Study of influence of ACPA in holographic reflection gratings recorded in PVA/AA based photopolymer

Rosa Fuentes^{1*} Elena Fernández¹, Celia García¹, Augusto Beléndez²
and Inmaculada Pascual¹

¹Departamento de Óptica, Farmacología y Anatomía, Universidad de Alicante,
Apartado 99, E-03080 Alicante, Spain

²Departamento de Física, Ingeniería de Sistemas y Teoría de la Señal, Universidad de Alicante,
Apartado 99, E-03080 Alicante, Spain

*E-mail: fuentes@ua.es

ABSTRACT

The performance of a holographic data storage system depends to a great extent on the quality and the physical properties of the recording medium. The storage capabilities of photopolymer materials are under constant study and for some applications a high spatial frequency material is necessary. In this work, we focus on the study of the influence of 4,4'-Azobis(4-cyanopentanoic acid) ACPA on holographic reflection gratings recorded in a polyvinyl alcohol/acrylamide-based photopolymer with the aim of recording reflection gratings with a spatial frequency of up to 5000 lines/mm. The experimental procedure used to examine the high spatial frequency response of this material is explained and the experimental results presented.

Keywords: Photopolymers, data storage holographic recording materials, reflection holograms.

1. INTRODUCTION

Holography has become a valid alternative to classic data storage technologies, both magnetic and optical, because it provides high storage density (since holography permits information to be recorded in the three dimensions of a bulky storage medium rather than on its surface only), as well as fast access times.

The conventional optical memory technologies, like CD-ROM's and DVD's, are two-dimensional surface-storage techniques, and so have almost arrived at the limit of their capacity. For this reason, in recent years a lot of attention has been centred on three-dimensional holographic disks¹⁻³. Recently, many studies have focused on the characterization and optimization of thick holographic recording materials in order to obtain the maximum data storage capacity⁴⁻⁶ and transmission holographic memories have been designed⁷. However, in order to obtain more compact systems it would be interesting to design reflection holographic memories to which the current technology for reading CDs and DVDs, designed for reflection holograms, may be applied.

A number of organic photopolymer materials for holographic data storage whose mechanism of recording is based on photoinduced polymerization have been introduced in recent years. Advances achieved in the last couple of years have

been impressive but there is still a strong demand for new application-optimized media for a variety of storage systems under development.

Photopolymers have been considered some of the most attractive materials for use as holographic recording media. They have excellent holographic characteristics, such as high refractive index modulation^{8,9}, large dynamic range^{2,10,11}, good light sensitivity, real time image development, high optical quality and low cost, they have been used as the base of new 3-D holographic disks. In addition, their properties like energetic sensitivity or spectral sensitivity can be easily changed by modifying their composition^{8,10,12,13}.

The photopolymer is composed of acrylamide (AA) as the polymerizable monomer, triethanolamine (TEA) as radical generator, yellowish eosin (YE) as sensitizer, a binder of polyvinyl alcohol (PVA) and 4,4'-Azobis (4-cyanopentanoic acid)^{14,15} (ACPA) which has a dual role as initiator and chain transfer agent.

Reflection holograms with an acrylamide-based photopolymer have been investigated previously with different compositions of recording material¹⁶. This type of hologram has also been used for different applications such as a bit-format holographic data storage¹⁷ and visual indication of environmental humidity¹⁸.

In this work, we focus on the study of the influence of 4,4'-Azobis (4-cyanopentanoic acid) on holographic reflection gratings recorded in a polyvinyl alcohol/acrylamide-based photopolymer with the aim of recording reflection gratings with a spatial frequency of up to 5000 lines/mm with the best results.

2. EXPERIMENTAL

2.1 Composition of the recording material

In recording materials based on polyvinyl alcohol (PVA) as binder, a solution of PVA in water forms the matrix and this is used to prepare the mixture of acrylamide (AA) as the polymerizable monomer, triethanolamine (TEA) as radical generator, yellowish eosin (YE) as sensitizer, and 4,4'-Azobis (4-cyanopentanoic acid)^{14,15} (ACPA) which has a dual role as initiator and chain transfer agent (CTA) enabling the length of the polymer chains formed during the recording process to be regulated.

The mixture is usually deposited in a layer on a glass plate and after evaporation of part of the water, a solid plastic film is formed which constitutes the holographic recording material. The conventional method of depositing the layer consists in using an automatic depositor, which by means of a rod with a calibrated opening deposits the solution in a uniformly thin layer on a glass plate.

In this method, the solution is prepared under red light, in standard laboratory conditions ($T \approx 20^\circ\text{C}$ and relative humidity, $\text{RH} \approx 35\%$) and left in the dark for 18-20 hours to allow the water to evaporate. These conditions of drying time are optimized to obtain the maximum diffraction efficiency of the gratings. The thickness of the material is measured using an ultrasonic thickness gage and the resulting layers are about $70 \pm 10 \mu\text{m}$ thick. Table 1 shows the component concentrations of the photopolymer composition.

Table 1: Concentrations of the photopolymer composition

Component	Concentration
Polyvinylalcohol	8.26% w/v
Acrylamide	0.44M
Triethanolamine	0.2M
Yellowish Eosin	$2.4 \cdot 10^{-4}\text{M}$
4,4' - Azobis (4-pentanoic acid)	0.026M

2.2 Mechanism of hologram recording

A photoreduction reaction is produced when the material is illuminated with a light beam of 514 nm. The dye is excited to the high-energy state and then reacts with the coinitiator TEA, which is an electron donor, to produce a dye radical anion and a triethanolamine radical cation. The dye radical is not usually reactive enough to initiate polymerization but the TEA radical will react with an AA molecule and polymerization may then occur. The reaction takes place in a PVA matrix and we consider that the PVA matrix is inert. When the material is exposed to an interference fringe pattern, more monomer is polymerized and the amount of polymer formed increases with exposure.

In some papers¹⁹ the spatial frequency response of an acrylamide polyvinylalcohol-based photopolymer is improved by adding CTA, for example sodium formate. CTA is added in order to reduce the length of the polymer chains formed, since this agent has the ability to stop the growth of a chain. Thus the spatially periodic concentration distribution in the material formed by polymer chains can disperse more rapidly. The results for transmission holograms analyzing a range of spatial frequencies from 500 to 2750 lines/mm are shown in reference 19. It is proposed that addition of a CTA will decrease the average length of the polymer chains formed and hence localize the chain growth to the bright regions of the interference pattern. Thus, more but shorter polymer chains are expected to be formed. If a reduction in the non-local effect can be achieved, the potential improvements in high-spatial frequency material response will be significant.

In this work, the influence of 4,4'-Azobis (4-cyanopentanoic acid) as CTA on reflection holograms with a spatial frequency of up to 5000lines/mm is studied

2.4 Determination of the refractive index of the recording material

Determination of the refractive index is one of the first steps in the characterization of a holographic recording material. There are several methods for measuring the refractive index of thin films. In these methods, the reflectance of s and/or p polarization at several different angles of incidence, polarization conditions, or phase differences must be measured.

In our laboratory, we do not have instruments necessary to measure the refractive index of polymers at the wavelengths with which we work. For this reason, the procedure used here is the one described in reference 20. This is a technique to evaluate the refractive index of holographic recording materials which are in the form of a thin layer of film deposited on a thick glass plate. The experimental reflectance of p-polarized light is measured as a function of the incident angle, and the values of refractive index, thickness and absorption coefficient of the layer are obtained using the theoretical equation for reflectance. The fitting of the theoretical reflectance to the experimental data is carried out by a computer program.

The thin-film structure considered here is shown in Fig. 1. A single layer of absorbing material with complex refractive index $\mathbf{n}_2 = n_2 - i\alpha_2$ where n_2 is the refractive index and α_2 is the absorption coefficient, and thickness d_2 is located between two semiinfinite regions of nonabsorbing materials with refractive index n_1 and n_3 , respectively. A laser beam, linearly polarized parallel to the plane of incidence (p-polarization) of wavelength λ in region 1 is incident at angle θ_1 on the film.

The reflectance is calculated by:

$$R = \frac{|U_1 + U_2|^2}{|U_0|^2} \quad (1)$$

where U_0 is the amplitude of the incident wave and U_1 , U_2 the amplitudes of the reflected waves represented in Fig. 1.

$$\begin{aligned} U_1 &= r_{12}U_0 \\ U_2 &= t_{12}r_{23}t_{21}\exp(-\alpha_2 s)\exp(i\beta)U_0 \end{aligned} \quad (2)$$

where $\beta = \frac{4\pi}{\lambda}d_2n_2\cos\theta_2$ is the phase difference between the waves reflected from the first and the second surface

of the film layer.

With the amplitudes U_0, U_1, U_2 and Eq. (1), the reflectance is given by Eq. (3).

$$R = r_{12}^2 + (t_{12}r_{23}t_{21})^2 \exp(-\alpha_2 s) + 2r_{23}t_{12}r_{23}t_{21} \exp\left(-\frac{\alpha_2 s}{2}\right) \cos(\beta) \quad (3)$$

where $s = d_2 / \cos \theta_2$ and r_{ij} and t_{ij} are respectively the Fresnel amplitude reflection and transmission coefficients of i-j interfaces, for p-polarization.

We obtain R as the following function: $R = f(n_1, n_2, n_3, d_2, \theta_1, \lambda)$

We suppose that the wavelength λ , thickness d_2 and refractive indexes n_1 and n_3 of the incident medium and the substrate, respectively, are known. On the other hand, we experimentally measure the reflectance R as a function of the incident angle θ_1 . Then there are two unknown parameters, the refractive index n_2 and the absorption coefficient α_2 of the layer. We obtain these parameters by comparing the experimentally measured reflectances $R_{\text{exp}}(\theta_{1j})$ where $j=1,2,3,\dots,J$, and J is the number of angles of incidence in medium 1, with the calculated reflectances $R(n_2, \alpha_2, \theta_{1j})$. We obtain the values of the optical constants n_2 and α_2 for which the sum of the root mean square χ^2 is minimum, where χ^2 is given by Eq. (4):

$$\chi^2 = \frac{1}{J} \sum_{j=1}^J [R(n_2, \alpha_2, \theta_{1j}) - R_{\text{exp}}(\theta_{1j})]^2 \quad (4)$$

Fig.1: Reflections from a single layer of photopolymer on a transparent substrate.

To obtain the experimental reflectivity values as a function of the incident angle, the light from the laser is divided into two beams by means of a beam splitter. One of the beams impinges on the holographic plate, whereas the other beam impinges on a picowatt digital optical power meter to record information about the fluctuations in laser power during the measurements. A mirror is mounted on the holographic plate perpendicular to it. The light intensity reflected by the holographic plate/mirror system was measured by means of another optical power meter.

The plate was mounted on a high-precision motorized rotation stage that was controlled electronically using a point-to-point motion controller connected to a personal computer across an IEEE-488 interface. The rotating device had an angular resolution of 0.001° . The plate was illuminated with a laser beam polarized parallel to the plane of incidence. The reflected intensity was measured varying the angle of incidence between 40° and 60° at intervals of 0.1° .

To efficiently eliminate the back-reflected light produced at the glass-air interface at the rear side of the holographic plate a black self-adhesive poly vinyl chloride (PVC) masking tape was stuck onto the back surface of the plane parallel glass plate.

The losses of the different optical components of the measurement system were taken into account in the data analysis. However, instead of measuring the losses for each optical component as a function of the incident angle, the experimental setup was calibrated with a parallel glass window. This parallel window had a clear aperture 50.8 mm in diameter and 10 mm thick. The glass window was manufactured by Newport from BK7, grade A, fine annealed optical glass with a refractive index of $n=1.52049$ at wavelength of 473 nm, $n=1.52339$ at wavelength 514 nm and $n=1.51509$ at wavelength 633 nm.

To carry out the fitting of the theoretical reflectivities to the experimental data, it is necessary to know the refractive index n_3 of the glass substrate, it is $n_3 = 1.509$ at a wavelength of 473 nm, $n_3 = 1.506$ at a wavelength of 514 nm and $n_3=1.510$ at a wavelength of 633 nm

Fig. 2(a,b,c) shows the reflectance as a function of the angle of incidence for the holographic plates with the composition shown in Table 1. The light was polarized parallel to the plane of incidence and three different wavelengths were used. Comparing the experimentally measured reflectances with the calculated curves, we can see that the agreement is satisfactory.

Fig. 2: Measured (empty circle) and calculated (continuous line) reflectances for photopolymer versus angle of incidence. The light was polarized parallel to the plane of incidence and three different wavelengths were used (a) $\lambda=473$ nm, (b) $\lambda=514$ nm, (c) $\lambda=633$ nm.

Using this method we were able to properly adjust the refractive indexes values shown in Table 2. We also have obtained absorption coefficient values that fall between $0.007 \mu^{-1}$ and $0.09 \mu^{-1}$ for the wavelengths used.

Table 2: Best fitting of the refractive indexes of photopolymer when different wavelengths are used.

Wavelength (nm)	n
473	1.536±0.001
514	1.526±0.002
633	1.510±0.002

2.5 Holographic setup

The basic setup used to record holographic reflection gratings is shown in Fig. 3. Two beams from an Ar-ion laser overlap at a photosensitive plate, producing an appropriate interference pattern at the plane of the material due to the formation of polyacrylamide in the regions of constructive interference.

Holographic reflection gratings were recorded using the output beam from the laser which was split into two beams and then spatially filtered, using a microscope objective lens and a pinhole, and collimated to yield a plane-wave source of light at 514 nm. The diameter of these beams was 1.5 cm. The two laser beams were spatially overlapped at the recording medium but reached the opposite sides of the holographic plate with symmetrical geometry. Both beams impinged at an angle $\theta = 45.1^\circ$ with respect to normal incidence. The grating period established by the experimental recording condition was $\Lambda = 0.190 \mu\text{m}$, applying Eq. (5) (therefore the spatial frequency of the recorded reflection gratings was 5260 lines/mm) where λ is the wavelength of the laser light used for the recording of a hologram, Λ is the closest separation between the fringes of the interference pattern in the recording layer, n is the refractive index of the photopolymer for the recording wavelength, 1.526, which was measured using the method described in the previous subsection and θ is the incident angle in air.

$$\Lambda = \frac{\lambda}{2\sqrt{n^2 - \sin^2 \theta}} \quad (5)$$

Figure 3: Experimental setup: HP holographic plate BS beam splitter, Mi mirror, Li lens, Di, diaphragm, SFi, spatial filter.

4. RESULTS

4.1 Reconstruction of the reflection gratings in real-time

The recording material includes yellowish eosin as dye in its composition. This is not sensitive to red light emitted by the He-Ne laser ($\lambda=633\text{nm}$) but the latter could not be used as the reconstruction beam in real-time because from Eq. (5) when $\lambda = 633 \text{ nm}$, $n=1.510$ and $\Lambda=0.190\mu\text{m}$ there is no solution for the Bragg angle. However by using blue light $\lambda=473 \text{ nm}$ (diode pumped solid state laser) with $n=1.536$ and $\Lambda=0.190\mu\text{m}$ there is a solution and the Bragg angle is 64.3° . The problem is that the recording material is a few sensitive at this wavelength so the reconstruction beam was used at low intensity to prevent the recorded grating from being destroyed.

The holographic reflection gratings recorded using the setup shown in Fig. 3 were symmetrical gratings then the direction of the diffracted beam matches with the direction of Fresnel's reflection and this is taken into account when calculating the diffraction efficiency.

Fig. 4 shows the real-time diffraction efficiency DE during exposure. The maximum diffraction efficiency is 2.4% which is obtained with an exposure energy $E= 0.6 \text{ J/cm}^2$. The intensity of each writing laser beam was 20.1mW/cm^2 . It may be seen that when the exposure energy increases, the diffraction efficiency does not increase linearly but in ripples.

Figure 4: Diffraction efficiency as a function of exposure energy.

4.2 Spectral analysis of the reflection gratings

Each reflection grating was optically characterized by measuring its transmission spectrum. Measurements were not carried out in real-time. The dependence of transmittance on the wavelength was measured using a double-beam spectrophotometer; therefore the transmittance values take into account Fresnel's reflections. The plate was placed perpendicular to the beam of the spectrophotometer ($\theta=0^\circ$). The depth of the reflection grating peak on the transmittance curve allowed us to calculate the diffraction efficiency (DE) of the recorded reflection grating, applying the Eq. (6) :

$$DE = \frac{T_p - T_{pg}}{T_p} \quad (6)$$

where T_p is the transmittance of the photopolymer layer without recorded grating and T_{pg} is the transmittance of the photopolymer layer with recorded grating.

Dimensional changes in the recording medium can occur after recording. Unfortunately during the photopolymerization process, the monomer used usually undergoes volumetric changes; conversion of the monomer molecules into a polymer network is accompanied by close packing of the growing polymer chains and a subsequent reduction in

volume. This phenomenon, known as photopolymerization shrinkage is particularly relevant in the case of reflection gratings, when effective period of the grating recorded in the sample after the photopolymerization process, Λ_{exp} , becomes lower than that defined by the geometrical conditions of the recording Λ_{th} . Shrinkage of the material induces a change in the fringe spacing $\Delta\Lambda = \Lambda_{\text{th}} - \Lambda_{\text{exp}}$ in the thickness of the material. The ultimate effect is a relevant shift of the wavelength at which the reflection grating peak should appear on spectral analysis.

It is possible to introduce a parameter called optical shrinkage²¹ as:

$$S_{\text{opt}} = \frac{\lambda_{\text{th}} - \lambda_{\text{exp}}}{\lambda_{\text{th}}} \quad (7)$$

where λ_{th} is the theoretical wavelength at which the reflection grating peak should appear if there was no shrinkage and λ_{exp} is the experimental wavelength at which the reflection grating peak appears when there is shrinkage. This parameter represents the ratio between the final reflection wavelength displacement observed on spectral analysis $\Delta\lambda = (\lambda_{\text{th}} - \lambda_{\text{exp}})$ and the theoretical reflection wavelength of the grating if there was no shrinkage.

The angle of incidence in the spectrophotometer was fixed at $\theta=0^\circ$, whereas the wavelength was now the free parameter. Therefore applying Eq. (5) with this incident angle and with both the refractive index and the period of the grating fixed by the geometrical writing conditions, we obtain a theoretical value of $\lambda_{\text{th}}=580\text{nm}$, at which the reflection grating peak should occur.

Fig. 5 shows the transmittance versus wavelength when a reflection grating was recorded in a plate. As can be seen, it was possible to separate the dye absorption peak ($\lambda=527\text{ nm}$) from the reflection grating peak ($\lambda= 575\text{ nm}$). The exposure energy was $E=0.6\text{J/cm}^2$.

Fig.5: Transmittance versus wavelength when reflection grating was recorded in a plate.

Fig. 6 shows the transmittance of the plate versus wavelength, and formation of the reflection grating peak and the wavelength displacement, observed on spectral analysis, may be seen. The exposure energy was $E=0.6 \text{ J/cm}^2$.

Fig.6: Transmittance as a function of wavelength for reflection gratings recorded in a plate. The reflection grating peak is shown.

The maximum diffraction efficiency obtained was 4.3% and in this case the shift of wavelength was 5 nm corresponding to 0.9% optical shrinkage. In one of our previous studies¹⁶, sodium formate was used as CTA, and reflection gratings were recorded with 1.5% of diffraction efficiency and also 1.5% of optical shrinkage. Comparing the results in both cases, it may be seen that the results with ACPA are better because the diffraction efficiency is higher and the shrinkage is lower than with sodium formate.

4. CONCLUSIONS

The capacity of acrylamide-based photopolymers with 4,4'-Azobis (4-cyanopentanoic acid) ACPA, as chain transfer agent to record holographic reflection gratings has been investigated for optical storage applications. The high spatial resolution of the photopolymer material is demonstrated by recording reflection gratings with a spatial frequency of 5260 lines/mm. The refractive index of the recording material is determined using a method in which the reflectance of p-polarized light is measured as a function of the incident angle. The refractive index was 1.536, 1.526 and 1.510 when the wavelength of the light was 473 nm, 514 nm and 633 nm respectively. Reconstructing reflection gratings in real-time was possible with $\lambda=473 \text{ nm}$ and in this case a maximum diffraction efficiency of 2.4% was obtained. The spectral analysis of the reflection gratings showed that the maximum diffraction efficiency obtained was 4.3 % and the optical shrinkage was 0.9%. These results are better than those obtained in previous studies when sodium formate was used as chain transfer agent. Therefore using ACPA the results improve. Experimentally, a range of concentrations of the chain transfer agent should be examined so as to better understand the process which takes place in our photopolymer material when holographic reflections gratings with high spatial frequency are recorded.

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