X-RAY DIAGNOSTICS OF THE STRUCTURE OF NEAR-SURFACE LAYERS OF ION-IMPLANTED MONOCRYSTALLINE MATERIALS

Розроблено методику отримання інформації про розподіл параметрів кристалічної структури по товщині приповерхневого іонно-імплантованого шару, типи та характеристики радіаційних дефектів (розмір, концентрацію та ін.). Встановлено вплив основних дифракційних параметрів на криві дифракційного відбивання, що дало можливість розробити алгоритм наближення експериментальних кривих дифракційного відбивання теоретично обчисленими. Показано, що при невеликих дозах імплантації на інтенсивність кривих дифракційного відбивання величина коефіцієнта екстинкції μ_{ds} найбільше впливає за межами додаткової осциляційної структури, а величина статичного фактора Дебая-Валлера E – в межах останніх осциляцій додаткової осциляційної структури, які відповідають максимальній деформації. При цьому для характеристики дефектної системи необхідно аналізувати дифузну складову, використовуючи частину кривої дифракційного відбивання, яка розміщена за додатковою осциляційною структурою і в якій вклад когерентної складової є мінімальним. Методику апробовано при аналізі імплантованих іонами бору плівок залізо-ітрієвого гранату. Представлений підхід дає можливість отримати велику кількість інформації про структуру іонно-імплантованого шару, оскільки використовує статистичну динамічну теорію розсіяння рентгенівських променів, яка враховує наявність дефектів кристалічної структури будь-яких типів та розмірів. Також даний підхід дає можливість використовувати всю інформацію, яку несуть в собі криві дифракційного відбивання, та оцінити ступінь однозначності визначених параметрів

Ключові слова: профіль деформації, дифракція рентгенівських променів, іонна імплантація, дефекти структури, статистична динамічна теорія розсіяння рентгенівських променів

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1. Introduction

X-ray structural analysis is one of the main methods for researching the structure of near-surface layers of monocrystalline materials with the ion-implanted near-surface layer [1]. Ion implantation leads to defects in the near-surface layer and, accordingly, change in the interplanar distance in it [2]. The effectiveness of using of X-ray diffractometry in the investigation of ion-implanted monocrystals and films is due to the fact that additional peaks and influences (additional oscillation structure) are formed near the main Bragg's peak from the ideal part of the single crystal. They are the result of the variable interplanar distance in the thickness of the implanted layer and the interference effects.

An analysis of the additional oscillatory structure on the rocking curve from ion-implanted monocrystals allows obtaining information on the distribution of radiation defects and deformations. Structural changes in the near-surface layer unambiguously affect the shape of the rocking curves. Solving the inverse problem, that is, determining

the structure of near-surface layers from the rocking curve, encounters considerable difficulties. Such a situation exists because for the unambiguous description of the structure of the near-surface layer from the rocking curve it is necessary to know the complex amplitude of reflection in the entire angular interval, and the experimentally measured coefficient is only a module of this magnitude.

Therefore, further research in the direction of X-ray structural diagnostics of near-surface layers of monocrystals is an actual task.

2. Literature review and problem statement

Although the analysis of this additional oscillatory structure is carried out for a long time [3–5], however, the problem of correctness and uniqueness of the results obtained in these articles remained open. It is also necessary to take into account the latest achievements in the description of X-ray scattering by crystals.

The analysis of near-surface layers of single crystals is currently carried out by means of the kinematic theory of X-ray scattering [6], dynamic theories based on the Takagi equations [7–9], and by means of the statistical dynamical theory of X-ray scattering [10, 11]. The statistical dynamical theory makes it possible to take into account the presence of specific types of defects in the structure and does not impose a limit on the size of the defect. It is worth noting that in the determination of strain profiles, diffuse scattering of X-rays on defects in a large number of publications is not taken into account [12].

The main approaches to the modeling of X-ray diffraction and algorithms for the determination of strain profiles are discussed in [13]. In this paper, an approach to rocking curve modeling is used, which is based on the semi-kinematic theory of X-ray diffraction in the case of Bragg geometry. Also the algorithm for the approximation of the theoretically calculated rocking curves to the experimental ones is described in detail. However, in this paper, as in most papers, diffuse scattering of X-rays on defects is not taken into account.

A very detailed and strict model of X-ray diffraction in single crystals with defects of any type and size was developed by the authors [10, 11] using a statistical dynamical theory. The types, sizes and profiles of defect concentration in the ion-implanted layer were determined. However, the method of minimizing the deviation of the theoretical rocking curves from the experimental ones is not specified. In general, in most publications this problem is limited to the phrase "the least squares method has been used" without analyzing the uniqueness of the results obtained. In determining the strain profiles, genetic algorithm [14] and annealing modeling algorithm [15] are also used. These methods allow determining the strain profile even without the "assumption" about its form. However, in analyzing the results published by the program, it is necessary to critically approve the physical validity of the obtained dependencies.

As practice shows, in the approximation of the theoretically calculated rocking curves to the experimental ones, often ambiguity arises in the determined parameters. This happens both through correlation between model parameters or intercompensation of some parameters, through the existence of experimental errors and errors in the calculations, and through stuck in local minima of the function

of the deviation of the theoretical diffractogram from the experimental one. Also, the source of ambiguity is the interference of X-rays on heterogeneities of the structure, in particular, in the amorphous layer.

Thus, in most publications the study of the ion-implanted layer is limited to the definition of deformation profiles and defect parameters are not determined. Also, the diffuse component of X-ray scattering on defects created by implantation is not taken into account and the analysis of the uniqueness of the determined parameters of the disturbed layer is not carried out. There is no analysis of the methods of approximation of the theoretically calculated rocking curves to the experimental ones. Therefore, there are reasons to believe that existing approaches do not fully cover all the problems that arise during the X-ray analysis of ion-implanted monocrystals and films. This is the reason for the need for further work towards the development of approaches for the correct determination of the structure of near-surface layers of implanted monocrystalline materials. To resolve these problems, this paper has been made.

3. The aim and objectives of the study

The aim of the work is to develop a method for obtaining information on the distribution of deformation in the thickness of the ion-implanted layer, the types and parameters of radiation defects, and the distribution of defects in the thickness of the implanted layer.

To achieve this aim, the following objectives were set:
– to analyze the influence of defect parameters in the ion-implanted layer on the form of rocking curves;
– to develop an approach to the analysis of the rocking curves, which would include determination of strain profiles and defect parameters of the crystalline structure of the ion-implanted layer, and evaluation of the uniqueness of the parameters obtained;
– to develop a method for minimizing the function of many variables for the given type of tasks to find the minimum of the function of the deviation of the theoretical diffractogram from the experimental one.

4. Method of determining the parameters of the crystal structure of the ion-implanted layer

4. 1. Modeling of theoretical rocking curves
For the realization of X-ray structural analysis of ion-implanted surface layers of monocrystals, various methods of scanning of the reciprocal-lattice points are used. One of the most common methods is ω-scanning with a wide slit in front of the counter – obtaining rocking curves.

To analyze the experimental rocking curves for defining parameters of defects in nonimplanted and implanted monocrystals and films, the simulation of diffraction of X-rays in nonperfect crystals was used. A statistical dynamic theory of X-ray scattering was used, which makes possible to take into account the presence of specific types of defects in the structure and does not impose restrictions on the size of the defects [16]. To do this, a special program has been developed in the C++ Builder, which allowed the modeling of theoretical rocking curves using the given defects parameters of the crystal. For the simulation of X-ray diffraction in the ion-implanted layer, the non-homogeneous near-surface layer

was divided into sublayers, in each of which defects were considered homogeneous distributed, and the parameters of defects, deformation and degree of amorphization were constant. Thus, the distribution of various parameters of the disturbed layer with depth (including deformation profile) was given in the form of stairs. In the figures, it is represented in the form of stairs or in the form of an averaging line.

For a comparison of the experimental and theoretical rocking curve, the convolution of the calculated theoretical rocking curve with the function of broadening of the spectrometer is made, and the polarization of the incident X-rays is taken into account.

Two models of the disturbed layer were considered:

– the strain profile (or defect profile) is set up functionally (use in cases, where some functional dependence is physically grounded) [17];

– the strain profile (or defect profile) is given in the form of stairs, each of which is characterized by width – sublayer thickness, and height – relative deformation or concentration of defects (use, when the profile is not known beforehand) [18].

For the analysis of experimental rocking curves, a complex of minimization of the function is used that characterizes the deviation of the theoretically calculated rocking curves from the experimental ones (in the simplest case, the mean square deviation (MSD)). In this case, the set of methods of minimization can be applied to several experimental rocking curves at the same time, and allows solving a multi-parameter problem.

Depending on the parameters of the defective subsystem, whose approximation should be carried out, two approaches are used for minimization:

– the first approach – when the approaching parameters are heterogeneous (this situation occurs, when the purposeful change of the values of the parameters of the functionally described strain profiles). This situation suits the first model of the disturbed layer;

– the second approach – when the approaching parameters are homogeneous and can be reduced to two types (this situation suits the second model of the disturbed layer).

4. 2. Algorithm for determining the parameters of the structure of the ion-implanted layer

For the initial determination of parameters of strain profiles, modeling of the ion implantation process was used. The modeling was done using the SRIM program. The profiles of defects obtained from modeling of ion implantation process can be described by a certain functional dependence. When implantation of ions of light and medium weight at low doses of implantation (the number of defects is negligible), it can be assumed that the strain profile is proportional to the profile of defects. Thus, the parameters of the defect profile that characterize the distances (for example, the thickness of the disturbed layer, the depth at which the maximum defects are located) can be directly used as parameters of the strain profile. From the relation between the parameters which characterize the concentration of defects, one can determine the relation between the parameters of strain profiles that characterize deformation in the ion-implanted layer (for example, maximum deformation, deformation on the surface).

From the experimental rocking curves from the angular distance $\Delta\Theta$ between the peak of the film and the last oscillation, by differentiating the Bragg's formula, the magnitude of the maximum deformation is approximately obtained

$$\frac{\Delta d}{d} = \Delta\Theta \, \mathrm{ctg}\Theta_B,$$

where Θ_B is the Bragg's angle for the undisturbed part of the single crystal. It is worth paying attention to the fact that this formula applies only to symmetrical reflections or in the case of the same crystal lattice deformation in all directions. When deformation occurs only in the normal to the surface, not only the distances between neighboring crystallographic planes, but also the angle of inclination of non-parallel to the surface of the planes change. This causes a change in the angular deviation of the diffracted ray:

$$\Delta\Theta = -\frac{\Delta d}{d}\big(tg\Theta_B \pm tg\psi\big),$$

where $\psi = \Theta_B - \Theta_{in}$, Θ_{in} is the angle between the plane of the surface of the single crystal and the incident ray. In the general case, when both the normal $\dfrac{\Delta d^{\perp}}{d}$, and the tangential $\dfrac{\Delta d^{II}}{d}$ components of the deformation depend on the depth, the angular deviation is equal to:

$$\Delta\Theta = -\left(\frac{\Delta d^{\perp}}{d} + \frac{\Delta d^{II}}{d}\frac{tg\Psi}{\cos\Psi}\right)tg\Theta_B \pm \left(\frac{\Delta d^{\perp}}{d} - \frac{\Delta d^{II}}{d}\right)tg\Psi.$$

A specially developed program was used to evaluate the unambiguity of the parameters of the strain profile. This program performs the sorting of all possible parameters of the strain profile P_1, P_2,..., P_p in a certain interval with a certain step. In this case, the multidimensional surface of the dependence MSD on the parameters P_i was obtained, which made it possible to estimate the uniqueness of the profile definition by the number of minima. The type of the profile chosen from certain considerations was further refined by the complex of gradient methods. In order to save time, it is possible to carry out parameter sorting using the semi-kinematic approximation and not to take into account the diffuse component if the value of the static Debye-Waller factor is close to unity. If the kinematic theory does not take into account absorption in the transition between sublayers, then the number of minima found by the kinematic theory is greater than the dynamic one. The reason for this is that when the sublayers change their places the result does not change, because in this method the usual sum of amplitudes from each of the sublayers is calculated. In the case, where the value static Debye-Waller factor is not close to unity or other parameters of the defective subsystem are not equal to zero and their depth change is also described by some function, the parameters of these functions may also be included in the sorting.

The specific local minimum of the MSD function was chosen from certain physical considerations, a satisfactory match between theoretical diffraction patterns and experimental or additional experimental studies. To clarify the parameters of the profile, a further "descent" into this minimum was carried out. As a starting profile, a set of parameters corresponding to the smallest MSD in this local minimum was chosen.

After choosing the starting approximation, the parameters of the disturbed layer are refined by minimizing the deviation of the theoretically calculated rocking curve from the experimental ones. This uses the first approach to minimizing the function of the MSD, which applies to the first

model of the disturbed layer. In this case, the refinement parameters are heterogeneous and describe the functional dependencies of the strain profiles or parameters of defects from the depth.

Given that the actual strain profile may differ somewhat from the used functional task, the next step of the approximation is used. This second stage applies to the second approach to minimizing the function of MSD, which is realized with the use of the second model of the disturbed layer. In this case, the strain profile was given in the form of stairs, which are described by two types of parameters: deformation and thickness of the sublayers [18]. Also, this approach to minimizing the function of MSD is used in the analysis of a mosaic layer on the surface of the crystal, where the parameters are the number of blocks and the angle of inclination of the blocks. In a model of a mosaic layer, it is assumed that the blocks are deformed in thickness, and some of the blocks are turned at some angles [19].

In the case of ω-scanning with a wide slit in front of the counter in the entire region of the additional oscillatory structure of the rocking curve, both coherent and diffuse scattering of X-rays from the near-surface disturbed layer is fixed. Correct separation of both components within an additional oscillatory structure is practically impossible. Therefore, for the analysis of the diffuse component of X-rays, a part of the rocking curve, which is located behind an additional oscillatory structure, and in which the contribution of the coherent component does not exceed 5 %, was used.

To describe the distribution and characteristics of defects with depth, the inhomogeneous near-surface layer was divided into sublayers, in each of which defects were considered homogeneously distributed. These are the same sublayers, to which the strain profile was divided. The distribution of defects in the ion-implanted layer is given in the form of stairs that describe two types of parameters: the concentration of defects and their radii. Minimization of the function of MSD was carried out by changing the concentration and radii of defects according to the second approach [20].

4. 3. The method of approximation of the theoretically calculated rocking curves to the experimental ones

Refinement of the parameters of the disturbed layer is carried out with the help of a set of methods for minimizing the deviation of theoretical diffractogram from the experimental ones. It is expedient to develop an algorithm that combines several different methods of minimization. In program realization of the given algorithm, three different methods of minimization were used which considerably increased the efficiency of use of minimization procedure. Therefore, on the example of the second approach to minimizing the function of MSD in determining the strain profile, we describe this algorithm in more detail.

This approach allows you to calculate the profile without using any predefined functional specification of the profile type, and correctly applied conditions allow minimizing the ambiguity that may arise in the calculation of strain profiles. By changing the thickness of the sublayers and relative deformation, the mean square deviation of the theoretical rocking curve from the experimental one was minimized.

The algorithm of MSD minimization is convenient to consider in a 2m-dimensional space whose coordinates are

the thickness of the sublayer of the disturbed layer L_i and the relative deformations D_i ($D_i = \frac{\Delta d_i}{d}$, $\Delta d_i = d_i - d$, where d_i and d, are the interplane distance in the sublayer and in the undisturbed part of the crystal, respectively). Each point of space (L_i, D_i) corresponds to a certain value of MSD.

To start the calculation, you can select the start profile of any type, that is a certain point of the *2m*-dimensional space L_i, D_i), and obtain the value of MSD for the chosen starting point. However, in order to choose the starting point, it is expedient to use the approximation results from the first approach of MSD minimization. Then, by changing the parameters L_i and D_i alternately to the certain value in the direction of the larger and smaller values, and taking into account the limitation on the parameters $L \geq 0$, the MSD for the new *4m* points is calculated. Among these points, a point (profile) with a minimum MSD is selected and remembered.

In view of the fact that most of the machine time is mainly used for the calculation of the MSD, the gradient method and the Gauss least squares method are also used to minimize the function of the MSD. Using a combination of these methods is discussed in detail in [21], where a similar algorithm was effectively used to analyze diffractograms from polycrystalline materials.

5. Determination of parameters of the ion-implanted layer on the example of ferrite-garnet films

The use of the described method can be demonstrated by an analysis of experimentally obtained rocking curves for reflections (444) and (888) from films of iron-yttrium garnet implanted by B^+ ions with energy $E=80$ keV and dose $D=3 \cdot 10^{14}$ cm^{-2}. X-ray structural investigations were carried out using the methods of two-crystal diffractometer in CuKα1 radiation.

The lattice parameter of the substrate determined by the Bond method is 12.382 Å, and the film is 12.376 Å.

For materials with a garnet structure, a number of features are characterized, in particular, the possibility of creating near-surface layers with large deformation and with a small amorphization and the presence of experimentally established two centers of disturbing [17, 22]. The defect profile has two components: defects, which result from collisions with the target nuclei (due to nuclear losses of ions energy) and defects, which result from electronic losses of ions energy. Modeling using the SRIM program allows only the first mechanism to be taken into account, therefore, defect formation with the second mechanism was calculated according to [22]. The calculated defect profile is presented in Fig. 1, *a*.

Taking into account that at small doses, the strain profile is proportional to the defect profile, which can be described by the sum of the asymmetric and decreasing Gaussian curves, the strain profile is also described as the sum of the asymmetric and decreasing Gaussian curves [22]:

$$D = \begin{cases} D_{max}^N \exp\left[-\left(z - R_P^N\right)^2 / \left(\sigma_1^N\right)^2\right], & \text{if} \quad z < R_P^N \\ D_{max}^N \exp\left[-\left(z - R_P^N\right)^2 / \left(\sigma_2^N\right)^2\right], & \text{if} \quad z \geq R_P^N \end{cases} + D_{max}^E \exp\left[-\left(z - R_P^E\right)^2 / \left(\sigma^E\right)^2\right],$$

$D = \frac{\Delta d}{d}$, h is the distance from the surface, R_P^N is the merging point of two Gaussian curves, and σ_1^N, σ_2^N and σ^E

are parameters that describe the Gaussian curve's width at half maximum. The upper indices N and E mean that these parameters characterize the components of the profile associated with nuclear and electronic losses of ions energy, respectively. The first term is described by the asymmetric Gaussian curve, which is part of the profile associated with nuclear losses of ions energy. The second term is described by the downward Gaussian curve ($R_p{}^E<0$), which is part of the profile associated with electronic losses of ions energy, which is monotonically declining with depth.
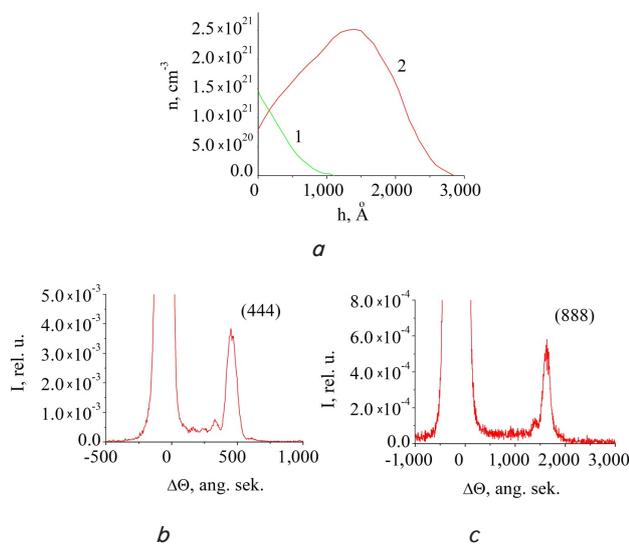

Fig. 1. Output data for determining the parameters of the ion-implanted layer: *a* — theoretical calculations distributions of the concentrations of defects formed as a result of electronic losses of ions energy (1) and nuclear losses of ions energy (2); *b, c* — experimental rocking curves from ion-implanted film of iron-yttrium garnet: reflexes (444) and (888), respectively

In materials with a garnet structure, an asymmetric reflex of high intensity is a reflex (880), corresponding to which planes lie at an angle of ≈35° to the plane of the film. In addition, the use of geometry of diffraction in which the angle of incidence ray ≈9° makes this reflex especially sensitive to the structure of thin near-surface disturbed layers. According to maps of reciprocal space of point (880), formed disturbed layer is stressed and deformation occurs only in the normal direction to the surface of the film.

From the rocking curve (Fig. 1, *b*) from the angular distance ΔΘ between the peak of the film and the last oscillation determine the value of the maximum relative deformation:

$$\frac{\Delta d}{d} = \Delta\Theta\, ctg\Theta_B \approx 0.61\%.$$

As practice has shown, for the best coincidence of theoretically calculated rocking curves to the experimental diffractograms, when fitting it is necessary to change the following parameters of the profile component associated with nuclear losses of ions energy: $\left.\frac{\Delta d}{d}\right|^N_{max}$ – maximum deformation of the profile component, $\left.\frac{\Delta d}{d}\right|^N_0$ – deformation on the surface, R_p^N – position of the maximum deformation of the profile component, L^N – the thickness of the disturbed layer of the profile component. Parameters of the profile component associated with electronic losses of ions energy should be changed as follows: $\left.\frac{\Delta d}{d}\right|^E_{max}$ – maximum deformation of the profile component, R_p^E – position of the maximum Gaussian curve, L^E – the thickness of the disturbed layer of the profile component. The fixed value $\left.\frac{\Delta d}{d}\right|_{min}$, is equal to the minimum value of the ordinate and determines the thickness of the disturbed layer as follows: $\{L^N, L^E\}$.

Using the theoretically calculated profile of defect concentration, which takes into account the components of the profile associated with nuclear and electronic losses of ions energy (Fig. 1, *a*), and the experimental rocking curves (Fig. 1, *b*, *c*), the following initial parameters of the profile are obtained: L^N=2,900 Å, L^E=1,100 Å, R_p^N=1,400 Å. Proceeding from (Fig. 1, *a*), only the component with nuclear losses of ions energy make a contribution to the maximum deformation. Then from the experimental rocking curve, it is obtained:

$$\left.\frac{\Delta d}{d}\right|^N_{max} = 0.61\%.$$

From Fig. 1, *a* the ratio of the maximum number of defects to the number of defects on the surface is equal to: 2.5/0.8=3.125. Considering that at small doses of implantation deformation is proportional to the number of defects, it is obtained:

$$\left.\frac{\Delta d}{d}\right|^N_0 = \left.\frac{\Delta d}{d}\right|^N_{max} / 3.125 = 0.20\%.$$

From Fig. 1, *a* the ratio of the maximum number of defects generated by nuclear losses of ions energy to the maximum number of defects generated by electronic losses of ions energy is equal to 2.5/1.4=1.786. Then:

$$\left.\frac{\Delta d}{d}\right|^E_0 = \left.\frac{\Delta d}{d}\right|^N_{max} / 1.786 = 0.34\%.$$

Parameter R_p^E from Fig. 1, *a* is about 1,500 Å.

The resulting starting strain profile is shown in Fig. 2, *a*, and the corresponding theoretical and experimental rocking curves are shown in Fig. 2, *b*, *c*. As we can see, the theoretical rocking curves in general describe additional oscillatory structures, but for better agreement between the theoretical and experimental rocking curves further clarification is necessary by correcting the parameters of the profile.

To clarify, we apply the first approach to minimizing the deviation of the theoretically calculated rocking curve from the experimental one used in the first model of the disturbed layer, according to which the function defining the strain profile is known.

After refinement of the parameters of the profile, we received the result, shown in Fig. 3, *a*, *b*. As we see from the figure, the film peak, substrate and additional oscillatory structure coincide very well. Therefore, a definite strain profile is reliable. However, outside of the additional oscillatory structure, there are no coincidences. This is due to the failure to take into account the radiation defects in the disturbed layer and the diffuse background created by them.

Therefore, in order to minimize the deviation of the theoretical rocking curve to the experimental outside of the additional oscillatory structure, the second approach was used, the parameters of which were the radius and

the concentration of the dislocation loops. Thus, from the slope of the background outside of the additional oscillatory structure, the radius of the dislocation loops R was recognized, and from the intensity the concentration of the dislocation loops n was recognized. It was established that at a radiation dose of $3 \cdot 10^{14}$ cm^{-2} the radius of dislocation loops is equal to $R=30$ Å, and the concentration of the dislocation loops in the maximally deformed layer is equal $n_{max}=1.2 \cdot 10^{15}$ cm^{-3}.
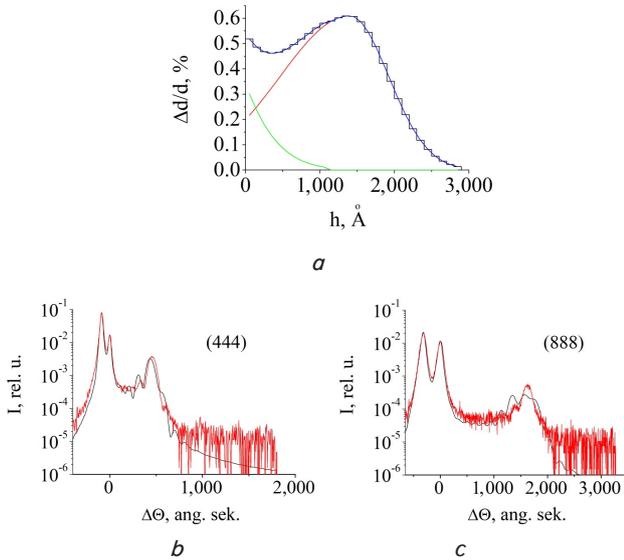

Fig. 2. The results of the X-ray diffraction modeling: a — the initial strain profile in the form of stairs (black line) and the averaging line (blue line), and its components, associated with the defect formation due to the electronic (green line) and nuclear (red line) losses of ions energy; b, c — corresponding experimental rocking curves (red line) and theoretically calculated rocking curves with taking into account spectrometer broadening (black line): reflexes (444) and (888) respectively
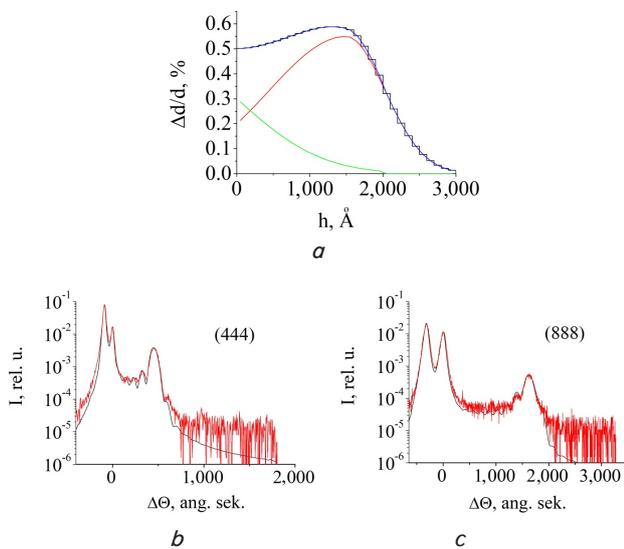

Fig. 3. Results of X-ray diffraction modeling: a — refined strain profile; b, c — corresponding experimental rocking curves and theoretically calculated rocking curves with taking into account spectrometer broadening. Explanation — as in Fig. 2

Using the obtained strain profile as a starting point and using the second model of the disturbed layer and the second approach to minimizing the function of MSD, the result was obtained presented in Fig. 4. Now there is a good coincidence between the theoretically calculated and experimental rocking curves in the entire angular range, and the strain profile is slightly different from the profile that was obtained at its set up functionally.
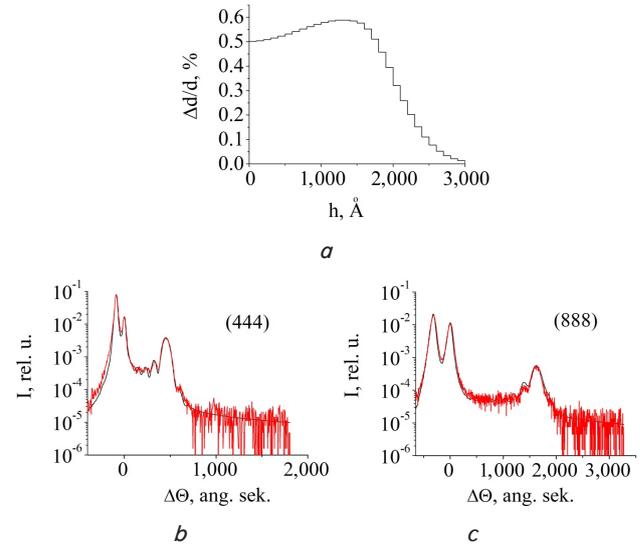

Fig. 4. Results of X-ray diffraction modeling: a — refined strain profile that was obtained without its set up functionally; b, c — corresponding experimental rocking curves and theoretically calculated rocking curves with taking into account the defects in the disturbed layer and the spectrometer broadening. Explanation — as in Fig. 2

All components of the theoretical rocking curves are presented in Fig. 5.
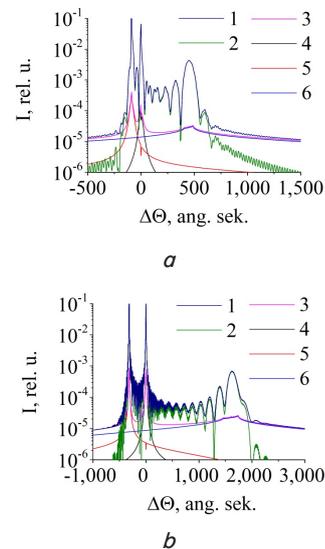

Fig. 5. The theoretical calculated rocking curve (1) and its components: coherent (2) and diffuse (3) (diffuse from the substrate (4), the film (5), and the disturbed layer (6)): a — reflex (444), b — reflex (888)

From Fig. 5, you can see that in some angular intervals on the rocking curve the intensity of diffuse scattering is com-

mensurable or exceeds the intensity of coherent scattering. It is also seen that the intensity of diffuse scattering from the substrate and the nonimplanted part of the film near the Bragg's peaks strongly exceeds the value of the background. This confirms the necessity of taking into account diffuse scattering from all parts of the layered structure, and not only from the ion-implanted layer.

6. Discussion of the method of determining the parameters of the crystalline structure of the ion-implanted layer and its practical realization

The choice of starting approximation is often crucial for the correct determination of the parameters of the ion-implanted layer. This is especially true for the uniqueness of the parameters. Therefore, an important role is given to modeling the process of ion implantation and taking into account all mechanisms of defect formation. In particular, when the results of formation of defects due to electronic losses of ions energy can be neglected, the second term in the formula, which described the strain profiles, will be equal to zero. With multiple ion implantation, additional terms will appear in this formula.

In ionic implantation, created point defects that can be combined in the dislocation loops are created. According to [16], the contribution of diffuse X-ray scattering by dislocation loops is much greater than diffuse scattering by point defects. In this case, the dominant contribution in diffuse X-ray scattering is from the dislocation loops.

The results of the X-ray diffraction modeling showed that the value of the extinction coefficient μ_{ds} influences most significantly on the intensity of the rocking curves outside the additional oscillatory structure. The value of the static Debye-Waller E factor influences most significantly on the intensity of the last oscillations of the additional oscillatory structure that correspond to the maximum deformation.

In the calculations, it was considered that the concentration of dislocation loops is proportional to the defect profile and the radius of the dislocation loops is the same in thickness of the disturbed layer. Prismatic dislocation loops in the ion-implanted layer are formed only in crystallographic planes which are parallel to the surface of the sample. For the studied film of iron-yttrium garnet, this is a plane (111). Therefore, the calculations took into account only this orientation of dislocation loops.

It is worth noting that in materials with a garnet structure with a cut plane (111) the most intensive reflex is (444), therefore, the additional oscillation structure from the ion-implanted layer is obtained with high accuracy. In some modes of implantation (significant amorphisation) and other reflexes, the accuracy of the obtained rocking curves can be much worse. In this regard, the simultaneous use of all reflexes in the initial stages of analysis can lead to deterioration of the accuracy of determining the parameters of the disturbed layer. To get out of this situation, you can enter the coefficient for the influence of each of the reflexes on the total MSD. You can also first analyze the most intensive reflex as much as possible, and then refine the obtained parameters from other reflexes that are sensitive to them. In particular, in the analysis of monocrystalline materials with the structure of garnet, one can first analyze in detail the coherent component of the scattering from the reflex (444), and then the diffuse component from the reflexes (888) and (880).

In general, this approach shows that in the study of the structure of ion-implanted layers of single crystals and films and in the simulation of diffraction of X-rays, both coherent and diffuse components for all parts of the layered structure should be taken into account. Failure to take into account the diffuse component and the use of only one reflex leads to a significant distortion of the parameters of the calculated strain profiles (displacement of the position of maximum deformation by more than 100 Å at the thickness of the damaged layer 5,000 Å). The use of the statistical dynamic theory of X-ray scattering [16] to all parts of the layered structure makes it possible to obtain information on specific types of defects (radius, concentration, orientation, and their changes in the thickness of the disturbed layer) without limitation on the size of the defects. The possibility of modeling X-ray diffraction by using other approaches (kinematic, semi-kinematic, and dynamic based on the Takagi equations [23]) is provided in the program. It allows comparing the results and minimizing inaccuracies in determining the strain profiles and other parameters of the disturbed layer. Unlike other works in this direction, in this approach all information about the sample (probable deformation distributions, defect parameters) is taken into account. In the given approach, the possibility of a discrepancy of the profiles to the selected starting physical model is taken into account and the uniqueness of the results obtained is determined.

The use of a complex of three different methods of minimization significantly increases the effectiveness of the minimization procedure in comparison with using only the least squares method or only the gradient method. If the target function of one of the methods falls into the local minimum or the zone of its ineffective use, it is possible to operate other methods and to continue the general minimization procedure. Due to the combination of minimization methods in the developed approach, the target functions of each of which can fall into local minima, the authors also consider it expedient in the future to include also minimization methods that use the genetic algorithm and the annealing simulation algorithm in the general complex.

This approach can be fully or partially used to analyze layered structures and near-surface layers after other types of modifications (studies of multilayer structures, diffusion saturation, laser annealing, etc.).

7. Conclusions

1. It has been established that at doses of implantation for which the strain profile can be considered proportional to the distribution of radiation defects, the value of the extinction coefficient μ_{ds} influences most significantly on the intensity of the rocking curves outside the additional oscillatory structure, and the value of the static Debye-Waller factor E influences most significantly on the intensity of the last oscillations of the additional oscillatory structure that correspond to the maximum deformation.

2. It was established that at the initial stages of the approach, the strain profile should be determined from the rocking curve region with an additional oscillatory structure by modeling a coherent component of X-ray scattering.

To characterize a defective system, it is necessary to analyze the diffuse component using a part of the rocking curve, which is located behind the additional oscillatory structure and in which the contribution of the coherent component does not exceed 5 %.

3. A method for defining the characteristics of defects in ion-implanted near-surface layers of crystals has been developed and tested, according to which:

– strain profiles are determined from reflexes for which the static Debye-Waller factor E and the extinction coefficient μ_{ds} are minimal;

– refinement of defects parameters is carried out by simultaneous analyzing of experimental rocking curves from a series of symmetric and asymmetric reflexes;

– the uniqueness of determination of strain profiles and defect parameters is checked.

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