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## GROWTH OF SINGLE CRYSTALS, PHOTOELECTRIC PROPERTIES AND THE ABSORPTION EDGE OF A NEW LAYERED $\text{CuGa}_{2.5}\text{In}_{2.5}\text{S}_8$ COMPOUND

Efim Arama <sup>1</sup>, ORCID: 0009-0005-8606-6266,  
Valentina Pinteau <sup>2</sup>, ORCID: 0000-0003-0137-4699,  
Tatiana Shemyakova <sup>3</sup>, ORCID: 0000-0003-1668-0082,  
Natalia Gasitoi <sup>4</sup>, ORCID: 0000-0002-5895-286X

<sup>1</sup> „Nicolae Testemitanu” State University of Medicine and Pharmacy, Chisinau, Republic of Moldova

<sup>2</sup> Technical University of Moldova, Chisinau, Republic of Moldova

<sup>3</sup> Institute of Applied Physics, Chisinau, Republic of Moldova

<sup>4</sup> Alecu Russo State University of Balti, Department of Mathematics and Informatics,  
Balti, Republic of Moldova

\* Corresponding author: Valentina Pinteau: [valentina.pinteau@fiz.utm.md](mailto:valentina.pinteau@fiz.utm.md)

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**Abstract.** In this article, the conditions for obtaining monocrystals of  $\text{CuGa}_{2.5}\text{In}_{2.5}\text{S}_8$ , were investigated, their optical absorption edge, electrical and photoelectric properties. Using the criteria for the formation of new layered chalcogenides with octahedral and tetrahedral coordination of cations, it was hypothesized that due to the replacement of half of the In atoms in the  $\text{CuIn}_5\text{S}_8$  spinel with Ga, which has a pronounced tendency to occupy the tetrahedral B sites in a denser packing of S atoms, a layered phase is formed. Melting of a stoichiometric mixture of  $\text{Cu}+2.5\text{Ga}+2.5\text{In}+8\text{S}$  led to the synthesis of a previously unknown single-phase product with a layered crystalline structure. Since copper, gallium, and indium sulfides are generally good photoconductors, it could be expected that the new compound would also exhibit high photosensitivity. The aim of this work was to investigate the conditions for obtaining of  $\text{CuGa}_{2.5}\text{In}_{2.5}\text{S}_8$  single crystals, their optical absorption edge, electrical and photoelectric properties.

**Keywords:** ternary semiconductors, optical absorption, photoluminescence, photoconductivity, chalcogenides.

**Rezumat.** În acest articol, au fost investigate condițiile pentru obținerea monocristalelor de  $\text{CuGa}_{2.5}\text{In}_{2.5}\text{S}_8$ , limita de absorbție optică și proprietățile lor electrice și fotoelectrice. Folosind criteriile pentru formarea noilor chalcogenide stratificate cu coordonare octaedrică și tetraedrică a cationilor, s-a admis că substituind jumătate dintre atomii de In din spinelul  $\text{CuIn}_5\text{S}_8$  cu Ga, care prezentau o tendință pronunțată de a ocupa spațiile tetraedrice B într-o împachetare mai densă a atomilor de S, s-a format o fază stratificată. Fuziunea unui amestec stoichiometric de  $\text{Cu}+2,5\text{Ga}+2,5\text{In}+8\text{S}$  a condus la sinteza unui produs monofazic necunoscut anterior, cu o structură cristalină stratificată. Având în vedere că sulfatul de cupru, galiu și indiu sunt, în general, buni fotoconductori, se putea aștepta că noul compus să prezinte, de asemenea, o sensibilitate fotoelectrică ridicată. Scopul acestei lucrări a fost de a investiga

condițiile de obținere a monocristalelor de  $\text{CuGa}_{2.5}\text{In}_{2.5}\text{S}_8$ , limita de absorbție optică și proprietățile lor electrice și fotoelectrice.

**Cuvinte cheie:** *compuși ternari semiconductori, absorbție optică, fotoluminescență, fotoconductivitate, calcogenizi.*

## 1. Introduction

Semiconductor sulfide compounds of the class  $A^{\text{II}}B_2^{\text{III}}C_4^{\text{VI}}$  (where A – Zn, Cd and B – In, Ga) are actively researched due to their unique physical properties – intense photoluminescence [1, 2] and high photosensitivity over a wide spectral range [2, 3]; they are high-resistance and stable under normal atmospheric conditions. In particular, the optical absorption, reflection, photovoltaic, and emission properties of ternary single crystals are described [2–4], which include various polytypic modifications [5, 6]. It has been demonstrated that  $\text{CuGa}_{2.5}\text{In}_{2.5}\text{S}_8$  single crystals in the form of low-resistance thin films are a promising material for use in n-p type solar cells [7].

Criteria for the formation of new layered chalcogenides with octahedral and tetrahedral coordination of cations are presented in [8-10]. The groups of layered crystals according to their crystallochemical properties are also described in the generalization works [11-14]. Another classification of layered crystals can be based on the type of the element that serves as the source of anions. Substances containing chalcogens (S, Se, Te) as anions also form groups of chalcogenides and are classified as semiconductors.

Layered chalcogenide structures occupy a special place in the theory and applicability of solid-state materials. Various technological methods are used to obtain high-quality single crystals of these compounds, and different experimental and well-established theoretical methods are employed for structural studies. One of the most accessible technological methods known in the literature is a method of chemical transport reactions. This method is widely used for growing single crystals of various compounds from this family.

Monocrystals of  $\text{CuGa}_{2.5}\text{In}_{2.5}\text{S}_8$  were obtained using chemical transport reactions in a closed system with iodine as a transportation agent. The advantage of this method is determined by the fact that chemical transport reactions occur at relatively low temperatures compared to other direct methods. The method of chemical transport reactions allows for the growth of single crystals with advanced parameters, of high quality, in the form of plates with an optically high-quality exterior surface. The perfection of the surface and the alignment of the main crystal faces enable the use of these crystals in devices, instruments, and for investigations of physical properties without the need for mechanical surface processing.

## 2. Experimental methodology

As a source of materials for crystal growth, the easily volatile components of chemical compounds can serve, which are subjected to thermal dissociation or appropriate recovery on the growth surface.

In this case, the crystallization processes occur in two successive stages: 1) the release (emission) of the substance as a result of the chemical decomposition reaction; 2) the incorporation of atoms into the crystalline lattice.

For the selection of substances, reversible heterogeneous reactions are used, the equilibrium constants of which, as usual, depend on temperature and the concentrations of all gaseous compounds. This means that even with some changes in conditions, the reversible

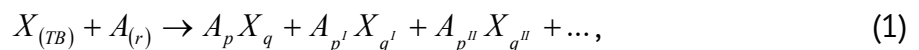
chemical process may occur, meaning that instead of crystallizing the substance, its dissolution may take place.

Since gaseous products are released during decomposition reactions, a steady and uniform process requires continuous removal, for which the use of flowing substances is appropriate.

The amount of crystallized substance emitted in a unit of time is determined by the yield of the decomposition reaction of the compound at a given temperature, the concentrations of the components involved in the reaction, and the flow rate of the gas mixture.

The method of transport reactions allows for the production of semiconductor compounds in the form of imperfect and very small crystals, the purity of which is unsatisfactory. Recrystallization of disaggregated compounds through melting or sublimation methods proves, for various reasons, to be ineffective or impossible. In these cases, interesting results can be achieved specifically by using the method of transfer reactions or gas transport reactions.

The essence of transfer reactions lies in the following: when the gaseous reagent  $A$  interacts with a solid, non-volatile substance that is to be processed at different temperatures and partial pressures, compounds of varying composition and concentrations of gaseous molecules, of the type  $A_pX_q$ , can be formed. This means the following reaction may occur:



where:  $X_{(TB)}$  - the solid and non-volatile substance,  $A_r$  - gaseous reagent,  $A_pX_q$  - the formed chemical compound,  $A_{p'}X_{q'}$ ,  $A_{p''}X_{q''}$  - different compounds based on the composition and concentrations of gaseous molecules.

Under these conditions, an equilibrium state is established among the different compounds  $A_pX_q$ , characterized by fixed partial pressures of all gaseous compounds. If the temperature of the system is instantaneously changed, the equilibrium state will be disrupted, and the composition of the compounds in the mixture will change. With certain fluctuations in temperature (pressure range), the decomposition of one of the gaseous products may occur, resulting in the release of substance  $X$ .

Under these conditions, a state of equilibrium is established among the different compounds  $A_pX_q$ , characterized by stable partial pressures of all gaseous compounds. If the temperature of the system is instantaneously changed, the equilibrium state will be disrupted, and the composition of the compounds in the mixture will change. During certain temperature fluctuations (pressure ranges), the decomposition of one of the gaseous products may occur, resulting in the elimination of substance  $X$ .

Based on the experiment, we can highlight the following thermodynamic features of the growth conditions we used to obtain quality compounds and their application in studying the modifications of the energy spectrum characteristics of charge carriers in the investigated single crystals: in the process of crystal formation, the transport rate must not exceed their growth rate; for the homogeneous growth of crystals, it is necessary to maintain a uniform temperature distribution in the growth chamber. When transport occurs via diffusion, single crystals with perfect faces are grown; a well-thought-out selection of transport conditions allowed for the obtaining of crystals that correspond to the initial stoichiometry of the compound [13, 15].

The container consisted of quartz ampoules with an inner diameter of 15 mm and a length of 15 cm. In some experiments, the starting material was a mixture of high purity elemental components taken in stoichiometric ratios, while in other experiments a pre-synthesized compound  $CuGa_{2.5}In_{2.5}S_8$  was used. In both cases, under a temperature regime of 870 – 820 °C, plate-like monocrystals of deep red color with thicknesses ranging from 2 to 100  $\mu\text{m}$  and areas up to 80  $\text{mm}^2$  were grown. The crystals, similar to mica, could be easily cleaved. The advantage of this method lies in the fact that chemical transport reactions occur at relatively low temperatures compared to other direct methods. The chemical transport reaction method enables the growth of single crystals with advanced parameters and high quality, in the form of plates with optically high-quality surfaces. The perfection of the surface and the alignment of the main crystal faces allow these crystals to be used in devices, apparatus, and for investigations of physical properties without the need for mechanical surface processing [16-19].

X-ray studies revealed that the obtained compound crystallizes in a hexagonal lattice with parameters  $a = 3.82$ ,  $c = 30.6 \text{ \AA}$ , and the  $\bar{c}$  axis is oriented perpendicular to the plates.

The crystal structure of most layered chalcogenides with octahedral and tetrahedral coordination of cations is described based on the closest hexagonal packing of anions, in which cations are arranged in layers within octahedral and tetrahedral voids [8, 9]. In this arrangement, a certain layer of voids remains completely free. The structural fragment (packet) between two layers of voids is connected to the adjacent packets by van der Waals forces. The obtained value of the  $c$  parameter for  $CuGa_{2.5}In_{2.5}S_8$  indicates the presence of two five-layer packets (in terms of sulfur layers) in the unit cell. Rewriting the chemical formula of the compound as  $Cu_{0.625}Ga_{1.5625}In_{1.5625}S_5$ , it is evident that the average (per packet) cation position filling factor amounts to 93.75%, meaning this substance is a type of defective layered chalcogenides with octahedral and tetrahedral coordination and is likely a structural analogue of the known crystal modification  $GaInS$  with unit cell parameters  $a = 3.8134$  и  $c = 30.656 \text{ \AA}$  belonging to the space group  $P6$  [20, 21]. In the latter substance, cations occupy different layers in the packet with varying density, and the average filling factor is 83.33%. The close values of the unit cell parameters of the two comparable compounds suggest that in the obtained compound the „excess” cations are distributed among the free vacancies in the known structure leading to packet densification.

For the measurements, samples with coplanar indium electrodes and surface-barrier structures (SBS)  $Pt-CuGa_{2.5}In_{2.5}S_8 - In$  were prepared. In these structures, the semi-transparent platinum and dense indium contacts were deposited on opposite faces of the plates by thermal vacuum evaporation. The indium electrode served as an ohmic contact, while the platinum electrode functioned as a rectifying contact. To enhance the dark conductivity of the  $CuGa_{2.5}In_{2.5}S_8$  crystals they were subjected to thermal treatment at temperatures of 600 – 650 °C for 10 - 15 min.

The dark specific resistance of specially undoped samples ranged from  $3 \cdot 10^{10} - 2 \cdot 10^{12}$  and  $10^6 - 10^{10} \text{ Ohm}\cdot\text{cm}$  along and perpendicular to  $c$ -axis, respectively. Annealing of the crystals resulted in a reduction in resistivity  $\rho$  by up to 10 times. Measurement of the thermoelectric voltage sign for the low-resistance samples indicated that the crystals exhibit n-type conductivity.

The rectification ratio of the surface - barrier structures (SBS) at a voltage  $U = 3 \text{ V}$  amounts to 10 - 100 and  $10^3 - 10^4$  for structures made from high-resistivity and low-resistivity

crystals, respectively. The forward voltage corresponds to a positive potential on the platinum electrode. The SBS structures generate a photovoltage of 0.47 – 0.86 V under illumination.

Based on the experiment, we can highlight the following thermodynamic features of the growth conditions we used to obtain high-quality compounds and their application in studying the modification of the energy spectrum characteristics of charge carriers in the researched single crystals: in the process of crystal formation, the transport rate must not exceed their growth rate; for the homogeneous growth of crystals, it is necessary to maintain a uniform temperature distribution in the crystallization zone. When transport occurs through diffusion, single crystals with perfect faces are grown; thoughtful selection of transport conditions allowed for the attainment of crystals that correspond to the initial stoichiometry of the compound.

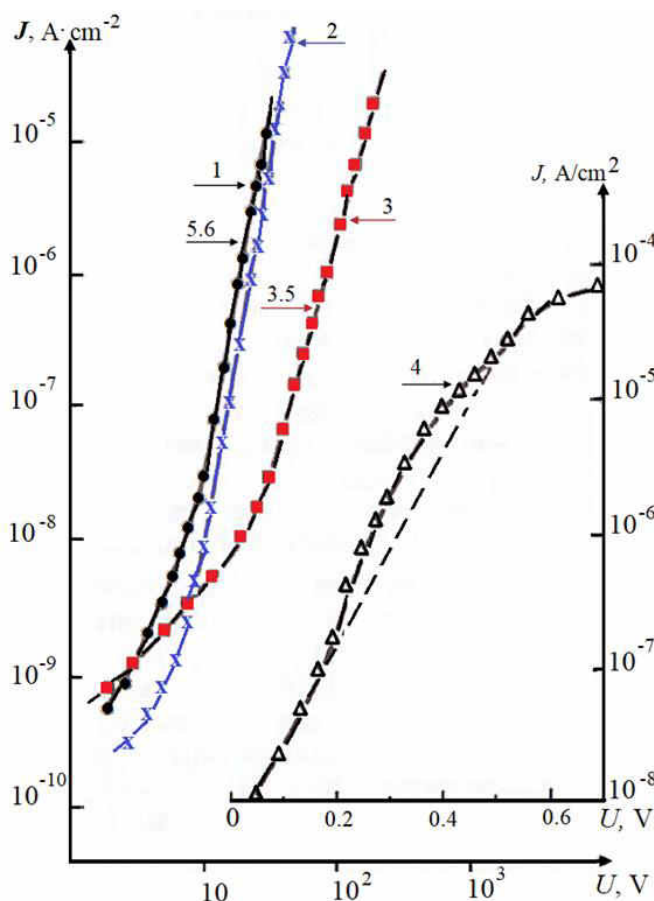
From the results obtained and the advantages of growth, the method of chemical transport reactions has proven to be the most technologically advanced for the studied materials [14].

### 3. Results and Discussion

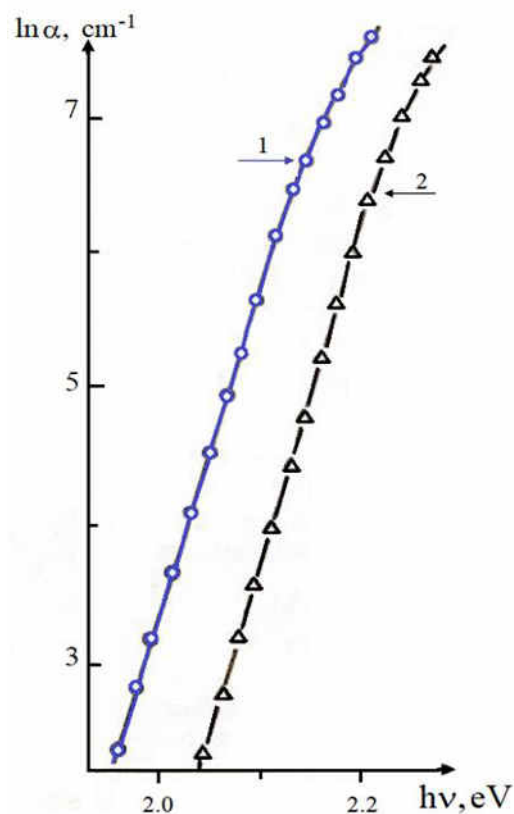
The forward branch of the current - voltage characteristic (I - U curve) of the SBS structures with a low - resistivity base at voltages below 1 V is described by an exponential dependence  $j \sim \exp(eV/\beta kT)$ , where the ideality factor  $\beta$  ranges from 1.6 to 2.3. When the voltage increases, a transition to an ohmic dependence  $j \sim U$  occurs. Larger values of the ideality factor indicate a tunnel-recombination mechanism for current flow through the space charge region at the platinum - crystal interface. For some surface - barrier structures, a region with a smoothly varying ideality factor was observed Figure 1, curve 4. This fact can be attributed to a complex nature of the distribution of local centers with respect to energy; however, detailing the charge transport mechanism requires further experiments and is a separate task.

The forward branch of the I - U characteristics of the samples with a high-resistivity base is also superlinear. When the voltage increases, it generally becomes linear and then follows a power law dependence  $j \sim U^n$ , where  $n = 3.5 \div 5.6$  (Figure 1, curves 1-3). This type of I - U characteristic indicates that at voltages  $U > 1V$ , the current flow is associated with charge transport through the SBS base and is determined by space charge limited currents with the presence of exponentially distributed traps in the crystal [21, 23]. The absence of a linear region in the I - U characteristics of some samples with a thickness less than 10  $\mu m$  is attributed to the fact that due to the low concentration of free electrons, the injection current exceeds the ohmic current even at low voltages.

The photocurrent spectra of SBS were studied in a photovoltaic mode using a load with a resistance of  $10^7$  Ohm, as well as with a direct bias of +4.5 V. The photoconductivity (PC) spectra were examined on coplanar  $CuGa_{2.5}In_{2.5}S_8 - In$  samples. The red PC edge (Figure 2, curves 1 and 2) of the crystals is approximately 1.9 eV, whereas for the SBS photovoltaic effect it is approximately 2.0 eV. This discrepancy indicates that this semiconductor contains photosensitive centers in the forbidden zone, which contribute to the photoconductivity for  $h\nu < E_g$ . The photoconductivity maximum at 2.2 eV corresponds to the excitation of electrons from these localized states into the conduction band.



**Figure 1.** Forward-bias region of the current-voltage characteristics of the Pt - CuGa<sub>2.5</sub>In<sub>2.5</sub>S<sub>8</sub> In structure at 300 K. Crystal thickness: (1) 5  $\mu\text{m}$ , (2) 7  $\mu\text{m}$ , (3) 27  $\mu\text{m}$ , and (4) 4  $\mu\text{m}$ .



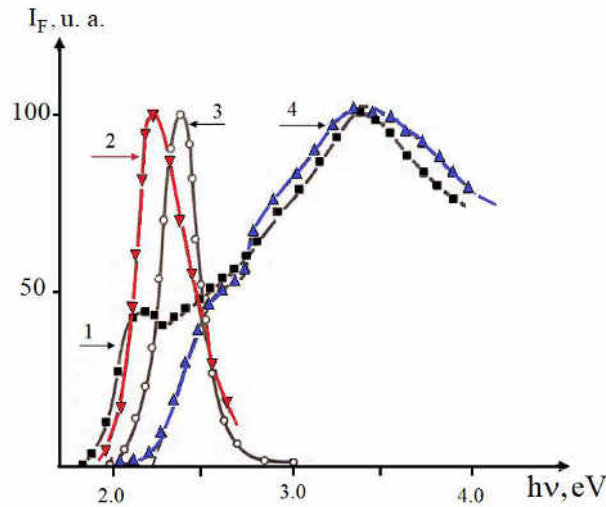
**Figure 2.** Spectra of photoconductivity of CuGa<sub>2.5</sub>In<sub>2.5</sub>S<sub>8</sub> single crystals (1) and the spectral distribution of the photoresponse of surface-barrier structures with a bias of +4.5 V (2).

The photocurrent of SBS with a low-resistance base is proportional to the number of photons absorbed in the depleted region of the crystal, namely, it is related to the optical absorption coefficient. By extrapolating the steep region of the low-energy wing of the photocurrent spectrum for such samples (Figure 3, curve 4), we obtain a value for the band gap width of  $\text{CuGa}_{2.5}\text{In}_{2.5}\text{S}_8$ , which is approximately 2.23 eV.

This spectrum exhibits a maximum at 3.4 eV and a feature at 2.7 eV. On the high-energy side of this feature the growth of photosensitivity with increasing photon energy ( $h\nu$ ) becomes more gradual. This characteristic of the photocurrent spectrum and the significant distance of the maximum from the photoresponse onset are apparently related to the structural features of the allowed energy zones of  $\text{CuGa}_{2.5}\text{In}_{2.5}\text{S}_8$  monocrystals.

The photoconductivity (PC) spectrum also exhibits a maximum at 3.4 eV and its slope changes around  $h\nu \approx 2.7$  eV. The possibility of observing features corresponding to interband electronic transitions in the spectral distribution of the PC is likely due to the low surface recombination rate, which allows the photocurrent to „track” changes in the optical absorption coefficient  $\alpha$ . Indeed, layered crystals are characterized by low adsorption capability, and at the (0001) face of the crystal the outer layer consists of anions with a 2-charge, whose ion - covalent bonds are closed within the structural package, similarly to the

bulk of the crystal. On such a surface, the conditions for recombination of non-equilibrium electrons are, in the first approximation, the same as those in the bulk.



**Figure 3.** Optical absorption edge of  $\text{CuGa}_{2.5}\text{In}_{2.5}\text{S}_8$  single crystals at 300 K (1) and 70 K (2), and in a photovoltaic mode (3, 4) at 300 K, with thicknesses of 47 (1), 4 (2), 5 (3), and 4  $\mu\text{m}$  (4).

The photocurrent spectrum of SBS devices fabricated from specially undoped crystals features a band with a full width at half maximum of 0.15–0.25 eV (depending on the crystal thickness  $a$ , with values of  $d = 20 \div 4 \mu\text{m}$ , respectively).

Since the base resistance in these structures is much higher than that of the load, the condition for photocurrent generation is the light flux penetration into the bulk crystal and excitation of photocurrent in the base.

The photocurrent spectrum of the SBS with a positive bias (Figure 2, curve 2) corresponds to the crystal (base) photocurrent when light propagates along the charge transport direction. Since for  $h\nu > E$  light is absorbed in the near-surface layer of the crystal, this results in a decrease in the base photocurrent and the photocurrent of SBS devices fabricated from specially undoped crystals.

The optical absorption edge of  $\text{CuGa}_{2.5}\text{In}_{2.5}\text{S}_8$  single crystals in the 1.95–2.2 eV region is described by an exponential dependence  $\alpha \sim \exp(h\nu/\Delta)$  where the parameter  $\Delta$  is temperature independent and equals 43 meV (Figure 3). The presence of an exponential region in the absorption spectrum is characteristic for chalcopyrite type crystals (ZnS, CdS, and other compounds based on these elements). However, the latter have generally higher  $\Delta$  values: such as 63 meV ( $\text{CdIn}_2\text{S}_2\text{Se}_2$ ), 65 meV ( $\text{HgGaInS}_4$ ), 44 meV ( $\text{HgGaInS}_4$ ),  $60 \div 77$  meV ( $\text{CdGaInS}_6$ ) [1, 2], and 98 meV ( $\text{ZnIn}_2\text{S}_4$ ) [23]. This type of edge absorption, typical for various multicomponent compounds, is attributed to the presence of the density of state tails near the allowed energy bands caused by disorder in the cation sublattice [24].

#### 4. Conclusions

The conditions for obtaining the new compound  $\text{CuGa}_{2.5}\text{In}_{2.5}\text{S}_8$  were experimentally established using the method of chemical transport reactions, with iodine as the transport agent. This method allowed for the production of perfect single crystals with a high-quality

optical surface. The advantage of this method lies in the fact that chemical transport reactions occur at relatively low temperatures compared to other direct methods.

Single crystals of a new  $\text{CuGa}_{2.5}\text{In}_{2.5}\text{S}$  layered compound, with n-type conductivity and good photoconductive properties, were grown using chemical transport reactions with iodine. It was demonstrated that vacuum thermal treatment can significantly increase their dark conductivity within a wide range. Based on this material, surface - barrier structures were fabricated that exhibit a high rectification ratio and excellent photosensitivity.

The parameters of the hexagonal unit cell were determined, and the bandgap width was assessed. The presence of photosensitive centers in  $\text{CuGa}_{2.5}\text{In}_{2.5}\text{S}_8$  crystals with a depth of approximately 1.9 eV was established. The absorption edge of this material features an extended exponential region with a characteristic parameter  $\Delta = 43$  meV.

**Conflicts of Interest:** The authors declare no conflict of interest.

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**Submission of manuscripts:**

[jes@meridian.utm.md](mailto:jes@meridian.utm.md)