NEW ROTATION ELECTRON-DIFFRACTION METHODS AND THEIR APPLICATIONS

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Electron diffraction patterns of the $CdInGaS_4$ and $Mg_{0.7}Ga_{1.4}In_{0.8}S_4$ single-crystal thin films were obtained by rotation around the normal to the plane of the crystal holder, which was a priori tilted relative to the position perpendicular to the incident electron beam. The thin single-crystal films in the first case was located on the surface of the crystal holder and in the second time was made a certain angle with it. For the second case, a diagram explaining the origin of some of the reflections positioned unconventionally (beyond the ellipse line) is presented. This scheme will help facilitate electron-diffraction studies of the crystal structures of other nano-samples in the case of their arbitrary orientation.

Keywords: New diffraction methods, Electron diffraction, Inorqanic materials
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INTRODUCTION

When shooting the stationary single-crystal films (SCF), only touching or projection of the site onto the Ewald plane (EP) can take place. When the crystal rotates, the EP passes through the entire volume occupied by each site of the reciprocal lattice (RL). If the rotation is uniformly and there is no superposition of reflections, the obtained information about the intensity of reflections in the electron diffraction patterns of rotation is right. In the proposed schemes, the angles of rotation (tilt) can be more than 70°. Therefore, it is especially important to study thin SCF by various electron diffraction methods of rotation.

When the single crystals films rotates around any axes (in particular, around the axis a of atomic lattice) lying in the film plane (FP) (before rotation, the electron beams fall perpendicularly on the FP), the site rows hk (hk=const, l-changes) of the reciprocal lattice are registered on the EP along lines perpendicular to the axis of rotation. Such electron diffraction patterns are imitated as electron diffraction patterns of needle like textures.

When the single crystals films rotate by an angle of ω=60° around the normal to the plane of the crystal holder, which was a priori (before the exposure) tilted by an angle of φ< 70° relative to the position perpendicular to the incident electron beam, obtained electron diffraction patterns are imitated as electron diffraction patterns of lamellar oblique textures.

EXPERIMENTAL AND DISCUSSION OF THE RESULT

Thin SCF were obtained by peeling off a thick crystal with adhesive tape. The electron diffraction patterns were obtained on an EG-400 electron diffraction recorder at an accelerating voltage of V=350 kV.

Two cases were considered. The thin single-crystal films in first case are located on the surface of the crystal holder, and in the second case make a certain angle with it.

By tilting and rotating a thin SCF in various ways (thin SCF lies exactly on the plane of the crystal holder), we obtain an electron diffraction pattern of the type of lamellar oblique textures (Fig. 1a) and acicular textures (Fig. 1b) [1]. From the reflections h00 we determine the value of the parameter a of the crystal lattice, and from the reflections 10*l* the value of the parameter c. By the distribution of reflections 11*l*, we determine the thickness of the package (layer, structural unit), and by the value of the second strong reflection (in this case, 116) in this series, we establish the structural type [2].

The diffraction patterns were interpreted using the well-known formulas for electron diffraction patterns of oblique textures [3]:

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

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

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

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

Here, L is the distance from the sample to the photoplate; λ is the wavelength of incident electrons; h, k, and *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 $L\lambda$ scale at *h, k* = const; ΔD is the distance between adjacent sites along the c* axis on the $L\lambda$ scale; and c* is the reciprocal lattice parameter.

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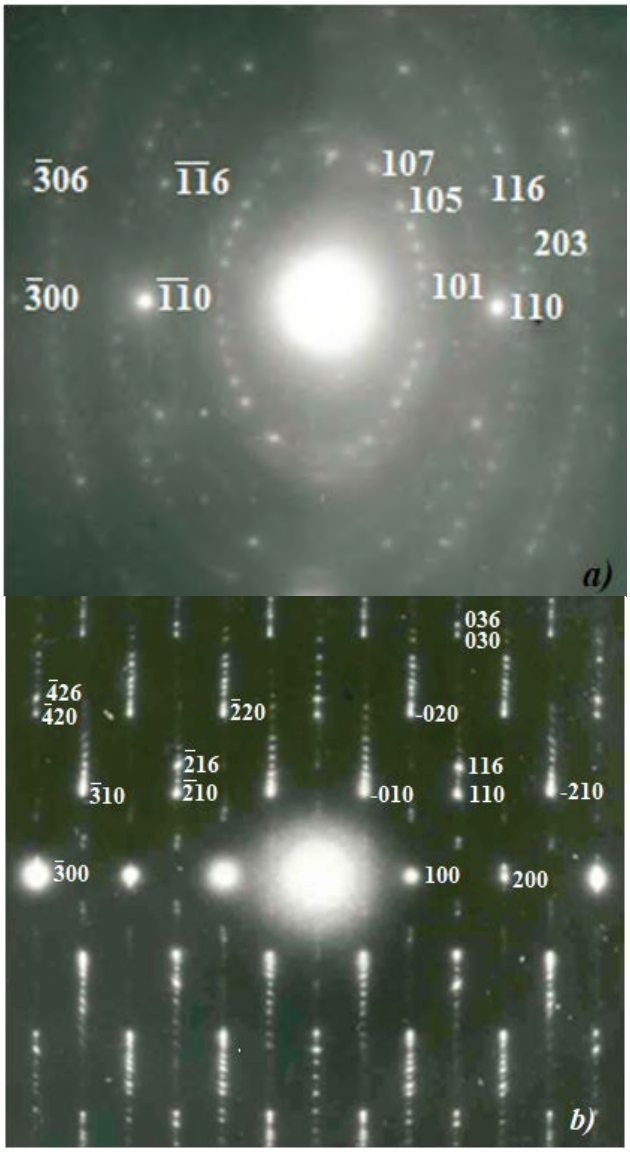
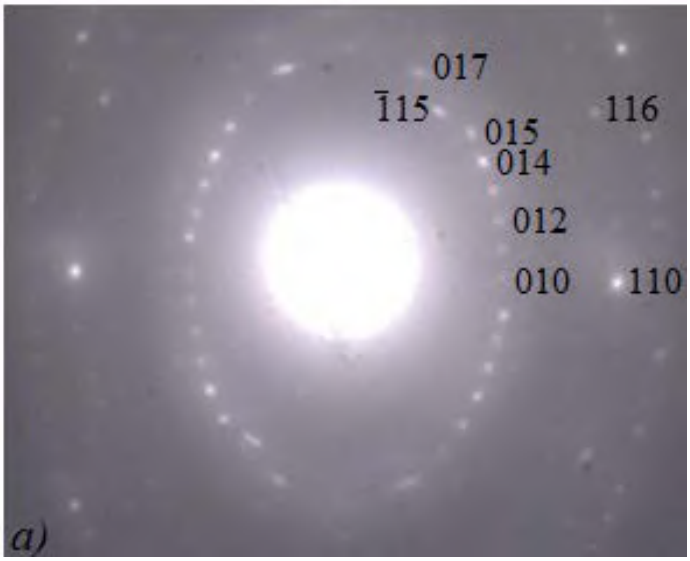


Fig. 1. Rotation electron diffraction pattern of the $CdInGaS_4$ single-crystal thin films: a) electron diffraction patterns imitating electron diffraction patterns of lamellar texture. b) electron diffraction patterns imitating electron diffraction patterns of acicular texture.



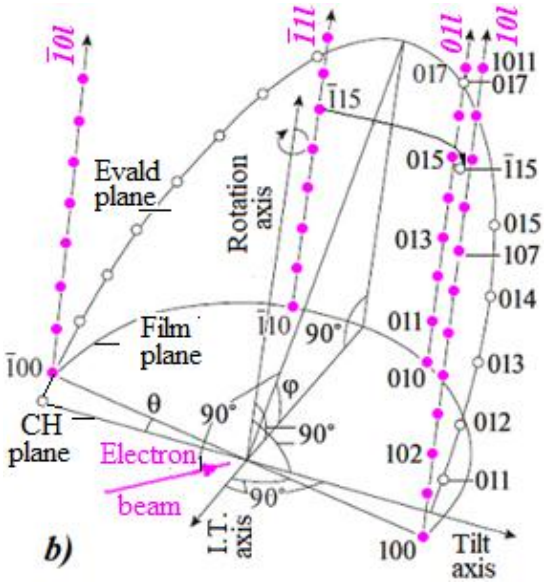


Fig. 2. 2H-polytype $Mg_{0.7}Ga_{1.4}In_{0.8}S_4$ (φ = 35°, ω = 60°) single crystal: a) rotation electron diffraction pattern (φ = 35˚, ω = 60˚) and b) scheme of rotation and detection of sites (and, consequently, reflections) of the reciprocal lattice in the Ewald plane. Red circles show reciprocal lattice sites located parallel to the 00*l* axis, open circles correspond to the 01*l* and *l* 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.

For some reason, the TSCF does not lie on the plane of the crystal holder (CH), and we are unable to correct it. In such cases, after tilting the CH and rotating around an axis perpendicular to the CH plane, in the obtained electron diffraction pattern, some reflections are mixed from the line of ellipses. The electron diffraction patterns simulated lamellar textures were obtained for the $Mg_{0.7}Ga_{1.4}In_{0.8}S_4$ single-crystal thin films. In this case, the film makes certain angle θ with the crystal holder plane. The electron diffraction patterns were obtained by tilting the crystal holder by an angle of ϕ = 35° and subsequent rotation by an angle of ω = 60° around the normal to the crystal holder plane (Fig. 2a). We give diagrams explaining such rotation and pointing (helping) to easy processing of the obtained electron diffraction patterns (Fig. 2b) [4]. The crystal lattice parameters determined from the electron diffraction pattern were a = 3.80 Å, c = 24.39 Å and sp. gr. $P6_3mc$.

CONCLUSION

In the proposed schemes, the angles of inclination can be more than 70°. Consequently, the number of registered nodes will be much greater than in the case of Precession Electron Diffraction when the inclination angle is ~3°.

Different site rows (series of reflections) come out separately, but when $a << c$ in the site rows hk (h,k=const, *l*-changes), reflections with small values of *l* are superimposed on each other, and the greater the distance of the site rows hk from the axis rotation, the more overlapping reflexes. How can these shortcomings be corrected?

By rotating the film around an axis perpendicular to the film plane (FP), it is possible to bring the studied site row hk closer to the rotation axis to such a distance that during rotation the individual reflections will take place.

In addition, with such rotations during exposure to the EP, different RL planes are recorded in turn. Thus, secondary electron diffraction can strongly affect the intensities of all sites of an individual plane of reciprocal lattice.

When the SCF rotates around not the coordinate axes, but the axes lying in the coordinate plane of reciprocal lattice, there is no superposition of reflections with large values of *l* on the obtained electron diffraction patterns. In addition, during such a rotation, only a few reflections are simultaneously recorded on the EP. Obviously, the kinematic scattering dominates in this situation.

Electron diffraction patterns of the $CdInGaS_4$ and $Mg_{0.7}Ga_{1.4}In_{0.8}S_4$ single-crystal thin films were obtained by rotation by an angle of ω 60° around the normal to the plane of the crystal holder, which was a priori (before the exposure) tilted by an angle of φ< 70° relative to the position perpendicular to the incident electron beam. Two cases were considered. The single-crystal films of the first compound are located on the surface of the crystal holder, and the film of the second compound makes a certain angle with it. For second case, a diagram explaining the origin of some of the reflections positioned unconventionally (beyond the ellipse line) is presented. This diagram can be used in further electron diffraction study of the structure of nanoobjects oriented arbitrarily on a crystal holder

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