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Influence of a bleaching post-exposure treatment in the performance of H-PDLC devices with high electric conductivity

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ABSTRACT

Holographic polymer dispersed liquid crystals (H-PDLC) are made by holographic recording in a photo-polymerization induced phase separation process in which the liquid crystal molecules diffuse to dark zones in the diffraction grating. The devices with H-PDLC materials develop a dynamic behavior that may be modified by means of an electric field. We study a photopolymer formulation with high diffraction efficiency but with the problem of high electric conductivity. We use a bleaching post-exposure treatment to obtain devices with a better electro-optical performance.

Keywords: Holographic polymer dispersed liquid crystals, holographic recording materials, photopolymer formulation, electro-optical polymer devices.

1. INTRODUCTION

Holographic polymer dispersed liquid crystals (H-PDLC) are made by holographic recording in a photo-polymerization induced phase separation process in which the liquid crystal molecules diffuse to dark zones in the diffraction grating. Afterwards they can be oriented by means of an electric field. The reorientation of the liquid crystal produces a refractive index variation which changes the diffraction efficiency. Therefore, the grating develops a dynamic behavior that may be modified by means of an electronic device. In this manner, it is possible to make dynamic devices such as tunable-focus lenses, sensors, phase modulators or prism gratings [1-7].

The objective of a H-PDLC material is to act as a support for an electro-optical dynamic device. Bearing this in mind, the material must have a low thickness in order to use a low electric field with the device. In this work, we study a photopolymer formulation with high diffraction efficiency [8]. We have identified a problem related to the high electrical conductivity of the formulation which implies a low voltage for the H-PDLC devices and therefore the electro-optical response is very poor. We have identified several ionic components of the photopolymer formulation that could be related to the high electric current intensity obtained when the voltage is increased. We use a bleaching post-exposure treatment in order to improve the electro-optical performance.

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2. EXPERIMENTAL SECTION

The photopolymer is composed of dipentaerythritol penta/hexa-acrylate (DPHPA) as monomer, N-vinyl-2-pyrrolidone (NVP) as crosslinker, N-phenylglycine (NPG) as radical generator and octanoic acid (OA) as surfactant and cosolvent [9]. We used ethyl eosin (YEt) as dye and N-methyl-2-pyrrolidone (NMP) to control the overmodulation during the hologram recording. This composition was optimized in a previous work [10]. Table 1 shows the composition of the material.

Table 1. Composition of the material.

component	concentration (wt%)
DPHPA	46.52
CL036	29.31
NVP	15.80
YEt	0.08
OA	4.22
NMP	3.13
NPG	0.94

We used the nematic liquid crystal CL036 from Qingdao Intermodal Co., Ltd. It is a mixture of 4-cyanobiphenyls with alkyl chains of different lengths. It has an ordinary refractive index n_0= 1.520, and a difference between extraordinary and ordinary index Δn= 0.250. Figure 1 shows the molecular structure of the components of the material.

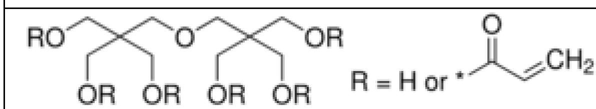
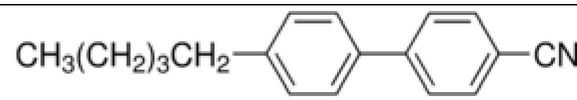
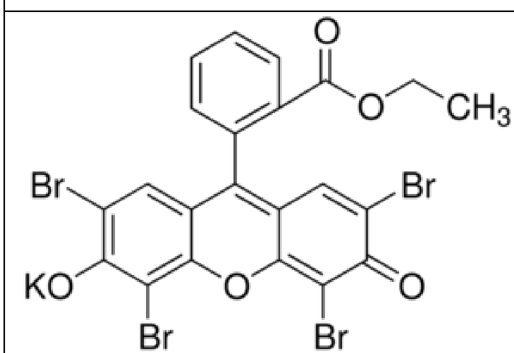
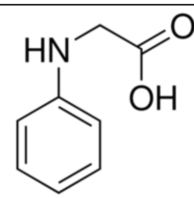
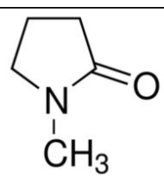
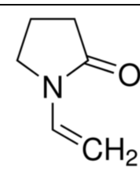
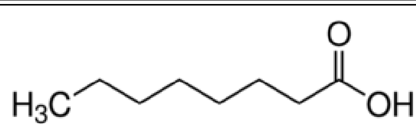
Dipentaerythritol penta/hexa-acrylate	4′-Pentyl-4-biphenylcarbonitrile (CL036)
	
Ethyl eosin	N-Phenylglycine
	
N-methyl-2-pyrrolidone	N-vinyl-2-pyrrolidone
	
Octanoic acid	
	

Figure 1. Molecular structure of the components of the material.

The material was made by mixing the components under red light where they are not sensitive. The solution was sonicated in an ultrasonic bath, deposited between two conductive ITO glass plates 1 mm thick and separated using 13 μm hollow glass microspheres as spacers (Figure 2). The device was exposed to a laser beam (λ=532 nm) in a holographic set-up in order to record a diffraction grating in the photopolymer layer (Figure 3). A photopolymerization reaction takes place in the bright zones of the diffraction grating and a highly reticulated polymer network is generated. The liquid crystal molecules diffuse to the unexposed region where they remain as droplets.

After recording, the diffraction grating in the H-PDLC is reconstructed (λ=633 nm) and the diffraction efficiency is obtained. The device with the optimized composition is exposed to a variable electric field in order to evaluate the electro-optical properties induced by the liquid crystal. We consider a bipolar square waveform, generated by a waveform generator connected to a voltage amplifier [9], whose voltage amplitude is modified. Figure 2 shows a scheme of the H-PDLC device. The polymer rich zone and liquid crystal rich zone are separated in the graph but the polymer network penetrates into the liquid crystal rich zones.

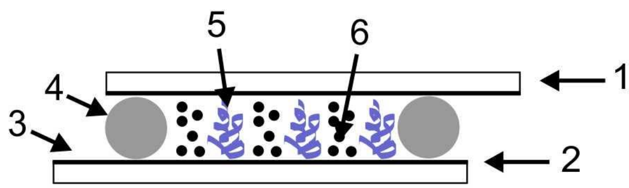


Figure 2. Scheme of the H-PDLC device: 1 glass plate, 2 ITO coating on glass plate, 3 electric connection zone, 4 glass microspheres, 5 polymer rich zone, 6 liquid crystal rich zone.

2.1 Holographic set-up

We obtained diffraction gratings using a holographic setup to study the behaviour of these photopolymers as a holographic recording material. The experimental device is shown in Figure 3. A Nd:YAG laser tuned at a wavelength of 532 nm was used to record diffraction gratings by means of continuous laser exposure. The laser beam was split into two secondary beams with an intensity ratio of 1:1. The diameter of these beams was increased to 1 cm by means of a lens, while spatial filtering was ensured. The object and reference beams were recombined at the sample at an angle of 16 degrees to the normal with an appropriate set of mirrors, and the spatial frequency obtained was 1036 lines/mm. The working intensity at 532 nm was 7 mW/cm^2. The diffracted and transmitted intensity were monitored in real time with a He-Ne laser positioned at Bragg's angle (19.1°) tuned at 633 nm, where the material does not polymerize. The diffraction efficiency was calculated as the ratio of the diffracted beam (I_D) to the incident power (I_0).

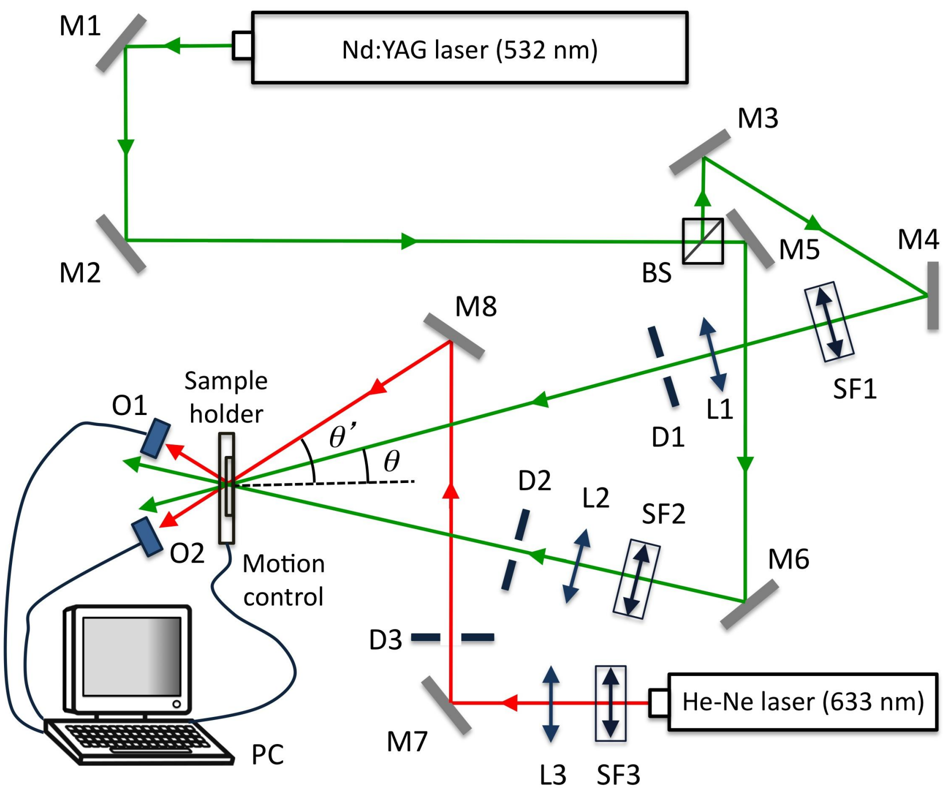


Figure 3. Experimental set-up. BS: Beamsplitter, Mi: mirror, SFi: spatial filter, Li: lens, Di: diaphragm, Oi: optical power meter, PC: data recorder.

2.2 Electro-optical response

The devices are exposed to an electrical field in order to evaluate the electro-optical response. Figure 4 shows the electro-optical set-up. It includes a Tektronik TDS1012B oscilloscope (A), Tektronic AFG3022B dual channel arbitrary function generator (B), N4L voltage amplifier (C), and an impedance control circuit designed in our laboratory (D). The signal applied to the device is adjusted to AC, 1 kHz bipolar square waveform. The impedance control circuit attenuates the high intensity spikes associated with capacitance effects produced by the steep voltage changes in the applied voltage. These intensity spikes reach values higher than the ones allowed by the amplifier protection circuit, thus switching it off and limiting the range of the applicable high voltage amplitude values. The capacitance is due to the capacitor structure of the H-PDLC cell, composed of the two ITO electrodes and the H-PDLC dielectric layer in between.

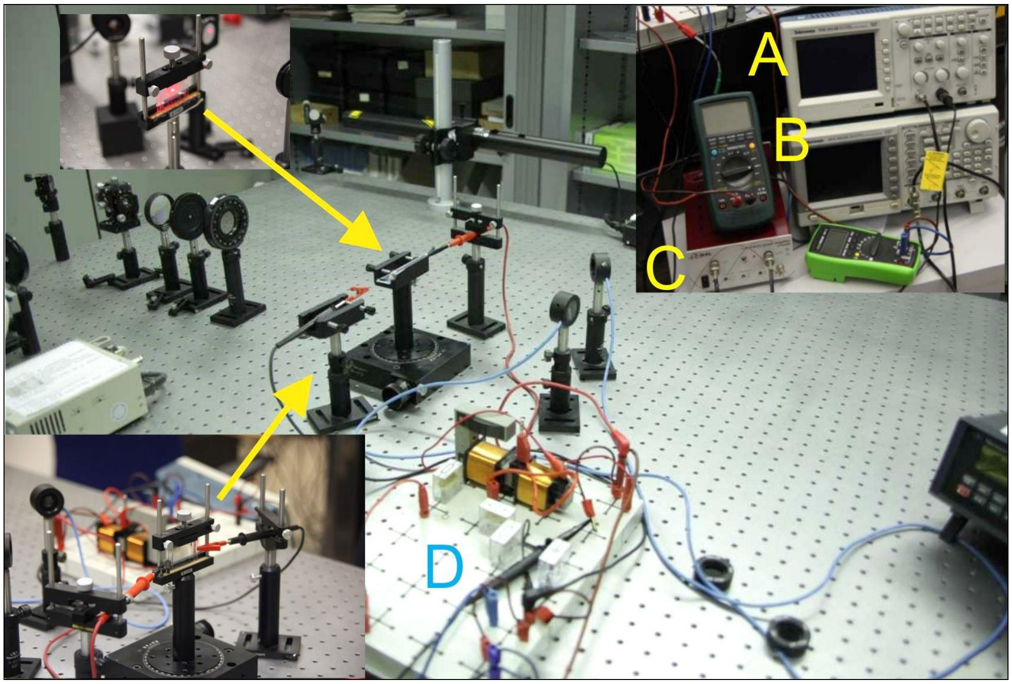


Figure 4. Electro-optical set-up: the upper arrow shows the diffraction when the hologram is reconstructed. The bottom arrow shows the H-PDLC device plate with the electric connections.

When the voltage is applied, the liquid crystal molecules are reoriented in the direction of the electric field. Due to the liquid crystal fraction the refraction index of the dark zones decreases to values closer to that of the bright zones. Therefore the index modulation decreases when the voltage increases.

3. RESULTS

3.1 Experiment without post-exposure treatment

After recording of the hologram, the device is placed into an electrical field in order to evaluate the electro-optical response. Figure 5 graph A shows the diffraction efficiency (DE) as a function of the applied RMS voltage. The device A without post-exposure treatment reaches a DEmin= 59% at 22 V. At this voltage, the electrical current intensity through the device reaches 100 mA (RMS value) and the protection circuit of the amplifier switches off the electric power (the impedance control circuit only affects the transient intensity values). Therefore is not possible to increase more the voltage. But this voltage is still very small to obtain changes of the diffracted intensity.

The relatively high value of electric intensity is due to the characteristics of the components in the photopolymer solution (Table 1). The molecular structure of each component can be seen in Figure 1. YEt, NPG and OA are ionic molecules with high solubility into the mix with the other components. When the voltage is increased, the ionic species support increasing values of electrical current intensity. Since the voltage used is AC, electrochemical reactions are avoided and the ionic species in the active layer are not wasted.

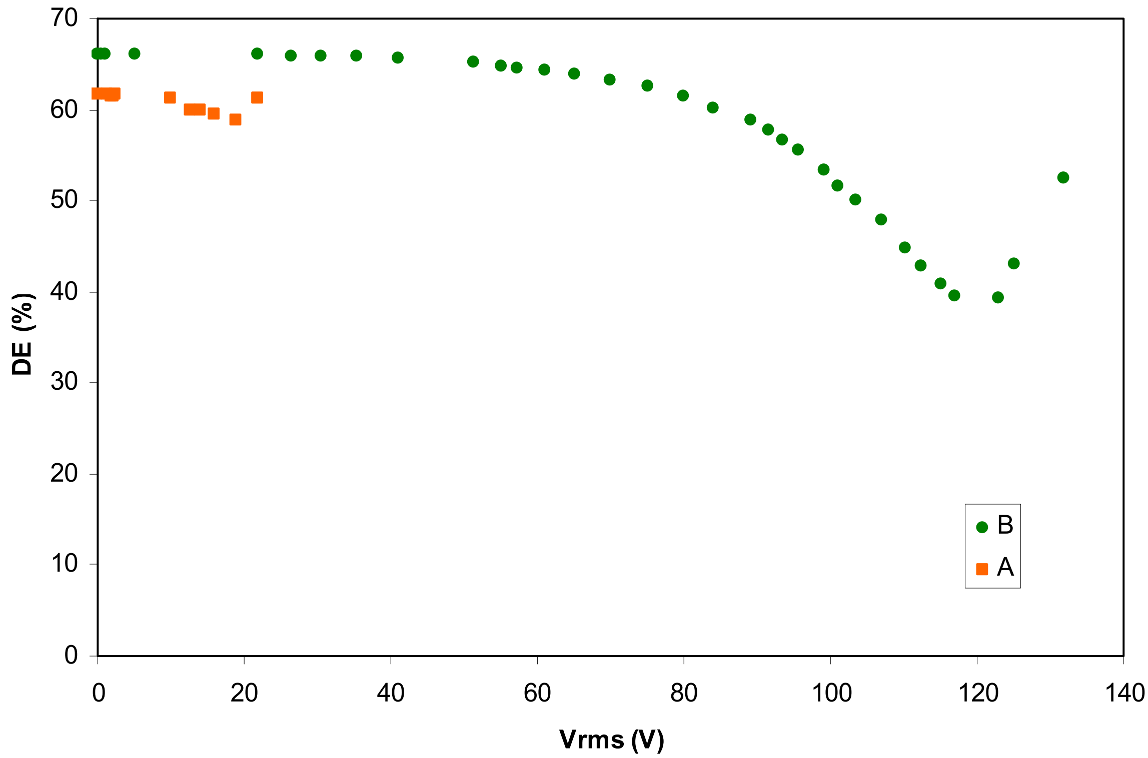


Figure 5. Diffraction efficiency as a function of RMS voltage. Graph A: device without post-exposure treatment. Graph B: device with post-exposure treatment.

3.2. Bleaching post-exposure treatment

In order to minimize the effect of the ionic species of this photopolymer, we use a bleaching post-exposure treatment by means of a halogen light. The devices are exposed to halogen light (90 W, 5 minutes) after hologram recording. This treatment has two effects. First the YEt concentration is reduced since the dye placed in the dark zones during hologram recording reacts with NPG. Second, the monomer placed in the dark zones is polymerized and therefore the mobility of the ions is reduced. Figure 5 graph B shows the diffraction efficiency as a function of the RMS voltage for device B with the bleaching post-exposure treatment. Figure 6 shows the electrical current intensity through the device as a function of the RMS voltage for the two devices.

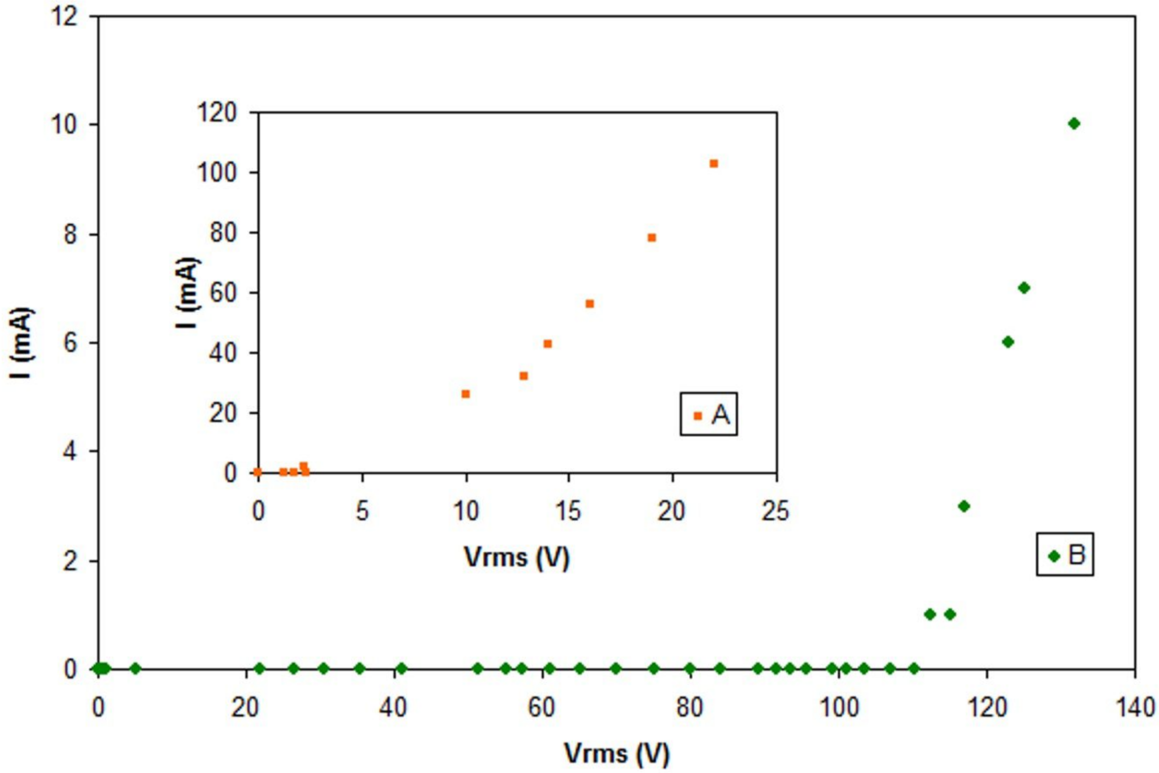


Figure 6. Electrical current intensity as a function of RMS voltage for both devices.

The device A reaches an electrical current intensity of 100 mA with a very low voltage of 22 V. Therefore, the diffraction efficiency decreases only from 62% to 59% (Figure 5). The diffraction efficiency starts to increase again when the amplifier automatically switches off (last dot of graph A in Figure 5). The voltage can be increased up to 132 V for the device B. The diffraction efficiency decreases from 66% to 39% when the voltage increases.

The electrical current intensity increases from zero at voltages higher than 123 V as can be seen in graph B. When the voltage of the amplifier is set at 125 V, i=7 mA and with this current between the electrodes of the H-PDLC cell the voltage starts to decrease. Therefore the DE starts to increase from the minimum value (Figure 5).

CONCLUSION

This photopolymer formulation obtains a high value of diffracted intensity during hologram recording (DE>60 %) but the device shows high electrical conductivity and the voltage only can be increased up to 60 V and at this voltage the electro-optical response is very poor. We have identified several ionic components of the photopolymer formulation that could be related to the high electric current intensity obtained at that voltage.

We use a bleaching post-exposure treatment that obtains a better result. Thus, the voltage can be increased up to 125 V and the device has a better electro-optical performance. The bleaching treatment has two effects: dye and initiator concentration decrease and mobility decrease of the ionic species. Therefore, we can conclude that the poor response of the untreated device is due to the high electrical conductivity of this formulation due to the molecules with ionic characteristics: YEt, NPG and OA.

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