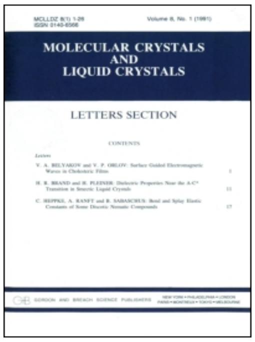

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Photoconductive, photovoltaic and information properties of new photochromic carbazole-based oligomeric film compositions doped with azo-dye

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ABSTRACT
The novel oligomeric thin film structures with photochromic properties were obtained. They based on carbazole-containing oligomer as a matrix and azo-dye 4-((4-nitrophenyl)diazenyl)phenol as a photosensitive additive. Spectral, photoconductive, photovoltaic and information properties of films were investigated. It was shown, that all investigated film composites exhibit photoconductive and photovoltaic properties under light irradiation correspond to the azo-dye absorption region. The features of the photoconductive and photovoltaic properties of the investigated compositions, as well as the possible mechanisms of the photovoltaic effect and charge carriers photogeneration, were discussed. It was shown that the prepared composites can be used as reversible recording media.

KEYWORDS
polymeric composite films; azo-dye; photoconductivity; photovoltaic effect; reversible holographic recording media; polarization holography; photothermoplastic holographic recording

1. Introduction

Polymer composite films (PCFs) doped with organic dyes 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 dyes and polymers. Thus, there is theoretical and practical interest in development of new photoactive media derived from organic dye-doped colored PCFs.

Oligo-9-(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 several azo dyes, azo polymers and azo dye-doped polymeric composites are widely used as optical recording media, in particular as HRM. Such HRM can be used for the polarization holography with information properties controlled by an external electric fields [9, 19–23], as organic photochromic materials [10, 20, 23, 24], promising materials for nonlinear optics applications [10, 24] and optoelectronic devices components [10, 19, 23–25].

Hence, it is reasonable to suppose that OGC-based PCF doped by appropriate azo-type sensitizers can be used as reversible HRM for both the polarization and PTP optical holography.

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The aim of this work is to develop new photosensitive thin film structures based on OGC sensitized by high concentration of the photochromic azo dye 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

4-((4-nitrophenyl)diazenyl)phenol. To a solution of 3.45 g (0.025 mol) of 4-nitroaminobenzene in 30 ml of water 9 ml of concentrated HCl was added. The solution was cooled to about 0°C in an ice bath and vigorously stirred for 30 min. Then solution of 1.75 g (0.025 mol) of sodium nitrite in 10 ml of water was added drop-wisely to the resulting solution throughout 15 min and stirred for another 30 min to yield a clear orange solution. The temperature was maintained between 0 and 5°C. The solution of 2.35 g (0.025 mol) of phenol and 1.4 g (0.25 mol) of KOH in 20 ml of water was added with vigorous stirring, and the color changed to dark red. This solution was stirred for 30 min. Then 15% KOH solution should be added until pH become 7.5 and the temperature was slowly raised to ambient. The product was separated by filtration as orange powder, washed several times with water, recrystallized from ethanol:water (1:1) and dried under vacuum at 60°C for 6 h. Yield: 5.49 g (90.37%). M.p.: 207–208°C. ^{1}H-NMR (400 MHz, DMSO-d_6) δ/ppm10.33 (s, 1H, —OH),: 8.36 (d, 2H, Ar), 7.99 (d, 2H, Ar), 7.84 (d, 2H, Ar), 6.94 (d, 2H, Ar).

2.2. Samples preparation

Photoconductive oligomer oligo-9-(2,3-epoxypropyl)carbazole (OGC) was used as a polymer-matrix ($T_{soft} \approx 80°C$) and the photochromic azo-dye 4-((4-nitrophenyl)diazenyl) phenol (Azo) was used as a photosensitive component (sensitizer of photoconductivity) of the investigated PCFs (Scheme 1).

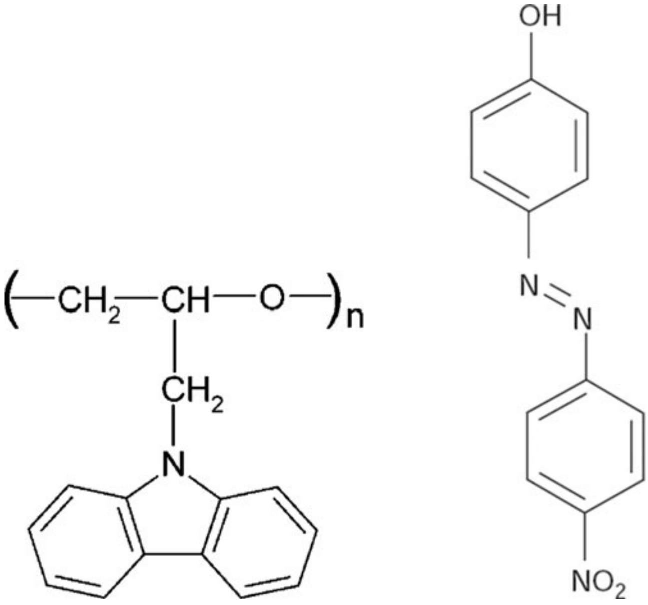
The samples were prepared as structures with a free surface [glass substrate – PCF], [glass substrate – transparent electroconducting ITO (SnO_2: In_2O_3) layer – PCF]. Concentrations of dye in all films was 15 mass%. The polymeric composite films were formed by pouring-evaporating of dichloromethane dye-polymer solutions at the substrates followed by drying at 80 °C for 2 days. The PCF films thickness, measured by MII-4 interference microscope, was $L \sim 1.0–2.0\ \mu$m.

All prepared azo-dye-doped oligomer film samples were characterized by measuring the spectral, photoconductive, photovoltaic and information characteristics.

2.3. Instrumentation

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 (electroconductive) properties of investigated PCFs were studied by a surface potential photodecay method (dark decay method respectively) in the electrophotographic mode with the use of the corona discharge at a positive charge of the surface [12, 25–28].



Scheme 1. Molecular structures of studied photoconductive oligomer and azo dye.

Photovoltage (surface) was measured by the modified Kelvin method [28–32] in the samples with a free surface. The oligomeric film composite samples were irradiated by respective light source in the active area of the probe from the side of glass substrate and transparent ITO electrode [32–33]. This method was chosen because the contact potential difference that arises in samples with sandwich structures [32] from electrodes of various materials did not affect the photoprocesses in the PFC films [29, 32–34].

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 1–50 W/m^2. The irradiation was realized at the conducting ITO layer side.

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 by 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. The corresponding planar-wavefront holograms were recorded by a semiconductor laser in the following two ways:

(1) In the first case, the surface relief gratings were fabricated using an interference of two linearly polarized beams from a laser at 532 nm (holograms of first type) according to [9, 20, 22], as shown in Figure 4b;

(2) In the second case, the respective holograms were recorded by the photothermoplastic method (PTP holograms) according to [7, 17–18], as shown in Figure 5b.

In all cases, the diffractive efficiency η of the recorded planar-wavefront hologram determined in the -1 diffraction order was used as the controlled parameter. It was measured in the corresponding diffraction order during the hologram development. Diffractive efficiency

η was defined as the intensity ratio of a diffracted beam to an incident beam as follows:

$$\eta = \mathrm{I}_{+1}/\mathrm{I}_0,$$

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

3. Results and discussion

The synthesis of 4-((4-nitrophenyl)diazenyl)phenol was carried out by a multi-step synthetic route as shown in Scheme 2.

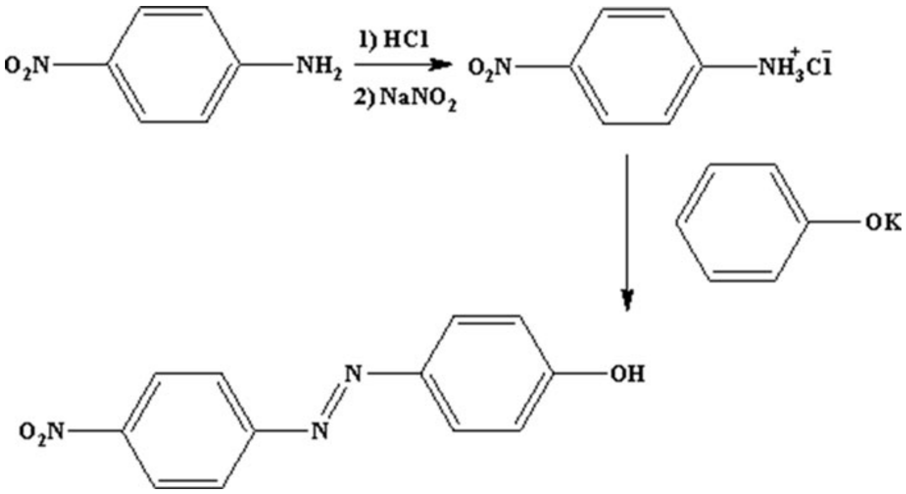
The film oligomeric composites based on photoconducting OGC oligomer doped by high concentration of azo-dye (Azo) have been obtained. The films of pure OGC are transparent in the visible and near infrared regions, and they haven't photoconductivity, photovoltaic and photorefractive properties in this area. In the PCFs which contain azo-dye additives, the bands of absorption (Figure 1) are defined by long-wavelength excitation and relaxation of the excited states of dye's molecules [8, 10, 23].

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

The kinetics of surface potential photoinduced decay 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.

It should be noted that the dark relaxation of the corona-charged samples surface potential rate in the electrophotographic mode is not significant $(dV_{surf}/dt)_{dark} \approx 0$ (see Fig. 2). On the contrary, the photoinduced decay of respective surface potential is relatively fast. This condition (low electrical conductivity and high photoconductivity within the spectral range of the used laser irradiation) is a necessary general requirement for PTP type HRM [7, 13, 17].

Figure 3 demonstrates the main features of photovoltaic response kinetics in the investigated PCFs. The growth and relaxation of photovoltaic response V_{PH} upon the light being



Scheme 2. Synthesis of 4-((4-nitrophenyl)diazenyl)phenol

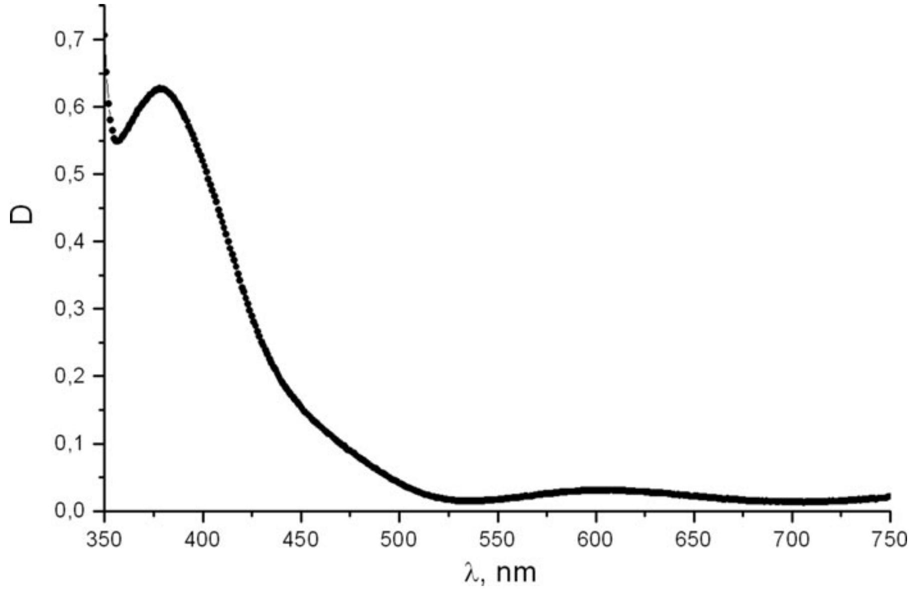


Figure 1. Absorption spectrum of oligomer composite film (PCF) based on OGC doped with 15 mass % Azo dye.

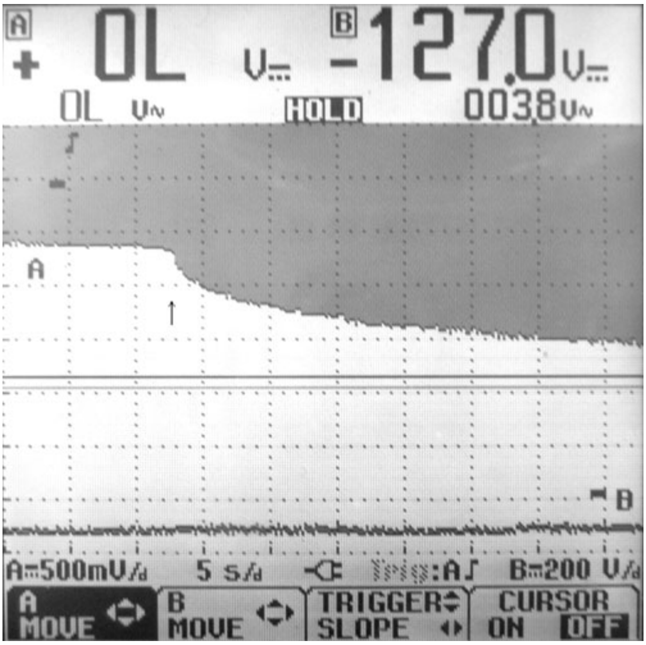


Figure 2. Experimental oscillogram of the dark and photodecay of a positive surface potential of the corona-charged sample with a free surface PCF based on OGC + 15 mass % Azo under white LED light irradiation via ITO-electrode (I = 40 W/m^2). Experiment is carried out in the electrophotographic mode. The starting and final electric surface potential values were $V_{surf, 0}$ = 127 V and $V_{surf, 50s}$ = 3,5 V respectively. The moment of switching light on is indicated with arrow.

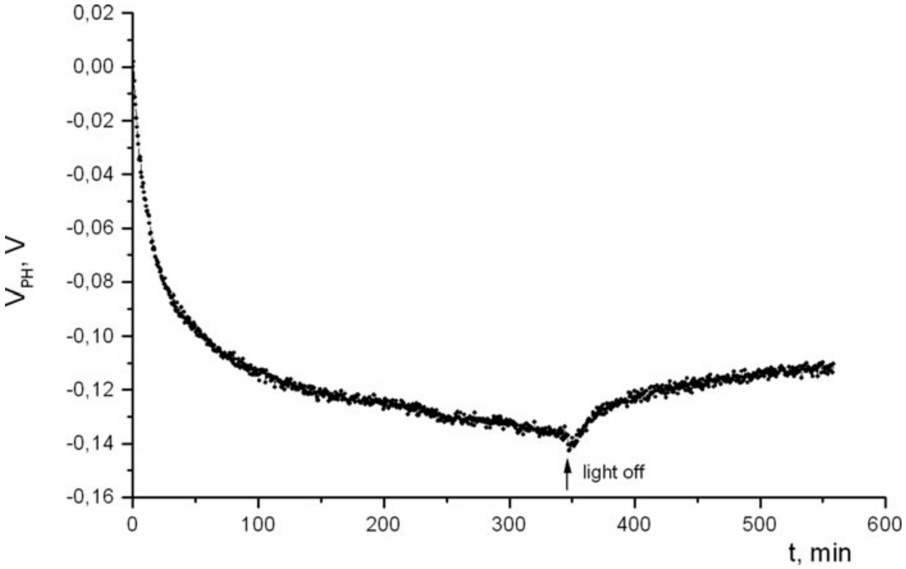


Figure 3. Kinetics of the surface photovoltage growth and relaxation in the sample with a free surface PCF based on OGC doped with 15% Azo under illumination of the ITO-electrode side of investigated sample by white light-emitting diode irradiation (I = 40 W/m^2; Ag-based probe material. The time moments of switching off the light are shown by vertical arrows).

switched on and off, respectively, is characterized by slow kinetics with a relaxation time constant, which is greater than the time constant of V_{PH} growth during the PCFs photoexposition.

The maximal attainable in these experiments photovoltaic response value V_{PH} was ~ 0,26 V.

In accordance with the earlier accepted model concepts of photogeneration and transport of charge carriers in amorphous photosemiconducting PCFs doped with organic dyes [1–3, 8, 10, 13, 15, 25, 34–36], in particular with azo-dyes [36], the internal photoeffect in these systems is related to the formation of nonequilibrium charge carriers after absorption of light by dye molecules and transport of these carriers in the polymer matrix. Based on previously developed for azo pigments-containing photoconductive amorphous composites [36] model representations, the charge carriers photogeneration in the studied film composites can be associated with two main possible mechanisms.

I. Extrinsic photogeneration in an Azo dye (Azo) in the presense of a donor hole transport molecules (OGC).

In this case, the photogeneration mechanism in the studied Azo-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 dye molecule. The structure of the EHP includes a hole, which corresponds to the cation-radical ($Cz^{\bullet +}$) of the oligomer OGC carbazolyl fragment, and the electron remaining in the molecule of the sensitizer 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 Azo and $Azo^{\bullet -}$. The corresponding reaction with electron transfer can be written as (1).

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

$$\mathrm{Azo} \overset{h\upsilon}{\rightarrow} \mathrm{Azo}^{*}$$

$$\mathrm{Cz} + \cdots + \mathrm{Cz} + \mathrm{Cz} + \mathrm{Azo}^{*} \rightarrow \mathrm{Cz} + \cdots + \mathrm{Cz} + \mathrm{Cz}\bullet^{+} + \mathrm{Azo}\bullet^{-} \rightarrow \tag{1}$$

$$\rightarrow \mathrm{Cz} + \cdots + \mathrm{Cz} + \mathrm{Cz} + \mathrm{Azo} + h\nu_{r}\,(\text{or heat})\,, \tag{2}$$

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

$$\begin{aligned}&\mathrm{Cz} + \cdots + \mathrm{Cz} + \mathrm{Cz}\bullet^{+} + \mathrm{Azo}\bullet^{-} \rightarrow \mathrm{Cz} + \cdots + \mathrm{Cz}\bullet^{+} + \mathrm{Cz} + \mathrm{Azo}\bullet^{-} \rightarrow \\ &\rightarrow \mathrm{Cz}\bullet^{+} + \cdots + \mathrm{Cz} + \mathrm{Cz} + \mathrm{Azo}\bullet^{-}\end{aligned} \tag{3}$$

As a result of EHP dissociation (Eq. 3), the electrostatic interaction between the hole and electron localized in Azo$\bullet^{-}$ anion-radical weakens. At the recombination stage (scheme 2), the valence electron of the sensitizer anion-radical form (Azo$\bullet^{-}$) transfers to Cz$\bullet^{+}$, and after relaxation of the excited state, the radiative energy ($h\nu_r$) or nonradiative heat is released. Then the azo-dye 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, 34, 36].

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

II. Intrinsic photogeneration in an Azo dye.

$$\mathrm{Azo} \overset{h\upsilon}{\rightarrow} \mathrm{Azo}^{*}$$

$$\mathrm{Azo}^{*} + \mathrm{Azo} \rightarrow \mathrm{Azo}\bullet^{-} + \mathrm{Azo}\bullet^{+} \rightarrow \tag{1'}$$

$$\rightarrow 2\mathrm{Azo} + h\nu_{r}\,(\text{or heat})\,, \tag{2'}$$

$$\text{or } \mathrm{Azo}\bullet^{-} + \cdots + \mathrm{Azo}\bullet^{+} + \mathrm{Azo} + \mathrm{Azo} \rightarrow \mathrm{Azo}\bullet^{-} + \cdots + \mathrm{Azo} + \mathrm{Azo}\bullet^{+} + \mathrm{Azo} \rightarrow$$

$$\rightarrow \mathrm{Azo}\bullet^{-} + \cdots + \mathrm{Azo} + \mathrm{Azo} + \mathrm{Azo}\bullet^{+} \tag{3'}$$

$$\text{and } \mathrm{Azo}\bullet^{+} + \cdots + \mathrm{Azo}\bullet^{-} + \mathrm{Azo} + \mathrm{Azo} \rightarrow \mathrm{Azo}\bullet^{+} + \cdots + \mathrm{Azo} + \mathrm{Azo}\bullet^{-} + \mathrm{Azo} \rightarrow$$

$$\rightarrow \mathrm{Azo}\bullet^{+} + \cdots + \mathrm{Azo} + \mathrm{Azo} + \mathrm{Azo}\bullet^{-} \tag{3''}$$

Where (1, 1′) - geminate EHP photogeneration, (2, 2′) - EHP recombination, (3, 3′, 3″) - geminate EHP dissociation.

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, 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 [37, 38]. 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 [38]. However, it is possible that other photoresponse formation mechanisms, e.g., azo chromophores photoorientation [23], were also partially responsible in this case [1–2, 10, 20, 23, 34].

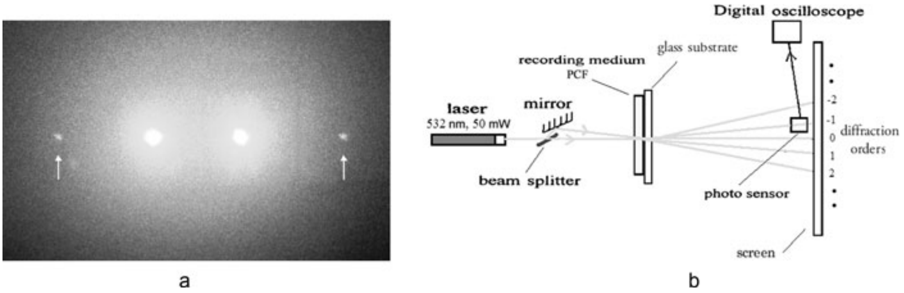


Figure 4.(a) The reconstructed hologram of a planar wavefront image (the respective diffraction pattern obtained by 532 nm laser beams) recorded in the composite sample with a free film surface (glass substrate – composite film) based on OGC doped with 15 mass % Azo (L ~ 1,5 μm) under sample illumination by green semiconductor laser (I = 15 mW, $t_{exposure}$ = 10 s) irradiation (the respective diffraction orders are shown by vertical arrows). Hologram was recorded at a 1:1 intensity ratio of the reference to the object beam. The spatial frequency of hologram recording was ~ 800 mm^{-1}; (b) the simplified scheme of respective measurement. The diffractive efficiency value was ~ 0,05%.

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 [2, 33, 35, 36, 39–41].

The significantly faster photoinduced decay rate of the corona-charged samples surface potential in the electrophotographic mode (Figure 2) than the respective photovoltaic response growth and dark relaxation (Figure 3) 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 ($E_{initial}$ ~10^8 V/m). The charge carriers in the photovoltaic effect were much less mobile, so their probability of 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, 40]) depends rather strongly on the electric field [1, 8, 13, 34–36, 41].

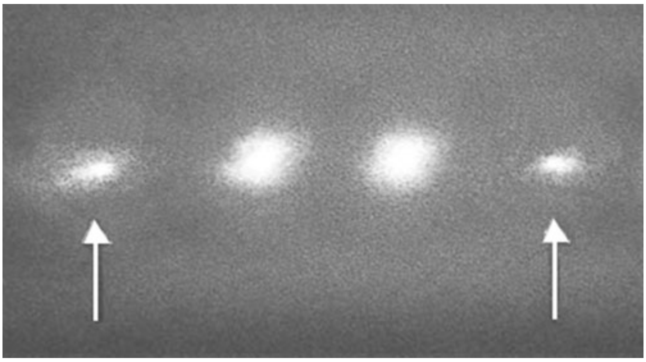


Figure 5a. The reconstructed photothermoplastic hologram of a planar wavefront image recorded in the composite sample with a free film surface (glass substrate – ITO – composite film) based on OGC doped with 15 mass% Azo (L ~ 1 μm) under sample illumination by green semiconductor laser (I = 15 mW, texposure = 10 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 ~ 800 mm^{-1}, η ~ 0,3% (the corresponding diffraction orders are shown by vertical arrows).

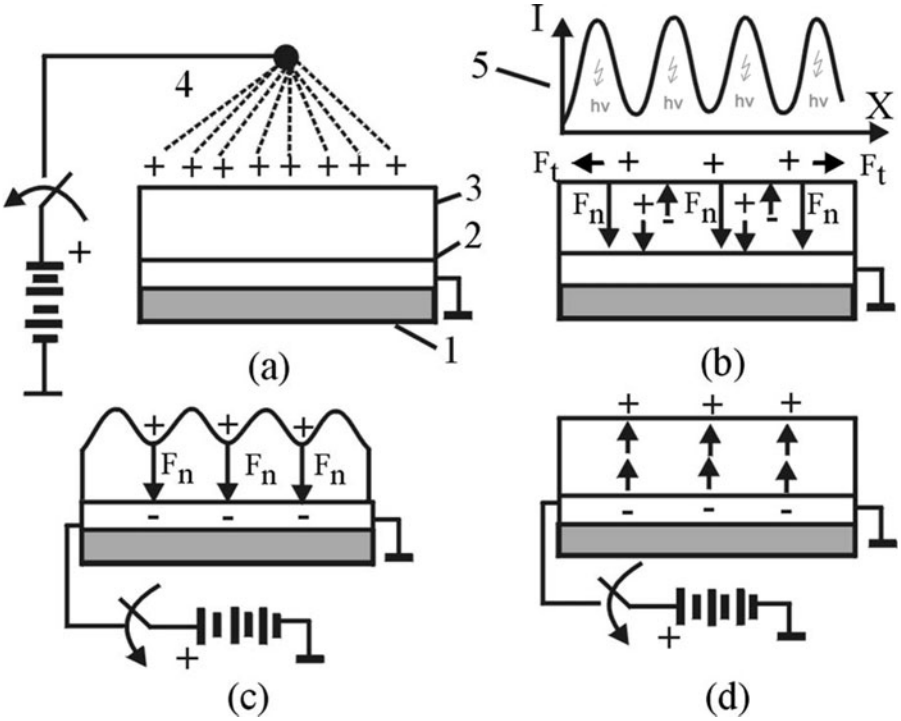


Figure 5b. Scheme of photothermoplastic holographic recording: a - charging of the surface of HRM in corona discharge; b - light exposure; c - latent image development; d - erasing of the recorded hologram; 1 - glass substrate; 2 - conductive ITO-layer; 3 - photoconductive thermoplastic film; 4 - corona discharge; 5 - modulated light.

As it could be expected, investigated PCFs are capable to form holograms of first type (see above). In this case, the diffraction grating formation is due to the well-known photoinduced trans-cis isomerization and molecular reorientation processes (photoinduced alignment of azo chromophores) in the azo dye molecules [9, 19, 23]. Figure 4 illustrates the reconstructed hologram image (Figure 4a) as a result of these experiments and general scheme of experiment (Figure 4b).

The respective PTP type holograms of a planar wavefront were also registered in the investigated oligomer media. Figure 5 shows results (Figure 5a) and the scheme (Figure 5b) of these PTP holographic recording experiments.

The maximal attainable in these experiments diffractive efficiency value was $\sim$ 1%. The possibility of the PTP surface relief grating formation is due to the following general requirements for PTP HRM: (i) 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_{soft} [7]. The investigated oligomeric films can be used again for recording a new hologram after erasing the previous hologram and after the film cools [7].

4. Conclusions

New photochromic oligomeric thin film structures ($L \sim 1\ \mu m$) based on carbazole-containing oligomer OGC doped by high concentration of the azo-dye 4-((4-nitrophenyl) diazenyl)phenol have been prepared. 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 azo dye absorption region. The diffusion nature of observed photovoltaic response 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 photodecay as compared with the respective characteristics of the photovoltaic response in these PCF. It 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 HRM for both the polarization and PTP optical holography. 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 shown a capacity for a long-term memory after preliminary irradiation of the films.

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