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# Photoconductive, photovoltaic and information properties of new carbazole-based oligomeric film compositions doped with 2,2-diphenyl-1-picrylhydrazyl

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## ABSTRACT

The novel photoconducting magneto-sensitive thin film structures based on carbazole-containing oligomer doped by stable organic radical 2,2-diphenyl-1-picrylhydrazyl as the photoconductivity sensitizer have been prepared. Their spectral, photoconductive, photovoltaic and information properties were investigated. It was shown, that the investigated composites exhibit photoconductivity and photovoltaic properties under illumination by light from the respective stable radical absorption region. The features of the photoconductive and photovoltaic properties of the investigated composites, as well as the possible mechanisms of the photovoltaic effect and charge carriers photogeneration were discussed. It was shown that the prepared films can be used as reversible holographic recording media.

## KEYWORDS


oligomer composite films; 2,2-diphenyl-1-picrylhydrazyl; photoconductivity; photovoltaic effect; reversible holographic recording media; photothermoplastic; holographic recording

## 1. Introduction

Photoconductive polymer composite films (PCFs) doped with dyes, organic electron donors or acceptors are commonly used in information recording, photorefractive, and electroluminescence media, as well as in photoelectric solar energy converters [1–11]. Considerable progress has been achieved in controlling the photophysical and electrophysical properties of PCFs by varying the chemical structure of the colored photoconductivity sensitizer and polymer. Thus, there is theoretical and practical interest in creating new photoactive media derived from organic photosensitizer-doped colored PCFs.

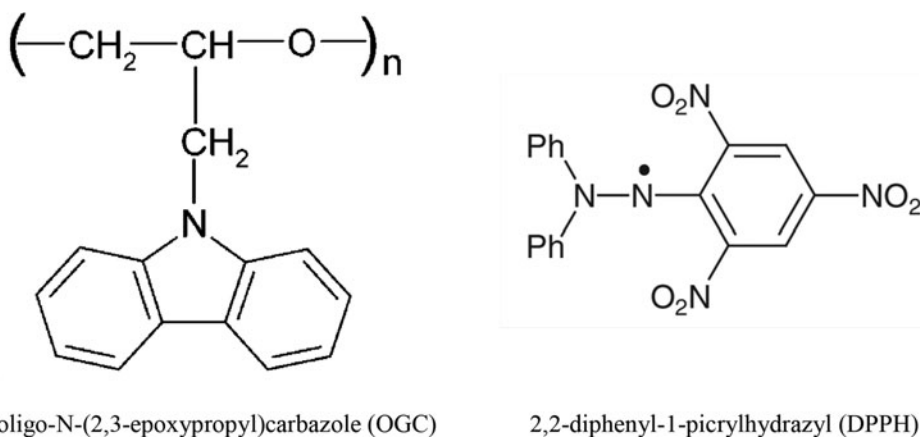
Oligo-N-(2,3-epoxypropyl)carbazole (OGC) (and its analogues) is a well-known photoconductive oligomer matrix for dye-sensitized electrographic and reversible photothermoplastic (PTP) holographic recording media (HRM) [12–18]. On the other hand, the well-known stable free organic radical 2,2-diphenyl-1-picrylhydrazyl (DPPH) is widely used as a standard substrate for ESR spectroscopy [19, 20], organic paramagnetic [21–22], and as a radical scavenger [23]. However, the DPPH is also used as the single electron type oxidizer and oxidation catalysts, in particular, in organic synthesis [24–29]. It is also well known, that the pure solid DPPH exhibits semiconductive and photoconductive properties [26–27].

In this connection, it is reasonable to suppose that OGC-based PCFs doped by DPPH radical as the photoconductivity sensitizer can be used as magneto-sensitive media for photonic applications, in particular as reversible HRM for the PTP type optical holography. The aim

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**Scheme 1.** Molecular structures of studied photoconductive oligomer and DPPH radical.

of this work is to develop new photosensitive thin film structures based on OGC sensitized by high concentration of the DPPH radical and to study the possibility of their application as reversible HRM, in particular as photothermoplastic HRM, as well as to investigate their photoconductive, photovoltaic and information properties.

## 2. Experimental

### 2.1. Materials

Photoconductive oligo-N-(2,3-epoxypropyl)carbazole (OGC) was used as a oligomer-matrix ( $T_{\text{soft}} \approx 80^\circ\text{C}$ ) and the stable organic radical 2,2-diphenyl-1-picrylhydrazyl (DPPH) was used as a photosensitive component (sensitizer of photoconductivity) of the investigated PCFs (scheme 1).

### 2.2. Instrumentation and samples preparation

The samples were prepared as structures with a free surface [glass substrate – PCF], [glass substrate – transparent electroconducting ITO ( $\text{SnO}_2$ :  $\text{In}_2\text{O}_3$ ) layer – PCF], and as the “symmetrical type” sandwich structures [glass – ITO – PCF – ITO – glass]. Concentrations of DPPH in all films was 10 mass %. The polymeric composite films were formed by pouring-evaporating of toluene DPPH - oligomer solutions at the substrates followed by drying at  $80^\circ\text{C}$  for 2 days. The PCF films thickness, measured by MII-4 interference microscope, was  $L \sim 1.0\text{--}2.0\ \mu\text{m}$ .

The prepared samples of oligomer DPPH-doped films were characterized by measuring the spectral, photoconductive, photovoltaic and information characteristics.

The optical density (D) spectra of investigated films were measured in free surface samples [glass substrate – PCF] in the wavelength range 400–900 nm. The absorption spectra of the prepared films were measured using a spectrophotometer (Varian Cary 50).

The photoconductive properties of investigated PCFs were studied by a surface potential photodecay method in the electrophotographic mode with the use of the corona discharge at a positive charge of the surface [12, 30–35].

Photovoltage (surface) was measured by the modified Kelvin method [34–38] in the samples with a free surface (under irradiation of the transparent ITO-electrode side). The additional photovoltaic measurements for the investigated PCFs were carried out in the

symmetrical type (glass – ITO – PCF – ITO – glass) sandwich structure samples using high resistance voltmeter based on CA3130 voltage follower scheme [35].

As a photoexcitation light source for the photovoltaic and electrophotographic mode measurements, we used a white (3 mW) semiconductor light-emitting diode (508H245WC-2.2-MD). The light intensity ( $I$ ) was varied by neutral light filters in the range of 1–50 W/m<sup>2</sup>. The irradiation was performed at the side of the conducting ITO layer.

The kinetics of photovoltaic response ( $V_{PH}$ ) and photodecay of corona-charged samples surface potential were measured using a storage oscilloscope Fluke 124 and a storage USB-oscilloscope BM 8020.

All these measurements were carried out at room temperature.

The PCFs informational characteristics measurements were carried out using known holograms of a planar wavefront recording methods [7, 9]. A semiconductor laser with the light emission wavelength  $\lambda = 532$  nm and an irradiation intensity of  $I = 35$  mW was used as a coherent light source for registration of the respective planar-wavefront holograms. In all cases, the respective holograms were recorded by the photothermoplastic method (PTP holograms) according to previously described procedure [7, 17–18], as shown in Figure 4b.

In these experiments, the controlled parameter was the diffraction efficiency  $\eta$  of the recorded planar-wavefront hologram determined in the  $-1$  diffraction order. It was measured in the corresponding diffraction order during the hologram development. Diffraction efficiency  $\eta$  was defined as the intensity ratio of a diffracted beam to an incident beam as follows:

$$\eta = I_{+1}/I_0,$$

where  $I_{+1}$  (or  $I_{-1} = I_{+1}$ ) and  $I_0$  are the intensities of a  $+1$  (or  $-1$ ) order (called first order) diffracted beam and a probe transmission beam, respectively. The spatial frequency was  $\omega \sim 800$  mm<sup>-1</sup>. The ratio between intensities of the object and reference beams was 1:1.

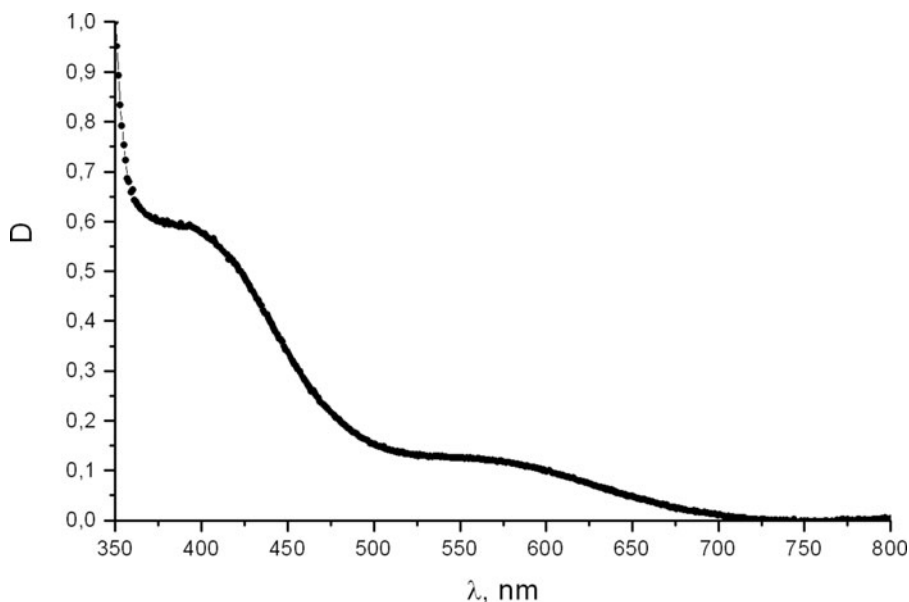
### 3. Results and discussion

The film composites based on photoconductive OGC oligomer doped by high concentration of stable organic radical DPPH have been obtained. The color of the respective dried oligomer films changed immediately from purple to brown, that caused by the fact that the partial dark electron transfers from carbazolyl fragments of OGC to DPPH proceeded in an instant due to the transition of DPPH in the anionic state (DPPH<sup>-</sup>) [24–27]. The films of pure OGC are transparent in the visible and near infrared regions, and they haven't photoconductivity and photovoltaic properties in this area. In the PCFs which contain DPPH additives, the absorption bands (Figure 1) are defined by long-wavelength excitation and relaxation of the excited states of DPPH molecules and its associates as well as DPPH<sup>-</sup> anionic form [8, 10, 24–27].

It was found, that all studied composite films exhibit photoconductivity and photovoltaic effect under the photoexcitation in the DPPH absorption region.

The kinetics of the dark and photoinduced decay of surface potential of investigated corona-charged sample with a free surface PCF [glass substrate – ITO – PCF] in the electrophotographic mode at a positive charge of the surface is presented in Figure 2 (curves 1 and 2, respectively).

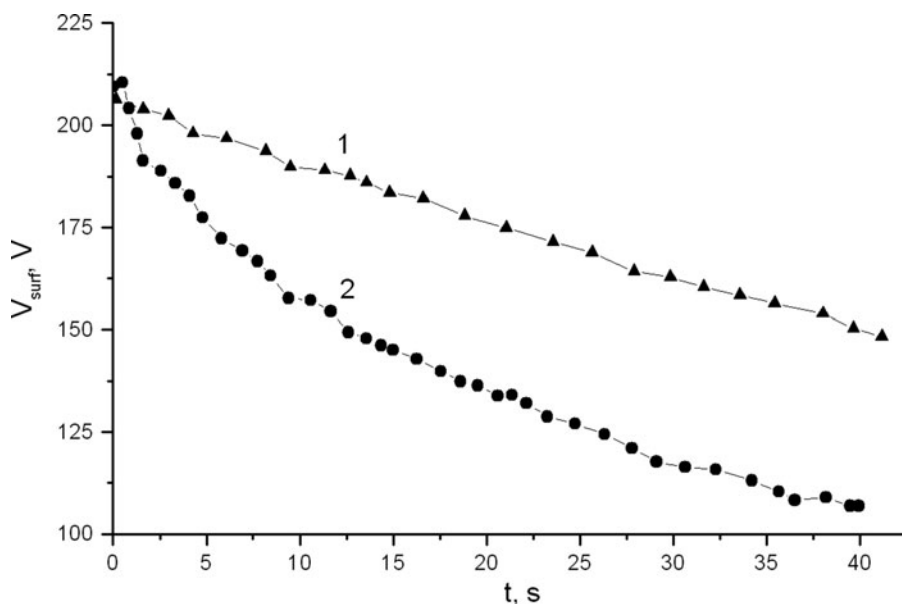
It should be noted, that the dark relaxation of the corona-charged samples surface potential rate ( $dV_{surf}/dt$ )<sub>dark</sub> in the electrophotographic mode is not very high (see Figure 2, curve 1). On the contrary, the photoinduced decay of respective surface potential is relatively fast (curve 2). This condition (low electrical conductivity and high photoconductivity within the spectral



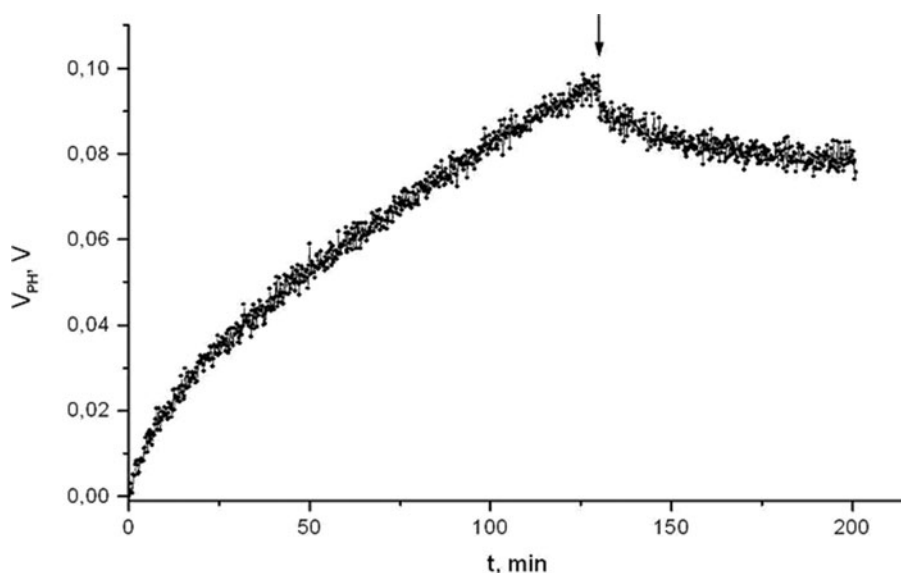
**Figure 1.** Absorption spectrum of oligomer composite film based on OGC doped with 10 mass % DPPH radical.

range of the used laser irradiation) is a necessary general requirement for PTP type HRM [7, 13, 17].

The relatively high dark electric conductivity value of the corona-charged samples (see Figure 2) can apparently be explained by the partial presence of  $\text{DPPH}^-$  anions (see above), which contain mobile weakly bonded excessive electron [24–27] in investigated PCFs.



**Figure 2.** Kinetics of the dark (1) and photodecay (2) of a positive surface potential of the corona-charged sample with a free surface PCF based on OGC + 10 mass % DPPH under white LED light irradiation via ITO-electrode ( $I = 40 \text{ W/m}^2$ ). Experiment is carried out in the electrophotographic mode.



**Figure 3a.** Kinetics of the surface photovoltage growth and relaxation in the sample with a free surface PCF based on OGC doped with 10 mass % DPPH under illumination of the ITO-electrode side of investigated sample by white light-emitting diode irradiation ( $I = 40 \text{ W/m}^2$ ; Ag-based probe material. The time moments of switching off the light illumination are shown by vertical arrows).

Figure 3a demonstrates the main features of photovoltaic response in the investigated PCFs. The growth and relaxation of photovoltaic response  $V_{\text{PH}}$  upon the light being switched on and off, respectively, is characterized by slow kinetics with a relaxation time constant, which is greater than the time constant of  $V_{\text{PH}}$  growth during the PCFs photoexposure. The maximal attainable in these experiments photovoltaic response value  $V_{\text{PH}}$  was  $\sim 0,165 \text{ V}$ .

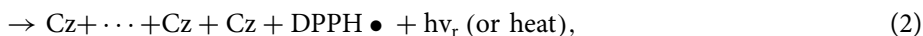
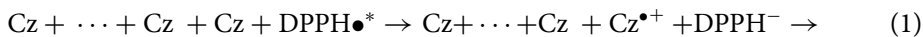
In accordance with the earlier accepted model concepts of photogeneration and transport of charge carriers in amorphous photoconducting PCFs doped with organic species as the photoconductivity sensitizers [1–3, 8, 10, 13, 15, 30, 39–41], the internal photoeffect in these systems is mainly due to the formation of nonequilibrium charge carriers after light absorption by photosensitizer molecules and transport of these carriers in the oligomer matrix. Based on previously developed for photoconductive dye-sensitized amorphous composites [41] model representations, the charge carriers photogeneration in the studied film composites can be associated with two main possible mechanisms.

### I. Extrinsic photogeneration in a DPPH radical in the presence of a donor hole transport molecules (OGC)

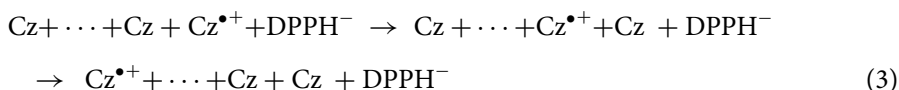
In this case, the photogeneration mechanism in the studied DPPH-doped OGC film composites can be represented as consisting of several steps.

At the first stage of photogeneration, the close (geminate) electron-hole pair (EHP) is formed after photon absorption by a DPPH molecule. The structure of the EHP includes a hole, which corresponds to the cation-radical ( $\text{Cz}^{\bullet+}$ ) of carbazolyl fragment of the oligomer OGC, and the electron remaining in the molecule of the sensitizer (DPPH) after leaving a hole. In simplified form, the unexcited molecule of sensitizer and the molecular sensitizer after electron capture from the carbazolyl unit can be represented as  $\text{DPPH}^{\bullet}$  and  $\text{DPPH}^-$ . Then, the corresponding reaction with electron transfer can be written as (1).

At the second stage of photogeneration, a hole on the carbazolyl fragment of the oligomer ( $\text{Cz}^{\bullet+}$ ) recombines with an electron in the same  $\text{DPPH}^-$  molecule in which it was formed (geminate recombination according to the scheme (2)):



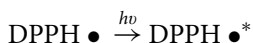
or removes from the electron through the transitions between neighboring Cz groups (EHP dissociation into free charge carriers (3)):



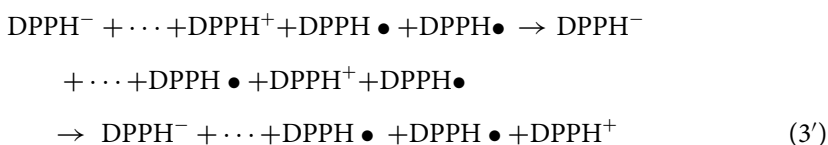
As a result of EHP dissociation (scheme 3), the electrostatic interaction between the hole and electron localized in  $\text{DPPH}^-$  anion weakens. At the recombination stage (scheme 2), the electron of an anion form of the sensitizer ( $\text{DPPH}^-$ ) transfers to  $\text{Cz}^{\bullet+}$ , and after relaxation of the excited state, the radiative energy ( $h\nu_r$ ) or nonradiative heat is released. Then the  $\text{DPPH} \bullet$  molecule can again absorb a photon and participate in EHP photogeneration. The probability of EHP dissociation increases with external electrical field strength  $E$  and temperature  $T$  [8, 39, 41].

Other possible mechanisms of charge carriers photogeneration also includes analogous stages.

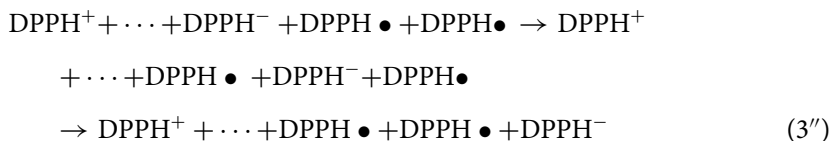
## II. Intrinsic photogeneration in a DPPH:



or

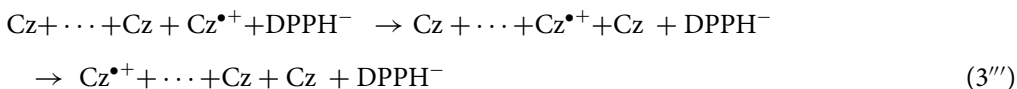
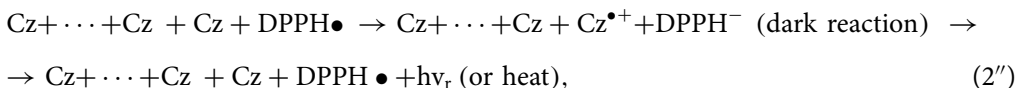
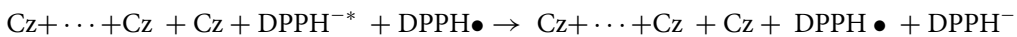
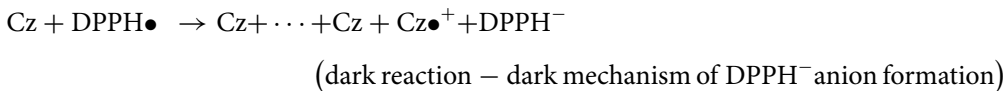


and



Where (1, 1') - geminate electron-hole pairs (EHP) photogeneration, (2, 2') - EHP recombination, (3, 3', 3'') - geminate EHP dissociation.

### III. Analogously, for DPPH<sup>-</sup> anionic form the possible charge carriers photogeneration scheme can be presented as [24–27, 41]:



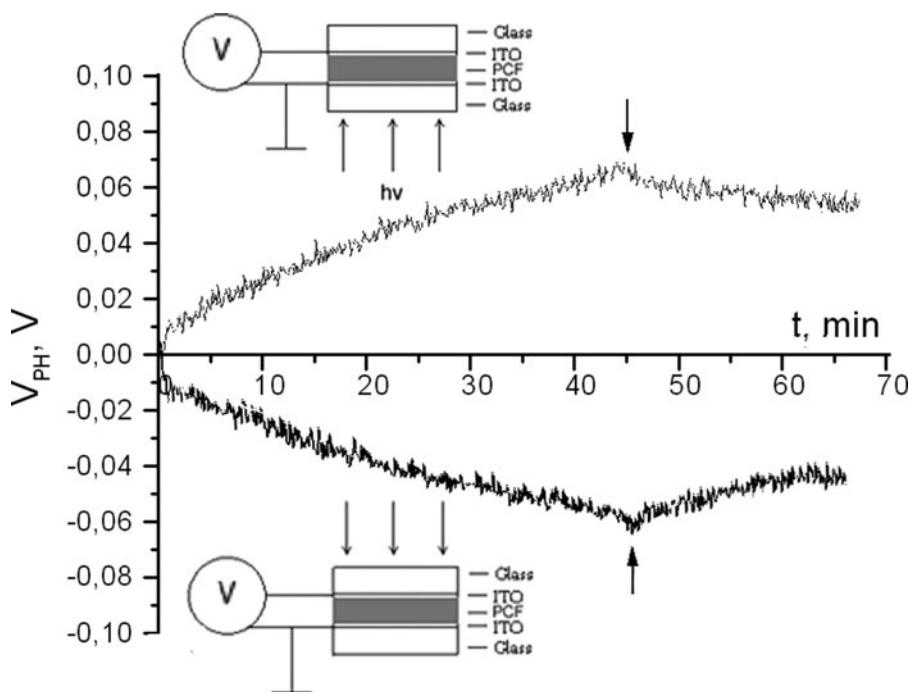
It is suggested that observed in PCFs photovoltaic effect phenomenon is caused by the Dember effect, i.e. discovered effect has diffusion nature. This assumption was based on fact that observed effect appears in the range of strong light-absorption of high concentration dye-doped PCFs. It is well known that the Dember effect can be observed in semiconductors when the electrons and holes (or, in the general case, electrons and holes, and ions of different types) have different mobilities and the light-illumination of the sample leads to a non-uniform distribution of charge carriers [42, 43]. The Dember effect (if the electrons and holes have different mobilities) is caused by the excitation light intensity and photogenerated charge (electron-hole) pairs concentration gradient in the film sample volume, respectively [43].

As a rule, the sign of the non-illuminated sample surface potential (with respect to irradiated surface) becomes the same as the charge sign of the photogenerated current carriers which have larger mobility [42–43]. Under the exciting light irradiation of the different sides of investigated samples the direction of this gradient becomes inverse. Therefore, under these conditions, the sign of the Dember's photovoltage also becomes inverse. To check the above-proposed assumption, the additional photovoltage measurements were carried out for symmetrical sandwich structure samples of investigated PCFs. Figure 3b illustrates the scheme and results of these experiments.

The Figure 3b shows that the sign of the respective photovoltaic response becomes inverse under exciting light illumination of the different sides of sandwiched samples. This fact confirms the correctness of our assumption about diffusion nature of the observed photovoltaic effect [38, 42]. In our systems case the potential of the non-irradiated (dark) surface becomes positive with respect to irradiated surface, because positive charged current carriers have larger mobility. This confirms the hole-type photoconductivity character of investigated PCFs [8, 13, 39–41]. However, it is possible that other photoresponse formation mechanisms were also partially responsible [1, 3, 10, 39, 40].

The relatively slow photovoltaic response growth and very slow dark photovoltage relaxation kinetics character can be explained by an influence of charge carriers capture on deep traps processes and relatively low mobility of non-equilibrium charges in the PCFs [1, 39, 41, 44–47].





**Figure 3b.** Kinetics of the surface photovoltage growth and relaxation in the symmetrical ITO-PCF-ITO sandwiched sample based on OGC doped with 10 mass % of DPPH under illumination of the different sides of sample by white light-emitting diode irradiation ( $I = 40 \text{ W/m}^2$ ), and the scheme of respective photovoltage measurements. The time moments of switching off the light illumination are shown by vertical arrows.

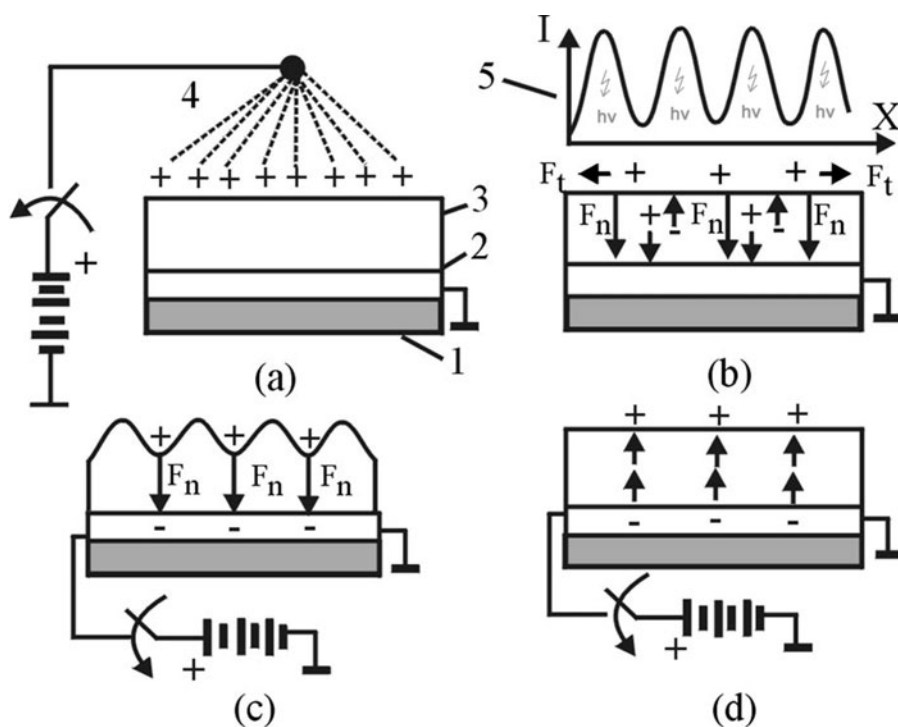
The significantly faster photodecay rate of the corona-charged samples surface potential in the electrophotographic mode experiment (Figure 2, curve 2) than the respective photovoltaic response growth and dark relaxation (Figure 3a) was explained by the greater mobility of non-equilibrium charge carriers in the investigated PFCs and the smaller contribution from their capture in corona electric fields ( $E_{\text{initial}} \sim 10^8 \text{ V/m}$ ). The charge carriers in the photovoltaic effect were much less mobile so that the probability of charge carriers being captured in energy traps was greater. This was related to the fact that charge carrier mobility for most amorphous molecular semiconductors (in particular, for OGC [8, 13, 15, 46]) depends rather strongly on the electric field [1, 8, 13, 39–41, 47].

As it could be expected, investigated PCFs are capable to form holograms of PTP type. The respective PTP holograms of a planar wavefront were registered in the investigated oligomer media. Figure 4 shows results (the respective diffraction pattern obtained by 532 nm laser beams) (Figure 4a) and the scheme (Figure 4b) of these PTP holographic recording experiments.

The maximal attainable in these experiments diffraction efficiency value was  $\sim 1.5\%$ . The possibility of the PTP surface relief grating formation is due to the following general requirements for PTP HRM: (i) respectively low dark electrical conductivity, (ii) high photoconductivity within the used spectral range of irradiation, and (iii) easy HRM deformation above the PCF softening temperature  $T_{\text{soft}}$  [7]. The investigated oligomeric films can be used again to record a new hologram after erasing the previous hologram and after the film cools [7].



**Figure 4a.** The reconstructed photothermoplastic hologram of a planar wavefront image recorded in the composite sample with a free film surface (glass substrate – ITO – composite film) based on OGC doped with 10 mass % DPPH ( $L \sim 1 \mu\text{m}$ ) under sample illumination by green semiconductor laser ( $I = 15 \text{ mW}$ ,  $t_{\text{exposure}} = 5 \text{ s}$ ) irradiation. Hologram was recorded at a 1:1 intensity ratio of the reference to the object beam. The spatial frequency of hologram recording was  $\sim 800 \text{ mm}^{-1}$ . The diffraction efficiency value  $\eta$  was  $\sim 0,25\%$  (the corresponding diffraction orders are shown by vertical arrows).



**Figure 4b.** Scheme of photothermoplastic holographic recording: a - charging of the surface of HRM in corona discharge; b - light exposure; c - latent image development; d - erasing of the recorded hologram; 1 - glass substrate; 2 - conductive layer  $\text{SnO}_2:\text{In}_2\text{O}_3$  (ITO); 3 - photoconductive thermoplastic film; 4 - corona discharge; 5 - modulated light.

## Conclusions

New photoconductive oligomeric thin film structures ( $L \sim 1 \mu\text{m}$ ) based on carbazoyl-containing oligomer OGC doped by high concentration of the stable organic radical 2,2-diphenyl-1-picrylhydrazyl (DPPH) have been obtained. Their spectral, electric, photoconductive, photovoltaic and information properties were investigated. It was shown, that all

investigated film composites exhibit photoconductive and photovoltaic properties under irradiation by light from the radical-sensitizer DPPH absorption region. The diffusion nature of observed photovoltaic response as well as the hole-type photoconductivity character of such compositions was proposed. It was suggested that the slow kinetics of the photovoltaic effect was determined mainly by the poor mobility of photogenerated charge carriers in the PCF and the important role of the mobile photogenerated charge carriers capture on traps in the PCF. The significantly faster character of the corona-charged PCFs surface potential photoinduced decay in comparison with the respective characteristics of the photovoltaic response in these PCF could be explained by the greater mobility of non-equilibrium charge carriers and the smaller influence of the non-equilibrium mobile charge carriers capture in the electrophotographic mode experiment electric fields of the order of  $\sim 10^8$  V/m in contrast with the photovoltaic measurement practically zero electric fields conditions.

It was shown that the prepared PCFs could be used to develop new reversible magneto-sensitive HRM for PTP optical type holography, as well as the media for other photonic applications. Furthermore, these PCF could possibly be used as recording media for optical information storage and molecular switches because of the significant growth and especially relaxation times of the photovoltaic response that were manifested as a long-term memory after preliminary irradiation of the films.

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