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# Effect of monomer diffusion on photoinduced shrinkage in photopolymer layers determined by electronic speckle pattern interferometry

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#### ABSTRACT

The aim of this study is to determine the effect of monomer diffusion on the photoinduced shrinkage profile in acrylamide based photopolymer layers during holographic recording. Using phase shifting electronic speckle pattern interferometry the displacement at each pixel in the image of the layer is measured. The complete displacement profile of the layer was obtained using phase shifting technique. We observed a reduction in shrinkage as a result of monomer diffusion from unexposed regions of holographic exposure. As a result of diffusion the maximum shrinkage was reduced by 26 % from 7.18 $\mu$ m to 5.28 $\mu$ m in a photopolymer layer of thickness160 ± 3  $\mu$ m after 84 seconds of recording.

Keywords: Holography, interferometry, photopolymer, shrinkage

#### 1. INTRODUCTION

Polymerisation induced shrinkage is one of the main reasons why photopolymer materials are not widely used in some holographic applications. Shrinkage occurring in an acrylamide based photopolymer developed at the Centre for Industrial and Engineering Optics<sup>1</sup> has been previously determined <sup>2-4</sup> by measuring the shift in the angular position of the Bragg peak. Incorporating of zeolite nanoparticles helps in reducing shrinkage in photopolymer layers<sup>4-6.</sup> Shrinkage was later measured in real time using holographic interferometry, a non-destructive technique that measures small static or dynamic changes occurring in an object <sup>6-9</sup>. Fringe counting techniques can give information regarding real time shrinkage but cannot give whole field information. Phase shifting electronic speckle pattern interferometry (ESPI) is a suitable technique widely used for determining whole field surface deformations and shapes of rough surfaces <sup>10-14</sup>. Temporal phase shifting technique in which the reference mirror is moved is one of the most common phase shifting technique. In our current work, we present the effect of monomer diffusion on the whole field displacement profile due to photoinduced shrinkage in an acrylamide based photopolymer layer during holographic recording. The shrinkage was determined using a (5, 5) phase shifting algorithm. The displacement at each pixel in the image of the layer was measured so that a complete shrinkage profile of the layer was obtained.

### 2. EXPERIMNTAL PROCEDURES

#### 2.1 SAMPLE PREPARATION

A green light sensitive photopolymer layer was prepared as previously described<sup>2</sup>. Briefly, 0.6 g of acrylamide monomer was added to 9 ml stock solution of polyvinyl alcohol (20% wt). Then 2 ml of triethanolamine was added. To this solution 0.2 g of *N*, *N*-methylene bisacrylamide and finally 4 ml of Erythrosine-B dye was added (0.11% wt. water stock solution). 0.06 ml of photopolymer solution was spread on a circular area of 1.2 cm diameter on a 25 mm x 35 mm glass plate coated on the opposite side with non-reflective paint. Similarly 0.12 ml of photopolymer solution

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was spread on a circular area of 1.7 cm diameter on a 25 mm  $\times$  35 mm glass plates. The samples were dried for 24 h. Sample thickness after drying was approximately 160±3  $\mu$ m.

#### 2.2 EXPERIMENTAL TECHNIQUE



Fig. 1. Schematic of the Electronic speckle pattern interferometry (ESPI) system

The ESPI system is as shown in Fig. 1. An Erythrosine B, green light sensitive photopolymer layer (Er.B sample) with different diameters was the object under study. The photopolymer substrate is a glass plate coated with non-reflective paint on the opposite side to the photopolymer layer. A similar glass plate was attached to a piezoelectric transducer (PZT) whose motion was precisely controlled by Labview software. A spatially filtered and collimated He- Ne laser (632.8 nm) beam to which the Er. B sample has negligible sensitivity was used in the interferometry system. The beam reflected from the photopolymer layer (object beam) and the beam reflected from the glass plate attached to the PZT (reference beam) were allowed to interfere and the interference pattern or specklegram was captured by a CMOS camera. The spatially filtered beam from a Nd-YVO<sub>4</sub> laser (emission wavelength 532 nm) was collimated and split into two beams which were allowed to interfere on the photopolymer sample for holographic recording. The diameter of the recording beam was 1.2cm. The spatial frequency of the recorded interference pattern was 1000 lines/mm. A constant phase difference of  $\pi/2$  between consecutive specklegrams was introduced by changing the input voltage to the PZT. The phase shifted specklegrams were used to obtain the phase map of the surface of the layer.

#### 3. EXPERIMENTAL RESULTS

The prepared sample as discussed in Section.2.1 was mounted in the ESPI system as shown in Fig. 1. Photopolymer layers of different diameters were exposed to a green light (532 nm) interference pattern formed by the two recording beams. Before holographic exposure, 5 phase shifted frames were stored for the initial state of the layer, and then the layer was exposed. In order to evaluate the shrinkage during exposure 5 phase shifted frames were captured and stored after 84 seconds of holographic recording. In order to study the effect of monomer diffusion on shrinkage holographic recordings were made in photopolymer samples of the same diameter as the recording beam. In this case there won't be any effect of monomer diffusion from unexposed regions. Another holographic recording was made in photopolymer sample of larger diameter than that of the recording beam in which case we may expect diffusion of monomer from outside the illuminated region. The phase maps corresponding to the sample surface before and after

exposure were calculated using a 5-frame algorithm. The error-compensating 5-step phase evaluation equation was used  $^{14}$ .

$$\phi = \tan^{-1} \left[ \frac{2(I_4 - I_2)}{I_1 - 2I_3 + I_5} \right]$$
(1)

where,  $I_1, I_2, I_3, I_4, I_5$  are the intensities of the phase shifted frames corresponding to phase steps of

$$-\pi, -\frac{\pi}{2}, 0, \frac{\pi}{2}, \pi$$

The phase map was calculated from these 5 phase shifted frames. The wrapped phase map contains  $2\pi$  phase discontinuities and phases are wrapped between  $-\pi$  and  $+\pi$  due to the nature of the arctangent function. The process of removing these  $2\pi$  discontinuities is called phase unwrapping or integrating the phase <sup>16,17</sup>. The wrapped phase maps before and after exposure were unwrapped using the 2D-SRNCP unwrapping algorithm <sup>18</sup>. The 2D SRNCP algorithm belongs to the class of quality guided path algorithms. In order to prevent error propagation, this algorithm will unwrap the highest quality pixels with highest reliability values first and lowest quality pixels with lowest reliability value last. The 2D SRNCP algorithm follows non-continuous or discrete paths for unwrapping. The phase maps still contain some errors which cannot be detected, but the algorithms are very robust in practice compared to continuous path unwrapping algorithms. The two unwrapped phase maps corresponding to the sample surface before and after 84 seconds of exposure to green light were subtracted from one another in order to get the phase map of the displacement profile due to shrinkage in the photopolymer.



Fig. 2. Unwrapped phase map (a) before exposure, (b) after 84s exposure, (c) phase map of shrinkage obtained by subtracting 3(a) from 3(b).

The unwrapped phase maps before and after exposure are shown in Fig. 2(a) and (b) respectively. The phase value at each pixel lying in the range  $[-\pi, +\pi]$  is represented by a grey level within the dynamic range of the CMOS camera enabling display of the phase map as a gray level image. The dark pixels correspond to a phase value of  $-\pi$  and white pixels which are saturated correspond to phase value of  $+\pi$ .

In order to determine shrinkage in photopolymer layers the phase map corresponding to the time of recording, in this case 84 sec, was subtracted from that before recording. The resulting unwrapped phase map is shown in Fig. 2(c). From the subtracted phase map, a 3D displacement map of shrinkage can be calculated

$$\Delta d = \left(\frac{\lambda}{4\pi}\right)\phi\tag{2}$$





Fig. 3. A 3D map of the absolute shrinkage photoinduced in a  $160\pm3$  µm thick sample after 84 sec holographic exposure for (a) without diffusion (b) with diffusion. 2D map of the absolute shrinkage (c) without diffusion (d) with diffusion. Recording intensity was 10 mW/cm<sup>2</sup>.

Fig. 3 (a) shows the shrinkage measured 84 s after the start of holographic recording with two beams with total intensity of 10 mW/cm<sup>2</sup>. The photopolymer layer used in this case was the same diameter as the recording beam. Fig. 3(b) shows the shrinkage measured after 84 s recording in a photopolymer layer of diameter larger than the recording beam. The thickness of both the samples was  $160\pm3 \mu m$ . Fig. 3 (c) and Fig 3(d) show the 2 dimensional displacement profiles of the samples as in Fig. 3(a) and Fig. 3(b). Inorder to compare the effect of diffusion the shrinkage profile is plotted along the central region of the layer designated by the black dotted line in Fig. 4.

The result is shown in Fig. 4. We can clearly see that the shrinkage is greater in the case of holographic recording where the recording beam and layer are the same diameter there is no diffusion. All monomer molecules in the photopolymer were polymerised during holographic recording and as a result higher shrinkage occurs whereas in the case of recording beams smaller in diameter than the layer, shrinkage is partially compensated by diffusion from outside the illuminated area. In the profiles 100 pixels corresponds to 1.2 cm. A detailed analysis of the data is currently being carried out and will be published elsewhere.



Fig. 4. Line profile showing the effect of monomer diffusion on shrinkage

#### 4. CONCLUSIONS

We have demonstrated the effect of monomer diffusion on the whole field deformation by photoinduced shrinkage of photopolymer layer, using a phase shifting ESPI system. Holographic gratings were recorded in  $160\pm3 \mu m$  thick layers with one sample having same size of the recording beam and another larger in size than that of the recording beams. Phase shifted specklegrams were captured before and after holographic recording. These phase shifted specklegrams were used to obtain the shrinkage profile. It was observed that the shrinkage is greater for samples where there is no effect of monomer diffusion from the unexposed region which could be related to the fact that all monomer molecules are polymerized during holographic exposure. The reduced shrinkage at the edges could primarily relate to the fact that the exposed beam profile is Gaussian and hence the intensity at the edges will be lower than that at the center. From the diffusion studies we can say that diffusion of material from outside the illumination area influences the final shrinkage and could be the reason why the shrinkage at the edge is lower than in the case of no diffusion. The current study will find useful application for the characterization of photosensitive polymer materials for holographic applications.

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