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Electrical and Optical Properties of Laser-Induced Structural Modifications in PbSe Films

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Abstract: PbSe chalcogenide films are widely used as photosensitive elements in gas analysis devices. High absorption in the IR spectrum region and low electrical resistance are important characteristics. Continuous laser radiation exposure of films in the near UV range makes it possible to achieve the desired characteristics, replacing oven heat treatment in the technological process. In the considered laser technology, PbSe films are subjected to photothermal action by a spot of focused radiation in the progressive scanning mode. In this work, changes in the optical and electrical film properties were studied, and the mechanism of structural laser modification was also considered.

Keywords: laser modification; PbSe films; optical characteristics; photodarkening mode; photobleaching mode; heat treatment

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Citation: Olkhova, A.A.; Patrikeeva, A.A.; Sergeev, M.M. Electrical and Optical Properties of Laser-Induced Structural Modifications in PbSe Films. *Appl. Sci.* **2022**, *12*, 10162. https://doi.org/10.3390/app121910162

Academic Editor: Cristian FOCSA

Received: 9 September 2022
Accepted: 8 October 2022
Published: 10 October 2022

1. Introduction

Currently, humanity is experiencing an active era and widespread use of gas, oil, and refined products such as diesel fuel, gasoline, bitumen, fuel oil, and kerosene. However, these components and their vapors are flammable and explosive, and often contain toxic substances, which require the safety of workers and industrial facilities. To solve this problem, gas analysis equipment is used. The detector in such devices is chalcogenide films, which detect the presence of harmful contaminants in the air, as they have high absorption in the IR range, whereas gas molecules have absorption peaks. Among the many ways to increase the photosensitivity of films, such as heat treatment and alloying, the most accessible and low-cost method is laser modification of the detector structure. Therefore, the method proposed in the work for the occurrence of photosensitivity to the chalcogenide films allows for reaching the goal comparable with heat treatment or surpassing it.

Due to their unique properties, chalcogenide films are widely used in the photonics field as sensitive photodetectors and sensors for various purposes [1,2]. One example of such materials is PbSe films, which are characterized by high absorption in the spectral range from 1 to 4 μm [3–6]. On the other hand, PbSe films have photoelectric characteristics, which allows them to be used in photoresists [7]. The combination of high optical absorption and photoelectric properties makes it possible to use these materials in express analysis devices of organic substances and gases [8–12].

The optical and electrical characteristics of PbSe films are largely affected by the oxidation process, which in most technological processes is activated during heat treatment in an oven. The nucleation and growth of a new oxide phase in the film leads to an increase in the band gap, for example, from 0.27 eV to 3.17 eV for $PbSeO_3$ [7]. Film gradual oxidation leads to a film clearing and, in some cases, to a deterioration in electrical characteristics. To control the change in the film's optical characteristics during their oxidation, the ellipsometry method was used [13]. It was shown that a PbSe film 50 nm thick was oxidized during heat treatment in an oven at 623 K for 10 min, its refractive index and extinction coefficient decreased, and its thickness increased. However, the oven heat treatment process remains difficult to control, especially for opaque films with a thickness of more than 0.5 μm.

In recent years, laser technologies have been resorted to for the thermal processing of semiconductor films, where nanosecond pulses of excimer lasers were first used, before switching to continuous radiation (CW—continuous wave) of semiconductor lasers with wavelengths from 380 to 450 nm (V—violet) and power radiation from 0.2 to 3.0 W [14]. The use of laser technologies has greatly simplified the conditions for modifying films and also made it possible to use commercially available optical elements and laser sources and also reduced the requirements for the safe operation of laser systems. The introduction of new technologies made it possible to study the structure modification under laser irradiation, and the concomitant change in both the electrical and optical film properties has acquired a new meaning [15].

The authors [16] present a study of photoinduced changes occurring during laser irradiation. Such modifications are one of the simple and environmentally friendly methods that make it possible to control morphological and optical changes in the studied films with minimal surface damage. In addition to the laser modification method ease used to change the optical и electrical characteristics, the method also leads to bond breaking and rearrangements within the material structure [16]. This leads to a modification of the amorphous material localized structures, which leads to changes in optical properties, such as transmittance, absorption and extinction coefficient, refractive index, and optical band gap [16,17]. Laser processing made it possible to reduce the film element resistance by several times, which has found wide application as photoelectric sensors [17,18]. Further study of the structure and properties of laser modification mechanisms of chalcogenide films opens up opportunities for increasing the sensor's photosensitivity in gas analysis, as well as for creating sensors in the various organic liquid microanalysis.

In the present study, the sample electrical characteristics change was carried out by modifying the PbSe chalcogenide film structure by continuous near-UV laser radiation. A change in the electrical and optical film properties was found by studying samples before and after laser exposure.

2. Experiments

2.1. Materials and Methods

The films were created by the VTS (vacuum thermal spraying) method on a plane-parallel glass composite (GC) of 1 µm thick, followed by heat treatment (activation, sensitization) in open-type furnaces under atmospheric conditions. The films were manufactured by Optosense.

For the experiments, film samples were prepared before and after heat treatment in an open furnace. The untreated samples were subjected to a continuous wave (CW) laser modification with a semiconductor laser at a wavelength of 405 nm. Laser was line scanned not more than once.

A preliminary analysis of the optical properties of the film before and after laser exposure was carried out using optical microscopy in bright and dark fields for transmission and reflection. For this purpose, a Carl Zeiss Axio Imager microscope (Germany) was used.

To assess the surface of the films using transmission electron microscopy, a high-resolution scanning Merlin Zeiss electron microscope (Germany) was used.

Reflection of PbSe films was measured in the range of 0.3 to 1.1 µm with an MSFU-K Yu-30.54.072 spectrophotometer, LOMO (St. Petersburg, Russia), where the minimum detection area is 2 µm. The reflectance Rmeas of all samples were measured under normal light incidence.

An FSM-1202 infrared Fourier spectrometer (Tekhnokom, Yekaterinburg, Russia) was used for quantitative analysis and quality control of PbSe films.

2.2. Laser Modification

Laser action on PbSe chalcogenide films was carried out by continuous radiation of LSR405CP-1W semiconductor laser with a wavelength of 405 nm. The laser beam central part after the diaphragm was directed to a 40× objective (NA = 0.65), after which it was focused in the film location plane fixed on a Thorlabs MTS50/M-Z8 three-coordinate table (Figure 1). The treatment zone was visualized using an ocular camera.

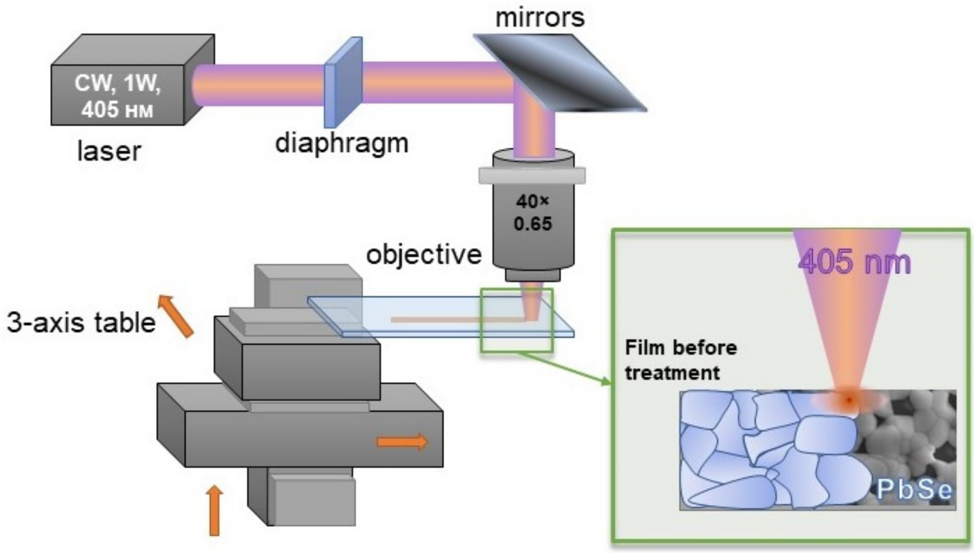

Figure 1. Experimental setup scheme for chalcogenide film's structure laser modification.

The radiation incident power on the film in the laser exposure mode was 22–33.6 kW/cm^2 with a spot diameter of about 10 µm. The radiation intensity profile in the spot cross-section was a beam with a flat top. The film structure was modified at a laser spot scanning rate of 950 µm/s.

2.3. Electrical Characterization

Measurements of the samples' electrical characteristics were carried out on the laser-treated and untreated sample areas. Figure 2 schematically shows the setup for measuring the film's electrical characteristics.

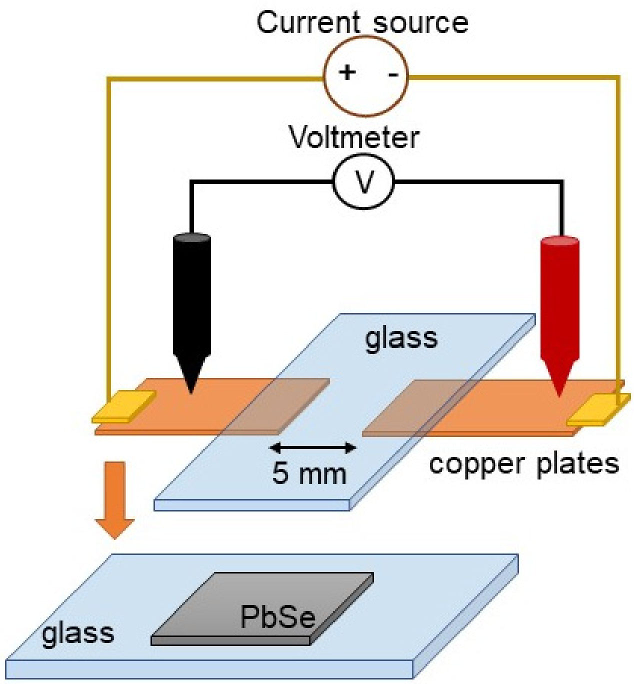

Figure 2. Experimental scheme for measuring the film electrical characteristics.

The circuit used copper plates that were soldered to the current source contacts with a voltage of 4 V. Voltmeter probes were connected to the copper plates to measure electrical characteristics. The sample was placed at the bottom and covered with a glass slide with plates to best ensure contact and also to avoid damage.

3. Results and Discussion

3.1. Laser Modification of the Structure

Optical characterization was carried out and the structure of untreated PbSe films, as well as films subjected to heat treatment, was studied. According to the results of optical microscopy, the untreated sample has an unstructured surface (Figure 3a,b), and the film itself contains light-scattering inhomogeneities with a size of about 1.0 μm—dark for transmission and bright for reflection. The untreated sample is not transparent (transmission is about 0.1) and has a high reflection in the UV region of the spectrum (reflection at a wavelength of 405 nm corresponds to 52%).

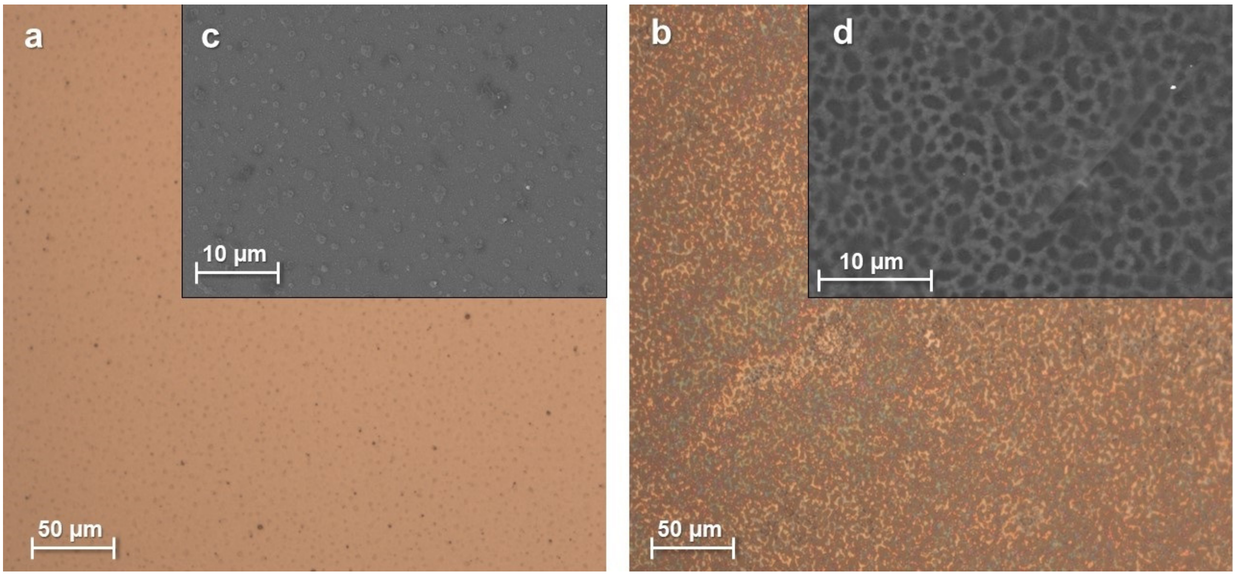

Figure 3. Image of PbSe film obtained using an optical microscope in a bright field of reflected light: (**a**) untreated, (**b**) after heat treatment. Image of PbSe film obtained using the SEM secondary electron detector: (**c**) untreated, (**d**) after heat treatment.

According to the results of optical microscopy (Figure 3a,b), the sample has a denser structure after activation in the furnace. Island formations (the second phase) with a size of more than 1.0 μm appear, which are transparent in transmitted light and randomly arranged. The film matrix itself becomes opaque. However, the spectral transmission of the sample increases compared with the transmission of the untreated sample. This feature is due to the properties of the second phase, an oxide that is transparent in the optical wavelength range. The high concentration of the second phase provides a significant increase in the transmission of the entire film (by 30 times). The film matrix in reflection becomes lighter. The second phase is dark in reflected light. Therefore, its reflection is less than the reflection of the film matrix, which ensures a decrease in the reflection of the entire sample.

According to the SEM results (Figure 3c,d), it can be seen that for the films after heat treatment, uncompensated stresses arise in the matrix due to the growth of the second phase. The second phase, being a material with a less dense structure, grows during the oxidation process. It increases in volume while maintaining mass, so compressive stresses arise, leading to the formation of cracks over the entire surface.

The reflection spectra of the untreated film and the film that has undergone activation are presented below (Figure 4).

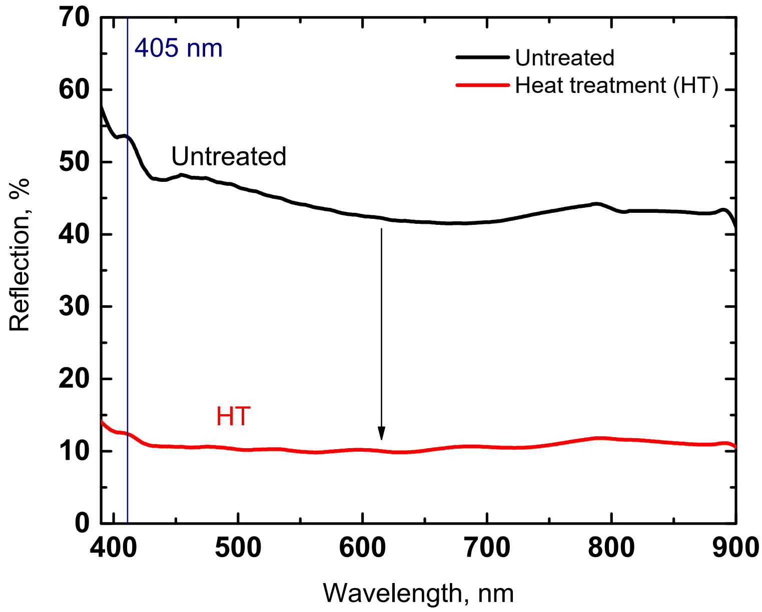

Figure 4. Reflection spectra of PbSe film: untreated (black curve), after heat treatment (red curve).

Based on the results obtained, it is possible to make an assumption about the mechanism of modification of the film's optical properties during heat treatment in an oven. During the heating of the film and its annealing, the phase transformation of the film matrix occurs: the inhomogeneities become centers for the nucleation of the second phase (lead oxide (Pb_3O_4)), and the matrix itself becomes denser, which leads to a decrease in its transmission. Since the oxide is transparent and the size of the regions is commensurate with the film thickness, the film becomes clear at a high concentration in the second phase. The reduction in the reflection of the acting laser radiation can be used to control the quality of film processing in the implementation of feedback.

During the formation of a structure with suitable optical characteristics, an increase in transmission at the wavelength of laser radiation is observed. In this case, the sample is clarified as a result of the formation of a transparent oxide and an increase in the concentration of the second phase, which affects the decrease in reflection to 1–2% with a subsequent increase to 5–7% at a constantly low transmission, approximately equal to 0.1.

The effect of continuous laser radiation on the structure and optical properties of PbSe chalcogenide films was studied under conditions of scanning with a focused beam. First, the laser treatment mode was found (Figure 5b), in which the material was observed to become photobleached and melted, followed by its destruction, leading to the formation of microcracks.

PbSe films that were not subjected to heat treatment in an oven were used as reference samples. To study the laser irradiation effect on the film's structure modification, sample optical characterization was carried out before and after exposure to radiation. According to the results of scanning electron microscopy (SEM), the untreated sample had an unstructured surface (Figure 3a,c), and the film itself contained inhomogeneities with a size of about 1.0 µm.

Structure modification after laser irradiation is shown in Figure 5b,d. Between the tracks, it can be seen that the film was not subjected to thermal action from the laser irradiation zone and retained the characteristics of the untreated sample (Figure 5b). Within the track, local modification regions were formed, spaced at the same distance from each other (Figure 5d). The mechanism of laser modification is associated with film local heating to a softening and melting temperature, followed by material redistribution entrained by a moving heat source with a high temperature gradient. At some point in time, the film within the track becomes thin, which is represented in the SEM image as periodic dark areas. Light, denser regions are film thickenings that form during sharp heating when the laser spot approaches and cools when it is removed [13]. As a result of the film material redistribution, concentric regions with local elevations are formed in the track center, and depressions are formed on its periphery. The structure morphology and the periodicity of light and dark regions depended on the scanning rate, laser spot size, and incident radiation power density. These parameters determined the heat source size, its maximum temperature, and the heating and cooling rate.

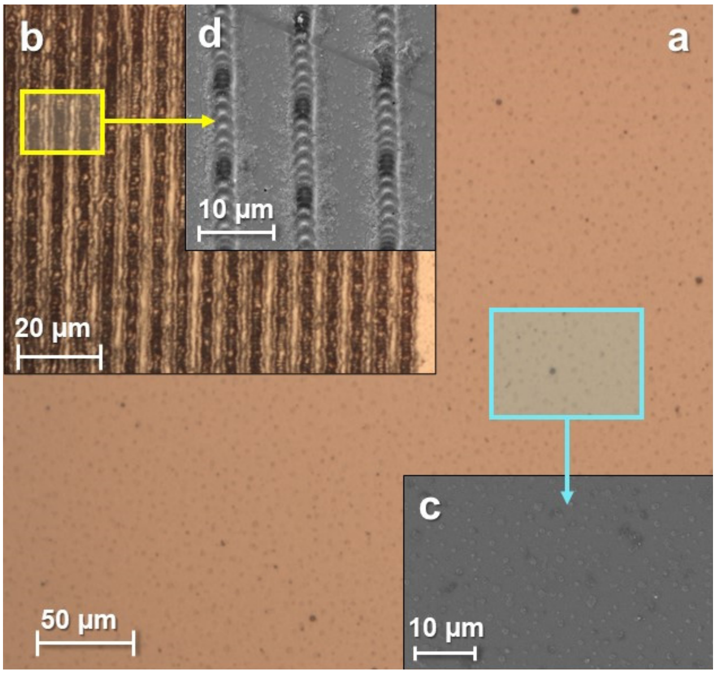

Figure 5. PbSe film optical microscopy a in a bright field of reflected light: (**a**) untreated, (**b**) after laser modification; PbSe film pictures obtained with the SEM secondary electron detector: (**c**) untreated, (**d**) after laser modification.

3.2. Electrical and Optical Characterization

The film's electrical characterization, including the measurement of electrical resistance and current, was carried out before and after sample structure laser modification. The measurement results are shown in Table 1. From the data obtained, it can be seen that with an external voltage in the area equal to 0.6 V, the current value in the modified region increased by 62.8%, compared with similar measurements of the untreated film. Under an external voltage of the measured area equal to 4 V, the current value for the modified areas increased by 40% in comparison with similar measurements of the untreated film.

Table 1. Electrical characteristics of PbSe films.

External Voltage	Untreated Film		Film after Laser Modification		Comparison: before and after Irradiation	
	R, Ohm	I, nA	R, Ohm	I, nA	dR, %	dI, %
U = 4 V	218	100	208	140	−4.59%	40.00%
U = 0.6 V	1400	86	1200	140	−14.29%	62.79%

The PbSe film optical characteristics study was carried out to explain the features of the band structure and material band gap E_g. The measured characteristics were used to determine the optical constants: refractive index n and extinction coefficient k [19]. The samples were opaque for the visible wavelength range. Therefore, the film reflectance spectra were measured before and after laser irradiation (Figure 6). For the film-modified sections in the selected spectral range, a sharp decrease in reflection was observed compared with untreated film. In terms of their optical properties, the modified regions approached an absolutely black body (Figure 6b).

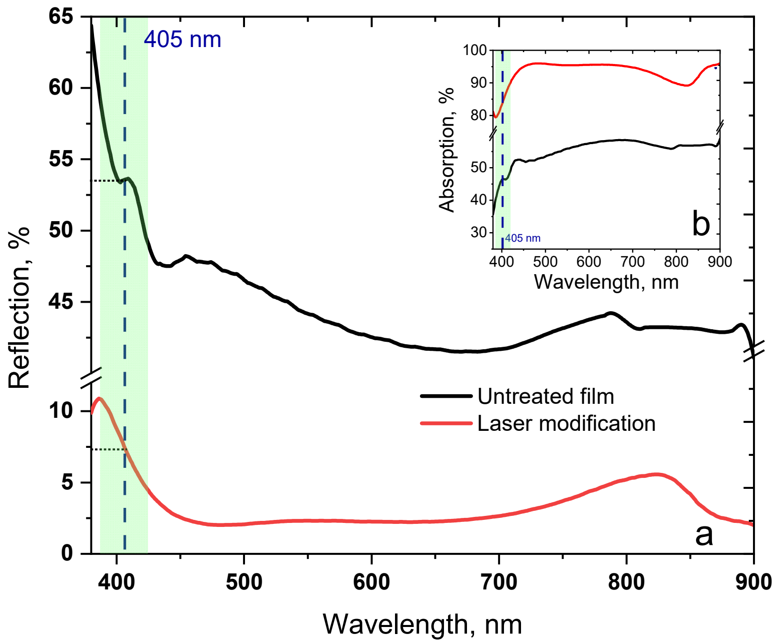

Figure 6. Dependence of (**a**) reflection, (**b**) absorption from wavelength for different PbSe films processing: untreated (black curve), laser modification (red curve).

The untreated and after laser modification film optical constants were calculated for a wavelength of 405 nm and presented in Figure 7. The absorption coefficient was calculated by the expression: $\alpha = A/d$, where A- absorption, d- film thickness. The extinction coefficient was calculated through a by the expression: $k = a\lambda/(4\pi)$, where λ is the wavelength.

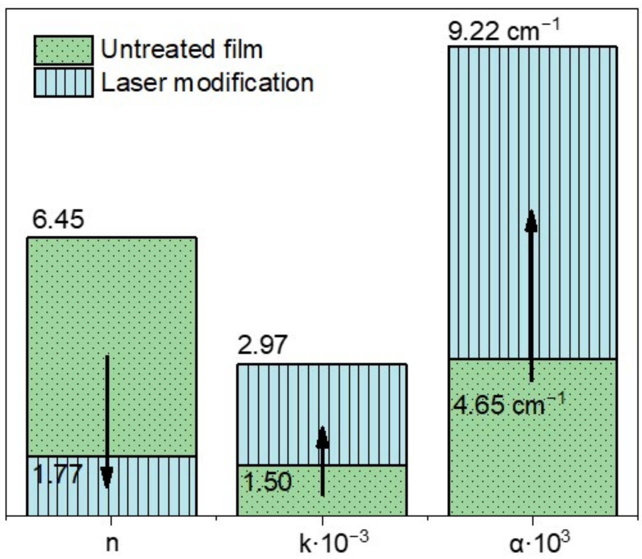

Figure 7. PbSe films optical characteristics at a wavelength 405 nm for untreated film (green) and film after laser modification (cyan): refractive index n, extinction coefficient k, absorption coefficient α.

The refractive index was calculated from the expression:

$$n = [(1 + R) + \{(1 + R)^2 - (1 - R)^2(1 + k^2)\}^{0.5}]/(1 - R)$$

where *R*—reflection.

Films laser modification resulted in a refractive index decrease and an increase in the absorption coefficient and extinction coefficient, which may be associated with the valence and conduction bands modification as a result of a high temperature gradient.

The regimes were also determined under which the effect of PbSe films photodarkening can be observed (Table 2). The power density of the acting radiation q was determined by Formula (1), where d is the laser spot diameter, equal to 10 µm. Using Formulas (2) and (3), we determined the exposure time t on the sample and the energy density w, where ν is the scanning speed.

$$q = P\pi r^2 = 4P\pi d^2 \tag{1}$$

$$t = d\nu \tag{2}$$

$$w = qt \tag{3}$$

Table 2. Scanning modes with continuous UV radiation.

	P, mW	V, mm/s	q, kW/cm^2	t, µs	w, µJ/cm^2	Regime Name
Regime 1	11.7	1.1	15	9	134	Modification threshold
Regime 2	17.2–26.4	0.9–1.2	22–33.6	8–11	241–269	Photodarkening
Regime 3	13.4	0.25	17	40	682	Burn

The PbSe film optical band gap was determined under various processing conditions by the expression: $(\alpha h\nu)^2 = B^2\ (h\nu - E_g)$, where $h\nu$ is photon energy, B is the Tauc parameter, E_g is the band gap [16]. As a result of two straight sections extrapolation, the values E_g = 1.33 and 1.38 eV were determined (Figure 8). The obtained values are in good agreement with the previously published results of the scientific group T.S. Shyju et al. [19], where the band gap varied from 1.46 to 1.62 eV. The decrease in the band gap is associated with an increase in the probability of charge carriers' passage from defect states in the modified regions of the film induced by thermal processes.

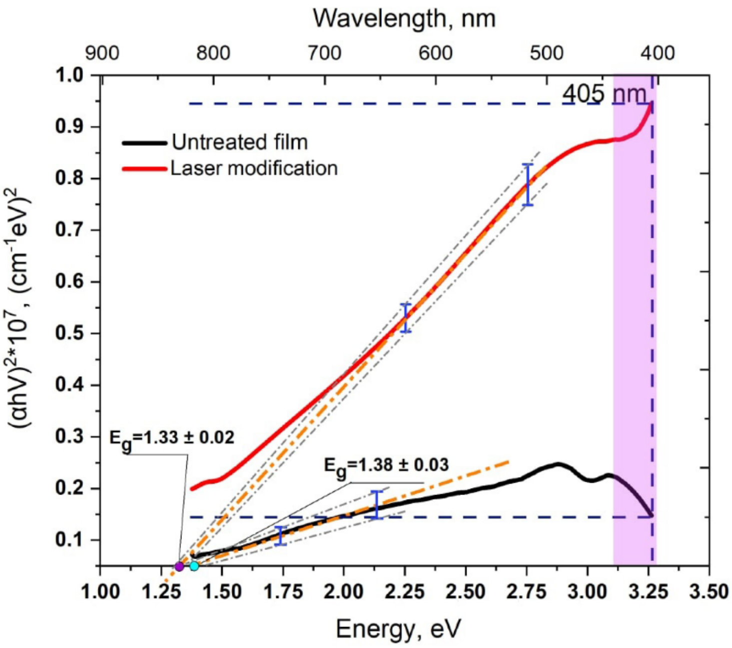

Figure 8. Optical band gap E_g graphical determination (orange line) for PbSe films various treatments: untreated (black curve), laser modification (red curve).

3.3. Modification Mechanism

Based on the above results, an assumption can be made about the mechanism of film structure laser modification (Figure 9).

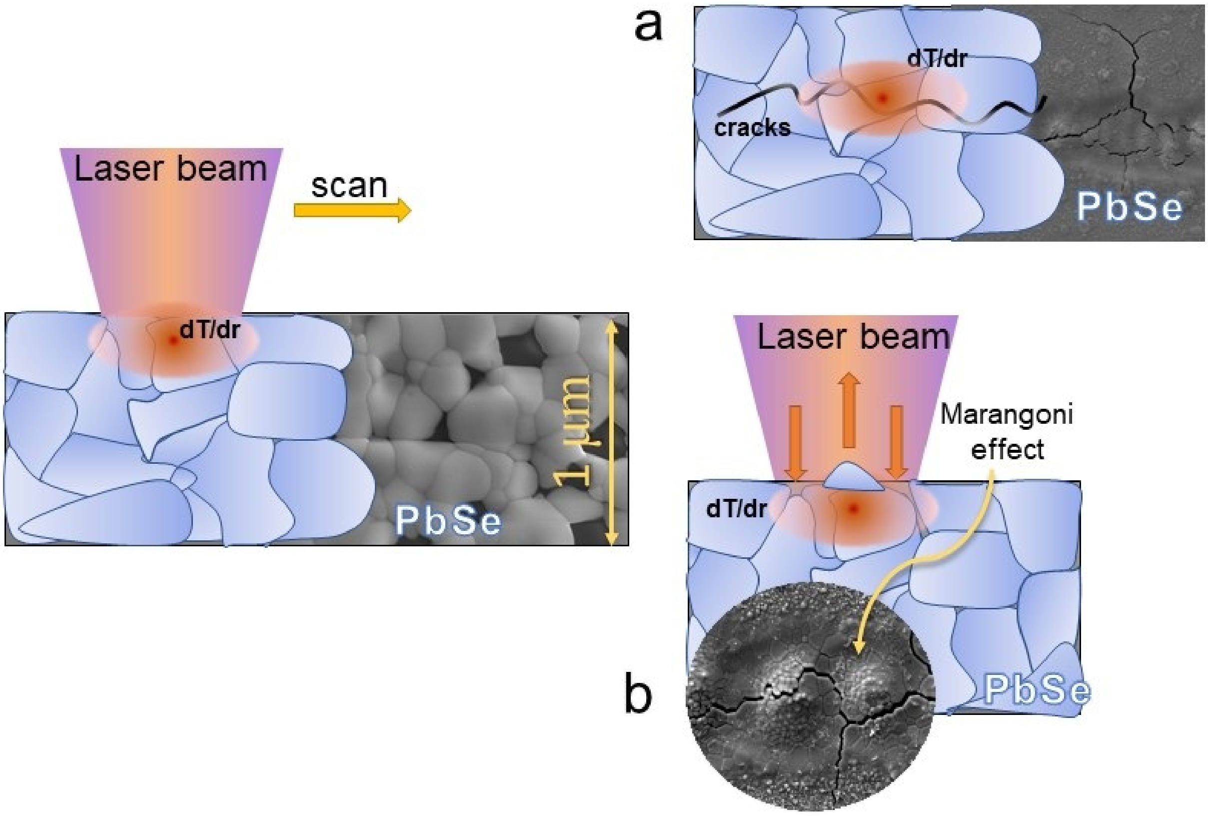

Figure 9. Mechanism of defective centers occurrence in the film modification area: (**a**) crack formation, (**b**) Marangoni effect.

The structure of the film in the track region was studied using scanning electron microscopy (SEM) (Figure 9). Within the track, local modification regions are formed during photodarkening. These regions are at the same distance from each other, which is associated with the formation of a film melt pool in the track area and the redistribution of the material in the mode of sharp heating–cooling with a moving local heat source (Figure 9b). The mechanism of the formation of periodic microdomains is related to the Marangoni effect [20,21] when a part of the material from the melt pool is periodically drawn into the center of a heat source bounded by a laser spot. As a result of the redistribution of the film material, local hills with a diameter of 1 μm at the top are formed in the center of the track, and depressions are formed on its periphery. The structure's morphology and periodicity depended on the scanning rate, the size of the laser spot, and the power density of the incident radiation, which determined the size of the heat source, its maximum temperature, and the heating–cooling rate. In some places of the track, cavities are formed with the removal of material (dark holes), under which there is a film substrate with fragments in the form of nanoparticles remaining from the film.

Cracks (Figure 9) are the result of an overheating of the film in the area of laser exposure due to the high temperature gradient dT/dr, which contributed to uneven thermal expansion and the occurrence of excessive stresses in the film. During the motion of the laser spot (Figure 9), the formed crack (Figure 9a) began to move, which led to its scribing. It is likely that the processing conditions were normal during the scan, however, a crack was initiated at the beginning of each track when the laser spot accelerated.

As a result of laser action on the film material, the concentration of defect centers increased, which became secondary sources of free charge carriers in the film. For this reason, after sample irradiation, the current increased and the resistance decreased. In addition, a decrease in the band gap indicated an increase in the probability of electron-hole pair transfer from defect states.

4. Conclusions

The effect of laser irradiation on the electrical and optical characteristics of PbSe films was studied. Laser radiation exposure in the scanning mode led to a film structure modification and decrease in reflection by a factor of 7 compared with the untreated sample. The structure of the film after laser irradiation differs significantly from the untreated sample due to the redistribution of the material within the track when the melting temperature is reached in the region of the laser spot.

Film structure laser modification led to a change in the sample's electrical characteristics. The value of the current after irradiation increased by more than 60% under an external voltage of 0.6 V, and by 40% when an external voltage of 4 V was applied. The resistance of the films after irradiation decreased by more than 10%.

The band gap energy in the film after irradiation decreased from 1.50 eV to 1.33 eV. This decrease is due to the defect center's appearance in the film-modified regions, which increased the electron transition probability from the bound to the free state.

The data obtained show that laser modification of films is a promising direction for improving electrical characteristics. In the future, it is possible to use modified chalcogenide films as sensors of organic substances, as well as in gas analysis applications.

Author Contributions: Conceptualization, A.A.O. and M.M.S.; methodology, A.A.O.; software, A.A.P.; validation, A.A.O., A.A.P. and M.M.S.; formal analysis, A.A.P.; investigation, A.A.P.; resources, A.A.P.; data curation, A.A.O.; writing—original draft preparation, A.A.O.; writing—review and editing, M.M.S.; visualization, A.A.O.; supervision, M.M.S.; project administration, A.A.O.; funding acquisition, M.M.S. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by the Russian Science Foundation grant (project no. 19-79-10208).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

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

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