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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

DETECTION OF NEW POLYTYPES IN $(Fe,Ga,In)_2S_3$ AND Cd(Mn,Mg)-In-Ga-S SYSTEMS BY IMPROVED ELECTRON-DIFFRACTION METHODS

Speciality: 2223.01 – Crystallography, crystal physics

Field of science: Physics

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GENERAL DESCRIPTION OF WORK

The actuality of the subject and degree of elaboration:

The high development of electronics mainly depends on providing its material base with new promising semiconductor materials. Layered semiconductor crystals have a wide range of different property parameters and these parameters are easily controlled. Due to the anisotropy of their physical properties, layered crystals are widely used in the production of photodetectors, switches, photovoltaic devices, etc. It has been shown that various ions and molecules can be placed between the packages of these compounds by chemical and electrochemical methods, and as a result, these materials become more useful for applications.

Structural studies play an important role in materials research. Thus, it is possible to explain many physical properties and processes observed in materials by knowing the crystal structure. However, in some cases, the lack of correct crystal structure data of materials leads to the spread of physical results that contradict each other in the literature about their physical properties. Therefore, for layered crystals, which often consist of a mixture of different polytypes, and in some cases even a mixture of polymorphic phases, the diagnostics of the crystal structure is very important.

Although the powder X-ray diffraction method has become a reliable X-ray structure analysis method in recent decades, it still has many serious disadvantages. In the X-ray powder method, three-dimensional diffraction data (reflexes) are all collected in one direction. For this reason, different reflexes with the same or close values of interplanar distances (d) can cover each other and in some cases even fall on top of each other (especially when the

values of the lattice parameters are large). During the study of nano powders, due to the broadening of reflexes, these covers and superimposes increase even more. When the studied samples consist of a multiphase mixture, the investigation of the crystal structure becomes even more difficult, since the X-ray powder diffraction include diffractions from all phases.

X-ray diffraction methods may not detect fine structure effects (for example: superlattices, modulations, etc.). Therefore, it is necessary to conduct electron diffraction studies in addition. Single crystal electron diffraction methods are an ideal method for studying such additional structures (superstructures) and effects.

The importance of the accurate determination of the crystal structure and the correct explanation of the physical properties stimulated the creation of new electron diffraction methods that overcome the disadvantages of the methods known so far. However, since the area of the electron flux is measured in nano-meters, the recent transition to nano-technologies makes the creation of new electron diffraction methods even more urgent.

Collection and analysis of X-ray and neutron diffraction data is performed using various programs. Automation of electron-diffraction data collection and analysis using various programs could greatly facilitate crystal structure studies.

Although many scientific groups in the world are engaged in this work, the collection and examination of diffraction data of electron beams is still semi-automatic. The goal is to collect as many reflexes as possible that do not superimpose and are fully recorded (not incomplete). For example, precession electron diffraction (PED)[1] reflections are located inside the circle (in the zero-order Laue zone) and inside the ring (in the second-order, third-order, etc. Laue zones). In this case (in PED), depending on the value of the crystal lattice parameter located perpendicular to the plane of the single crystal thin film, such tilt angles (smaller than 2^{o}-3^{o}) are selected for the electron beam so that different Laue zones do not superimpose each other. Therefore, the choice of

[1] R.Vincent, P.A.Midgley. Ultra-microscopy, 1994, 53, p. 271-282.

crystal in this case also depends on the value of its lattice parameter. Electron diffraction patterns contain a large number of partially marked reflexes at the boundaries of each Laue zone (in this case, the Ewald plane rotates, recording only a fraction of the volume of superlattice (SL) sites).

In the works[2,3,4] various rotation electron diffraction patterns obtained from thin single crystal thin films placed parallel to the surface of the crystal holder are presented. But often, for some reason, it is not possible to place a thin single crystal thin films or a nano-sample completely parallel to the surface of the crystal holder. We have to make do with what we have: to study samples that are not exactly parallel to the crystal holder surface, to rotate the samples in various ways, to give schemes that explain these rotations and help to easily investigate non-standard reflexes (for example, reflexes located outside the lines of ellipses)[5].

The choice of research object and subject is related to the ease of layering of layered crystals, and therefore it is possible to prepare textured (oriented polycrystalline) and single-crystal thin films with nano-thickness very easily from them.

The object and subject of the research:

To develop known electron diffraction rotation methods, to create new electron diffraction rotation schemes, and to reveal new polytypes by taking electron diffraction patterns from crystals included in the $(Fe,Ga,In)_2S_3$ and Cd(Mn,Mg)-In-Ga-S_4 systems, applying these methods and schemes.

Purposes and tasks of research:

The purpose of the dissertasion - to discover new polytypes by developing rotational electron diffraction methods and developing new rotational schemes and applying them to the study

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[2] B. B. Zvyagin, A. P. Zhukhlistov, M. G. Kyazumov, and A. M. Fominenkov, Kristallografiya, 1990, 35 (3), p.602.

[3] M. G. Kyazumov, B. B. Zvyagin, A. P. Zhukhlistov, and A. M. Fominenkov, USSR Inventor's Certificate no. 1649397 (1991).

[4] M.G. Kyazumov, Crystallogr. Rep. 2014, 59 (4), p.486-492.

[5] M.G. Kyazumov, S. M. Rzayeva, A. S. Avilov, Crystallogr. Rep. 2022, 67 (6), p. 833-837.

of crystal structures of arbitrarily oriented single-crystal films.

To achieve the goal, the **following issues** were resolved:

1. Obtain single crystal films less than 50 nm thick from thick samples of $CdInGaS_4$, $CdInAlS_4$, $MnGaInS_4$, $Ga_{0,8}In_{1,2}S_3$ layered compounds and $Fe_{0,25}Ga_{0,5}In_{1,25}S_3$, $Fe_{0.75}Ga_{0.25}InS_3$, $Mg_{0.7}Ga_{1.4}In_{0.8}S_4$ solid solutions, as well as oriented polycrystalline films (textures) composed of nano-thick particles.

2. To study the single crystal thin films of these crystals by developing the proposed rotational electron diffraction methods by solving the technical issues.

3. Taking into account the disadvantages of known diffraction methods, to create new electron diffraction schemes for the study of arbitrarily oriented films that eliminate this deficiency.

4. To determine the crystal lattice and structure types of crystals using these schemes, to determine the lattice parameters and space groups, and also to study the crystal structure.

5. To study the superlattice and superstructure properties of these crystals.

6. To show the advantages of received eloctron diffraction patterns over electron diffraction patterns received by other methods.

Research methods:

Crystals were synthesized by various methods. Single crystal thin films which located parallel to the surface of crystal holder and forming a certain angle with it were studied. Part of the research was performed on the basis of obliqued electron diffraction patterns obtained lamellar textures, and on the basis of single-crystal rotation electron diffraction patterns, which are imitating of electron diffraction patterns obtained from lamellar and acicular textures, but have special advantages. Electron diffraction patterns which obtained from single crystal thin films which located on the surface of crystal holder were also studied by developing single crystal rotation electon diffraction methods.

One of the important parts of electron diffraction studies is the acquisition of thin single-crystal films, which the accuracy and reliability of the determination of the crystal structure depend on.

Single crystal thin films were obtained by lifting off from thick films of layered crystals with adhesive tapes. Due to existence of two tilt axes on goniometer stage of EG-400 and ER-102M electron diffractometer under the electron beam using different methods by tilting and rotating a crystal holder, it were obtained rotation electron diffraction patterns imitating electron diffraction patterns of lamellar or acicular textures. To obtain electron diffraction patterns with a desired set of reflections, a single-crystal film was rotated around the axes lying in the plane of the crystal holder and perpendicular to it. Each of the methods used in the dissertation is related to diffraction of parallel electron beams.

The main provisions submitted to the defense:

1. Experimental justification of the advantages of new electron diffraction methods during the study of polytype mixtures.

2. Schemes explaining the rotations of the single-crystal film or crystal holder around the axes perpendicular when single crystal thin films located on the surface of the crystal holder, as well as the packing of the reciprocal lattice sites on the Ewald plane located obliquely to it. A new 2H polytype revealed on the basis of electron difraction patterns obtained from $Fe_{0.75}Ga_{0.25}InS_3$ by rotation electron diffraction methods, around an axis perpendicular to the single-crystal films (single-crystal located parallel to the surface of the crystal holder) after rotating from the position perpendicular to the electron beam to an angle of $\varphi<60°$.

3. A new 2H polytype of $Ga_{0,8}In_{1,2}S_3$ crystals detected from electron diffraction patterns obtained by rotation of the single-crystal film around the axes located on the plane of the CH (single-crystal located parallel to the surface of the crystal holder).

4. Schemes that explain the origin of reflexes located in non-standard positions (outside the lines of ellipses) in electron diffraction patterns obtained from $Mg_{0.7}Ga_{1.4}In_{0.8}S_4$ and $MnGaInS_4$ crystals and also facilitate their easy investigation.

These schemes are important in future electron diffraction studies of arbitrarily oriented nanopatterns on CH.

5. New 2H polytypes observed in $MnGaInS_4$ and $Mg_{0.7}Ga_{1.4}In_{0.8}S_4$ crystals and also 3R polytype known from the

literature observed in $Fe_{0,25}Ga_{0,5}In_{1,25}S_3$ and $Fe_{0.75}Ga_{0.25}InS_3$ crystals based on electron diffraction patterns obtained from arbitrarily oriented samples. The role of new schemes in research.

Electron diffraction patterns were obtained by rotation the crystal holder together with the single crystal film (the single-crystal film located at an angle θ on the surface of the crystal holder) from the position perpendicular to the incident electron beam to an angle of $\varphi<60°$ before starting the recording, and rotating around the axis perpendicular to the plane of the crystal holder during the recording.

Scientific novelty of the study:

1. Detection of a new 2H polytype based on electron diffraction patterns obtained from rotation around an axis perpendicular to its surface during recording of a single-crystal thin film of $Fe_{0.75}Ga_{0.25}InS_3$ tilted by an angle of $\varphi<60°$ from the position perpendicular to the incident electron beam before rotation.

2. A new 2H polytype was detected based on electron diffraction patterns obtained from $Ga_{0,8}In_{1,2}S_3$ crystals during rotation around the axis located on the surface of the crystal holder of the single-crystal nano-thick film which located parallel to the surface of the crystal holder. Electron diffraction patterns obtained by this method imitate electron diffraction patterns obtained from acicular textures, but give specific information related to a single crystallinity, rather than the generalized information related to many crystallinities;

3. Detection of new 2H polytypes based on electron diffraction patterns obtained rotation of single-crystal films of $MnGaInS_4$, $Fe_{0,25}Ga_{0,5}In_{1,25}S_3$ and $Mg_{0.7}Ga_{1.4}In_{0.8}S_4$ (the single-crystal film is first located on the surface of the crystal holder at an angle $\theta \leq 5^o$) from the position perpendicular to the incident electron beam up to an angle of $\varphi<60°$ before rotation and recording;

4. In the electron diffraction patterns obtained from $Mg_{0.7}Ga_{1.4}In_{0.8}S_4$ and $MnGaInS_4$, schemes are given that explain to which series the reflections located outside the lines of the ellipses belong and help to easily index them. Such schemes can be used in future electron diffraction structure studies of nano-samples

arbitrarily placed on the surface of the crystal holder;

5. Electron diffraction phase analysis for $CdInGaS_4$ crystals confirmed its effectiveness in studying layered crystals consisting of polytype mixtures. During the combined application of inclined texture electron-diffraction and single-crystal rotation methods around different axes, it was determined that the studied $CdInGaS_4$ samples are not 3T polytype, but a mixture of 1T and 3R polytypes, and not a 6T polytype, but a mixture of 2H and 3R polytypes;

6. Schemes of creation of superlattices and reciprocal lattices (with $A_{1,2}=\sqrt{7}\,a$, $A_3=2a$ and $A_1\hat{}A_2=22°$ parametres) of $Ga_{1.29}In_{3.38}S_7$ crystals with lattice parameters a=3.82 Å, c=63.41 Å, sp. gr. R3m and structural type $TO\overline{T}POOP$ are given and justified. It was determined that in $Ga_{1.29}In_{3.38}S_7$ crystals with a new composition formed due to sulfur deficiency in $Ga_{1.30}In_{3.40}S_7$ and $Ga_{1.32}In_{3.45}S_7$ crystals, the reflexes from the superlattice with parameter $A_3=2a$ are correspondingly, weakened and disappear.

Theoretical and practical significance of the study:

1.When a thin single crystal layer forms an angle with the CH surface, schemes are given that explain the rotations around the axis perpendicular to the CH surface, and also the marking of the reciprocal lattice (RL) nodes on the Ewald plane located inclined to the CH. These schemes can be used in future electronic-structure studies of arbitrarily oriented nanoparticles on the surface of CT.

2. Single-crystal rotation electron diffraction patterns, unlike electron diffraction patterns obtained from texture , have greater quality and information and, most importantly, provide compact and fine structure information related to a single crystallinity, rather than generalized data related to multiple crystallinities;

3. New electron diffraction methods expand the capabilities of electron difraction patterns due to the improvement of the accuracy and fineness of diffraction data obtained compared to electron microscopes, as well as obtaining more complete structural information. These methods can be successfully applied in physics, chemistry, crystallography, biology, metallurgy and other fields;

4. The specified lattice parameters, space group, structure type, anion packed type and crystal structure of the mentioned new

crystals are important information materials for researchers studying these crystals and their isostructural analogs.

5. The given and justified formation schemes of superlattices of $Ga_{1.29}In_{3.38}S_7$ crystals with parameters $A_{1,2}=\sqrt{7}\,a$, $A_3=2a$ və $A_1\hat{}A_2=22°$ and their reciprocal lattices can be successfully applied in future studies of layered crystals with several super structures .

Approbation and application:

The main results of the dissertation work were presented at the following international and republican scientific conferences:

"XXVIII Russian Conference on Electron Microscopy (RCEM 2020") (Chernogolovka, 2020), "V Russian meeting on clays and clay minerals", dedicated to the 100th anniversary of the birth of B.B. Zvyagin) (Moscow, 2021) "1st International symposium on recent advances in fundamental and applied sciences" (Erzurum, 2021), "International Conference on Molecular Spectroscopy dedicated to the 100th anniversary of academician L.M. Imanov" (Baku, 2022), XXIX Russian Conference on Electron Microscopy (RCEM 2022) (Chernogolovka, 2022, online), "Dedicated to the 100th anniversary of National Leader Heyder Aliyev Conference on Development of the Theoretical and Applied Physics" (Baku, 2023), The Third International Forum "Physics -2024" (Samarkand, 2024).

The list of articles published at the end of the dissertation on the topic of the dissertation was published in the following journals:

Azerbaijan National Academy of Sciences, Transactions, Physics and Astronomy, 2020; Azerbaijan Journal of Physics 2022; "Scientific works" journal of the Nakhchivan Brunch of ANAS, 2021; "Gənc tədqiqatçi" Scientific-practical journal 2021; "Кристаллография" and Crystalloqraphy Reports 2021, 2022; Crystalloqraphy Reports 2023;

15 scientific works have been published in local and foreign scientific publications guaranteed by SAC of Azerbaijan on the subject of the dissertation work. 7 of them are articles (3 of them in the journal of "Кристаллография", as well as in "Crystallography Reports") and 8 are conference materials.The list of published works is given at the end of the dissertation.

The name of the organization where the dissertation work is

done:

The dissertation work was performed at the Institute of Physics of the Ministry of Science and Education Republic of Azerbaijan.

The structure and volume of the dissertation :

Dissertation consists of 150 pages - introduction, 4 chapters, conclusions, 5 tables, 16 pictures and a list of 227 references. The volume of the dissertation (excluding spaces in the text and pictures, tables, graphs and the bibliography) is 189390 characters (introduction - 29988, chapter I - 57596, chapter II - 32809, chapter III - 29766, chapter IV - 32510, , result – 2907 characters).

THE MAIN CONTENT OF THE DISSERTATION

Introduction. The relevance of the chosen topic is substantiated, the purpose and issues are formulated, the scientific innovation, practical and scientific importance of the work is shown, the main propositions submitted to the defense are listed, and the chapters of the dissertation are briefly interpreted separately.

The first chapter is devoted to the investigation of electron diffraction methods and crystal structures of multi-component layered semiconductors. The methods of obtaining lamellar and acicular textures and the methods of obtaining and examining experimental data from them are presented. Also, the discrete and continuous electron diffraction methods of single crystal were investigated, their differences and advantages from each other and also from inclined texture method, were shown. The irreplaceable advantages of continuous electron diffraction methods have been confirmed, especially for our studies.

Various experimental devices and data collection methods, including precession electron diffraction (PED), automated diffraction tomography (ADT), electron diffraction tomography (EDT), single crystal electron diffraction (SCED), precession electron diffraction tomography (PEDT), rotating (discrete) electron diffraction (RED, DRED), continuous rotation electron diffraction (CRED), microcrystal electron diffraction (MicroED), crossed beam electron

diffraction (CBED) was investigated.

The advantages and disadvantages of three-dimensional electron diffraction (3ED) methods, especially 3ED methods with continuous rotation, have been discussed since the 80s of the last century by M.H. Kazimov et al.[6,7,8], it has been shown that a large number of continuous rotation electrongrams obtained from single crystals, reminiscent of electrongrams of needle-shaped and plate-shaped textures, can be easily analyzed. Rotations were carried out around axes located on the surface of the crystal holder or perpendicular to it. The coordinate axes of the crystal and the reciprocal lattice were brought to these axes and rotations were performed. In the presented work, since it is not always possible to precisely place the nanosamples on the surface of the crystal holder, rotations were also performed around arbitrary axes (non-coordinate) of the crystal lattice, and schemes were presented that easily explain the origin of reflexes located in non-standard places, and with the help of these schemes, all reflexes were easily indexed[9].

This chapter also discusses the basic principles of close pack and polytypism. Crystal structures of polytypes of layered crystals and polytype phenomena in general Ramsdel, Hegg, Zhdanov, Belov (and at the same time Pauling, Wyckoff and Yagodzinski) number of double, triple, and quadruple layered semiconductor lattice parameters and crystal structures are given in the literature.

The second chapter presents the results of crystal structure studies of polytypes of $Fe_{0.75}Ga_{0.25}InS_3$, $Ga_{0,8}In_{1,2}S_3$ and $CdInAlS_4$ layered semiconductors. In the studies presented in this chapter, single- crystal films are placed parallel to the surface of the crystal holder.

When the single crystal film rotates under the incident electron

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[6] B. B. Zvyagin, A. P. Zhukhlistov, M. G. Kyazumov, and A. M. Fominenkov, Kristallografiya, 1990, 35 (3), p. 602

[7] M. G. Kyazumov, B. B. Zvyagin, A. P. Zhukhlistov, and A. M. Fominenkov, USSR Inventor's Certificate no. 1649397 (1991).

[8] M.G. Kyazumov, Crystallogr. Rep. 2014, 59 (4), p.486-492.

[9] M. G. Kyazumov, S. M. Rzayeva, and A. S. Avilov, Crystallogr. Rep., 2022, 67 (6), p. 833-837.

beam obliqued to its surface (around the axis perpendicular to the surface of the single crystal layer), the different site rows of the reciprocal lattice are marked along ellipses in the Ewald plane.

The number of recorded sites depends on the value of the tilt angle φ and the rotation angle ω. Due to the increase in the value of the rotation angle ω (ω≥60º), the different site rows of the reciprocal lattice are marked in the same places of the Ewald plane (ellipses), and their intensities are collected.

Schemes of rotation of a single crystal film around the axes located both on the surface of the crystal holder and perpendicular to its surface and registration of reciprocal lattice sites on the obliqued Ewald plane are presented.

When ω ≤60º, different parts of different site rows (for example: 10, 01, $\bar{1}1$, $\bar{1}0$, $0\bar{1}$ və $1\bar{1}$) equidistant from the rotation axis (c* axis of the reciprocal lattice) are assembled into different parts of the ellipses without superimposed each other. As the value of angle ω (ω>60º) increases, different parts of different site rows begin to superimposed with each other. These reflex series marked in hexagonal structures all have the same intensity, but the reflex series marked in trigonal and rhombohedral structures have different intensities, and therefore it is necessary to know which part of the ellipse belongs to which series. They can be determined by comparing the experimental values of the reflex intensities with their calculated values for the selected structural model.

When the single-crystal film is rotated, depending on the position of the tilt axis, along the ellipses in the Ewald plane, first a part of the sites belonging to one site row, and then other parts of the sites belonging to other site rows are recorded. If the single-crystal film is located exactly in the plane of the crystal holder, the site rows are perpendicular to the plane of the crystal holder, and therefore, during rotation, the sites of the reciprocal lattice are marked on the line of ellipses in the Ewald plane and replace each other equally.

The study of $Fe_{0.75}Ga_{0.25}InS_3$ crystals by powder X-ray diffraction method showed that they consist of the main known polytype and a small amount of an unknown phase. Electron diffraction patterns were obtained from thin single-crystal films of

$Fe_{0.75}Ga_{0.25}InS_3$, after being rotated to an angle of φ<70 from the position perpendicular to the incident electron beam before exposure, and by rotating to an angle of $\omega \leq 60°$ around the axis perpendicular to the plane of the crystal holder during recording (exposure).

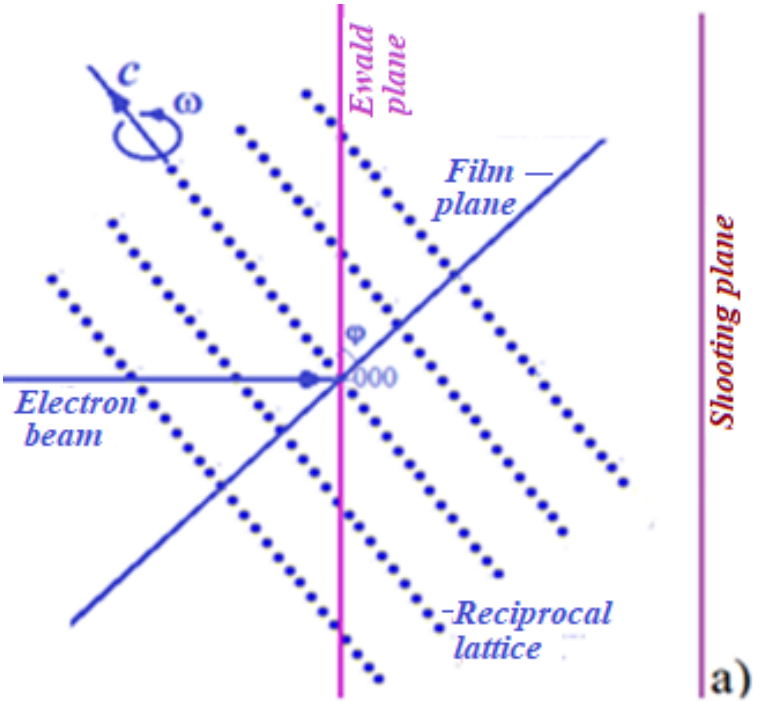
The investigation of diffraction patterns were interpreted using the well-known formulas for electron diffraction patterns of oblique textures[10]:

$$d_{100} = \sqrt{3}\, a/2 = 2L\lambda h/2R_{h00} \quad (1)$$

$$D_{hk\,l} = (R^2_{hkl} - R^2_{hk0})^{1/2} \quad (2)$$

$$\Delta D = c^*L\lambda = (D_{hkl} - D_{hk(l-1)}) \quad (3)$$

$$d_{001} = c = 1/c^* = L\lambda/\Delta D \quad (4)$$



[10] B. Zvyagin, Electron Diffraction and Structural Crystallography of Clay Minerals (Nauka, Moscow, 1964) [in Russian].

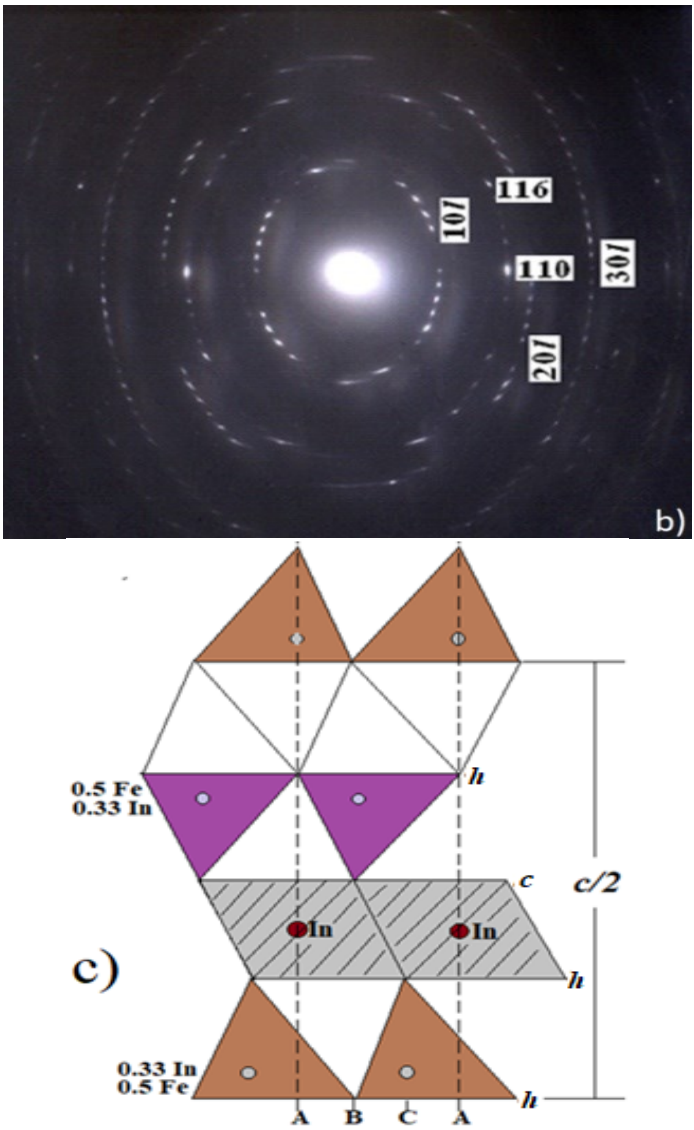


Figure 1. a) Rotation of the reciprocal lattice around the c axis and assembly scheme of its sites obliquely located Ewald plane. Here, ω is the rotation angle, φ is the angle of tilt of the thin single crystal film from the position perpendicular to the electron beam until the diffraction recording, b) rotation

electron diffraction patterns obtained from thin single crystal films of $Fe_{0.75}Ga_{0.25}InS_3$ (φ=40°, ω=45°), c) is the projection of the two-pack hexagonal (2H) polytype of $Fe_{0,75}Ga_{0,25}InS_3$ onto the (11 $\bar{2}$ 0) plane of the crystal structure.

Here and *h*, *k*, *l* are the Miller indices; 2R are the distances between reflections on the electron diffraction pattern; R_{hk0} are the minor axes of ellipses; d_{hkl} is the interplanar distance; D_{hkl} is the distance between sites hkl and the (hk0) plane of the reciprocal lattice on the Lλ scale at h, k = const; ΔD is the distance between adjacent sites along the c* axis on the Lλ scale; and c* is the reciprocal lattice parameter.

The ratio of R_{hk0} distances $1:\sqrt{3}:2:\sqrt{7} = R_{100}:R_{110}:R_{200}:R_{210}$ determined the crystal lattice to be hexagonal. Using the distances $L\lambda\Delta D_{10l}$ between the reflections lying in the first ellipse (the 10 $\bar{1}$ *l* series), the lattice parameters a=3.78 Å and c=24.44 Å were found and, according to the extinction of general-type reflections, the sp. gr. $P6_3mc$ was determined. The absence of reflections with odd indices *l* (the 11 $\bar{2}$ *l* series) in the second ellipse indicates that the structure of this sample consists of two packages. Using the distance $L\lambda\Delta D_{11l}$ between reflections in the second ellipse (the 11 $\bar{2}$ *l* series), the thickness package (12.22 Å) was determined and, by an index of *l* = 6 of the second strong 11 $\bar{2}$ 6 reflection, the $_hT_hO_\kappa T_hP$ structural type was determined[11]. Thus, a new two-package hexagonal (2H) polytype was discovered. Here, T and O are the two-dimensional layers of tetrahedra and octahedra, respectively, P is the interpacket void space, and h and k are the hexagonal and cubic packing of sulfur layers, respectively.

This chapter also presents a new two-package hexagonal 2H polytype discovered on the basis of electron diffraction patterns obtained by rotating nano-sized single crystal films of $Ga_{0,8}In_{1,2}S_3$ (the film is located parallel to the surface of the crystal holder)

[11] M.G. Kyazumov, Crystallogr. Rep. 2014, 59 (5), p.705-712.

around axes located on the plane of the crystal holder. The crystal lattice parameters are a=3.82Å, c=24.53Å and the space group is $P6_3mc$.

The electron diffraction patterns obtained by this method imitate the electron diffraction patterns obtained from acicular textures.

Packet-packet transitions occur according to the hh type in structures whose structural unit is one of the TOTP, TOTTTP, and TTOTTP type packets. Therefore, the centers of the interpacket tetrahedra in the direction of the c axis coincide with the centers of the tetrahedra within the adjacent packet.

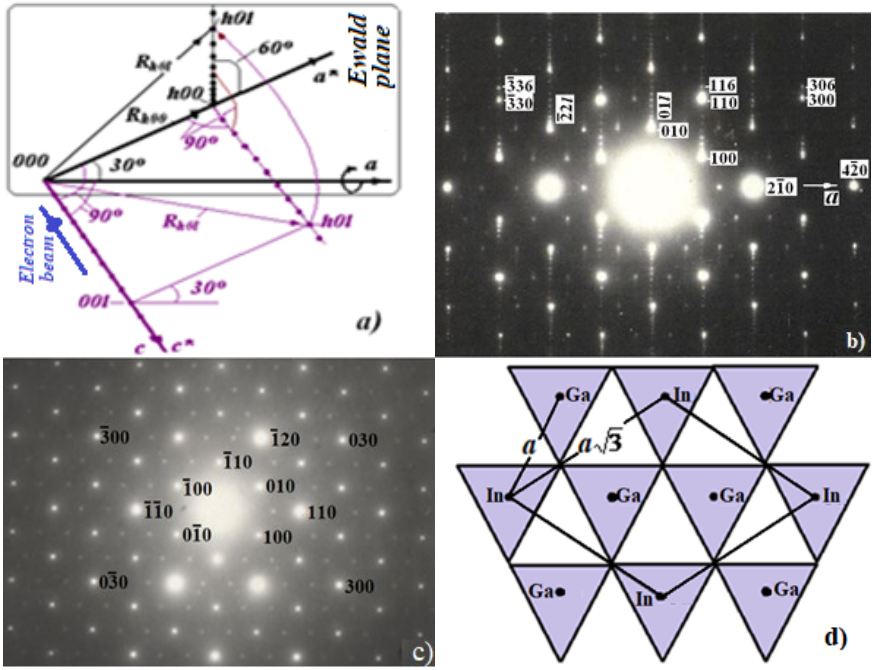


Figure 2. a) Detection of site rows of the reciprocal lattice of a hexagonal crystal (colored sites) and registration of site rows in the Ewald plane (black sites) during rotation of the reciprocal lattice around the a axis. The electron beam is perpendicular to the axis of rotation, b) the electron diffraction pattern of

rotation from the 2H polytype of $Ga_{0.8}In_{1.2}S_3$ c) the electron diffraction patterns corresponding to the hk0 plane of the reciprocal lattice, d) the vertical projection of the structure of the upper layer of tetrahedra.

This helps to partially occupy the tetrahedral positions between the packages with cations and, as a result, significantly strengthen the connections between the packages.

The third chapter presents the results of electron diffraction studies of single-crystal films of $Mg_{0.7}Ga_{1.4}In_{0.8}S_4$, $MnGaInS_4$ and $Fe_{0,25}Ga_{0,5}In_{1,25}S_3$ crystals. Single-crystal films are lyied at a certain angle θ on the surface of the crystal holder.

Most of the reflections are located along ellipses and are easily indexed in electron diffraction patterns from $Mg_{0.7}Ga_{1.4}In_{0.8}S_4$ (fig.3a) and $MnGaInS_4$. Diagrams explaining the origin of additional reflexes that are not on the lines of the ellipses are presented (Fig. 3b). These schemes are of great importance in the study of thin single-crystal films and films systems located at an arbitrary angle on crystal holder.

If the single-crystal film is at an angle to the plane of the crystal holder, the hk0 sites are located at different heights from the plane of the crystal holder, and the site rows are not perpendicular to the plane of the crystal holder. In this case, it can be determined by visual evaluation that the distances between the reflections on the ellipses are disturbed, which erroneously indicates the low symmetry of the crystal lattice. Some reflections fall outside the line of ellipses, leading to the erroneous conclusion that other phases or phase mixtures are present. But in fact, the lengths of the radius vectors of the sites do not change. Therefore, as the height of the reflexes from the minor axis of the ellipses decreases, they (reflexes) move further away from the major axis of the ellipses and remain outside the line of the ellipses.

Three different positions of the site rows of the reciprocal lattice can be distinguished (noted):

1. When the crystal holder is rotated by an angle $\varphi<70°$ from the position perpendicular to the incident electron beam and rotated

around an axis perpendicular to the plane of the crystal holder during recording, the $\mathbf{R}_{hk0}$ (h=nh_1, k=nk_1, h_1,k_1= const., n=1,2,3…) radius vectors of the site rows of the hexagonal reciprocal lattice perpendicular to the axis of natural tilt (priori tilt) (for example, in Fig. 3b: 1$0l$ və $\bar{1}\,0l$) will move along the surfaces of the cones.The vertices of the cones will be located at a distance ($h\mathbf{a}^*$ + $k\mathbf{a}^*$)ctgθ from the center of the reciprocal lattice on the axis of rotation. The inclined cross-sections of these opposite cones with Ewald plane are ellipses, shifted alternately to opposite sides along the major axes of the ellipses. At small values of θ the distance (ha* + ka*)ctgθ is very large, so they can be considered as deformed cylinders, but the deflections (slips) of ellipses and reflexes, respectively, should be taken into account.

2. When the crystal holder is rotated by an angle φ<70° from the position perpendicular to the incident electron beam and rotated around the axis perpendicular to the plane of the crystal holder during recording, the $\mathbf{R}_{hk0}$ (h=nh_1, k=nk_1, h_1,k_1= const., n=1,2,3…) radius vectors of the site rows of the hexagonal reciprocal lattice on the natural tilt axis (for example: $\bar{1}\,2l$, $\bar{2}\,4l$ etc., are not shown in fig. 3b), will move along the surfaces of the hyperboloids, whose centers are at the center of the reciprocal lattice and whose vertices are the real axes of the hyperboloids, at a distance ($h\mathbf{a}^*$ + $k\mathbf{a}^*$) from the axis of rotation. The oblique sections of these equiaxed hyperboloids with the Ewald plane consist of concentric ellipses centered at the center of the reciprocal lattice.

3. When the crystal holder is rotated by an angleφ<70° from the position perpendicular to the incident electron beam and rotated around an axis perpendicular to the plane of the crystal holder during recording, the $\mathbf{R}_{hk0}$ (h = nh_1, k = nk_1, h_1,k_1= const., n=1,2,3…) radius vectors on the natural tilt axis and also not perpendicular to it, hk site rows of the hexagonal reciprocal lattice (for example, Fig. 3b: 01l və $\bar{1}\,1l$), will move along the surfaces of hyperboloids, whose centers are on the axis of rotation but not at the center of the reciprocal lattice, and whose vertices are located at different distances from the axis of rotation.

At small angles θ (θ<3°), these cones and hyperboloids can be represented as deformed coaxial cylinders, and their oblique sections as deformed concentric ellipses centered at the center of the reciprocal lattice.

Oblique sections of these hyperboloids (at significant values of θ) with the Ewald plane give different groups of concentric ellipses that undergo different shifts along the major axes of the ellipses in opposite directions.

Electron diffraction patterns imitating electron diffraction patterns of lamellar textures obtained from thin single-crystal films of $MnGaInS_4$ and $Mg_{0.7}Ga_{1.4}In_{0.8}S_4$ were obtained. Electron diffraction patterns were obtained after tilting single crystal film up to angles $\varphi=40^0$ and φ=35°, respectively, and with subsequent rotations around the axis perpendicular to the plane of crystal holder, up to angles ω=70° and ω = 60°, respectively.

If a thin single-crystal film were located parallel to the crystal holder surface with its surface, during rotation around an axis perpendicular to the crustal holder surface, the 015 and $\bar{1}15$ reciprocal lattice sites, which are the same distance from the c-axis and from the film plane, would superimpose. In the present case (Fig. 3), since the single crystal film of $Mg_{0.7}Ga_{1.4}In_{0.8}S_4$ is located with its surface inclined to the surface of crystal holder, the 015 and $\bar{1}15$ reciprocal lattice sites move at different heights and along circles with different radii, and are marked in different places of the Ewald plane (Fig. 3b). As can be seen, the first ellipse of the rotation electron diffraction pattern of $Mg_{0.7}Ga_{1.4}In_{0.8}S_4$ (Fig. 3a) has only $\bar{1}15$ reflections from the $\bar{1}1l$ series. In the first ellipse of the rotation electron diffraction pattern of $MnGaInS_4$, there are two reflections from the $\bar{1}1l$ series, $\bar{1}15$ və $\bar{1}16$ (the image is presented in the dissertation).

Based on these electron diffraction patterns, new two-package hexagonal (2H) polytypes with structural modulus $_hT_hO_\kappa T_hP$, space group $P6_3mc$, and lattice parameters a=3.80 Å, c=24.55 Å and a=3.80 Å, c=24.39 Å were discovered, respectively.

Most reflexes in electron diffraction patterns are located along ellipses and are easily indexed. Diagrams explaining the origin of additional reflexes located outside the line of the ellipses are presented. In principle, these schemes should help to study the crystal structures of nanometer-thick thin films, as well as thin-film systems, nanopatterns, nanotubes, etc., located on the surface of crystal holder in arbitrary orientation.

Researches with the above mentioned electron diffraction rotation method were also carried out for $Fe_{0,25}Ga_{0,5}In_{1,25}S_3$ crystals. In this case, the single-crystal film is placed on the crystal holder surface at a certain angle ($4°<\theta<5°$). In this case, an easy-to-examine scheme is given, despite the increased complexity of the rotation. It was determined that the crystal structure of $Fe_{0,25}Ga_{0,5}In_{1,25}S_3$ crystals corresponds to the TOTP structural type 3R polytype with lattice parameters a=3.78 Å, c=36.78Å and space group R3m.

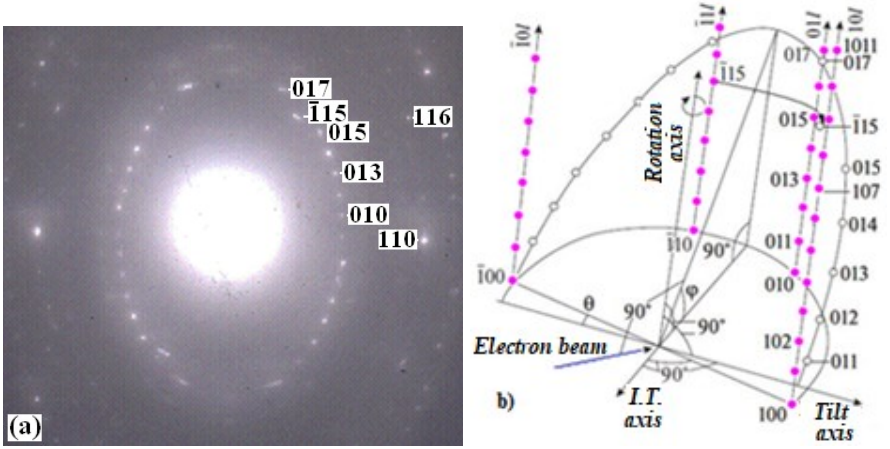


Figure 3. a) rotation electron diffraction patterns obtained from a thin single-crystal film of the 2H-polytype of $Mg_{0.7}Ga_{1.4}In_{0.8}S_4$ (φ=35°, ω=60°), (b) - scheme of rotation of the reciprocal lattice and detection of its sites on the Ewald plane. The reciprocal lattice sites are marked in red and are parallel to the *000l* axis. Closed circles show reciprocal lattice sites located parallel to the 01*l* axis, open circles correspond to the $\bar{1}1l$ and $\bar{1}15$ sites detected in the Ewald sphere cross section, I.T. is the axis of the

initial tilt of the film relative to the plane of the crystal holder, and θ is the angle between the film and crystal holder planes.

Knowing at least one R_{hk0} (h,k = const.) distance in the case of a hexagonal crystal, we can find other R_{hk0} distances. By applying the Pythagorean formulas to the entire series of reflexes, it is possible to find the parameter c and index all the reflexes.

The fourth chapter presents the results of polytypism research in layered crystals of $CdInGaS_4$ compound obtained by chemical transport reaction method. Electron diffraction patterns obtained from obliqued lamellar textures and also rotational electron diffraction patterns obtained from thin single-crystal films were studied. It has been shown that, in addition to the known polytypes 1T, 2H and 3R, polytypes 3T and 6R are also incorrectly identified when using electron diffraction patterns obtained only from obliqued texture. By examining rotation electron diffraction patterns obtained from thin single-crystal films, it was determined that the erroneously detected 3T (Fig. 4a) and 6R polytypes are actually a mixture of 1T and 3R (Fig. 4b), 2H and 3R polytypes, respectively[12].

The results of the conducted studies show that not only crystals selected from different ampoules and different parts of the same ampoule can differ in terms of structure, but also the same crystal can be composed of a mixture of different polytypes:

Different polytype modifications of 2H and 3R, 1T and 3R or 1T and 2H $CdInGaS_4$ crystals grow in the direction perpendicular to the layers. It was found that more than 50% of the samples in the same ampoule consisted of mixtures of different polytypes.

It is known from the literature that many polytypes are observed in layered crystals whose elementary lattices have a large c parameter. Among them there are both proven and unproven polytypes. The results of this study allow us to ask a logical question: are some new polytypes with large c parameters presented

[12] M. G. Kyazumov, S. M. Rzayeva, A. S. Avilov. Crystallography Reports, 66 (6) 2021, p. 906–912.

in the literature not mixtures of different polytypes with small c parameters?

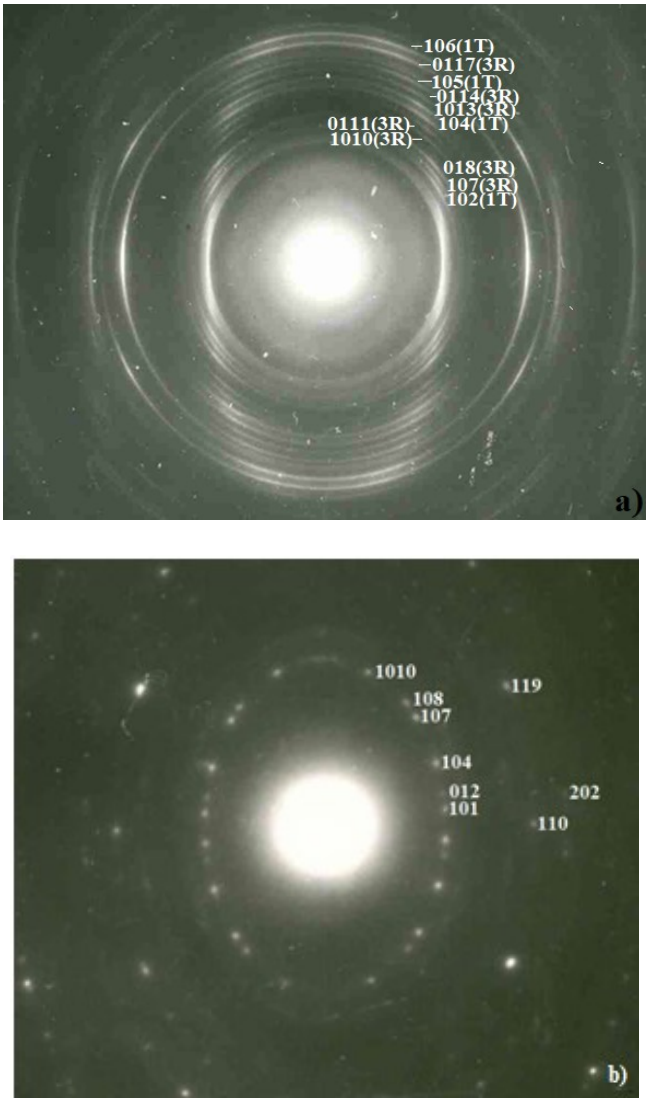


Figure 4. Electron diffraction patterns of $CdInGaS_4$: a) – textures mixtures, 1T and 3R polytypes; b) – thin single-crystal films, 3R-polytype.

It is known that in many cases during the synthesis of layered crystals, at least a few chalcogen atoms remain in the ampoule, and their amount varies depending on the synthesis conditions. Anions are close packed in the mentioned structures. Therefore, they cannot have a large amount of anion deficiency (gap). It should be noted that anion deficiency can also be seen as cation excess.

Electron diffraction patterns were obtained from three samples taken from one ampoule. In these electron diffraction patterns, in addition to nets formed by strong reflexes obtained from the **unit cell** with parameters α=3.826 Å, which are formed by the close packed of sulfur atoms, there are also nets formed by weak reflexes, which have regular connections with this net.

Based on the electron diffraction patterns obtained from $Ga_{1.29}In_{3.38}S_7$ single crystals, the superlattice with $A_{1,2}=\sqrt{7}\,a$ parameter were discovered as a result of forming 6/7 of the octahedra included in the OOP package touching the OOP package with indium atoms and their bases, and 1/7 of the inter-package P_1(T) and $P_2(\overline{T})$ tetrahedra being occupied by gallium atoms . Also, on the basis of this electron diffraction patterns, superlattice with parameter A_3=2a was discovered as a result of filling 3/4 of the octahedra included in TO$\overline{T}$P packages with In atoms and leaving 1/4 of them empty.

Due to the lack of sulfur atoms, the amount of which varies from crystal to crystal, the amount of cation filling of octahedral (O) sites in TOTP packages varies. Quantitative analysis of the next two samples showed that their chemical composition was similar to $Ga_{1.30}In_{3.40}S_7$ and $Ga_{1.32}In_{3.45}S_7$. The results of examining the electron diffraction patterns obtained from the textures of $Ga_{1.30}In_{3.40}S_7$ and $Ga_{1.32}In_{3.45}S_7$ showed that their crystal structure is the same as that of $Ga_{1.29}In_{3.38}S_7$.

The strong reflections obtained from $Ga_{1.29}In_{3.38}S_7$ and related to the superstructure with superlattice parameter A=2a are weakened in the electron diffraction patterns obtained from $Ga_{1.30}In_{3.40}S_7$, $Ga_{1.30}In_{3.40}S_7$, and completely disappear in the electron diffraction patterns obtained from $Ga_{1.32}In_{3.45}S_7$.

In the case of metal excess, part of the ¼ empty octahedral (O) sites in TOTP packages are filled with cations, and as a result, the reflexes belonging to the superlattice with parameter A=2a are weakened (or completely disappear). In this case, the amount of positive charges on the package is greater than the amount of negative charges. To ensure the electroneutrality of the package, part of the cations in the typical T and $\overline{T}$ tetrahedra move to the atypical $P_1(\overline{T})$ and $P_2(T)$ interpack tetrahedra, respectively. Metallic bonds are formed between the same atoms, and as a result, electroneutrality in the packages is restored.

The cation excess in $Ga_{1.32}In_{3.45}S_7$ samples is X = 4.77 – 4.67 = 0.1.

$OP_1(T,\overline{T})TO\overline{T}P_2(T,\overline{T})O$…arrangement of atoms along the c-axis is as follows:

$$S_1 6/7In_O\, S_2\, 1/7Ga_T + 1/2X_{\overline{T}}\, S_3(Ga,In)_T - 1/2X_T S_4\, (3/4In_O + XIn_O)$$

$$S_5\, (Ga,In)_{\overline{T}} - 1/2X_{\overline{T}}\, S_6\, 1/2X_T + 1/7Ga_{\overline{T}}\, S_7\, 6/7In_O\ldots$$

In the $TO\overline{T}P$ package, tetrahedra T $[(Ga,In)_T - 1/2X_T]$ and $\overline{T}$ $[(Ga,In)_{\overline{T}} - 1/2X_{\overline{T}}]$ are connected to the vertices of octahedra filled with $(3/4In_O + XIn_O)$ on both sides with their own vertices. .T and $\overline{T}$ tetrahedra are connected to each other by the center of inversion. Here, $P_1(T,\overline{T})$and $P_2(T,\overline{T})$) are close packed anions, oriented opposite to each other and filled no more than $1/3[1/7Ga_T + 1/2X_{\overline{T}}]$ and $[1/2X_T + 1/7Ga_{\overline{T}}]$ are tetrahedral sites.

Since In and Ga atoms have 3 positive valences, the $TOTP_1OOP_2$ package has 4.77 (number of positive ions) × 3 (valence) = 14.31 (amount of charge), positive charge and 7 × 2 = 14 negative charges. Therefore, the number of positive charges is greater than the number of negative charges (14.3 – 14 = 0.3). To restore the electroneutrality of the package, trivalent metal in the amount of 0.5X = 0.05 from each of the T and $\overline{T}$ tetrahedra of the TOTP package should pass to $P_1(\overline{T})$, $P_2(T)$ and P(O). $P_1(\overline{T})$ ilə $P_1(T)$ and $P_2(T)$ metals create metallic connections (dumbbells) and each of them uses one of their valence electrons. As a result, excess positive charges are lost. Cations in the P(O) octahedron, on the contrary, restore the charges lost in the OOP

package. As a result, the electroneutrality of the package (structural unit) is restored[13].

THE MAIN RESULTS:

1.After rotating by an angle φ=40° from the position perpendicular to the incident electron beam, based on electron diffraction patterns obtained by rotating a thin single-crystal film of $Fe_{0.75}Ga_{0.25}InS_3$ through an angle ω=45° around an axis perpendicular to the surface of the crystal holder during recording, a new 2H polytype with lattice parameters a=3.78 Å, c=24.44 Å and sp. gr. $P6_3mc$ was discovered. (The single-crystal film is located parallel to the crystal holder surface);

2. Based on electron diffraction patterns obtained by rotating a thin single-crystal film of $Ga_{0,8}In_{1,2}S_3$on the surface of the crystal holder and perpendicular to the incident electron beam at an angle of φ=70° during recording, a new two-package hexagonal (2H) polytype with lattice parameters a=3.82 Å, c=24.53 Å, sp.gr. $P6_3mc$, $_hT_hO_кT_hP$ structure module and superlattice parameter $A=\sqrt{3}\,a$ was discovered (The single-crystal film is located parallel to the crystal holder surface);

3. Schemes explaining the rotation of the single-crystal film around an axis perpendicular to the surface of the crystal holder while the single-crystal film is located at an arbitrary angle on the surface of the crystal holder and the registration of the sites of the reciprocal lattice on the Ewald plane which oblique located are presented. Based on these schemes, a large number of electron diffraction patterns were recorded and investigated. The schemes can be used in future studies of the electronic structure of nanoobjects oriented at an arbitrary angle to the surface of the crystal holder;

4. A new 2H-polytype (sp. gr. $P6_3mc$) of the following crystals was detected with the help of electron diffraction patterns obtained by

[13] M. G. Kyazumov, S. M. Rzayeva, A. S. Avilov. Crystallography Reports, 2023, 68 (7), p. 1005–1009.

special tilt and subsequent rotation of the crystallite together with single-crystal film:

- $Mg_{0.7}Ga_{1.4}In_{0.8}S_4$, lattice parameters: a=3.81 Å, c=24.39 Å,
- $MnGaInS_4$, lattice parameters: a=3.80 Å, c=24.55 Å

In both cases, schemes are given that indicate the origin of reflexes located in non-standard places and facilitate the examination of electron diffraction patterns very easily (single-crystal films are located at an angle to the surface of crystal holder).

Also known 3R-polytypes (sp. gr. R3m) of the following crystals were identified:

- $Fe_{0,25}Ga_{0,5}In_{1,25}S_3$, lattice parameters: a=3.78 Å, c=36.78 Å,
- $Fe_{0.75}Ga_{0.25}InS_3$, lattice parameters: a=3.78, c=36.66Å;

Single-crystal films are placed at an arbitrary angle to the surface of the crystal holder.

5. The results of the study of polytypism in layered crystals of $CdInGaS_4$ compound obtained by the method of chemical transport reaction showed that if only the electron diffraction patterns obtained from the obliqued texture are used, in addition to the known polytypes 1T, 2H and 3R, polytypes 3T and 6R are incorrectly identified. By examining rotation electron diffraction patterns from thin single-crystal films, it was determined that the erroneously detected 3T and 6R polytypes are actually a mixture of 1T and 3R and 2H and 3R polytypes, respectively.

6. Super crystal lattices of $Ga_{1.29}In_{3.38}S_7$ crystals with lattice parameters a=3.82 Å, c=63.41 Å, sp.gr. R_3m and structural type $TO\overline{T}$ POOP with parameters $A_{1,2}=\sqrt{7}\,a$, $A_3=2a$ and $A_1\hat{}A_2=22°$ and schemes of formation of reciprocal lattices are given and justified. It was determined that $Ga_{1.30}In_{3.40}S_7$ and $Ga_{1.32}In_{3.45}S_7$ new composition crystals formed in $Ga_{1.29}In_{3.38}S_7$ crystals due to the lack of sulfur, reflections from the superlattice with parameter $A_3=2a$ with the partial filling of 1/4 empty spaces in the octahedra included in the $TO\overline{T}P$ package accordingly weakens and disappears.

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