MAGNETIC PROPERTIES AND INTERPARTICLE MAGNETIC INTERACTIONS IN POWDER SAMPLES OF MAGNETITE AND MAGNESIUM OXIDE MIXTURES

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Abstract

In this paper, magnetic properties and interparticle magnetic interactions of the powder mixtures consisting of a ferromagnetic magnetite (mean particle size about 50 nm) and diamagnetic MgO (micrometric particles) are presented. Mixtures with concentrations of Fe₃O₄ from 10 to 80 wt. % were prepared using two methods: ultrasonic homogenization and manual mechanical mixing. Additionally, the powder mixtures were mechanically pressed in a specially developed device to produce samples in form of pills with the diameter of 10 mm. An EV9 MicroSense vibrating sample magnetometer (VSM) was used for measurements of the virgin curves and magnetization loops. Both measured quantities were used for construction of the Henkel plots that indicate the behavior of the magnetic interparticle interactions of prepared samples. We have discovered that the sample with 30 wt. % of Fe₃O₄ exhibited the most intensive interparticle interactions and at the same time the highest value of the remnant and saturation magnetization.

Keywords: Magnetite nanoparticles; vibrating sample magnetometer, Henkel plot; ultrasonic homogenization

1. INTRODUCTION

Magnetic nanoparticles are a frequent point of attention especially in terms of their usage in technological areas such as data storage or chemical and biomedical applications. Magnetic nanoparticles on the Fe basis belong among the most frequent used types of nanoparticles [1-5]. Such mixed magnetic structures, e.g. magnetite dispersed in a diamagnetic matrix, have been discovered in several biological systems, where single domain and/or superparamagnetic magnetite are parts of cells or bacteria – objects which interact in organisms.

Existence of such structures is not always clear, but it is often attributed the role of a magneto sensor. In such case, these structures would have to interact with a very weak geomagnetic field of the size of 30−60 µT and use a so far unknown amplifying mechanism. Such mechanism may be based on interparticle interactions of a single domain magnetite, since it was discovered in animal bodies and its single domain form appears to be very favorable for biomagnetic sensors [6-9].

This contribution deals with the measurement of magnetic interparticle interactions of powder mixtures of a single domain magnetite (Fe₃O₄) and diamagnetic MgO. Basic magnetic parameters were determined from measured hysteresis loops and interparticle magnetic interactions were evaluated using the Henkel plots [4,5]. From the measured concentration dependences we attempted to find a potential amplifying mechanism or parameter which initiates it and which could play an important role in applications in biomagneticsensorics.
2. MATERIAL AND EXPERIMENTAL METHODS

Powder mixtures of ferromagnetic magnetite Fe₃O₄ (Sigma Aldrich company, product No. 637106, particle size about 50 nm) and diamagnetic MgO powder (Sigma Aldrich company, product No. 220361, particle size about several μm) in magnetite concentrations from 10 to 80% wt. % were prepared [10-12]. For the preparation of the mixed samples, two different methods were used: with the first method (denoted in the following text as non-homog), both powders were merely manually mechanically stirred for approximately 3 minutes, while the latter method (denoted as homog) included an extra process of sample homogenization by means of rotation in ultrasound field.

Samples for magnetometric measurements were subsequently prepared by mechanical pressing of the powder mixture into the form of a pill with a diameter of 10 mm and fixed with paraffin layer. Finally they were attached to a glass sample holder of a magnetometer using a teflon tape. We suppose that influence of the diamagnetic components (teflon tape, glass sample holder) is negligible in comparison to the magnetic response of the samples.

Measurements on vibrating magnetometer VSM EV 9 Microsense (resolution of magnetic moment 10⁻¹⁰ A·m² and adjustable resolution of magnetic field 10⁻⁴ T) were performed at room temperature. Altogether, 16 measurements were performed, of which 8 on samples prepared with the first method and the other 8 on samples with the latter one. At the beginning of every measurement, careful demagnetization of a sample was done. Then, measurement of virgin curve followed. Finally, hysteresis loop was measured in magnetic field ranging from -2T to +2T.

3. RESULTS AND DISCUSSION

Table 1 summarizes basic magnetic parameters (Bc - coercive field, Mr - remnant magnetization, and Ms - saturation magnetization) that were obtained from measured hysteresis curves. Fig. 1 shows the dependence of Mr and Ms on the magnetite concentration and also on the way of preparation process (non-homog/homog).

Table 1. Chosen magnetic parameters of samples depending on wt% Fe₃O₄ and on the preparation method (non-homog/homog).

<table>
<thead>
<tr>
<th>Fe₃O₄ [wt%]</th>
<th>Bc [mT]</th>
<th>Mr [A·m²/kg]</th>
<th>Ms [A·m²/kg]</th>
<th>ΔMmin [A·m²/kg]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>nonhomog</td>
<td>homog</td>
<td>nonhomog</td>
<td>homog</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>8.21</td>
<td>8.75</td>
<td>73.03</td>
<td>58.17</td>
</tr>
<tr>
<td>20</td>
<td>8.67</td>
<td>7.86</td>
<td>80.13</td>
<td>70.39</td>
</tr>
<tr>
<td>30</td>
<td>8.14</td>
<td>7.42</td>
<td>101.91</td>
<td>104.03</td>
</tr>
<tr>
<td>40</td>
<td>8.54</td>
<td>7.43</td>
<td>68.34</td>
<td>46.62</td>
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<tr>
<td>50</td>
<td>7.98</td>
<td>7.32</td>
<td>75.91</td>
<td>56.60</td>
</tr>
<tr>
<td>60</td>
<td>8.12</td>
<td>6.82</td>
<td>77.62</td>
<td>79.07</td>
</tr>
<tr>
<td>70</td>
<td>7.93</td>
<td>7.11</td>
<td>76.04</td>
<td>68.15</td>
</tr>
<tr>
<td>80</td>
<td>8.09</td>
<td>6.60</td>
<td>72.55</td>
<td>50.70</td>
</tr>
</tbody>
</table>

Henkel plots, which describe interparticle magnetic interactions in powder samples, were constructed using the following equation [4, 5]:

\[ \Delta H(B) = M_{vir}(B) \cdot \frac{1}{2} \cdot [M_{up}(B) + M_{down}(B)] \]

where \( M_{vir} \) is the value of magnetization on the virgin curve, \( M_{up} \) is the value of magnetization while amplifying a positive external field and \( M_{down} \) while reducing it. Intensity of interparticle interactions is determined by the
minimum $\Delta M_{\text{min}}$ on the curve $\Delta M = f(B)$ [13,14], its value for different Fe$_3$O$_4$ concentrations is presented in Table 1.

![Graph](image1.png)  

**Fig. 1:** $M$ and $M_s = f$(wt. % Fe$_3$O$_4$) dependence for non-homogenized and homogenized samples.

Examples of Henkel plots for samples containing 10 and 30% wt. % Fe$_3$O$_4$, prepared by both methods, are shown in Fig. 2.

![Graph](image2.png)  

**Fig. 2:** Dependence $\Delta M = f(B)$. Interparticle magnetic interactions of non-homogenized and homogenized samples with 10 and 30 wt. % Fe$_3$O$_4$ in MgO.

From Henkel plots we can see that interparticle interactions are the strongest in the range of external magnetic field, from 0.013 to 0.02 T. The value of $\Delta M_{\text{min}}$ fluctuates significantly depending on the concentration of Fe$_3$O$_4$ and reaches its maximum with 30 wt. % Fe$_3$O$_4$ (see Fig. 3).
Fig. 3: Dependence of $\Delta M_{\text{min}} = f(\text{wt. \% Fe}_3\text{O}_4)$ for nonhomogenized and homogenized samples.

It is interesting that the character of $\Delta M_{\text{min}} = f(\text{wt. \% Fe}_3\text{O}_4)$ follows the process of remnant magnetization $M = f(\text{wt. \% Fe}_3\text{O}_4)$ and saturation magnetization $M_s = f(\text{wt. \% Fe}_3\text{O}_4)$. Higher values of $M$ and $M_s$ correspond with a higher intensity of magnetic interactions and vice versa. Maximum values of $M$ and $M_s$ are reached with 30 wt. \% $\text{Fe}_3\text{O}_4$, i.e. with maximum intensity of interparticle magnetic interactions. The only exception is a homogenized sample with 60 wt. \% $\text{Fe}_3\text{O}_4$ (see Fig. 2 and 3). $B_c = f(\text{wt. \% Fe}_3\text{O}_4)$ dependence does not demonstrate this quality. The measured processes indicate a possibility to influence the intensity of interparticle magnetic interactions in powder mixtures using an appropriate ratio of concentrations of both components.

CONCLUSION

In this contribution, we examined magnetic parameters and interparticle magnetic interactions of powder samples of magnetite nanoparticles dispersed in magnesium oxide. Samples were generated with two different methods: the first group of samples was prepared by means of manual mechanical stirring, while the other samples were furthermore homogenized using ultrasound. In all samples, hysteresis loops were measured, from which coercive field, remnant magnetization and saturation magnetization were determined. Interparticle magnetic interactions were evaluated using the Henkel plots.

It was demonstrated that these interactions are the strongest with magnetic field ranging from 0.013 to 0.02 T. The intensity of these interactions changes significantly with the change of magnetite concentration and reaches its significant maximum with 30 wt. \% $\text{Fe}_3\text{O}_4$. The measured dependencies indicate a possibility to influence the intensity of interparticle magnetic interactions in powder mixtures using an appropriate ratio of concentrations of both components. The dependence of the intensity of interparticle magnetic interactions on the concentration of $\text{Fe}_3\text{O}_4$ corresponds with the dependence of remnant magnetization and saturation magnetization on the concentration of $\text{Fe}_3\text{O}_4$. The method of sample preparation influences the values of all examined quantities, but it does not significantly change the character of their concentration dependences.

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REFERENCES


