Study of Magnetite Nanoparticles by the Method of Mössbauer Spectroscopy

G. I. KRIVEN[1,2], YING SUN[3],
[1]Moscow Aviation Institute (National Research University),
Moscow, Volokolamskoe shosse, 4, 125993,
RUSSIA
[2]Institute of Applied Mechanics of RAS,
Moscow, Leningradsky prt. 7, 125040,
RUSSIA
[3]Department of Mechanical Engineering,
Hangzhou Xiaoshan Technician College,
Hangzhou Zhejiang, 448 Tonghui South Road, Xiaoshan District, 311200,
CHINA.

Abstract—In this work, we studied the Mössbauer spectra of magnetite samples of various compositions. To protect magnetite from oxidation, the resulting particles are coated with protective shells, among which silanes are promising, which polymerize on the surface of magnetite nanoparticles, forming strong covalent bonds. The coating of nanoparticles protects them from aggressive environmental influences, evens out their size distribution, and also protects the environment from the possible toxic effects of the particles themselves.

It was shown that the magnetite phase predominates in the sample of native particles, the coating of native particles with alkoxysilane does not lead to fundamental changes in the phase state of the sample particles, and oxidation with nitric acid leads to the complete transformation of magnetite into maghemite. It is obvious that the reason for the distortions of the relaxation nature in the Mössbauer spectra of the samples is the small sizes of the iron-containing domains, which allows us to consider the description of the spectra within the framework of the model of multilevel superparamagnetic relaxation.

Keywords—Nanoparticles, magnetite, spectroscopy, silanes.

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1. Introduction

It document is known that magnetite particles are sensitive to oxidation, which can lead to the appearance on the particle surface of a modified layer, the magnetic properties of which may differ from the particle core, leading to a decrease in the saturation magnetization. In addition, due to Van der Waals forces, particles tend to agglomerate. Thus, there is a need to stabilize magnetite nanoparticles [1],[2],[3],[4],[5], [6],[7],[8],[9],[10],[11],[12],[13]. This will make it possible to control the size of the resulting particles, prevent their aggregation after synthesis, protect the resulting particles from the aggressive oxidative effects of the environment, and the environment itself from the toxic effect of the nanoparticle. There is a wide range of substances capable of forming a protective shell on the surface of magnetite nanoparticles; among them, alkoxysilanes are of interest as inert, biocompatible, and functional inorganic ligands. There are many publications devoted to the synthesis and characterization of silica-coated iron oxide nanoparticles.

There are several ways to synthesize composite nanoparticles [14],[15],[16],[17],[18],[19],[20],[21],[22],[23],[24],[25],[26], [27],[28],[29],[30].

Modern composites have not only a wide range of physical and mechanical properties, but are also capable of directionally changing them, for example, increasing fracture toughness, regulating rigidity, strength, and other properties. These possibilities are expanded when fibers of different nature and geometry are used in composites, i.e., when creating hybrid composites. In addition, these materials are characterized by the appearance of a synergistic effect (coordinated joint action of several factors in one direction) [31],[32],[33], [34],[35],[36],[37],[38],[39],[40],[41],[42],[43],[44],[45],[46], [47].

The properties of the interface or interfacial zone, first of all, the adhesive interaction between the fiber and the matrix, determine the level of properties of composites and their retention during operation. Local stresses in the composite reach their maximum values just near or directly at the interface, where material destruction usually begins. The interface must have certain properties to ensure efficient

transfer of the mechanical load from the matrix to the fiber. The adhesion bond at the interface should not be destroyed under the action of thermal and shrinkage stresses arising from the difference in the temperature coefficients of linear expansion of the matrix and fiber or as a result of chemical shrinkage of the binder during its curing.

When creating nanocomposites, the key tasks are the development of efficient, reliable, and affordable production technologies for mass production, which make it possible to obtain materials with stable characteristics. The hand lay technique, also called wet lay, is the simplest and most widely used process for producing flat reinforced composites. The process consists of laying layers of a polymer in successive layering using an epoxy matrix. Wet-laying is a molding process that combines layers of reinforced carbon fiber with epoxy to create a high-quality laminate. Before starting the installation process, you must prepare the appropriate form. This preparation consists of cleaning the table and applying a release agent to the surface. The manual laying process can be divided into four main steps: mold preparation, epoxy coating, laying and curing. Form preparation is one of the most important steps in the installation process. This process requires dry reinforcement layers and the application of a wet epoxy matrix. They are connected together - reinforcing material, impregnated with a matrix

Nanoparticles, even with a very low volumetric content (less than 1%), are contained in such a fragment in a very large amount, and it is impossible to model their effect at this scale level. For example, a cubic fragment of a 1 μm matrix contains more than thousand nanoparticles for a given volumetric content. Therefore, in particular, the nano-modified binder is white, while the usual binder is yellow. To model such materials, it is necessary to resort to multiscale approaches and to carry out a consistent determination of effective properties at various scale levels. This task is greatly simplified if the properties of the nanomodified matrix are known from experiments. In particular, it is known that its Young's modulus is 2.5 GPa. The missing characteristic is Poisson's ratio, which can be approximately taken unchanged, or estimated on the basis of analytical calculations using the found value of the "effective" volumetric content of the filler, which was done. Further, it suffices to numerically solve the averaging problem on a representative fragment containing only nanoparticles.

Coatings on nano and micro-sized particles can serve for many purposes. First of all, modification of the surface with coatings makes it possible to make the particles compatible with various matrixes [14],[15],[16],[17],[18], [19],[20],[21],[22],[23],[24],[25],[26],[27],[28],[29],[30]. For medical purposes, the biocompatibility with the environments of a living organism is of crucial importance. It is equally important that coatings can significantly enhance or decrease the sorption properties of magnetically controlled sorbents. This provides prerequisites for the creation of magnetically controlled particles with specific sorption properties. It is also known that the coatings prevent the core from leaching out. The presence of a coating also often facilitates the stabilization of particles in an environment with an alkaline pH or significant salt concentration. For example, the isoelectric point of SiO_2 is reached at pH 2-3. Therefore, the particles coated with silica are negatively charged at the pH of the blood, which causes electrostatic repulsion, which avoids the formation of clumps.

In order to derive the equation of the adsorption isotherm, a number of simplifications are introduced. All the places where the adsorbed particles are fixed are the same, and adsorption on one of them does not affect the state of the other. The interaction between the adsorbed particles is negligible. The adsorption layer is monomolecular, i.e. it consists of one layer of molecules. In this case, the bond of the adsorbate with the adsorbent is sufficiently strong, which excludes the movement of the adsorption complex along the surface of the adsorbent (localized adsorption).

Active (well absorbing) adsorbents have a very large specific surface area. For example, the absorbers most often used for scientific purposes and industrial practice - activated carbon, silica gel, zeolites – have s_0 up to several hundred and even thousands of square meters per 1 g.

The ability of the adsorbent to absorb the adsorbate is characterized by the amount of adsorption. The amount of adsorption is the excess mass of the adsorbate in the boundary layer over its mass in an equal volume of the environment, referred to the unit surface of the adsorbent.

Sometimes the adsorption value is expressed in moles of adsorbate per 1 m^2 (or 1 cm^2) of the adsorbent surface. Since quite often the surface of the adsorbent is unknown, the value of adsorption is expressed in moles of adsorbate per 1 g of adsorbent (mol/g). It is customary to evaluate the process of toxin sorption by the adsorbing surface using the curves of Langmuir sorption isotherms.

Silanol binding agents are applied directly to the surface of Fe_3O_4 nanoparticles by copolymerization of monomers or by direct silanization. The developed surface of nanoparticles leads to a high density of surface functional groups [48],[49], [50],[51],[52],[53],[54],[55],[56],[57],[58],[59],[60],[61],[62], [63],[64],[65],[66],[67],[68],[69],[70],[71], which can fix a large number of biologically active substances [32]. The most common way to obtain LF Fe_3O_4/SiO_2 with a core-shell structure is the sol-gel method (Stober method), which consists in hydrolysis and polycondensation under alkaline conditions in ethanol [33].

In this regard, in this work, we performed a comparative analysis of the microstructure of magnetite nanoparticles synthesized by various methods before and after their modification with 3-aminopropyltriethoxysilane under various reaction conditions (in argon and during oxidation).

2. Study of magnetite samples

Methods for obtaining the studied samples of magnetite are presented in Table 1. Chemical analyses of the synthesized samples were carried out by the standard procedure described in our previous studies [32],[33],[34].

Table 1. List of obtained samples of magnetite nanoparticles.

Sample code	Sample description
M - EN	Synthesis of magnetite from FeSO4 · 7H2O and EDTA with heating T = 80°C
M - C	Synthesis of magnetite from FeCl3·6H2O and FeCl2·4H2O
M - A	Modification of sample M - With APTES coating
M - AO	Oxidation of sample M - A with nitric acid HNO3

An analysis of the sample M - EN, obtained by oxidation of Fe^{2+} with the formation of a complex with EDTA, was carried out by IR spectrometry. The image of the obtained spectrum is shown in Figure 1.

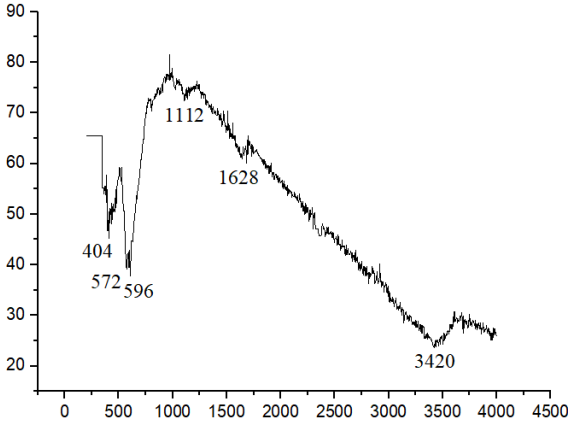

Fig. 1 ir spectrum for sample M - EN.

The characteristic absorption band at a wavelength of 572 cm^{-1} refers to the characteristic absorption bands of the Fe-O bond in Fe_3O_4, which is evidence of the formation of magnetite nanoparticles. A low intensity absorption band at a wavelength of 1628 cm^{-1} corresponds to asymmetric vibrations of the carboxylate group COO-, indicating binding with free Fe^{2+}/Fe^{3+} ions or surface magnetite ions. The broad absorption band at 3420 cm^{-1} is a characteristic region of vibration of the OH– groups of water.

Mössbauer absorption spectra were obtained on an MS1104EM express Mössbauer spectrometer. The noise / signal ratio in the obtained spectra did not exceed 1%. The experimental Mössbauer spectra were mathematically processed for high-resolution spectra (1024 points) using the SpectRelax 2.4 software. Chemical shift values are given relative to α-Fe. The spectra were described using two models based on the literature data on the Mössbauer parameters most likely for the considered samples of iron oxides: magnetite-Fe_3O_4 and maghemite γ-Fe_2O_3.

Mössbauer spectra at room temperature for all samples have the form of distorted asymmetrically broadened sextets. In this case, the sextets are not symmetrical both in intensity and in width. So, for a sample of native particles 1-3, the resonance lines have a noticeably wider width and lower intensity than lines 4-6 (Figure 2). In this case, the intensities of the lines are the same within groups 1-2 and 5-6.

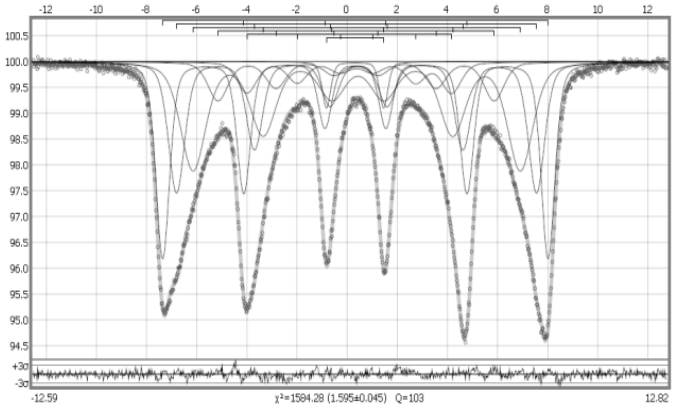

Fig. 2 Mössbauer spectrum of sample M - C at 295 °C.

All spectra can be satisfactorily described within a single model of five (295 K) or four (78 K) nested symmetric sextets and one symmetric doublet. In general, the models for different samples are similar to each other, and differ mainly in the ratio of the contributions of each of the subspectra for an individual sample. Thus, in the high-temperature spectra, the first, second, and third sextets have the largest areas (subspectra 1, 2, and 3, respectively). In the low-temperature spectra, the largest areas have 1 and 2 subspectra, and the areas of subspectra 3 and 4 that are close to each other in the samples of native and coated particles do not differ much from the area of subspectrum 2. These features, characterizing the distribution of iron atoms in crystallographic positions with different degrees of ordering of the local environment, distinguish these samples from magnetite, in which in the high-temperature spectrum one of the "internal" sextets (similar to subspectrum 4) had the largest area in addition to the outer sextet (similar to subspectrum 1) and the areas in the low-temperature spectrum decreased monotonically from outer to inner.

As it can be also seen from the Figure 2, the ultrafine parameters of the subspectra of the samples of native and coated particles are close at room temperatures. At the same time, based on the data at room temperature, all sextets can be divided into three groups. The first combines subspectra 1 and 4 with isomeric shifts corresponding to Fe^{3+} atoms in an octahedral oxygen environment. In the second, subspectrum 5 with an isomeric shift corresponding to Fe^{3+} atoms in a tetrahedral oxygen environment. And, finally, subspectra 2 and 3, which have too large values of isomeric shifts for Fe^{3+} iron atoms in the octahedral environment, but too small for $Fe^{2.5+}$ atoms in the octahedral voids of magnetite. Obviously, these sextets belong to iron atoms in octahedral voids of the oxidized form of nanomagnetite - δ-Fe_3O_4, in which the

proportion of Fe^{3+} significantly exceeds that of Fe^{2+}. At low temperatures, sextets regroup (including due to the Verwey transition), but they can also be divided into Fe^{3+} atoms and partially reduced $Fe^{(3-x)+}$.

The values of magnetic splitting obtained at room temperature are lower than those expected for bulk samples of magnetite and maghemite, which is typical for nanosized materials. The isomeric shifts of all subspectra of the sample of oxidized particles, obtained at room temperature, indicate that all atoms were oxidized to the Fe^{3+} oxidation state (octahedral positions). Even with low-temperature shooting, the isomeric shift of sextet 4 decreased significantly. Otherwise, the Mössbauer parameters did not undergo significant changes, except perhaps a noticeable increase in the widths of almost all subspectra, which may be associated with a decrease of the probability functions of the magnetic field distribution obtained for low-temperature Mössbauer spectra for all samples have the form of asymmetric unimodal peaks strongly shifted in the region of high fields (Figure 3).

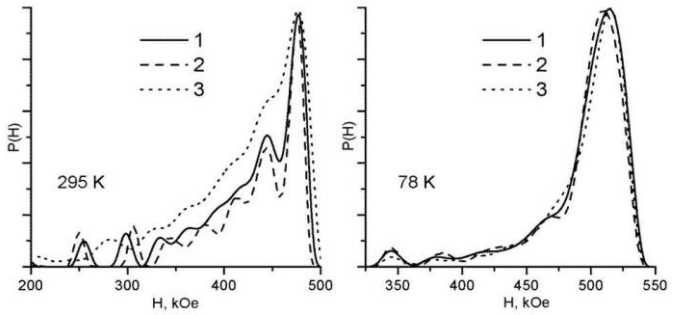

Fig. 3 Probability functions of magnetic field distribution: 1 - sample M - C, 2 - sample M - A, 3 - sample M - AO.

As it can be seen from the Figure 3, the parameters of the probability functions practically do not differ from each other, except that for the sample of APTES-coated particles, the distribution maximum is located at a slightly lower field strength.

From the data presented in the Figure 3 one can also conclude that the probability functions of the distribution of hyperfine parameters for high-temperature spectra are different for different samples. Thus, for a sample of native particles, the field distribution has the form of a predominantly asymmetric bimodal peak, with maxima localized in the region of high fields, and an extended "wing" extending into low fields (Figure 3). In the sample of coated particles (with the same predominant asymmetric bimodal peak), this wing is slightly more resolved - three local maxima are clearly manifested on it. Before the beginning of this "wing", two weak "satellites" are observed for both samples in the region of 255 and 300 kOe. In the sample of oxidized particles, in contrast to their predecessors, there is practically no "fine structure" on the dependence of the probability of the distribution of magnetic fields - the distribution has the form of a noticeably wider unimodal peak, with the weighted average shifted to lower fields. This indicates a much wider size distribution of the sample particles (variants of the local environment). It is obvious that the reason for the distortions of the relaxation nature in the Mössbauer spectra of the samples is the small sizes of the iron-containing domains, which allows us to consider the description of the spectra within the framework of the model of multilevel superparamagnetic relaxation.

3. Conclusion

An examination of the data obtained makes it possible to show that systematic changes are observed in the experimental spectra, which are the same at different temperatures. Moreover, when passing from a sample of native particles to a sample of coated particles, these spectra changes are less than from a sample of coated particles to a sample of oxidized particles. Thus, this gives us possibility to make a conclusion that the treatment of the initial sample of magnetite with APTES leads to a narrowing of the external profile of the relaxation sextet, apparently due to the sharpening of the internal sextets. The processing of a sample of APTES coated particles with nitric acid caused the removal of components from the right side of the spectra, and the appearance of components in the left side of the spectrum, i.e. in fact, we are talking about a shift of some sextet to the region of low isomeric shifts, i.e. oxidation of iron.

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