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GROWN, CRYSTAL STRUCTURE AND MECHANISM OF PHOTOCONDUCTIVITY OF BiOCl SINGLE CRYSTALS

Purpose. Oxyhalides of bismuth BiOX (X = Cl, Br, I) are very interesting materials which find various applications as X-ray luminescent screens, as anti-Stokes converters, photocatalyst, usual luminophors and as photoconductive analyzer of linear polarized radiation in the 0.24–1.2 μ m spectral region. The great interest for these materials is strongly related to the influence of dimensionality on the behaviour of physical properties (they are 2D structured materials). Bismuth oxyhalides are one of the V–VI–VII group compound semiconductors belonging to the tetragonal system.

Methods. The BiOX single crystals were grown by the chemical gas transport reactions method in the closed volume. The lattice parameters of the crystals were determined by X-ray powder diffraction with a Si internal standard (high purity) spectral dependences of the steady-state photocurrent (photoconductivity) were recorded using a LOMO MDR3 monochromator. Low temperatures were obtained by mounting the sample in an UTREKS continuous flow cryostat. The decay time measurements were performed using pulse UV-laser LGI-21.

Results. The best bismuth oxyhalide crystals were obtained at the 8-10 Torr pressure of transport agent. The layered structure BiOX can provide the space large enough to polarize the related atoms and orbitals. The induced dipole can separate the hole–electron pair efficiently, enhancing photocatalytic activities. BiOX has an indirect-transition band-gap so that the excited electron has to travel certain k-space distance to be emitted to valence band

Conclusions. The optimal temperature region on BiOCl crystals based photodetectors is in the range 80- 400 K. BiOCl single crystals are stable in air, and their parameters do not depend on the environment, which eliminates the need for sealing. The selectivity of BiOCl spectral characteristics make them good radiation detectors of the nitrogen, argon and helium-cadmium lasers, as well as some of the semiconducting light-emitting diodes (LEDs). Therefore, they can be used in optoelectronic circuits in conjunction with these lasers and LEDs.

Keywords: bismuth oxyhalides, layer single crystals, photoconductivity, semiconducting light-emitting diodes (LEDs.)

Introduction

In recent years, two-dimensional (2D) materials, such as single crystals, nanoplates and nanosheets, have attracted much attention because of not only their unique electronic, magnetic, optical, and catalytic properties, which mainly arise from their large surface areas, nearly perfect crystallinity, structural anisotropy, and quantum confinement effects in the thickness [1, 2].

Oxyhalides of bismuth BiOX (X = Cl, Br, I) are very interesting materials which find var-

ious applications as X-ray luminescent screens, as anti-Stokes converters, photocatalyst, usual luminophors and as photoconductive analyzer of linear polarized radiation in the 0.24–1.2 μ m spectral region [3]. Bismuth oxyhalides provides a very convenient matrix for activation by various rare-earth and 3d-elements: by doping this matrix with different, is possible to obtain a wide variety of optical, luminescent and photoconductive properties. The great interest for these materials is strongly related to the influence of dimensionality on the behaviour of physical properties. Bismuth oxyhalides are one

of the V–VI–VII group compound semiconductors belonging to the tetragonal system [4–6].

Bismuth oxychloride crystals exhibit many intriguing and interesting properties such as photoluminescence, photoconductivity, and thermally stimulated conductivity [7]. The luminescent photoconductivity bands at 1.6–2.2 eV are a result of c-band free electrons capture by recombination r-centres. Heat conductivity measurements as a function of temperature showed that the electronic component of heat conductivity is negligibly small, since the crystals are nearly insulators in the temperature range 90–300 K [8].

Experimental details

The BiOX single crystals were grown by the chemical gas transport reactions method in the closed volume (typically of the order $10 \times 10 \times 0.5 \text{ mm}^3$). The crystals exhibit tetragonal matlockite (PbFCl) type structure (space group $D_{4h}^7 - P_{4/n}^2 mm$; $Z = 2$). The trivalent Bi^{3+} ion is surrounded at one side by four oxygen ions and at the other side by five halide ions, four of which are in a plane, whereas fifth is situated on the fourfold axis. There is only one crystallographic site available for the trivalent ion and this site has C_{4v} symmetry.

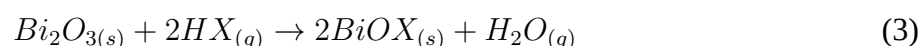
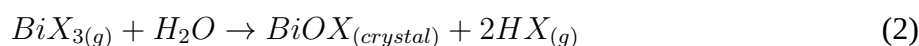
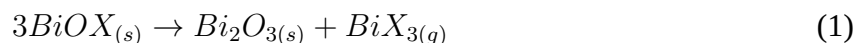
The lattice parameters of the crystals were determined by X-ray powder diffraction with a Si internal standard (high purity). The spectral dependences of the steady-state photocur-

rent (photoconductivity) were recorded using a LOMO MDR3 monochromator. Low temperatures were obtained by mounting the sample in an UTREKS continuous flow cryostat. The decay time measurements were performed using pulse UV-laser LGI-21 (pulse width 8-9 ns; excitation wavelength $\lambda = 0.337 \text{ nm}$; filters UFS-2 and UFS-6 were used). The change in the intensity was done by a platinum reducer.

Results and discussion

Grown and crystal structure of bismuth oxyhalide single crystals

The BiOX samples were prepared in several ways. BiOX pure (starting material for a BiOX single crystals growth) was prepared by dissolving 99,99% pure bismuth oxides in hydrohalides acid, evaporating to form the hydrated halides, dehydrating the latter under vacuum, heating at $t \sim 380\text{--}700^\circ\text{C}$ in a X_2 atmosphere, cooling to room temperature, and washing the product to remove BiX_3 . The BiOX crystals were grown by the vapour gas transport reaction method in closed volume. The compounds BiOX of 99,99% purity was loaded into a high quality polished quartz ampoule (150-165 mm length and 15 mm diameter; combination of cylinder and cone). The ampoule was evacuated under vacuum of 10-4 Torr. After pumping the ampoule was filled with a (H_2O and HX) transport agent (TA). The system of chemical gas-transport reactions are as follows [1, 3]:



During transport involved three gaseous compounds: H_2O , HX and BiX_3 . In the solid state exists a bismuth oxyhalides in polycrystalline and single crystal form. Reaction (1) describes the dissociation process of bismuth oxyhalides. The reaction (2) is a direct gas transport reaction with the water vapour as a transport agent. Reaction (3) describes the halogenation of bismuth oxide to bismuth oxyhalides.

The (1-3) reactions cycle is closed and carried to the final transport in a BiOX single crystal. The decomposition (1) and hydrolysis (2) reactions of BiOX are endothermic. Therefore, transport is possible only in the $T_2 \rightarrow T_1$ direction ($T_2 > T_1$). A two-zone resistance heated furnace was used for crystal growth. The axis of the ampoule was kept slightly inclined to the horizontal to enhance the transport of the vapour

from the source zone to the growth zone during growth. We selected the pressure of transport agent in the ampoule on the basis of optimal mass transfer calculation by the convection method according to the equation (2). The

best bismuth oxyhalide crystals were obtained at the 8-10 Torr pressure of transport agent. The some growth parameters of the BiOX crystals obtained are given in the table 1.

Table 1: Temperature of source and growth zones, transport agent pressure and size of BiOX crystals obtained

Substance	Zone temperature (°C)		TA pressure (Torr)	Morphology and size	Growth period (days)
	Source	Growth			
BiOCl	720	600	8-10	Platelets 7x7x3 mm ³	5
BiOBr	700	600	8-10	Platelets 6x7x2 mm ³	6
BiOI	580	450	8-10	Platelets 5x5x1 mm ³	7

The crystallinity and morphology of the BiOX crystals was examined by X-ray diffraction (XRD), and field effect-scanning electron microscopy (FE-SEM). The lattice parameters were refined by X-ray powder diffraction data with a Si internal standard (high purity) and by using a least square method. All of the sharp diffraction peaks in the XRD patterns were perfectly indexed as pure phase of BiOCl, in good agreement with the standard JCPDS file (No. 06-0249) of BiOCl. A Laue photograph taken parallel to the large face of the platelets showed clearly a fourfold symmetry axis characterizing (001) planes of a matlockite tetragonal PbFCl-like structure (No. 129) [9,10] with two formula units in the unit cell.

Fig. 1a shows the clinographic projection of the unit cell of BiOX crystals. Each Bi atoms is eight-coordinated by four O atoms and four X (halogen) atoms in the form of an asymmetric decahedron [10]. Each atom (Bi, O, X) occupies a special position with multiplicity 2. The faces of the decahedron are 2 rectangles (O-O-O-O, X-X-X-X), which are parallel to the (110) plane and 8 isosceles triangles (four X-O-X and four O-X-O). The decahedra are linked to each other by a common O-X edge along the a and b axes forming infinite layers. The coordination of O and X are follows. Each O atom is linked to four Bi atoms and forming a tetragonal pyramid with the O atom at its apex. Also each X atoms forms with the neighbouring Bi atoms tetragonal pyramid with the X atom at

its apex. Neighbouring decahedra form layers along (001) which are connected by common O-X edges. Neighbouring layers of decahedra are connected by common O-O or X-X edges.

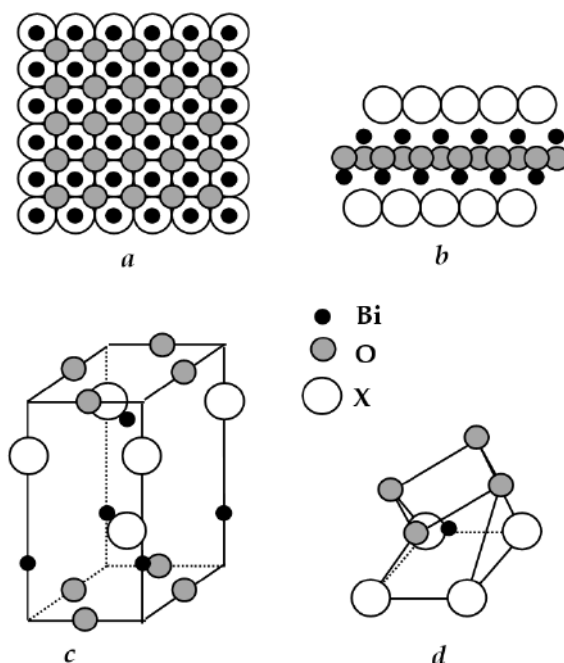


Figure 1: Crystal structure of bismuth oxyhalides: (a) projection to (001) plane; (b) projection to (100) or (101) plane; (c) clinographic projection of the unit cell; (d) coordination polyhedron of the bismuth.

The X atom is bonded with four Bi atoms in a planar square to form a pyramid and with its nonbonding (lone pair) electrons pointing to the other side of the square. These nonbonding electrons convert the three-

dimensional fluorite-like structure into a two-dimensional layered structure. In the three-dimensional Bi₂O₃, the Bi atoms are coordinated to six O atoms. This structural difference between them is the major reason for BiOX to have a wider optical band-gap. As shown in the Fig. 1, the [BiOX] layers are stacked together by the nonbonding (van der Waals) interaction through the X atoms along the c-axis. Therefore, the structure is not closely packed in this direction. When one photon excites zone

electron from X np states (n = 3, 4, 5 for Cl, Br, I respectively) to Bi 6p states in BiOX, one pair of a hole and an excited electron appear. The layered structure BiOX can provide the space large enough to polarize the related atoms and orbitals [11]. The induced dipole can separate the hole–electron pair efficiently, enhancing photocatalytic activities. BiOX has an indirect-transition band-gap so that the excited electron has to travel certain k-space distance to be emitted to valence band (Table 2).

Table 2: Lattice parameter (a, c), relaxed atomic position (z, z'), Debye temperature (θ_D) and band gap (E_g) of bismuth oxyhalide crystals [7, 8]

Substance	Latt. const., Å		Relaxed atomic position		θ_D , K	E_g , eV
	a	c	z	z'		
BiOCl	3.883	7.347	0.645	0.170	205	3.455
BiOBr	3.915	8.076	0.653	0.154	168	2.924
BiOI	3.984	9.128	0.668	0.132	146	1.89

Mechanism of photoconductivity in BiOX (X=Cl, Br, I) single crystals

The original specimens had a dark resistivity of $\rho_d = 10^{10}$ to 10^{11} Ω -cm and significant photo-sensitivity [12]. At a temperature 293 K and an illumination of 104 lx, the ratio $\frac{\rho_d}{\rho_{ph}}$ (ρ_{ph} is the resistivity in the light) reached 103; cooling of the specimen led to an increase of 3-4 orders of magnitude in the steady-state photocurrent such that at 90 K, $\frac{\rho_d}{\rho_{ph}} > 10^7$. Thus, there was an intense temperature (T) suppression of the photocurrent (see Fig. 2a).

The spectral dependence of the photoconductivity $\sigma_{ph}(h\nu)$ for a typical specimen of BiOCl recorded at 95 K (curve 1) and 293 K (curve 2) is illustrated in Fig. 2b. In addition to the intrinsic maximum $h\nu_1 = 3.54$ eV (350 nm) which corresponds to the band gap of $E_g = 3.5$ eV at T = 100 K a significant impurity photoconductivity is observed due to the transfer of

an electron from the r-centre to the conduction band [1]. On heating, the main maximum is shifted towards the long-wave side at a rate of $\frac{dE_g}{dT} = -6.3 \cdot 10^{-4}$ eV/K, while the photoconductivity in both the impurity and intrinsic regions falls sharply. The depth of the level which produces the impurity photocurrent maximum was found to be $E_{cr}^0 = 2.2$ eV [3].

The lux-ampere characteristics of the photocurrent (Fig. 3) at low temperatures up to the region of T-suppression are linear in the experimental illumination range $\alpha = 1$ ($\alpha = \frac{dL}{dI_{ph}} \frac{L}{I_{ph}}$; $I_{ph} \sim L^\alpha$). In the T-suppression region the lux-ampere characteristics become superlinear and the value of α depends on the concentration of photocurrent carriers n, at a fixed temperature. The maximum value of α observed in the experiment reached $\alpha =$ up 3 to 4. At low temperatures, an optical IR suppression of the photocurrent was observed [3, 4, 12].

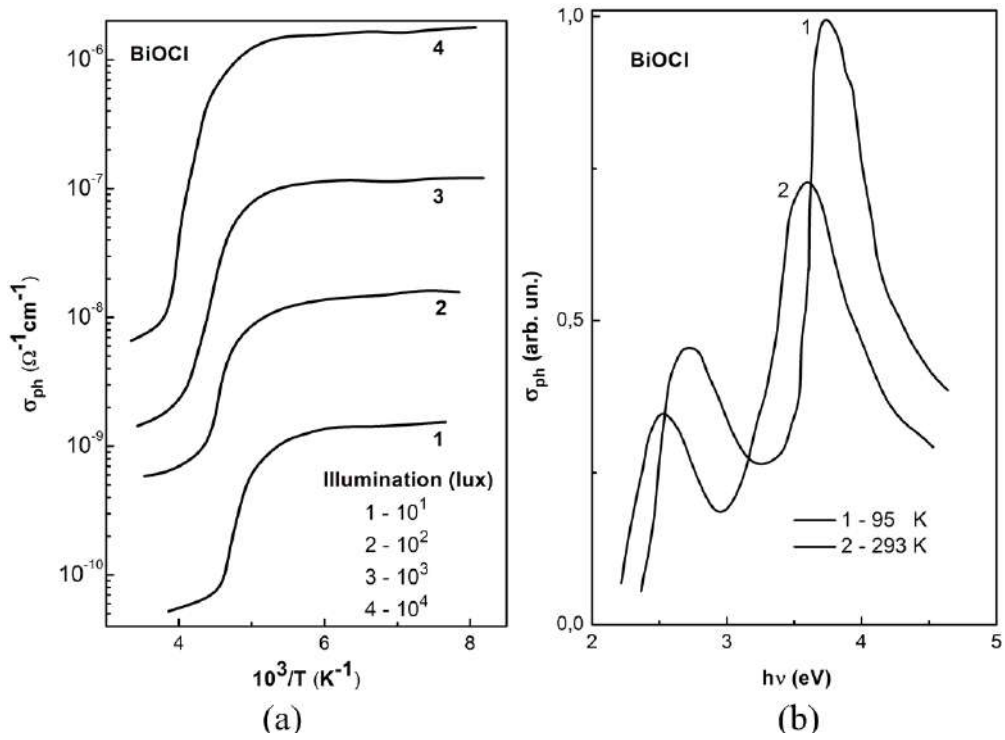


Figure 2: The temperature dependence (a) and the spectral dependence (b) of photoconductivity BiOCl single crystal

A study of the photoconductivity kinetics was made in the linear regime: $\Delta n \ll n$, where Δn is the change in n caused by a short pulse of light in the intrinsic excitation region. The curve of the decay of the photocurrent during the illumination by a pulse of intrinsic light consisted, of course, of two sections with clear by different times: $\tau_r = (2.5 - 6.6) \cdot 10^{-3}$ and $\tau_s = (0.8 - 4) \cdot 10^{-5}$ s.

The presence of temperature and IR suppression of the photocurrent, the superlinearity of the lux-ampere characteristics, and the two sections observed on the relaxation curves of the photoconductivity, indicate unequivocally that in these particular crystals, the recombination processes are determined by two types of centres: "sensitizing" r centres and fast s centres.

The energy distance of the r centres from the valence band is E_{Vr}^T and the thermal value was determined from the dependence on the carrier concentration of the temperature at which the thermal suppression of photocurrent begins as in [4, 12]: $E_{Vr}^T = 1.3$ eV.

Another more accurate method of determining E is to measure the dependence of $\frac{\Delta L}{L}$ on $\frac{1}{T}$, where ΔL is the change in intensity of the exciting light L which is necessary for maintaining the electron concentration n , with a change

in the temperature in the T-suppression region,

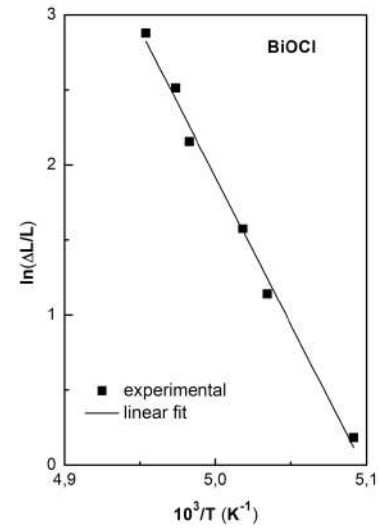


Figure 3: Temperature dependence of the $\ln \frac{\Delta L}{L}$ value

$$\frac{\Delta L}{L} \sim q_s Q_\nu \frac{c_{rp}}{c_{rn}} \exp - \frac{E_{Vr}^T}{kT} \quad (4)$$

where q_s is the portion of the total carrier-recombination current passing through the s centres; c_{rp} and c_{rn} are the probabilities of hole or electron trapping at an r centre; and Q_ν is the static factor of the valence band. From the curve

of $\ln \frac{\Delta L}{L} = f(10^3/T)$ (Fig. 3) was determined $E_{Vr}^T = 1.3$ eV [1, 3].

Conclusion

The optimal temperature region on BiOCl crystals based photodetectors is in the range 80-400 K. BiOCl single crystals are stable in air, and their parameters do not depend on the environment, which eliminates the need for sealing. It was established experimentally that the adsorption of oxygen leads to a slight increase even values of photosensitivity [1, 3, 4]. Other adsorbents do not cause changes in the photoelectric properties. Crystal structure of the sam-

ple reduces the intrinsic noise of the radiation detector. The functional photoresistors based on BiOCl single crystals, included in electronic circuits as a two-terminal networks, what is their great advantage. They can follow the rotation of polarization plane or work in schemes of the maximum signal finding. Besides, the BiOCl crystals can be used as photo-potentiometers. The selectivity of BiOCl spectral characteristics make them good radiation detectors of the nitrogen, argon and helium-cadmium lasers, as well as some of the semiconducting light-emitting diodes (LEDs). Therefore, they can be used in optoelectronic circuits in conjunction with these lasers and LEDs.

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РОСТ, КРИСТАЛЛИЧЕСКАЯ СТРУКТУРА И МЕХАНИЗМ ФОТОПРОВОДИМОСТИ МОНОКРИСТАЛЛОВ BiOCl

Монокристаллы оксигалогенидов висмута представляют значительный интерес в различных областях применения: в качестве рентгеновских люминесцентных экранов, анти-стоксовских преобразователей излучения, фотокатализаторов, обыкновенных люминофоров и фотопроводящих анализаторов линейно-поляризованного излучения в спектральном диапазоне 0.24–1.2 микрона. Вышеуказанные материалы являются слоистыми кристаллами с четко выделенным двухмерным проявлением физических свойств (структурированные 2D-материалы). Оксигалогениды висмута относятся к группе V–VI–VII широкозонных полупроводниковых материалов тетрагональной сингонии. Монокристаллы BiOCl обладают четко выраженным слоистым строением за счет комбинации слоев ионов галогена, кислорода и висмута. В данной работе представлены результаты исследования спектров фотопроводимости монокристаллов BiOCl.

Ключевые слова: оксигалогениды висмута, слоистые монокристаллы, фотопроводимость, полупроводниковые светоизлучающие структуры

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РІСТ, КРИСТАЛІЧНА СТРУКТУРА ТА МЕХАНІЗМ ФОТОПРОВІДНОСТІ МОНОКРИСТАЛІВ BiOCl

Монокристали оксигалогенідів висмуту становлять значний інтерес в різноманітних галузях застосування: в якості рентгенівських люмінесцентних екранів, анти-стоксівських перетворювачів випромінювання, фотокатализаторів, звичайних люмінофорів та фотопровідних аналізаторів лінійно-поляризованого випромінювання

в спектральному діапазоні 0.24-1.2 мікрона. Вищевказані матеріали є шаруватими кристалами з чітко вираженим двовірним проявом фізичних властивостей (структуровані 2D- матеріали). Оксигалогеніди вісмуту відносяться до групи V-VI-VII широкозонних напівпровідникових матеріалів тетрагональної сингонії. Монокристали BiOCl мають чітко виражену шарувату будову за рахунок комбінації шарів іонів галогену, кисню й вісмуту. В даній роботі представлені результати дослідження спектрів фотопровідності монокристалів BiOCl .

Ключові слова: оксигалогеніди вісмуту, шаруваті монокристали, фотопровідність, напівпровідникові світло-випромінюючі структури

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