

FİZİKA-RİYAZİYYAT VƏ TEXNİKA ELMLƏRİ

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PHASE ANALYSIS OF GaSe SINGLE CRYSTALS CARRIED OUT BY NEW ELECTRON DIFFRACTION'S METHOD OF ROTATION

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The electron diffraction patterns of GaSe single – crystal films (SCF) obtained by their rotation around the axis perpendicular to the film's plane which are inclined at an angle φ from perpendicular position to the incident electron beam as well as electron diffraction patterns obtained by rotation of SCF around axis α^* of reciprocal lattice perpendicularly positioned to the electron beam have been studied. On contrary the electron diffraction patterns of oblique textures in which the reflection series superimpose and therefore it is impossible to diversify ε and β polytypes, we can clearly determine different polytype modifications on SCF obtained by flaking off the crystal adhesive tape synthesized by Bridgman method. These are ε (sp. gr. $P\bar{6}m2$) and β (sp. gr. $P6_3mc$) ones with lattice parameters: $a=3,76^0 \text{ \AA}$, $c=15,95^0 \text{ \AA}$.

Keywords: new rotation methods, electron diffraction, inorganic compound structure

Introduction

Close-packed prismatic layers filled by X-M-M-X typed metal atom vapors form the structures of GaS, GaSe, InSe polytype sets. Polytype 2H is elementary with chalcogen arrangement (position A,B and C in close packing) and metals (position α,β,γ) – $A\beta\beta AB\alpha\alpha B$, sp.gr. $P6_3/mmc$, 2T, $A\beta\beta AB\gamma\gamma B$ ($P\bar{6}m2$) polytype and 3R, $A\beta\beta AB\gamma\gamma BC\alpha\alpha C$ ($R3m$). There have been also 4H, 6H, 9R, 12R, 18R₁, 18R₂, 21R polytypes and others [1]. The crystals chosen from different parts of the same ampoule can be differed by structure so that different polytype modifications can co-exist in the same crystal.

Among the well-known diffractometric methods the electronographic method of oblique textures is the most valuable for investigation. But of course there might be some deficiencies as well. Primarily textured samples can be studied by this method. Dispersion can disturb not only the crystal lattice perfection but lead also to the phase transitions in some cases. Electron diffraction patterns of single crystal

rotation (EDPSCR) have some advantages in compare with the electron diffraction patterns of texture. EDPSCR gives the local diffraction and structural information relating to the individual crystals but electron diffraction patterns of textures give us just only averaged information about on a number of micro- and nanodimension crystals. That is why in this paper we have presented new electron diffraction's method of crystal rotation which have been used to investigate GaSe single crystals.

Experimental part and result's discussion

GaSe crystals synthesized by Bridgman method are cleaved perpendicular to layers into two parts. Crystal structures of one of them have been investigated by method of oblique texture. Experimental samples were obtained by microdice deposition from aqueous suspension (derived by easy crushing with subsequent ultrasonic dispersion) in the aqueous form on the metal gauze covered by celluloid films. Because

se the crystals were layered after depositing them from the suspension on the film one of the crystal orientation remained. The experiment was carried out on a high-voltage electron diffractometer EG-400(V-350kV, $2L\lambda=33,2 \text{ mm}^0 \text{ \AA}$). In Fig.1a there has been shown electron diffraction pattern from GaSe textures. Even the most conservative crushing can partially result in a destruction of crystal structure.

The second part of GaSe crystals was used to obtain thin single crystal films (SCF) being appropriated in the electron diffraction study. Thin SCF have been prepared by adhesive tape flaking off the heavy crystal and then investigated by electron diffraction method of rotation developed by M.G. Kyazumov [2,3].

In Fig.1b there has been shown electron diffraction pattern of GaSe thin single crystal film (SCF), where SCF is inclined at an angle $\varphi = 55^\circ$ from perpendicular position to the incident electron beam and rotating through $\omega = 50^\circ$ around axis perpendicular to the film plane is made out. As it can be seen from the figure the diffraction field is considerably bigger here than in electron diffraction pattern taken from textured polycrystalline sample (Fig.1a).

Lattice parameters are: $a=3,76 \text{ \AA}$, $c=15,95 \text{ \AA}$, respectively. At the second (series 11 ℓ) and fifth (series 30 ℓ) ellipses reflections with add values of ℓ do not extinguish. This indicates on presence of ε (sp. gr. $P\bar{6}m2$) polytype modification of GaSe.

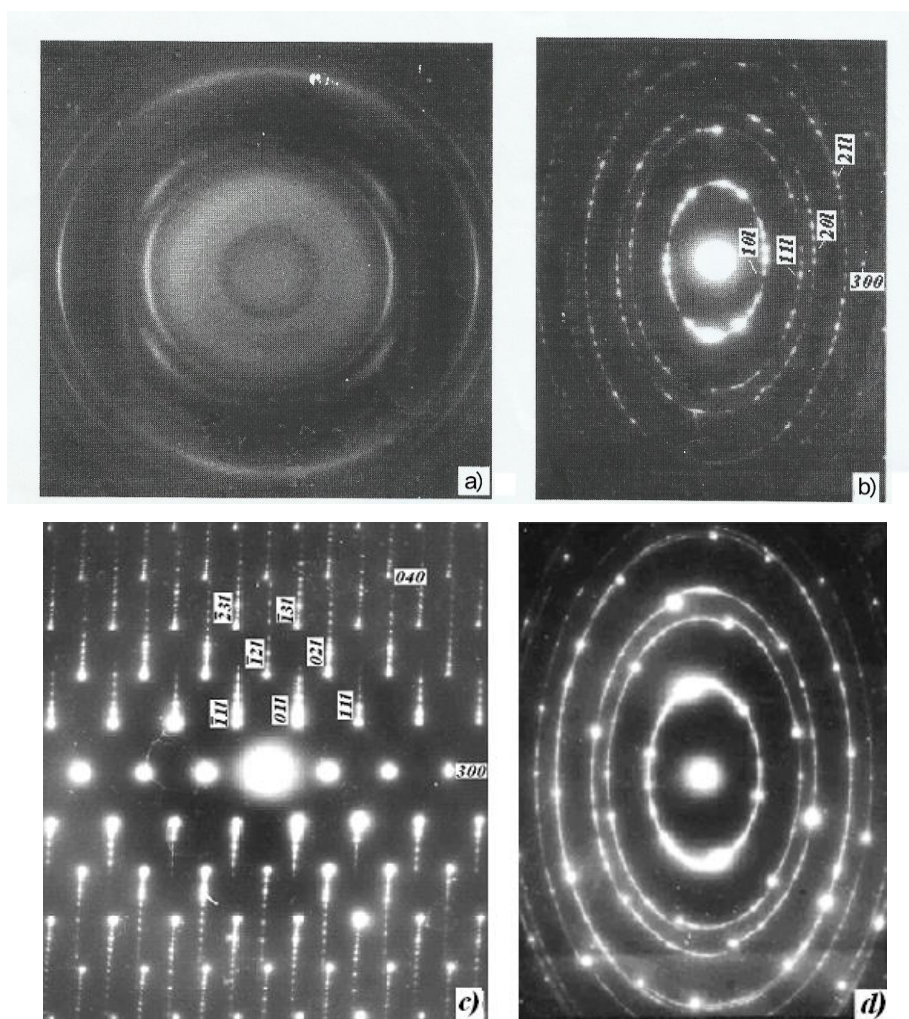


Fig.1. GaSe electron diffraction patterns:
a) of textures; b),c), d) of single crystal nanofilms

In Fig.1c there has been given electron diffraction pattern from GaSe thin single crystal film (SCF) obtained by SCF rotation around α^* axis of reciprocal lattice perpendicularly positioned to the electron beam. Initial position complies with the $hk0$ plane of the reciprocal lattice (this position is held well under electron beam and hence emerges more highly). Thus it is easy to measure R_{hk0} having big value when determining lattice parameters and establishing three-dimensional crystal lattice. Lattice parameters agree with: $a=3,76 \text{ \AA}$, $c=15,95 \text{ \AA}$. There has been seen six symmetry, symmetry plane and sharply seen $l \neq 2n$ types of extinctions for reflexes $12l$. Therefore given electron diffraction pattern fits β - phase of GaSe with space group $P6_3mc$.

In Fig.1d there has been presented oblique-angled mesh of point reflections. Azimuthal rotation around the normal to the plane of sample holder reveals ellipses system to which point reflexes belong. These ellipses unite the reflexes which are representatives of six knots of reciprocal lattice rows parallel to rotation axis and equidistant from it. As a result, they correspond to hexagonal basis of elementary cell and are characteristic of constant values of h^2+k^2+lk to which small lengths of their axis are proportional.

Structural disorder of two-layered GaSe polytype (Fig.1d) manifest itself apparently by marking continuity section of ellipses where point and ruling maxima indicating the small thickness of coherent scattering regions are denoted.

Conclusions

By surveying the stationary crystal only the contact or imaging the knot site on Evald's

sphere might be taken place. By rotating the crystal Evald's sphere passes through the whole volume taken by each knot site of reciprocal lattice.

Therefore the obtained information on reflexes intensity in electron diffraction patterns of rotation is closely true if the rotation occurs uniformly and reflexes are not superimposed each other.

In suggested patterns tilting angles can be over 70° . Hence the number of registered knot sites is rather more than in the case of precessional electron diffraction (PED) where tilting angle is $\sim 3^\circ$.

Unlike the electron diffraction patterns of oblique textures where the dissimilar series of reflections superimpose and therefore there is no possibility to distinguish ϵ and β polytypes in the electron diffraction patterns obtained by new methods of rotation, various series emerge separately. In different SCF obtained by adhesive tape flaking off the crystal synthesized by Bridgman method the different polytypic modifications have been determined, i.e. ϵ and β polytypes.

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REFERENCES

1. Terhell J.C. Polytypism in the III-VI layer compounds //Polytype structures. Pergamon press, 1983, pp.55-110
2. Kyazumov M.G. New diagrams of electron diffraction pattern production of hexagonal and monoclinic crystals. Kristallografiya 59(2014), pp.549-555
3. Zvyagin B.B., Zhukhlistov A.P., Kyazumov M.G., Fominenkov A.M. Electron diffraction patterns of crystal rotation. Kristallografiya, 35, N 3 (1990), pp. 602-609.

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YENİ FIRLANMA ELEKTRON -DİFRAKSİYA ÜSULU İLƏ GaSe MONOKRİSTALLARININ FAZA ANALİZİ

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Elektron şüasına perpendikulyar vəziyyətdən φ bucağı qədər döndərilərək səthinə perpendikulyar ox ətrafında fırlandırılan GaSe nazik monokristallik təbəqələrindən (MT) alınmış elektronogrammlar və həmçinin elektron şüasına perpendikulyar yerləşdirilmiş tərs qəfəsin α oxu ətrafında fırlandırılan MT-dən alınmış elektronogrammlar öyrənilmişdir. Müxtəlif sıralara aid olan reflekslərin üst-üstə düşməsi səbəbindən ϵ və β politiplərini bir- birindən fərqləndirmək mümkün olmayan maili teksturadan alınan elektronogrammlardan fərqli olaraq, yeni fırlanma metodları ilə alınmış

elektronoqrammalarda müxtəlif sıralara aid olan reflekslər üst-üstə düşür. Bricmen metodu ilə sintez olunmuş kristallardan alınmış müxtəlif MT-lərdə müxtəlif politip modifikasiyalar, b.s. qəfəs parametrləri $\alpha=3,76 \text{ \AA}$, $c=15,95 \text{ \AA}$ olan ε (f.qr. $P6m2$) və β (f. qr. $P6_3mc$) politipləri aşkar edilmişdir.

Açar sözlər: yeni fırlanma üsulları, elektronların difraksiyası, qeyri-üzvi birləşmələrin strukturu

ФАЗОВЫЙ АНАЛИЗ МОНОКРИСТАЛЛОВ GaSe, ПРОВЕДЕННЫЙ С ПОМОЩЬЮ НОВЫХ ЭЛЕКТРОННО-ДИФРАКЦИОННЫХ МЕТОДОВ ВРАЩЕНИЯ

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Изучены электронограммы монокристаллических пленок (МП) GaSe, полученные вращением МП вокруг оси, перпендикулярной к плоскости пленки, которая заранее наклонена на угол φ от перпендикулярного положения к падающему электронному пучку, а также электронограммы, полученные вращением МП вокруг оси α обратной решетки, перпендикулярно расположенной к электронному пучку. В отличие от электронограмм косых текстур, где различные серии рефлексов накладываются друг на друга, и потому невозможно различить ε и β политипы, на разных МП, полученных отслаиванием липкой лентой от кристалла, синтезированного методом Бриджмена, установлены разные политипные модификации, т.е. политипы ε (пр. гр. $P6m2$) и β (пр. гр. $P6_3mc$) с параметрами кристаллической решетки: $\alpha=3,76 \text{ \AA}$, $c=15,95 \text{ \AA}$.

Ключевые слова: новые методы вращения, дифракция электронов, структура неорганических соединений