



Characterization and comparison of different photopolymers for low spatial frequency recording



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ABSTRACT

Free-radical photopolymer materials can be fabricated using a wide range of monomers, binders, dyes, etc. It was shown that in some photopolymers the surface and the internal diffusion for acrylamide materials are very different and also it was demonstrated the viability of acrylamide materials to achieve 2π phase depth for low spatial diffractive optical elements. Building on the work developed previously, in this paper, a characterization and a comparison is carried out for three different photopolymers: cross-linked acrylamide based photopolymers, a biophotopol and an H-PDLC material. We have measured the motion of the components inside the material and through the surface using a new index matching component and through the surface. We have studied the introduction of different crosslinkers in order to increase the refractive index modulation and to reduce the thickness required to achieve a phase depth higher than 2π in the diffraction optical elements.

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1. Introduction

The correct fitting of the monomer diffusion was the aim of the several works carried out by different research groups, specially, for acrylamide based photopolymers. Recently it was proposed the using of index matching methods to decoupling the surface and internal changes. As discussed in a previous papers [1,2], the method proposed can be applied when new chemical compounds are introduced, even for different photopolymer families such as Biophotopolymers or HPDLC ones. The previous studies can be widely applied to analyze the viability of different photopolymers to record diffractive optical elements (DOEs) like lenses, axicons or blazed gratings [3–7]. Moreover the velocity of surface changes and the diffusion of the monomer inside the layer can be fitted too using the method described in [1,2]. The many applications of such free-radical photopolymer materials [8–12] make the analysis of the diffusion process very interesting.

In the commented manuscript [1], the influence of index matching to measure the rate of diffusion of monomer in an acrylamide material, AA, was developed. The results indicated that this type of material is a very appealing candidate to record DOEs when

the thickness is higher than 105 μm . Nevertheless large values of thickness occasionally produce an increase in the scattering and instabilities in the final hologram or diffractive optical elements stored [13]. Therefore on the first place, in this paper, we report the experiments for AA photopolymer, we show as the phase modulation can be increased avoiding layers thicker than 100 μm for AA materials. AA based photopolymer is one of the most studied in the literature [4,5,14–16]. The main problem of this type of photopolymers is the important toxicity of the main monomer, AA. Some efforts have been made to substitute this component in the chemical formulations so as to design highly compatible environmental photopolymers [17–20]: one of the greenest photopolymers is called Biophotopol [19], BIO. A photopolymer based in this material will be the second material analyzed in this work. In particular we study some different chemical variations of Biophotopol incorporating different crosslinker monomers in order to study the optimum thickness to fabricate 2π phase depth elements for each chemical variation of BIO photopolymers. The Biophotopol is a material with non-toxic nature and high environmental compatibility, wide dynamic range and high holographic sensitivity, together with its self-processing nature and low cost of production; make it a potentially excellent candidate for applications such as holographic sensors [21], diffractive optics or data storage [22].

On the last place we have evaluated a photopolymer with dispersed liquid crystal molecules, H-PDLC [23–27], for recording

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DOEs in the low spatial frequency range. Nowadays new components have been included in the standard formulation of classical photopolymer systems to arise unexpected properties, some examples of these components are nano-particles or dispersed liquid crystal molecules (LC), by combining polymers and dispersed liquid crystals a new spectrum of interesting applications was opened. The opportunity to design new switchable or tunable holographic displays due to phase separation between photopolymer and LC has attracted great attention. For example, the ability to control the diffraction efficiency of holographic optical elements by applying an electric field leads to the possibility of using holographic optical elements in dynamic applications for agile beam steering, nonlinear optics and optical switching devices [23–25]. This family of polymers is known as H-PDLC. In the chemical formulation multifunctional monomers are used in order to induce a phase separation process (PIPS) in which the liquid crystal molecules diffuse to dark zones in the diffraction grating where, once the corresponding element has been recorded, they can be oriented by means of an electric field. The orientation 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 electronic means. In this manner, it is possible to make dynamic devices such as tunable-focus lenses, sensors, phase modulators or prism gratings. H-PDLCs are characterized by high values of refractive index modulation that provide diffraction efficiencies close to 100% for an optical thickness around 10 μm , and the main drawback is the high value of scattering. The novelty presented here is the application of H-PDLCs to fabricate low spatial frequency EOD. To obtain this goal, on the last part of the paper we characterize the observable monomer diffusion, refractive index modulation and the optimum thickness required to fabricate 2π phase depth DOE in a standard H-PDLC photopolymer [27]. To summarize, along this work, we report the complete characterization to produce optimized DOE in three different photopolymer families.

Let us introduce Fig. 1, diffraction efficiencies, DEs, of the main diffracted orders for phase thin gratings as a function of the phase depth, thus enabling readers to evaluate directly the phase modulation achieved just comparing the experimental results to this figure.

2. Material and preparation

As we have said in a previous paper the standard photopolymer is composed of AA as polymerizable monomer, triethanolamine (TEA) as coinitiator and plasticizer, yellowish eosin (YE) as dye,

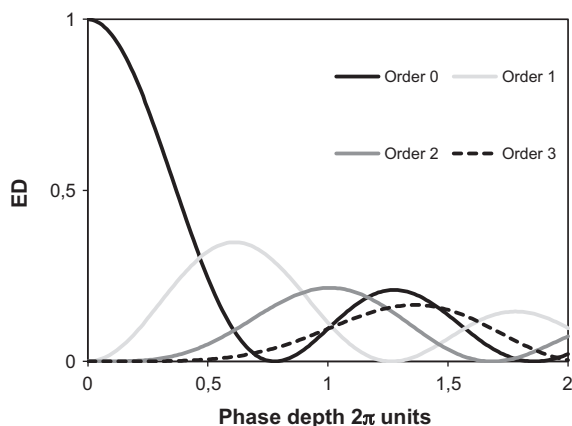


Fig. 1. Diffraction efficiency for the first four orders for a sinusoidal grating as a function of the phase depth.

PVA as binder and a small proportion of water as additional plasticizer. It may also contain N,N'-methylene-bis-acrylamide (BMA) as crosslinking monomer. AA and BMA are toxic monomers, the former more than the latter. YE also introduces problems due to the four Br atoms in its molecule, but it is clearly the less toxic component. In this sense in the chemical formulation analyzed in this work we keep YE as a dye, to obtain a better comparison of the monomer importance.

For monomer substitution it is necessary to use another vinyl monomer that is less toxic than AA. We used sodium acrylate, NaAO, prepared in situ by means of a one-pot reaction with acrylic acid and sodium hydroxide in the prepolymer solution used to prepare the layers. The toxicity of sodium acrylate is lower than that of AA. We replaced AA by NaAO in the photopolymers BIO.

BMA, a known also toxic, but clearly less than AA, is the crosslinker usually used in a standard AA-based photopolymer. We used N,N'-(1,2-dihydroxyethylene) bisacrylamide (DHEBA) in biophotopol as an alternative to BMA. DHEBA has occasionally been used in hydrophilic photopolymers due to its good solubility in water. This molecule is suitable for the new photopolymer because its two hydroxyl groups are compatible with the structure of sodium polyacrylate generated in the photopolymerization. In this manner, hydrogen bonds may be formed with the PVA binder and TEA and water plasticizers. Although there are no studies in the literature suggesting that this substance is toxic, future research may show it has a certain level of toxicity; however, it is less toxic than BMA.

Biophotopol has also a hydrophilic binder as the AA-based standard photopolymer and this implies that during its production the main solvent used is water. Biophotopol does not use any additional co-solvent. Any products and devices made with this photopolymer can also be eliminated, once their useful life is over, by dissolving in water. Therefore, this material has an advantage over hydrophobic photopolymers because it avoids the use of petroleum-based solvents, which are toxic and flammable.

Analyzed solutions for AA and Bio, whose compositions can be seen in Table 1, are deposited, using the force of gravity, on a glass substrate (25 cm \times 20 cm), and left in the dark (RH = 40–45%, $T = 20\text{--}23\text{ }^\circ\text{C}$). When part of the water has evaporated (after about 36 h), the layer has enough mechanical resistance and can be cut without deforming. The final “solid” film has a physical thickness around $90 \pm 5\ \mu\text{m}$. This final thickness can be modified changing the quantity of the syrup deposited on the glass with the same method of preparation thicknesses between 30 and 250 μm had been successfully fabricated [26].

To fabricate H-PDLC photopolymer, the monomer used is dipentaerythritol penta/hexa-acrylate (DPHPA) with a refractive index $n = 1.490$. We use the nematic liquid crystal, licristal[®] BLO36 from Merck. It is a mixture of 4-cyanobiphenyls with alkyl chains of different lengths. It has an ordinary refractive index $n_0 = 1.5270$, and a difference between extraordinary and ordinary index $\Delta n = 0.2670$ [26]. There is a difference of 0.037 between the ordinary refractive index of the liquid crystal and that of the monomer. The liquid crystal concentration was set at 28 wt% as the starting point for

Table 1
Composition of photopolymer AA and BIO, liquid solutions.

Composition name	AA1	AA2	BIO1	BIO2	BIO3
NaAO (g)	–	–	1	1.5	1.5
TEA (ml)	2.0	2.0	2	3.0	3.0
PVA (ml) (8% w/v)	25	25	30	25	25
YE (0.8% w/v) (ml)	0.6	0.6	0.84	0.6	0.6
DHEBA (g)	–	–	–	0.18	–
BMA (g)	–	0.2	–	–	0.18
AA (g)	0.96	0.84	–	–	–
Thickness ± 4 (μm)	90	90	90	90	90

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