Optical Patterning of Photopolymerisable Materials

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Optical patterning of photopolymerisable materials

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\textbf{Abstract.} Holographic recording is an effective approach for photopolymer surface patterning. It has been previously utilised in acrylamide-based photopolymers and a spatial frequency limit of 200 l/mm has been observed. We report the successful inscription of submicrometer resolution patterns. The spatial frequency response has been extended to 1550 l/mm by introduction of thermal post recording treatment. Initial results from the optical patterning utilising a spatial light modulation (SLM) reveal that the amplitude of the photoinduced surface relief structures is larger in comparison to the amplitude obtained by holographic recording.

\textbf{Keywords:} photopolymers, photoinduced surface relief, optical patterning, acrylamide based photopolymer

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\textbf{INTRODUCTION}

Self-processing acrylamide-based photopolymers for volume holography have been successfully developed at the Centre for Industrial and Engineering Optics for more than a decade [1-4]. It has been observed that during holographic recording in these materials in addition to the volume holograms, surface relief modulation is inscribed [5-7]. Optically recorded surface relief modulation has been previously observed and characterised in other photopolymer systems [8-11]. A common feature of the surface relief structures in self-processing photopolymers is their limited range of spatial frequency. It has been observed that the amplitude of the surface relief profile decreases with the increase of the spatial frequency and the upper resolution limit in these materials doesn’t exceed 500 l/mm.

For many practical applications such as fabrication of switchable electro-optical devices and design of optical sensors sub-micron resolution is required. Such resolution is achievable by photolithography or electron beam lithography, but both techniques are expensive and the first requires wet post processing of the material while the second one has a small throughput.

In the present paper two different approaches to the optical patterning of the surface of a photopolymerisable material – holographic recording and illumination through a spatial light modulator (SLM) have been adopted. 3D surface relief structures have been inscribed and studied using white light surface profilometry and atomic force microscopy. Sub micrometer size structures have been inscribed by introduction of post exposure thermal treatment of the holographically recorded photopolymer layers. In addition, surface relief structures inscribed by illumination through a SLM are compared to these obtained by holographic recording.

\textbf{EXPERIMENTAL}

\textbf{Materials}

The investigated acrylamide-based photopolymer layer consists of a polyvinyl alcohol binder, two monomers- acrylamide and N,N\textsuperscript{'}-methylenebisacrylamide, triethanolamine as an initiator and Erythrosine B photosensitive dye. Once mixed, 0.3 ml of the solution was spread on a 75mm x 25mm glass substrate. The films were ready for use after drying for 5 hours. The typical layer thickness was 25±2 µm.
**Recording set-up**

The photoinduced surface relief was inscribed by recording transmission holographic gratings ranging from 200 – 1550 lines/mm. A Verdi™ laser (532 nm) was used for holographic recording. The optical set-up is shown in figure 1. The spatial frequency of recording was controlled by moving the sample along a rail as well as adjusting the mirror (see Fig. 1). The recording intensity was 10 mW/cm². Exposure time was varied from 50 to 100 seconds.

**FIGURE 1.** Optical set up for holographic patterning.

**Experimental procedure**

The experimental procedure is shown in fig.2. After exposure to light the layers were bleached and exposed to high temperature. The temperature was varied from 160 to 210°C with a rate of 1°C/min. The photopolymer surface was studied using a white light interferometric (WLI) surface profiler MicroXAM S/N 8038 and an atomic force microscope AFM Easy Scan model 2. These two methods both reveal the surface profile of the structure, with each providing attractive qualities. The WLI delivers fast and reliable information with resolution limit of 1000 l/mm, whereas the AFM delivers a much higher resolution picture, albeit requiring more time.

**FIGURE 2.** Experimental procedure

**RESULTS AND DISCUSSION**

**Holographic patterning**

AFM scans of the photopolymer layers were carried out immediately after holographic exposure. No periodical surface relief was formed when the interference pattern was of frequency above 500 l/mm. When the exposed area was probed with one of the recording beams it was observed that a volume phase holographic grating had been recorded. After baking at high temperature this latent pattern was developed and a periodical surface relief profile was observed in the AFM pictures (Figure 3). A set of samples recorded at the same conditions was transferred to the oven which was initially set at 120°C. The temperature was set to rise by 1°C/min. Two samples were removed from the oven every 10min and their surface was examined by AFM. The amplitude of the surface relief profile

![Diagram](image-url)
(SRA) increased with the increase of baking temperature whose maximum of 210°C was limited by the oven used in this experiment. The maximum surface relief amplitudes were observed in samples baked at 210°C. Control measurement of the weight of the photopolymer layers revealed that the increase in the surface amplitude is accompanied by decrease in the sample mass. Most probably the mechanism of surface relief formation after thermal treatment is related to spatially modulated evaporation of TEA whose boiling temperature is 208°C.

**FIGURE 3** Holographically inscribed surface relief gratings of spatial frequency a) 500 l/mm; SRA = 260 nm; b) 700 l/mm; SRA = 150 nm; c) 1000 l/mm; SRA = 35 nm.

**Spatial Light Modulator experiment**

The aim of this experiment was to study the properties of the surface relief modulation inscribed by imaging the pattern on a SLM onto the photopolymer layer. An asymmetric pattern (Figure 4a) of white rectangles on a black background was chosen in order to allow correlation of the location of the surface relief peaks and the locations of the illuminated areas. As one can see in Fig 4b the surface relief peaks correspond to the illuminated areas. This is in agreement with our previous studies of the location of the surface relief peaks in holographically recorded structures [5].

**FIGURE 4.** Pattern on the SLM a) and surface relief profile inscribed in the photopolymer layer.
Comparison of the SRA achieved by illumination through a SLM and the amplitude of holographically inscribed patterns of the same spatial frequency of 16 l/mm shows that the first process is more effective. The SRA at 16 l/mm achieved using holographic recording was less than 1µm while by using a SLM SRA of 3.5µm was achieved.

CONCLUSIONS

In summary, a post recording thermal treatment is required for achievement of sub-micrometer surface relief profiles. The development of the surface relief profile is accompanied by a mass loss. The highest SRA was observed after baking above 200°C. This suggests that the mechanism of high temperature surface relief formation could be related to a spatially modulated evaporation of TEA. A successful fabrication of surface relief profiles with amplitude of 15nm and spatial frequency of 1550 lines/mm by holographic patterning was demonstrated. The initial results from surface patterning by utilising a SLM demonstrate that when compared to holographic recording this technique at the same geometry and recording conditions leads to a higher surface relief amplitudes. Further improvement of the optical set-up is required to test the high spatial frequency response of the material using this technique.

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