Magnetic nanocomposites based on borate glasses doped with Fe and Mn

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Magnetic properties, including magneto-optical Faraday Effect (FE), have been investigated in potassium-aluminum-boron glasses doped with iron and manganese oxides. The formation of magnetic nanoparticles has been proved using high resolution electron microscopy. A wide variety of particle size, shape, structure, and distribution in the matrix has been observed depending on the technological conditions. The narrow region of conditions has been established providing formation of almost ideal manganese ferrite single crystals of about 40 nm size distributed rather homogeneously in the matrix. Therefore, the glasses can be considered as a nano-composite material. Thanks to the low concentration of paramagnetic elements, the glasses kept the optical transparence and demonstrated high FE in the infrared and a part of the visible spectral regions, thus making it possible to develop a new magneto-optical material for the 1.0–1.5 μm spectral region.

Исследованы магнитные свойства, включая магнитооптический эффект Фарадея (FE), в калиево-алюмо-боратных стеклах, допированных оксидами марганца и железа. С помощью электронной микроскопии высокого разрешения доказано формирование магнитных наночастиц. Наблюдалось большое разнообразие размеров, формы, структуры частиц и их распределения в матрице в зависимости от технологических условий. Установлена узкая область условий, обеспечивающих формирование почти идеальных монокристаллов марганцевого феррита размером 40 нм, довольно равномерно распределенных в матрице. Поэтому исследованные стекла можно рассматривать как нано-композитный материал. Благодаря низкому содержанию парамагнитных элементов, стекла сохраняют оптическую прозрачность и демонстрируют высокие значения FE в инфракрасной и части видимой области спектра, что позволяет предпринять усилия по созданию нового магнитооптического материала для области спектра 1,0–1,5 мкм.

Nanosized magnetic particles in nonmagnetic matrices attract considerable attention because of their importance both from fundamental and practical viewpoints. Manganese ferrite is among compounds used most often to prepare nanoparticles [1–7] both in the powder state and embedded in various matrices. Various methods of particle preparation and various matrices used give rise to a wide variety of magnetic characteristics observed in systems containing nanoparticles. Great attention is currently paid to magnetic particles dispersed in glassy matrices. Thanks to its high flexibility and relatively low cost, the glass technology seems to be proper one to prepare nanoparticles with purposely variable properties. To obtain magnetic particles, a rather high amount of paramagnetic oxide (about 20 to 40% by mass) is to be introduced into glass composition (see, e.g., [8–15]). Potassium-aluminum-boron glass system occurred to be an exclusion of this rule: in spite of a low Fe_2O_3 content (less than about 3.0 % by mass), the magnetic susceptibility [16, 17] and Mossbauer spectra [18] singularities therein allowed to suppose hematite (α–Fe_2O_3) particles arising in

Table. Some characteristics of samples: heat treatment temperature, concentrations of Mn and Fe oxides in the charge, average crystallite dimensions according to X-ray diffraction spectra, FR (α) value in magnetic field $H = 0.1$ T for 1.5 μm, and the relative value of the remnantl FR (α_0/α_s) in zero magnetic field.

Sample No.	$t_{treatment}$, C°	n_{MnO}, % mass	n_{Fe2O3}, % mass	$D_{average}$, Å	α, grad/cm	α_0/α_s
1	520	1	1.5	–	0.09	0
2	520/6 h			–	0.18	0.14
3	520	1.5		–	1.11	0.27
4	560			247	4.32	0.55
5	600			247	3.77	0.50
6	500 + 560			113	1.75	0.19
7	520/6 h			88	1.7	0.50
8	560			165	3.45	0.37
9	600	2	3	219	0.29	0.02
10	560 + 600			308	4.36	0.39

the glass. Later, glasses of this system containing MnO together with Fe_2O_3 were synthesized, which were characterized by a non-linear magnetic field dependence of magnetization with hysteresis and magnetic saturation [19]. At the same time, the glasses were transparent in the visible and near infrared spectral range and demonstrated high magneto-optical Faraday rotation (FR) [19, 20]. The peculiar magnetic properties of the glasses were explained by formation of manganese ferrite nanoparticles during the glass synthesis and additional thermal treatment. A further study of particle characteristics depending on technological conditions and correlations between these characteristics and magnetic properties of the particle assembles could provide the development of a new material for magneto-optical devices. This work is aimed at the characterization of particles and systematic investigation of magnetic properties of the particle assembles as functions of their dimensions, structure, composition and volume distribution for the system $22.5Al_2O_3 \cdot 22.5K_2O \cdot 55B_2O_3$ doped with Fe_2O_3 and MnO in low concentrations.

KNO_3, Al_2O_3, and H_3BO_3 were used as initial materials to prepare glasses of the basic composition $22.5Al_2O_3 \cdot 22.5K_2O \cdot 55B_2O_3$ using the technique described in [20]. Before the synthesis, Fe_2O_3 in the concentration of 1.5 or 3.0 % (mass) and MnO in various concentrations ranging from 1.0 to 2.0 % (mass) over 100 % (mass) of the basic composition were added to the charge. The glasses were melted at 1100−1300°C in platinum crucibles under various oxidation conditions. The

melt was poured onto steel sheets, and the glass plates were cooled spontaneously in air to 380°C and then hold at this temperature for several hours. Additional heat treatment was carried out at different temperatures from 520°C to 600°C during 2, 4, or 6 hours. Manganese and iron oxide concentrations in the charge, temperatures and the additional heat treatment duration for some samples are presented in Table 1.

X-ray diffraction (XRD) spectra were obtained using a D8ADVANCE (Bruker) instrument in CuK_α emission ($\lambda = 0.15406$ nm). The phases were identified basing on the diffraction peak sequence by comparison with that for a $MnFe_2O_4$ single crystal using the PDF-2 database [21]. The particle size was estimated using the Scherrer equation. The visualization of separate particles in glasses and determination of their dimensions, shape, and volume distribution were carried out using a JEM-2010 high resolution transmission electron microscope (HRTEM) having resolution 0.14 nm and accelerating voltage 200 keV. To identify the crystal phase, the phase contrast method and selected area electron diffraction (SAED) with the locality up to 100 nm were used. Energy-dispersion atomic X-ray (EDAX) spectra were used for the elemental analysis. The locality used here was about 10−25 nm. For the HRTEM investigation, the glass samples were ground, dispersed in ethanol, applied onto perforated carbon substrates attached to a standard copper grid, and placed into the microscope chamber. The Mossbauer spectra were recorded at room temperature with the [57]Co in Cr γ-ra-

diation source in the constant acceleration mode. The $\alpha-Fe_2O_3$ standard was used to calibrate the spectrometer. Magneto-optical Faraday rotation was measured as a function of the electromagnetic wavelength, temperature, and external magnetic field. The plane polarization modulation of the light wave provided the FR measurement accuracy of ±0.2 min within limits from the absorption edge of each sample (about 700 nm) to 2200 nm. An external magnetic field was directed normal to the sample surface and varied from −0.5 to +0.5 T. The temperature was varied from 90 to 300 K at an accuracy of ±1 K. The magnetization was measured in the external magnetic field up to 5 T using a vibrating sample magnetometer.

Two types of X-ray diffraction spectra were observed for the glasses listed in Table depending on the heat treatment temperature and the Mn concentration: (a) spectra similar to that of an amorphous medium and (b) those characterized by distinct Bragg peaks. The broad halo observed for all samples is related to the amorphous matrix. Several narrow peaks are seen against the background of the amorphous halo (samples 4 through 8). The peak angular coordinates correspond to $MnFe_2O_4$ [21]. The peaks widths and intensities varied from sample to sample reflecting the difference in particle sizes. The average crystallite sizes are presented in Table. Though some samples (1 through 3) did not exhibit diffraction peaks, the large enough FR value and, especially, the finite value of the remnent FR (α_0/α_s) for those samples could not be ascribed to the isolated paramagnetic ions. Therefore, magnetic particles could be expected to arise in those samples, too. In this case, the particles could be too small or amorphous.

For all samples investigated, HRTEM images contain small regions of different density that can be attributed to magnetic particles. Typical HRTEM images of the glass fragments are illustrated in Fig. 1. For most samples, aggregates of 30−40 nm size consisting of several very small irregularly shaped or oval particles (of about 2 nm size) are observed (Fig. 1a). Isolated small particles are also presented. HRTEM images of some glasses show poly-disperse features (Fig. 1b). For a narrow region of technological conditions, isolated particles of cubic habitus are observed, relatively uniform in size (~50 nm), and distributed rather homogeneously in the matrix (Fig. 1c). In all the glasses, particles of 30−50 nm, as analyzed

by SAED (Fig. 1d) and compared to the bulk $MnFe_2O_4$, could be identified as ferrite of the cubic spinel structure. According to EDAX, the Mn/Fe ratio in the large particles is close to that in stoichiometric Mn ferrite. These elements are presented also in the matrix in the dissolved state. Thus, the HRTEM has proven the formation of nanosized particles close in structure to $MnFe_2O_4$ in the studied glasses. Therefore, these glasses can be considered as glass ceramics with nanosized crystal particles. A more regular structure was obtained for 1.5 % (mass) of Fe_2O_3 and 1.5 % (mass) of MnO in the charge and heat treatment at 560°C. This case seems to be unexpected. In fact, almost ideal ferrite single crystals distributed rather homogeneously in the matrix are formed at low content of paramagnetic additives and under conditions very far from those being used to synthesize the bulk ferrite crystals. Insignificant deviations in any of the technological conditions result in a dramatic change of particle characteristics, as is seen in Fig. 1 and Table. The Mossbauer spectrum for the sample No.4 containing the most regular crystal particles is shown in Fig. 2b. It consists of the sum of Zeemann sextets and the relax component. The spectrum has been analyzed by comparison with that of the bulk Mn-ferrite single crystal with partly inverse spinel structure. Each spectrum was computer-fitted assuming the minimal number of the non-equivalent Fe positions. Four non-equivalent Fe^{3+} positions were revealed in the bulk ferrite (Fig. 2a). The sextet with the isomer chemical shift $\delta = 0.30$ mm/s is ascribed to Fe^{3+} ion occupying the tetrahedral (A) positions. Three others ($\delta = 0.40$ mm/s) correspond to the octahedral [B] positions. According to the positions occupancies, the bulk ferrite structural formula could be presented as $(Mn_{0.78}Fe_{0.22})[Mn_{0.22}Fe_{1.78}]O_4$. The appearance of three non-equivalent octahedral positions in the partly inverse spinel is a well known phenomenon associated with the different surrounding of [B]-ions with magnetic ions of the second coordination sphere [22].

In the bulk ferrite, 32 % of B-cations have (4Mn + 2Fe) ions as first neighbors with the hyperfine field $H_{hf} = 43.7$ T. 42 % of B-cations have (5Mn + 1Fe) ions as first neighbors with the hyperfine field $H_{hf} = 41.8$ T. 18 % of B-cations have 6 Mn ions as first neighbors with the hyperfine field $H_{hf} = 39.8$ T. In spite of the same magnetic moment value of Fe^{3+} and Mn^{2+},
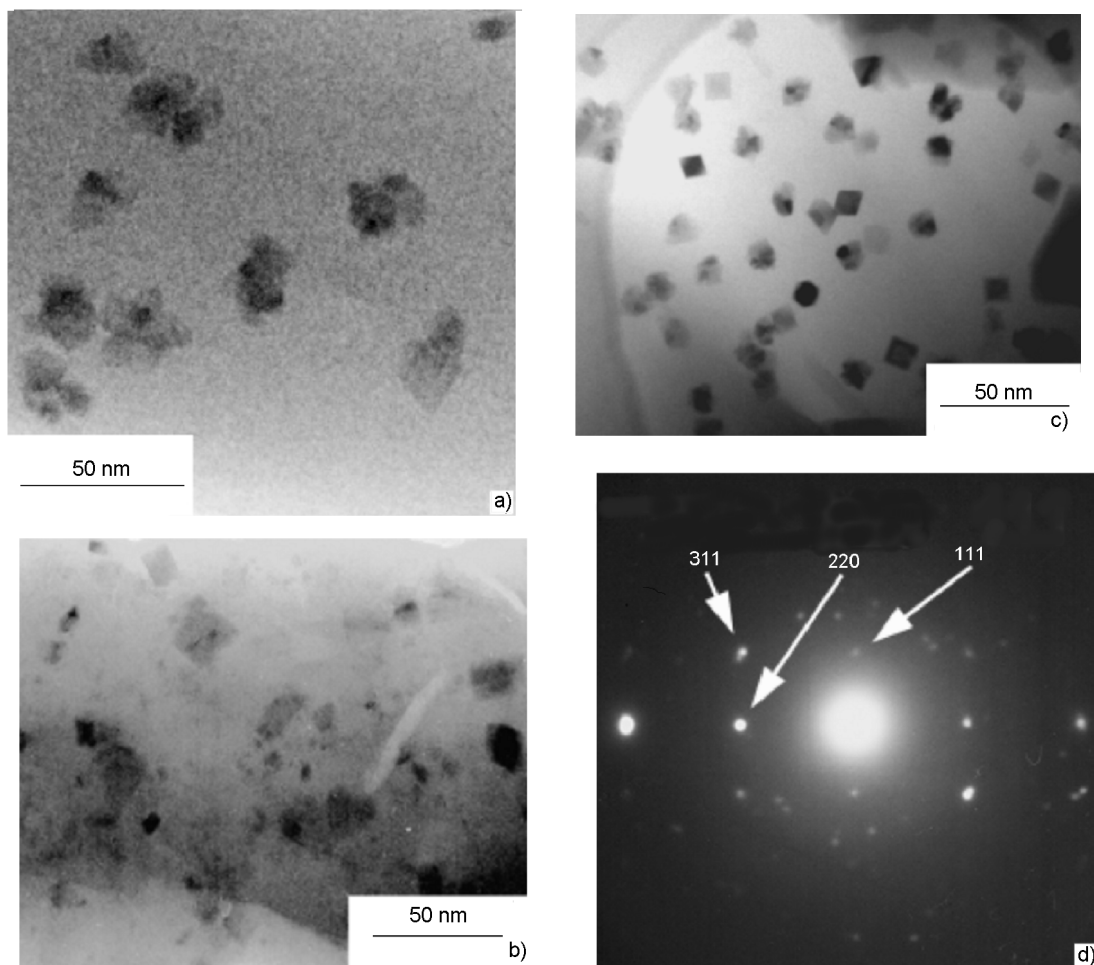
Fig. 1. TEM images of the particles for samples Nos.2, 10, 4(a–c, respectively) and SAED patterns from the separate particle for sample No.4.
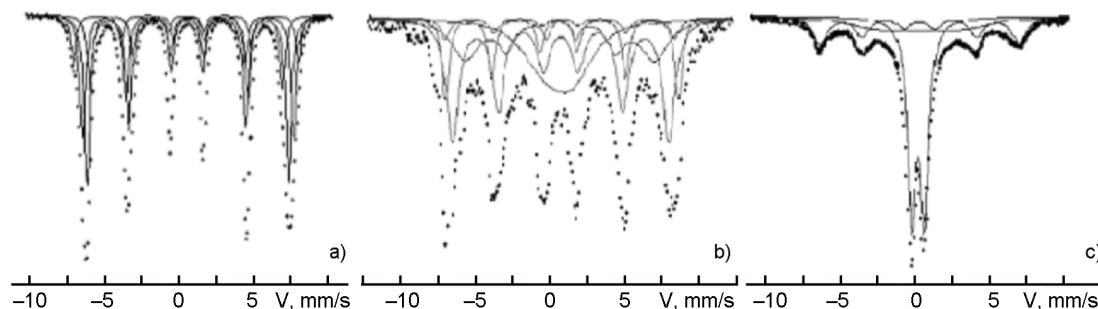

Fig. 2. Room temperature Mossbauer spectra of $MnFe_2O_4$ single crystal and samples Nos.4 and 10 (a–c, respectively).

$5\mu B$, interactions between Fe^{3+} and its nearest neighbors are different in the cases of $Mn(A) <-> Fe[B]$ and of $Fe(A) <-> Fe[B]$ from the viewpoint of the indirect exchange because the energy of the $O^{2-}-Mn^{2+}$ electron excitation is about 1.0 eV lower than that for $O^{2-}-Fe^{3+}$. Therefore, the exchange interaction integral of Mn^{2+} with neighbors is less than that of Fe^{3+}. So, the B-cation "distinguishes" Mn^{2+} and Fe^{3+} among A-type first neighbors, and this results in a series of sextets associated with this cation.

The spectrum of the glass 4 (Fig. 2b) can be approximated as the sum of four sextets and one relax component. The four different sextets evidence the formation of the defect Mn-ferrite. The rather wide spectral lines and smaller values of hyperfine fields
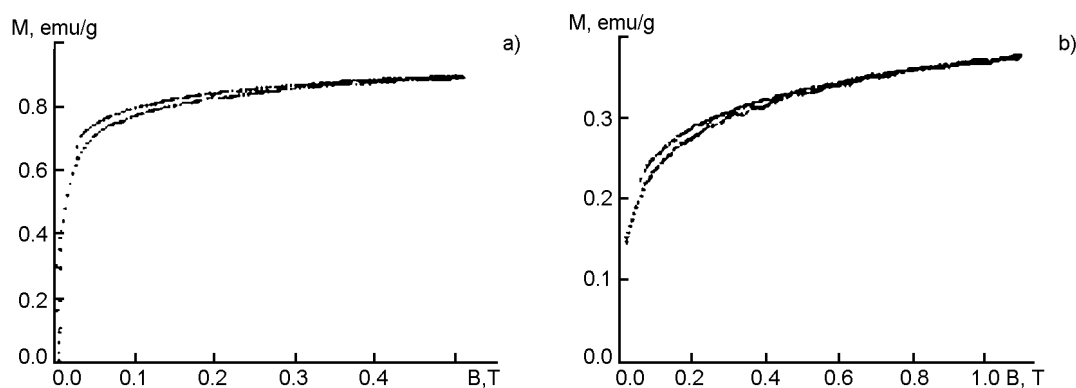
Fig. 3. Room temperature magnetization curves for samples: 4 (a) and 1 (b).

for the ferrite particles in this case testify to essential irregular environment of the Fe cations, for which the following factors may be responsible: variation of Mn/Fe ratio from particle to particle, entering Al into the ferrite composition, and different cation distribution among positions of spinel. For samples with less regular particles structure, intensity of sextet lines decreases. At the same time, a doublet appears that is typical of superparmagnetic state (Fig. 2c). The smaller are the particle dimensions, the higher is the intensity of doublet and lower intensity of sextet.

So, according to Mossbauer effect study, the crystalline particles possess a magnetic order, and this order is close to that of the bulk manganese ferrite, at least for the largest size particles. The difference in the first neighbors of the metal ions involved in magnetic interaction is shown to take place even for samples of the most regular particle structure. Dilution of ferrites by Al ions of the matrix may also take place here resulting in the change of the Mossbauer spectrum as compared to that of the bulk ferrite.

For all glass samples studied, nonlinear field dependences of the magnetization (M) are observed at room temperature with the sharp M increase in the relatively low magnetic field and the approach to saturation in 0.2–1.0 T fields (Fig. 3). Such a behavior is typical of materials possessing ferromagnetic or ferrimagnetic order. Two limiting cases are distinguished: (i) magnetization increases sharply in low fields about 0.01– 0.02 T and approaches saturation at 0.1– 0.2 T, and the magnetic hysteresis is observed with the coercive field H_c of about 0.01 T (Fig. 3a); and (ii) magnetization increases gradually but nonlinearly with increasing external magnetic field (Fig. 3b)

but does not reach the saturation at magnetic field values up to 1.0 T, and the magnetization curve can be described by the Langevin law. The first type magnetization curve is observed for samples containing large crystalline particles, e.g., sample 4. This case is most interesting from the applied viewpoint: it is possible to prepare magneto-optical components with the finite Faraday rotation value in zero magnetic fields. The second type curve is typical of the samples containing small particles. For the majority of samples, the magnetization curves of intermediate shape between (i) and (ii) types are observed. Rectangular shape of the magnetization curve and regular distribution of particles in the sample 4 (Fig. 1c) allowed estimating of the particle magnetic moment. It has been found to be about 50 emu/g, i.e. it is essentially lower than the bulk Mn ferrite magnetization (80 emu/g at room temperature [23]). The lower particle magnetization comparing to that of the bulk ferrite corresponds to the lower hyperfine field determined using the Mossbauer spectrum. The second type magnetization curve seems to be due to the superparamagnetic behavior of the particles.

The Faraday rotation of oxide glasses containing paramagnetic inclusions is defined by two contributions: diamagnetic rotation of the glass matrix and the rotation associated with paramagnetic ions. The diamagnetic contribution of the glass matrix $22.5Al_2O_3 \cdot 22.5K_2O \cdot 55B_2O_3$ was shown before to be independent of the heat treatment and at least two decimal orders lower than in glasses containing paramagnetic additives and subjected to additional heat treatment. The matrix contribution was not taken into account here. Typical FR spectral dependences are shown in Fig. 4 together with FR spectrum of the thin $Mn_{1.6}Fe_{1.4}O_4$ film pre-
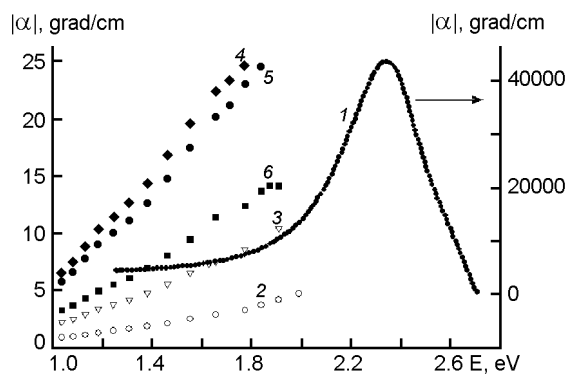
Fig. 4. FR spectra for samples 2–6 (left-hand scale) and for $Mn_{1.6}Fe_{1.4}O_4$ thin film [24] (right-hand scale).

Fig. 5. Some FR characteristics of samples 3–8 as functions of the particle average size estimated according to HRTEM images: FR value at $H = 0.35$ Å (2), FR value at $H = 0$ (3), and temperature coefficient C_t (1).

pared by the magnetron sputtering [24]. The FR sign is negative in all the cases. The FR value decreases monotonously as the light wavelength increases. Thanks to the low thickness of the ferrite film, authors [24] have managed to observe a maximum at 520 nm which was ascribed to the $^6A_{1g}(^6S) \rightarrow {}^4A_1$, ^4E_g (^4G) electron transition in Fe^{3+} ions. As it is seen from Fig. 4, the FR spectra of various samples contain no peculiarities in the spectral region investigated and differ from each other only in the FR decrease rate at increasing light wavelength. Strong changes of FR value are seen between different samples: similar to magnetization, the higher FR value is observed for the samples containing larger particles.

The nature of magneto-optical effects in ferrites remains unclear up to now because those are due to different electron transitions in different ions occupying different crystal sites. Maximum in the spectrum of magneto-optical effect squared in magnetization in $MnFe_2O_4$ epitaxial thin film [25] was observed near 520 nm which was associated with the same electron transition $^6A_{1g}(^6S) \rightarrow {}^4A_1$, ^4E_g. The supposition on the prevailing contribution in FR of Fe^{3+} ions occupying octahedral positions made in [24] is consistent with the larger amount of these ions as compared to Mn^{2+} ions in manganese ferrite. Coincidence of the FR sign and its spectral dependence for manganese ferrite with that for weak ferromagnetic $FeBO_3$ containing only Fe^{3+} ions in octahedrons [26] can be an argument for this supposition, too.

An interesting observation is now noteworthy, i.e. the strong FR increase with the temperature lowering (Table) that is characterized by the temperature coefficient $C_t = \alpha_{105}/\alpha_{300}$. In the interval 300–100 K,
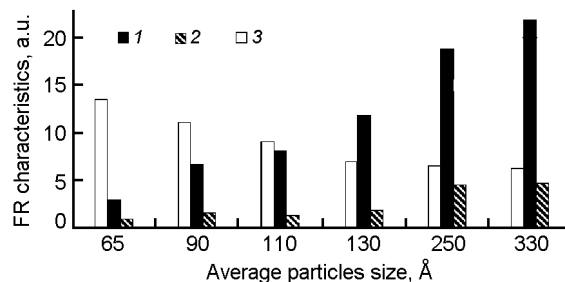
C_t is linear in temperature. For all samples, this increase exceeds essentially the magnetization increase observed when cooling the bulk manganese ferrite [23]. The higher C_t and lower FR value correspond to smaller and worse-shaped particles. Such a behavior can be compared with an idea of the spin disorder in the particle surface layer [27]: the surface layer contribution into the particle magnetic behavior increases as the particle size decreases. Fig. 5 demonstrates the correlation of the magneto-optical parameters with the average particle size D_{ev} for series of samples which differ from each other only by the heat treatment conditions: FR (α) and remnant FR (α_0/α) increase while C_t decreases with increasing D_{ev}. The correlations of such kind allows to suppose the magnetic behavior of glasses studied to be associated with individual characteristics of particles rather than with particle interaction.

The formation of magnetic particles in the glass matrix permits to define the material studied as a glass ceramics with unique combination of magnetic and magneto-optical properties. It is worth to make efforts to develop a new magneto-optical material on this basis. In this connection, the FR values (α) for the wavelength $\lambda = 1.5\,\mu m$ in the magnetic field $H = 0.02$ T are shown in Table. The maximal FR value of 4.2 grad/cm observed for the sample 4 in the magnetic field 0.02 T can be compared with data for magnetic semiconductors. For example, for $Hg_{1-x}Mn_xTe$ that was proposed as a cheap magneto-optical insulator for the 1.3–1.5 μm region such FR value is reached in magnetic field of 0.3 T [28].

To conclude, we have studied the magnetic and magneto-optical properties of assembly of ferrite nanoparticles dispersed in

potassium-aluminum-boron glasses doped with low concentrations of Fe and Mn oxides depending on the particle size, structure, composition, and spatial distribution in the matrix. In spite of a low concentration of paramagnetic elements, the glasses demonstrate magnetic and magneto-optical properties being typical of the magnetically ordered media. High resolution transmission electron microscopy (HRTEM), selected area electron diffraction (SAED), energy dispersive X-ray analysis (EDAX), X-ray diffraction spectra, and Mossbauer spectroscopy have been used to characterize the particles. The particles of 20−30 nm size have been shown to be crystalline. In particles of about 5 nm size, no distinct crystal structure has been revealed. The structure and composition of larger particles are close to those of $MnFe_2O_4$ manganese ferrite. The particle characteristics have been shown to depend strongly on the technological conditions. The conditions have been determined providing the formation of almost ideal ferrite single crystals. The correlation was also obtained between particle characteristics and magnetic and magneto-optical properties of the glass. Some features of these properties have been revealed, in particular, an increase of FR value with decreasing temperature exceeding essentially the magnetization increase in the bulk Mn ferrite [23] in the corresponding conditions. The increase extent depends on the average particle size and reaches 200 % for the smallest particles. Some of the glasses investigated are shown to have large FR in IR spectral region what could be of interest for the application in magneto-optical devices.

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Магнітні нано-сполуки на основі боратного скла, легованого залізом та марганцем

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Магнітні властивості, включаючи магнето-оптичний ефект Фарадея (ЕФ), досліджено на калій-алюміній-боратному склі, легованому окислами заліза та марганцю. Формування магнітних нано-часток доведено за допомогою електронного мікроскопа високої роздільної здатності. Спостерігався широкий діапазон розмірів часток, форми, структури та розподілення у матриці, в залежності від технологічних умов. Встановлено вузьку область умов, в якій формувалася майже ідеальна монокристалічна структура марганцевого фериту, розміром 40 нм, достатньо випадково розподілена у матриці. Отже, скло можна вважати нанокомпозитним матеріалом. Завдяки низькій концентрації парамагнітних елементів, скло зберігало оптичну прозорість та мало високий ЕФ в інфрачервоній та видимій спектральних областях, що дозволило створити нові магнето-оптичні матеріали для спектрального діапазону 1.0–1.5 μм.