

Effect of film thickness on the performance of photopolymers as holographic recording materials

Joel E. Boyd, Timothy J. Trentler, Rajeev K. Wahi, Yadira I. Vega-Cantu, and Vicki L. Colvin

An important issue in developing applications for photopolymers in holography is the effect of film thickness on recording properties. Now it is possible to create these samples with a much wider range of thickness ($d = 20\text{--}1400\ \mu\text{m}$) than was previously available. We exploit these recent advances in photopolymer processing to systematically evaluate how the dynamic range of a photopolymer depends on its thickness. The results illustrate that sample performance increases linearly with thickness as predicted by standard models of volume holography. However, above a critical thickness sample performance degrades, and the angular response of recorded plane-wave holograms shows evidence of grating curvature. These distortions are likely the result of photopolymer shrinkage, which in thicker samples occurs in a nonuniform fashion. This problem limits the performance of these photopolymers and is likely to be an issue for any photopolymer that undergoes comparable polymerization shrinkage.

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1. Introduction

Photopolymers are photoactive materials capable of recording spatial variations in light intensity through irreversible changes in their refractive index. When exposed to interfering light beams, as in hologram formation, the systems undergo photopolymerization reactions that encode the fine spatial interference pattern of the incident light. Both the phase and the amplitude information needed for hologram recreation are stored. Photopolymers have been exploited in a variety of applications requiring versatile holographic storage media, such as heads-up displays,¹ data storage,^{2,3} holographic optical elements,^{4–7} and waveguides.⁸ Although other holographic storage materials, particularly inorganic photorefractive crystals,⁹ have been used to develop these technologies, it is widely believed that the high sensitivity, low cost, and versatility of photopolymeric media would enable more widespread commercial applications of holography.^{8,10,11} Until recently, however, the routine use of photopolymers as holographic recording media has been hampered by pho-

topolymer shrinkage, which distorts the patterns of the recorded holograms. Another important issue has been the limited thickness of the materials, which leads to lower dynamic range and greater angular bandwidth than with thicker materials.

Recently, advances in the chemistry and in the processing of photopolymers have enabled the production of photopolymer samples with significantly lower shrinkage and greater thickness.^{11–21} For many applications the availability of photopolymers with film thicknesses in excess of $250\ \mu\text{m}$ is particularly important, since it is expected that a hologram's diffraction efficiency will increase quadratically with material thickness^{22,23} unless it is limited by high sample optical absorption.^{24–26} Thus, for a grating of a given contrast, thicker samples allow for higher diffraction efficiencies per hologram. This enables more holograms to be stored in a given area and increases the storage capacity of the material. Recent reports of high-performance photopolymers illustrate the potential of these new systems^{12,16,20,21}; however, the recording behavior of photopolymers has not been explored over the wide range of thickness now available. In this study we systematically evaluate how an important measure of photopolymer dynamic range, the $M/\#$,²⁷ depends on film thickness. These data illustrate that an issue specific to photopolymers, namely, the dimensional changes that occur during photopolymerization, reduces the single-hologram diffraction efficiency in thicker pho-

The authors are with the Rice Quantum Institute and the Department of Chemistry—MS 60, Rice University, Houston, Texas 77005. V. L. Colvin's email address is colvin@ruf.rice.edu.

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topolymers and limits their overall recording capacity.

2. Experiment

This study is made possible by the development of a new multicomponent acrylate photopolymer; the polymerization chemistry, namely, free-radical polymerization of acrylates and vinyl monomers, as well as the mechanism of grating formation, is quite similar to that found in commercial photopolymers.^{12,14,28} Moreover, it is a widely studied system whose net sensitivity, absorption properties, hologram formation mechanism, photopolymerization shrinkage, and performance have been characterized in prior studies.^{11–15} Exposure of a hologram ($\lambda = 532$ nm) initiates a spatially varying photopolymerization reaction that results in a periodic variation in the refractive index of the material ($\Delta n \sim 0.001$). For the thicker samples used in these experiments a single grating of this size is large enough to result in hologram diffraction efficiencies approaching 100%, and strong interference between the diffracted and the incident light (overmodulation) complicates the assessment of the material. For this reason, sample performance is measured by recording of many smaller holograms ($\Delta n \sim 3.5 \times 10^{-5}$) at different angles; the sum of the square roots of the diffraction efficiencies, referred to as $M/\#$, can then be calculated. This parameter, rather than the net refractive-index contrast in the material (Δn), is a more appropriate figure of merit in gauging the performance of thicker holographic recording media.^{29,30}

Of particular importance for this research is that both sample thickness and shrinkage during hologram recording can be systematically controlled in this photopolymer. Unlike other materials in which the photoactive monomer is present in an inert matrix, all the chemical components in this system are photoactive and initially liquid. Thus sample thickness can be dictated through the use of spacers that define the separation between two glass plates that encase the unreacted monomer. Before recording, this viscous fluid must be exposed to a flood illumination to create a solid and stable matrix. Variations in the duration of this preexposure treatment provide a method for controlling the amount of monomer available for hologram formation and thus the amount of shrinkage during hologram development.

Samples are prepared, and plane-wave holograms are recorded and read in accordance with established procedures.^{12,31} The materials are formulated as disks approximately 1 cm in diameter and 20–1400 μm thick. Two different formulations with different composition, performance, and sensitivity were studied—one containing N-vinylcarbazole (NVC), and the other containing N-vinyl-2-pyrrolidinone (NVP). Chemical compositions for each photopolymer are similar to those found in Ref. 12 (samples designated NVC and VNVP). They are partially polymerized between glass slides that function to exclude oxygen (a free-radical polymerization inhibitor) and to provide substrates of high optical quality.

The material dynamic range is measured by angle multiplexing of 20 holograms to determine the $M/\#$. A sample rotation of 2° between holograms prevents any appreciable interhologram coupling that might distort the holograms or reduce diffraction efficiencies.^{32,33} The holograms are recorded at 532 nm, and the angle of intersection of the writing beams is 40° external to the sample. The sample is again exposed to flood illumination after the final hologram is written to assure completion of photopolymerization and render the sample optically clear. Holograms are read out with an angular resolution of 0.002° .

Although this photopolymer material has the resolution required to record images,¹³ plane-wave holograms are employed here for two reasons. First, we can measure the precise thickness of the sample by determining the full width at half-height of the 0° hologram and comparing it with a well-established model.^{22,31} Thickness determined in this way correlates well with the results obtained by use of mechanical calipers for samples between 100 μm and the thickness at which the $M/\#$ deviates from linearity; however, for samples thicker than this critical measurement, mechanical calipers provide a more reliable measure of film thickness. The second reason for using plane-wave holograms is that they provide a quantitative measure of the photopolymerization shrinkage. When this shrinkage occurs uniformly, and perpendicular to the glass substrates, the angular positions of multiplexed holograms are shifted. This effect is common to many photopolymer materials and demonstrates that material shrinkage manifests itself in similar ways in different photopolymer systems. The magnitude of these shifts can be related to the net percentage of this transverse shrinkage.^{10,34} For the data shown in this study, this shrinkage is calculated to be $1.5\% \pm 0.5\%$ over the range of film thickness studied.

3. Results and Discussion

One of the primary motivations for creating thicker photopolymers is that single-hologram diffraction efficiencies are expected to increase substantially with thickness. Figure 1 demonstrates this trend experimentally. The $M/\#$ of these samples increases linearly with thickness from less than 20 μm to more than 500 μm in the NVC material and to as much as 300 μm in the NVP material. We can understand this behavior by using the standard model for volume holography, which describes the strength of scattering from a spatially periodic dielectric variation.²² For perfect gratings the diffraction efficiency is predicted to scale as the square of the material thickness; thus the $M/\#$ depends linearly on sample thickness. Once sample thickness exceeds approximately 520 μm in the NVC system and 310 μm in the NVP, linear scaling is no longer observed. Attempts at more precise definition of the critical thickness at which the deviation from linear behavior occurs indicate that other contributing factors, such as the thickness of the glass substrates and the proximity of

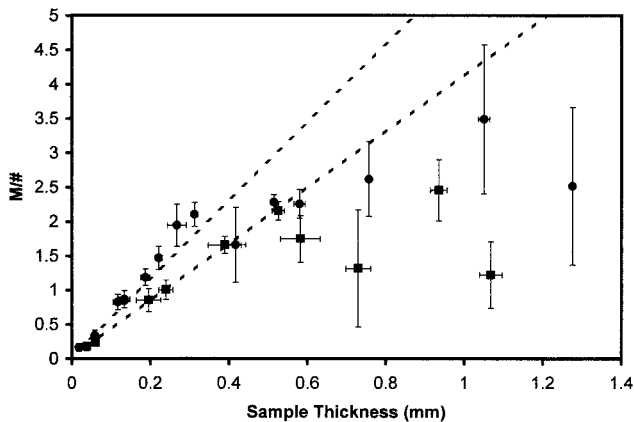


Fig. 1. $M/\#$ versus sample thickness. $M/\#$'s are scaled to correct for small deviations in preexposure conditions when required. Sample thickness was determined by the full width at half-height of the 0° hologram for samples that have not deviated from the linear $M/\#$ versus thickness relationship, and by mechanical calipers for thicker samples. Points indicated by squares refer to the photopolymer NVC, and points indicated by circles refer to the photopolymer NVP. The displayed line (slope, 4.13; y intercept, 0.017) represents a best fit for samples with thickness as great as $520\ \mu\text{m}$ for the NVC samples, and the displayed line (slope, 6.87; y intercept, -0.0192) is for the NVP samples with thickness as great as $320\ \mu\text{m}$. Each point represents an average of several samples, and the error bars depict one standard deviation.

the hologram to the sample edge, are important for sampling performance in thicker photopolymers.

An examination of the holograms written in these thicker samples illustrates that they are distorted in ways that are characteristic of holograms with curved (chirped) gratings. Figure 2 shows the angle-dependent diffraction efficiency of holograms in three different NVC samples of varying thickness. Although each sample was used to record 20 angularly multiplexed holograms, only one, written at 0° sample rotation, is shown for clarity. For the thinner sample [Fig. 2(a)] the response function is symmetric; moreover, with the known thickness ($200\ \mu\text{m}$) it is possible to predict the bandwidth of the angular response curve from theory and compare it with the experimental data. (In this case we define bandwidth as the angular separation between the first null points.) The vertical dashed lines represent these predictions and agree with the data. Figure 2(b) depicts the angular response of a hologram in a thicker sample ($560\ \mu\text{m}$); in contrast to Fig. 2(a), the data exhibit some asymmetry but still have a bandwidth in reasonable agreement with theory. The angular response curve for the thickest sample ($950\ \mu\text{m}$) is much more distorted [Fig. 2(c)], and its bandwidth exceeds the theoretical prediction. The general features of Fig. 2(c) (e.g., loss of symmetry, lack of well-defined null points, and broad width) are all consistent with the reduced scattering efficiency from chirped holograms.³⁵⁻³⁹

There are a number of possible origins for the emergence of chirped holograms in thicker photopolymers, including high sample absorbance, poor beam

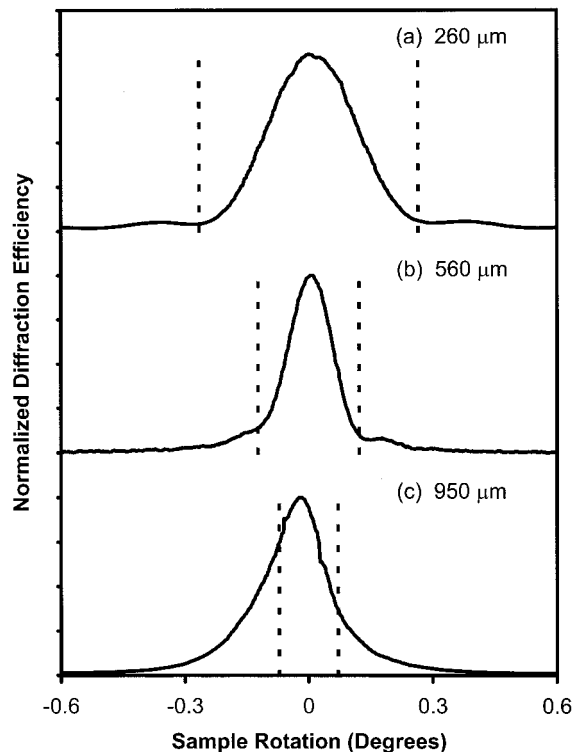


Fig. 2. Angular response curves for 0° holograms from multiplexed holograms recorded in photopolymers of varying thickness. In these data the vertical dashed lines represent the location of the first nulls of the response functions calculated following Kogelnik²² using thickness measured by mechanical calipers. [(a) $260\text{-}\mu\text{m}$ sample, (b) $560\text{-}\mu\text{m}$ sample, (c) $950\text{-}\mu\text{m}$ sample]. The excessive bandwidth and asymmetry caused by shrinkage is apparent in these data. (a) and (b) are from NVC samples that did fall on the regression line from Fig. 1, and (c) was well below its projected $M/\#$ value.

collimation during exposure, and photopolymerization shrinkage. To evaluate the relative importance of these issues, a weak single hologram is recorded in 600- and $200\text{-}\mu\text{m}$ NVC samples. The exposure levels for the recording, as well as the subsequent readout, are kept low so as to ensure that the material experiences little polymerization shrinkage. As can be seen in Fig. 3 (solid curves), the resulting holograms display angular response curves with narrow bandwidth and the expected oscillatory wings. The lack of chirp in these holograms illustrates that distortions do not arise during the exposure process itself. The samples are then subjected to a uniform light exposure during which the material becomes fully photopolymerized, a process that causes both film shrinkage and bleaching.¹² After this treatment [Fig. 3(a), dashed curve], the hologram written in the $600\text{-}\mu\text{m}$ sample is distorted with the reduced diffraction efficiency and broadened angular response characteristic of a curved grating. The $200\text{-}\mu\text{m}$ sample identically exposed and flood cured shows no evidence of hologram chirp [Fig. 3(b), dashed curves].

These results indicate that hologram chirp is

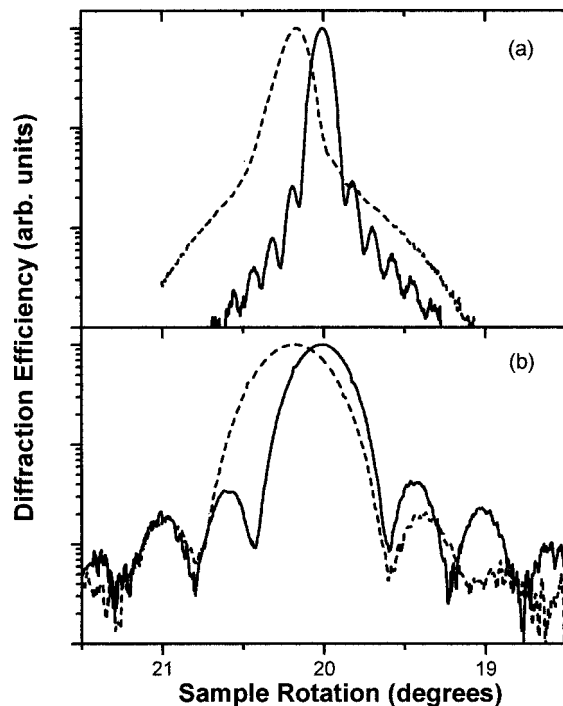


Fig. 3. Angular response curves for weak single holograms before and after flood exposure. Single holograms were written in two NVC samples of different thickness. These holograms were written at 20° sample rotation and read out before (solid curve) and after (dashed curve) complete flood cure. The sample exposures during the write process were 68 mJ as compared with the total of 2260 mJ normally used to react all monomer. Each readout provided an additional 34 mJ of exposure. These data are displayed with diffraction efficiency on a log scale to emphasize changes in the hologram response, specifically the nature of the changes to the sidelobes that occur with shrinkage. (a) A weak hologram recorded at 60° in a $600\text{-}\mu\text{m}$ -thick sample and read immediately after recording (solid curve) and read again after flood cure (dashed curve). (b) A weak hologram recorded at 20° in a $200\text{-}\mu\text{m}$ -thick sample read prior to flood cure (solid curve) and read again after a uniform flood cure (dashed curve).

present only *after* the flood exposure and only in thicker samples. Since the change in material optical density at the writing wavelength during flood cure is small, from 0.16 to 0.00 in a $200\text{-}\mu\text{m}$ sample, it is unlikely that sample absorption effects are responsible for the hologram distortion described by Lessard and co-workers.^{32,33,40} Holograms recorded in other thick photopolymers, with even smaller optical densities, show the same distorted response curves. Material shrinkage during the flood exposure provides a better explanation for the observed phenomena. Changes in the samples' shape brought about by shrinkage could cause holograms to distort; indeed, in thin photographic emulsions, which experience as much as 20% shrinkage during processing, shrinkage-induced chirp is a common problem.³⁷ Moreover, hologram chirp is more readily apparent in photopolymers, which receive less preexposure and thus experience more shrinkage during hologram recording. One important feature

of the sample behavior is that, although polymerization shrinkage occurs in both thin and thick samples, it leads to chirped holograms only in thicker materials.

Apparently, thicker samples undergo different dimensional changes from those in thinner samples as a result of the shrinkage process. For example, uniform shifts of the peak location (Fig. 3, $\sim 0.17^\circ$) are observed in both samples after flood cure; as discussed above, these rotations are known to arise from shrinkage along the direction perpendicular to the glass substrates.^{10,34} The rotations are of the same magnitude in thick and in thin samples and correspond to a decrease of approximately 1.5% in the sample thickness. As was shown recently, this type of shrinkage can be counteracted during hologram writing in some instances.⁶ A more serious problem, however, is the distortion of the peak shape observed in the $950\text{-}\mu\text{m}$ sample after a flood cure [Fig. 3(c)]. These changes are the hallmarks of nonuniform distortions in the fringes of plane-wave holograms. Whereas transverse shrinkage has little effect on the measured diffraction efficiency, chirped holograms experience a substantial decrease in scattering efficiency.³⁸

These data suggest that in thicker samples the dimensional changes brought about by photopolymerization occur both *parallel and perpendicular* to the glass substrates. If completely unconstrained the photopolymer would shrink uniformly in all dimensions; however, in thinner samples the adhesion of the polymer to the rigid glass substrates provides a force that opposes any changes in the polymer's shape in the direction parallel to the glass. Thus, the substrate-photopolymer interface acts to restrain shrinkage and effectively places the polymer under tensile stress parallel to the plates. Our results suggest that, as samples become thicker, the force required for maintaining the shape of the polymer grows sharply, whereas the interfacial constraints remain roughly constant. At some critical thickness, sample distortion in the plane of the plates becomes possible, leading to either delamination of the sample from the plates, curvature of the plates themselves (lensing), or nonuniform shrinkage where the middle section of the photopolymer contracts while the edges remain fixed by the substrate.

Not surprisingly, the relative magnitude of these three effects is sensitive to the thickness of the glass supports. If very thin glass slides ($200\text{ }\mu\text{m}$) are used as substrates, then samples exhibit strong lensing after flood illumination. In this instance the force required for bending the substrates is minimal, and the samples are able to shrink to some extent in all dimensions. If thicker glass substrates ($\sim 3\text{ mm}$) are employed to prevent bending of the glass substrates, then the films delaminate from the glass. Here the adhesive force between the polymer and the glass is not sufficient to hold the polymer under tension parallel to the plates. For the intermediate glass thickness used in this study ($d = 1\text{ mm}$) nonuniform shrinkage rather than sample delamination or lens-

ing is observed. The center of the sample contracts while the sample edges remain fixed by the substrate. Such a distortion in the shape of the sample could explain the observed angular response functions after flood cure. If these nonuniform changes in sample shape are indeed occurring in these materials, then the shear strength of the photopolymer would be expected to influence sample performance. This is consistent with recent research³⁸ illustrating that increasing the tensile strength of photopolymer materials ($d \sim 200 \mu\text{m}$) can reduce the magnitude of nonuniform shrinkage and the resulting hologram chirp.

A quantitative analysis of the mechanical forces and constraints in this sample geometry could provide insight into the complex interplay between photopolymer thickness, substrate thickness, sample shrinkage, and glass-photopolymer adhesion. Such analyses have been performed on somewhat similar systems, namely, polymeric coatings on glass fibers subject to various mechanical and thermal stresses.^{41,42} In future studies these models will be adapted to shrinkage-induced stresses in photopolymer disks of the type studied here.

4. Conclusions

In this study we have illustrated that, as photopolymers become thicker, their sensitivity to polymerization shrinkage increases. For this reason the theoretical scaling of dynamic range with sample thickness cannot be assumed for this class of materials without experimental verification. It is important to note that this result was observed in two different photopolymer formulations with different compositions and performance characteristics and is likely to be observed with any photopolymer that undergoes photopolymerization shrinkage. Relative to other commercially available photopolymers, which exhibit transverse shrinkage of $\sim 5\%$,^{2,3,6} the shrinkage in this system is quite small ($\sim 1.5\%$). The fact that even this low level of shrinkage has such a striking effect on photopolymer performance illustrates the importance of this issue in the design of practical systems. Some new photopolymer formulations have been reported that undergo little or no polymerization shrinkage.^{16,20} In the current study we have suggested that the advantages provided by the use of thicker samples for holographic applications could be fully realized in lower shrinkage materials that are not limited in thickness by other concerns.

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