STRUCTURAL INVESTIGATIONS OF $Zn_{1,5}In_3Se_6$ POLYTYPE 3R BY MEANS OF NEW ELECTRON-DIFFRACTION ROTATION METHODS

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The electron-diffraction pattern of monocrystalline films (MF) $Zn_{1.5}In_3Se_6$,obtained by rotation of MF round axis perpendicular to film plane, which earlier is inclined on φ angle from perpendicular position to incident electron beam and also electron-diffraction pattern obtained by MF rotation round a^* axis of reciprocal lattice perpendicular located to electron beam, are studied. The thin structural effects and different series appear separately on electron-diffraction patterns obtained by new rotation methods as opposed to electron-diffraction patterns of oblique textures where the thin structural effects appear and different series of reflexes superimpose one on another. The three-package rhombohedral polytype (3R) with crystal lattice parameters a = 4.046 Å, c = 59.292 Å, sp. gr. $R3m$ and also superlattice parameter $A_{s.l.} = \sqrt{3}\, a$ are found.

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

The electron-diffraction methods are more effective ones to investigate of layered crystals. The development of nanotechnology stimulates the design of new electron-diffraction methods having the specific advantages to investigate of nanosamples. The present paper is devoted to study of nanothick monocrystalline films (MF) $Zn_{1,5}In_3Se_6$ by new electron-diffraction rotation methods [1-4]. Earlier the three-package rhombohedral polytype (3R) is defined by electron-diffraction patterns of $Zn_{1,5}In_3Se_6$ textured samples [5].

EXPERIMENTAL PART AND RESULT DISCUSSION

$Zn_{1,5}In_3Se_6$ crystals synthesized by ChTR (chemical transport reaction), are divided into two parts perpendicularly to layers. The crystalline structure of one of them is studied by method of oblique texture. The samples for experiment are obtained by precipitation of micro-crystals (obtained by easy comminution with further dispergation by ultrasound) from suspension in water on metallic grid covered by celluloid film. The one orientation remains constant, in connection with layered structure of crystals, after the crystal precipitation from the suspension in water on the film. The experiment is carried out on high-voltage electronograph EG-400 (V=350kV, 2Lλ=33,2mmÅ).

The electron-diffraction patterns from $Zn_{1,5}In_3Se_6$ textures is shown in fig.1. The electron-diffraction pattern interpretations are made by the following formulas for oblique textures [6]:

$$d_{100} = 3a/4 = 2L\lambda h/2R_{h00}, \quad (1)$$

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

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

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

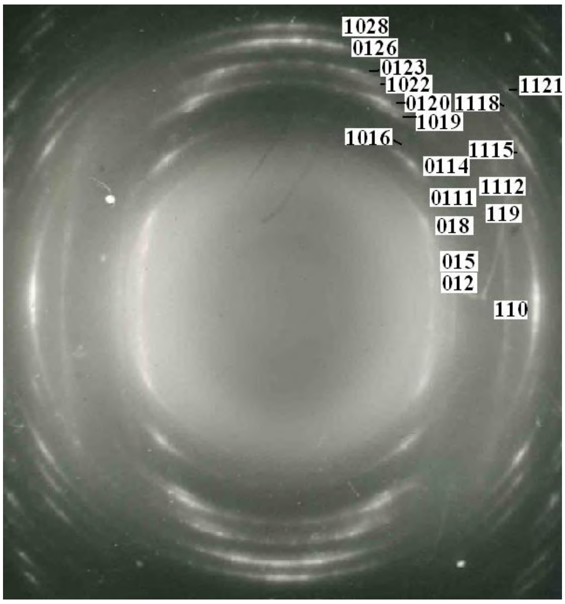

Fig.1. Electron-diffraction pattern of $Zn_{1,5}In_3Se_6$ textures.

The polytype 3R with elementary cell parameters a = 4.046, c = 59.292 Å, sp. gr. $R3m$ and with structure module $\ldots_h T_c T_h O_h T_c T_h P\ldots$, where T and O are two-dimensional tetrahedral and octahedral layers, P is empty interlayer, h and c are hexagonal and cubic package of Se atomic planes, is established.

The metal disposition (a,b,c positions) in compact selenium package (A,B,C positions) is following:

…AbBaCcApCaAcBbCpBcCbAaBp…,

…Se_1 2/3In;1/3Zn Se_2 1/3In;0,42Zn Se_3 In Se_4 1/3In;0,42Zn Se_5 2/3In;1/3Zn Se_6 P…

The other part of $Zn_{1,5}In_3Se_6$ crystal is used for obtaining of thin MF, suitable for electron-diffraction investigation.

The thin MF are obtained by film exfoliation from thick crystal by the adhesive tape.

The thin MF are studied by electron-diffraction rotation methods developed by M.G.Kazumov [1-4].

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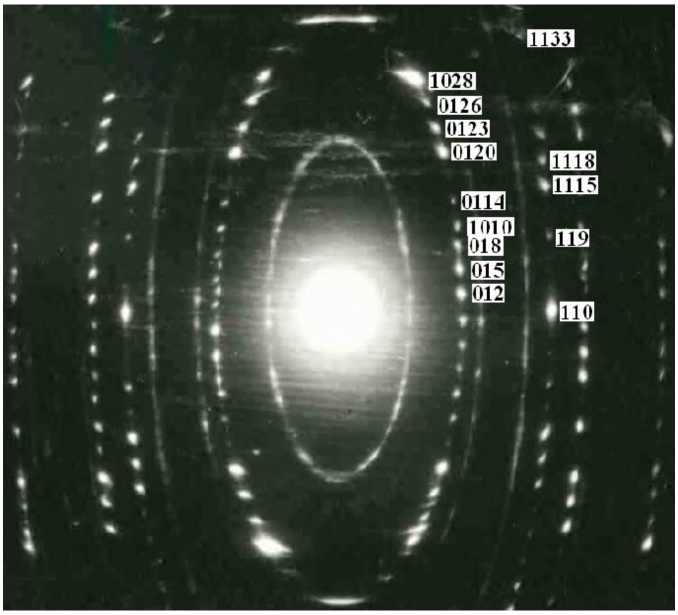

Fig.2. Electron-diffraction pattern of monocrystalline rotation imitating the electron-diffraction patterns of $Zn_{1,5}In_3Se_6$ oblique texture type.

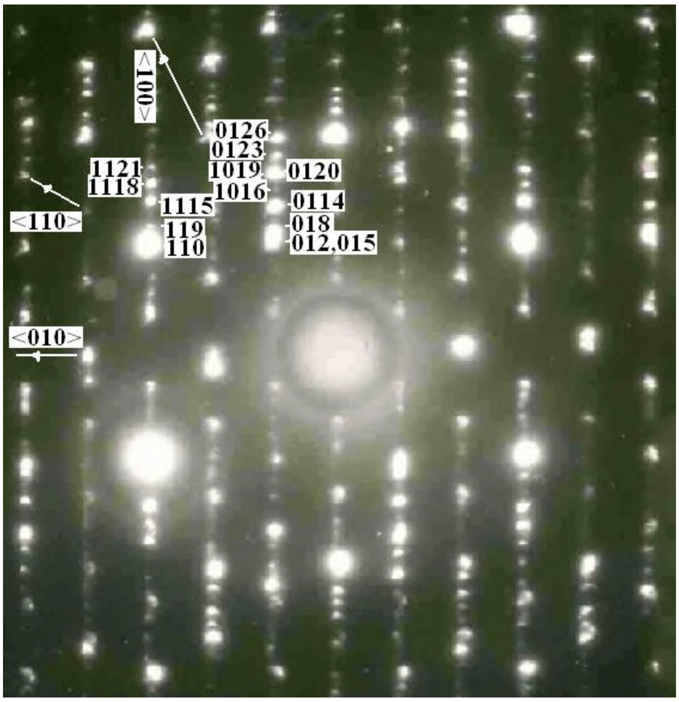

Fig.3. Electron-diffraction pattern of 3R-polytype $Zn_{1.5}In_3Se_6$ rotating round axis of *a** reversal lattice.

The electron-diffraction pattern obtained by rotation (during exposition) of MF film on $\omega = 60^0$ angle round axis perpendicular to film plane which is previously inclined to incident electron beam on angle $\varphi = 60^0$ (reciprocal lattice plane *hk0* is in MF plane), is shown in fig.2. The main crystal lattice parameters a = 4.046 Å, c = 59.292 Å, sp. gr. $R3m$ and also superlattice parameter $A_{s.l.} = \sqrt{3}\,a$ in basic plane of main lattice, are found. The reflexes being on weak ellipses which are first, third, fourth, seventh, eighth and other ones are to superlattice. The reflexes being on the strong ellipses, which are second, fifth, sixth, ninth and other ones are to main lattice. The reflex intensities in fig.1 coincide with ones of corresponding reflexes of main lattice (fig.2) and it shows the structure identity (fig.2).

The electron-diffraction pattern obtained by rotation of MF film $Zn_{1.5}In_3Se_6$ round a^*axis of reversal lattice perpendicularly situated to electron beam, is shown on fig.3. The registration is begun from *hk*0 plane; the essential delay of this plane under electron beam is admitted. The polytype 3R with above mentioned lattice parameters and super lattice is observed. The different node series (series of reflexes) appear separately but the nodes with *l* small values join each other in each node series *hk* (*h*, *k* = const, *l* changes). The quantity of joined reflexes depends on value and on distance of node row and on rotation c^* axis, i.e. on sp. R_{hk0}.

CONCLUSION

The electron-diffraction patterns of monocrystal rotation (EMR) have the additional advantage series besides the texture ones have. EMR give the concrete-local diffraction and structural information relating to isolated crystals and electron-diffraction patterns from textures give the integral averaged information relating to crystal variety. In EMR the higher sensitivity to weak diffraction effects, which can't be observed in electron-diffraction patterns of textures and polycrystals, is observed. The reflexes in EMR localize in the point form and they spread in the form of parenthesis or total rings in electron-diffraction patterns of textures and polycrystals.

The other advantage is the possibility to avoid the consequence of object dispergation accompanying to preparation of polycrystals and textures.It can not only destroy the crystalline structure perfection, but lead sometimes to phase transformations. So, for example, the graphite comminution causes the polytype transition 2H-3R.

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