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Study of ultrafine magnetic parameters of Fe_2O_3@$NdFeO_3$ nanocomposites depending on the Nd_2O_3 dopant concentration[1]

Abstract: the article presents the results of a study of the ultrafine magnetic parameters of Fe_2O_3@$NdFeO_3$ nanocomposites obtained by solid-phase synthesis followed by thermal sintering of samples at a temperature of 1000 ° C. The study of hyperfine magnetic parameters was carried out by changing the Mossbauer spectra of the studied nanocomposites depending on the concentration of dopants. The resulting dependences of the hyperfine magnetic parameters were studied for nanocomposites depending on the Nd_2O_3 dopant concentration, the content of which varied from 0.1 to 0.5 mol. As shown earlier, varying the dopant concentration leads to phase transformations of the Fe_2O_3@$NdFeO_3$/Fe_2O_3 type, followed by the dominance of the $NdFeO_3$ phase at high dopant concentrations of 0.4-0.5 mol. During studies of the hyperfine parameters of the magnetic field for nanocomposites under study, it was found that the obtained parameters are characteristic of the hematite (Fe_2O_3) structure, with a high structural and magnetic ordering degree. The obtained dependences of the hyperfine parameters indicate that the decrease in the hyperfine magnetic field depending on the dopant concentration is due to structural deformations of the crystal lattice, as well as the dominance of the $NdFeO_3$ phase, which is the substitution phase.

Keywords: nanocomposites, magnetic characteristics, Mossbauer spectroscopy, substitution effect, hematite.

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Introduction. Over the past few years, much attention has been paid to the development of new types of magnetic micro-and nanocomposites based on iron oxide compounds. Interest in these structures is due to great prospects in the practical application of composites in microelectronics, magnetic information carriers, targeted drug delivery, as well as various catalysts for decomposition of organic dyes, etc. [1-5]. At the same time, special attention is paid to the study of phase transformations and changes in structural, magnetic, or conductive parameters, as well as their relationships, which are interesting from a fundamental point of view, to obtain new unique data on the properties of nanomaterials. Obtaining new data on the relationship between structural and magnetic parameters, in turn, can open even greater prospects for practical application [6-8]. Thus, knowledge of the influence of structural parameters, including structural distortions and deformations, which are an integral part of any nanomaterials, on magnetic parameters will provide data on the possibilities of controlling magnetic properties, which opens broad prospects in the future [9,10]. Moreover, in the case when nanocomposites are two-three-phase structures, the presence of substitution or interstitial phases can play an important role in determining the magnetic parameters, as well as their further practical application.

So, for example, doping of iron oxide nanoparticles with magnetic components leads to the formation of a spinel structure that has unique magnetic characteristics and allows a significant crystal lattice rearrangement, which leads to the appearance of additional vacancy defects. The presence of

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vacancy defects in the structure of nanocomposites leads to a change in the material properties, due to the possibility of filling them in the case of external influences. At the same time, the nanoscale dimensions of oxide composites contain many structural defects, the presence of which leads to the formation of metastable states, and in the case of additional vacancy defects, any external action leads to a significant crystal lattice rearrangement.

Among the variety of magnetic nanocomposites, iron oxide compounds doped with various components such as nickel, cobalt, or neodymium, as well as their oxide forms, have significant differences from other magnetic composites, which leads to a large potential for their use as a base for water purification, photocatalysts, absorbents, etc. Interest in this direction in magnetic structures is primarily due to the possibility of reusing nanocomposites over several cycles since the magnetic properties allow them to be captured and removed from aqueous solutions by simple magnets, after which they can be used again. However, this field of application requires the knowledge of magnetic components, as well as their resistance to degradation in case of long-term operation.

Based on the foregoing, the purpose of this work is to study the effect of Nd_2O_3 dopant concentration on ultrafine magnetic parameters of Fe_2O_3@$NdFeO_3$ nanocomposites, as well as to conduct a comparative analysis of structural distortions and deformations with changes in the magnetic properties of nanocomposites [11, 12]. Interest in these types of nanocomposites is due to their great prospects for application in photocatalysis and decomposition of organic dyes, as well as purification of aqueous media from pollutants, which is a very important area of research in the field of improving the environmental situation in industrial production [13-15].

Experimental part. Fe_2O_3@$NdFeO_3$ nanocomposites obtained by solid-phase synthesis were chosen as objects of study. Samples were synthesized by mixing Fe_2O_3 nanoparticles obtained by chemical precipitation with $_2O_3$ nanoparticles and subsequent grinding in a PULVERISETTE 6 planetary mill (Fritsch international, Idar-Oberstein, Germany) for 1 hour at a grinding speed of 400 rpm. The concentration of the Nd_2O_3 dopant was varied in the range from 0.1 to 0.5 mol. [16]. Grinding of the initial structures was carried out in a grinding cup made of tungsten carbide, the use of which leads to the absence of impurities during stirring.

After grinding, the resulting mixtures were subjected to thermal annealing for 5 hours at a temperature of $1000\,^{\circ}$C. The cooling of the samples, to avoid the effect of hardening, was carried out for 24 hours together with the furnace. Annealing was carried out in a SNOL muffle furnace (SNOL, Moscow, Russia), the heating rate was $10\,^{\circ}$C/min.

The study of the effect of Nd_2O_3 doping depending on the dopant concentration on the magnetic characteristics, including the values of hyperfine parameters of the magnetic field, was carried out using the Mossbauer spectroscopy method. Mossbauer studies were carried out on a Mossbauer spectrometer MS1104Em (Rostov-on-Don, Russia). The source was 57Co in an Rh matrix, the activity of which was 50 mCi. At the same time, the use of the Mossbauer spectroscopy method for estimating hyperfine magnetic parameters is one of the most relevant and reliable methods for determining the magnetic properties of iron-containing structures, and also makes it possible to determine the relationships between magnetic parameters and structural distortions resulting from phase transformations or structural changes with high accuracy.

Results and discussion. According to previous studies, it was found [16] that the doping with Nd2O3, depending on the dopant concentration, leads to the formation of phase transformations of the $Fe_2O_3 \rightarrow NdFeO_3/Fe_2O_3$ type, moreover, with the dominance of the Nd_2O_3 phase at dopant concentrations of 0.4-0.5 mol. The formation of the $NdFeO_3$ phase also led to coarsening of particles, as well as an increase in the degree of structural ordering. The formation of this phase at high concentrations of the Nd_2O_3 dopant is due to the substitution of Fe atoms in the octo- and tetrahedral positions of the crystal lattice by Nd atoms. At the same time, such a substitution undoubtedly affects not only the structural characteristics, expressed in a change in the crystal lattice parameters, crystallite sizes but also the ultrafine magnetic texture, as well as the magnetic characteristics of nanocomposites.

The study of the magnetic properties of the hyperfine magnetic parameters of the Fe_2O_3@$NdFeO_3$ nanocomposites under study was carried out using the Mossbauer spectroscopy method. Figure 1 shows the results of a model interpretation of the Mossbauer spectra of the obtained nanocomposites depending on the dopant concentration. The general view of the spectra is characterized by the presence of a Zeeman sextet with symmetrical lines, the width, and intensity of which are characteristic of the magnetic structure of hematite. The used model for interpretation has the values of the functional $\chi 2$= 0.9-1.1, which indicates the absence of systematic deviations of the experimental data from the model representation used for the analysis of the obtained data. At the same time, the absence of deviations characteristic of disordered regions characterized by the presence of quadrupole doublets indicates a high structural and magnetic ordering degree of the synthesized nanocomposites.

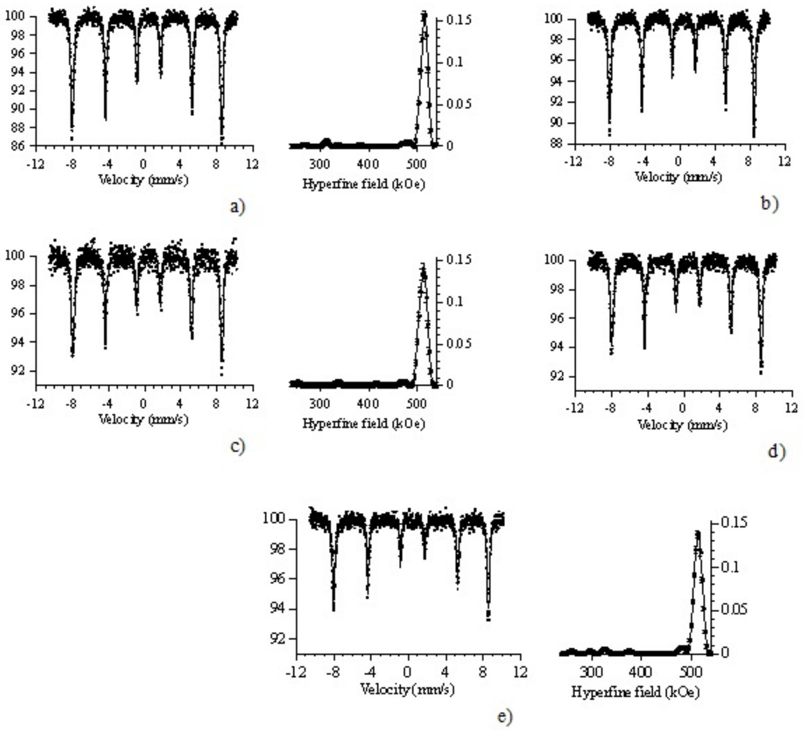

FIGURE 1 – Mossbauer spectra and distributions of the hyperfine magnetic field in Fe_2O_3@$NdFeO_3$ nanocomposites depending on the concentration of the Nd_2O_3 dopant: a) 0.1 mol; b) 0.2 mol; c) 0.3 mol; d) 0.4 mol; e) 0.5 mol

An analysis of the obtained dependences of the distributions of the hyperfine magnetic field showed that the characteristic maximum observed in the region of 510-519 kOe is characteristic of the values of the hyperfine magnetic field of the hematite structure (517 kOe). At the same time, the presence of small peaks in the region of 250-350 kOe may be due to the presence of substituted iron atoms in the structure of the composites. The general tendency for the maximum to shift to lower magnetic fields indicates a change in the structure and the appearance of magnetic disordering, which maybe because of the formation of the $NdFeO_3$ substitution phase and its dominance at high dopant concentrations. Also, the influence of the dopant and the dominance of the $NdFeO_3$ phase in the structure is evidenced by the formation of asymmetry in the distributions of the hyperfine fields for samples with a dopant concentration of 0.4-0.5 mol.

Figure 2 shows the results of changes in the hyperfine parameters depending on the Nd_2O_3 dopant concentration in nanocomposites based on iron oxide obtained by thermal annealing at a temperature of 1000 ° C.

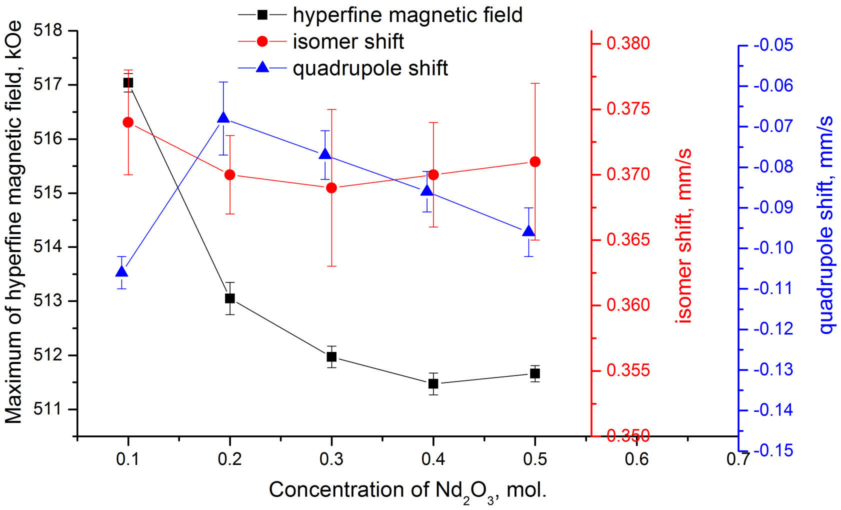

Figure 2 – Dependence of the change in the hyperfine parameters on the Nd_2O_3 dopant concentration

The general trend of changes in the hyperfine parameters can be divided into two components. The first trend of changes is associated with an exponential decrease in the hyperfine magnetic field from 517.04 kOe for nanocomposites with a dopant concentration of 0.1 mol. up to 513-511 kOe for all other dopant concentrations. Such a decrease maybe because of the formation of the NdFeO3 phase because of the replacement of iron atoms by neodymium atoms, which leads to the disordering of magnetic characteristics. At the same time, the change in the values of the quadrupole shift and the isomeric shift for the Zeeman sextet practically does not change.

Figure 3 shows the results of changes in the values of distortions of the crystal lattice and hyperfine magnetic field depending on the Nd_2O_3 dopant concentration. The analysis of crystal lattice distortions was carried out by determining the ratio of c/a parameters for the Fe_2O_3 hematite phase, which reflects the deformation of the crystal structure upon substitution of atoms at the lattice sites. The distortion of the hyperfine magnetic field was evaluated in comparison with the value of the hematite phase characteristic of the standard.

The general appearance of the dependences obtained has a general trend of changes; however, as can be seen from the presented data, the dominance of the $NdFeO_3$ phase at concentrations of 0.4-0.5 mol. has a greater effect on the crystal lattice distortion than on the hyperfine magnetic field distortion. This effect maybe because of the substitution of iron atoms in the structure of the hematite phase, leading to the formation of the $NdFeO_3$ phase, and its subsequent dominance leads to large structural distortions. In turn, changes in the magnetic field hyperfine parameters are characterized by the nearest environment and the formation of locally heterogeneous regions, the presence of which was not identified by Mossbauer spectroscopy.

At the same time, the general appearance of changes in the value of structural distortions has a pronounced dependence on the phase ratio of nanocomposites. At low dopant concentrations, when the Fe_2O_3 phase dominates in the structure, the amount of distortion is quite small and does not exceed 3-5 % compared to the initial values. However, the dominance of the $NdFeO_3$ phase, which is observed according to [16] at concentrations above 0.4 mol. there is a sharp increase in the structure deformation associated with the substitution effect, as well as the formation of a solid solution from two phases in the structure. At the same time, the effect of disordering and deformation of the crystal structure, as well as magnetic parameters, can be due to a change in the concentration of vacancy defects and dislocation density in the structure of nanocomposites. Figure 4 shows the

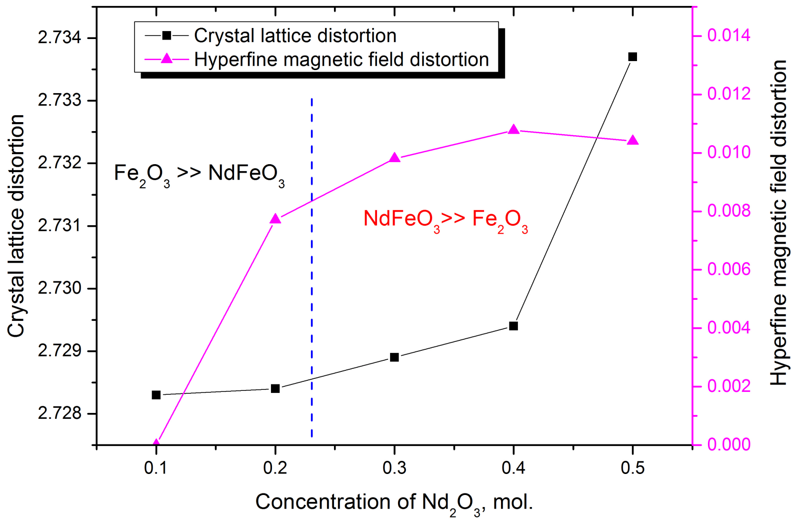

Figure 3 – Dependence of crystal lattice distortion and ultrafine magnetic field (the dashed line indicates phase separation depending on the dopant concentration)

results of calculations of the concentration of vacancy defects in the structure resulting from the substitution effect.

Vacancy defects were calculated using calculation formula (1):

$$\delta = \frac{1}{L^2} \tag{1}$$

where L is the grain size, which was determined from the analysis of X-ray diffraction patterns.

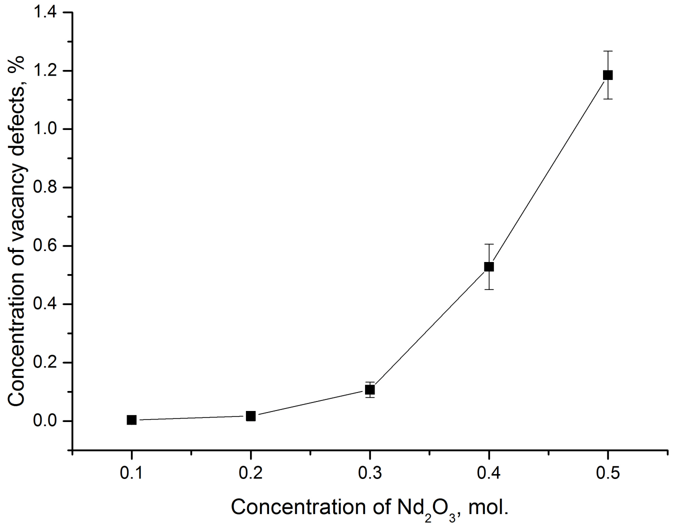

Figure 4 – Results of changes in the vacancy defect concentration depending on the Nd_2O_3 dopant concentration

As can be seen from the results obtained, a change in the phase composition, followed by the dominance of the $NdFeO_3$ phase, leads to a sharp increase in vacancies in the crystalline sublattices, which in turn hurts magnetic parameters of the nanocomposites and increases the disordering degree. An increase in the concentration of vacancy defects with an increase in the Nd_2O_3 dopant concentration can be due to the partial substitution of neodymium atoms for iron atoms in the Fe_2O_3 structure, followed by the formation of the $NdFeO_3$ substitution phase, which in turn leads to deformation of the structure due to the formation of additional interfacial boundary effects.

At the same time, an increase in the concentration of vacancy defects has a good correlation with the θ angle, which characterizes the direction of the magnetization axis and orientation of magnetic domains, the change of which varies in the range of θ =50-54 °, which corresponds to a state close to the absence of a preferred magnetization direction. Such magnetic disordering caused by the absence of a preferred direction of the magnetization axis can somewhat complicate the control of magnetic particles during operation, however, the presence of additional vacancy defects can compensate for this effect by increasing the rate of absorption or photocatalytic reactions, depending on the type of practical application of nanocomposites. In the case of heavy metal adsorption from aqueous solutions, the presence of additional structural distortions and vacancy defects leads to the presence in the structure of many active centers, which can serve as activators for the absorption of heavy metals.

Conclusion. The article presents the results of a study of the ultrafine magnetic parameters of Fe_2O_3@$NdFeO_3$ nanocomposites. During the study, the dependences of the influence of structural distortions on the magnetic parameters of nanocomposites, as well as the relationship between the phase composition and hyperfine parameters of the magnetic field, were established. The obtained dependences of the hyperfine parameters indicate that the decrease in the hyperfine magnetic field depending on the dopant concentration is due to structural deformations of the crystal lattice, as well as the dominance of the $NdFeO_3$ phase, which is the substitution phase. At the same time, the crystal lattice deformation due to an increase in the $NdFeO_3$ phase leads to an increase in the disordering of magnetic parameters, as well as a decrease in the hyperfine magnetic field value.

The obtained data will be used in the further development of catalysts for the decomposition of organic dyes, as well as purification of aqueous media from heavy metals and wastes of the textile industry.

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Nd_2O_3 допант концентрациясына байланысты Fe_2O_3@$NdFeO_3$ нанокомпозиттерінің өте жұқа магниттік параметрлерін зерттеу

Аннотация. Бұл жұмыста Fe_2O_3@$NdFeO_3$ нанокомпозиттерінің өте жұқа магниттік параметрлерін зерттеу нәтижелері ұсынылған, қатты фазалық синтез әдісімен алынған, содан кейін үлгілерді 1000 ° C температурада термиялық синтездеу. Өте жұқа магниттік параметрлерді зерттеу допант концентрациясына байланысты зерттелетін нанокомпозиттердің Мессбауэр спектрлерін өлшеу арқылы жүргізілді. Алынған ультра жұқа магниттік параметрлердің тәуелділігі Nd_2O_3 допантының концентрациясына байланысты нанокомпозиттер үшін зерттелді, оның мазмұны 0.1-ден 0.5 mol-ге дейін өзгерді. Бұдан бұрын көрсетілгендей, допант концентрациясының озгеруі $Fe_2O_3 \rightarrow NdFeO_3 / Fe_2O_3$ сияқты фазалық өзгеру процестеріне әкеледі, 0.4-0.5 mol жоғары допант концентрациясы кезінде $NdFeO_3$ фазасының үстемдігімен. Зерттелетін нанокомпозиттер үшін магнит өрісінің өте жұқа параметрлерін зерттеу барысында алынған параметрлер жоғары құрылымдық және магниттік реттілікпен гематит құрылымына (Fe_2O_3) тән екендігі анықталды. Алынған ультра жұқа параметрлердің тәуелділігі допант концентрациясына байланысты ультра жұқа магнит өрісінің төмендеуі кристалдық тордың құрылымдық деформацияларымен байланысты екенін көрсетеді, сондай-ақ $NdFeO_3$ фазасының үстемдігі, алмастыру фазасы.

Түйін сөздер: нанокомпозиттер, магниттік сипаттамалар, Мессбауэр спектроскопиясы, алмастыру әсері, гематит.

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Изучение сверхтонких магнитных параметров Fe_2O_3@$NdFeO_3$ нанокомпозитов в зависимости от концентрации допанта Nd_2O_3

Аннотация. В работе представлены результаты исследования сверхтонких магнитных параметров Fe_2O_3@$NdFeO_3$ нанокомпозитов, полученных методом твердофазного синтеза с последующим термическим спеканием образцов при температуре 1000 ° C. Изучение сверхтонких магнитных параметров проводилось путем изменения мессбауэровских спектров исследуемых нанокомпозитов в зависимости от концентрации допантов. Полученные зависимости сверхтонких магнитных параметров были изучены для нанокомпозитов в зависимости от концентрации допанта Nd_2O_3, содержание которого варьировалось от 0.1 до 0.5 mol. Как было показано ранее, варьирование концентрацией допанта приводит к процессам фазовых трансформаций типа $Fe_2O_3 \rightarrow NdFeO_3 / Fe_2O_3$, с последующим доминированием фазы $NdFeO_3$ при больших концентрациях допанта 0.4-0.5 mol. В ходе изучения сверхтонких параметров магнитного поля для исследуемых нанокомпозитов было установлено, что полученные параметры характерны для структуры гематита (Fe_2O_3), с высокой степенью структурного и магнитного упорядочения. Полученные зависимости сверхтонких параметров свидетельствуют о том, что снижение величины сверхтонкого магнитного поля в зависимости от концентрации допанта обусловлены структурными деформациями кристаллической решетки, а также доминированием фазы $NdFeO_3$, являющейся фазой замещения.

Ключевые слова: нанокомпозиты, магнитные характеристики, мессбауэровская спектроскопия, эффект замещения, гематит.

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