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Laser modification of ferrite-garnet films

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High-power laser irradiation influence on the magnetic and structural parameters of ferrite-garnet films was investigated. It was shown that regardless of the laser irradiation method a decrease in the maximum deformation value and its gradient from the surface side occurs (caused by the movement of defects to the film surface). That is, during laser irradiation, its thermal effect is the determining factor for the relaxation and movement of defects, when a certain temperature gradient is formed as a result. Laser irradiation of ion-implanted FGF leads to electromagnetic waves' transmission coefficient increase by almost 20% in a wide spectral range (3500-400 cm^{-1}) that indicates reduced concentration of absorption centres. Crystal structure partial restoration as laser irradiation result leads to *a*- and *d*- sublattices superexchange interaction ordering that exhibited an increase of about 5-20% in the effective magnetic fields at the Fe^{57} nuclei for all used implantation doses.

Keywords: ferrite-garnet films, laser irradiation, magnetic and structural parameters.

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Introduction

Epitaxial single-crystal ferrite-garnet films (FGFs) constitute a wide range of magnetic media that are being intensively developed and researched for a wide variety of applications in information recording and processing devices and applied magneto-optics. [1]. Despite a comprehensive study of the properties of these materials, specific requirements for sensor devices require both the development of ideas about magnetization reversal processes and the physical phenomena associated with them, and the creation of a new class of FGFs that differ from the widely known ferrogarnets in magnetic anisotropy, in particular, have a direction of spontaneous magnetization close to the film plane. Ferrite garnets are described by the general formula $\{SR_i\}_3[Fe,Me]_2(Fe,D)_3O_{12}$, where the elements in curly brackets correspond to the dodecahedral, square-octahedral, and round-tetrahedral sublattices of garnet (c-, a-, and d-sublattices, respectively); R_i – rare earth elements; Me – elements that replace Fe in the a-sublattice (Al, Ga, Pb, rare earth elements with a small ionic radius); D – elements that replace Fe in the d-sublattice (Si, Ge, V, Al, Ga). Isomorphic substitution of Fe^{3+} and R^{3+} ions in the composition of ferrite garnets allows garnets with different magnetic properties to be obtained. In particular, the introduction of Bi^{3+} ions into the composition of ferrite garnet films leads to a gigantic increase in the polarization plane Faraday rotation. In turn, the development of new materials requires determining the necessary set of properties and accordingly establishing their optimal chemical composition and the possibility of targeted modification of physical characteristics. One of such modification possibilities is the use of powerful laser radiation (within the transparency range for these materials in particular) which is absorbed only by structural imperfections. This, in turn, allows optimizing such conditions and modes of laser irradiation where the imperfections influence on the specified characteristics of the structures will be minimal.

During laser annealing of ion-implanted layers by pulsed irradiation, a significant redistribution of the implanted impurity in the recrystallized layer occurs, and in most cases the impurity redistribution profile has the form of a step, i.e. an optimal distribution is formed for the impurities. Also concentration of impurities in the near-surface layer can have a value that significantly exceeds their equilibrium concentration in the solid solution.

Millisecond pulse annealing of implanted layers can be conditionally divided into annealing of the material, which under the ion bombardment has transitioned into an amorphous state and annealing of a partially disordered crystal lattice. Implanted layers are characterized by a very complex set of radiation damages, therefore, currently there is no simple mathematical model that would adequately describe the process of their annealing. In particular, annealing of a partially disordered structure is accompanied not only by the disappearance of defects but also by their rearrangement and the formation of new imperfections, more resistant to temperature increase [2].

I. Main part

Analysis of the influence of laser irradiation on magnetic and structural parameters was carried out using magneto-optical methods and X-ray analysis using a double-crystal diffractometer. The characteristics of the magnetic and structural parameters of the studied FGFs depending on the radiation energy density are given in Table 1.

Here E is the energy density of laser radiation, θ_F – specific Faraday angle of rotation of the plane of polarization, H_u – magnetic saturation field. $\Delta\theta_F$ and ΔH_u – absolute errors of the corresponding values,
$\varepsilon_0 = |\Delta d/d|$ – the value of the maximum micro-deformation in the film, $\varepsilon_0/\varepsilon$ – the ratio of the micro-strain value of the original sample to the micro-strain value of the sample after irradiation.

Changes in the interplanar distance and integral half-widths of the oscillation curves, as indicated by the ω-scanning results, at the same energy densities did not exceed the error limits. ($\Delta a = \pm\ 0.0005$ Å and $\Delta\beta = \pm\ 0.05\cdot 10^{-4}$ rad).

Based on the irradiated FGFs' characteristics analysis, it can be concluded that in the E = 10-34 J/cm^2 range the properties of garnet structures practically do not change. E increase in range 40-70 J/cm^2 and few irradiations (2 or 3 times) with the same energy lead to garnet films' crystal structure changes, that has been exhibited by X-ray curves analysis. Curves were built using ω-, θ- and θ/2θ-scanning schemes obtained from the plane (444) in comparison with the source sample (Table 2).

Growth of the integral half-width for the sample irradiated at E = 45 J/cm^2 (Table 2) may be associated with the decay of the complex defect and insufficient pulse time for annealing of secondary components [3]. Therefore, even with double irradiation with the same energy density, a decrease in the integral half-width of the oscillation curves is observed.

In the samples irradiated with single and double energy densities of 60 J/cm^2 (Table 2), integral half-width decrease of the reflection curves is observed compared to the original sample. The line broadening at triple irradiation is most likely due to the creation and accumulation of thermal shock-induced defects at the indicated energy densities in the pulse.

Irradiation of FGF with energy densities greater than 70 J/cm^2 leads to their destruction.

The interest in the destruction process is primarily due to technological needs, since this is one of the main factors limiting the maximum power of laser irradiation. In this regard, it is important to clarify the laser destruction mechanisms. There are two main mechanisms of destruction of different types of transparent materials under the influence of powerful laser radiation: 1) the mechanism of laser destruction associated with the heating of absorbing defects and 2) the inherent mechanism of laser destruction associated with impact and multiphoton ionization. Since Bi-substituted garnet is a dielectric, the dominant source of laser destruction will be absorbing impurities and inclusions, which are present in sufficient quantities in these magneto-optical materials.

Table 1.

Magnetic characteristics and structural parameters of laser-irradiated FGFs

E, J/cm^2	θ_F, deg/µm	θ_F, deg/µm	Saturation field H_u, A/m	ΔH_u, A/m	ε_0,×10^{-4}	$\varepsilon_0/\varepsilon$
0	1.20		220		7.94	1.00
10	0.96		231		7.94	1.00
17	1.04	±0.05	226	±5	7.22	1.10
24	1.10		226		7.78	1.02
34	1.08		228		7.94	1.00

Table 2.

Relative changes in the integral half-widths of X-ray diffraction curves from laser-irradiated FGFs

E, J/cm^2	(444), ω-scan.		(444), θ-scan.	(444), θ/2θ-scan.	
	film	substrate	film	film	substrate
0	1.00	1.00	1.00	1.00	1.00
45	1.11	1.16	1.49	1.29	1.14
2x45	0.93	0.79	1.05	1.16	0.93
E, J/cm^2	(444), □-scan.		(444), θ-scan.	(444), θ/2θ-scan.	
	film	substrate	film	film	substrate.
0	1.00	1.00	1.00	1.00	1.00
60	0.66	0.66	0.98	0.93	0.80
2x60	0.53	0.48	0.59	0.75	0.61
3x60	0.58	0.59	0.93	1.03	0.98
Error limits	±0.05				

An important aspect of laser destruction is the probabilistic nature of this process, since it is due to the random spatial distribution of absorbing defects, which have different characteristics and therefore lead to different thermal breakdown thresholds.

Defects present in garnet have a significant absorption coefficient and cause local heating, which leads to the destruction of the material in the vicinity of the defect.

The presence of metastable defects is characteristic of the structures of Bi-substituted FGFs, and they can be detected using absorption spectra in the IR region. Interestingly, metastable defects are characterized by a narrow stability region and will disappear at low energy densities in the pulse. When transitioning to the ground state, an irreversible rearrangement of the defect occurs and the metastable states disappear.

The results of studying the nature of electro- and mass transfer in complex oxides with a garnet structure show that the most disordered in them are the sublattices of the components of the *a*-, *d*-positions and oxygen. If the *d*-position contains a *p*-element (Si, Ge), then atomic defects (vacant sites, atoms displaced from a site to an interstitial site in the form of impurity atoms) dominate among others. At the same time, the nature of the dominant types of atomic defects does not change, since the latter are a function of the crystallochemical and energy features of the garnet structure.

Fig. 1 and Fig. 2 show the transmission spectra in the region of 0.19-1.10 μm, where all local minima are responsible for individual contributions from the absorption of individual impurities. Impurity contributions in some cases overlap, which complicates the interpretation of the spectra. The formation of transmission spectra, in addition to substituted atoms and impurities, is influenced by dislocations at densities of 10^7-10^8 cm^{-2}, deviations from the stoichiometry of cations and oxygen, surface irregularities are due to the peculiarities of epitaxial growth. Absorption by dislocations is associated with a decrease in the symmetry of the oxygen environment of Fe^{3+}.

The experimental results are explained by the presence of a large number of impurity atoms and vacancies present in Bi-substituted FGFs. The most common impurities in Bi-substituted garnets, in addition to bismuth, include Pb^{2+}, Pb^{4+}, Fe^{2+}, Fe^{4+} ions. They are mainly formed during the growth of these single crystals and are determined by the technology used, the composition of the charge and solvent, the purity of the starting components, etc.

From Fig. 1 it is seen that the presence of bismuth ions leads to intense additional absorption in the region of *l* < 0.6 μm. The most probable contribution of bismuth to the additional absorption has a maximum in the region of *l* = 0.29-0.34 μm (29000-34700 cm^{-1}), which is due to the transition of ions Bi^{3+} – ${}^1S_0 \rightarrow {}^3P_1$, as well as an intense transition with charge transfer between ions. Bi^{3+} i Fe^{3+}.

Local minima present at l = 0.55 μm (18000 cm^{-1}) and 0.31 μm (32000 cm^{-1}) and they are responsible for the additional absorption due to intense $Pb^{2+} + Pb^{4+} + h\nu \rightarrow Pb^{3+} + Pb^{3+}$ transitions that emerge only at high Pb concentrations (0.05 per formula unit). An abnormally high concentration of lead could have appeared as a result of samples overcooling during

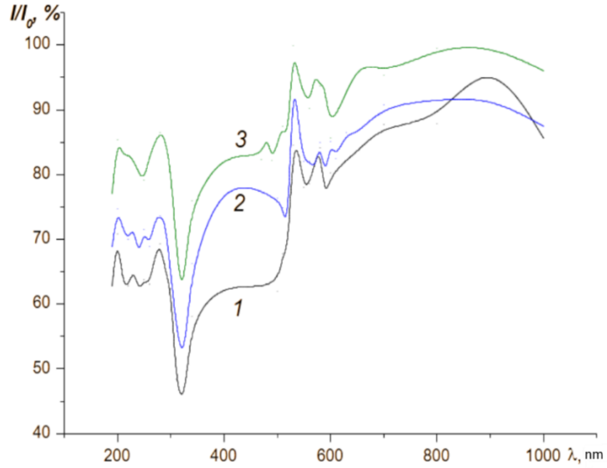

Fig. 1. Transmission spectra of FGF samples: 1 – original unirradiated; 2 – irradiated with pulse energy density of 45 J/cm^2; 3 – double irradiation of 45 J/cm^2.

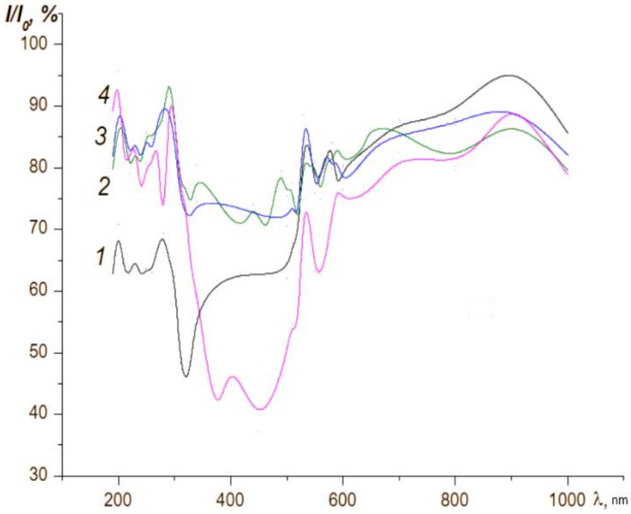

Fig. 2. Transmission spectra of FGF samples: 1 – original unirradiated; 2 – energy density in the pulse 60 J/cm^2; 3 – double irradiation of 60 J/cm^2; 4 – triple irradiation of 60 J/cm^2.

their growth by the method of liquid-phase epitaxy, which leads to a significant occurrence of Pb (where PbO was used as a solvent). Since lead occupies dodecahedral positions in garnet, part of it will be located in the c-position, and part will act as interstitial atoms. Taking into account the above, the chemical composition would be more correct to write as $(YSmCaBi)_{3-x}Pb_x(FeGeSi)_5O_{12}$, where $x \geq 0.01$.

Laser irradiation of FGF leads to a decrease in absorption (for which Bi^{3+} ions are responsible) while the absorption responsible for lead remains unchanged (Fig. 1 and Fig. 2), i.e., laser irradiation with the specified parameters cannot change the energy state of impurity lead.

Si^{4+} and Ca^{2+} ions and also oxygen non-stoichiometry leads to Fe^{2+} and Fe^{4+} ions emergence. Additional absorption by Fe^{2+} ions causes the appearance of an intense band in the region *l* < 0.6 μm and a shallow tail ending in the IR region. The intense absorption in the long-wave region of *l* < 0.6 μm is caused by the enhancement of pair transitions of Fe^{3+} ions in the octahedral and tetrahedral positions of the garnet lattice due to the mixing of the wave functions of the excited states of Fe^{3+} and Fe^{2+} ions. The shallow tail of additional absorption in the region l > 0.6 μm is associated with the Verwey hopping mechanism in $Fe^{3+} – Fe^{2+}$ pairs.

The appearance of a deep absorption minimum in the region l ≤ 0.6 μm for triple-irradiated (60 J/cm^2 energy density) samples is most likely associated with the appearance of Fe^{4+} ions.

Thus, the changes obtained demonstrate the fact that during irradiation not only the energy density magnitude in the pulse is important but also the method of irradiation, i.e. the use of multiple irradiations. The considered absorption mechanisms allow not only to modify the structure of Bi-substituted garnets using laser irradiation but also to correlate the properties of the films.

In order to eliminate radiation defects formed as a result of ion irradiation and increase the thermal stability of the structure, also partially remove mechanical stresses and accordingly improve magnetic characteristics, La and Ga-substituted FGFs (irradiated with YAG: Nd^{3+}- laser pulses) were studied. Laser annealing was carried out by a laser operating in the Q-switched mode with a pulse radiation energy of E = 0.04 J, a pulse duration τ = 15 ns and a pulse repetition frequency f = 56 Hz. The irradiation duration varied within 25–35 seconds. The films were irradiated with a laser both from the implanted and from the opposite side.

Given that for the studied films $hc/\lambda < E_g$ (λ = 1.06 μm – wavelength of laser radiation, E_g = 2.8 eV – band gap width), the energy of laser radiation is absorbed mainly by imperfections of the crystal structure, formed both during the growth of ferrite-garnet films and generated by ion implantation. It is obvious that the defects concentration in the ion-implanted FGF layer is several orders of magnitude higher than in the non-implanted one and therefore, the effect of laser irradiation is most fully manifested in this layer.

The condition $hc/\lambda < E_g$ (absorption is specific to the defective layer) allows the implanted layer to be irradiated from the non-implanted side as well that provides corresponding advantages in terms of energy efficiency of the process and eliminates the influence of the environment.

Laser irradiation stimulates diffusion and recombination processes and thus partial or complete elimination of radiation defects formed as a result of ion irradiation occurs. It is possible either by recombination of near pairs (most likely oxygen-anion vacancy) and by the defects movement to the drain surface. The most likely cause of radiation defect migration is considered to be diffusion that can be caused by excessive vacancy concentration or by the movement of radiation defects in interstitials.

In order to study the relative change in the concentration of defects of different types formed in the films during implantation and their relaxation under the action of laser irradiation of the studied samples [4], IR transmission spectra of films of the composition $Y_{2.8}La_{0.2}Fe_{4.545}Ga_{0.455}O_{12}$ with a thickness of 2.44 μm were recorded in the region of 2.5-25 μm (4000-400 cm^{-1}) before and after laser irradiation. Fig. 3 presents IR spectra from implanted films subjected to laser irradiation from the implanted and non-implanted sides, respectively. For comparison, the figure shows the IR spectrum from the non-irradiated implanted sample.

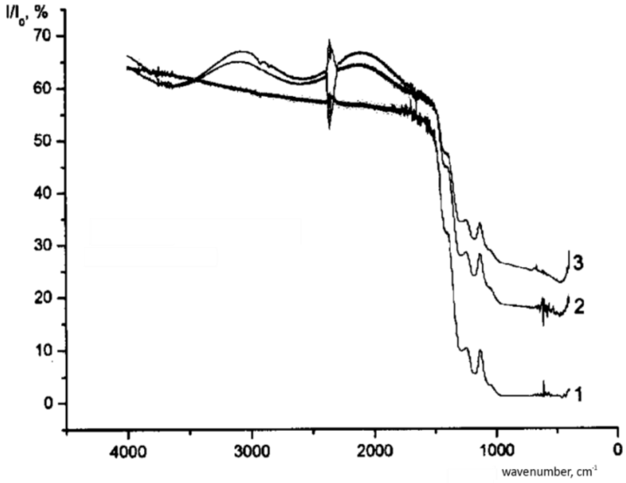

Fig. 3. IR transmission spectra of films of the type $Y_{2.8}La_{0.2}Fe_{4.545}Ga_{0.455}O_{12}$: 1 – unirradiated implanted sample (D = 4·10^{13} cm^{-2}, E = 90 keV); 2 – irradiated with a laser from the implanted side (D = 2·10^{13} cm^{-2}, E = 90 keV); 3 – irradiated with a laser from the non-implanted side (D = 2·10^{13} cm^{-2}, E = 90 keV).

ZIG single crystals have a transparency window in the region of 1-6 μm. Several narrow absorption peaks in this range are possible due to the substitution of yttrium in the c-sublattice by rare-earth metals (крім Lu^{3+}, Gd^{3+}, La^{3+}). The increase in transmission after laser treatment in the region of 2.5–6 μm (4000–1660 cm^{-1}), is probably due to the fact that against the background of a “cold” lattice, the energy absorbed by the defect allows it to be transferred to a different charge and energy state that leads to its annihilation with another defect. In the region of 4.3 μm (2300 cm^{-1}) an interference reflex is observed: apparently due to the interference maximum of transmitted waves on a film with a thickness of 2.44 μm.

In the range of 7-25 μm (1400-400 cm^{-1}) for the non-irradiated laser-implanted fluorine sample with a dose of 4·10^{13} cm^{-2}, a sharp decrease in transmittance is observed. It is apparently associated with crystal lattice defects that include growth mechanical stresses caused by a mismatch between the parameters of the film and the substrate, a decrease in the crystal lattice parameter of the ferrite-garnet film caused by the introduction of small-sized Ga^{3+}, a deviation from stoichiometry in the anionic sublattice caused by fluorine implantation. After laser irradiation, the transmittance of the films in this range (7-25 μm) increases by almost 20-25% (Fig. 3, curves 2 and 3), which indicates a significant decrease in the absorption centers’ number. They are probably anionic vacancies formed during ion implantation and as a result of laser irradiation they annihilate with oxygen and fluorine ions moving towards the surface. The transmittance of films irradiated by a laser from the implanted side is lower than that of films irradiated from the non-implanted side, which is associated with a smaller number of defects that relax under a given irradiation geometry.

Partial recovery of the crystal lattice of the FGF due to relaxation of radiation defects leads to the recovery of the magnetic microstructure [5], which is reflected in the Mössbauer spectra obtained from implanted *La, Ga*-substituted ferrite garnet films subjected to laser irradiation from both the implanted and non-implanted sides.

Mössbauer spectra of Fe^{57}, were obtained at room temperature in the constant acceleration mode. A source of γ-quanta Co^{57} in a chromium matrix with an activity of ~90 mKu was used. Registration of conversion electrons was carried out by a flow counter (gas mixture composition 96% He + 4% CH_4); calibration of the spectra was carried out relative to α-Fe.

The experimental spectra (Fig. 4) were approximated by two sextets (one a-position and one *d*-position) and a doublet D, which corresponds to Fe^{57} atoms that do not participate in the superexchange interaction and are in the paramagnetic state.

The choice of the background parabolic shape was due to the manifestations of geometric factors when collecting the experimental spectrum, $t_{collection}$ = 48 h. Qualitative approximation of the unimplanted film spectrum is possible under the condition that the Fe^{57} nuclei with the number of magnetic neighbors ≤ 2 are in the paramagnetic state.

A comparative table of decoding of Mössbauer spectra before and after laser irradiation is given in Table 3.

As can be seen from Table 3, the effective magnetic field on the iron nuclei for the a- and d-sublattices as a result of laser irradiation increases by 5-20%. With increasing ion implantation dose the monotonically decreasing nature of the magnetic field dependence on the implantation dose becomes practically unchanged, that indicates ordering in the system caused by laser irradiation. Since the effective magnetic fields are practically the same for samples irradiated with a laser both from the implanted and non-implanted sides (Table 3), it can be stated that laser radiation is absorbed by impurities and defects in the crystal structure and is transparent to the rest of the crystal region.

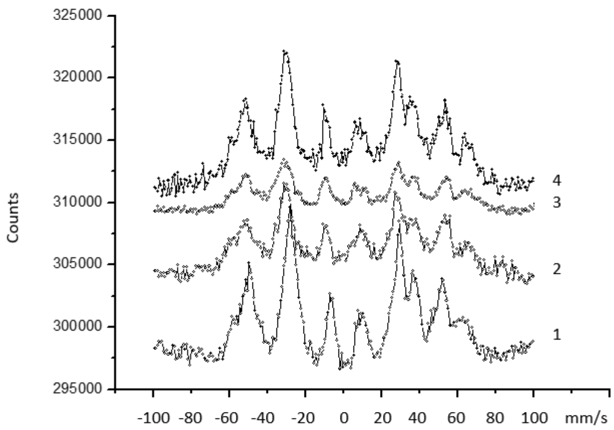

Fig. 4. Mössbauer spectra of laser-irradiated La, Ga-substituted FGFs implanted with F+ ions at a dose of $1{\cdot}10^{13}$ cm^{-2}: 1 – original unirradiated sample; 2-4 – irradiated with a laser from: 2 – non-implanted side; 3 – implanted side; 4 – non-implanted side (study from the implanted side).

As a result of laser irradiation, there is an intensities redistribution of the spectrum magnetic components from the *a*- to the *d*-sublattice, while the intensity of the paramagnetic doublet remains practically unchanged. Thus, it can be stated that upon laser irradiation of F^+-implanted FGFs, there is a redistribution of Ga^{3+} and Fe^{3+} ions between tetrahedral and octahedral positions in the lattice. The relative number of d-positions filled with Fe^{3+} grows while the additional filling of a-positions with Fe^{3+} decreases. It is obvious that upon laser irradiation, the knocked-out Fe^{3+} and Ga^{3+} ions do not occupy their previous positions, and Fe^{3+} prefers the *d*-position, and Ga^{3+} prefers the *a*-position. At the same time, the specified

Table 3.

Calculated parameters of partial Mössbauer spectra of iron FGF of composition $Y_{2.8}La_{0.2}Fe_{4.545}Ga_{0.455}O_{12}$ irradiated by laser

Dose, cm^{-2}	*H, kE*				*S, %*		
	a_1-position	a_2-position	a_3-position	*d*- position	*a*- position	*d*- position	doublet
For samples not irradiated with laser							
0	429±3	385±4	306±2	352±1	40.7	56.0	3.3
1×10^{13}	430±2	395±2	303±2	352±1	39.0	55.8	5.2
2×10^{13}	420±2	381±2	301±2	347±0,5	39.1	55.3	5.5
6×10^{13}	380±2	347±2	271±3	331±2	39.1	54.9	5.9
1×10^{14}	335±2	322±2	213±3	290±2	25.7	52.6	11.7
For samples irradiated with laser from the implanted side							
1×10^{13}	437±1	–	–	368±1	33.47	60.74	5.79
2×10^{13}	422±1	–	–	358±1	32.93	63.19	3.88
6×10^{13}	405±1	–	–	348±1	34.42	59.29	6.29
1×10^{14}	377±1	–	–	346±1	29.34	58.26	12.4
For samples irradiated with laser on the non-implanted side							
0	436±1	–	–	366±1	29.83	64.31	5.86
1·10^{13}	426±1	–	–	365±1	29.34	64.08	6.57
2·10^{13}	437±1	–	–	358±1	29.18	65.79	5.04
6·10^{13}	396±1	–	–	351±1	26.26	68.34	5.39
1·10^{14}	367±1	–	–	349±1	26.19	61.06	12.75

redistribution occurs more actively during laser irradiation from the non-implanted side that confirms our conclusions about more intense diffusion during irradiation in this way. As a result of the redistribution of ions, the saturation magnetization at room temperature increases. The characteristic temperature ranges in which various mechanisms of magnetization change under the action of laser irradiation dominate can be divided into three ranges: high-temperature - near the Curie point (T_C), intermediate - from 300 K to T_C and low-temperature - below 300 K. From the analysis of the studies it follows that in the first and second areas the model of the thermal mechanism of magnetization change under the action of laser irradiation is dominant, and therefore the thermal model of laser irradiation, in which the temperature gradient is the determining factor of relaxation and movement of defects, well explains most of the experimental facts obtained when considering Mössbauer spectra, including the effects of diffusion redistribution of F^+ ions introduced into the film under the action of laser pulse energy.

Conclusions

1. It was established that regardless of the laser irradiation method of FGF, there is a decrease in the deformation maximum value and its gradient from the surface side which is caused by the defects movement to the film surface, i.e., during laser irradiation, the temperature gradient is the determining factor of relaxation and movement of defects.

2. Laser irradiation of ion-implanted FGFs leads to electromagnetic waves' transmission coefficient growth by almost 20% in a wide spectral range (3500-400 cm^{-1}), that indicates an absorption centers' concentration decrease.

3. Partial restoration of the crystal structure due to laser irradiation leads to ordering of the superexchange interaction of *a*- and *d*-sublattices, that exhibited in an increase of ~5-20% of the Fe^{57} nuclei effective magnetic fields at all used implantation doses.

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Лазерна модифікація ферит-ґранатових плівок

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Досліджено вплив потужного лазерного випромінювання на магнітні та структурні параметри ферит-гранатових плівок. Показано, що незалежно від методу лазерного опромінення відбувається зменшення максимальної величини деформації та її градієнта з боку поверхні (зумовлене рухом дефектів до поверхні плівки). Тобто під час лазерного опромінення визначальним для релаксації та переміщення дефектів є його тепловий вплив, у результаті якого утворюється певний температурний градієнт. Лазерне опромінення іонно-імплантованого ФГП призводить до підвищення коефіцієнта пропускання електромагнітних хвиль майже на 20% у широкому спектральному діапазоні (3500-400 см$^{-1}$), що свідчить про зниження концентрації центрів поглинання. Часткове відновлення кристалічної структури в результаті лазерного опромінення призводить до надобмінного впорядкування a- і d-підграток, що демонструє зростання ефективних магнітних полів на ядрах Fe^{57} приблизно на 5-20% для всіх використаних доз імплантації.

Ключові слова: ферит-гранатові плівки, лазерне опромінення, магнітні і структурні параметри.