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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

OBTAİNİNG METAL (Ga,In,Cd) CHALCOGENIDES (Se, S, Te) NANOPARTICLES BY LASER ABLATION METHOD AND THEİR OPTICAL PROPERTIES

Specialty: 2220.01- Semiconductor Physics

Field of Science: Physics

Applicant: **Turana Akif Mammadova**

Baku - 2025

The work was performed at Department of Semiconductors Physics of Baku State University.

Scientific supervisors: Doctor of Physics and Mathematics Sciences, Professor
Vagif Museyib Salmanov

Doctor of Physics and Mathematics Sciences, Professor
Maarif Ali Jafarov

Official opponents: Doctor of Physics and Mathematics Sciences, Associate Professor
Vugar Amir Aliyev

Doctor of Physics and Mathematics Sciences, Professor
Rauf Madat Sardarli

Doctor of Physics Sciences, Associate Professor
Mehriban Shirin Hasanova

Dissertation Council of Supreme Attestation Commission under the President of the Republic of Azerbaijan operating at the Institute of Physics of the Ministry of Science and Education of the Azerbaijan Republic under number ED 1.14.

Chairman of the Dissertation Council: Academician
Arif Mammad Hashimov

Scientific Secretary of the Dissertation Council: Doctor of Physics Sciences, Professor
Rafiga Zabil Mehdiyeva

Chairman of the scientific Seminar: Doctor of Physics and Mathematics Sciences Professor
Huseyn Behbud Ibragimov

GENERAL DESCRİPTİON OF THE WORK

Relevance and degree of development of the topic. Recently, nanoparticles have become one of the important research objects of semiconductor physics, both as a field of fundamental science and due to their practical importance. The fact that semiconductor nanoparticles have unique physical properties is due to the emergence of quantum-scale effects in them. The dependence of the optical and electronic properties of semiconductor nanoparticles on their geometric dimensions creates broad opportunities for the creation of new generation light-emitting diodes, transistors, solar cells and quantum lasers. Despite the intensive study of semiconductor nanostructures, the preparation and study of nanostructures based on currently little-studied and at the same time promising semiconductor compounds of the $A^{III}B^{VI}$ and $A^{II}B^{VI}$ types is of great interest. [1,2,3]. One of the newest methods for obtaining semiconductor nanoparticles is the ablation method of solid bodies located in a vacuum, gas or liquid medium using pulsed lasers. The laser ablation method is a fairly simple, fast and direct method for the synthesis of nanoparticles. This method does not require chemical reactions, as well as high temperatures and pressures, or long multi-stage processes characteristic of chemical synthesis. The properties of the synthesized nanoparticles depend on the shape, size, composition and structure of the material from which they are taken, the parameters of the laser used in ablation (radiation wavelength, pulse duration, frequency and energy), as well as environmental conditions (vacuum, gas or liquid).

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[1] В.М. Салманов, А.Г. Гусейнов, Р.М. Мамедов, А.А. Салманова. Нелинейные оптическое поглощение в GaSe при лазерном возбуждении. Оптика и спектроскопия 2020, т. 128, в.4, 513-516.

[2] D.A.Bandurin, A.V.Tyurnina at al. High electron mobility, quantum Hall effect and anomalous optical response in atomically thin InSe. Nature Nanotech., 2016, 3, 223-227.

[3] N.G.Semaltianos, S.Logothetidis, W. Perrie at al. Appllied physics letters 2009, 95, 033301-033306

Another important problem in the dissertation work is the method of determining the main parameters of a multilayer structure consisting of various semiconductors under the influence of laser beams. In the preparation of optoelectronic devices based on semiconductor structures with a variable band gap, it is very important to know the composition of these structures, the width of the band gap, the thickness of the layer, the concentration of deep and shallow impurities, the nature of the recombination processes and the properties that determine the efficiency of the resulting structures.

Aims and objectives of research:

Synthesis of nanoparticles of semiconductor compounds InSe, GaSe, GaS, $GaSe_{1-x}S_x$, CdTe, CdS and $Cd_{1-x}Zn_xS$ by laser ablation method, structural analysis, detection and study of optical and photoluminescence phenomena in them, including nonlinear optical effects, and determination of the mechanism of two-photon optical absorption in multilayer semiconductor structures.

In accordance with the goal set in the dissertation work, the following issues were solved::

- Synthesis of nanoparticles of semiconductor compounds InSe, GaSe, GaS, $GaSe_{1-x}S_x$, CdTe, CdS and $Cd_{1-x}Zn_xS$ by laser ablation;
- Study of the surface morphology and internal structure of synthesized nanoparticles by X-ray diffraction analysis (XRD), scanning electron microscope (SEM), atomic force microscope (AFM) and X-ray energy dispersive spectroscopy (EDAX) methods
- Determination of the optical absorption mechanism and plasma temperature of laser beams in the semiconductors studied during the laser ablation process;
- Determination of the optical absorption and luminescence parameters (absorption coefficient, band gap, exciton binding energy, etc.) of InSe, GaSe, CdS and CdTe nanoparticles;

- Detection of the forced radiation effect in InSe nanoparticles;
- Detection of nonlinear optical absorption processes in InSe, GaS and CdS nanoparticles at high optical excitation intensity;
- Determination of the parameters of multilayer semiconductor structures (gap width, layer thickness, carrier concentration, nature of recombination processes) by the two-photon excitation method.

Research objects. Nanoparticles of semiconductor compounds InSe, GaSe, GaS, CdTe, CdS, $GaSe_{1-x}S_x$ and $Cd_{1-x}Zn_xS$ solid solutions and thin layers of GaS/GaSe and GaSe/InSe heterostructures were taken as research objects. This choice of research object was determined, first of all, by the features of the crystal structure of these materials, their layered structure and strong anisotropy, transparency in a wide frequency range and the availability of developed technology for obtaining perfect crystals. On the other hand, the choice of the research object was significantly influenced by the compatibility of the bandgap width of the semiconductors used with the energy of the laser beams**.**

Research methods. The issues raised in the dissertation were carried out on the basis of experimental and theoretical studies. The light sources used were Helium-Neon (He-Ne) and Nitrogen (N_2) gas lasers, a nanosecond Nd:YAG laser with three harmonics (1060 nm, 532 nm and 335 nm), a Rhodamine 6G liquid laser with a beam source in a wide wavelength range (594-642 nm), and a collimated light lamp. During the research, a technology for obtaining nanoparticles of the studied semiconductors by laser ablation was developed, the surface morphology and structural analysis of nanoparticles were carried out using modern structural analysis methods such as X-ray diffraction analysis, scanning electron microscopy, atomic force microscopy, and X-ray dispersive energy spectroscopy, and the optical, luminescence, nonlinear optical, photoconductivity, and other physical properties of the studied nanoparticles under high optical excitation were detected and studied using modern research methods of laser spectroscopy such

as pump-probe spectroscopy, beam distortion, and other methods of nonlinear optics, and a computer system with special software for analytical analysis of the results (board Master 800 ABI 8).

The main provisions of the defense:

1. Synthesis and structural analysis of nanoparticles of semiconductor compounds InSe, GaSe, GaS, CdS and CdTe by laser ablation;

2. Detection of nonlinear optical phenomena in InSe, GaS and CdS nanoparticles at high optical excitation intensity;

3. Determination of nonlinear refractive index in InSe nanoparticles under the influence of laser beams;

4. Determination of parameters of multilayer semiconductor structures by two-photon optical absorption method;

5. Determination of plasma temperature arising in the laser ablation process;

6. Detection of quantum-scale effect in GaSe nanoparticles;

7. Detection of forced radiation effect in InSe, GaS and CdS nanoparticles;;

Scientific novelty of the research:

1. Nanoparticles of semiconductor compounds InSe, GaSe, GaS, CdS and CdTe were synthesized by laser ablation;
2. InSe, GaSe and CdS nanoparticles were synthesized by laser ablation using In, Ga and Cd elements and SeO_2, $(NH_2)_2CS$ solutions;
3. The surface morphology and internal structure of the synthesized nanoparticles were studied using X-ray diffraction analysis, Scanning electron microscope, Atomic force microscope and X-ray energy dispersive spectroscopy methods;
4. The optical absorption mechanism of laser beams and plasma temperature in the semiconductors studied during the laser ablation process were determined;
5. The main parameters of InSe, GaSe, CdS and CdTe nanoparticles (absorption coefficient, band gap width, exciton binding energy) were determined by the action of laser beams;
6. The forced radiation effect was discovered in InSe, GaS and CdS nanoparticles;

7. The nonlinear optical absorption process was discovered in InSe, GaS and CdS nanoparticles at high excitation intensity;
8. The main parameters (band gap, layer thickness, impurity concentration, nature of recombination processes) of multilayer semiconductor structures were determined by the two-photon excitation method;

Theoretical and practical significance of the research:

- Obtaining nanoparticles of semiconductor compounds InSe, GaSe, GaS, $GaSe_{1-x}S_x$, CdTe, CdS and $Cd_{1-x}Zn_xS$ by laser ablation and studying their physical properties may allow these nanoparticles to be used as basic materials and elements of optoelectronics;

- The phenomenon of two- and three-photon photoluminescence discovered in $GaSe_{1-x}S_x$ solid solutions can be used as a basic method for studying multilayer semiconductor epitaxial heterostructures;

- The effect of forced radiation discovered in InSe nanoparticles can allow the creation of laser radiation generators based on them;

- The "transparency phenomenon" discovered in the exciton absorption region in InSe nanoparticles can allow the creation of optical modulators of laser radiation based on them;

- The change in refractive index of InSe nanoparticles at high optical excitation intensity may allow them to be used as a frequency converter of radiation in nonlinear optics.

Publications. The topic of the dissertation is reflected in 22 publications. The number of published articles on the dissertation is 13. Of these, 6 were published in impact factor journals in the Web of Science database ("Оптика и спектроскопия", "Physics,Chemistry, Mathematics", "Physics of Solid State", "Semiconductors", "Russian Journal of Physical Chemistry A"), and 7 in local journals. He made 9 presentations at international (3) foreign and (6) republican conferences.

Approbation. The materials of the dissertation were presented and discussed at the International Scientific Conference "International conference Modern Trends in Physics" (Baku 2023), the International Scientific Conference "Current problems of modern natural and economic sciences" dedicated to the 100^{th}

anniversary of the birth of the national leader Heydar Aliyev (Ganja 2023), the International Scientific Conference "Current problems of modern natural and economic sciences" dedicated to the 101st anniversary of the birth of the national leader Heydar Aliyev (Ganja 2024), the Republican Scientific and Practical Conference "The role of universities in the development of innovative ecosystems" (Ganja 2024), the XIV International Scientific and Technical Conference "Micro-and nanotechnology in electronics" (Russia, Nalchik 2024) and the 8th International Scientific Conference "Scientific Results" (Rome, Italy 2024).

The structure and volume of the dissertation. The presented dissertation work consists of 13035 characters in the introduction, 56730 characters in chapter I, 11357 characters in chapter II, 31666 characters in chapter III, 64678 characters in chapter IV, and 3151 characters in the conclusions, for a total of 181494 characters. The dissertation work consists of 148 pages, including 102 pages of text only (181494 characters), 20 images, 51graphs, and 6 tables.

Name of the organization where the dissertation research was performed:

The dissertation was performed at the Department of Semiconductor Physics of Baku State University.

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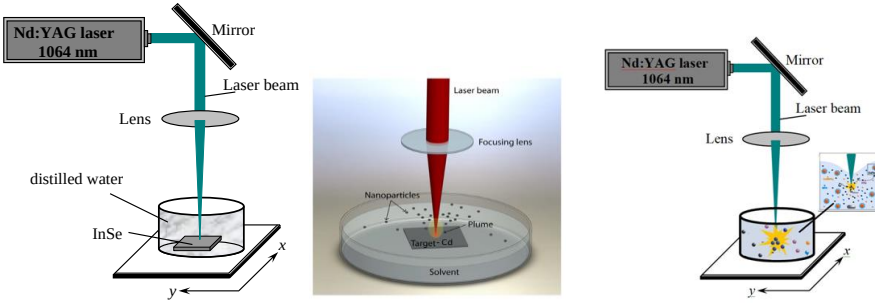
CONTENT OF THE DİSSERTATİON

In the introduction, the relevance and degree of development of the topic of the dissertation work, the main goal of the dissertation work, scientific novelty, scientific and practical significance, the set goal and the provisions put forward for the defense are justified.

The first of chapter is devoted to the synthesis of semiconductor nanoparticles InSe, GaSe, GaS, CdS və CdTe by the method of laser ablation in a liquid. An Nd:YAG laser was used as a light source. The laser pulse duration was 10 ns, the pulse repetition frequency was 10 Hz, and the maximum power was ~12

MW/cm^2. The intensity of the laser beams was controlled using neutral light filters. Laser radiation with a wavelength of 1064 nm, focused by a collecting lens (f =11 cm), is focused on the surface of the studied substance with a diameter of ~1.5 cm. Laser ablation was performed in a quartz cuvette, in distilled water, without the addition of additional active substances. The ablation process was carried out at normal pressure with laser beams with an energy of 135 mJ, with a frequency of 10 Hz and an ablation time of 10 minutes (Fig. 1).



a **b** **c**

Figure 1. Synthesis of semiconductor $A^{III}B^{VI}$ və $A^{II}B^{VI}$ semiconductor compounds from liquid by laser ablation method: a- obtaining nanoparticles from InSe, GaSe, GaS crystals by laser ablation method, b - obtaining nanoparticles using high purity (99%) In, Ga and Cd elements and SeO_2 and $(NH_2)_2CS$ solutions mixed with distilled water for the synthesis of InSe, GaSe and CdS nanoparticles, c- obtaining CdTe nanoparticles in solution by reactive laser ablation method.

In addition to the synthesis of semiconductor nanoparticles by the well-known laser ablation method, a new method for the synthesis of nanoparticles of the semiconductors under study by using their constituent atoms directly has been proposed for the first time.

For the synthesis of InSe, GaSe and CdS nanoparticles, high purity (99%) In, Ga and Cd elements and solutions of SeO_2 and $(NH_2)_2CS$ mixed with distilled water were used.

It was found that the color of suspensions of colloidal solutions depends on the intensity of laser irradiation. The light orange color of GaSe nanoparticles synthesized at low laser intensity turned into dark orange at high laser intensity. The nanoparticles were obtained according to the following reactions:

$Ga+SeO_2+H_2O=GaSe+H_2O+O_2$ (1)
$İn+SeO_2+H_2O=İnSe+H_2O+O_2$ (2)
$Cd+(NH_2)_2+CS+H_2O=CdS+(NH_2)_2CO+H_2$ (3)
$2CdCl_2+ 2TeO_2 + 2H_2O = 2CdTe + 4HCL + 3O_2$ (4)
$2CdCl_2+2TeO_2+Na_2S_2O_3+H_2=2CdSTe+2NaCL+2HCL+4O_2$ (5)

Figure 2 shows colloidal solutions of GaSe, GaS, InSe, CdS, CdSTe, and CdTe nanoparticles obtained by laser ablation.

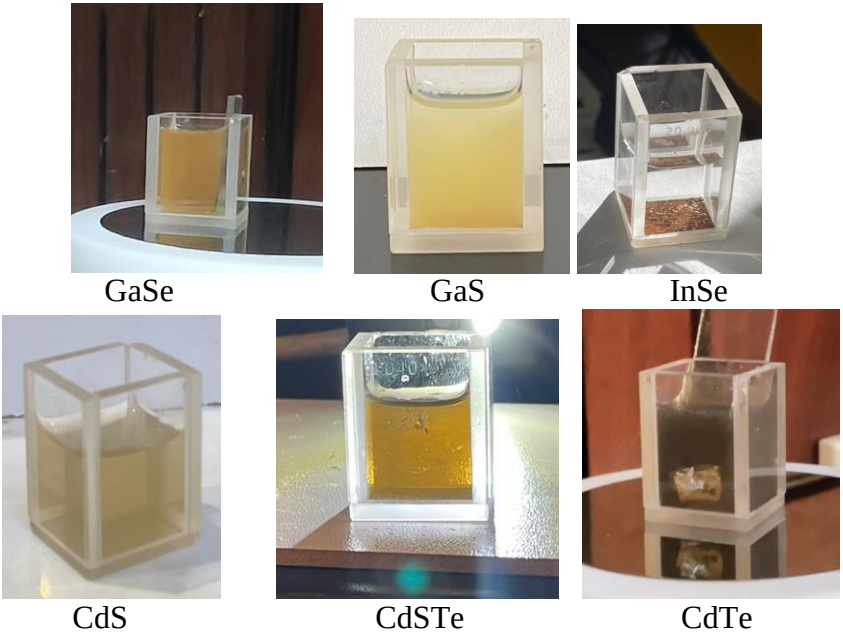


Figure 2. Colloidal solutions of nanoparticles obtained by laser ablation.

In the second chapter, information on the experimental study of the surface morphology and structural analysis of InSe, GaSe, GaS, CdS and CdTe nanoparticles synthesized by the laser ablation method in liquid is presented. For this purpose, modern research methods such as X-ray diffraction analysis (XRD), Scanning electron microscope (SEM), Atomic force microscope (AFM) and Energy dispersive X-ray spectroscopy (EDAX) were used. The sizes of the nanoparticles obtained on the basis of X-ray diffraction analysis were calculated using the Debye-Scherrer formula[4]:

$$D = \frac{k\lambda}{\beta \cos\theta} \qquad (6)$$

where D is the size of the nanoparticles, k = 0.9 is the form factor, β = 0,035 A^0 - is the full width at half maximum (FWHM-FullWidth at HalfMaximum), λ – is the wavelength of the X-ray radiation (λ=1,54 A^0), θ – is the Bragg angle ($\cos\theta$=0,727).

Calculations show that the sizes of the studied nanoparticles vary in the range of ~(7-50) nm.

Figure 3,a shows the SEM image of InSe nanoparticles deposited on a glass substrate. The SEM image shows that the obtained material consists of spherical nanocrystals with a size of (7-30) nm, collected in a polydisperse form. The homogeneous distribution of particles is not observed in the presented AFM image (Figure 3,b). EDAX analysis of InSe nanoparticles shows that the amount of indium atoms in the material is in the ratio In:Se=1:1, which indicates that the composition has stoichiometry. (Figure 3, c).

[4]S.S. Mao, Nanolasers: lasing from nanoscale quantum wires. Int.J.Nanotech. 2004, 1, 42-85.

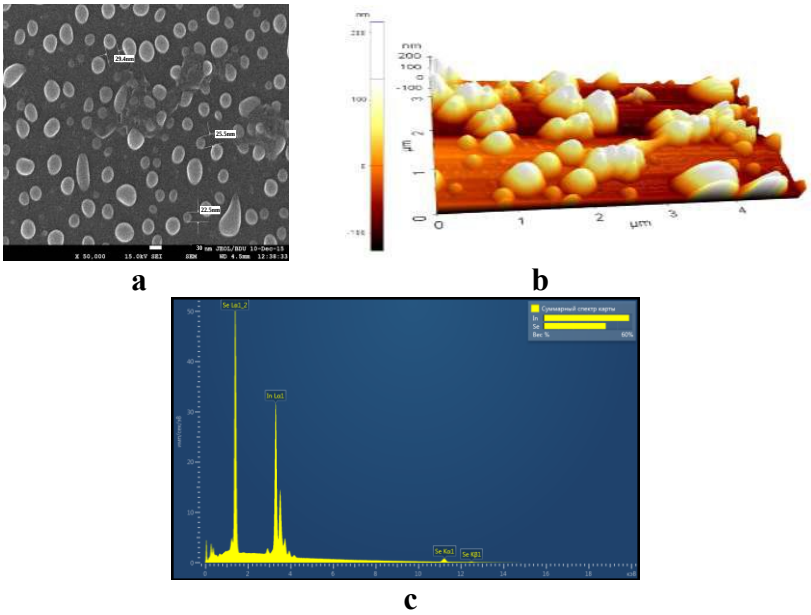


Figure 3. SEM (a), AFM (b) and EDAX(c) images of InSe nanoparticles.

Figure 4 shows the XRD analysis of CdTe nanoparticles deposited from a nanocolloidal solution onto a clean glass substrate.. CuK_{α}, λ=1,544178A^0 SSFOM: F17-610.0.5.10.60 was used as the radiation source. The XRD results show that the 2θ diffraction angles of the nanoparticles are 23.620, 27.890, 38.970, 45.280, 49.340, 56.750, 63.600, 73.220, 76.840, corresponding to the Miller indices (111), (200), (220), (311), (222), (400), (331), (422) and (511), which are consistent with the cubic (zinc blend) structure of the CdTe crystal..

The sizes of the nanoparticles obtained based on the XRD results were calculated using the Debye-Scherrer formula. The calculations show that the average size of the CdTe crystallites is 27.434 nm..

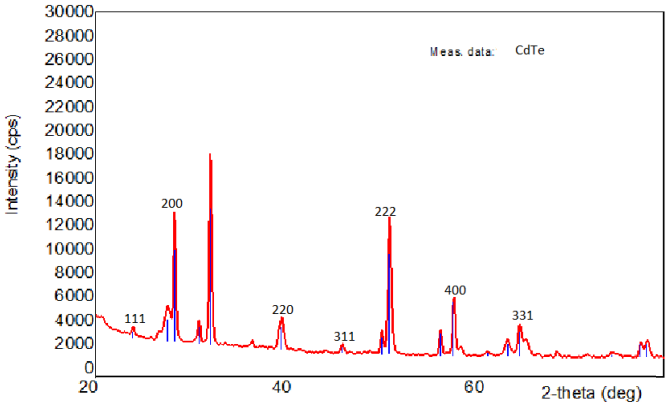


Figure 4. X-ray diffraction curves of CdTe nanoparticles dried on a glass substrate.

Figure 5,a shows the SEM image of CdS nanoparticles synthesized by laser ablation. It is shown that the size and morphology of the nanoparticles depend on the intensity of the laser beam. Figure 5,b shows the three-dimensional AFM image of CdS nanoparticles. As can be seen from the figure, a homogeneous distribution of nanoparticles is not observed. The size distribution histogram of nanoparticles is shown in Figure 5,c. As can be seen from the figure, the average size of nanoparticles calculated by the software is 60 nm. Analysis of the structure of CdS nanoparticles by energy dispersive X-ray spectroscopy (EDAX) shows that the ratio of cadmium and sulfur is Cd:S =1:1, which indicates that the composition of the substance is stoichiometric.

The heat transfer equation was used to determine the temperature of the plasma cloud formed on the surface of the sample during laser ablation

$$\frac{\partial Q}{\partial t} + div J_Q = 0, \qquad (7)$$

where Q- is the amount of heat in a certain volume, J_Q- is the density vector of the heat flux.

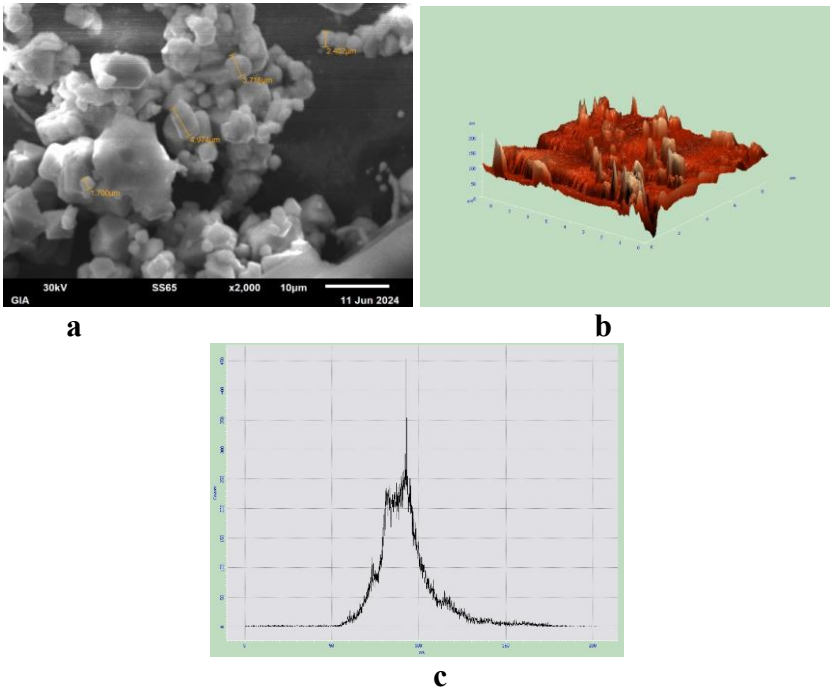


Figure 5. SEM(a), AFM(b) and histogram(c) images of CdS nanoparticles.

Expression (7) is a continuity equation and shows the change in the amount of heat released in the volume.

$Q = \rho c T$ və $J_Q = -\chi \frac{\partial T}{\partial x^2}$, since equation (7) can be written as follows

$$\rho c \frac{\partial T}{\partial t} - \chi \frac{\partial^2 T}{\partial x^2} = 0 \qquad (8)$$

where T- is the temperature, ρ- is the density of the material, c-is the specific heat capacity, and χ- is the thermal conductivity.

In the case of an external source, the limit $(rkI_0 \hbar\omega)$ must be added to expression (8). where r- is the reflection coefficient, k is the absorption coefficient of light, I_0-is the intensity of the incident light and $\hbar\omega$-is the energy of the incident quantum..

Then equation (8) will be as follows

$$\rho c \frac{\partial T}{\partial t} = \chi \frac{\partial^2 T}{\partial x^2} + rkI_0 \hbar \omega e^{-kx} \qquad (9)$$

The last expression shows the relationship between the parameters of the laser pulse, the main characteristics of the removed material, and the temperature released during ablation. From the solution of equation (9), the temperature of the heat released during the ablation process can be calculated

$$T = \frac{2(1-r)E}{\sqrt{\pi c \rho \chi t_0}} \frac{1}{S} \qquad (10)$$

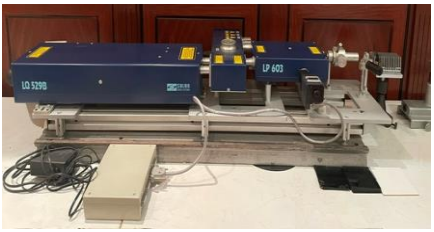
where E is the energy of the pulse, S is the area covered by the light.

Calculations show that the temperature on the surface of InSe nanoparticles increases to ~10^5K. The following parameters of InSe were used:

c = 1.3 C/K•cm^3, χ = 8.2 W/m·K, r=0.3, ρ=2.3 g/cm^3, t_0=12·10^{-8} sec, S=5.10^{-3} cm^2 . In addition, as can be seen from expression (10), the energy E required to vaporize a substance by a laser pulse is proportional to the square root of the pulse duration, E~$\sqrt{t_0}$.

The third chapter presents the optical absorption and photoluminescence properties of InSe, GaSe, GaS, CdTe, CdS nanoparticles and $Cd_{1-x}Zn_xS$ solid solution obtained by laser ablation. Figure 6 shows a description of the devices used to measure the optical and photoelectric properties of semiconductor nanoparticles. An Nd:YAG laser was used as a light source. The wavelengths of the laser pulses with three harmonics were 1064, 532 and 335 nm, the power was ~ 12 MW/cm2, and the pulse duration was 10 ns. The intensity of the laser beams was adjusted with calibrated neutral light filters. Optical absorption and luminescence spectra of nanoparticles were performed using an automatic M833 monochromator with a resolution of ~0.024nm at 600nm, a double dispersion system, and a detector in the 350-2000nm wavelength range. A Le Croy 900 oscilloscope with a

memory capable of measuring nano-picosecond current pulses was used for photoconductivity measurements.



a)

b)

c)

Figure 6. Illustration of the devices used to measure the optical (b) and photoelectric (c) properties of the Nd:YAG laser (a) and nanoparticles.

Figure 7,a shows the absorption curve of GaSe nanoparticles. Starting from the wavelength value λ=550nm a sharp increase in the absorption coefficient is observed. Since GaSe is a semiconductor with a flat optical transition, $\alpha^2 \sim f(h\nu)$ the bandgap width of the nanoparticles was calculated from the dependence Eg = 2.62eV (Figure 7,b). This figure is 0.62 eV larger than the bandgap width of bulk GaSe crystals,Eg(crystal)=2.02eV).

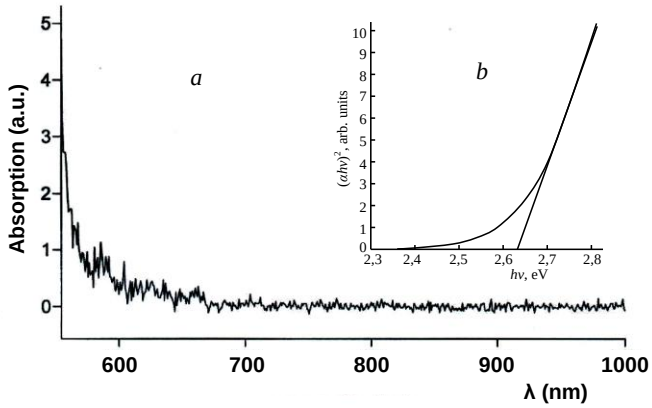
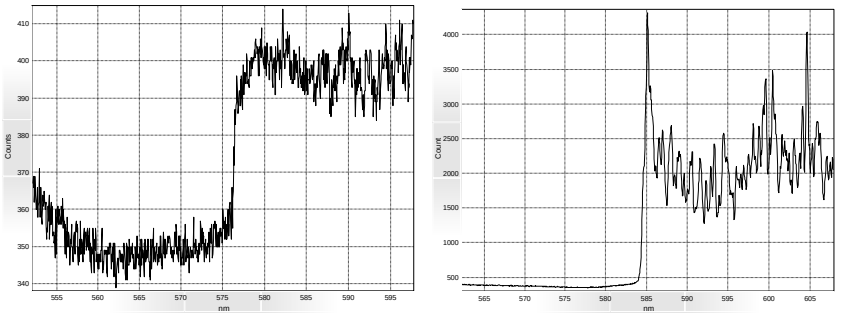


Figure 7. Optical absorption spectrum (a) and dependence $\alpha^2 \sim f(h\nu)$ (b) of GaSe nanoparticles obtained in colloidal solution.

Figure 8,a shows the photoluminescence spectrum of GaSe nanoparticles excited by the second harmonic ($\hbar\omega = 2.34$eV) of the Nd:YAG laser. As can be seen from the figure, the maximum emission of the nanoparticles corresponds to a wavelength of λ=473 nm (2,62eV). Figure 8,b shows the photoluminescence spectrum of GaSe nanoparticles obtained as a result of the interaction of the laser radiation with the Ga element and the SeO_2 solution. The semi-width of the emission line is ~$7A^0$. The fact that the observed luminescence line is arranged in several angstroms indicates that stimulated emission is detected in GaSe nanostructures upon laser excitation. As can be seen from the comparison of Figures 8,a and 8,b, the luminescence spectrum of nanoparticles obtained directly from GaSe crystals during the laser ablation process differs significantly from the luminescence spectrum of nanoparticles obtained from a mixture of Ga element and SeO_2 solution.

Gallium sulfide (GaS) crystals occupy a special place among semiconductors with a layered structure due to their unique physical and chemical properties. This group differs from other layered crystals (for example, GaSe and InSe) in that they have a large band

gap: at T=300 K, the oblique transition is Eg ~ 2.5 eV; the forward transition is Eg ~ 3.0 eV. Thus, GaS crystals fill the gap between semiconductors and insulators, which can be used for the implementation of color-tunable blue/UV light-emitting diodes and ultraviolet (UV)-selective photodetectors.



a **b**

Figure 8. Photoluminescence spectra of nanoparticles synthesized by laser ablation from a mixture of Ga element and SeO_2 solution (a) and directly from GaSe crystals (b).

The width of the forbidden band for oblique transitions $\alpha^{1/2} \sim f(h\nu)$ (Fig. 9,b, curve 1) and for straight transitions (Fig. 9,b, curve 2) was determined from the dependence of $\alpha^2 \sim f(h\nu)$. As can be seen from the figure, the width of the forbidden band for oblique and straight transitions of GaS nanoparticles was equal to Eg ≈ 3.0 eV and Eg ≈ 4.0 eV, respectively. It is known that these values are larger than those of crystalline GaS (0.5-1.0 eV). Comparison of the luminescence spectra of GaS nanoparticles obtained with the second harmonic of the Nd:YAG laser ($\hbar\omega = 2{,}34\,\text{eV}$) with their optical absorption spectra suggests that the short-wavelength luminescence line observed in the luminescence spectra is associated with straight optical transitions, and the longer-wavelength line is associated with oblique optical transitions.

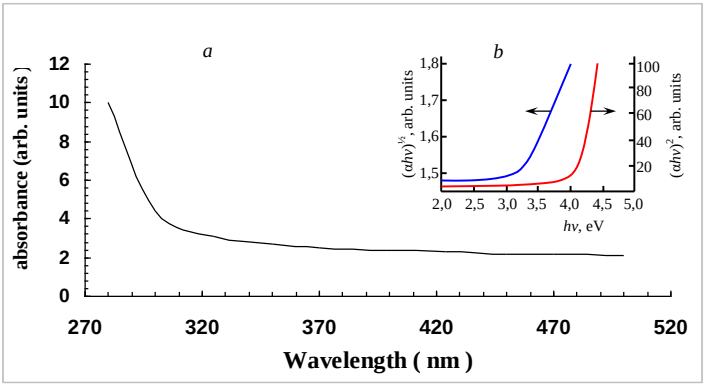
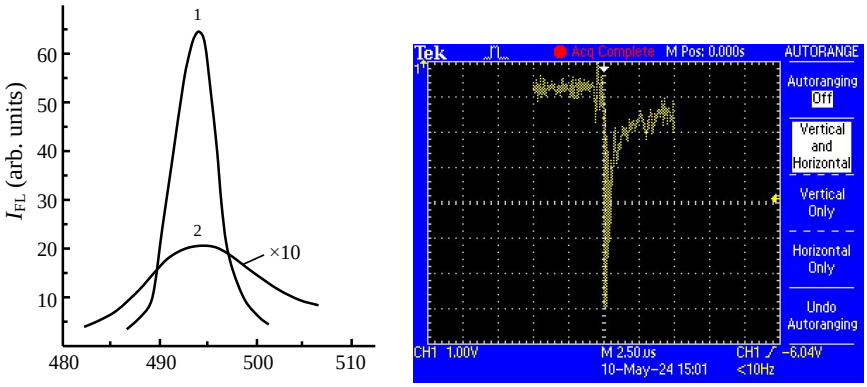


Figure 9. a- Absorption spectrum of GaS nanoparticles, b- $\alpha^{\frac{1}{2}} \sim f(h\nu)$ $\alpha^2 \sim f(h\nu)$ dependencies.

The photoluminescence spectra of CdS thin films were measured with a Rhodamine 6G liquid (dye) laser. The pulse power was 120 kW, the duration was 3 ns, and the repetition rate was 20 Hz. The photoluminescence spectra of CdS nanoparticles are shown in Figure 10,a. As can be seen from the figure, increasing the laser beam power from 8 MW/cm^2 to 12 MW/cm^2 leads to a significant increase in the photoluminescence intensity, ~30 times..

The dependence of the photoluminescence intensity ($I_{люм}$) of CdS nanoparticles at the maximum wavelength ($\lambda = 499$ nm) on the intensity of laser radiation ($I_{лаз.}$) is expressed by the formula $I_{люм.} \sim I_{лаз.}^{3.5}$. Such a nonlinear dependence allows us to say that laser irradiation occurs in CdS thin films. Figure 10,b shows the kinetics of photoluminescence in CdS thin films. As can be seen from the figure, the relaxation time is of the order of $\tau \approx 5 \cdot 10^{-7}$sec. The presence of a fast relaxation time can be explained by the presence of a laser effect in CdS thin films.

In the dissertation work, the photoconductivity and luminescence spectra of $Cd_{1-x}Zn_xS$ solid solutions were also experimentally studied. Figure 11,a shows the photoconductivity spectra of $Cd_{1-x}Zn_xS$ solid solutions under the influence of laser irradiation at T=300 K..

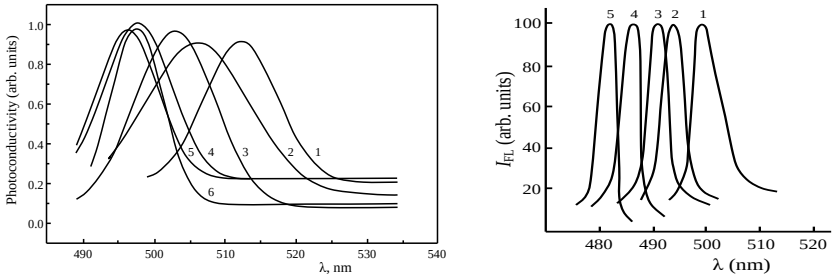


a **b**

Figure 10. a-Photoluminescence spectra of CdS thin films at different intensities of laser irradiation, $I_{лаз.}$(MW/cm^2): 1-8; 2-12; b-Relaxation curve of photoluminescence in CdS nanoparticles.

As can be seen from the figure, the photosensitivity spectra of $Cd_{1-x}Zn_xS$ thin films (x=0÷0.30) cover the wavelength range (490-520)nm. Figure 11,b shows the photoluminescence spectra of $Cd_{1-x}Zn_xS$ solid solutions at a temperature of T=80K. As can be seen from the figure, with an increase in the amount of Zn in $Cd_{1-x}Zn_xS$ solid solutions, the photoluminescence spectra also shift to shorter wavelengths. The features observed in the photoconductivity spectra of $Cd_{1-x}Zn_xS$, depending on the resistance of the studied samples, indicate that the photoconductivity is not due to the effect of additives, but is due to zone-by-zone optical absorption.

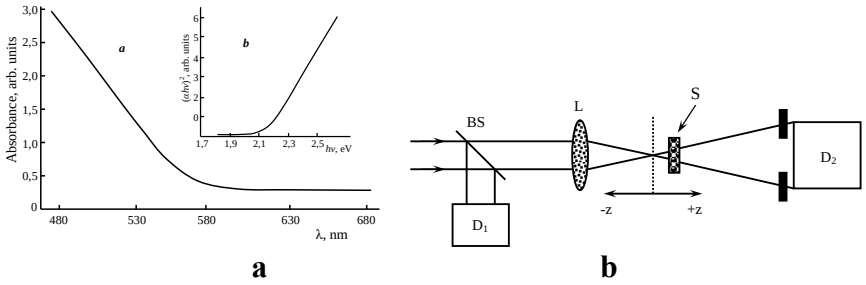
The fourth chapter of the dissertation is devoted to the theoretical and experimental study of nonlinear optical phenomena in thin films and nanoparticles of semiconductor compounds InSe, GaSe, GaS, CdS and CdTe, CdS. The absorption spectrum of InSe nanoparticles is given in Figure 12,a. Since InSe crystals have a flat band gap, it was determined by the formula $\alpha^2 \sim f(h\nu)$ that the band gap of nanoparticles is equal to Eg=2.15 eV. The obtained results show that the band gap of InSe nanoparticles is ~ 0.9 eV larger than the band gap of bulk InSe crystals (Eg=1.25 eV).



a **b**

Figure 11. a- Photoconductivity spectra of $Cd_{1-x}Zn_xS$ (x=0÷0.30) solid solutions: 1 – 0; 2 – 0.05; 3 – 0.10; 4 – 0.15; 5 – 0.25; 6 – 0.30. T=300 K, b-Photoluminescence spectra of $Cd_{1-x}Zn_xS$ solid solutions under the influence of laser beams at different values of x, x: 1– 0; 2 – 0.05; 3 – 0.15; 4 – 0.25; 5 – 0.30. T=80 K.

The beam distortion (z-scan) method was used to study the nonlinear optical properties of InSe nanoparticles (Figure 12,b). Using the open aperture technique, the intensity transmitted through the sample (the transmittance of the sample) was measured as a function of the position of the sample (Figure 13,a).

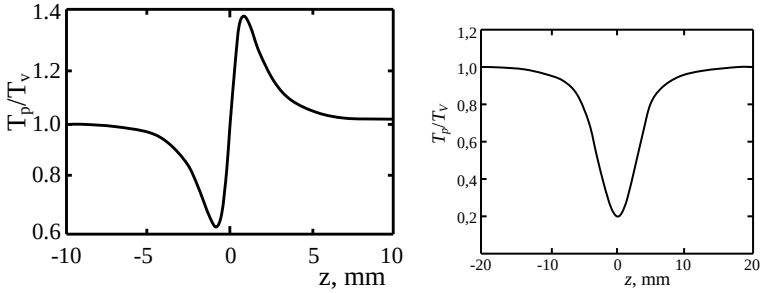


a **b**

Figure 12. a- Absorption spectrum of InSe nanoparticles, b- Schematic diagram of the Z-scan device.

As can be seen from the figure, this dependence is symmetric with respect to the focus of the lens (z=0 mm) when the minimum transmittance is observed. T_v is the transmittance of the sample at

low intensity of radiation (in the absence of nonlinear effects), T_R is the transmittance of the sample at high intensity of radiation. The closed diaphragm technique was used to determine the nonlinear refractive index. Since the size of the light spot in the detector plane changes due to the self-focusing effect, placing a finite diaphragm in front of the detector provides measurement of the nonlinear refractive index. The characteristic shape of the transmittance functio is shownin Figure13,b.



a **b**

Figure 13. a- Transmittance as a function of the position of the sample relative to the focus of the lens in the case of an open diaphragm, b- Transmittance as a function of the position of the sample in the case of a closed diaphragm.

If a sample with a positive nonlinear refractive index (Δn) is located far from the focus of the lens, the intensity of the radiation passing through the sample is low, and since the sample is not very thick, the transmittance changes slightly as the sample moves. As the sample approaches the focus, the intensity of the beam is sufficient for self-focusing to occur in the sample. The value of Δn(ω) can be determined by analyzing the transmittance as a function of the position of the sample relative to the focus of the lens.

The change in refractive index causes a change in phase, $\Delta\Phi_0 = (2\pi/\lambda)\Delta n(\omega)L_{eff}$, where $L_{eff} = (1-\exp(-\alpha l)/\alpha$ - the effective length of the sample, and λ is the laser wavelength. The change in phase and hence the change in refractive index can be

determined from the measured quantity ΔT_{pv}, which is the difference between the maximum and minimum, $\Delta T_{pv} = T_p - T_v$, where $T_p(T_v)$ is the peak (valley) transmittance. Since $\Delta T_{pv} \approx 0.45(\Delta\Phi)$, the nonlinear refractive index $\Delta n(\omega)$ is given by:

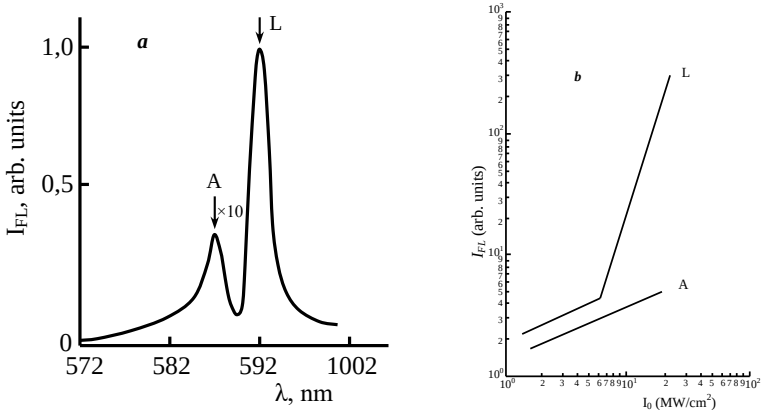
$$\Delta n(\omega) = \frac{\Delta T_{PV} \cdot \lambda \cdot \alpha}{0.405 \cdot 2\pi(1 - \exp(-\alpha l)} \qquad (11)$$

where $\lambda = 535$ nm is the wavelength of the laser, $\alpha \approx 10^3$ cm^{-1} is the optical absorption coefficient of the studied substance, $l = 20$ nm is the thickness of the sample.

Calculations show that when InSe nanoparticles are excited by laser beams with a power of W~10 MW/cm^2, the change in the refractive index as a result of the occurrence of a nonlinear optical phenomenon is $\Delta n(\omega) \approx 0.24$.

A nonlinear optical absorption phenomenon was detected in the exciton absorption region of InSe nanoparticles. The luminescence spectrum of InSe nanoparticles excited by the second harmonic ($\hbar\omega = 2{,}34\,\text{eV}$) of the Nd:YAG laser is given in Fig. 14,a. As can be seen from the figure, two emission lines with maxima at 587 nm (A line) and 592 nm (L line) are observed in the spectrum. As the intensity of the exciting rays increases, the L line becomes dominant in the emission spectrum, at an excitation power of 10 MW/cm^2 the intensity of the L line is 2 orders of magnitude higher than the intensity of the A line. It should be noted that the position of the L line changes depending on the excitation intensity, a shift towards long wavelengths is observed, at a laser beam power of ~8 MW/cm^2 the shift of the L line is ~15 meV. Figure 14,b shows the dependences of the luminescence intensity of the A line and the L line on the laser radiation power. As can be seen from the figure, the intensity of the A line depends linearly on the laser beam power, while in the L line the linear dependence observed at low excitation intensities is replaced by a quadratic dependence at high excitation intensities. In our opinion, such a superlinear dependence indicates the observation of the stimulated emission phenomenon in InSe nanoparticles. Comparison of the luminescence spectrum of nanoparticles with the absorption spectrum of InSe suggests that the

A line observed in the luminescence spectrum is due to the annihilation of free excitons. This is evidenced by the stability of the luminescence line, its narrow width (~10 A^0), and the fact that this line is located at a distance equal to the exciton binding energy (Eb=20meV) from the edge of the fundamental absorption band in the long-wavelength region of the spectrum..



a **b**

Figure 14. a- Luminescence spectrum of InSe nanoparticles excited by the second harmonic of the a-Nd:YAG laser ($\hbar\omega = 2{,}34\,\text{eV}$), dependences of the intensity of the б- A and L luminescence lines on the laser irradiation power.

The study of the nature of the L band, where stimulated emission occurs, is of great importance. In our opinion, this band can be associated with the exciton-exciton interaction that occurs in semiconductors at high levels of optical excitation. Indeed, as the excitation intensity increases, the concentration of excitons increases, and when it reaches a certain critical value, interaction occurs between excitons, which leads to the dissociation of excitons and the formation of free electron-hole pairs. This phenomenon is called the Mott criterion. Knowing the effective masses of electrons and holes (m_e=0.12m_0, m_h = 0.6 m_0), as well as the Bohr radius for

InSe a_{ex} = 37A^0, we can determine the concentration of excitons (n_{Mott}):

$$n_{Momm} = \frac{\pi}{3}\left(\frac{1{,}46}{4a_{экс.}}\frac{m_0}{m_e + m_h}\right)^3 \qquad (12)$$

The critical exciton concentration for InSe calculated using formula (12) was equal to n_{Mott} ~$2{,}5\times10^{16}$ cm^{-3}. Calculations show that the non-equilibrium carrier concentration created by a laser with intensity $I_0=1{,}5x10^{25}$ photon/cm^2·sec and pulse duration $\Delta t=3\times10^{-9}$ sec is equal to the following value:

$$\Delta n = \alpha I_0 \Delta t = 4{,}5\times10^{19}\ cm^{-3} \qquad (13)$$

where $\alpha \approx 10^3\ cm^{-3}$ –is the absorption coefficient at the fundamental absorption edge.

As can be seen from the comparison of formulas (12) and (13), the concentration of non-equilibrium carriers obtained is 3 orders of magnitude higher than the concentration required for the Mott transition. The exciton-exciton interaction can be expressed by the following formula:

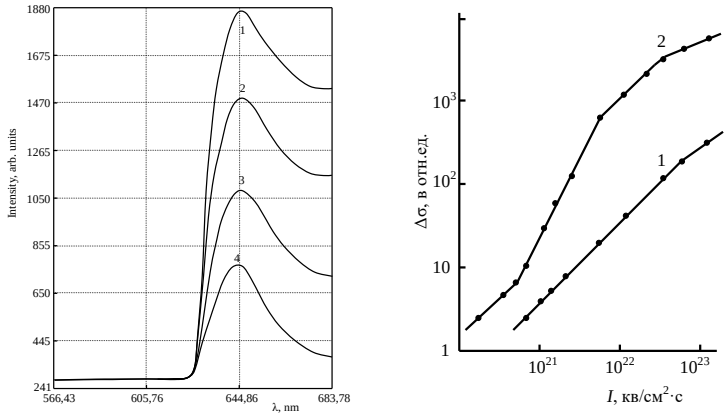
$$h\nu = E_g - 2E_b - E_k E_{k'} - \frac{\hbar k^2}{2\mu} = E_g - 2E_{cв} - \Delta E \qquad (14)$$

where E_g – is the width of the forbidden band, $\mu = \left(\frac{1}{m_e} + \frac{1}{m_h}\right)^{-1}$ – is the effective mass of the electron-hole pair, $\frac{\hbar^2 k^2}{2M}$ – is the total energy of the electron-hole pair, $M = m_e + m_h$, $E_{св}$ – is the binding energy of the exciton, $\hbar k_e$ (*baş.son.*) – is the momentum of the electron before (after) the collision, $E_{kk'}$ – is the kinetic energy of the exciton before the collision, ΔE is determined by the following expression:

$$\Delta E = \frac{1}{\mu}\frac{h^2}{8}\left(\frac{3}{8\pi}\right)^{2/3}\left(\frac{N}{V}\right)^{2/3} \qquad (15)$$

Experiments show that the dependence of ΔE on the excitation intensity is of the form ΔE ~ $I_0^{1/3}$.(Calculations using formula (16) show that with m_e=0.12m_0, m_h=0.6m_0 and $I_{лазер}$≈10MW/cm^2 , ΔE is equal to ~15meV, which is in agreement with the experimentally found values for ΔE.

t is known that structural defects present in GaSe crystals with a layered structure have a strong effect on their electrical, optical and photoelectric properties. It is of great interest to determine the influence of structural defects on the physical properties of GaSe nanoparticles. For this purpose, in the dissertation work, the optical and photoelectric properties of GaSe thin films and nanoparticles under the influence of laser beams were experimentally studied. Thin films with a thickness of (100-200) μm were obtained from the bulk crystal by mechanical cleavage. Figure 15,a shows the emission spectra of GaSe thin films. As can be seen from the figure, the optical absorption edge corresponds to the energy value of ~2.02 eV.



a **b**

Figure 15. Emission spectrum (a) and lux-ampere characteristic of photoconductivity of GaSe thin films.

The increase in the intensity of the laser beams leads to a decrease in the emission spectrum. Thus, a change in the intensity in the interval of 0.1-10 MW/cm^2 leads to a 35% decrease in the emission coefficient. Figure 15,b shows the dependence of the photoconductivity ($\Delta\sigma$) on the laser radiation intensity (I) in the directions perpendicular to the layers (curve 1) and parallel (curve 2). As can be seen from the figure, in the case of, E⊥c curve 1), the photoconductivity in a wide intensity range linearly depends on the intensity I of the laser beams, while in the case of E//c (curve 2) a quadratic dependence is observed in a certain intensity range $I_1 \le I \le I_2$ ($I_1 = 6.5 \cdot 10^{22}, I_2 = 6.5 \cdot 10^{23}$ kV/cm^2•sec) $\Delta\sigma \sim I^2$. The features observed in the emission spectrum and lux-ampere characteristics of the photoconductivity of GaSe thin films are related to the potential barrier created by structural defects in the direction parallel to the optical axis “c”. These features observed in GaSe thin films are not observed in nanoparticles. In our opinion, the absence of structural defects existing between layers in GaSe thin films in their nanoparticles creates opportunities for the wide application of these materials in optoelectronics..

The phenomenon of two-photon photoconductivity under the influence of laser beams has been discovered in CdS thin films. Figure 16 shows a measurement scheme of photoconductivity under the influence of laser beams in CdS thin films.

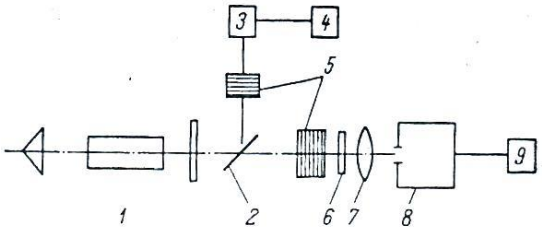
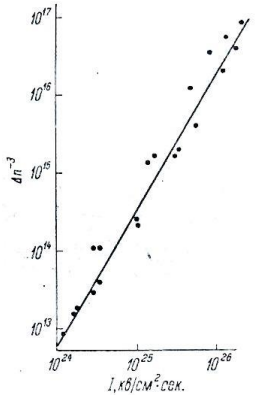
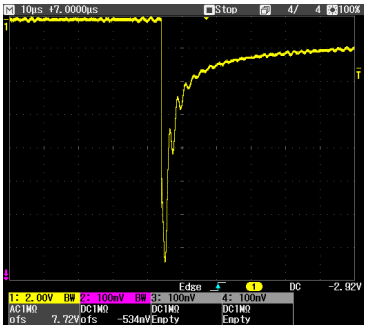


Figure 16. Measurement scheme of photoconductivity in CdS thin films: 1-laser; 2-bias plate; 3-nanosecond photodiode; 4,9-memory oscillographs; 5-neutral light filters; 6-edge light filters; 7-collecting lens; 8-camera with sample.

The bandgap width of CdS thin films is ~2.45 eV, therefore, under the influence of the second harmonic of the Nd:YAG laser ($\hbar\omega = 2{,}34\,\text{eV}$), a two-photon absorption phenomenon can occur in them. Figure 17,a shows the dependence of Δn - the concentration of non-equilibrium charge carriers formed under I_0 - the influence of laser beams on the intensity of the laser beams. As can be seen from the figure, Δn the dependence of I_0 on has a quadratic character. The quadratic dependence is satisfied in a wide range of intensity. At a laser beam intensity of ~$2\cdot10^{26}$ kV/cm^2·sec., the concentration of charge carriers is of the order of ~10^{17}cm^{-3}.





a b

Figure 17. a- Dependence of the concentration of non-equilibrium charge carriers formed in CdS thin layers under the influence of laser beams on I_0 the intensity of the laser beams, b- Oscillogram of photoconductivity in CdS thin layers.

In our opinion, the observed quadratic dependence of on indicates that non-equilibrium charge carriers Δn are formed as a result of two-photon photoconductivity under the influence of laser beams- I_0. It is known that the concentration of non-equilibrium charge carriers is determined by the following formula

$$\Delta n = \beta k I \mu \tau (1 - e^{-t/\tau}) \quad (16)$$

where β - quantum yield, I- light intensity, κ- light absorption coefficient, μ - carrier mobility, τ - carrier lifetime.

When the laser pulse duration (Δt =10ns) is shorter than the characteristic lifetime of the charge carriers τ ($\Delta t < \tau$), the value of the photoconductivity is determined solely by the rate of generation.

$$\Delta n = kI\Delta t \quad (17)$$

Since in the multiphoton absorption process $k^n \sim I^n$ olduğundan, n the two-photon absorption process κ~ I will be. The concentration of non-equilibrium charge carriers formed in it will depend quadratically on the intensity of the laser beams

$$\Delta n = k(I) I \Delta t = k I^2 \Delta t \quad (18)$$

Thus, the quadratic dependence of the concentration of non-equilibrium charge carriers on the intensity of the laser beam ($\Delta n \sim I^2$) allows us to say that two-photon photoconductivity occurs in the sample. Figure 17,b shows the relaxation curve of photoconductivity in CdS thin films. As can be seen from the figure, the relaxation curve consists of two components: fast and slow components. At high optical excitation intensity, the slow component almost completely disappears as a result of the filling of the bonding centers with charge carriers.

The dissertation also uses the multiphoton optical absorption method to determine the parameters of multilayer structures, which are widely used in optoelectronics. For this purpose, X-ray spectral microanalysis and single-photon excitation of individual layers are currently used. These methods violate the integrity of the sample and require considerable time for measurements. In contrast, the two-photon absorption method presented in the dissertation allows determining the composition of the layers, the width of the forbidden band of the semiconductor material present in each layer, the concentration of deep and shallow impurities, and the thickness of the layers.

It is known that when semiconductors are exposed to laser beams with an energy smaller than the bandgap width ($\hbar\omega < E_g$), a

multiphoton absorption phenomenon occurs. Layered $GaSe_{1-x}S_x$ semiconductor compounds are suitable objects for conducting this type of research. The bandgap width of $GaSe_{1-x}S_x$ varies in the range of 2.02÷2.21eV depending on the composition (x=0÷0.25), therefore, the 1st harmonic of the Nd:YAG laser ($\hbar\omega$=1.17eV) can cause a two-photon absorption phenomenon. The multilayer $GaSe_{1-x}S_x$ structure was created by the optical contact method. The essence of the optical contact method is that the two semiconductor materials in contact must have an almost "ideal" surface. In other words, the surface roughness of the materials for optical contact should not exceed a quarter of the wavelength of light. Naturally, not all semiconductors meet these requirements. The $GaSe_{1-x}S_x$ crystals we used are ideal for this purpose. These crystals have a layered structure with a natural mirror surface. In this case, there is no need for mechanical and chemical processing of the samples. $GaSe_{1-x}S_x$ crystals with a thickness of 20÷100μm were used to prepare the multilayer structure. Then the samples are pressed together and kept under pressure for several hours. The surfaces of the samples in contact adhere due to the intramolecular forces arising between them. Since the lowest layer is GaSe (E_g=2.02 eV), and the remaining layers are located with increasing values of E_g, the recombination radiation resulting from two-photon excitation occurs over a wide range without weakening from the sample. Various numbers of layers (from two to five) prepared on the basis of $GaSe_{1-x}S_x$ solid solutions (x=0÷0.25) were experimentally measured. Experiments were carried out under pressure to implement dense contact areas of the multilayer structure. Figure 18,a shows the volt-ampere characteristics of the $GaSe_{0,5}S_{0,5}$ structure at various applied external pressures. These layers were subjected to mechanical pressure directed perpendicular to the surface plane (along the “c” symmetry axis of the crystals). The pressure varied in the range (5-500) kg/cm^2. As can be seen, the value of the current in the forward direction increases with increasing pressure in the volt-ampere characteristic. However, starting from the pressure value of 500 kg/cm^2, the volt-ampere characteristic deteriorates.

Figure 18-b shows the photoluminescence spectra of the five-layer $GaSe_{1-x}S_x$ structure under two-photon excitation with the first harmonic of the Nd:YAG laser (ħω=1.17 eV). As can be seen from the figure, the number of lines in the photoluminescence spectra coincides with the number of layers in the structure. To determine the nature of the observed emission lines, the optical absorption spectra of individual layers at the fundamental absorption edge were measured. Comparison of the absorption spectra and luminescence spectra shows that there is a correspondence between them. Thus, based on the studies conducted, it can be noted that the values of 2.020 eV, 2.084 eV, 2.127 eV, 2.175 eV and 2.210 eV were obtained for the band gap of each composition in the $GaSe_{1-x}S_x$ multilayer structure, respectively. x: 1-0; 2=0.5; 3=0.1; 4=0.2; 5=0.25. It should also be noted that the band gap width determined for $GaSe_{1-x}S_x$ solid solutions is in satisfactory agreement with the data given about them in the literature.

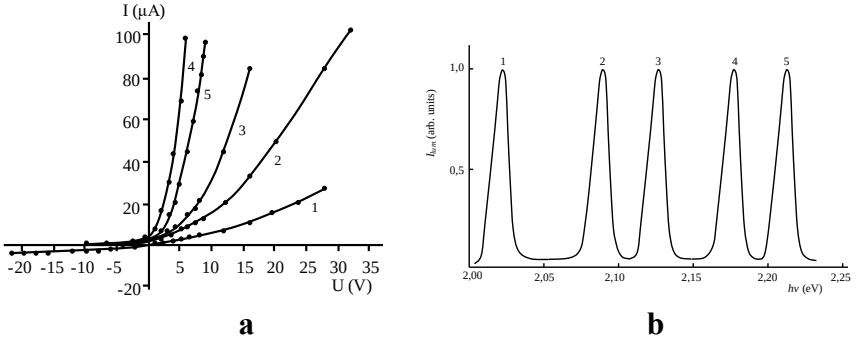


Figure 18. a- Volt-ampere characteristics of GaSe thin films at different external pressures (P), P (kg/cm^2): 1-50, 2-100; 3-300; 4–500; 5–600, b-Photoluminescence spectra of the multi-layered $GaSe_{1-x}S_x$ structure. x: 1-0; 2=0.5; 3=0.1; 4=0.2; 5=0.25.

As can be seen from Fig. 18,b, the intensity of radiative recombination is almost the same throughout the entire volume of the multilayer structure, which suggests that the intensity of the lines in the luminescence spectra is proportional to the thickness of the layer. In this case, it is possible to compare the relative

thicknesses of the layers in the structure by comparing the ratio of the intensities of the lines in the luminescence spectrum and, knowing its total thickness, determine the absolute values of the layer thickness. According to our measurements, the total thickness of the layers was equal to ~500 μm, which allows us to say that the thickness of each layer in the multilayer structure is equal to ~100 μm.

CONCLUSIONS

1. Nanoparticles of semiconductor compounds InSe, GaSe, GaS , CdS and CdTe were synthesized by laser ablation using a Nd:YAG nanosecond pulse laser. In addition to the traditional synthesis of nanoparticles from single crystals by ablation, for the first time InSe, GaSe and CdS nanoparticles were obtained using their constituent atoms, high purity (99%) In, Ga and Cd.

2. The surface morphology and structure InSe, GaSe, GaS , CdS və CdTe nanoparticles were studied using modern research methods such as X-ray diffraction analysis (XRD), scanning electron microscope (SEM), atomic force microscope (AFM) and energy dispersive X-ray spectroscopy (EDAX). It was shown that the obtained nanoparticles completely reflect the structure and stoichiometry of the semiconductor crystals used.

3. The mechanism of laser light absorption and plasma temperature in the semiconductors studied during the laser ablation process were determined. As a result of non-radiative recombination of electron-hole pairs formed in semiconductor crystals under the influence of powerful laser beams, the crystal lattice heats up to a temperature of ~10^5K.

4. The optical and luminescence parameters (absorption coefficient, band gap, exciton binding energy) of InSe, GaSe, GaS, CdS and CdTe nanoparticles were determined. It was found that the band gap of nanoparticles is 0.5-1.0 times larger than the band gap of the semiconductors studied. In GaS crystals with a wide band gap (Eg=2.52eV), the band gap width is 4 eV.

5 A photoluminescence line with a maximum at $\lambda = 592$ nm was detected in the luminescence spectrum of InSe nanoparticles excited

by the second harmonic ($\hbar\omega = 2{,}34\,\text{eV}$) of a Nd:YAG laser. The fact that the half-width of the photoluminescence line at high optical excitation (I_0=1.5x10^{25} photons/cm^2 x sec) is on the order of several angstroms, the quadratic dependence of the luminescence intensity on the laser power, and the high concentration of electron-hole pairs created by laser beams (Δn=4.5×1019$см^{-3}$) indicate the occurrence of a forced radiation effect in InSe nanoparticles.

6. A nonlinear optical absorption process was detected in InSe nanoparticles at high laser irradiation intensity. At a laser excitation intensity of ~10MW/cm^2, the refractive index change of InSe nanoparticles was observed to be $\Delta n(\omega) \approx 0.24$. At high optical excitation, exciton-exciton interaction leads to annihilation of excitons, which leads to a decrease and elimination of optical absorption in the exciton region.

7. The main parameters of the multilayer semiconductor structure, including the bandgap width, layer thickness, dopant concentration, and the mechanism of recombination processes, were determined by the two-photon excitation method with laser beams.

Published scientific articles on the topic of the dissertation

1.Салманов,В.М. Особенности фотопроводимости и люминесценции тонких пленок CdS и твердых растворов $Cd_{1-x}Zn_xS$ при лазерном возбуждении / В.М. Салманов, А.Г.Гусейнов,М.А.Джафаров [и др.] // Оптика и спектроскопия, – 2022. 130(10), – с. 1567-1570.

2. Салманов, В.М. Спектры поглощения и люминесценции наноструктуры GaSe и гетероперехода GaSe/InSe при лазерном возбуждении / В.М. Салманов, А.Г. Гусейнов, Г.Б.Ибрагимов [и др.] // Azərbaycan Milli Elmlər Akademiyasının Xəbərləri, – 2023. 5, –s.16-22.

3. Jafarov, M.A. A new method for synthesis of CdTe nanoparticles by laser radiation / M.A. Jafarov, V.M. Salmanov, A.G. Guseinov [et al.] // AJP FİZİKA–2023. 29 (3), – s. 13-18.

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5. Jafarov M.A. CdS nanoparticles synthesized by laser ablation / M.A. Jafarov , V.M. Salmanov, A.G.Huseynov [et.al.] // 8th International Conference Modern Trends in Physics, – Baku: – 30 nov.-1 dec., – 2023, – p. 47-48.
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7. М.А. Джафаров, Т.А.Мамедова. Оптические и фотоэлектрические свойстватонких пленок CdS и гетеропереходов $CuInSe_2$/ $Cd_{1-x}Zn_xS$. “Müasir təbiət və iqtisad elmlərinin aktual problemləri “ adlı Beynəlxalq elmi konfransı, Gəncə 2023, səh. 14-17.
8. Салманов В.М., Гусейнов А.Г., Мамедов Р.М., Мамедова Т.А., Ахмедова Ф.Ш. Оптическое поглощение и люминесценция в наночастицах İnSe, полученные методом лазерной абляции. “ Müasir təbiət və iqtisad elmlərinin aktual problemləri “ adlı Beynəlxalq elmi konfransı, Gəncə 2024, səh. 30-33.
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The defense will be held on "8" october 2025 at 9^30 at the meeting of the Dissertation Council ED 1.14 of Surpreme Attestation Commission under the President of Republic of Azerbaijan operating at Institute of Physics of the Ministry of Science and Education of the Republic of Azerbaijan.

Address: AZ-1073, Azerbaijan, Baku, G.Javid Ave., 131.Institute of Physics of the Ministry of Science and Education of the Republic of Azerbaijan.

Dissertation is accessible at the Institute of Physics of the Ministry of Science and Education of the Republic of Azerbaijan Library.

Electronic versions of the abstract is available on the official website of the Institute of Physics of the Ministry of Science and Education of the Republic of Azerbaijan.

Abstract was sent to the required addresses on "5" september 2025.

Signed for print: 30.06.2025

Paper format: A5

Volume: 35 370

Number of hard copies:20

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