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Nonlinear optical and quanta-dimensional effects in monoselenide of gallium and indium

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Nonlinear light absorption and its time evolution at high optical excitation levels in GaSe and InSe layered crystals have been experimentally investigated. It is shown that the nonlinear absorption observed in InSe in the region of exciton resonance is due to the exciton-exciton interaction. The effect of filling the zones detected in GaSe at high excitation intensities leads to a change in the absorption coefficient and the refractive index. For InSe nanoparticles obtained by the chemical deposition method, a quanta-dimensional effect was observed; the width of the forbidden band was dependent upon the dimensions of the nanoparticles.

Keywords: nonlinear absorption, GaSe, InSe, bandfilling effect, nanoparticles.

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1. Introduction

In semiconductors, photogenerated electron-hole pairs rapidly thermalize and relax into levels close to the band gap. Direct electron-hole pairs normally have a short recombination lifetime which, thus at low crystal lattice temperatures T_l, thermalization with the crystal lattice is not allowed. Thus, the temperature T_e of the electron-hole pairs is usually higher than T_l. Therefore, they will not only occupy bound states (excitons) which have the lowest energies, but also higher dissociated states (ionized excitons). The electron-hole pairs' density can be easily raised by increasing the photoexcitation intensity. A high electron-hole pair density will screen the Coulomb interaction between electrons and holes. For very strong screening, no bound electron-hole pair states exist [1, 2]. Thus, at low temperatures and with increasing pair density, the electron-hole fluid turns from an insulating gas of mostly bound electron-hole pairs or excitons into a metallic plasma of dissociated pairs. This phase transition is called the Mott transition of the excitons [3], even though the carriers are not at zero temperature.

GaSe and InSe crystals belonging to III-V compounds have received considerable attention recently as an interesting class of nonlinear optical materials. Possessing layered structure, high polarizability, optical homogeneity and naturally mirror-like surfaces, a number of nonlinear optical phenomena such as harmonic generation [4, 5], parametric light generation [6, 7], electron-hole plasma [8–12] and stimulated emission in the visible and terahertz region [13–19], etc., have been observed in these crystals.

Investigations of dimensional quantum phenomena in these semiconductors open up great prospects for constructing on their basis new devices with a wide range of functional capabilities. Scientists have established that the ultrathin nanosilic indium and gallium monoselenides have unique properties that qualitatively distinguish them among the remaining two-dimensional crystals. In the obtained samples of indium monoselenide, the electron mobility is the highest. This material parameter is extremely important in terms of improving the performance of devices that can be created on its basis. Another interesting property of indium monoselenide is that, unlike dichalcogenide and silicon, this crystal is a so-called direct-gap semiconductor. This makes it particularly promising for use in optoelectronics. In addition, the width of the InSe band gap depends essentially on the thickness of its layers. The ability to vary the width of the band gap of this material by selecting nanofilms of various thicknesses opens up wide possibilities for its application in optoelectronic devices that can function over a wide spectral range from the near infrared to the visible regions. Such an effect cannot be achieved in other graphene-like two-dimensional semiconductors [20]. This paper is devoted to an experimental study of nonlinear absorption and quanta-dimensional effects in gallium and indium monoselenides at high optical excitation levels.

2. Experimental method

The investigated single crystals were obtained by the Bridgmen method. GaSe and InSe have a layered structure, where each layer contains two gallium (indium) and two selenium close-packed sublayers in the stacking sequence Se-Ga(In)-Ga(In)-Se. The bonding between two adjacent layers is of the Van der Waals type, while within the layer, the bonding is predominantly covalent. The ingots were cleaved along the planes of layers, obtaining slices about (10-50) μm thick. The presence of exciton line in the absorption spectrum is indicative of a good quality material [21,22]. Mobility and concentration of charge carriers measured by conventional methods at room temperature were $\sim$ 20 cm^2/V·s, 1×10^{14} cm^{-3} and $\sim$ 1.2×10^3 cm^2/V·s, $\sim$ 7×10^{14} cm^{-3} GaSe and InSe, respectively. The samples were put into a helium cryostat equipped with a temperature controller which allows any temperature between 4.2 and 77 K to be maintained.

As an excitation source, a picosecond YAG:Nd laser with a pulse duration of 30 ps were used to excite InSe crystals. In this case, both the single beam and also double beam excitation were used [23]. In the first method, resonant excitation of excitons is performed by a parametric light generator. The second method (double beam method) uses pump-probe spectroscopy. After amplification, the light pulse is divided into two parts and two laser beams are focused on the same spot of a semiconductor sample. The laser labeled pump passes through a KDP crystal and doubles in frequency ($\hbar\omega = 2.34$ eV). The pump pulse has a relatively large intensity and is usually tuned to an energy within the absorption region of the InSe. The pump beam is absorbed, resulting in the generation of electron-hole pairs. The second light beam, referred to as the probe beam, is used to monitor the changes in the optical properties caused by the pump. The probe intensity is very small, not inducing any changes by itself. The transmission spectrum of the probe beam detected in the presence of the pump beam is compared to the spectrum without pump beam, giving the frequency–dependent absorption of the sample for different intensities of the pump. For this purpose, it is convenient to have a probe beam that is either spectrally broad or easily tunable in wavelength, making it possible to monitor the entire band-edge absorption region. In our case, the probe pulse was formed by passing the second laser beam through deuteroxide, after which it was converted into a pulse having a wavelength in the range 0.75–1.5 μm. The pump-probe technique described above may also be employed to study the time evolution of the absorption spectrum. The time delay Δt between the probe and pump pulses is caused by a change in the path length of the pump pulse. Spectral distribution of probe pulse passing through the sample was investigated by a double-monochromator. All experimental data were processed and computer analyzed.

As an excitation source, Rhodamine 6G dye laser (PRA, LN-107) pumped by the output of a N_2–laser (PRA, LN-1000), tuned through the region 594–643 nm with a repetition frequency of 10 Hz and a pulse width of 1 ns was used in the case of excitation of GaSe crystals. Lower excitation intensities were obtained by means of suitable calibrated neutral filters. Transmission spectra were obtained by shifting filters from the front to the rear of the samples and checking the experimental reproducibility in the region of transparency. The output signals were detected by a silicon photodiode and recorded by a storage oscilloscope (Le Groy 9400).

2.1. Nonlinear light absorption in InSe crystals at high optical excitation

Fig. 1(a) illustrates dependence of the magnitude of transmission coefficient on the emission intensity for an InSe single crystal excited by laser light having an energy $\hbar\omega = 1.327$ eV (resonant excitation of exciton) at 77 K. As it is seen from the figure, a nonlinear absorption in the exciton resonance region and occurrence of sample bleaching in the indicated light frequency at high excitation levels are observed. The observed bleaching saturates at the incident light intensity of $\sim$300 MW/cm^2. Diminishing of the magnitude of exciton absorption may be explained by the process of screening (Mott transition) for a high density exciton system (Fig. 1(b)). The density of the electron-hole pairs in our experiment reached $\sim$ 10^{20} cm^{-3} which exceeds the density necessary for the Mott transition in InSe (n_{Mott}= 2.5×10^{16} cm^{-3}) [24]. The detailed investigation of the bleaching and dynamics of nonlinear absorption in the exciton resonance region have been realized by using double beam method at 4.2 K. Similar to single beam excitation, in this case, bleaching is also observed in the exciton absorption region at $T = 4.2$ K.

Figure 1(c) illustrates clearly dependence of optical density on the excitation intensity in a frequency where the exciton absorption is maximum (the time delay between the probe and pump pulses is zero). The observed bleaching saturates at higher excitation levels with respect to the case of resonant excitation of exciton. This can be ascribed to the spatial inhomogenity of the excitation in the sample. Typical absorption length of the excitation power is of the order $1\sim1/\alpha\sim10^{-4}$ cm, α=10^4 cm^{-1} for $\hbar\omega = 2.34$ eV [5]. Disappearance of the exciton peak in this case may be explained by screening of the Coulomb interaction by free charge carriers. The screening length

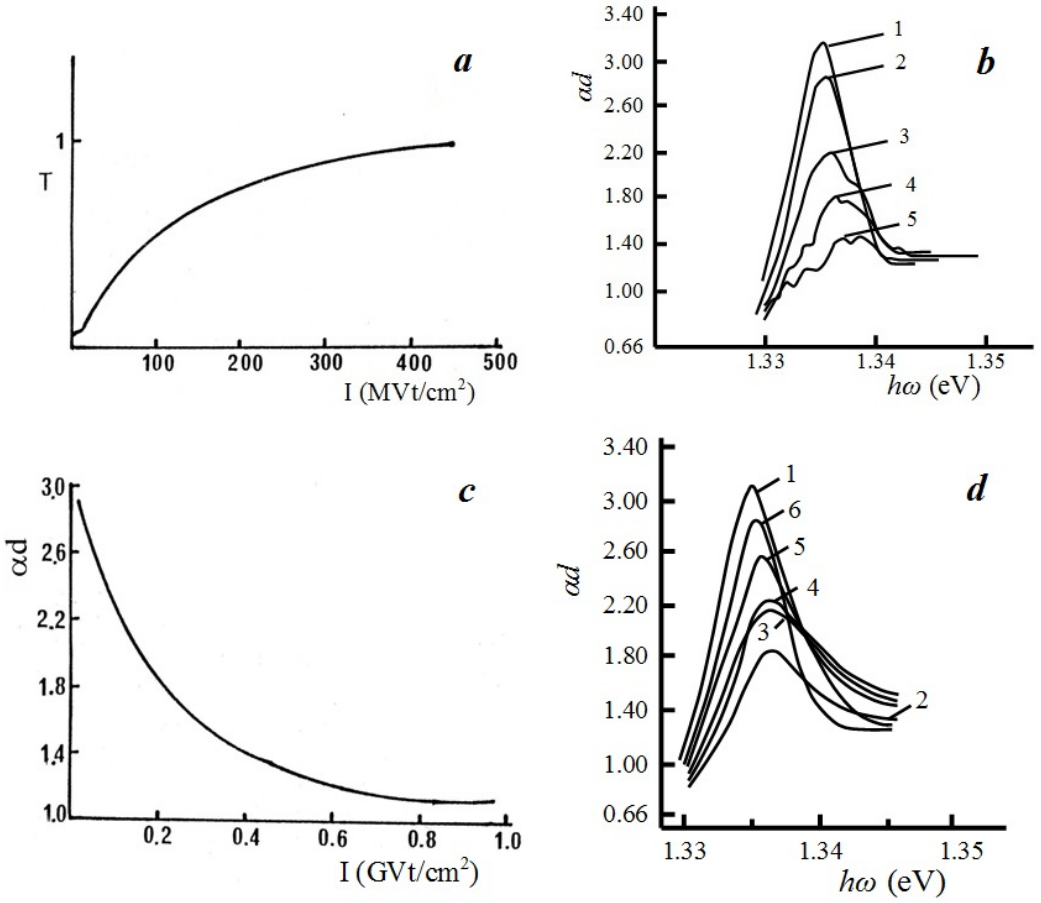

FIG. 1. (a) Dependence of the transmission coefficient on the excitation intensity in InSe (in the case of resonant excitation of exciton, $\hbar\omega = 1.327$ eV) at 77 K. (b) Absorption spectra of InSe at various excitation intensities I_{pump}. (MVt/cm^2): 1 – 0; 2 – 12; 3 – 60; 4 – 250; 5 – 600, $T = 4.2$ K. (c) Dependence of the optical density on the excitation intensity ($\hbar\omega_{pump} = 2.34$ eV, $\hbar\omega_{probe} = 1.336$ eV, $\Delta t = 0$) at 4.2 K. (d) Absorption spectra of InSe for different time delays between the probe and pump pulses: 1 – $I_{pump.} = 0$; 2 – $\Delta t = 24$ ps; 3 – $\Delta t = 98$ ps; 4 – $\Delta t = 297$ ps; 5 – $\Delta t = 660$ ps; 6 – $\Delta t = 910$ ps, $I_{pump} = 600$ MVT/cm^2, $h\nu_{pump.} = 2.34$ eV, $T = 4.2$ K

can be defined by the following equation [25]:

$$L = \frac{\hbar}{2}\left(\frac{\pi}{3}\right)^{1/6} N^{-16}\frac{\varepsilon^{1/2}}{em^{*1/2}},$$

where, ε is the dielectric constant of the crystal and m^* is the effective mass. By substituting these values from [26, 27] it is found that, L$\sim$10 Å which is less than the exciton radius ($\sim$37 Å) [12]. In Fig. 1(d) the absorption spectra of InSe crystals for different time delays between the probe and pump pulses are shown. It is clear from the figure that, the exciton absorption peak broadens and shifts toward higher energies with respect to the nonexcitation case. In the energy region between the exciton level and edge of the conduction band, an induced absorption is appeared. It should be noted that at a light intensity $I \sim$600 MW/cm^2, complete disappearance of the exciton peak is not observed. Thus, a situation is realized experimentally in which both the electron-hole plasma (EHP) and high density exciton gas are present in the sample.

3. Nonlinear light absorption in GaSe crystals at the fundamental absorption edge

The absorption spectra of GaSe at low (curve 1) and high (curve 2) excitation intensities are given in Fig. 2(a). As it is seen from the figure, at high excitation levels, the absorption coefficient is decreased, and along with the onset of absorption is also shifted towards higher energies. The change in the absorption coefficient $\Delta\alpha$ can be

obtained by direct subtraction of curves 1 and 2 in Fig. 2(a). The result is plotted in Fig. 2(b). It is seen that the maximum absorption change takes place in the vicinity of the band gap.

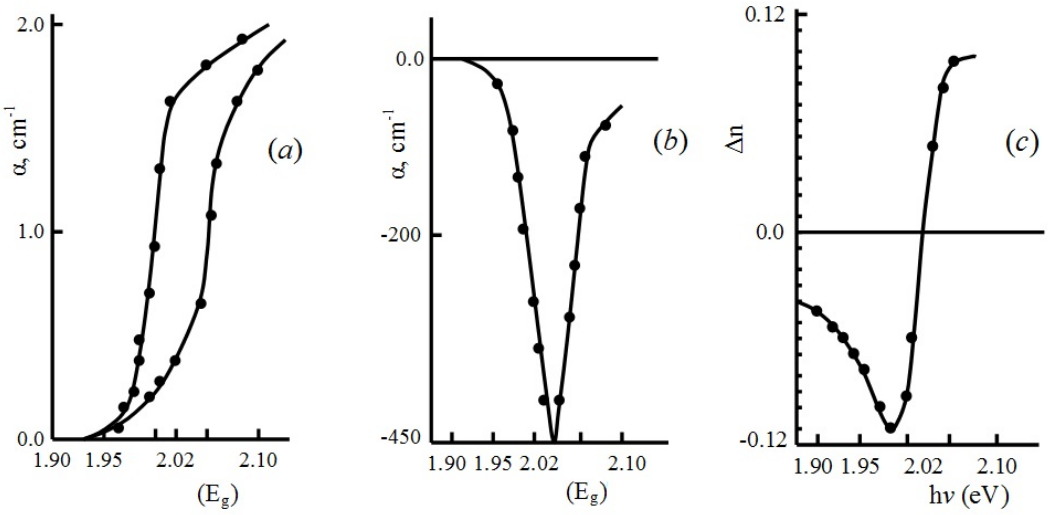

FIG. 2. (a) Absorption spectra of GaSe at low (∼3,5 mW/cm^2, curve 1) and high (∼12 mW/cm^2, curve 2) intensity excitations. (b) The change in the absorption coefficient $\Delta\alpha$. (c) The change in the refractive index $\Delta n(\omega)$

The observed nonlinear absorption near the band gap at high excitation intensities can be attributed to optical saturation effects in GaSe, i.e. electrons and holes generated by absorption of light which relax rapidly to a thermal distribution, blocking absorption states near the band edge. Effectively, this is like a shift of the band edge to higher energies with increasing laser intensity, which causes the absorption at the vicinity of the band edge to decrease. From Fig. 2(b), it is clear that, the absorption change becomes negative. Negative absorption means amplification as can be seen from Beer-Lambert's law:

$$I(t) = I_0 \exp(-\alpha x). \tag{1}$$

For $\alpha < 0$, the transmitted intensity is higher than the input intensity. This optical gain can give the possibility of producing a semiconductor laser based on GaSe crystals.

The bandfilling effect predicts that the change in absorption coefficient due to free carriers (neglecting exciton interaction) at photon energy $\hbar\omega$' above the band-gap energy is given by [28]:

$$\Delta\alpha(\hbar\omega') = -\alpha_0(\hbar\omega')2^{1/2}\left(\frac{\pi h}{k_B T}\right)^{3/2} \times \left[n_e m_e^{-3/2}\exp(-\Delta E_c/k_B T) + n_h m_h^{-3/2}\exp(-\Delta E_v/k_B T)\right], \tag{2}$$

where:

$$\Delta E_c = (\hbar\omega' - E_g')/(1 + m_e/m_h), \tag{3}$$

$$\Delta E_v = (\hbar\omega' - E_g')/(1 + m_h/m_e), \tag{4}$$

$\alpha_0(\hbar\omega')$ is the low-power absorption coefficient at photon energy $\hbar\omega'$, m_e is the effective mass of an electron, m_h is the effective mass of a hole, n_e, n_h are the integrated population density of free – electron and free – hole, respectively, E_g' is the renormalized band gap which results from exchange and correlation effects at high carrier densities [29]. Taking $m_e = 0.3m_0$, $m_h = 0.2m_0$ and calculated values of n_e, n_h, E_g', ΔE_c, ΔE_v from Eqs. (4)–(11) of Ref. [30], the percentage relative absorbance change $\Delta\alpha 100\%/\alpha_0$ is evaluated to be ∼12% in GaSe. This is in good agreement with the corresponding observed value of 15%. The small difference between these values can be due to the fact that, the exciton interactions were neglected in Eq. (2), while in wide-band-gap materials (such as GaSe). Coulomb electron-hole correlation effects should be taken into account which can eventually lead to the enhancement of the nonlinear absorption. Such saturation leads both to nonlinear absorption and to a strong intensity dependence of the refractive index. From the Kramers – Kronig relation [31] we may write the change in

refractive index at photon energy $\hbar\omega$ as:

$$\Delta n(\hbar\omega) = \frac{hc}{\pi} \int_0^\infty \frac{\Delta\alpha(\hbar\omega')}{(\hbar\omega')^2 - (\hbar\omega)^2} d(\hbar\omega'). \tag{5}$$

Using Eq. (5) to compute the index change related to the absorption change of Fig. 2(b), we obtain the result plotted in Fig. 2(c). As can be noted from Fig. 2(c), the change in the refractive index leads to nonlinear effects. $\Delta n(\omega)$ is negative at frequencies below the absorption edge and positive on the high-energy side. The laser-induced negative index change is referred to as a self-defocusing optical nonlinearity. The positive $\Delta n(\omega)$ on the high-energy side of the band gap corresponds to a self-focusing optical nonlinearity.

4. Quantum-dimensional effects in GaSe nanoparticles

The GaSe nanoparticles were obtained by a modified method of chemical deposition (Successive Ionic Layer Adsorption and Reaction-SILAR). Using the Debye-Scherer formula, the sizes of the nanoparticles obtained were calculated [32]. Estimates show that the dimensions of GaSe nanoparticles range from 7–20 nm. Using the energy-dispersive X-ray spectroscopy (EDAX) and the scanning electron microscope (SEM), the internal structure and structure of the GaSe nanoparticles were studied. Images obtained with SEM show that the obtained substances consist of spherical nanocrystals that are collected in a polydisperse form (Fig. 3(a)). The laser irradiation of GaSe nanoparticles results in a homogeneous distribution of the nanoparticles. In this case, the dimensions of the nanoparticles practically become the same (Fig. 3(b)). The image of the GaSe and InSe nanoparticles, obtained from AFM studies, shows that the homogeneous particle distribution is not observed (Fig. 4(a,b)). The EDAX method shows that the ratio of gallium to selenium (Ga:Se) is 1:1, hence the composition of the substance is in the stoichiometric ratio. Images obtained by an atomic force microscope show that a homogeneous distribution of nanoparticles is not observed on the substrate surface.

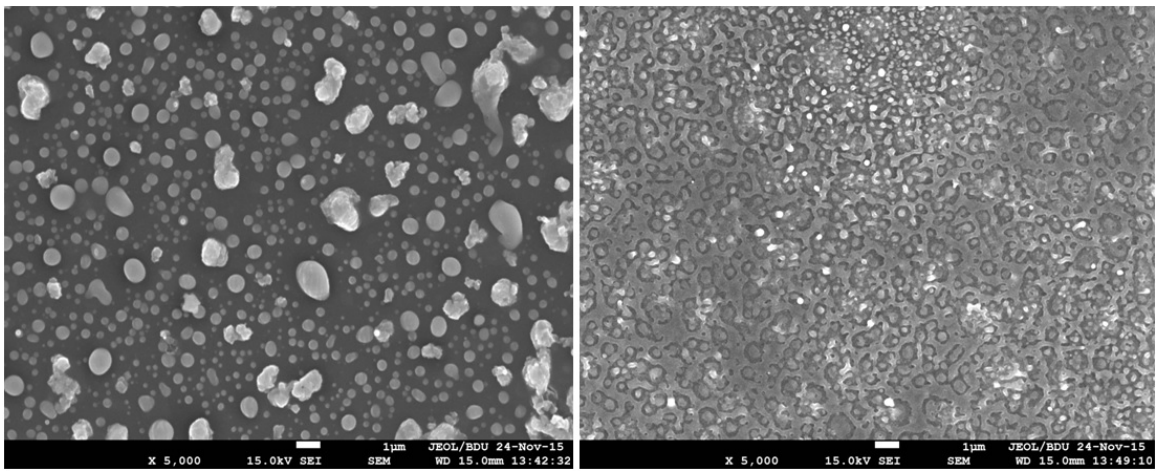

FIG. 3. SEM images of the GaSe nanostructure, grown on a glass substrate before (a) and after (b) laser light irradiation with a power of 6 MVt/cm^2

As is known, many mechanical, thermodynamic and electrical characteristics of a substance are altered in nanoparticles. Their optical properties are not an exception to this behavior. Correspondingly, the frequency of light emitted by nanoparticles increases with decreasing particle size. Experiments conducted by us showed that a quasi-dimensional effect is observed in GaSe nanoparticles, the width of the forbidden band depends on the dimensions of the nanoparticles.

Calculation of the width of the band gap was carried out according to the following formula:

$$E_g = E_g^{(bulk)} + E_b \left(\frac{\pi a_B}{D}\right)^2, \tag{6}$$

where E_g is the width of the forbidden band of nanostructures, $E_g^{(bulk)}$ is the width of the same substance without nanostructures, E_b is the binding energy of the exciton, a_B – the Bohr radius of the exciton, and D is the size of the nanoparticles (∼4–20 nm).

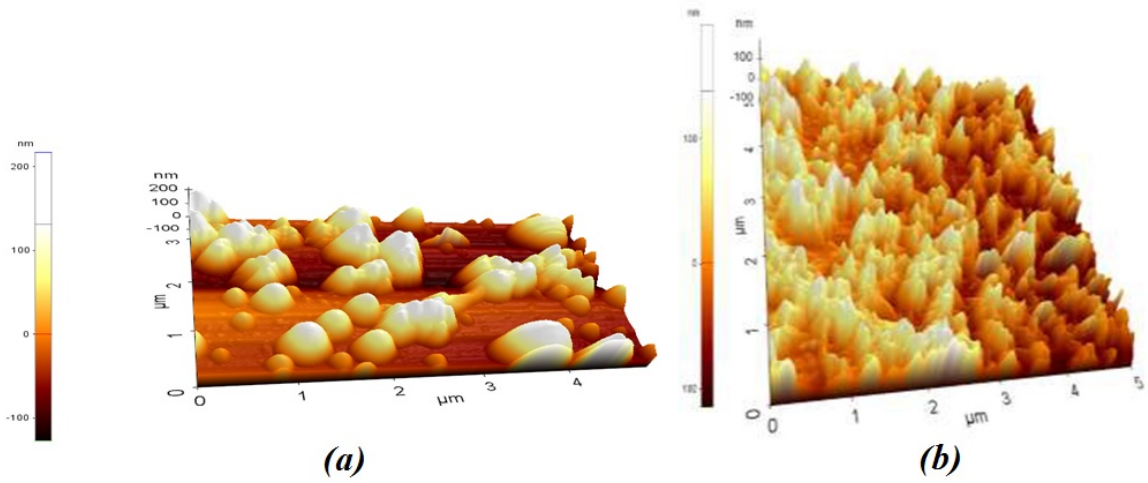

FIG. 4. AFM images of the nanostructure of GaSe (a) and InSe (b) grown on a glass substrate

The above parameters in GaSe have the following values: $E_g^{(bulk)} = 2.02$ eV, $E_b = 20$ meV, $a_B = 37$ Å. Fig. 5 shows the dependence of the width of the forbidden band of GaSe nanoparticles on the dimensions of nanoparticles. As can be seen from the figure, the quantum-size effect begins to significantly affect the width of the forbidden band when the dimensions of the nanoparticles become smaller than 10 nm.

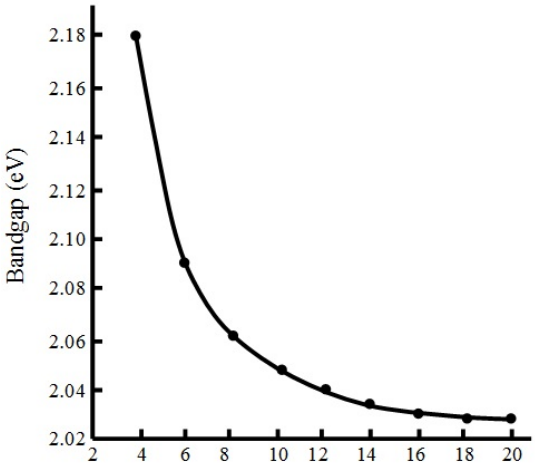

FIG. 5. Dependence of the width of the forbidden band of GaSe on the dimensions of nanoparticles

5. Conclusion

We note in conclusion that the nonlinear absorption observed in high-level optical excitation of InSe crystals in the region of exciton resonance is due to the exciton-exciton interaction. The density of the pairs generated by laser light in InSe (3×10^{19} cm^{-3}) is much higher than the density necessary for the Mott transition in these crystals. The induced absorption which appeared in the energy region between the exciton level and edge of the conduction band is due to the appearance of continuum states caused by shifting the energy band edges for InSe. The time dynamics of bleaching in the both the exciton absorption and induced absorption regions may be explained by recombination processes taking place in EHP and high density exciton gas. The illumination of the edge of the absorption band of GaSe, with its simultaneous shift to the high-energy region of the spectrum, is associated with the filling of energy bands at high optical excitation levels. It is shown that the observed band-filling effect allows a semiconductor laser to be created on the basis of GaSe crystals. In the nanoparticles of GaSe, obtained by a modified method of chemical deposition, a quanta-dimensional effect was observed. Reducing the size of nanoparticles from 20 nm to 4 nm leads to an increase in the width of the forbidden band from 2.02 eV to 2.18 eV.

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