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# Holographic Analysis of Photopolymers

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## ABSTRACT

Two-beam holographic exposure and subsequent monitoring of the time-dependent first-order Bragg diffraction is a common method for investigating the refractive index response of holographic photopolymers for a range of input writing conditions. The experimental set up is straightforward, and Kogelnik's well-known coupled wave theory (CWT)[1] can be used to separate measurements of the change in index of refraction ( $\Delta n$ ) and the thickness of transmission and reflection holograms. However, CWT assumes that the hologram is written and read out with a plane wave and that the hologram is uniform in both the transverse and depth dimensions, assumptions that are rarely valid in practical holographic testing. The effect of deviations from these assumptions on the measured thickness and  $\Delta n$  become more pronounced for over-modulated exposures. As commercial and research polymers reach refractive index modulations on the order of  $10^{-2}$ , even relatively thin ( $< 20 \mu\text{m}$  thick) transmission volume holograms become over-modulated. Peak  $\Delta n$  measurements for material analysis must be carefully evaluated in this regime. We present a study of the effects of the finite Gaussian write and read beams on the CWT analysis of photopolymer materials and discuss what intuition this can give us about the effect other non-uniformities, such as mechanical stresses and significant absorption of the write beam, will have on the analysis of the maximum attainable refractive index in a material system. We use this analysis to study a model high  $\Delta n$  two-stage photopolymer holographic material using both transmission and reflection holograms.

**Keywords:** holography, photopolymer, coupled wave theory

## 1. INTRODUCTION

Photopolymers are attractive recording materials for applications in holography ranging from solar energy and security to holographic optical elements (HOEs) [2,3,4,5]. In particular, HOEs are an emerging interest due to the advent of Augmented Reality (AR) consumer devices and their associated stringent constraints and requirements. Specifically, the use of photopolymers as materials for HOEs is gaining traction due to the attractive features of photopolymers such as: cost-effective scalability, self-processing nature, design tunability, optical clarity and high refractive index modulation. A major design consideration for the practical use of photopolymers as recording materials for HOEs is the achievable refractive index modulation ( $\Delta n$ ). With higher  $\Delta n$ , high diffraction efficiency can be achieved in thin ( $< 20 \mu\text{m}$ ) films, resulting in flexible and lightweight optical elements. Thin samples are also less likely to suffer from mechanical stresses due to shrinkage and diffusion of the mobile writing monomer or absorption losses through the depth of the samples.

Holographic plane wave testers are a common method for evaluating the refractive index response of holographic photopolymers for improving materials design. The experimental set up is straightforward, and Kogelnik's well-known coupled wave theory [1] can be used to separate measurements of the change in index of refraction ( $\Delta n$ ) and the thickness of a transmission hologram. However, practical difficulties in experimental holographic systems, such as the finite size of Gaussian read and write beams as well as non-uniformities in the resulting hologram due to mechanical stress or absorption in the sample can lead to incorrect analysis of the maximum  $\Delta n$  achievable in a given material. These effects become pronounced as the hologram efficiency approaches 100% and are critical for over-modulated exposures.

We present a study of the effects of the finite size and non-uniform intensity profile of the write and read Gaussian beams used in most plane wave testers. This study reveals how non-ideal recording and readout conditions result in errors in the extracted material performance. We report the holographic characterization of a simple two-stage chemistry formulation capable of a  $\Delta n$  of at least 0.01 with a spatial frequency response capable of writing reflection holograms in the blue. Our formulation comprises a relatively low refractive index thermoset polyurethane network and a second stage high refractive index acrylate monomer that is free to diffuse through the polyurethane matrix. Upon exposure to an

interference pattern and subsequent photopolymerization, the diffusion of the acrylate monomer from the dark to bright areas results in a permanent refractive index change. While this diffusion of monomer can result in high refractive index changes, the movement of mobile species and subsequent swelling and shrinkage of the material can cause non-uniformities in the hologram recorded into the material. We discuss the effects of these non-uniformities, we fit the over-modulated transmission holograms resulting from high  $\Delta n$ , and present the real-time growth of a high diffraction efficiency reflection hologram.

## 2. NON-UNIFORM HOLOGRAM ANALYSIS

### 2.1 Gaussian write & read beams

Characterization of the refractive index response of holographic photopolymers as a function of writing intensity, exposure time, and material design properties is often done using a holographic plane wave tester. A laser of the appropriate wavelength to initiate photopolymerization is split into two and interfered at the sample plane to write a transmission hologram with a pitch on the order of 1  $\mu\text{m}$ , and the thickness of the sample sufficient to result in a Bragg hologram [6]. The hologram is analyzed during recording using a longer wavelength to which the material is not sensitive and the diffraction efficiency is measured as a function of input angle. Kogelnik's CWT is often used to fit this data according to [6]

$$\eta = \frac{\sin^2(\Phi\sqrt{1+X^2/\Phi^2})}{1+X^2/\Phi^2} \quad (1)$$

$$\Phi = \frac{\pi n_1 d}{\lambda \cos\theta}$$

$$X = \frac{Kd}{2\cos\theta} \left[ \Delta\theta \cos(\theta - \psi) - \frac{\Delta\lambda}{2\Lambda} \right]$$

where  $\eta$  is the diffraction efficiency of the hologram and depends on  $d$ , the thickness of the holograms,  $n_1$ , the peak to mean index modulation,  $\lambda$ , the wavelength used to read the hologram,  $\theta$ , the Bragg angle at which the largest diffraction efficiency occurs,  $K = 2\pi/\Lambda$ , where  $\Lambda$  is the period of the hologram,  $\Delta\theta$ , the difference between the input angle and the Bragg angle, and  $\Delta\lambda$ , the difference between the current read wavelength and the wavelength corresponding the Bragg angle. All quantities are defined within the material. For transmission holograms, we generally keep  $\Delta\lambda = 0$ , and scan over many input angles,  $\Delta\theta$ . The diffraction efficiency is measured as a function of input angle and the data is fit to give  $n_1$  and  $d$  for a measured hologram.

The assumptions made in this set of equations is that the hologram was written by two interfering plane waves with infinite extent and uniform intensity, there is no loss of intensity through the depth of the writing sample, the read beam is also a plane wave, and the recorded hologram is an exact replica of the interference pattern created by the two plane waves used to write it. While these assumptions are clearly never exactly true, it is important to understand under what conditions they are approximately valid, and under what conditions the assumptions break down sufficiently that this fit no longer giving us an accurate measurement of  $n_1$  and  $d$ .

Real plane wave testers use Gaussian laser beams to write the holograms and Gaussian beams to read the holograms. Depending on the complexity of the system, some beam shaping might be done to make the write beam more uniform, and the read beam is generally smaller than the write beam to interrogate the center of the recorded hologram. We investigate here the effect of the Gaussian intensity profile of the write beam. This will result, for a material with a linear response, in an index profile that is highest in the center of the hologram and goes to zero at the edges. Ideally, the read beam would be infinitely small so that it would only measure the center of the hologram with the peak in  $\Delta n$ . However, as the read beam becomes smaller, the divergence of the beam increases. When this divergence approaches the same angular scale as the angular selectivity of the hologram, this prevents an accurate Bragg selectivity curve from being measured [7]. This can be used to do real time measurements of the hologram [8], but in general is not desired. As the read beam increases in size, the read beam begins to measure not just the peak  $\Delta n$  of the hologram, but also the lower  $\Delta n$  of the edges of the hologram. If the read beam is not well aligned to the center of the hologram, it may not measure the peak  $\Delta n$  at all.

We calculated the effect of the relative sizes of the Gaussian write and read beams by splitting the hologram into small, uniform regions of  $\Delta n$ , assuming that coupled wave theory holds over these small areas, and summing the diffraction efficiency of each of these areas of the hologram for the overlapping read beam. Because our holograms are thin ( $< 50 \mu\text{m}$ ), the angular selectivity, the angular width between the first nulls of the Bragg selectivity curve, is on the order of a few degrees, and so we do not include the effect of the angular spread of the Gaussian beam in this work. Figure 1 shows the effect of the relative sizes of the write and read beams. The example chosen is for a low  $\Delta n$ , low diffraction efficiency (DE) ( $< 10\%$ ) hologram. The graph on the left shows that as the read beam radius approaches zero, we see the expected plane wave DE, but as the read beam radius approaches the radius of the write beam, the measured DE falls to  $\sim 1/3$  of the expected DE. In a material test system, this would result in an underestimation of the peak  $\Delta n$  achievable in each material. On the right of Figure 1, we show the effect of misalignment of a very small Gaussian read beam (one tenth the size of the write beam), on the measured DE. While plane wave testers are generally carefully aligned, this demonstrates how important that alignment is and how much misalignment will result in significant underestimation of the peak  $\Delta n$  of the hologram.

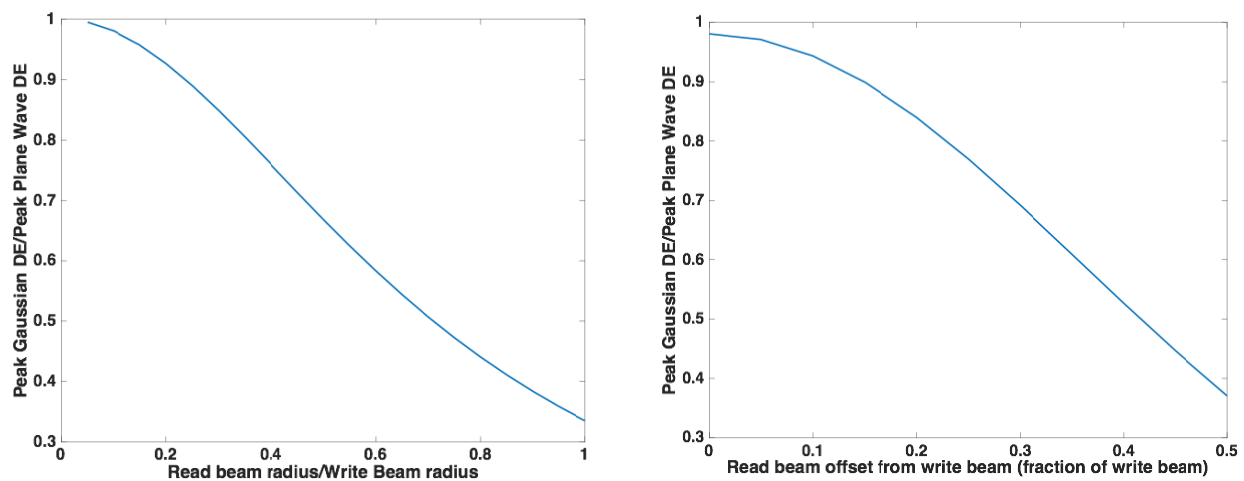


Figure 1. The effect of the relative sizes of the Gaussian read and write beams on the measured diffraction efficiency (DE) of the hologram. Left: Peak measured DE/Peak expected DE for the maximum  $\Delta n$  of the hologram vs. the read beam radius/write beam radius. As the read beam radius approaches zero, we see the expected plane wave DE, but as the read beam radius approaches the radius of the write beam, the measured DE falls to  $\sim 1/3$  of the expected DE. Right: Peak measured DE/Peak expected DE for the maximum  $\Delta n$  of the hologram vs. the read beam misalignment as a fraction of the write beam. As expected, measured DE falls off dramatically with misalignment of the read beam.

The above calculations are done for the low DE limit. As the peak  $\Delta n$  or the thickness of the sample increase to the point of 100% DE and over-modulation, the mismatch between the fit  $\Delta n$  and the peak  $\Delta n$  becomes more pronounced. In the case of over-modulation, the DE at the center of the hologram decreases, while the edges, with a lower  $\Delta n$ , continue to increase. To demonstrate this situation, we have modeled the DE across a Gaussian read beam for a transmission hologram with an  $n_i = 0.02$ ,  $d = 20 \mu\text{m}$ , a read beam radius equal to  $1/3$  of the write beam radius, and a period of  $1 \mu\text{m}$ , shown in Figure 4. The center of the hologram, where  $\Delta n$  is highest, is over-modulated to the point where the DE is close to zero. The  $\Delta n$  decreases with distance from the center, and so the DE increases. Integrating over this beam with a detector, we will find an average DE that is representative of an average  $\Delta n$  of the hologram, and not the peak  $\Delta n$ .

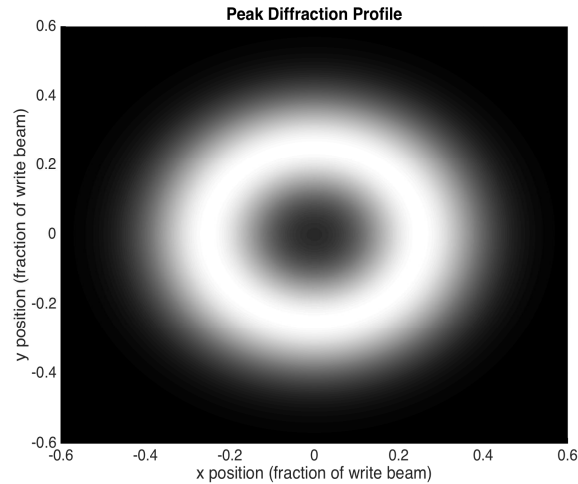


Figure 2. Image of an example diffracted beam profile for the over-modulated case. The Gaussian read beam has a radius equal to  $1/3$  of the radius of the write beam and is plotted as a fraction of the write beam. The center of the hologram, where  $\Delta n$  is highest, is over-modulated to the point where the DE has returned to zero. The  $\Delta n$  decreases with distance from the center, and so the DE increases. Integrating over this beam with a detector, we will find an average DE that is representative of an aver  $\Delta n$  of the hologram, and not the peak  $\Delta n$ .

The maximum achievable DE will depend on the relative sizes of the write and read beams for holograms written with Gaussian beams. Again, using a Gaussian read beam radius equal to  $1/3$  of the write beam radius, we plot the peak DE of the hologram as a function of the modulation parameter,  $\Phi$ , defined in Equation 1. The results are shown in Figure 3 (left). Integrating the DE over an area of the hologram that does not have a uniform  $\Delta n$  results in a DE measurement that does not accurately represent the peak  $\Delta n$  of the hologram. This effect becomes more pronounced at higher modulation and for read beams that are a larger fraction of the write beam. For a Gaussian read beam radius that is  $1/2$  the radius of the write beam, the effect becomes a significant problem.

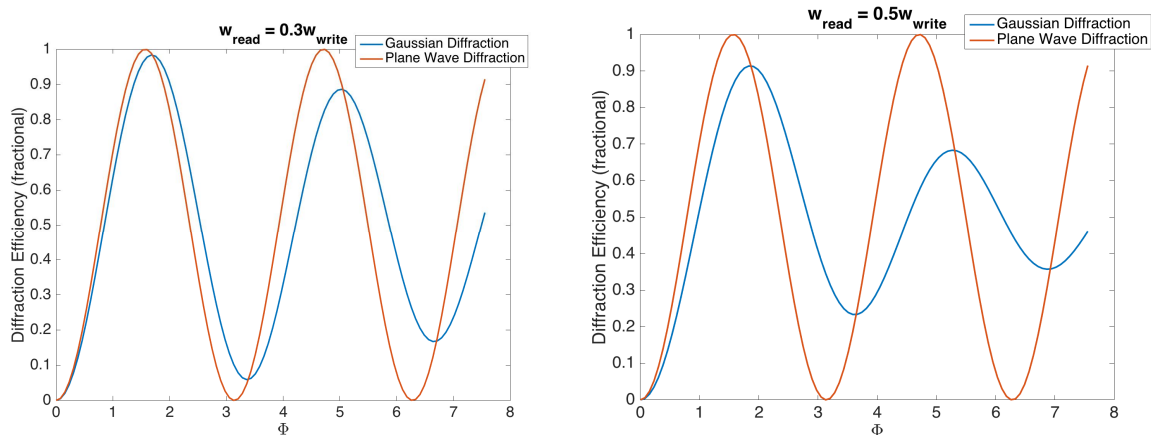


Figure 3. Diffraction Efficiency vs.  $\Delta n$  for a plane wave read/write system as well as a Gaussian read/write system where (left) the read beam is  $1/3$  the size of the write beam. Integrating the DE over an area of the hologram that does not have a uniform  $\Delta n$  results in a DE measurement that does not accurately represent the peak  $\Delta n$  of the hologram. This effect becomes more pronounced at higher modulation and for read beams that are a larger fraction of the write beam. The graph on the right shows the effect of a read beam radius that is  $1/2$  of the size of the write beam.

For accurate measurements of peak achievable  $\Delta n$  in a materials test system, the hologram and read beams must be carefully measured and compensated either through homogenization optics to create a more uniform hologram, or through the fit parameters that include the Gaussian read and write beam sizes. Especially in cases where the DE is over-modulated and these effects become more pronounced.

## 2.2 Other non-uniformities

While the non-uniformity of the hologram written with a Gaussian beam is straightforward to characterize and compensate, there are other factors that lead to holograms being written that are non-uniform, such as mechanical stresses, diffusion-limited processes in the material, and significant absorption of the writing light in the depth of the sample. The magnitude of these effects depends on the beam sizes, the thickness of the samples, and the pitch of the hologram for open films, so each experimental factor must be carefully considered when comparing material systems for design of optimal systems for a given application.

Two chemistry photopolymer systems consist of a thermoset matrix with a higher refractive index monomer and photoinitiator that are free to diffuse through the matrix. When writing a hologram, the bright fringes of the interference pattern cleave photoinitiator, generating radicals that lead to the polymerization of the writing monomer. The resulting monomer gradient causes diffusion of monomer from the dark to the bright fringes. The initial polymerization of monomer causes local shrinkage, the final flood cure causes large scale shrinkage that has been well studied in the literature [9], and diffusion of the monomer from dark to light fringes causes local swelling of the matrix. The effect of the shrinkage and swelling depend on the boundary conditions. For holograms written between two 1 mm glass microscope slides, attachment at the surfaces prevents significant shrinkage or swelling of the material in the depth of the samples due to the constraint of the surrounding polymer, which is also anchored to the glass. The forces caused by the polymerization and diffusion of monomer are still present, however, and the stress caused by the writing the hologram likely causes transverse strains that affect the uniformity of the period across the writing beam. We have observed that when the photopolymer is prevented from adhering to the glass slides using Rain-X, the polymer will locally delaminate from the glass slides upon polymerization. We have also observed surface relief gratings in open films, especially with low spatial frequency gratings. Any stress in the development of the hologram that results in a surface feature or non-uniform hologram period will result in a decrease in DE and an underestimation of the peak  $\Delta n$ . These are real decreases in coupling of light into the diffracted order and affect the performance of holograms, but especially at high DE, they also couple the issue of mechanical stress with actual  $\Delta n$ , making analysis of holographic material properties confusing.

While the materials presented here have very low absorption profiles at both the read and write wavelengths, the dyes and photoinitiators used to initiate polymerization in some materials can also be highly absorbing at the write wavelengths. This results in an index profile that decays in depth just as the intensity of the write beam decreases due to Beer-Lambert absorption. This will result in a similar issue as seen with the Gaussian beam. For a hologram that decreases in  $\Delta n$  over the depth, the resulting DE and subsequent fit parameters will be an average of the index modulation in depth. This will cause measurements of DE to underestimate the peak  $\Delta n$ , especially for over-modulation, where the front of the sample has a  $\Delta n$  that leads to over-modulation and a decreased DE, while the back of the sample has a higher DE. Polymers with lower absorption and/or thinner films will prevent this, but is not always possible in practical systems.

## 3. EXPERIMENTAL METHODS

### 3.1 Materials

The crosslinked matrix of the two-component photopolymer system consists of a stoichiometric ratio of polyisocyanate (Desmodur N3900, Covestro AG) and polyol (polycaprolactone-block-polytetrahydrofuran-block-polycaprolactone, Sigma Aldrich) that forms a flexible polyurethane. The photoreactive component contains of a 1:10 molar ratio of photoinitiator TPO (2, 4, 6-Trimethylbenzoyl-diphenyl-phosphineoxide, Sigma Aldrich) and commercially purchased tribromophenyl acrylate (TBPA, Monomer Polymer), a monofunctional acrylate. Samples were made with 30 wt% of this writing monomer. The matrix and photoreactive components were mixed together in their liquid form at 60°C, degassed, and then cast between two 1 mm glass microscope slides. Sample thicknesses were set using polyester spacers ranging between 15 and 25  $\mu\text{m}$ . Polymerization occurred overnight in an oven at 60°C.

### 3.2 Holography systems

Single transmission holograms are recorded using a 405 nm laser diode and read out with a 633 nm HeNe laser to measure the live diffraction efficiency development as well as the angular selectivity profile. The transmission and reflection holography systems used in this work are shown in Figure 4. For the transmission system, the 405 nm laser is conditioned through anamorphic prisms and a spatial filter to provide good beam quality, the power is controlled through an attenuator, and a shutter is used to control the exposure times. The relative beam powers of the two writing beams are controlled through a set of half waveplates and beamsplitters and the fringe visibility at the sample plane is optimized by imaging onto a CCD camera. The 633 nm laser is spatial filtered, aligned to the appropriate Bragg angle, and then the transmitted and reflected beams are monitored with power meters. In the transmission system, the 405 nm write beam has a  $1/e^2$  diameter of 4.3 mm and the 633 nm read beam has a  $1/e^2$  diameter of 1.26 mm. On the right side of Figure 4, the reflection system has similar initial optics for conditioning the 405 nm write beam, and the beam is split in two then interfered inside the polymer sample, with each beam entering from opposite sides of the sample to form a reflection hologram. In the reflection system, the 405 nm write beam has a  $1/e^2$  diameter of 1 cm. The wavelength selectivity and DE of the resulting hologram is monitored with an Ocean Optics flame spectrometer in transmission using a low power halogen light source. Both transmission and reflection systems use an intensity of  $5 \text{ mW/cm}^2$ .

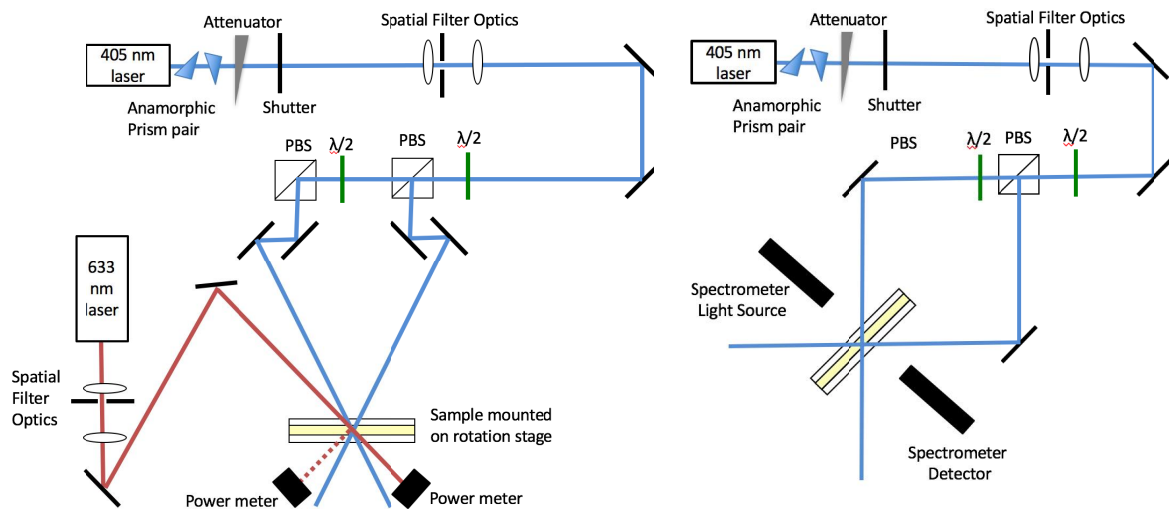


Figure 4. Left: Transmission holography experimental set up. The 405 nm laser is conditioned through anamorphic prisms and a spatial filter to provide good beam quality, the power is controlled through an attenuator, and a shutter is used to control the exposure times. The relative beam powers of the two writing beams are controlled through a set of waveplates and beamsplitters and the fringe visibility at the sample plane is optimized by imaging onto a CCD camera. The 633 nm laser is spatial filtered, then aligned to the appropriate Bragg angle and the transmitted and reflected beams are monitored with power meters. Right: Reflection holography experimental set up. The write system is similar to the transmission system and the wavelength selectivity is monitored using an Ocean Optics spectrometer real time during writing and development of the hologram.

## 4. EXPERIMENTAL RESULTS

We analyze the refractive index change for a two-chemistry photopolymer system with a monofunctional acrylate, TBPA, compensating for the known loss in DE due to the Gaussian write beam used in our transmission system. Both transmission and reflection holograms are recorded using the same writing intensity of  $5 \text{ mW/cm}^2$  and exposure time of 1 second. The transmission holograms are written with a period of  $1 \mu\text{m}$ , while the reflection hologram is written with a period of  $150 \text{ nm}$ .

The angular selectivity curves are shown in Figure 5 for the transmission holograms written into samples of two different thicknesses with identical writing intensities and exposure times. The transmission hologram shown on the left is over-modulated, as demonstrated by the higher 1<sup>st</sup> order side lobes. The thickness is found to be  $23 \mu\text{m}$  with an  $n_I = 0.0124$ . If the Gaussian write/and read beams are not accounted for, a standard plane wave fit gives an  $n_I = 0.0118$ , a

difference of approximately 5%. If careful fits are not made, it is also possible to fit the main peak to the under-modulated case, which will dramatically underestimate the actual  $n_I$ . The central peak DE does not contain enough information to determine if the hologram is over- or under-modulated [10,11]. For example, if a fit is made to the same data shown in Figure 5(left) using the plane wave approximation and fitting the central peak to the case of under-modulation, the resulting fit parameters are  $d = 25 \mu\text{m}$  and  $n_I = 0.006$ , underestimating  $n_I$  by a factor of 2. Thicker holograms written in the same formulation become over-modulated at lower values of  $n_I$  and suffer from mechanical stresses, making fits much more challenging, as shown in Figure 5 (right). The data can clearly not be fit nearly as well, leading to larger uncertainties. For the fit shown on the right, the thickness is found to be  $42 \mu\text{m}$  with an  $n_I = 0.0125$ .

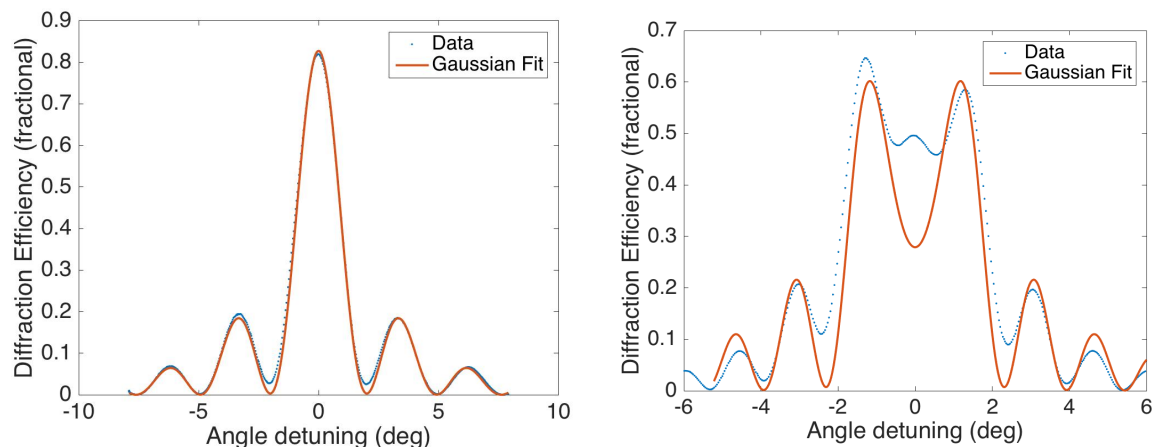


Figure 5. Bragg selectivity curves for holograms with identical writing parameters, but different thickness films. While careful fits of the data give very similar values for  $n_I$ , over-modulation in thick samples with non-uniformities likely due to mechanical stress can cause large uncertainties in the resulting fits.

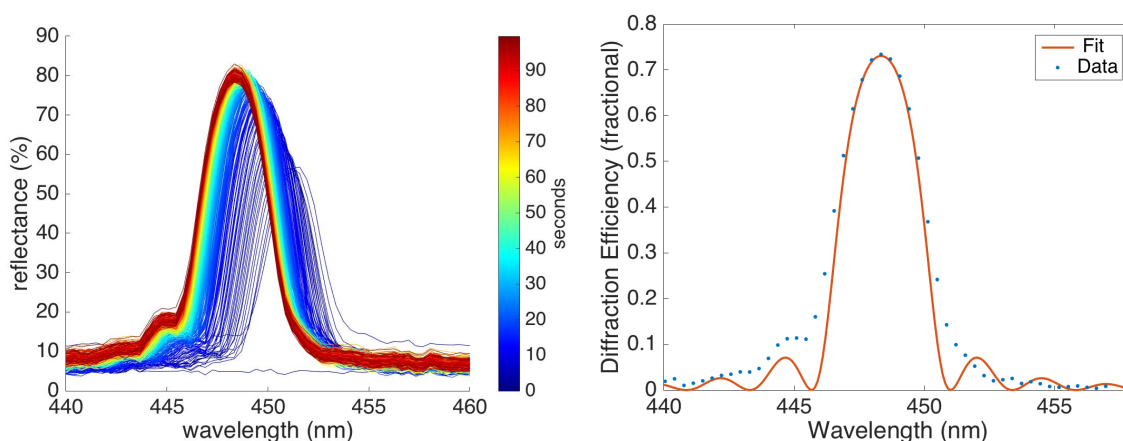


Figure 6. Left: wavelength selectivity curve for reflection hologram written with 405 nm writing beam,  $5 \text{ mW/cm}^2$ , and a 1 s exposure time. The hologram is recorded in real time as the strength of the hologram grows due to diffusion of monomer over 100 seconds. The peak wavelength shifts likely due to shrinkage of the material. Right: Fit to the final DE vs. wavelength of the reflection hologram. Without clear side lobes, this fit is not very accurate, but can give a rough measurement of  $d$  and  $n_I$ .

As reflection holograms have higher wavelength selectivity, the reflection hologram is monitored vs. wavelength instead of angle detuning, shown in Figure 5 (left). The growth of the reflection hologram is monitored over the course of 100 seconds and shows a blue shift of the peak wavelength from the start of writing to the full growth of the hologram. This decrease in peak wavelength over the time of diffusion indicates shrinkage of the material. This leads to a non-uniformity in the grating as seen in the fit in Figure 5 (right). Without clear side lobes in the data, the fit is challenging. The width of the central peak could be due to either thickness of the sample or non-uniformity in the grating pitch. From the fit shown,  $d = 18 \mu\text{m}$  and  $n_I = 0.0067$ , but the uncertainty in this calculation is large. Angular scans of the reflection



hologram, smaller probe beams, and a study of how the width of the peak changes with thickness would help differentiate the various factors affecting the wavelength selectivity of the reflection hologram. Also, the  $n_1$  measured here is significantly lower than that measured for the transmission hologram. This discrepancy in refractive index change from the transmission case may be caused by fit uncertainty, shrinkage, mechanical stresses in writing or a limitation of the spatial frequency response of the material and will require further investigation.

## 5. CONCLUSIONS

Analysis of transmission and reflection holograms using plane wave testers is a common method for analyzing and optimizing material formulations. While plane wave testers give accurate information on the DE of a hologram under test, they do not take into consideration the non-uniformities of the hologram when calculating the thickness and refractive index modulation of the hologram. When using these systems to optimize material performance for holography and other applications, careful attention must be paid to the assumptions that go into the standard CWT fits, especially in the case of high  $\Delta n$ , over-modulated holograms. We demonstrate the effect of Gaussian write and read beams on the output measurements of the thickness and refractive index modulation and correct for the non-uniformities resulting from the Gaussian write beam. The intuition gained from this analysis can inform further studies of non-uniformities due to absorption and mechanical stress. We present a model material with  $n_1 > 0.01$  in transmission holograms along with high DE reflection holograms as a demonstration of the effect these non-uniformities can have on extraction of material parameters from an experimental plane wave tester.

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