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INVESTIGATION OF THE OPTICAL PROPERTIES OF METAL ION-INTERCALATED GaSe SEMICONDUCTOR MONOCRYSTALS

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In this study, the XRD method was used to characterise the structural and phase properties of GaSe monocrystals, and the impact of Cu ion intercalation on their optical properties was investigated. An increase in absorption (ABS) was observed in the 200–400 nm region as a result of the addition of Cu ions, and new optical transitions occurred around 600 nm. Overall, observable variations in absorption levels have been observed over the 200–800 nm spectral range. Tauc analysis revealed that the band gap narrowed from approximately 2 eV to about 1.88 eV upon intercalation and slightly widened to about 2.15 eV with photo-intercalation. These results suggest that Cu ion intercalation can be applied to modify the optical properties of GaSe monocrystals, increasing their potential for use in nonlinear optical devices, photonics, and sensor technologies. The findings also demonstrate that intercalation is an appropriate technique for regulating the physical characteristics of layered materials.

Keywords: *Band gap; Absorption; Intercalation; XRD diffraction*
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INTRODUCTION

Due to their significant potential in optoelectronics, photonics, and sensor technologies, layered semiconductor materials have been the subject of extensive research in recent years. Gallium selenide (GaSe) is unique among these materials due to its unique layered crystal structure, broad band gap (around 2 eV), and significant nonlinear optical characteristics. GaSe may crystallise in both orthorhombic and hexagonal systems, and its layered structure makes it possible to adjust its electrical and optical characteristics using a variety of modification techniques [1]. GaSe is a potential material for use in laser technologies, photodetectors, and nonlinear optical devices because of these characteristics [2–5].

The process of intercalation, which is the insertion of atoms, molecules, or ions into the interlayer spaces in such layered crystals, can have a major impact on the electronic structure and functional characteristics of the material [6]. The research suggests that intercalation may result in the creation of new localised energy states, band gap change, optical absorption spectrum broadening, and photoluminescence intensity regulation. In GeI_2 crystals intercalated with pyridine molecules, for example, band gap widening has been seen, whereas electrochemical intercalation has enhanced electrical conductivity in materials like MoS_2 and $ZrSe_2$, even causing a change from semiconducting to metallic behaviour [7–10].

The combined results of these investigations show that the intercalation process is a popular and efficient way to intentionally adjust the electrical and optical characteristics of materials with little structural harm. Intercalated layered materials have a wide range of potential applications in optoelectronic devices, as confirmed by the literature now under publication. This highlights the need for more study in this area [11-12,13].

However, the effects of intercalation on the optical properties of layered semiconductors like GaSe, such as absorption spectra, optical transitions, and energetic modifications of the band gap, have not yet been thoroughly examined. Previous study published in the scientific literature [1, 14-18] has concentrated chiefly on structural features and electrical conductivity. Electrochemical intercalation was chosen for this investigation because to its quick, energy-efficient, and economical application, as well as its repeatable and controllable nature. Additionally, it provides a viable approach for the creation of novel functional nanostructures. Because of their potential to improve electrical conductivity, modify the band gap, contribute to memory, energy storage, and more effectively thermal and chemical stability, copper ions were specifically chosen as intercalants. Nevertheless, little is known about the specific optical changes brought about by the intercalation of transition metals like Cu into GaSe crystals, especially with regard to wavelength-dependent effects and structural changes of the band gap. This emphasizes the necessity of additional experimental research. Therefore, by investigating the impact of Cu intercalation on the optical properties of GaSe crystals and exploring the potential applications of the ensuing alterations, this work aims to fill the existing gap.

EXPERIMENTAL METHODOLOGY

The layered GaSe single crystals investigated in this study were grown using the Bridgman–Stockbarger directional crystallization technique [1]. This method was specifically chosen due to its ability to produce high-quality

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single crystals with a homogeneous structure and excellent crystallinity, yielding large, optically smooth GaSe crystals with a characteristic bright red appearance. The technique enabled the growth of samples with high purity and well-preserved layered morphology, which are essential for accurate optical investigations.

A detailed sample preparation protocol was implemented before measurements. High-quality specimens were selected based on their mirror-like surface morphology and scanned along the layer planes to ensure structural uniformity. Structural and compositional analyses were performed, including X-ray diffraction (XRD), to verify the crystalline order and homogeneity of the samples. These characterizations were essential not only to confirm the composition of the GaSe crystals but also to monitor changes induced by the intercalation process.

The intercalation itself was carried out via electrochemical intercalation under a constant electric field. The GaSe single crystal samples were placed between electrodes separated by a 2.5 cm distance and subjected to the process for 1 hour. Experiments were conducted under two different conditions – illuminated and dark environments – to examine the effect of external excitation on ion diffusion. The electrolyte consisted of a 4% aqueous copper sulfate solution ($CuSO_4 \cdot 5H_2O$)+H_2O (distilled), prepared by dissolving 30 g of copper sulfate in 700 ml of distilled water. Before the intercalation, the solution was heated to 70°C, and the process was initiated at this temperature. During the experiment, a potential difference of 0.3 V was applied, while a constant current of 0.22 mA was maintained between the electrodes, enabling the effective insertion of copper ions into the GaSe crystal lattice. A copper sulfate ($CuSO_4$) aqueous solution was employed as the electrolyte, facilitating the insertion of copper ions into the GaSe crystal lattice.

To investigate the influence of intercalation on the optical properties of GaSe single crystals, the optical spectra of the samples were measured across a wide spectral range of 190–1100 nm, encompassing both ultraviolet (UV) and visible regions. A high-sensitivity Varian Cary 50 UV-Visible spectrophotometer was used for spectrometric analysis, allowing precise measurement of the samples' absorption and transmission characteristics. The results revealed notable changes in the optical spectra due to the incorporation of copper ions into the crystal lattice, particularly the shift of the absorption edge and the modulation of the optical band gap energy. These findings provide critical insights into how intercalation affects the electronic structure of GaSe crystals, offering a valuable foundation for assessing their potential in future optoelectronic applications.

Absorbance spectra in the range of 200–800 nm were calibrated by subtracting the dark signal prior to analysis. The absorption coefficient was calculated as:

$$\alpha(\lambda)=\frac{2.303A(\lambda)}{d}$$

where d is the sample thickness (uncertainty: ±3%). Wavelength calibration uncertainty was ±1 nm, as specified by the manufacturer, corresponding to ±0.005–0.02 eV in the 300–600 nm range.

The optical bandgap (E_g) was determined under the assumption of a direct allowed transition using Tauc analysis, plotting $(\alpha h\nu)^2$ versus hν. The linear fitting region was selected by visual inspection (pure GaSe: 1.95–2.15 eV; Cu-intercalated: 1.75–1.95 eV) and fitted using the least-squares method. Eg values were obtained from the x-intercept of the linear extrapolation.

Total uncertainty was estimated as the quadratic sum of contributions from: (i) absorbance noise and fitting error (95% confidence interval of the linear fit), (ii) wavelength calibration, and (iii) thickness measurement. For each sample, three replicate spectra were recorded; where applicable, the 95% CI from the fit and the standard deviation across replicates were combined.

3. RESULTS AND DISCUSSION

3.1. Structural Characterisation of the Layered GaSe Single Crystal by XRD

To analyse the structural properties of the studied GaSe single crystals, X-ray diffraction (XRD) measurements were carried out in the angular range of 2θ = 0°–120°. The XRD technique plays a crucial role in identifying the crystallographic phase and structural order of materials. In the diffraction pattern, the 2θ angle provides information about the atomic arrangement. At the same time, the intensity (measured in counts per second, cps) reflects the strength of the diffracted signal and indicates how densely atoms are packed along specific planes.

In the recorded diffraction pattern, a dominant peak with an intensity of approximately 300,000 cps appears near $2\theta \approx 20°$, which corresponds to the primary crystallographic phase of GaSe and confirms the high degree of ordering within the material (Fig. 1). The sharpness and narrow width of the peaks across the entire scan range are indicative of a well-ordered single-crystalline structure. We identified seven distinct diffraction peaks, each corresponding to specific crystallographic planes within the GaSe lattice.

The most intense peak observed around 20° is indexed as the (002) reflection plane. This peak is characteristic of layered semiconductors, such as GaSe, and reflects the strong periodicity along the crystallographic c-axis. A weaker peak located between 30° and 40° corresponds to the (004) plane, which typically exhibits lower intensity in layered compounds due to the nature of interlayer interactions. Another significant peak appears around 60°, associated with the (006) plane, further validating the material's layered periodicity. The (008) reflection is detected near 70° and is generally observed in high-quality GaSe single crystals.

In addition, a peak near 80°, indexed as the (101) plane, reveals information about the lateral atomic arrangement within the basal layers. Two further low-intensity reflections observed between 90° and 100°, corresponding to the

(112) and (114) planes, provide evidence for the full three-dimensional crystallographic symmetry of the crystal lattice. These reflections, though less intense, help affirm that the crystal exhibits long-range order in multiple directions.

Figure 2 presents the compositional analysis derived from the XRD pattern of the investigated GaSe crystal. In this plot, the red and green peaks correspond to the experimental reflections obtained from the sample, while the blue markers represent the standard diffraction data for the GaSe phase (ICDD card: 00-037-0931). A comparison of the measured and reference patterns reveals a high degree of correspondence, confirming that the studied material is consistent with the stoichiometric GaSe phase. This agreement indicates that the crystal is single-phased and exhibits high purity.

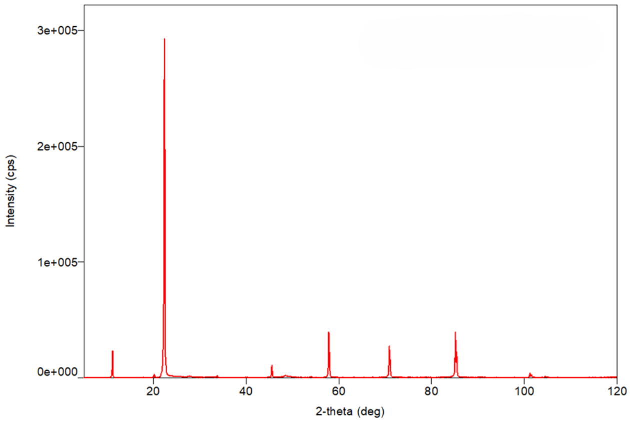


Figure 1. XRD image of a GaSe crystal

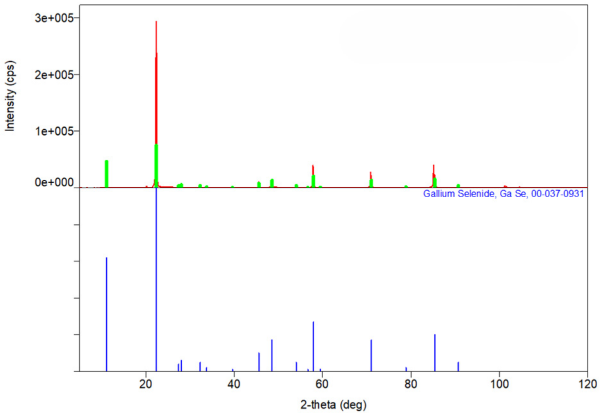


Figure 2. Compositional analysis of GaSe crystal

Gallium selenide (GaSe) is a III–VI group layered semiconductor that crystallizes in an orthorhombic system and typically displays p-type conductivity. The presence of acceptor-like defects, particularly selenium vacancies, is a characteristic feature contributing to its p-type behavior. The confirmation of a monocrystalline nature implies that the entire crystal has grown uniformly along a single crystallographic direction, exhibiting minimal grain boundaries or internal fractures.

The orthorhombic symmetry of the GaSe structure implies that the crystal possesses three mutually perpendicular axes of unequal lengths (ϒ-modification, $a = 3.755\times10^{-4}$ μm and $c = 2.392\times10^{-3}$ μm.). This structural system is distinct from the more symmetric hexagonal configuration and is considered a more specific and directionally anisotropic phase. A major advantage of single-crystalline materials over polycrystalline or nanocrystalline forms is their superior and more predictable physical properties, as the uniform crystal orientation enables more coherent charge transport and reduced scattering at interfaces.

In the case of p-type GaSe, the charge transport is primarily governed by positively charged carriers (holes), resulting from an intrinsic deficit of electrons. This type of conductivity is particularly relevant for tuning the material's electronic response under external stimuli. The combination of a layered structure and intrinsic p-type behavior makes GaSe a promising host material for intercalation-based modifications, enabling targeted tuning of its physical and optoelectronic properties.

3.2. Effect of electrochemical intercalation with copper ions on optical properties

In Figure 3a, the wavelength-dependent absorption (ABS) of the layered GaSe single crystal is presented. The main features of the graph can be described as follows:

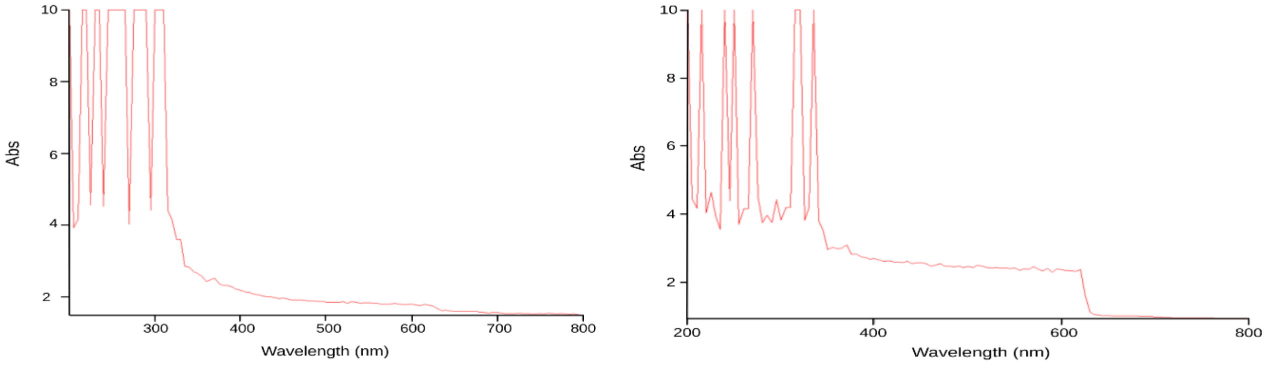
- At short wavelengths (high absorption region) – in the range of 200–350 nm, a strong absorption is observed. This indicates the spectral region where electronic transitions from the valence band to the conduction band occur intensely in the GaSe single crystal. The sharp peaks observed during the measurement are attributed to the sensitivity of the device.
- Sharp decrease in absorption (300–400 nm region) – after 300 nm, a noticeable drop in absorbance is observed. This suggests that the material's band gap lies within this spectral range. In this region, the photon energy is no longer sufficient to induce major electronic transitions in the GaSe crystal, leading to a decrease in absorption.
- Low absorption region (beyond 400 nm) – for wavelengths greater than 400 nm, the absorbance appears to stabilize around 2. This indicates that the material does not significantly absorb light in this region and is mainly transparent.
- Stable absorption up to 800 nm – at longer wavelengths (approaching the infrared spectral region), the absorption remains very low. This is due to the increase in optical transmission of the material and the fact that the photon energy is insufficient for electronic transitions.

Figure 3b shows the wavelength-dependent absorption of the GaSe single crystal after electrochemical intercalation with copper ions.

High absorption region (200–350 nm) – compared to the pure GaSe, intense absorption peaks are also observed at short wavelengths in this spectrum. These peaks are associated with changes in the electronic structure of the crystal due to the incorporation of copper ions between the GaSe layers.

Sharp decrease in absorption (350–600 nm) – after intercalation, the absorption decrease shows a slightly different behaviour. Compared to the previous (non-intercalated) spectrum, it can be seen that absorption persists to some extent even at longer wavelengths. This is explained by the modification of energetic levels and the emergence of new optical transitions as a result of copper ion incorporation into the crystal structure.

New features (600–800 nm) – in this region, a sharp change is observed around 600 nm in the intercalated sample. This is related to the influence of newly formed energy levels, induced by copper ions between the GaSe layers, on the optical properties. The fact that absorption drops to nearly zero beyond 600 nm indicates that the material becomes less sensitive to light in this region.



a) b)

Figure 3. a) Wavelength dependence of absorption before intercalation; b) Wavelength dependence of absorption after intercalation with Cu ions

In Figure 4, the Tauc method is used to analyze the effect of intercalation on the band gap more precisely. By applying the Tauc method, the band gap energy of the GaSe single crystal is accurately determined. For this purpose, the Tauc equation is used:

$$(\alpha h\nu)^n = A(h\nu - E_g)$$

Here:
α - absorption coefficient, hν - photon energy, Eg - band gap width, A - proportionality coefficient, n - a parameter that depends on the type of transition. Since this coefficient is a direct transition for GaSe, n=2 is taken.

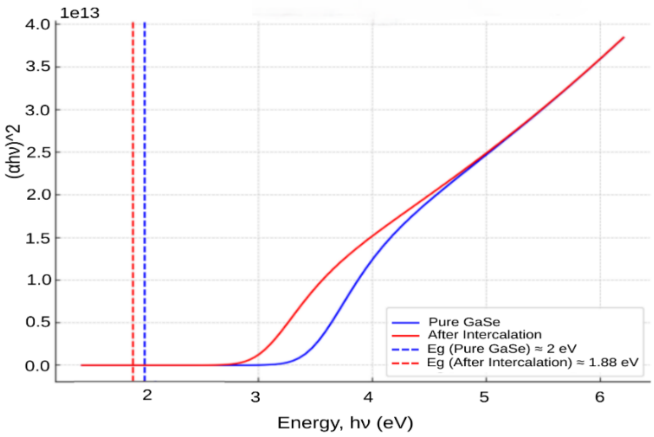


Figure 4. Determination of the prohibited zone according to the Tauc method

The linear fit windows are shown as gray bands; solid lines represent least-squares fits; the shaded area indicates the 95% confidence interval. The band gaps extracted from the intersections are: E_g (pure) = 2.00 ± 0.03 eV, E_g (Cu-intercalated) = 1.86 ± 0.03 eV.

In Figure 4, the narrowing of the band gap in the GaSe crystal is related to structural and electronic changes in the crystal lattice caused by intercalation with copper ions.

Before intercalation, the band gap width of the GaSe crystal (blue line) was observed to be approximately 2 eV. This value corresponds to the expected band gap of pure GaSe. For copper-intercalated GaSe (red line), the band gap is approximately 1.88 eV. As shown, the band gap narrows due to copper intercalation.

The insertion of copper ions between the layers leads to changes in the energetic levels and gives rise to new optical transitions at lower energy states. In the spectrum of pure GaSe, the transmittance (Tm%) sharply decreases at high energies (short wavelengths), indicating the absorption edge and the band gap energy (Eg) of the material. This steep drop is a clear indicator of the band edge position.

During the electrochemical intercalation process, copper ions enter between the layers of the GaSe single crystal and affect the crystal lattice. As a result, lattice deformation occurs, where the incorporation of copper ions alters interatomic distances, thereby influencing the mobility of electrons and the configuration of the energy bands. At the same time, new localised energy states are formed.

When copper ions (Cu^{2+}) are intercalated into the GaSe crystal lattice, the type of energy levels they generate depends on several factors. These factors are as follows:

- The electronic configuration of Cu^{2+} ions – The electron configuration of the Cu^{2+} ion is $3d^9$. The d-orbital electrons of this ion create localized energy states within the GaSe crystal lattice.
- Increase in conductivity and narrowing of the band gap – As seen from the graph, copper intercalation enhances conductivity and narrows the band gap by approximately 0.1 eV. This implies that Cu^{2+} ions are more likely to form acceptor levels rather than donor levels.
- Type of levels formed by copper ions – Acceptor levels (energy levels near the valence band) – When Cu^{2+} ions are intercalated into the layered GaSe structure, they tend to attract electrons, which leads to the formation of new energy levels just above the valence band. In this case, the valence band effectively expands, resulting in a narrower band gap. The energy required for electrons to transition into the conduction band decreases, which accounts for the 0.12 eV narrowing of the band gap. As a result, conductivity increases because electrons in the valence band can more easily reach the conduction band.
- Spectral manifestation of band gap narrowing – In the spectrum of pure GaSe, the absorption edge is located at a higher energy (around 2 eV). However, after copper intercalation, this edge shifts toward lower energies (around 1.88 eV), indicating a narrowing of the band gap, as the material begins to absorb lower-energy photons.

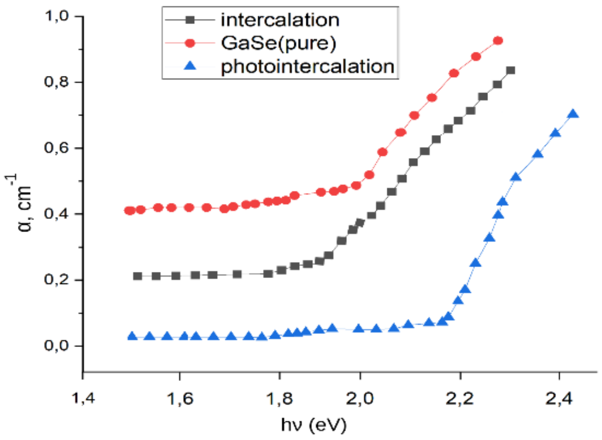


Figure 5. Dependence of the absorption coefficient (α) on photon energy (hν) for pure GaSe (black squares), Cu-ion-intercalated (red circles), and photo-intercalated (blue triangles) GaSe single crystals

Figure 5 presents the dependence of the absorption coefficient (α) on photon energy (hν) for pure GaSe, Cu-ion-intercalated GaSe, and photo-intercalated GaSe single crystals. Layered semiconductor GaSe, in its pure state, possesses high crystalline perfection and a relatively low defect density. High-energy photons (in the UV region) induce strong absorption; however, as the photon energy approaches the band gap, the absorption decreases sharply. The absorption edge is observed at approximately 2.0 eV, which corresponds to the typical band gap of pure GaSe. The incorporation of Cu^{2+} ions into the interlayer spaces creates localized energy states in the crystal lattice. These states enable additional transitions between the valence and conduction bands, allowing electrons to be excited by photons of lower energy (~1.88 eV) (red shift). At the same time, defects and lattice deformation caused by the ions increase light scattering and the probability of carrier recombination, which in turn reduces the overall absorption coefficient. During photo-intercalation, both the positioning of the ions and the formation of photo-induced defects occur under illumination. The photo-induced effect generates additional lattice strain and changes the relative positions of atoms, thereby increasing the distance between the valence and conduction bands (~2.15 eV) (blue shift). In this process, some defects act as "trapping centers" in the conduction band, reducing the absorption of low-energy photons. As a result, the overall absorption coefficient becomes even lower compared to ion intercalation.

Figure 6 shows the dependence of light transmittance on photon energy for all three samples.

As seen in the pure sample, the transmittance remains relatively high (~65%) up to approximately 2 eV, followed by a sharp drop. This drop corresponds to the fundamental band gap of the material, indicating that the GaSe single crystal absorbs light with photon energies above ~2 eV and does not transmit it.

After intercalation, the initial transmittance level starts around ~43%, and a steep decrease is observed near 1.88 eV. This indicates that the intercalation slightly narrows the band gap and introduces changes in the crystal structure or electronic states.

In the case of photo-intercalation, the transmittance is further reduced (~35%), and the onset of absorption appears around ~2.15 eV. This suggests that even lower-energy photons are now being absorbed by the material, resulting in an earlier and more pronounced drop in transmittance.

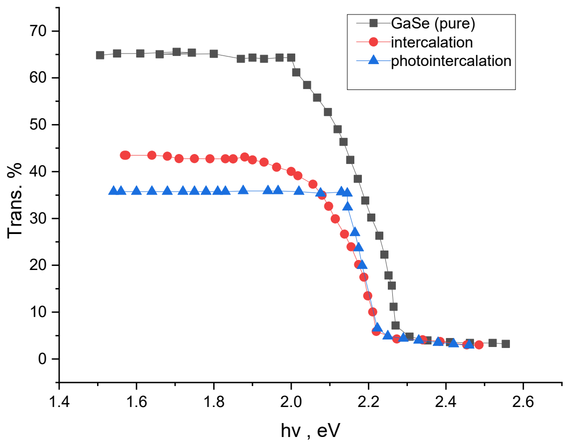


Figure 6. Dependence of light transmission rate on energy

CONCLUSIONS

The experimental investigations revealed that the most intense diffraction peak (002) in the XRD pattern confirms the layered structure and high degree of ordering along the c-axis. Additional major diffraction peaks such as (004), (006), and (008) indicate a well-organised layered phase, while reflections corresponding to planes like (101), (110), and (112) demonstrate the lateral structural arrangement of the crystal. The narrow and sharp nature of these peaks reflects the high crystalline quality of the single crystal (99.99999%).

As a result of Cu ion intercalation into GaSe single crystals, the band gap decreased from 2 eV to 1.88 eV, leading to the formation of new energetic levels. This caused a notable increase in the absorption coefficient within the 200–400 nm range and the appearance of new optical transitions around 600 nm. In the case of photo-intercalation, approximately 35% of the incident light is transmitted through the material, while the remaining 65% is absorbed, with the band gap widening slightly to about 2.15 eV.

Using the applied approach, the band gap for pure GaSe was determined as $E_g=2.00\pm0.03$ eV and for Cu-intercalated GaSe as $E_g=1.86\pm0.03$ eV (95% CI). Thus, intercalation reduces the band gap by $\Delta E_g=-0.14\pm0.04$ eV.

DISCUSSION

This work presents results for a single intercalation level; nevertheless, considering the measurement uncertainty, the observed narrowing of the band gap (Eg) is clear and exceeds the experimental errors. In the future, we plan to prepare a series of samples with varying Cu intercalation levels (confirmed by EDS) to investigate changes in Eg over a broader range.

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ДОСЛІДЖЕННЯ ОПТИЧНИХ ВЛАСТИВОСТЕЙ МОНОКРИСТАЛІВ НАПІВПРОВІДНИКІВ GaSe, ІНТЕРКАЛЬОВАНИХ ІОНАМИ МЕТАЛІВ

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У цьому дослідженні для характеристики структурних та фазових властивостей монокристалів GaSe було використано метод рентгенівської дифракції, а також досліджено вплив інтеркаляції іонів Cu на їх оптичні властивості. Збільшення поглинання (ABS) спостерігалося в області 200–400 нм в результаті додавання іонів Cu, а нові оптичні переходи відбувалися близько 600 нм. Загалом, спостережувані варіації рівнів поглинання спостерігалися в спектральному діапазоні 200–800 нм. Таус аналіз а показав, що ширина забороненої зони звузилася приблизно з 2 еВ до приблизно 1,88 еВ при інтеркаляції та дещо розширилася до приблизно 2,15 еВ при фотоінтеркаляції. Ці результати свідчать про те, що інтеркаляцію іонів Cu можна застосовувати для модифікації оптичних властивостей монокристалів GaSe, збільшуючи їхній потенціал для використання в нелінійних оптичних пристроях, фотоніці та сенсорних технологіях. Результати також демонструють, що інтеркаляція є відповідним методом регулювання фізичних характеристик шаруватих матеріалів.

Ключові слова: *ширина забороненої зони; поглинання; інтеркаляція; рентгенівська дифракція*