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## EFFECT OF GAMMA RADIATION ON LUMINESCENCE AND PHOTOCONDUCTIVITY OF MEH-PPV – LEAD SULFIDE NANOCOMPOSITE

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The thin-film properties of polymeric nanocomposite MEH-PPV/PbS (with PbS quantum dots (QD)) and PbS-QD gamma-irradiated by 0.5–24.4 kGy doses have been studied. The film samples for photoluminescence (PL) investigation were prepared from solution using spin-coating technique, and for photoconductivity measuring were made using slot technique. The threshold doses for irreversible sharp PL-degradation of nanocomposite determined by the degradation of PbS-QD were found. The behavior dynamics of MEH-PPV/PbS photoconductivity after irradiation was analyzed. The conduction relaxation observed in the MEH-PPV/PbS was shown to be due to restoration of damages in the conjugate chains of the MEH-PPV polymer. Moreover, the absence of conductivity and photoconductivity relaxations in the colloidal PbS was revealed.

**Keywords:** gamma radiation, photoluminescence, photoconductivity, quantum dot, lead sulfide, conducting polymer

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## ДЕЙСТВИЕ ГАММА-ИЗЛУЧЕНИЯ НА ЛЮМИНЕСЦЕНЦИЮ И ФОТОПРОВОДИМОСТЬ НАНОКОМПОЗИТА МЕН-PPV–СУЛЬФИД СВИНЦА

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Исследованы свойства тонких пленок полимерного нанокompозита MEH-PPV/PbS (полимер MEH-PPV – квантовые точки (КТ) сульфида свинца PbS) и отдельно КТ PbS, подвергнутых гамма-облучению дозами до 24,4 кГр по H<sub>2</sub>O. Образцы пленок для изучения их фотолюминесценции (ФЛ) были приготовлены из раствора с помощью технологии спин-коатинга, а для изучения фотопроводимости – по технологии «на щели».

Найдены пороговые дозы резкой необратимой деградации ФЛ нанокompозитного полимера MEH-PPV/PbS, определяемые деградацией КТ PbS. Исследована динамика поведения фотопроводимости нанокompозита после облучения. Показано, что наблюдаемые процессы релаксации проводимости в нанокompозите связаны с восстановлением разрывов в сопряженных цепях полимера MEH-PPV. Обнаружено отсутствие релаксации проводимости и фотопроводимости в коллоидных КТ PbS.

**Ключевые слова:** гамма-излучение, фотолюминесценция, фотопроводимость, квантовая точка, сульфид свинца, проводящий полимер

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## Introduction

Optoelectronic devices, in particular, those based on thin-film polymer nanocomposites, are widely used in diverse industries [1]. Nanocomposite structures with the required parameters can be synthesized by modifying the polymer matrix with different components [2]. A high-molecular compound as a matrix should actually have the characteristics allowing to obtain the nanocomposite with the given properties [3]. The conductive polymer МЕН-PPV has good electrical characteristics, such as conductivity and charge carrier mobility and can be used to fabricate electroluminescent light-emitting structures [4]. Materials with tunable properties can be made by adding semiconductor quantum dots (QDs) as inorganic inclusions [5]. For example, materials produced with lead sulfide (PbS) QDs have found numerous practical applications in optical communications involving near infrared spectroscopy (NIRS); in particular, NIRS probes are used in agribusiness, pharmaceuticals and biosciences [6]. PbS quantum dots are probably one of the most promising dopants for efficient absorption of solar energy in different types of solar cells, combining simplicity and low production costs, making it possible to synthesize nanocomposites of a wide spectral range [7].

QD-based solar cells are not commercially available because degradation of QD properties poses a major challenge that has yet to be overcome [1]. Such materials are unstable mostly due to oxidative degradation of QDs in oxygen- or moisture-containing environments. Poor conditions for synthesizing QDs or for fabricating photovoltaic devices based on these QDs in turn deteriorate the optical and electronic properties of finished products [8, 9]. While several comprehensive reviews have been dedicated to the effect of oxidation on PbS QDs [10], very few studies have discussed the effect that gamma radiation has on QDs and on QD-based nanocomposite materials. Stability of gamma-irradiated materials is, without doubt, one of the key issues in

research on lead sulphide nanocrystals, which are among the most promising colloidal materials for creating photodetectors of electromagnetic radiation in the near IR region.

This study continues our previous investigations [13], refining the threshold doses for sharp degradation of the optical characteristics of QDs and tracing the dynamics of photoluminescence (PL) spectra for four weeks (at weekly intervals) after irradiation. We have also considered the effect of different doses of gamma irradiation on the electrical properties of a polymer/QD nanocomposite for the first time, analyzed the temperature dependences of conductivity and the overall changes in the optical and electrical properties of the polymer/QD nanocomposite.

## Experimental procedure

Commercially available МЕН-PPV powder from Sigma Aldrich with a number-average molecular weight of about 40,000 – 70,000 was used for the experiment. The structural unit of the polymer is (poly[2-methoxy-5-(2-ethylhexyloxy)-1,4-phenylenevinylene]) whose chemical formula is  $(C_{18}H_{28}O_2)_n$ . PbS QDs were synthesized by the method identical to that used in [11] from an organometallic solution based on lead oxide (PbO) dissolved in oleic acid ( $C_{18}H_{34}O_2$ ). Bis-trimethylsilyl sulfide in trioctylphosphine was introduced into this solution. The given PbS QDs allow to synthesize nanocomposites with tunable spectral parameters in the range from 1000 to 1600 nm [11].

Film samples with a thickness of about 1  $\mu\text{m}$  deposited on glass substrates were prepared for studying the photoluminescent properties of the МЕН-PPV/PbS nanocomposite. These samples were obtained from solution by spin-coating.

Samples for studying the electrical properties were fabricated by the slot technology: a thin (about 10  $\mu\text{m}$ ) layer of electrically conductive material (we used the Contactol conductive adhesive on silver) was applied to a dielectric (we used glass) substrate, followed by drying for 24 h.



Contactol is a low-friction composite containing various synthetic resins as binders and fine (.999) silver powder serving as conductive filler. The adhesive has a specific volume resistivity of about  $0.01 \Omega \cdot \text{cm}^3$ . A through slot of the order of  $10 \mu\text{m}$ , filled with an excess of the test substance (nanocomposite solution), was formed in the conductive layer. The solution was prepared by mechanical mixing of the MEH-PPV powder (weighing 0.1 g), followed by 15-minute treatment in an ultrasonic bath and then adding toluene in a volume of 0.5 ml. The resulting solution was mechanically mixed with the prepared colloidal solution of PbS QDs and treated in the ultrasonic bath for 10 min.

PL spectra of the samples provide data on the electronic structure of the materials and allow to analyze the migration of photoexcited excitons. Recording the optical transmission of samples is limited due to changes in the optical characteristics of the substrates exposed to light, causing different defects to evolve, including ionization of the material; at the same time, presence of ions in the substrates leads to attachment or detachment of electrons, and this in turn changes or deteriorates useful optical properties.

PL spectra of the samples were recorded using the Horiba Jobin Yvon automated system comprising a FHR 640 monochromator with a 1200 gr/mm diffraction grating and a Symphony II 1024\*256 Cryogenic Open-Electrode CCD detector with the necessary optical filters. Photoluminescence of the samples was excited by a semiconductor laser with a wavelength  $\lambda = 405 \text{ nm}$ .

The electrical properties (current-voltage characteristics and photoconductivity) were studied with a setup consisting of a KEITHLEY 2601A source meter with an integrated Test Script Processor combined with a laboratory probe station. The measurements were carried out in the temperature range from 77 K (boiling point of liquid nitrogen) to 300 K (room temperature). For photoconductivity measurements, the samples were exposed to radiation from a 100 W halogen lamp (with a continuous spectrum close to blackbody spectrum).

A setup consisting of 84 tubes filled with  $^{137}\text{Cs}$  powder was used as a source of gamma radiation. This  $^{137}\text{Cs}$  isotope continuously emits gamma rays with an energy of 661 keV. Irradiation was carried out in a quasi-closed chamber (atmospheric oxygen and moisture were not pumped out of it). For this reason, the medium ionized and had a

negative effect on the experimental samples. As a result of gamma irradiation, the experimental samples received the following total exposure doses, kGy: 0; 0.5; 1.0; 1.5; 3.1; 6.1; 12.2 and 24.4 (with respect to  $\text{H}_2\text{O}$ ). The dynamics of the changes in PL spectra after irradiation was monitored for four weeks (with weekly intervals). After irradiation, the samples were stored under normal conditions (without using any special equipment). All measurements were also performed under standard conditions.

### Experimental results and discussion

Ionizing radiation can change the properties of nanocomposites. Gamma radiation has a high penetrating power, interacting with matter by electronic excitation, electron ionization, and production of electron-hole pairs [12].

**Effect of radiation on PL spectra.** We have established in our previous study [13] that PL of the given nanocomposites is generally characterized by photoluminescence of quantum dots (Fig. 1). This means that the effect of gamma irradiation on PL of nanocomposites can be regarded solely as the effect of gamma irradiation on PL of quantum dots.

Changes in the PL spectrum of the conductive polymer as a result of its incorporation into the nanocomposite are due to the fact that charge carriers produced in the polymer matrix are trapped in QDs and recombine with emission of infrared rays. Changes in the PL spectrum of a conductive polymer induced by gamma radiation may be associated with structural changes in the polymer (Fig. 2). The reasons for the PL drop and different competing processes occurring in a conductive polymer exposed to gamma radiation were discussed in detail in [13].

Analyzing the experimental data and the results obtained in [13], we can conclude that the threshold exposure dose is identical to that for PbS QDs; it lies in the range from 12.2 to 18.4 kGy with respect to  $\text{H}_2\text{O}$ . The PL emission peak virtually does not shift with a sharp drop in PL intensity after the threshold exposure dose. Notably, a monotonic decrease in PL intensity was observed in [14, 15] with an increase in the radiation dose rather than the threshold dependence we have observed in the experimental samples. It was argued in [14] that gamma irradiation reduces the size of QT micelles and of the organic molecules surrounding them, and this change in size depends on the conditions in which the

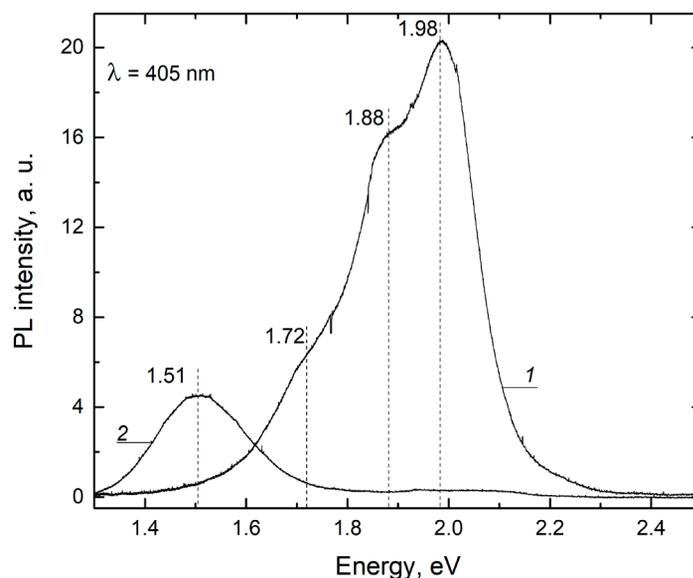


Fig. 1. Photoluminescence spectra for MEH-PPV (1) and PbS (2) QDs  
Laser wavelength was 405 nm

specific QDs were obtained. A decrease in the size of QDs was established depending on the level of micelle hydration, leading the authors to conclude that gamma irradiation can be used to control the size of CdS QDs. This is inconsistent with our data for PbS ODs. PL of colloidal CdSe/

ZnS QDs was shown to depend on the dose of gamma irradiation in [15]; poor radiation hardness of the given QDs was also discovered in that study. Analysis of perovskite CsPbBr<sub>3</sub> QDs also confirmed the dependence on the exposure dose. Such ODs exhibit much better radiation hard-

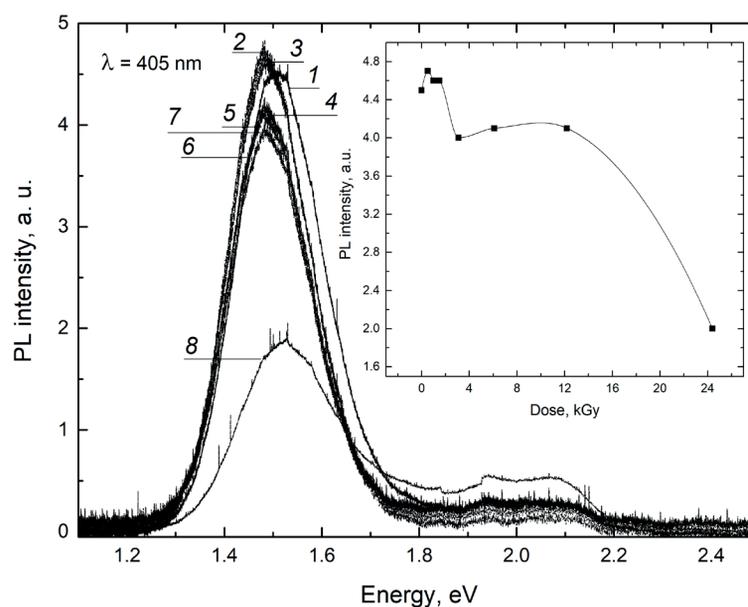


Fig. 2. PL spectra for the samples of the MEH-PPV/PbS nanocomposite, non-irradiated (1) and gamma-irradiated (2–8); the inset shows the dependence of maximum PL intensity of the sample on the total exposure dose  $D$  of gamma irradiation;  
 $D$ , kGy: 0.51 (2), 1.00 (3), 1.5 (4), 3.1 (5), 6.1 (6), 12.2 (7), 24.4 (8) (with respect to H<sub>2</sub>O).  
Laser wavelength was 405 nm

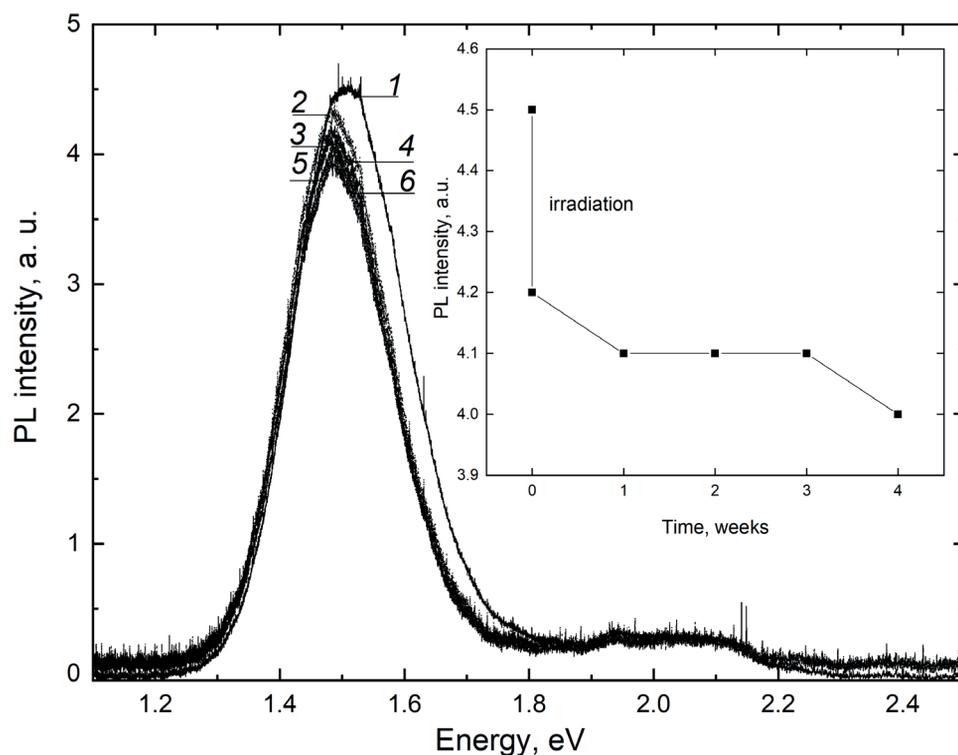


Fig. 3. PL spectra for samples of the MEH-PPV/PbS nanocomposite, non-irradiated sample (1) and gamma-irradiated with 12.2 kGy (2–6); the inset shows the dependence of the maximum PL intensity of the sample on time  $t$  after irradiation

Time  $t$ , weeks: 0 (2), 1 (3), 2 (4), 3 (5), 4 (6)

ness against gamma rays than the commercially available samples of CdSe/ZnS QDs. CsPbBr<sub>3</sub> quantum dots showed greater stability to gamma irradiation than the PbS QDs considered (irradiated in solutions).

Weak degradation with time was observed in the PL spectra recorded after irradiation with 12.2 kGy and at different times after the irradiated samples were left at room temperature (Fig. 3). This is due to the post-radiation effect of gamma rays on the organic molecules surrounding the QDs. In combination with the oxygen-containing environment where the samples were stored, this leads to nonradiative recombination centers and electric charge trapping centers forming in the nanocomposites.

**Effect of radiation on photoconductivity.** The dependences of photoconductivity versus exposure dose for the given films (Fig. 4) complement the data obtained from the PL spectra.

A sharp increase in photoconductivity is observed in the initial segment of the curve for the nanocomposite film (Fig. 4, *a*); in contrast with the corresponding curve for films of pure QDs (Fig. 4, *b*), this increase is associated with

the processes occurring in the polymer, namely, with crosslinking of polymer chains. This result agrees with the results obtained by analyzing the PL spectra. As the dose of gamma irradiation increases, conformational defects evolve in polymer chains, which leads to gradual decrease in photoconductivity. The actual degradation of QDs at doses in the range of 6.1–12.2 kGy causes a sharp drop in photoconductivity, which also agrees with the results obtained in the study of PL spectra.

A slight decrease in photoconductivity in PbS QD films in the initial segment of the photoconductivity curve might be due to recombination centers induced by gamma irradiation and trapping of the charge by the molecules surrounding the QDs. In the first case, recombination leads to a decrease in the concentration of excited carriers. In the second case, charge trapping leads to additional increase in the random potential and, as a result, to a decrease in the hopping conductivity of charge carriers, and, therefore, their partial Anderson localization.

The described effect is also observed in the nanocomposite, with the transfer of holes mainly

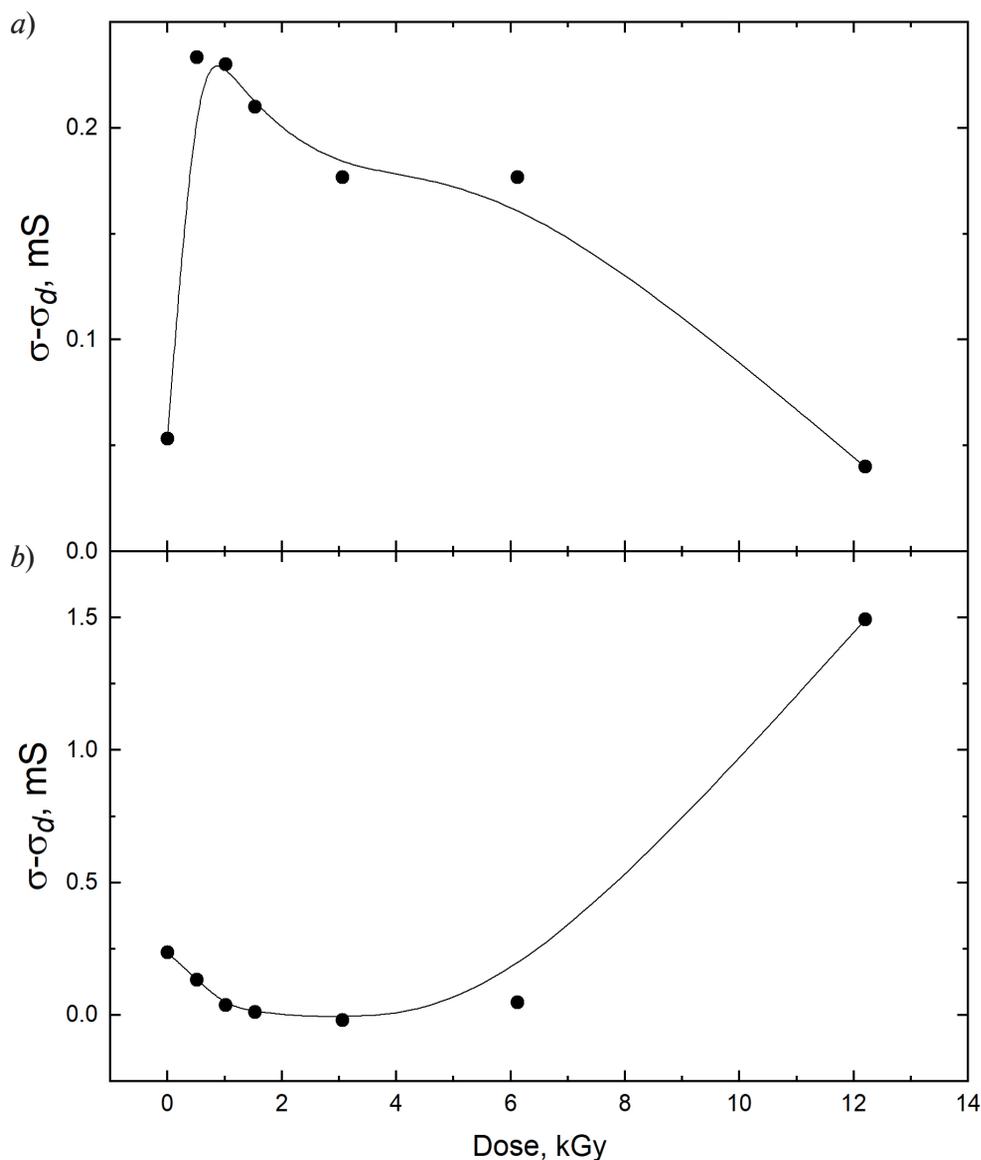


Fig. 4. Dependences of photoconductivity for thin films of MEH-PPV/PbS (a) and PbS (b) on the dose of exposure to gamma irradiation  
The films were synthesized using the slot technology, illuminated with a 100 W halogen lamp;  
 $\sigma_d$  is the dark conductivity

occurring in the polymer matrix, and the transfer of electrons in the QD chains. This means that photoconductivity of the nanocomposite substantially depends on the processes occurring both in the polymer and in the QDs.

We plotted the changes in photoconductivity for films that were first exposed to a large dose of gamma irradiation, and subsequently gamma-irradiated with a total exposure dose of 12.2 kGy (these graphs are not given in this paper). Relaxation of either electrical conductivity or photoconductivity was not observed after irradiation by

this scheme in films of colloidal PbS QDs.

Analyzing the dynamics of photoconductivity relaxation after irradiation with gamma rays (the graphs are not given in this paper), we established partial recovery of photoconductivity in nanocomposite films. We assume that this effect is due to crosslinking of ruptured conjugated chains of the MEH-PPV polymer.

Fig. 5 shows the temperature dependences of electrical conductivity and the effect of illumination on electrical conductivity for polymer, QD and nanocomposite films. Evidently, electri-

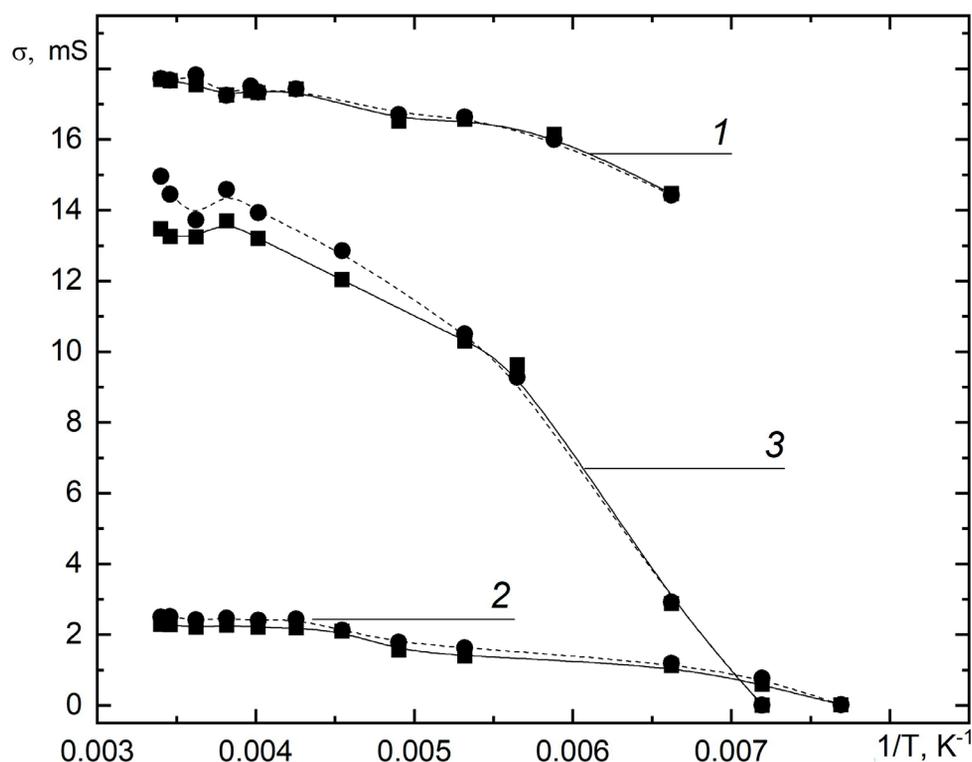


Fig. 5. Temperature dependences of the dark (solid line) and light (dashed line) conductivities for MEH-PPV (1), PbS (2) and MEH-PPV/PbS (3) films

cal conductivity increases in all samples with an increase in temperature. This is due both to an increase in charge carrier concentration and an increase in carrier mobility concordant with an increase in the number of phonons. The difference between the conductivity of QD and nanocomposite films and the conductivity of polymer films is due to saturation of polymer conductivity at temperatures close to room temperature. It can be also seen from Fig. 5 that there is practically no difference in “dark” and “light” conductivities in polymer films. This is because the recombination rate of excitons with photon emission is higher than their decay rate with production of free electrons and holes. It is known that hole mobility is higher than electron mobility in the given polymer, and that current flow in the sample is mainly due to hole conductivity. At the same time, conductivity has a more complex mechanism in nanocomposites: it is associated both with hole conductivity of the polymer matrix and with hopping conductivity along the chains of PbS quantum dots. In contrast, conductivity is exclusively hopping along the lattice of an infinite QD cluster in films of PbS QDs. As evident from the PL spectra of the nanocomposite (see Fig. 2),

QDs effectively trap electrons and holes from the polymer, contributing to decay of excited excitons; as a result, hopping conductivity along QD chains can make a significant contribution to the conductivity of the film. The difference in conductivities of the polymer in the dark and in the light is insignificant and practically does not depend on temperature, while photoconductivity of QD and nanocomposite films increases with increasing temperature (Fig. 6).

This result indicates a significant distinction between the photoconductivity of the nanocomposite and the photoconductivity of a conventional semiconductor photoresistor, where this value usually decreases with increasing temperature due to an increase in the dark concentration of carriers.

The increase in the photoconductivities of the nanocomposite and QD films is due to increased hopping mobility of charge carriers with increasing temperature. Similar behavior exhibited by photoconductivity of QD and nanocomposite films with temperature points to the same nature of the process, and this means that photoconductivity of the nanocomposite is mainly governed by conductivity along QD chains under illumination.

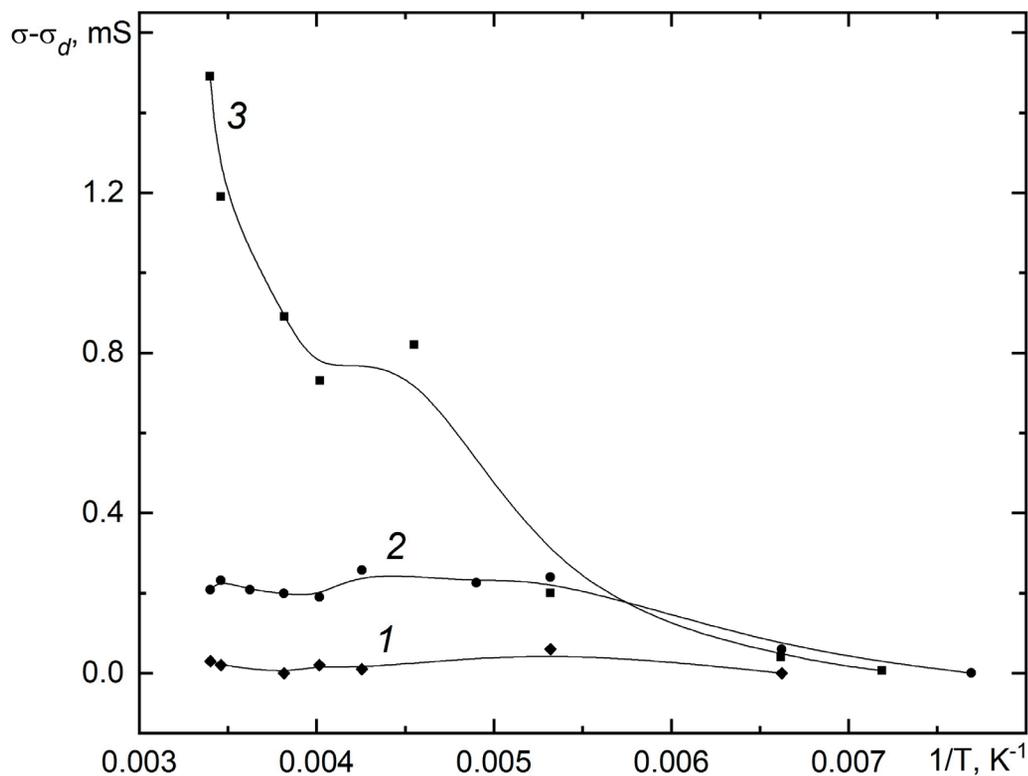


Fig. 6. Temperature dependences of photoconductivity for thin films of MEH-PPV (1), PbS (2), MEH-PPV/PbS (3)

However, photoconductivity of the nanocomposite exceeds that of QDs, due to increased probability of exciton decay (with the production of free electrons and holes) at the interface between the QDs and polymer chains. Mathematical analysis of the graphs has revealed that the temperature dependence of electrical conductivity is generally consistent with Mott's law:

$$\sigma = \sigma_0 \exp[-(T_0/T)^{1/(1+d)}],$$

where  $T$  is the absolute temperature;  $d$  is the dimension of the space of the conductive system;  $\sigma_0$ ,  $T_0$  are constants.

However, the system is peculiar in that the dimension  $d$  is not an integer, since the value  $1/(1+d)$  is not equal to  $1/4$ ,  $1/3$  or  $1/2$ .

### Conclusion

Our experimental studies have yielded the following results.

The threshold exposure dose for a sharp drop in photoluminescence (PL) of the MEH-PPV/

PbS nanocomposite depends solely on the radiation hardness of PbS QDs and lies in the range from 12.2 to 18.4 kGy (with respect to  $H_2O$ ). While the intensity of the PL spectra of the samples does not decrease immediately after irradiation, weak degradation with time has been observed. These effects are due to the effect of gamma irradiation and the post-radiation effect of gamma rays on the organic molecules surrounding the QDs.

At least two competing processes occur in the polymer matrix: crosslinking of polymer chains and defects evolving in polymer chains (polymer chain conformation), indicated by nonlinear behavior of the dependence of photoconductivity of the thin films manufactured by the slot technology on the exposure dose.

Conductivity is partially recovered by regeneration of ruptured conjugated polymer chains. In addition, we have found by analyzing the temperature dependence of electrical conductivity that the Mott law is satisfied both for films of the nanocomposite and for films of the nanocomposite components.



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