

Production of photosensitive structures based on CdSe nanoparticles and carbon nanotubes by physical methods

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Abstract

The paper shows the possibility of producing photosensitive structures using physical methods without the use of chemical synthesis and vacuum techniques. Suspensions of CdSe nanoparticles were produced by pulsed laser ablation in a liquid medium (chloroform), and layers of semiconductor material (CdSe) and conductive layers (carbon nanotubes) were formed at the gas-liquid interface and transferred to solid substrates by the Langmuir–Schaeffer method. The size of CdSe nanoparticles in the layers ranged from 60 nm to 200 nm, and the bandgap was 2.3 eV. Multilayer film structures consisting of a layer based on synthesized CdSe nanoparticles and a layer of conductive carbon nanotubes, that demonstrated photosensitivity in the optical region, were obtained. The value of current density under lighting at 1 V and –1 V equals 1.25 mA/cm² and –1.35 mA/cm², respectively. The maximum ratio of the illumination current density to the dark current value was 13.5 times (at a voltage of 1 V).

Keywords

CdSe, conductive carbon nanotubes (CNTs), laser ablation, Langmuir–Blodgett technology, photodiode, semiconductor

1. Introduction

Development of simpler and cheaper technological processes is one of the most important stages in the development cycle of any technology. This also applies to production techniques that can be used to obtain multilayer functional structures for photosensors and photo-conversion devices. Technological processes that exclude the use of vacuum technology and involve only the most

simple chemical processes that are less demanding on the purity of reagents are especially interesting for potential application.

Most methods can be classified as chemical or physical. Typical examples of methods of the first group are sol-gel synthesis, synthesis in micelles, chemical precipitation, etc. Such methods are difficult to implement, they often use expensive starting components and tools, in addition, the synthesis takes quite a long time. Physical

methods (such as the molecular beam epitaxy method and other vacuum methods), as well as a group of physico-chemical methods (which can include mechanochemical synthesis, solvothermal synthesis and synthesis in supercritical solvents) require special technological equipment and a combination of complex conditions – pressure, temperature, rate of deposition of materials on the substrate, duration of exposure to the material, creation of a special deposition/synthesis environment [1, 2].

Laser ablation is a well-known method that involves ejection of macroscopic amounts of materials from a surface of a solid usually induced by the interaction of short ($\sim 10^{-13}$ to 10^{-8} s) and intense ($\sim 10^6$ to 10^{14} W/cm²) laser pulses with the surface [3].

According to various sources [4, 5], the greatest advantage of laser ablation is the fact that this technique does not require any additives or precursors, thus, resulting in very high purity of the products of synthesis. In addition, laser ablation is capable of stoichiometric transfer of target material to the substrate (even for very complex materials) and is very versatile as the high-intensity focused pulsed laser can ionize a majority of materials to produce either single- or multielement compounds, multilayers, nanoparticles and nanostructures. It provides an opportunity to accurately control the growth rate and flexibility of various experimental parameters, i.e., ambient gas nature and pressure, gas flow rate, substrate temperature, laser incident intensity, number of applied pulses, pulse repetition rate, duration and wavelength, the substrate-to-target separation distance, target composition and structure and power density. It is capable of depositing multicomponent layers, shows an unlimited degree of freedom in the geometry of the experimental set-up, and is a clean and safe technique due to the use of light [5]. Laser ablation is often used to produce both thin films and nanosized objects of various composition [3, 6]. The composition of produced films mostly depends on the target composition. Thus, it is an attractive technology for the synthesis of nanoparticles (NPs) and composite materials, such as CdSe or similar semiconductor-doped glasses [7–9]. Another important factor affecting the nature of products is the chemical composition of medium where the laser ablation is performed. Pulsed laser ablation in a liquid (PLAL) has many specific features and advantages [3]. The liquid medium provides for a more effective heat dissipation from the impact zone of laser radiation. Swift supply of a large amount of energy results in formation of a very small size reaction zone. The overall dimensions of nanoparticles formed within this zone depends on the quantity of substance located there. This makes laser ablation quite similar to micelle-based NPs synthesis [10–13].

Abd A.N. et al. [14] described the method of producing colloidal spherical NPs of CdSe by laser ablation of cadmium selenide target in methanol and toluene solutions and showed that the size and shape of produced particles have a strong dependence on the liquid chosen as a medium for synthesis. Laser ablation in toluene produced

well-defined CdSe NPs with an approximate diameter of 50 nm, while NPs ablated in methanol had an approximate diameter of 34 nm. In liquids, sedimentation of synthesis products to solid surfaces is complicated and can be viewed as a separate independent process.

Another promising physical method of production of nanosized film structures is Langmuir–Blodgett (LB) technique. There are many sources noting that this technique can become a suitable alternative to vacuum methods [15]. The key feature of this technique is the presence of amphiphilic properties of the molecules of substances forming a floating layer. These properties uniquely determine the position of molecules at the “gas–liquid” interphase and contribute to the formation of a monomolecular crystalline 2D-layer [15, 16].

Amphiphilic properties and low solubility in water determine the stability of floating layers and their technological characteristics. To form a floating layer from materials that do not have amphiphilic properties, it is necessary to integrate these substances into amphiphilic matrices and prepare nanocomposites with the required parameters. This way allows the formation nanofilms containing such large objects as conductive carbon nanotubes (CNTs) [17–19] or CdSe NPs [20–23].

Often, oleic acid or trioctylphosphine oxide are used as stabilizers and surfactant matrices for nanoparticles, and they are added at the NP synthesis stage. And, as a rule, the chemical synthesis of nanoparticles assumes the presence in the solution of an excess of surfactant that is not bound to the surface of the nanoparticles. But this is precisely the significant drawback of chemical synthesis methods. There is no way to accurately calculate the amount of free (i.e. not bound to the surface of the NP) surfactant in the solution. Therefore, an additional procedure to remove excess surfactant is often required. [24]. In this regard, laser ablation methods are a much “cleaner” alternative.

Returning to the LB method, one cannot ignore another important feature: it allows the application of conductive layers without creating short circuits. Of course, there are some difficulties and contradictory points here. Amphiphilic substances are usually dielectrics, but molecules of conducting materials do not have amphiphilic properties. To resolve these contradictions, composite compositions based on an amphiphilic matrix and a conductive component as inclusions are used, as mentioned above.

Such large nanosized objects as CNTs (if their surface has not been specifically modified) are hydrophobic, insoluble in water and at the same time are molecules that conduct electrical current well. This makes them a good choice for Langmuir layer formation. Of course, their complex structure and the lack of prominent amphiphilic properties makes it impossible to produce layers with high degree of order. But due to the chaotic interweaving of nanofibers, CNT layers have anisotropic structural and electrophysical properties. This allows avoiding additional problems associated with the manifestation of isotropy

of electrophysical properties, which exists, for example, in sheet graphene. And the stability of the CNT layer can be increased by including in a surfactant matrix or additives that functionalize the CNT surface. Such functionalization leads to an increase in the solubility of CNTs in organic solvents, minimization of interactions between CNT molecules, and therefore to a more uniform distribution of molecules on the water surface and their greater order in the floating layer [17–19].

Floating layers of NPs or CNTs formed at the surfaces of a water subphase can be transferred to solid substrates using Langmuir–Blodgett or Langmuir–Schaeffer (LS) techniques (with horizontal or vertical substrate orientation during application, respectively). In this way, we can create CNT and NP films layer by layer, being able to control the individual composition and molecular organization of each layer at our discretion. [19, 22, 25, 26].

The following achievements can be noted as some examples demonstrating the capabilities of the LB method. High stability of the layers formed on the surface of water and the local order of CNTs in films on solid substrates can be directly caused by the modification of the CNT surface with crown ethers. This also allows for the successful transfer of Langmuir layers of multi-layer and single-layer CNTs onto hydrophilic silicon substrates [27, 28]. Jia et al. [29] showed that, in addition to functionalization of CNT surfaces with an amphiphilic compound, octadecylamines, the length of CNT also affects the structural orientation of CNTs in the film. The authors studied the CNT-based films and layers with length ranging from 500 to 3000 nm. They showed that the lower is the length of CNT the greater is their capacity for ordering and orientation in any particular direction. Fedoseeva et al. [30] showed the results of CdS NPs synthesis on the surface of vertically oriented CNTs. The X-ray photoelectron spectroscopic analysis showed that annealing and formation of a hybrid structure leads to the transfer of electron density from CdS to CNTs.

Thus, it can be seen that the interest in films based on NP and CNT is consistently high. This concerns both the preparation of solutions, the methods of formation and transfer of layers, and the influence of post-processing on the properties of these films. The aim of our research is to develop a technology for creating multilayer photosensitive structures based on CdSe nanoparticles and CNTs by combining PLAL with LS technique.

2. Materials and methods

The initial reagents included CdSe powder with a particle size of 44–150 μm (CdSe medium, 99–99.999 wt.% purity, Hunan Fushel Technology Limited, China), multiwalled CNT (>95 wt.% purity; diameter $\approx$ 8–50 nm; length $\sim$ 1–50 μm ; ash <1.5 wt.%) acquired from CTI materials (GRAFTON Company, USA); chloroform with 99.95% purity (Component-Reactive, Russia), a square FTO (Fluorine-doped Tin Oxide) glass sample sheet with

resistivity of $\sim$ 10 $\Omega/\text{sq.}$ and an area of 2.25 cm^2 size and a square silicon (*p* type <111> orientation) sheet with an area of 0.25 cm^2 and resistivity 1–10 $\Omega\cdot\text{cm}$ and thickness (400 μm).

The target was made by pressing 5 g of CdSe powder into a tablet with a diameter of 15 mm and a thickness of about 3 mm using a 5 t press. The tablet was placed to the bottom of a 25 ml glass beaker that was then filled with chloroform. The height of the chloroform layer above the tablet was close to 5 mm.

Irradiation was performed with a Nd:YAG laser at a energy of 700 mJ with wave length of 1064 nm, a pulse width of 10 ns, and a pulse train repetition rate set to 2 Hz. Laser ray (diameter – 1.2 mm) was focused from the source by a focusing lens onto a point on the target with approximate size 20 μm . The estimated radiation energy density at the focus was $0.22\cdot 10^6 \text{ J/cm}^2$.

The suspension with CNTs was homogenized using the Hielscher Ultrasonics UP200Ht (Hielscher, Germany) handheld ultrasonic homogenizer operating at the output of 200W and frequency of 26 kHz.

The transmission and absorption spectra were registered with the Shimadzu UV-1800 spectrophotometer (Shimadzu, Japan). The photoluminescence spectra were recorded using the installation based on the Ocean Optics QE Pro-FL broadband spectrometer capable of recording PL spectra in the wavelength range of 380–1100 nm using the laser beam excitation at $410\pm 10 \text{ nm}$ KN2002 Langmuir–Blodgett (LB) troughs (KSV Nima, Finland) were used to transfer the layers to solid surfaces.

Characterization of surface morphology of samples with CdSe NPs and CNTs was performed with Mira II LMU Scanning Electron Microscope (SEM) (AztecLive Advanced Ultim Max 40 Inca Wave 500, TESCAN, Czech Republic) and Inspect F50 (Spain) Field Emission Scanning Electron Microscope (FESEM).

The samples reaction to lighting was studying by subjects the samples to Solar simulator LSS9000 IV characterization system (Fytronix Electronic Technologies, Turkey) as a white light source with density of luminosity ranging from 1 to 100 mW/cm^2 . The current-voltage curves (CVC) were registered with Agilent (Keysight) B1500A parameter analyzing equipment and PM-5 probe station. The module of the parameter analyzing equipment allowed making precise measurements with current resolving power of 10 fA (if the power source voltage is up to 100 V).

3. Results and discussion

3.1. Production of a CdSe NPs-based suspension via laser ablation

To produce a suspension of CdSe NPs, we put a pure CdSe tablet in 5 ml of chloroform and ablated it with 250 laser pulses over a period of 2 min. Then we left the solution

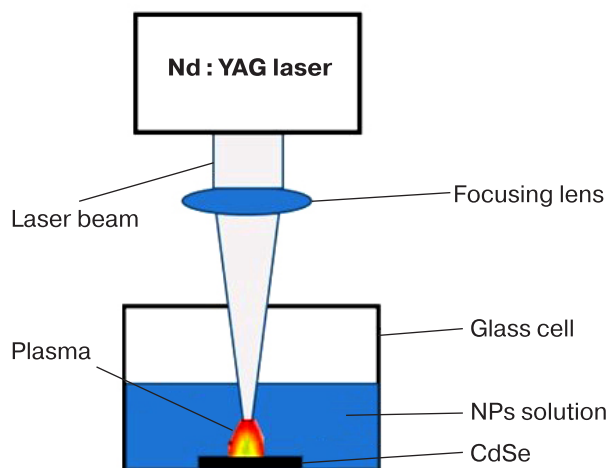


Figure 1. The experimental setup for laser ablation of CdSe tablet immersed in chloroform

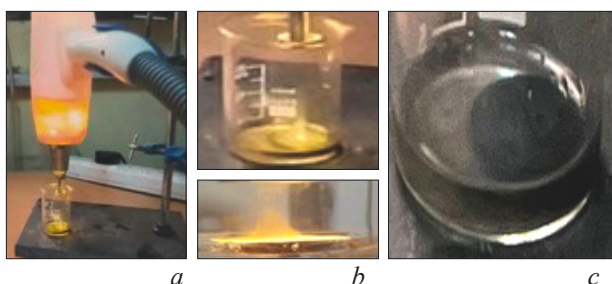


Figure 2. Light emission forming as a result of laser's interaction with CdSe target: (a) PLAL emission during the pulse; (b) formation of a cloud of ablated particles as a result of PLAL; (c) CdSe suspension after ablation

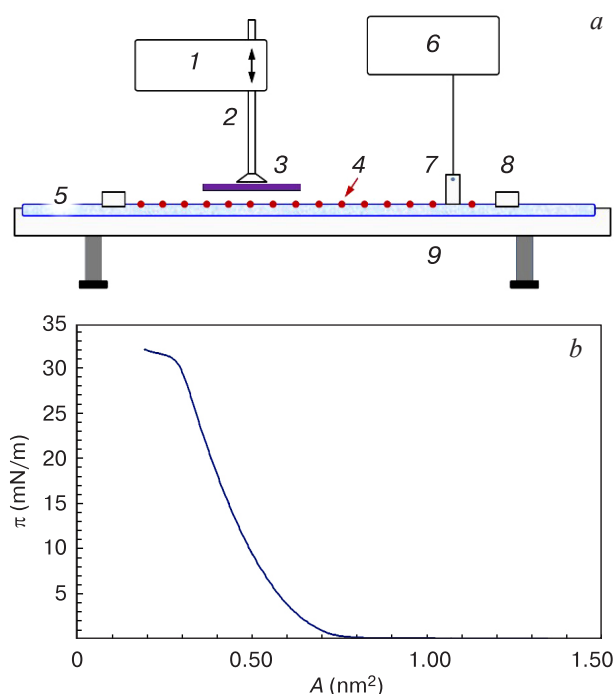


Figure 3. Schematic diagram of the LS method (a) and isotherm CdSe layer on the water subphase (b): 1 – dipper, 2 – substrate holder, 3 – substrate (ITO/FTO coated glass plate); 4 – CdSe NPs layer; 5 – distilled water; 6 – surface tension sensor; 7 – Wilhelmy plate; 8 – movable barriers; 9 – Langmuir trough

for some time to let the heavier particles to sediment at the bottom of a glass beaker. After that, we transferred the homogeneously gray supernatant containing lighter (nano-sized) CdSe particles to another beaker and removed the residues of heavier particles from the beaker into a separate container. We rinsed the glass beaker which contained a CdSe tablet with chloroform, then returned the CdSe tablet; after that we poured fresh chloroform into the beaker and repeated the ablation process. We removed the newly formed suspension and added fresh chloroform instead. Then we repeated the procedure (Fig. 1).

Then we added the tablet and chloroform to the container again and subjected it to ablation by another pulse. We repeated this procedure 20 times to produce 100 ml of NPs suspension.

Characteristic cracking and formation of a dense cloud above the CdSe table (Fig. 2b) during ablation indicates of an explosive nature of light interaction with the target resulting in formation of microbubbles containing the evaporated materials. This serves as an indirect confirmation of the right choice of ablation parameters (pulse power and velocity, distance between source and target etc.).

3.2. Formation and characteristics of thin films

3.2.1. CdSe films

The CdSe films were deposited using the LS deposition technique. 100 μ l of the suspension of CdSe in chloroform was applied drop-wise and distributed among the surface of aqueous subphase with the area of 243 cm^2 . After 7 min have passed and chloroform has completely evaporated, we compressed the layers of nanoparticles to the area of 5 cm^2 . After that, we transferred the layer to a horizontally oriented substrate using the LS method as shown in Fig. 3.

We clearly observed the formation of a homogenous CdSe layer and successfully transferred it to the FTO substrate and silicon (Fig. 4).

To further investigate the morphology of synthesized CdSe particles, we examined them with SEM. Scanning electron microscopy images demonstrated a good deposition and uniform distribution of CdSe composition in the

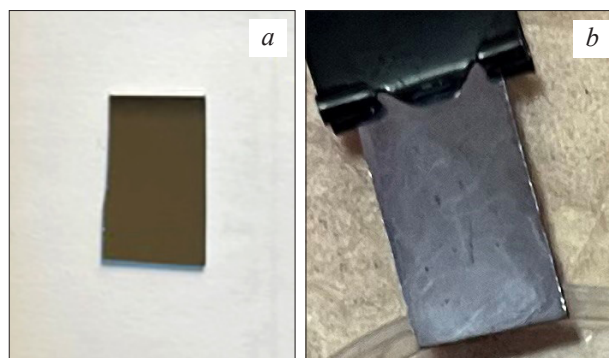
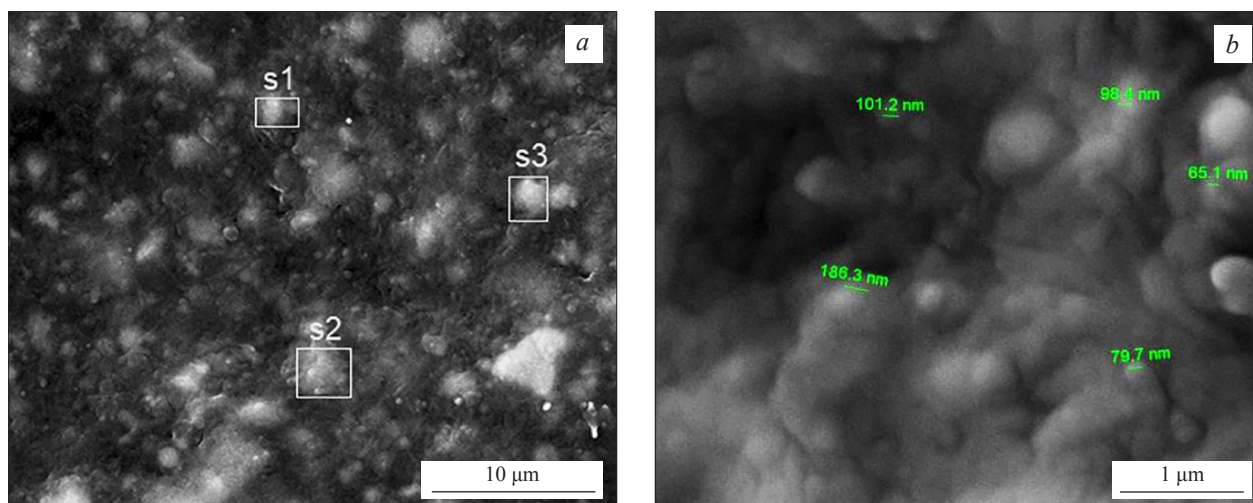


Figure 4. Silicon substrate before (a) and after (b) deposition of thin films using LS method: CdSe thin films deposited to the silicon substrate

Table 1. EDX analysis of Cd and Se atomic ratio Statistical characteristics of found atomic values

Detected elements	Label of the selected point			Statistics			
	S1	S2	S3				
	Atomic (%)			Max	Min	Average	St. deviation
Se	48.90	51.77	50.51	51.77	48.90	50.39	1.44
Cd	51.10	48.23	49.49	51.10	48.23	49.61	1.44

**Figure 5.** SEM image of CdSe films morphology

multilayer structure. Scanning electron microscopy measurements clearly show that the size of CdSe nanoparticles range from 60 to 200 nm (Fig. 5).

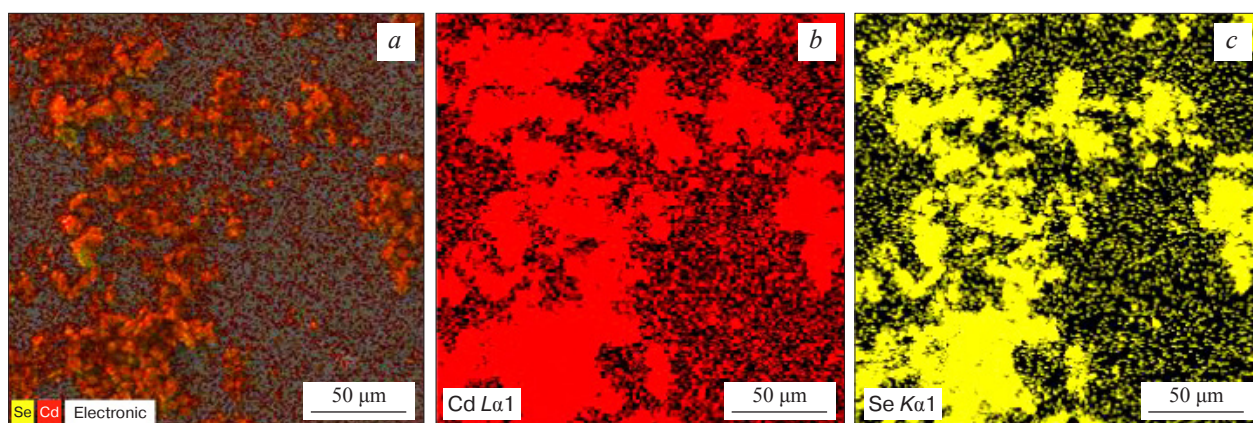
The results of energy dispersive X-ray analysis (EDX) shows that the average atomic percentage of Cd and Se atoms in CdSe film is approximately equal (see Fig. 5, Table 1). We selected three points (S1, S2, S3) and investigated the composition of the layer with the help of SEM.

Surface element mapping showed that the Cd и Se atoms are distributed unevenly with the maximal amount located inside larger nanosized objects (greater than 60 nm in size); at the same time, close stoichiometric quantities of Cd and Se are observed in various nano- and submicron-sized objects. The EDX spectroscopy confirmed that the CdSe had been deposited on the substrate (Fig. 6).

The absorption and transmission spectra (Fig. 7) of ablated solution were registered in a glass cuvette with an optical path of 1 cm. At the 350–360 nm range the transmittance of CdSe sharply increased to 61%, then it decreased to 57%, thus corresponding with the absorbance peak of the CdSe nanoparticles, and then again it increased up to 70% (starting from 575 nm for the ablated structures) and stabilized at 73% starting from 950 nm. Thus the absorbance can be determined according to the relation below

$$A = 2 - \log T,$$

where A is the absorbance, T is the transmittance, the results match the laboratory table and chart provided by

**Figure 6.** EDX images of CdSe multilayers

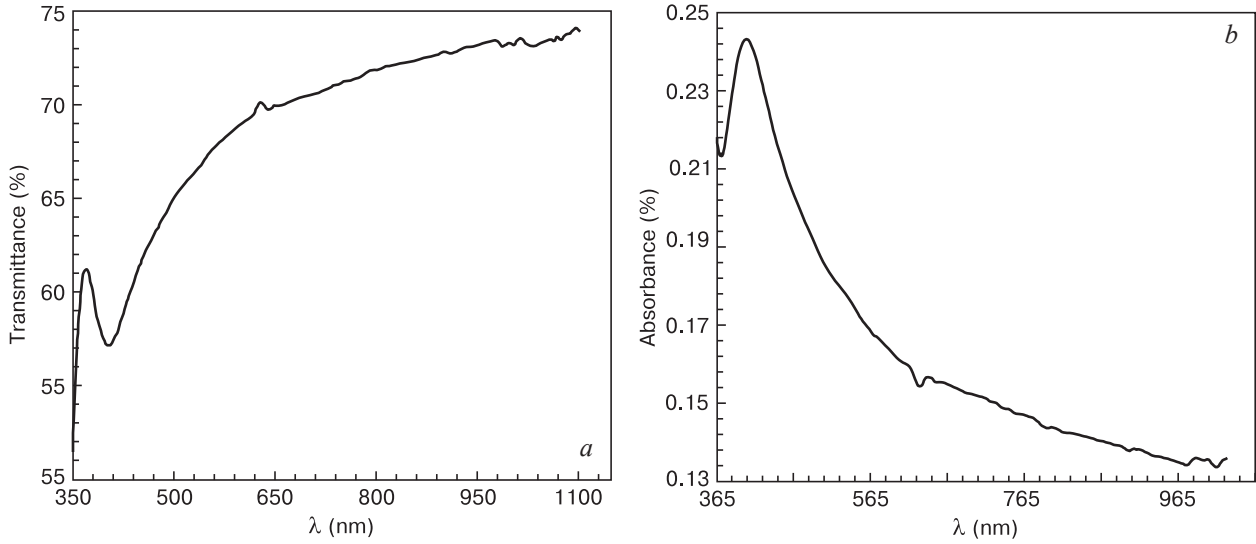


Figure 7. Transmittance (a) and absorbance (b) spectra of CdSe NPs solution

Sigma–Aldrich [31]. The absorption coefficient α can be calculated from relation below

$$\alpha = 2.303(A/T).$$

Thus, we can see that minimal transmittance (57%) for ablated structure is observed at 400 nm.

The optical band gap E_g can be determined from the absorption coefficient $\alpha(h\nu)$. For this purpose we used the known Tauc relation for the direct optical transition between valence and conduction bands [32]:

$$(\alpha h\nu)^2 = A(h\nu - E_g).$$

Figure 8 provides a graph explaining the determination of the optical bandgap energy for CdSe nanoparticles.

The optical bandgap energy E_g is deduced from extrapolation of the straight line to $(\alpha h\nu)^2 = 0$. The estimated

energy gap value is 2.3 eV. This can attributed to the quantum confinement effect [14, 33], therefore, we can state that synthesized CdSe nanoparticles have semiconductor properties.

We can see, that the photoluminescence (PL) spectra of the ablated nanoparticles in thin films on solid substrates have two peaks at 700 nm (more intensive) and 531 nm (less intensive) (Fig. 9). The 531 nm peak may be caused by the presence of CdSe nanoparticles with smaller diameter, that are present in the film in smaller quantities, thus, confirming the value of the generated energy gap through the following law $E = hc/\lambda$.

3.2.2. CNTs films

We used a two stage process to make CNT suspension. At the first stage, we dissolved $12 \cdot 10^{-5}$ g of CNT powder in 0.01 l of chloroform. The concentration of resulting suspension was $12 \cdot 10^{-3}$ g/l. In the second stage, we dilut-

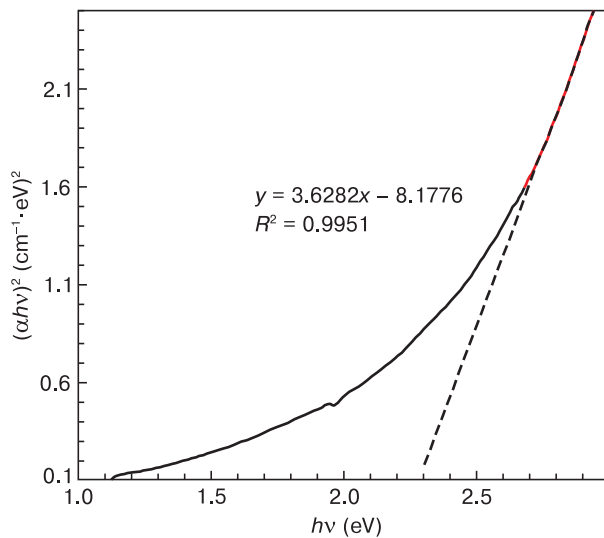


Figure 8. Plot of $(\alpha h\nu)^2$ vs $h\nu$ for CdSe nanoparticles

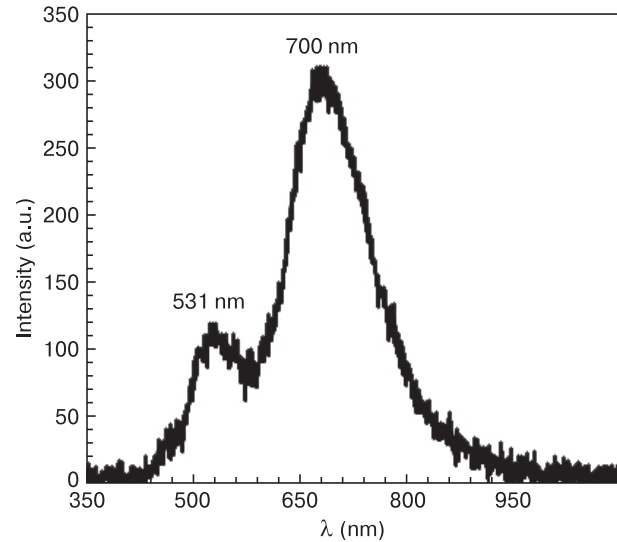


Figure 9. Photoluminescence spectra of LB films deposited on solid substrates from the resulting suspensions of CdSe nanoparticles after ablation

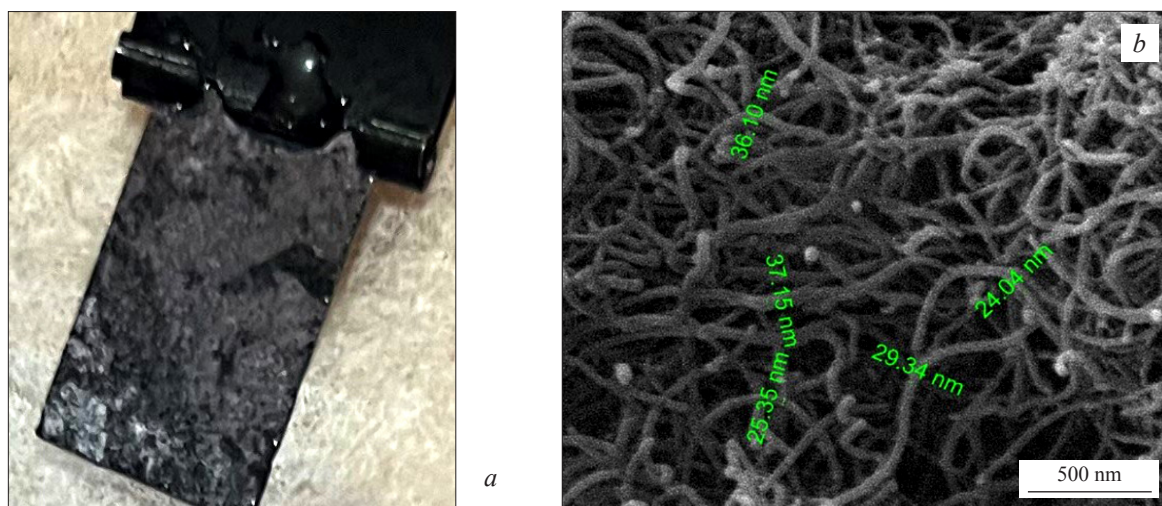


Figure 10. Photo of the sample after deposition of CNT layers over the CdSe film: (a) general view; (b) FESEM image of surface morphology

ed 1 ml of the CNT suspension with 9 ml of chloroform. As a result, we produced a suspension with a concentration of $12 \cdot 10^{-4}$ g/l, that was later used to obtain CNT films. To obtain a more homogenous suspension, we placed it into an ultrasonic homogenizer for 15 min.

We added 200 μ l of the suspension of CNTs in chloroform drop-wise to the surface of aqueous subphase with an area of 243 cm² so that it disturbed evenly across the surface. After 7 min, which is necessary for the complete evaporation of chloroform, we compressed the layers of nanoparticles to the area of 5 cm². After compressing, we transferred the layer to a horizontally oriented substrate using the LS technique as shown in Fig. 3, i.e. to deposit the CNT films onto a solid substrate, we used the same process as for CdSe layers (Fig. 10a).

FESEM analysis showed a homogenous distribution of CNT thin films at solid substrate. Due to the effect of homogenizer, the final suspension was deposited on the water surface and the compression barrier was used to produce a homogenous thin film of CNTs. Fig. 10b shows

randomly selected CNTs with diameter of 24–40 nm, that correspond to the data stated in their technical specifications.

3.3. Conductivity properties of multilayer CNT/CdSe structures

We have engineered a device consisting of CNT/CdSe/p-Si (111) junction diode. The CVC characteristics of the multilayer structures are shown in Fig. 11, where the current density of produced multilayer particles has been measured both in the dark and by the radiation from the “white light” source – Solar Simulator, with a controlled power density of 100 mW/cm². As we can see from Fig. 11, the CVC characteristics within the measured range (–1 to +1 V) change non-linearly. In the dark, CVC change asymmetrically that can be attributed to the lack of symmetry in the multilayered structure. CVC measured after illumination differ from those measured in the dark by appr. 5 times for negative voltage and by 13.5 times for positive voltage values. This can be explained by generation of charge carriers in photosensitive material, i.e. CdSe nanoparticles. At the same time, due to big value of photocurrent, the asymmetry of CVC becomes practically imperceptible (current density at 1 and –1 V equals 1.25 and –1.35 mA/cm², respectively). This suggests that the contribution of photocurrent to the total current flowing through the multilayer structure is decisive/determining.

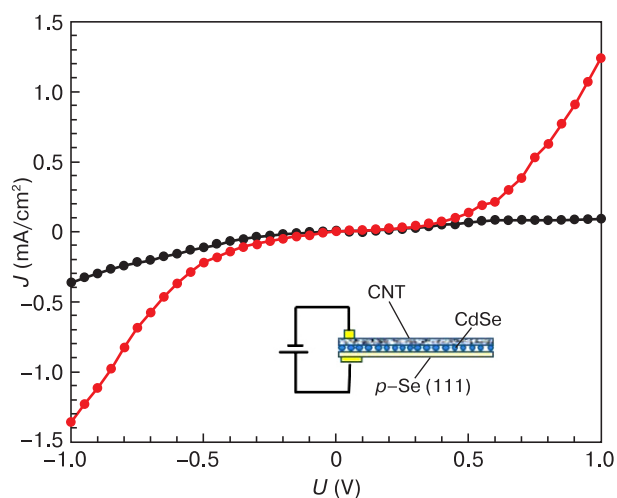


Figure 11. Typical CVC characteristics measured in the dark (black line) and after illumination (red line) on linear scales

4. Conclusions

Thus, we successfully produced photosensitive film structures consisting of CdSe NP layer (active layer) and CNT layer (counter electrode) obtained via Langmuir technique. The upper CNT layer had good conductivity – sufficient for a contact layer. At the same time, the application of the CNT layer did not lead to short-circuiting

or any disruption of the structure of the NP layer located under the CNT layer (as is often the case when applying the upper contact by vacuum methods on single layers of nanoparticles with a thickness of several nanometers).

We successfully used PLAL to produce CdSe NPs for these structures and did not use any chemical synthesis techniques. Thus, we showed the possibility of producing nano-sized CdSe suspensions (with nanoparticle size ranging from 60 to 200 nm) in non-polar solvent without the need of a stabilizing agent.

The photocurrent density exceeds the dark current by 13.5 times at a voltage of 1 V. Thus, the obtained structures containing CdSe NPs and CNTs can be used in photoelectric devices, while elements of the presented production technique can be used to make potential

photosensitive devices (such as photoresistors, photovoltaic equipment, and the like).

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Conflict of interest

The authors declare that they have no conflicts of interest.

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