparticles in the superparamagnetic phase several different FMR lineshapes have been considered [32]. Taking into account the non-zero absorption at zero magnetic field only the Callen lineshape seems appropriate in our case. The Callen lineshape follows from the Callen equation containing two damping terms [32].
The first damping term coincides with damping term in Landau-Lifshitz equation while the second with the Bloch-Bloembergen one. The following equation for the Callen lineshape is obtained in case of the linear polarization of the microwave field and for a perfect ferromagnet [32]:

$$I(H) \propto \frac{H^2}{(H - H_0)^2 + (|H\Delta + H_0\delta|^2)} \left( \frac{H_0^2 + \Delta_0^2}{H_0^2 + (|H\Delta + H_0\delta|^2)} \right) \Delta_0$$

where $H_0$ is the true resonance field, $\Delta_0$ is the true linewidth connected with relaxation of the Landau-Lifshitz type, and $\delta_0$ a true linewidth connected with relaxation of the Bloch-Bloembergen type.

The registered FMR spectra of our samples (black line) were fitted to the sum of four Callen lines (grey line), two component lines for each nanoparticle type. The result of fitting for all six samples is presented in Fig. 4. In general, almost perfect fitting is achieved. The FMR parameters of two components lines and two different magnetic centres obtained from the fittings are presented in Table 2. $A_{pp}$ is the peak-to-peak amplitude, $\Delta_{pp}$ is the linewidth connected with relaxation of the Landau-Lifshitz type, $\delta_p$ is the linewidth connected with relaxation of the Bloch-Bloembergen type, $I_{int}$ is the integrated intensity. The FMR integrated intensity is calculated as the product of the amplitude and a squared linewidth and is assumed to be correlated with the concentration of nanoparticles. Fig. 5 presents the calculated FMR integrated intensity of $\alpha$-Fe nanoparticles as a function of that phase concentration, while Fig. 6 shows a similar dependence for the cementite component in our samples. For $\alpha$-Fe nanoparticles approximately linear dependence is obtained whereas for Fe$_3$C nanoparticles this function seems to be nonlinear. It should be pointed out that

### Table 2. The fitting of FMR spectra parameters of the investigated samples. $A_{pp}$ is the peak-to-peak amplitude, $\Delta_{pp}$ is the linewidth connected with relaxation of the Landau-Lifshitz type, $\delta_p$ is the linewidth connected with relaxation of the Bloch-Bloembergen type, $I_{int}$ is the integrated intensity.

<table>
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<tr>
<th>Sample</th>
<th>Nanoparticle Component</th>
<th>$A_{pp}$ [a.u.]</th>
<th>$H_0$ [kG]</th>
<th>$\Delta_{pp}$ [kG]</th>
<th>$\delta_p$ [kG]</th>
<th>$I_{int}$ [a.u.]</th>
<th>$I_{int}$ [a.u.]</th>
</tr>
</thead>
<tbody>
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<td>A</td>
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<td>5.5</td>
<td>3.7</td>
<td>19.8</td>
<td>25.9</td>
</tr>
<tr>
<td></td>
<td>(∥)</td>
<td>5.0</td>
<td>1.7</td>
<td>1.1</td>
<td>3.3</td>
<td>6.1</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Fe$_3$C</td>
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<td>7.3</td>
<td>147.0</td>
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</tr>
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<td></td>
<td>(⊥)</td>
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<td>2.1</td>
<td>0.3</td>
<td>4.3</td>
<td>13.2</td>
<td></td>
</tr>
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<td>$\alpha$-Fe (⊥)</td>
<td>4.6</td>
<td>2.5</td>
<td>5.6</td>
<td>3.6</td>
<td>21.7</td>
<td>27.7</td>
</tr>
<tr>
<td></td>
<td>(∥)</td>
<td>5.9</td>
<td>1.8</td>
<td>1.1</td>
<td>3.0</td>
<td>6.0</td>
<td></td>
</tr>
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<td>Fe$_3$C</td>
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<td>73.6</td>
<td>84.8</td>
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<td>4.0</td>
<td>11.2</td>
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<tr>
<td>C</td>
<td>$\alpha$-Fe (⊥)</td>
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<td>5.5</td>
<td>3.4</td>
<td>19.7</td>
<td>26.4</td>
</tr>
<tr>
<td></td>
<td>(∥)</td>
<td>6.7</td>
<td>1.9</td>
<td>1.0</td>
<td>3.0</td>
<td>6.7</td>
<td></td>
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<tr>
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<td>0.1</td>
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<td>11.8</td>
<td></td>
</tr>
<tr>
<td>D</td>
<td>$\alpha$-Fe (⊥)</td>
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<td>6.4</td>
<td>3.2</td>
<td>33.8</td>
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<td>(∥)</td>
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<td>2.4</td>
<td>1.0</td>
<td>3.2</td>
<td>12.4</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Fe$_3$C</td>
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<td>~0</td>
<td>4.6</td>
<td>8.5</td>
<td>26.8</td>
</tr>
<tr>
<td></td>
<td>(⊥)</td>
<td>9.9</td>
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<td>0.1</td>
<td>4.3</td>
<td>18.3</td>
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<tr>
<td>E</td>
<td>$\alpha$-Fe (⊥)</td>
<td>7.8</td>
<td>3.5</td>
<td>6.8</td>
<td>2.8</td>
<td>42.2</td>
<td>51.1</td>
</tr>
<tr>
<td></td>
<td>(∥)</td>
<td>10.7</td>
<td>2.6</td>
<td>1.0</td>
<td>2.7</td>
<td>8.9</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Fe$_3$C</td>
<td>3.0</td>
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<td>0.2</td>
<td>3.6</td>
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<tr>
<td></td>
<td>(⊥)</td>
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<td>3.9</td>
<td>~0</td>
<td>3.8</td>
<td>12.9</td>
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</tr>
<tr>
<td>F</td>
<td>$\alpha$-Fe (⊥)</td>
<td>8.5</td>
<td>3.7</td>
<td>6.5</td>
<td>2.0</td>
<td>36.3</td>
<td>39.9</td>
</tr>
<tr>
<td></td>
<td>(∥)</td>
<td>8.4</td>
<td>2.9</td>
<td>1.2</td>
<td>1.7</td>
<td>3.6</td>
<td></td>
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<tr>
<td></td>
<td>Fe$_3$C</td>
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<td>0.5</td>
<td>1.9</td>
<td>1.0</td>
<td>5.5</td>
</tr>
<tr>
<td></td>
<td>(⊥)</td>
<td>6.6</td>
<td>3.8</td>
<td>~0</td>
<td>2.6</td>
<td>4.5</td>
<td></td>
</tr>
</tbody>
</table>
the range of $\alpha$-Fe concentrations variation is rather narrow (3.9 wt.%) while for the cementite phase almost a whole range of concentrations (7.81 wt.%) is realized.

Two FMR components associated with a particular type of nanoparticles are identified as parallel ($\parallel$) and perpendicular ($\perp$) components, reflecting the orientation of each nanoparticle relative to the external magnetic field. This is only a rough approximation as the shape of the FMR spectrum is concerned because statistical orientation of nanoparticles in the studied samples is expected. The difference of resonance fields in perpendicular
and parallel orientations, \( H_0 = H_0^p - H_0^c \), is a measure of magnetic anisotropy of a specific nanoparticle system. Calculated values of anisotropy field \( H_0 \) for all investigated samples and for \( \alpha\)-Fe and Fe\(_3\)C phases are presented in Table 3. As could be easily noticed the anisotropy field has roughly the same value in all studied samples (for a particular phase) independent on carburization level so an average value can be calculated for \( \alpha\)-Fe and Fe\(_3\)C nanoparticles. It turned out that \( <H_0> \) is 0.78 kG and 2.87 kG for \( \alpha\)-Fe and Fe\(_3\)C, respectively.

The remarkable properties of magnetic nanoparticles are due to their anisotropy. The anisotropy has its origin in the non-spherical shape of the particle (shape anisotropy) or in the magnetocrystalline anisotropy. In the simplest model, the anisotropic nanoparticle can be represented by an ellipsoid of revolution. The resonance field \( H_0 \) depends on the angle \( \theta \) between the external magnetic field and the main axis of revolution of the ellipsoid shape of a nanoparticle and is given by a well known formulae introduced by Kittel [33];

\[
\left( \frac{\omega}{\gamma} \right) = \left[ H_0 + M \left( N_\perp - N_\parallel \right) \cos \theta \right],
\]

\[
\left[ H_0 + M \left( N_\perp - N_\parallel \right) \cos 2\theta \right]^{1/2},
\]

where \( \omega \) is the frequency of the spectrometer, \( \gamma \) is the gyromagnetic ratio, \( M \) is the saturation magnetization, \( N_\perp \) and \( N_\parallel \) are the demagnetization form factors.

In the magnetic field directed parallel to the axis of revolution of the ellipsoid (\( \theta = 0 \)) Eq. (3) takes the form

\[
\left( \frac{\omega}{\gamma} \right) = \left[ H_0 + M \left( N_\perp - N_\parallel \right) \right],
\]

where \( H_0^p \) is the resonance field in parallel configuration.

For the magnetic field aligned perpendicularly to the axis of revolution of the ellipsoid (\( \theta = 90^\circ \)) Eq. (3) can be written in the form

\[
\left( \frac{\omega}{\gamma} \right) = \left[ H_0^c - M \left( N_\perp - N_\parallel \right) \right]^{1/2},
\]

where \( H_0^c \) is the resonance field in perpendicular configuration.

From Eqs. (4) and (5), the product \( M \cdot (N_\perp - N_\parallel) \) can be calculated

\[
M \cdot (N_\perp - N_\parallel) = M \cdot \Delta N = -2H_0^{c} - H_0^{p} \pm H_0^{c} \left( 5 + 4 \left( \frac{H_0^{0}}{H_0^{c}} \right) \right)^{1/2}.
\]

The values of that product calculated for \( \alpha\)-Fe and Fe\(_3\)C nanoparticles for all investigated samples are placed in the sixth column in Table 3. For both nanoparticles these values do not show any significant correlation with the amount of those nanoparticles and concentrate around average values of 0.514 kG and 1.851 kG for \( \alpha\)-Fe and Fe\(_3\)C, respectively. Assuming that the shape anisotropy of both types of nanoparticles is roughly the same (and thus \( \Delta N \) has a comparable value for \( \alpha\)-Fe and Fe\(_3\)C nanoparticles) it would follow that the saturation magnetization \( M \) for cementite would be approximately over three times larger than for \( \alpha\)-Fe.
Table 3. Magnetic properties of nanoparticles in the investigated samples. Anisotropy field is defined as \( H_a = H_0^+ - H_0^- \), total linewidth as \( DH = \Delta H + \delta f \), the product \( M \cdot \Delta N \) was calculated from Eq. (6), and \( H_{mc} \) from Eq. (10).

<table>
<thead>
<tr>
<th>Sample</th>
<th>Nanoparticle</th>
<th>Anisotropy field ( H_a ) [kG]</th>
<th>Total linewidth [kG] ( DH(\perp) ) ( DH(\parallel) )</th>
<th>( M \cdot \Delta N ) [kG]</th>
<th>( H_{mc} ) [kG]</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>( \alpha )-Fe</td>
<td>0.8</td>
<td>9.2</td>
<td>4.4</td>
<td>0.523</td>
</tr>
<tr>
<td></td>
<td>Fe(_3)C</td>
<td>3.1</td>
<td>8.5</td>
<td>4.6</td>
<td>1.987</td>
</tr>
<tr>
<td>B</td>
<td>( \alpha )-Fe</td>
<td>0.7</td>
<td>9.2</td>
<td>4.1</td>
<td>0.459</td>
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<td>Fe(_3)C</td>
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<td>C</td>
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<td>8.9</td>
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<td>0.459</td>
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<td>D</td>
<td>( \alpha )-Fe</td>
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<td>E</td>
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<td>F</td>
<td>( \alpha )-Fe</td>
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<td>8.5</td>
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<tr>
<td>Average values</td>
<td>( \alpha )-Fe</td>
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<td>3.9</td>
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<td>-</td>
<td>-</td>
<td>1.851</td>
</tr>
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</table>

nanoparticles. This is in direct contrast to the published data on the value of that parameter because the saturation magnetization of \( \alpha \)-Fe is reported to be almost two times bigger than for Fe\(_3\)C [34]. Thus it could be concluded that the mechanism of shape anisotropy is not capable to account for the observed magnetic anisotropy field \( H_a \) in our samples.

In case of magnetocrystalline anisotropy the resonance field depends on the orientation of the external magnetic field and an easy axis of magnetization of a nanoparticle, given by two angles \( \theta \) and \( \varphi \). The resonance condition could be written in the following form [35]

\[
\left( \frac{\omega}{\gamma} \right) = \left[ H_0 + H_{mc} \left( 2 - \left( 5/2 \right) \sin^2 2\theta - \left( 5/2 \right) \sin \theta \sin \varphi \right) \right],
\]

where \( H_{mc} \) is the magnetocrystalline anisotropy field. For an external magnetic field aligned along the easy axis of magnetization (parallel orientation) of a nanoparticle this leads to the following equation for the resonance field \( H_0^+ \):

\[
\left( \frac{\omega}{\gamma} \right) = \left[ H_0^+ + 2H_{mc} \right],
\]

while for perpendicular orientation the resonance field \( H_0^- \) fulfills the following equation:

\[
\left( \frac{\omega}{\gamma} \right) = \left[ H_0^- - (1/2)H_{mc} \right],
\]

Thus the magnetocrystalline anisotropy field \( H_{mc} \) could be calculated if \( H_0^+ \) and \( H_0^- \) are known:

\[
H_{mc} = (2/5) \left( H_0^+ - H_0^- \right) = K_1 / M.
\]

In Eq. (10) \( K_1 \) is the first-order uniaxial anisotropy constant. In the seventh column in Table 3 calculated values of \( H_{mc} \) field are given for all samples. No correlation with the nanoparticles
concentrations could be detected for both kinds of magnetic nanoparticles. The average values of $H_{mc}$ field calculated from Eq. (10) are 0.31 kG and 1.15 kG for $\alpha$-Fe and Fe$_3$C, respectively. From Eq. (10) it could be seen that $H_{mc}$ field is inversely proportional to the saturation magnetization $M$. As $M(\alpha$-Fe) $>$ $M(\text{Fe}_3\text{C})$ than it is expected that $H_{mc}(\alpha$-Fe) $<$ $H_{mc}(\text{Fe}_3\text{C})$ what is indeed observed in our samples. This strongly suggests that the magnetocrystalline anisotropy is largely responsible for the observed magnetic anisotropy of our samples.

The total anisotropic linewidth $D_H$, defined as $D_H=\Delta_{Hr}+\delta_{Hr}$ has been calculated for each sample and for each nanoparticle type (see Table 3). For $\alpha$-Fe nanoparticles, the values of $D_H(||)$ and $D_H(\perp)$ seem to be independent on the concentration of this phase, so the average values can be calculated and they are 3.9 kG and 9.2 kG, respectively. The situation is quite different for Fe$_3$C nanoparticles. As the concentration of that phase in our samples is generally greater than for $\alpha$-Fe nanoparticles and changes in a much wider range, $D_H(||)$ and $D_H(\perp)$ display significant dependence on wt(Fe$_3$C) (see Fig. 7). In case of a dipole-dipole interaction between relatively close nanoparticles separated by a distance $d$, the linewidth is expected to vary as $DH\sim d^3$, and in consequence $DH\sim wd$, where $w$ is the weight percentage of that magnetic phase. As could be seen in Fig. 7 $D_H(\perp)$ varies roughly linearly with the concentration of Fe$_3$C component, indicating on a dominant contribution of the dipole-dipole interaction to the FMR linewidth of cementite.

4. CONCLUSIONS

Six samples produced during different stages of carburization of iron nanoparticles, containing different concentrations of $\alpha$-iron, iron carbide and carbon have been investigated by FMR method. The FMR spectra were fitted very well by four Callen-type lineshape functions (two different magnetic centers, each with magnetic anisotropy approximated by parallel and perpendicular components). The FMR parameters (resonance field, linewidth, integrated intensity) have been calculated for each component and discussed in terms of underlying magnetic interactions. It was determined that the magnetocrystalline anisotropy is the main contributor to the observed anisotropy, the shape anisotropy being of secondary importance. The linewidth is determined primarily by the dipole-dipole interaction in case of Fe$_3$C nanoparticles.

REFERENCES


