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Diffusion parameters estimation of holographic memories based in PVA/Acrylamide photopolymer

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ABSTRACT

The hard research about the models to predict and understand the behaviour of photopolymers have as a results many interesting works for 2-dimensional cases. These studies permits obtain the mains parameters that governs the process in photopolymers with maximum thickness about $200\text{ }\mu\text{m}$. Historically this materials, dry layers of photopolymers, have been used in many attractive devices and now a new application of this type of material is being developed: the application of photopolymers as holographic memories. The main characteristics of these layers of photopolymers are their high thickness (higher than $600\text{ }\mu\text{m}$). In order to optimize this layers the original photopolymer composition has been changed, then a new parameters estimation has be done. In this work this study is made using PVA/Acrylamide photopolymer with layers around $800\text{ }\mu\text{m}$ of thick. The values of monomer and polymer diffusion are obtained and the values of polymerization rate constant and residual monomer are calculated too using and first harmonic diffusion constant. The validity of this model to study the layers with high thickness is evaluated, because this type of materials only a few percent of the initial light arrive to the depth zones of photopolymer.

1. INTRODUCTION

In recent years, the research to achieve optimized holographic memories has received much attention [1-4]. This type of devices takes advantage of the thickness of the recording material to record data, a characteristic that is inherent in holography, as opposed to currently available optical devices such as CDs or DVDs based on surface recording. For this reason, the theoretical information storage capacity of holographic memories largely exceeds the capability of current techniques: 1000 times greater than CDROM, with a random access time of only 10% of the latter.

One of the basic requirements for holographic memories to be competitive is that the thickness of the recording material layer must be $500\text{ }\mu\text{m}$ or thicker [1]. A greater number of holograms may be recorded with thicker layers, since in this case the angular Bragg selectivity is higher and the width of the angular response curve is very small [3]. Some different techniques for multiplexing holograms have been developed [1]. On the other hand one problem of multiplexing methods is the choice of an exposure schedule that maximizes the uniformity and capacity of a holographic recording medium [5-6]. To applied some of these optimized schedules in necessary to know the parameters that's govern the behaviour of the material during the recording process and the exactly diffusivity of the species involves in the grating formation.

It is not easy to find such a great thickness with the recording materials currently available. In spite of the large number of materials that have been employed as holographic memories (photorefractive polymers, bacteriorhodopsin, photopolymers...)[2,5,7]. Photopolymers based on acrylamide monomer (AA) are one of the materials of interest due to their good properties, in particular their acceptable energetic sensitivity compared with that of other available materials, the

possibility of easily adapting their spectral sensitivity to the type of recording laser used by simply changing the sensitizer dye, high diffraction efficiency, together with an acceptable resolution and signal/noise ratio [8]. Specifically, in our research team we use polyvinylalcohol/acrylamide (PVA/AA) compounds [9-10]. Their low price, easy preparation and the fact that complicated developing processes are not necessary make them even more attractive for use on a large scale in read only WORM (write once read many) type memories.

Photopolymers have many attractive applications in addition to holographic data storage. For example optical imaging processing by Bragg diffraction [11], generation of diffraction elements [12], etc. For this reason many models have been proposed in order to simulate the behavior of this type of materials [13-16]. The usefulness of these models have been demonstrated in different works (to predict the cut off spatial frequencies, the residual monomer...) [17-18], but the thickness of the layers analyzed by the models is only around 100 μm . In this paper the main idea is to study the behaviour of the holographic memories recorded in PVA/AA (layers with effective optical thickness higher than 500 μm) using a diffusion model. The first goal of the work is the estimation of the parameters (diffusivity, polymerization rate...) that characterize the hologram formation in the material. The values obtained are compared with the parameters obtained in gratings with a thickness of 100 μm . The second goal is to compare the polymer diffusivity between thin and thick layers. For this analysis we use a method based on a model proposed by O'Neill et al [19]. These values are important to know the properties of the photopolymer (chain length, viscosity, etc). In order to conserve the information stored in photopolymers and stopped polymer diffusion crosslinker monomers are introduced in the composition [20]. Nevertheless the values of the material viscosity are similar in two cases because the crosslinker concentration are very low [20-21]. The third goal of this paper is the analysis of the behaviour of the material using a simple exposure schedule (exposure time constant for each hologram).

2. THEORETICAL MODELS

2.1 HOLOGRAM FORMATION

In order to analyze the photopolymer characteristics we will use the diffusion model described in reference [18]. This is a first harmonic diffusion model, thus higher harmonics are not considered in the Fourier expansion of the monomer and polymer concentrations. This approximation can be justified because the higher harmonics of the refractive index are negligible in gratings recorded in PVA/AA as demonstrated in reference [22]. In a first harmonic diffusion model a sinus (or cosinus) distribution is considered for the refractive index (n), monomer concentration ($[M]$) and polymer concentration ($[P]$).

$$n(x,t) = n_0(t) - n_1(t) \cos(K_g x) \quad (1)$$

$$[M](x,t) = [M]_0(t) - [M]_1(t) \cos(K_g x) \quad (2)$$

$$[P](x,t) = [P]_0(t) + [P]_1(t) \cos(K_g x) \quad (3)$$

where K_g is the grating period and $n_1(t)$, the refractive index modulation, depends on the refractive indexes of the different components of the material as [23]:

$$n_1 = \frac{(n_{dark}^2 + 2)^2}{6n_{dark}} \left[- \left(\frac{n_m^2 - 1}{n_m^2 + 2} - \frac{n_b^2 - 1}{n_b^2 + 2} \right) [M]_1 + \left(\frac{n_p^2 - 1}{n_p^2 + 2} - \frac{n_b^2 - 1}{n_b^2 + 2} \right) [P]_1 \right] \quad (4)$$

where:

n_p , is the polymer refraction index.
 n_m , is the monomer refraction index.
 n_b , is the binder refraction index.
 n_{dark} , is the refraction index of the layer before the exposure.

In the material used in this work the different refractive indexes take the following values:

$$\begin{aligned} n_m &= 1.486 \\ n_b &= 1.474 \\ n_{dark} &= 1.478 \end{aligned}$$

If a sinusoidal interference pattern is considered the polymerization (F) can be obtained as:

$$F(x, t) = k_R I_0^\gamma \left(1 + V \cos(K_g x) \right)^\gamma = k_R I^\gamma \quad (5)$$

where V is the visibility of the fringes, I is the intensity, γ indicates the relationship between the intensity and the polymerization and k_R is the polymerization rate. The polymerization rate decreases by the Trommsdorff effect and is given by:

$$k_R(t) = k_R \exp(-\varphi I_0 t) \quad (6)$$

where φ is the attenuation coefficient of the polymerization rate.

The differential equations to solve are:

$$\frac{\partial [M](x, t)}{\partial t} = \frac{\partial}{\partial x} D \frac{\partial [M](x, t)}{\partial x} - k_R(t) I^\gamma(x) [M](x, t) \quad (7)$$

$$\frac{\partial [P](x, t)}{\partial t} = k_R(t) I^\gamma(x) [M](x, t) \quad (8)$$

Where D is the monomer diffusion constant. In this paper a value of 0.5 for γ is considered [13].

The limitation of the diffusivity by the increases of the polymer concentration is also considered and can be seen in following equation:

$$D(t) = D \exp(-\varphi' I_0 t) \quad (9)$$

where φ' is the attenuation coefficient of the polymerization rate and is similar than φ .

The physical thickness of the material, d' , is larger than the real thickness (d) occupied by the recorded grating. We call d the optical thickness of material. This difference is due to the existence of an exponential decay of the light across the depth of the hologram. An algorithm based in Rigorous Couple Wave (RCW) method [22, 24] was developed to study this decay and obtain the optical thickness of the holographic memories [25] and obtain the thickness where the grating is recorded. Then, this parameter is introduced in the diffusion model.

2.2 EFFECTIVE OPTICAL THICKNESS OF THE THICK LAYERS

Important difficulty provided by these layers of a high thickness is the accurate determination of the optical thickness: The reason is the existence of an attenuation of the index profile in depth [25]. The initial transmittance (for the wavelength of 514 nm) of this type of layers is around 1%, and the main portion of the information is stored in the vicinity of the entrance side of the layer. This prevents from obtaining narrow angular responses.

In RCW method the profile of the dielectric permittivity will only be supposed to vary in the x and z directions, therefore a planar diffraction grating is studied. The study will also be restricted to the case of unslanted gratings so the dependence of dielectric permittivity with x, z will assume to be in the form:

$$\varepsilon(x, z) = \sum_n \varepsilon_n(z) \exp[jhKx] \quad (10)$$

K is the modulus of the grating vector, which is related to the period of the interference fringes, Λ , as follows:

$$K = 2\pi/\Lambda \quad (11)$$

We consider the following index decay for the index modulation inside the grating:

$$n_1(z) = n_1 \exp(-\alpha z) \quad (12)$$

2.3 DIFFRACTION EFFICIENCY DECAY

In PVA/AA based photopolymers (without crosslinker monomers in their composition), the estimation of the polymer diffusion, D , is a simple work using the model proposed by O'Neill et al [19]. The value of this parameter indicates the viscosity of the material and provides useful information to the general behaviour of the photopolymer during the hologram formation and also when the hologram has been stored. If crosslinker monomers are used the monomer chains are very large, the diffusion is very slow and this effect can not be seen. The values of the polymer diffusion depends basically on the same factors as the monomer diffusion: photopolymer composition (PVA, monomer...), humidity, temperature and drying time. The method used to calculate the polymer diffusivity inside the material is simple. In the model an exponential decrease of the index modulation after the hologram recording is assumed using a constant value of the diffusivity. This idea can be expressed in the following form:

$$n_1(t) = n_1(t \rightarrow \infty) + \Delta n_1 \exp\left\{-D\left(\frac{2\pi}{\Lambda}\right)^2 t\right\} \quad (13)$$

We measure the diffraction efficiency decay in the recorded gratings from the fitting of the angular responses.

3. EXPERIMENTAL

The photopolymerizable solution was prepared, by adding yellowish eosin (the dye) together with a solution of acrylamide (the monomer) and triethanolamine (the co-initiator) to a PVA (the binder) solution. In this study the solutions were prepared using a conventional magnetic stirrer, under red light and in standard laboratory conditions (temperature, pressure, relative humidity). If the viscosity is too high to use a magnetic stirrer, we stir the solution slowly by hand to prevent the formation of air bubbles, followed by the application of a vacuum atmosphere to both the prepared solution and the coated molds so that

any eventual air bubbles are completely eliminated. The solutions are deposited, by gravity, in polystyrene circular molds, and left in the dark to allow the water to evaporate, while recording in the laboratory conditions (temperature and relative humidity, RH) during the process. When a high percentage of the water content has evaporated, the “dry” material is removed from the mold, cut into squares and deposited, without the need of adhesive, onto the surface of glass plates measuring $6.5 \times 6.5 \text{ cm}^2$. The plates are then ready for exposure, which takes place immediately. In table 1 both the composition of the solution of the holographic memories (more than $500 \text{ }\mu\text{m}$) and the layers of $100 \text{ }\mu\text{m}$ of thickness can be seen. To introduce the monomer concentration in the dry film the water evaporated is calculated [9]. The values (in volume fraction units) of the initial monomer concentration in the dry holographic memories used in this work are 0.19 and 0.25 for thick and thin layers respectively. These are the values to be introduced in the model.

Table 1. Composition of the solution used to prepare layers of the recording material according to the thickness to be obtained

component	More than $500 \text{ }\mu\text{m}$	Around $100 \text{ }\mu\text{m}$
Polyvinyl alcohol $M_w=130000$	13.30% w/v	8.5% w/v
Triethanolamine	0.15 M	0.4 M
Acrylamide	0.34 M	0.48 M
Yellowish eosin	$9.00 \times 10^{-5} \text{ M}$	$2.4 \times 10^{-4} \text{ M}$

To study the behavior of the photopolymeric layers, we obtained unslanted diffraction gratings using a holographic setup (figure 1). An Argon laser at a wavelength of 514 nm was used to store 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 diameters of these beams were increased to 1.5 cm with an expander, while spatial filtering was ensured. The object and reference beams were recombined at the sample at an angle of 16.8° to the normal with an appropriate set of mirrors, and the spatial frequency obtained was 1125 lines/mm . The working intensity at 514 nm was 5 mW/cm^2 . The diffracted and transmitted intensity were monitored in real time with a He-Ne laser positioned at Bragg's angle (20.8°) tuned to 633 nm , where the material is not sensitive. To obtain the transmission and diffraction efficiency as a function of the angle at reconstruction we placed the plates on a rotating stage. The transmission and diffraction efficiency (TE and DE respectively) were calculated as the ratio of the transmitted and diffracted beam power, respectively, to the incident power. We consider the losses due to the Fresnel coefficients of the refraction.

Fig. 1. – Experimental setup. Where M_i are the mirrors. L_i are the lenses. SF_i the spatial filters to expand the beams. D_i represent the diaphragms. BS is the beam splitter.

4. RESULTS AND DISCUSSION

4.1 HOLOGRAM FORMATION

In this section, we use the diffusion model proposed in reference [16] to characterize the holographic behaviour of layers with a thickness larger than $500\text{ }\mu\text{m}$. To obtain a deeper insight, first we analyzed the characteristics of thin layers, then we compare these values with the results provided by thick layers.

In figure 2 the diffraction efficiency as a function of the exposure time for two gratings recorded in holographic memories (layers thickness greater than $500\text{ }\mu\text{m}$) is represented. The gratings studied have different optical thickness: the first (white dots) is $650\text{ }\mu\text{m}$ thick and the second (squares) is $700\text{ }\mu\text{m}$ thick as can be seen in the figure. The model predicts properly the behavior in both cases. After the first diffraction efficiency maximum the effects of the overmodulation of the refractive index can be observed [29]. The exposure has been stopped near this maximum because the scattering increases dramatically for high exposures and the attenuation of the index profile across the depth of the layer increases too [9]. The combination of these two factors produce a high distortion of the angular response. As a result we can not fit properly the value of the optical thickness, which is needed in the simulations of the diffraction efficiency.

Fig. 2. Diffraction efficiency versus the exposure time at Bragg angle for thick layers using two different thickness.

The parameters obtained using the first harmonic diffusion model for the holographic memories can be seen in table 2.

Table 2. Parameters obtained by diffusion model when the experimental data are fitted.

Parameters	More than $500\text{ }\mu\text{m}$	Around $100\text{ }\mu\text{m}$
D	$7 \times 10^{-10}\text{ cm}^2/\text{s}$	$2 \times 10^{-10}\text{ cm}^2/\text{s}$
k_R	$0.016\text{ cm}^2\text{ mW}^{-1}\text{ s}^{-1}$	$0.025\text{ cm}^2\text{ mW}^{-1}\text{ s}^{-1}$
n_p	1.504	1.504
φ	$0.005\text{ }\mu\text{m}^{-1}$	$0.005\text{ }\mu\text{m}^{-1}$
Residual Monomer	25%	60%

As can be seen the values of the parameters are similar to those obtained when thin layers were studied, although there are some slight changes because the composition and the thickness are different. In the first place, this type of layers

presents a low diffusivity and in the second place the values of the initial polymerization rate is low too. This second aspect occurs because the concentrations of dye and TEA are low decrease the light absorption so that we obtain high values of the optical thickness [9-10]. On the other hand the concentration of PVA is high in the holographic memories, what produces stronger layers but a small polymerization rate too [9].

Let us analyze the stability of recorded gratings low values of the polymerization rate and short exposure times create higher values of the residual monomer, and this can be considered as a drawback to the conservation of the information stored in this type of holographic memories. The evolution of the free monomer ($[M_0]$ see equation 2) as a function of time can be analyzed in figure 3. In this figure, after 20 s (the optimum exposure time for this type of gratings), the concentration of residual monomer is around 60 %. This high value is an important drawback for the stability of the information stored: under white light the monomers will react, but is a good advantage to record a lot of hologram using multiplexing methods.

Fig. 3. The normalized residual free monomer ($[M_0]$ see equation 2) as a function of the exposure time.

4. The angular response fitted by the algorithm proposed in reference [25] for a grating of $650 \mu\text{m}$ is plotted in figure

Fig. 4. The angular scan around the first Bragg angle is plotted for hobgraphic memories ($650\text{ }\mu\text{m}$ of thickness). The parameters obtained fitting the angular response are: optical thickness, $d = 650\text{ }\mu\text{m}$, average index modulation, $n_1 = 0.00035$, and exponential decay of the index modulation $\alpha = 0.005\text{ }\mu\text{m}^{-1}$.

4.2 POLYMER DIFFUSION

4.2.1 Thin layers

First let us analyze the results for the thin layers ($\sim 100\text{ }\mu\text{m}$ thick). In the particular composition used in this work the diffraction efficiency after 500 minutes is very low. Using equation (9) the figure 5 can be obtained. From the fitting of the angular responses we obtain that the polymer diffusivity is around $8.7 \times 10^{-15}\text{ cm}^2/\text{s}$, which is smaller than the value obtained by O'Neill et al for photopolymer films with similar thickness ($D \sim 3 \times 10^{-13}\text{ cm}^2/\text{s}$). The value of R^2 indicates the correlation and the quality of the fit, this value is not optimal because the layer is has not finished the drying process [9], thus, D is still not stabilized.

Fig. 5. The natural logarithm of the exponential decay of n_1 , modulation of the refractive index, as a function of time is plotted for thin layers. The correlation value for the line fit is also presented.

4.2.2 Thick layers

When the same study is applied to thick gratings, the value obtained for the polymer diffusion in thick layers is around $1.6 \times 10^{-14} \text{ cm}^2/\text{s}$. The value of R^2 is similar to that obtained in the thin layers case. This value of polymer diffusivity is higher than the one for thin layers but lower than the value obtained by O'Neill et al. An interesting situation occurs when in the thick layers composition a new component (liquid in laboratory conditions) is introduced. The function of this liquid is to increase the diffusivity of the material. In the experiment shown in this work the liquid used is glycerine, because it does not react with the other substances. In figure 6 the behaviour of the grating after the exposure can be observed. The value estimated of the polymer chains diffusivity is around $1.4 \times 10^{-13} \text{ cm}^2/\text{s}$. For this type of gratings the diffraction efficiency is already very low 25 minutes after the recording process. Analyzing these results it is clear that the diffusivity for the different substances is higher when a liquid is introduced in the polymer composition. In this case the value of the correlation coefficient is close to 1, what indicates the quality of the fit.

Fig. 6. The natural logarithm of the exponential decay of n_1 , modulation of the refractive index, as function of the time is plotted for thick layers. The correlation value for the line fit is also presented.

This diffusivity analysis is important to know the stability of the recorded holograms in dark conditions [18]. In general, the addition of a crosslinking monomer will stabilize the hologram. However in high fluid photopolymer systems the high values of diffraction efficiencies achieved during recording time can be loosen after.

4.3 MULTIPLEXED HOLOGRAMS

To study the usefulness of thick layers as holographic memories some holograms were recorded using Bragg's multiplexed method. To store seven holograms we tried a simple exposure schedule (a constant exposure time of 8 seconds), the results of the angular scan after the recording process are presented in figure 7. As can be seen the first recorded gratings smaller lower diffraction efficiency, that occurs because there is an induction period in the material. This is often related to the presence of inhibitors [30-31]. Trace quantities of transition elements that are present impurities in the monomer used in the composition of the photopolymer or the presence of the oxygen that is occluded in the material may deactivate the excited state of the dye or the polymer chains that are being formed [32]. Specifically, oxygen is known to be an inhibitor of radical polymerization because it bonds with the propagation radicals to form peroxide radicals, of low reactivity. Once the inhibitor has been used up, polymerization progresses normally. This phenomenon has to be considered when new exposure schedules are proposed to maximize the stored capacity of the material.

Fig. 7. Diffraction efficiency for each multiplexed grating as function of the rotation angle using a layer of $700\text{ }\mu\text{m}$ thick layers.

5. CONCLUSIONS

The validity of the first harmonic diffusion model to predict the behaviour of the gratings stored in holographic memories based in PVA/AA photopolymers is demonstrated. A first estimation of parameters that governs the formation of the hologram in photopolymers (monomer diffusivity and polymerization rate) has been done. These values are compared with the values for thin layers (around $100\text{ }\mu\text{m}$). The values of the polymerization rate in the holographic memories are low, what produces higher values of the residual monomer concentration. The values of the monomer diffusion are higher for thick layers.

As in the cases studied the crosslinker monomers are omitted in the material composition, the polymer diffusion can be observed and measured. Holographic memories present a higher polymer diffusion than thin layers. Nevertheless, in the bibliography similar materials with higher values of the polymer diffusion can be observed. The diffusion of the different species can be increased using an inert liquid in the polymer composition to better observe the polymer diffusion, which generates more accurate information about the behaviour of the thick photopolymer materials. In order to obtain more accurate information about the behaviour of the holographic memories based in PVA/AA photopolymers during the recording process it is necessary to employ models that take into account the attenuation of the intensity in depth.

When multiplexed gratings was recorded using same exposure time the first recorded gratins presents lower diffraction efficiency. This effect must be consider in the new exposure schedules are designed for PVA/AA holographic memories.

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