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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

ELECTRICAL AND OPTICAL PROPERTIES OF THIN-LAYER AND NANO-SIZED InSe AND GaSe SEMICONDUCTORS

Speciality: 2220.01- Semiconductor physics

Field of science: Physics

Applicant: **Fidan Shirvan Ahmedova**

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The dissertation work was carried out at the Department of "Semiconductor Physics" of Baku State University.

Scientific supervisor:

Doctor of Physical and Mathematical Sciences, professor
Vagif Museyib Salmanov

Doctor of Physical and Mathematical Sciences, professor
Nizami Mammad Gasanli

Official opponents:

Doctor of Physical and Mathematical Sciences, professor
Karim Rahim Allahverdiyev

Doctor of Physical and Mathematical Sciences, professor
Huseyn Behbud Ibrahimov

Doctor of Physical and Mathematical Sciences, professor
Rauf Madat Sardarli

Dissertation council ED 1.14 of Supreme Attestation Commission under the President of the Republic of Azerbaijan operating at Institute of Physics of Azerbaijan National Academy of Sciences

Chairman of the Dissertation council:

Active member of ANAS,
doctor of physical and mathematical sciences, professor
Nazim Timur Mammadov

Scientific secretary of the dissertation council:

Doctor of physical sciences, associate professor
Rafiga Zabil Mehdiyeva

Chairman of the scientific seminar:

Doctor of physical and mathematical sciences,
associate professor
Talat Rzagulu Mehdiyev

General description of work

Relevance of the research topic. The dissertation is devoted to the theoretical and experimental study of the interaction of layered GaSe and InSe thin films and nanoparticles with laser beams. Carrying out research in this area required great responsibility. On the one hand, it was necessary to take into account the long-term research of these crystals at the Institute of Physics and Baku State University, as well as in the scientific centers of the former Soviet Union and a number of foreign countries, and on the other hand. Recent advances in semiconductor physics, especially the study of the interaction of semiconductors with laser beams, have led to the emergence of new fields of physics. It is safe to say that GaSe and InSe crystals have become key elements in quantum electronics and nonlinear optics. In these crystals, the generation of harmonics, the phenomenon of multi-photon absorption, the parametric transformation of light, and so on. such effects have been found. The formation of semiconductor thin films and nanoparticles and the associated new physical phenomena have re-emphasized research in GaSe and InSe semiconductor compounds. The laser, miniature teraherst lasers, and nanoscale tubes in the quantum dot found in these substances are clear examples of this. In view of all this, it was planned to conduct complex research on GaSe and InSe thin films and nanoparticles..

Object and subject of research: Thin-layer and nano-sized InSe and GaSe semiconductors, their non-linear optical, photoelectric and luminescence properties.

Research aims and objectives: The main purpose of the dissertation is to obtain thin layers and nanoparticles of InSe and GaSe crystals by means of discrete instantaneous thermal evaporation and modified chemical precipitation (SILAR) in vacuum, structural and compositional analysis, their electrical, optical, photoelectric and photoelectric properties under the influence of laser beams. by identifying the features and showing the aspects that are of practical importance.

Objectives of the research are to obtain thin layers and

nanoparticles of InSe and GaSe crystals by selecting optimal technological methods and modes, to acquire theoretical knowledge to explain nonlinear optical, photoelectric and luminescence phenomena that can occur in samples at high optical excitation, and to interpret experimental results.

Research methods:

Modern research methods based on perfect theories and tested modern technical equipment were used in the implementation of the dissertation.

InSe and GaSe single crystals were grown by the Bridcman-Stockbarger method. Obtaining thin layers by thermal evaporation method 10-7 mm.c.st by instant evaporation method. carried out in a vacuum chamber capable of delivering pressure. Sequential ion implantation and reaction (SILAR) method was used to obtain nanoparticles and nanoparticle thin films. The obtained InSe and GaSe thin-film and nanoparticles were studied using modern research methods such as X-ray Diffraction (XRD), Scanning Electron Microscope (SEM), Atomic Force Microscope (AFM) and X-ray Dispersed Energy Spectroscopy (EDAX).

Nd^{+3}: YAG laser, Rhodamin dye laser, He-Ne gas laser and halogen light bulb were used as light sources. The Nd^{+3}: YAG laser operating in pulse mode had 3 harmonics (1064, 535 and 355 nm), 12 MWt / cm^2 power and a duration of 10 ns. The active medium Rodamine 6G liquid laser had a wavelength of 594-643 nm. The power of the laser beam was 120 kWt, the pulse duration was 10 ns, and the pulse repetition frequency was 20 Hs. The He-Ne gas laser, operating in continuous mode, had a wavelength of 632.8 nm and a power of 20 watts. Optical absorption and luminescence spectra were measured using a double dispersion M833 monochromator with a resolution of 600 nm at a wavelength of 0.024 nm, a computer-operated detector capable of receiving rays in the wavelength range of 350-2000 nm. Memory oscilloscopes such as Le Croy and Textronic, which can measure nanosecond current pulses, were used to measure photoelectric events.

In addition to traditional methods for measuring nonlinear optical phenomena, two-beam laser spectroscopy and beamdistortion

techniques were used.

The main provisions for the defense:

1. Acquisition of GaSe and InSe thin film and nanoparticles by chemical decomposition and structural analysis of the obtained substances by modern research methods,
2. Experimental study of optical absorption and luminescence spect -ra of GaSe and InSe thin film and nanoparticles,
3. Determination of the anisotropy of GaSe thin films by measuring the photoconductivity at high optical excitation,
4. Experimental study of current pulse relaxation in 2D InSe crystal,
5. Experimental determination of nonlinear optical absorption coefficient and refractive index in GaSe and InSe thin layers under the influence of laser beams,
6. Nd: GaS determination of characteristic features of thermo-luminescence of crystals and preparation of p-GaS/n-InSe heterocurrents,
7. Detection of inversion of the electrical conductivity of thin layers of InSe under the influence of laser beams,
8. Development of optical filters that control the intensity of laser beams based on thin layers of GaSe,
9. Measurement of nanosecond laser pulses in thin layers of InSe.

Scientific novelty of the research:

1. GaSe and InSe thin films and nanoparticles were obtained on glass and GaSe crystal plates by sequential ion lamination and reaction (SILAR) method using a solution of sodium selenosulfate anionic solution, a solution of $GaCl_3$ and $InCl_3$ salts as a cationic solution,
2. Physico-chemical analysis of InSe and GaSe thin film and nanoparticles by XRD, SEM, AQM and EDAX methods.
3. GaSe and InSe thin-film and nanoparticles have been found to occur with free electron transitions of optical absorption and luminescence spectra,
4. It was found that the width of the band gap of InSe nanoparticles increased due to the quantum effect on solid crystals,
5. The anisotropy of GaSe thin films was determined by measuring

the photoconductivity at high optical excitation,

6. Relaxation of photocurrent pulse at high optical excitation in 2D InSe crystal $i = Ae^{-\frac{t}{\tau}} - A'e^{-\delta t}\cos\omega t$ variability is defined by law,
7. Nonlinear optical absorption coefficient and refractive index of high optical excitation in GaSe and InSe thin layers were determined experimentally,
8. Characteristics of thermoluminescence of Nd:GaS crystals were determined and p-GaS / n-InSe heteroconduct was developed,
9. Inversion of electrical conductivity under the influence of laser beams in thin layers of InSe was detected,
10. Optical filters based on thin layers of GaSe have been developed to control the intensity of laser beams,
11. The possibility of measuring nanosecond laser pulses based on InSe thin layers has been discovered.

Theoretical and practical significance of the research:

1. The change in the refractive index under the influence of laser beams in thin layers of GaSe can be explained by the effect of optical filling of zones at high optical excitation.
2. The change in optical absorption and refraction coefficients under the influence of laser beams in thin layers of InSe can be explained by the transfer of the energy of the electron-hole pair generated by high optical excitation to the crystal lattice by radiation of phonons as a result of non-radioactive recombination.
3. The energy of the interlayer potential barrier that causes the anisotropy of GaSe crystals can be explained by the photoconductivity of the charge carriers at high optical excitation $\Delta E_0 = kT \ln\left[\frac{\Delta\sigma_2}{\Delta\sigma_1}\left(\frac{I_1}{I_2}\right)^{\alpha}\right]$,
4. By applying the Maxwell-Kattaneo equation for the dissipative flood to the process of relaxation of the photocurrent pulse in 2D-InSe crystals, we can derive a formula expressing the time dependence of the current, $i = Ae^{-\frac{t}{\tau}} - A'e^{-\delta t}\cos\omega t$,
5. The p-junction can be created by changing the type of electrical

conductivity of InSe thin layers under the influence of laser beams.

6. The narrow (~ 10nm) luminescence line observed at a wavelength of $\lambda = 562$nm in the n-InSe /p-GaS heterogeneity makes it possible to obtain a laser effect based on them.
7. The possibility of developing optical filters based on GaSe and InSe thin layers, which can continuously adjust the value of the intensity of the corresponding laser beams, is shown,
8. It may be possible to develop detectors for measuring nano-second laser pulses using high-speed photocurrents generated in thin layers of InSe.

Approbation and application:

The results of the dissertation were presented at the following conferences: Materials of the dissertation XXI Republican Scientific Conference of Doctoral Students and Young Researchers (Baku, 2017), International Conference "International Conference Modern Trends in Physics" (Baku 2017), "Actual of modern natural sciences" International Scientific Conference (Ganja 2017), the International Scientific Conference of Doctoral Students and Young Researchers dedicated to the 100th anniversary of the Azerbaijan People's Republic (Baku 2018), Ganja State University International Scientific Conference (Ganja 2018), Masters and International Scientific Conference of Young Researchers "Problems of Physics and Astronomy" (Baku 2018), International Scientific Conference on Applied Physics and Energy (Sumgayit 2018), International Scientific and Technical Conference on Micro and Nanotechnology in Electronics (Nalchik, Russia 2021) and Baku State University Department of Semiconductor Physics Reported and discussed at scientific seminars of the Academy of Sciences of the Republic of Azerbaijan.

25 works (17 articles, 8 theses) on the topic of the dissertation were published. Of these works, 8 articles and 1 thesis, 9 of which were published abroad, in journals with an impact factor and 1 conference material, 9 articles and 7 theses were published in local publications and conference proceedings.

The name of the organization where the dissertation work was perfomed:

Department of Semiconductor Physics of Baku State University **The structure and scope of the dissertation:**

The dissertation consists of an introduction, four chapters, results and a bibliography of 147 titles, 178 pages. There are 84 pictures and 1 table in the dissertation. Excluding figures, tables and references, the introduction consists of 12280, chapter I 36287, chapter II 65427, chapter III 31697, chapter IV 42506, results 2145, and abbreviations and symbols 685. The total volume of the dissertation consists of 196939 characters.

Maın content of the dıssertatıon

The **introduction** substantiates the topicality of the dissertation, the goals and objectives of the research, research methods, the main provisions, scientific novelty, theoretical and practical significance of the work, the degree of approbation, publications, as well as a brief explanation of the main content of the dissertation.

In the **first chapter**, the production of thin layers and nanoparticles of InSe and GaSe, structural analysis of the obtained samples are given. It is known that various methods are used to obtain semiconductor thin film and nanoparticles: discrete evaporation method, epitaxial flow of molecules, laser ablation, chemical precipitation method, etc. Although we prepared different samples using some of these methods, in the end we decided to prefer the chemical precipitation method. This is due to the production of nanoparticles of various semiconductors by chemical precipitation, experiments at room temperature, precise regulation of the resorption rate of the material, purposeful selection of the ratios of the components of the material and, most importantly, maintaining the stoichiometric composition of the substance. Taking into account the above, the technology of obtaining thin film and nanoparticles by modified chemical precipitation (Silar) method of GaSe and InSe semiconductor compounds has been developed. Se, In, Ga, Na_2SO_3,

HCl and bidistilled water were used as raw materials for the supply of liquid solutions in the process of obtaining nanoparticles by modified chemical precipitation. Elemental Se, In and Ga substances had a purity of 99.99998%, while Na_2SO_3 and HCl acids had a purity of the "special pure substance" category. The following reaction was achieved by adding Se particles to Na_2SO_3 acid:

$$Na_2SO_3 + Se \xrightarrow{60^0C} Na_2SeSO_3 \qquad (1)$$

The combination of $InCl_2$ and $GaCl_2$, called cation solution, was obtained by the following reaction:

$$Ga + 3HCI \leftrightarrow GaCl_3 + \frac{3}{2}H_2$$
$$In + 3HCI \leftrightarrow InCl_3 + \frac{3}{2}H_2 \qquad (2)$$

GaSe and InSe nanoparticles, the end products of these reactions and using the hydrogen activity of aqueous solutions of InCl2 compounds as a starting product by increasing the pH to 5, were obtained.

Structural morphology and surface morphology of InSe and GaSe thin films and nanoparticles by X-ray diffraction (XRD), Scanning Electron Microscope (SEM), Atomic Force Microscope (AQM) and X-ray Dispersed Energy Spectroscopy (EDAX). The XRD method determined that the GaSe and InSe nanoparticles had a hexagonal structure, and the radiographs showed that the GaSe and InSe nanoparticles were in the range of 4 ÷ 20nm. From the SEM images of the samples obtained, it was found that the thin layers consist of a discrete structure, and the particles that make up the structure are identical in shape and structure. Homogeneous distribution of nanoparticles was observed in the AFM images of GaSe and InSe nanoparticles grown on glass plates. It has been shown that the ratio of indium atoms to selenium atoms in the InSe nanoparticle by the EDAX method corresponds to the stoichiometric ratio of the substances taken.

İn the **second chapter** presents the theoretical and

experimental study of the optical, photoelectric and luminescence properties of InSe, GaSe and GaS thin films and nanoparticles under the influence of laser beams [6, p.90-100].

Figure 1 shows the optical absorption spectrum of a thin layer of GaSe. As can be seen in the figure, it consists of a very narrow peak corresponding to a maximum absorption spectrum λ=620nm.

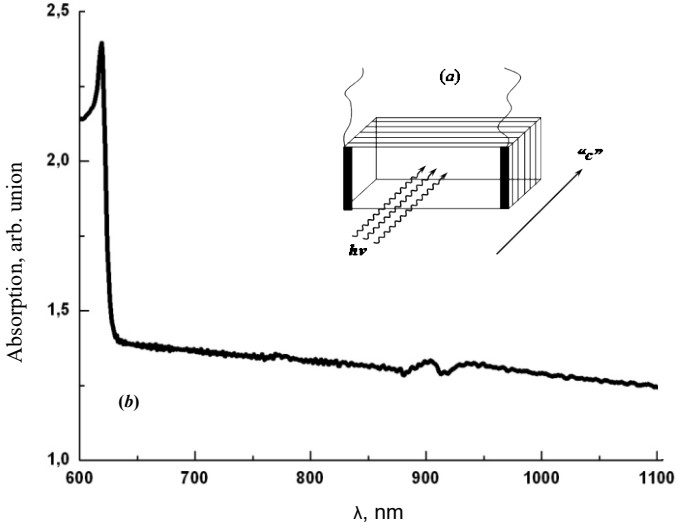


Figure 1. Optical absorption spectrum of a thin layer of GaSe.

There is a sharp decrease in the absorption coefficient on the longitudinal side. From the dependence of α^2~f(hν) the width of the forbidden band of the thin layer of GaSe was determined and it was shown that it is equal to Since the contact energy of the excitons is E_{eks}.- 20meV, the maximum observed in practice λ=620nm can be associated with free exciton transitions. We would like to note that the observation of a free exciton peak in the GaSe thin layers with a semicircle of several angstroms and a sharp decrease in the absorption edge in the longitudinal region indicate that these thin layers are extremely clean.

The optical absorption spectrum of InSe nanoparticles is shown

in Figure 2 [13, p.60-63]. The fundamental absorption edge of InSe crystals corresponds to the near-infrared region of the spectrum. The fundamental absorption edge of InSe crystals corresponds to the near-infrared region of the spectrum. As can be seen from the figure, the absorption spectrum covers the wavelength range of 400-1100 nm. In

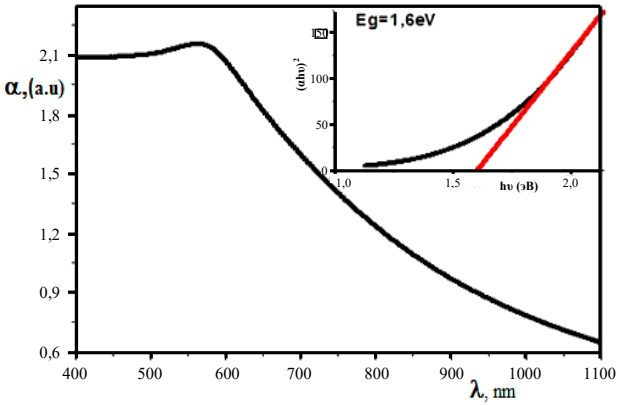


Figure 2. Optical absorption spectrum of InSe nanoparticles obtained on a glass substrate.

the absorption spectrum, the maximum is observed at the $\lambda = 580$ nm value of the wavelength. Since InSe crystals belong to semiconductors with a straight band gap, it has been determined that the width of the band band of nanoparticles is equal to E_g=1.60eV due to the $\alpha^2 \sim f(h\upsilon)$ dependence. As can be seen, the bandwidth of InSe nanoparticles is much higher than that of massive crystals (E_g = 1.34 eV).

It is known that the width of the stranded band of semiconductor nanoparticles varies depending on its thickness, which indicates that they have a quantum effect. The dependence of the width of the band gap of semiconductor nanoparticles on its thickness can be expressed by the following formula:

$$E_g = E_g^{(kristal)} + E_b\left(\frac{\pi a_b}{D}\right)^2 \qquad (3)$$

where E_g -is the width of the blocked band of the nanostructure, $E_g^{(kristal)}$- is the width of the blocked band of the massive crystal, the binding energy of the E_b-excitons, the radius of the boron of the a_b-exciton, is a measure of D-nanoparticles.

Calculations show that if we take into account that InSe crystals $E_g^{(kristal)}$=1,34eV, E_b=25meV, a_b=37 Å, D=12nm, the width of the band gap of nanoparticles will be equal to E_g = 1.55eV. As can be seen, the calculated value of E_g corresponds to the experimental value.

The presence of exciton junctions in the thin layers of GaSe is also reflected in their luminescence spectra. Thus, the observation of a peak with a maximum $\lambda = 615$ nm, narrow (~ 10Å) in the luminescence spectra is associated with free exciton transitions at the fundamental absorption edge. Indeed, the high-concentration electron-hole pair created by the laser beam causes free excitons to form near the fundamental absorption edge as a result of the Coulomb attraction. Calculations show that the concentration of the electron-hole pair formed by the action of laser beams $\Delta n = \alpha \dot{I}_0 \Delta t = 4{,}5x10^{19} cm^{-3}$ is in order (here α =1x10^{3}sm^{-1}- fundamental absorption edge absorption coefficient, $\dot{I}_0$=1,5x10$^{25}\frac{photon}{cm^2 \cdot sec}$ - intensity of laser beams, Δt =3x10^{-9}sec – is the duration of the laser pulse). Similar dependencies are observed in the luminescence spectra of InSe thin layers. However, their luminescence lines are located in the near-infrared region of the optical range (λ = 935nm).

GaSe crystals have a layered structure, they have a strong anisotropy. In most cases, the anisotropy of a crystal is determined by studying its electrical properties. Due to the high resistance of the thin layers under study, it is not possible to determine the nature of the potential interlayer barrier from electrical measurements. The same can be said about optical and luminescence methods.

The layered structure of GaSe crystals does not allow for optical measurements in the directions parallel to the layers, and the radiation lines observed in the luminescence spectra cannot always be interpreted unambiguously. To overcome the difficulties listed

above, it is more convenient to choose the method of studying the photoconductivity in layered crystals under conditions of high optical excitation. By changing the configuration of the contacts on the sample, it is possible to apply an external electric field in the directions parallel and perpendicular to the layers. On the other hand, a high-concentration electron-hole pair generated by high optical excitation allows the height of the potential barrier to be controlled to the point of extreme shrinkage. Thus, the method of measuring photoconductivity under high optical excitation in GaSe crystals with a layered structure is very suitable for the study of anisotropy. The experiments were performed on thin layers of GaSe with a thickness of 30-100 microns. Optical excitation was performed with a liquid laser "Rodamin 6G". Figure 3, a shows the spectrum of the photocurrent in the thin layers of GaSe in accordance with the orientations $\vec{E} \perp \vec{c}$ and $\vec{E} \parallel \vec{c}$ of the electrical contacts [3, p.31-35].

As can be seen from the figure when both contacts are located on the illuminated surface, the surface illumination $\vec{E} \perp \vec{c}$ and (curve 1) has an exciton peak with a high photosensitivity of the electromagnetic waves (1.95÷2.05)eV in the energy range eV. When the direction of the electric field is parallel to the axis ($\vec{E} \parallel \vec{c}$), the spectrum of the photocurrent changes at a reasonable speed (curve 2). The sample is photosensitive at lower energies than the previous spectrum and is observed at the maximum $\hbar\omega = 1.967eV$ peak energy of the spectrum. With the increase in energy starting from this price, a sharp decrease in the photocurrent is observed.

In Figure 3, b, the orientations of the contacts in the directions $\vec{E} \perp \vec{c}$, $\vec{E} \parallel \vec{c}$, the change in the intensity of the laser beam in the range 10^{21}-10^{25} quantum / cm^2 · s, the wavelengths corresponding to the maximum values of photosensitivity ($\lambda_1 = 620nm, \lambda_2 = 630nm$) luxury of photoconductivity -ampere characteristics are given. As can be seen from the figure, in the case of $\vec{E} \perp \vec{c}$ the dependence of the photocurrent ($\Delta\sigma$) on the intensity of the laser beam (I) is linear (curve a), and in the case of $\vec{E} \parallel \vec{c}$ in a certain intensity range the linear dependence is replaced by a stronger dependence $\Delta\sigma \sim I^{2,5}$

(curve b). The intensity of the laser beam is in the range of 10^{23}-10^{25} quantum / cm^2 · s, both characteristics are the same, and it is observed that the photoconductivity is weakly dependent on the intensity.

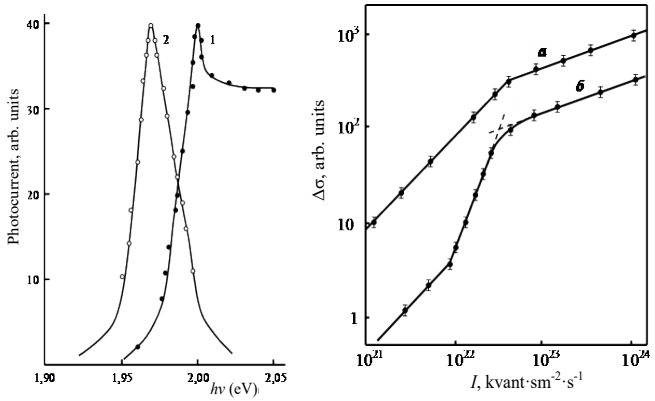


Figure 3, Spectral characteristics of photoconductivity at different orientations of electrical contacts in a-GaSe thin layers 1- $\vec{E} \perp \vec{c}$, 2- $\vec{E} \parallel \vec{c}$; b- dependence of photoconductivity of GaSe crystals on laser beam intensity: α- $\vec{E} \perp \vec{c}$, б- $\vec{E} \parallel \vec{c}$

The properties observed in the spectral and lux-ampere characteristics of the photoconductivity in the thin layers of GaSe are due to the delocolization of unbalanced charge carriers in the direction parallel to the axis from potential holes formed by the fluctuation of the forbidden zone.

The dependence of the potential barrier energy on the photoconductivity of the carriers and the intensity of the laser beam is expressed by the following formula

$$\Delta E_0 = kT \ln\left[\frac{\Delta\sigma_2}{\Delta\sigma_1}\left(\frac{I_1}{I_2}\right)^{\alpha}\right] \qquad (4)$$

where $\Delta\sigma_1$ and $\Delta\sigma_2$ are the values of photoconductivity at intensities I_1 and I_2, indicating the dependence of $\alpha - \Delta n \sim I^{\alpha}$.

Calculations show that it is in $\Delta E_0 = 50meV$ order.

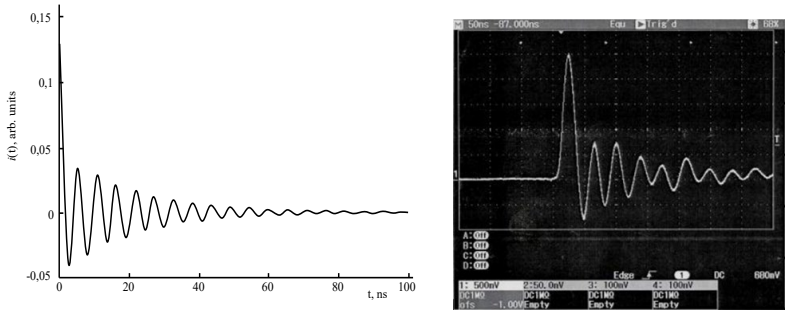
Among the 2D-sized materials, the atomic thin InSe layer is considered the most promising material for nanoelectronics and photonics. The main advantage of 2D InSe crystals over 2D materials such as graphene, Mo_2S and others is that the conductivity of conductive electrons in thin InSe layers is very large ($\sim$7000 $sm^2V \cdot s^{-1}$), the lifetime of unbalanced carriers is very short. ($\sim 10^{-9}s$) happens. The mechanism of current flow in 2D crystals differs sharply from that of crystals. The passage of current through such thin layers in special cases creates a drift capacity within the layer. A prerequisite for the formation of drift capacity is a sharp difference in the loads of the carriers within the layer. The mobility of electrons in the thin layers of InSe is about two orders of magnitude greater than the mobility of holes. Relaxation of the current pulse was found in the thin layers of InSe. When a laser beam strikes a thin layer of InSe whose electrons have a velocity greater than the velocities of the two composite holes, high-concentration charge carriers are formed within the layer.

The rapid extraction of high-velocity electrons and the restriction of electron injection of "slow" holes at the sample inlet lead to the accumulation of positive and negative charges at the sample inlet, thus forming the drift capacity. In the case of stationary current flow, the drift capacity plays a small role [17, p.9-13]. Therefore, the relaxation of the photocurrent can be considered as a relaxation-type dissipative flood and the Maxwell-Kattaneo equation can be applied to it:

$$\mathrm{i} = \mathrm{A}\mathrm{e}^{-\frac{\mathrm{t}}{\tau}} - \mathrm{A}'\mathrm{e}^{-\delta \mathrm{t}} \cos \omega \mathrm{t} \qquad (5)$$

Figure 4a shows the dependence I (t) obtained from expression (5). The kinetics of the photocurrent generated by a laser beam with a duration of 12 ns and $\lambda = 535nm$ a wavelength in the thin layer of InSe (d = 1.3 μm) is shown in Figure 4b. As can be seen, there is a very good correspondence between the theoretically calculated and experimentally obtained i (t) curves. The neutral position of neodymium additives in GaS crystals and nanoparticles and the compensation of shallow donor additives led to the idea that

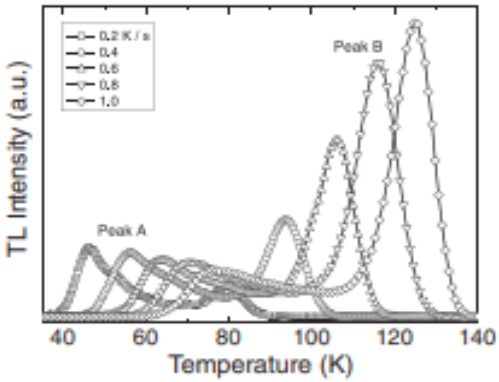
alloying GaS crystals with neodymium atoms and obtaining a blue light source by preparing



a **b**

Figure 4, a- relaxation curve calculated by equation (5), b- kinetics of the photocurrent obtained experimentally in the thin layer of InSe.

an n-InSe-p-GaS <Nd> heteroconductor they are. It is known that the width of the forbidden band of the GaS crystal at room temperature is 2.53 eV, and the width of the straight band is 3.04 eV. Temperature dependences of the intensity of TL at different heating rates are given in Figure 5 [10, p,15].



Şəkil 5. GaS: Nd crystal at different heating temperatures brightness curves of TL

Thus, by creating radiation centers in the broadband GaS crystal, luminescence can be obtained in a wide range from blue to red. As can be seen from the figure, two peaks were observed in the TL brightness curves, peaks A and B. Peaks A and B behave differently depending on the heating rate. As the heating rate increases, the intensity of peak A decreases and peak B increases. The behavior of a peak is an expected outcome and corresponds to a capture center-one recombination model. Peak B is thought to have been caused by specific defects in the GaS crystal.

In the **third chapter** of the dissertation, the nonlinear optical absorption coefficient and refractive index of GaSe and InSe crystals under the influence of laser beams were determined experimentally. The attitude of the environment to optical excitation is characterized by its optical parameters. At high optical excitation, the optical properties of substances are modified when the electromagnetic field voltage is in the order of the intra-atomic field voltage or greater (10^9–10^{12} V / m), ie the reaction of the medium to light depends on the intensity of excitation. At high optical excitation, almost all substances have nonlinear characteristics. In this case, the physical characteristics of the environment (dielectric constant, absorption coefficient, refractive index, etc.) depend on the intensity of light passing through the environment. [21, p.121-131].

A liquid laser with Rodamine 6G was used to study the phenomenon of nonlinear optical absorption in thin layers of GaSe. Beam distortion (Z-scan method) was used to determine the optical constants. This method consists of moving the sample under focus around the focus of the lens and measuring the intensity of the light rays passing through the sample. Figure 6 shows an experimental scheme of the beam distortion method.

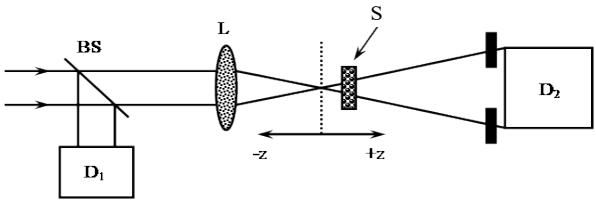
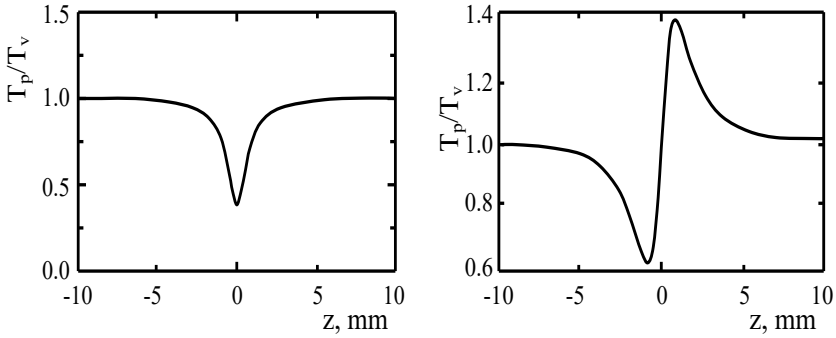


Figure 6. Experimental scheme of Z-scan method.

Sample S is moved along the Z-axis and the rays passing through the sample are recorded by detector D_2. The intensity of the laser beam is controlled by the detector D_1. The change in the intensity of the laser beams passing through the sample is related to the nonlinear refractive index of the sample.

The Z-scan method measured the dependence of the intensity of the laser beam passing through the sample in the "open aperture" mode on its displacement relative to its focal point (Figure 7a).



a **b**

Figure 7. Dependence of the emission factor on the displacement of the sample relative to the focal point in the open (a) and closed (b) aperture modes in the thin layers of GaSe. T_v -is a low-intensity emission factor, and T_p -is a high-intensity emission factor.

As shown in the figure, this dependence is symmetrical with respect to the focus of the lens (z = 0 mm). T_v is the emission factor at low intensity, in the absence of a nonlinear effect, and T_p is the emission factor at high intensity, in the absence of a nonlinear effect. A "closed aperture" mode was used to determine the nonlinear refractive index. As the intensity of light in the detector plane changed as a result of the self-focusing effect, it was possible to measure the nonlinear refractive index through the diaphragm placed in front of the detector. Figure 7, b shows the dependence of the emission factor on the displacement of the sample relative to the

focal point in the "closed aperture" mode. As shown in the figure, when the sample is far from the focal point, the intensity of light passing through the sample decreases. As the sample approaches the focal point, the intensity of the light increases, causing the sample to self-focus. A change in the refractive index causes a change in the phase of the laser beam.

$$\Delta\Phi_0 = (2\pi/\lambda)\Delta n(\omega)d \qquad (6)$$

here d- effective length of the sample, λ- is the wavelength of the laser beam.

The change in the refractive index as a result of the phase change is determined by the quantity ΔTpv = Tp - Tv measured in practice. Knowing the phase dependence of ΔTpv, we can determine ΔTpv ≈ 0.45 (ΔΦ), Δn (ω) by the following formula:

$$\Delta n(\omega) = \frac{\Delta T_{PV}\cdot\lambda\cdot\alpha}{0.45\cdot 2\pi(1-\exp(-\alpha l)} \qquad (7)$$

where λ = 620nm is the wavelength of the laser beam, α ≈ 10^3 cm^{-1} is the optical absorption coefficient at the fundamental absorption edge of the GaSe crystal. Calculations show that the change in the refractive index Δn (ω) ≈ 0.12 is equal to the power of the laser beam at W ~ 10 MWt/cm^2. Of course, as the intensity of the laser beam decreases, the change in the refractive index will also decrease. Thus, the change in the refractive index of the power of the laser beam at W ~ 0.5 MWt / cm^2 is equal to Δn (ω) ≈ 0.069.

It should be noted that another nonlinear optical absorption may occur in semiconductors under the influence of laser beams. It is known that in semiconductors, under the influence of laser beams, it is possible to create a highly concentrated electron-hole pair (~ 10^{18}-10^{19} cm^{-3}). Recombination of these carriers can occur in two main ways, radioactive (irradiated) or non-radioactive (without radiation). Typically, the process of radioactive recombination is associated with optical transitions at the zonal, exciton, and additive levels.

The intensity of radioactive recombination is significantly smaller in semiconductors with a relatively large bandwidth (1-2eV). In the process of non-radioactive recombination, the energy of the electron-hole pair generated by the laser beam is transmitted to the crystal lattice by the radiation of phonons, resulting in an increase in the temperature of the crystal. The increase in the temperature of the crystal lattice may be due to the energy released during the non-radiation recombination of the charge carriers directly into the crystal lattice, through plasmons, or due to Oje recombination. All these processes will cause the fundamental absorption edge of semiconductors to shift towards the longitudinal region. Thus, it is possible to determine experimentally the change in the optical absorption coefficient and the refractive index through the process of nonlinearity of heat generated by the action of laser beams. In the dissertation work, the nonlinear thermal effect in InSe thin layers was discovered and its theoretical and experimental research was carried out.

The experiments were performed using two-beam laser spectroscopy in thin layers of InSe with a size of 20-150 μm (Figure 8). The second harmonic ($\lambda = 532nm$) of Nd:YAG-laser was used as a laser beam. A halogen lamp was used as the second radiation source. The optical emission spectra of the samples were measured using these beams [22, p.148-153; 24, p.63-69].

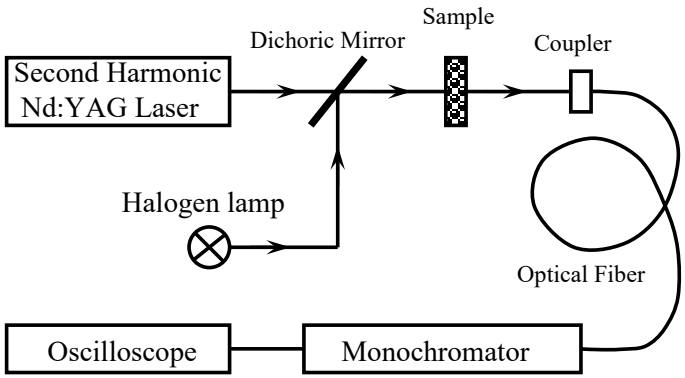


Figure 8. To measure the nonlinear effect of heat on InSe

crystals diagram of the device used.

Figure 9, a shows the energy dependence of the absorption coefficient of the InSe crystal (in the absence of the effect of laser beams and in the presence of the effect of laser beams). As can be seen from the figure, under the influence of laser beams, the optical absorption edge shifts towards a small energy region of the spectrum (curve 2).

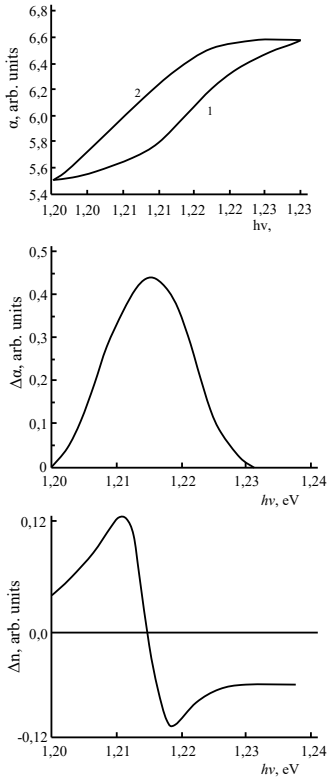


Figure 9. Nonlinear optical phenomenon under the influence of laser beams in InSe crystals. (a) - optical absorption spectra at small (0.5 MWt / cm^2, 1 curve) and high (10 MWt / cm^2, 2 curve) excitation, (b) - change in absorption coefficient ($\Delta\alpha$), (c) - change in refractive index (Δn) [22, p.148-153].

As can be seen, the maximum change in the absorption coefficient occurs in the immediate vicinity of the forbidden lane. According to the Kramers-Kronig expression, the change in the refractive index at a given energy of a photo can be expressed as follows:

$$\Delta \mathrm{n}(\hbar\omega) = \frac{\mathrm{hc}}{\pi}\int_0^{\infty} \frac{\Delta\alpha(\hbar\omega')}{(\hbar\omega')^2-(\hbar\omega)^2}\,\mathrm{d}(\hbar\omega') \qquad (8)$$

Using the Kramers-Kronig relationship, it was determined that the refractive index of InSe crystals changed under the influence of laser beams (Figure 9, c). As can be seen from the figure, the change in the refractive index has a positive value at the small energies and a negative value at the large energies from the fundamental absorption edge.

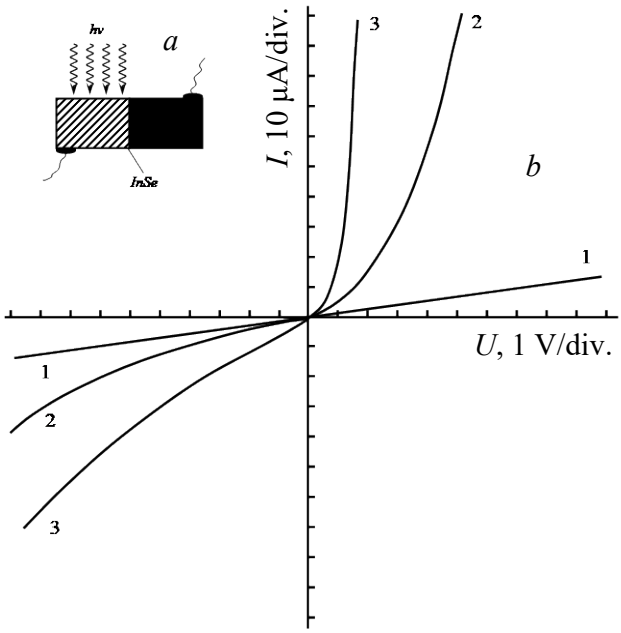
An induced negative change in the refractive index under the influence of a laser beam creates a self-defocusing effect in the case of different distributions of intensity in the area of the beam front. $\Delta n(\omega) > 0$ is called a self-focusing non-linear optical effect, and the environment resembles a collecting lens. This is because the intensity of the laser beam is greater at the center of the beam than at the edges. Thus, when a single-mode laser beam (λ = 535nm) passes through an InSe crystal, the refractive index changes because the intensity is high at the center of the radiation, but the relatively low intensity at the edges keeps the refractive index unchanged.

It is known that the increase in the temperature of the crystal lattice in semiconductors leads to a decrease in the width of the band gap. The reduction of the bandwidth due to the nonlinear thermal effect can be calculated by the following formula:

$$\mathrm{E_g(T)} = 1250\mathrm{meV} - \frac{0.58\mathrm{T}^2}{\mathrm{T}+226\mathrm{K}}\frac{\mathrm{meV}}{\mathrm{K}} \qquad (9)$$

Experience has shown that the shift of the fundamental absorption edge towards long waves under the influence of laser beams is in the order of 10nm. The last expression (9) shows that an increase in temperature of 50-60^{0}C leads to a decrease in E_g - 0.01eV. This figure is consistent with the value obtained in practice.

İn the **fourth chapter** of the dissertation is devoted to the practical significance of scientific research in the thin layers of GaSe and InSe. Experiments have shown that it is possible to achieve an inversion of the electrical conductivity of n-InSe thin layers when exposed to laser beams. The experiments were performed using Nd: YAG pulse laser. Figure 10a shows a schematic of the experiment. When a part of the n-InSe thin layer used is exposed to laser beams, the type of electrical conductivity changes to p-type [20, p.599-603].



Şəkil 10. a- The position of the current contacts in the thin layer of InSe relative to the laser beams, b-volt-ampere characteristics of the laser beams at different power: 1- before the laser beam, 2- after the laser beam W ~ 6.0 MWt / cm^2, 3- after the laser beam W ~ 10 MVt / cm^2.

At the same time, a significant change in the volt-ampere characteristic (VAC) of the sample was observed (Figure 10, b). As can be seen from the figure, the VAC to the laser beam has a

symmetrical characteristic (curve 1). After exposure to laser beams, VAC has the characteristics of a diode (curves 2.3). In this case, the correction factor was ~ $2 \cdot 10^2$ at a value of 2V of the voltage given to the sample (curve 3).

Structural analysis of X-rays of the InSe thin layer by dispersed spectroscopy (EDAX) showed that the composition of the substance did not change after exposure to laser beams. This means that the mass of each component, which forms a thin layer of InSe, allows to maintain the stoichiometric composition of the substance as a whole.

The scanning electron microscope of the InSe thin layer shows that the composition of the thin layer is homogeneous after exposure to laser beams. In our opinion, the change in the type of conductivity of InSe thin layers under the influence of laser beams may be due to changes in the dynamics of defects present in the crystal or the absorption of laser beams in structural heterogeneity. In both cases, the absorption of the lattice causes the temperature of the crystal to rise.

One of the most pressing problems in laser physics is the control of laser beam intensity. Calculations show the duration of modern laser pulses 1fs. (1fs=10^{-15}san), of energy 1Mj (1Mj=10^6j) and the density of the laser energy flux, given that the focal area is ~ 10-4 cm^2 $q = \frac{E}{S\tau} = 10^{25} Wt/cm^2$ we see that it is in order.

It is practically impossible to direct a laser beam of such a high intensity (power) to a single studied sample, because, depending on the power of the laser beam, the substance will melt, evaporate, form plasma, and so on. may occur. Therefore, there is still a great need today for elements that can change the intensity of laser beams over a wide range and at the same time do not distort the shape of the pulse. It is true that some progress has already been made in this area. Various neutral filters, controlled mechanical diaphragms, optical polarizers, and integrated systems are currently used to reduce the intensity of laser beams. However, all of these devices have certain drawbacks. The dissertation presents another method for changing the intensity of laser beams. This method is based on the phenomenon of electrocution observed in the thin layers of GaSe and

InSe. It was found that the fundamental absorption edge of the thin layers of GaSe and InSe, which have a layered structure, is subject to anomalous sliding towards the longitudinal region of the spectrum under the influence of an external electric field.

Experiments show that at a voltage of ~ 20V (10^3 V / cm) applied to thin layers of GaSe, the maximum shear is ~ 16 nm or 50 meV (Figure 11) [12, p.18-25].

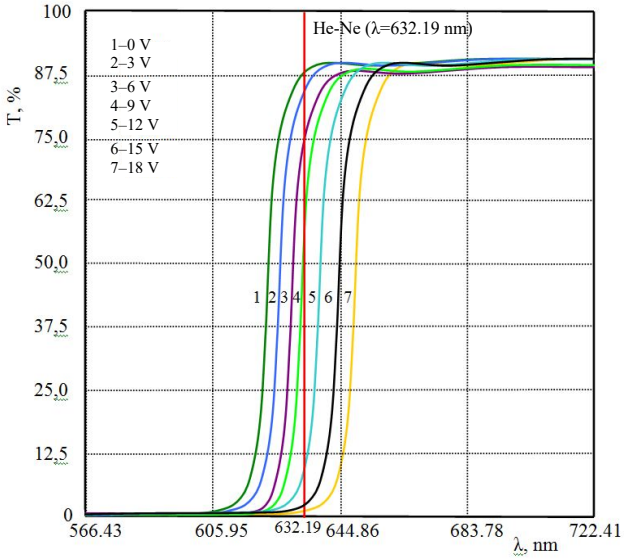


Figure 11. The emission spectrum of a thin layer of GaSe is applied to it dependence on the voltage of the electric field.

This allows you to modulate the intensity of the He-Ne gas laser. Thus, since the maximum radiation of the He-Ne gas laser is λ = 632.8nm, the emission factor will be zero at U = 18V of the external voltage applied to the sample.

Similar experiments were performed on InSe, GaS thin films, GaSxSe1-x solid solutions, and GaSe (B) thin films with boron atoms, and it was determined that the He-Ne gas laser (λ = 632,8nm), Nd: YAG laser (λ = 1060nm) and various wavelengths were based on these semiconductor compounds. The

intensity of a liquid laser (625, 629, 632.8, 634nm) can be changed in two ways.

According to experimental studies and calculations, the shift of GaSe thin films towards the longitudinal region of the optical absorption edge can be explained as follows: the electric field applied to the sample causes its temperature to increase and the absorption edge to slide towards the longitudinal region. Calculations show that ~ 1.9 Wt / cm^2 of energy will be released when an electric field of the order U = 20V is applied to a sample with a specific resistance $\rho \sim 10^3 Om \cdot cm$. In this example, the temperature will be ~ 45-50^0C. Thus, as a result of the sliding of the thin layers of GaSe towards the longitudinal region of the optical absorption edge, it was possible to control the intensity of the pulses of the He-Ne laser, the liquid laser with some wavelengths.

Of course, the shift of the fundamental absorption edge of layered A^3B^6 semiconductor compounds (GaSe, InSe, GaS, $GaS_{0,5}$ $Se_{0,5}$) to an abnormally large distance (~ 160 Å or 50 meV) in the longitudinal region of the spectrum under the influence of an external electric field has aroused great interest. Similar experiments with classical semiconductors Ge, Si, GaAs, and others show that this shift is explained by a very small number (a few millielectron volts) and a Franz-Keldish effect. At present, it is impossible to approach this issue unanimously. It is likely that such an anomalous large slip is due to the fact that these semiconductor compounds have a layered structure, ie they are covalent along the layer and have a weak Van der Vaals interaction between the layers. It is the weak Van der Waals interaction between the layers that causes the width of their forbidden band to slip towards the longitudinal region depending on the temperature. The above is confirmed by our experiments on thin layers of GaSe alloyed with boron atoms. Thus, the sliding in the thin layers of GaSe (B) is very small (1-3 meV). The point is that since B atoms have a smaller tetrahedral covalent radius than Ga atoms, the replacement of Ga atoms with B atoms at the node of the crystal has a fundamental effect on its mechanical and optical properties.

The reason for this is that in the GaSe crystal structure, Ga atoms are replaced by B atoms, resulting in a stronger bond with Se

atoms. The development of receivers capable of measuring nanosecond laser pulses is one of the most pressing problems in laser physics. Currently, several methods are used for this purpose. Vacuum photocells based on the phenomenon of external photoeffect have a very complex structure. At the same time, a high voltage current source is required for the operation of this device. Geodiometers based on Ge and Si are less efficient, and their manufacturing technologies are more complex. On the other hand, photoresistors with a simple structure have great inertia, so it is impossible to measure fast light signals through them.

Our research has shown that nanosecond laser pulses can be measured at room temperature using a receiver (Schottky diode) with a simple structure made of thin layers of InSe. Figure 12 shows an oscillogram of a photocurrent generated by Nd: YAG laser beams in thin layers of InSe. As shown in the figure, the relaxation time of the photocell is ~ 10 nanoseconds [9, p.9-13].

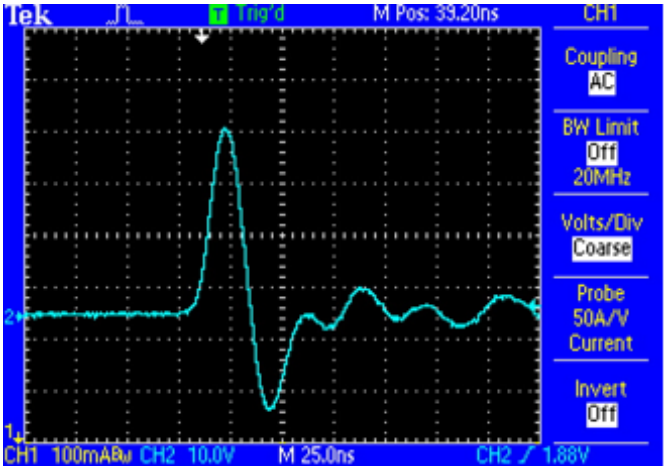


Figure 12. Nd: YAG in thin layers of InSe under the influence of laser beams oscillogram of the resulting photocurrent.

In our opinion, the reason for the rapid photocurrent in the thin layers of InSe is the presence of a fast S recombination center in

their forbidden $\Delta E_i = 0{,}23eV$ band. The direct involvement of the S-center in the formation of ultra-fast photocells in the thin layers of InSe is also reflected in our luminescence event. Thus, when the thin layers of InSe are excited by the 2nd harmonic of the Nd: YAG laser (eV), a luminescence line with a maximum nm and a hemisphere of ~8 Å is observed (Figure 13).

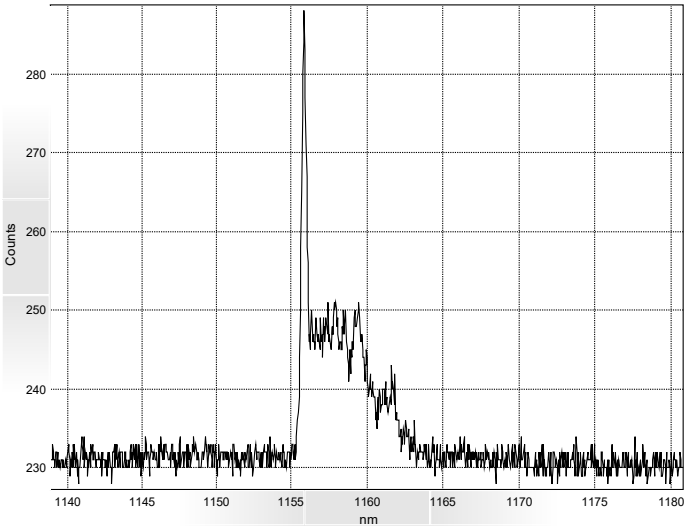


Figure 13. Nd: InSe thin layers under the influence of YAG-laser luminescence spectrum, T = 300K

If we consider that the width of the band gap of the InSe crystal is Eg≈1.30 eV, then we can say that the luminescence radiation ($\hbar\omega = 1{,}073$ eV) observed in the experiment is related to the center S at a distance of $E_S \approx 0.23$eV from the conducting zone. The presence of such an intense and semi-intense ~8 Å radiation spectrum at room temperature in the thin layers of InSe indicates that these crystals can be used as a light source in the near-infrared region of the spectrum.

Result

1. The technology of obtaining GaSe and InSe thin films and nanoparticles by chemical precipitation (Silar) method has been developed and it has been determined that the thickness of the obtained samples is in the range of 4 ÷ 20nm.
2. Structure and morphology of InSe and GaSe thin layers and nanoparticles by X-ray diffraction (XRD), Scanning Electron Microscope (SEM), Atomic Force Microscope (AFM) and X-ray Dispersed Energy Spectroscopy (EDAX) has been shown to have a homogeneous distribution and stoichiometric composition.
3. A quantum-dimensional effect was found on InSe nanoparticles, with an increase in the bandwidth of a 10nm-thick nanoparticle from 1.34eV to 1.60 eV relative to massive crystals.
4. By measuring the anisotropy of transfer events in GaSe thin layers with a layered structure under conditions of high optical excitation, it was determined that the potential barrier energy perpendicular to the layers is equal to ~ 50meV.
5. It was found that the relaxation of the photocurrent pulse in high-level optical excitation in InSe-type semiconductor 2D-crystals $i = Ae^{-\frac{t}{\tau}} - A'e^{-\delta t}\cos\omega t$ occurs by law.
6. It has been shown that the nonlinear optical absorption event detected in the GaSe and InSe thin layers at high optical excitation is due to changes in their optical absorption and refractive indices. It was found that the change in the refractive index lazn (ω) ≈ 0.12 was equal to the power of the laser beam in the thin layers of GaSe at W ~ 10 MW / cm^2.
7. It was found that the inversion of the type of electrical conductivity observed in the thin layers of InSe is due to changes in the dynamics of defects in the crystal as a result of an increase in the temperature of the crystal lattice under the influence of laser beams.
8. It has been shown that the anomalous displacement of the emission spectra of the thin layers of GaSe, InSe, GaS, GaS0,5Se0,5 and GaSe (B) under the influence of an external electric field towards the longitudinal region of the spectrum

determines the intensity of different laser beams (He-Ne, GaAs-diode, liquid laser). allows you to change approximately two layouts.

9. It has been shown that the presence of a rapid recombination center inside the thin layers of InSe allows the measurement of nanosecond laser pulses through a receiver based on them.

List of scientific works for which the main scientific results of the dissertation are published

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