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Investigation of the photoinduced surface relief modulation in acrylamide-based photopolymer

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Abstract: A surface relief diffraction grating is inscribed in acrylamide photopolymer by holographic recording with spatial frequency below 300 l/mm. The periodic surface modulation appears in addition to a volume phase holographic grating. Due to the nature of the presented photopolymerisable material the gratings are induced by light only and no post-processing is required.

An investigation of the dependance of the amplitude of the photoinduced relief modulation on spatial frequency of recording and on sample thickness has been carried out utilising white light interferometry. A model of the mechanism of surface relief formation is proposed on the basis of the measured dependencies.

The possibility for inscription of surface relief modulation implies different applications of the photopolymer: fabrication of diffractive optical elements, recording of computer generated holograms and biosensors.

Keywords: photopolymer, surface relief grating, photoinduced relief, holography

Introduction:

Photoinduced single step inscription of a surface relief modulation in photopolymer systems opens attractive perspectives for applications such as design of diffractive optical elements^{1,2}, recording of computer generated holograms³ and design of biosensors. Important characteristics of the material in relation to these applications are its spatial resolution, the amplitude of photoinduced surface relief, the required recording time for achievement of maximum surface modulation and its long-term stability. For optimisation of all these characteristics a thorough understanding of the mechanism of surface relief formation under light illumination is necessary. Although the photopolymer systems have been a subject of constantly increasing interest, especially after the development of self-processing photopolymer systems, only a few investigations dedicated to the surface relief formation have been reported in the literature^{1,2,4,5}.

In the present paper, for the first time to our knowledge, we report an investigation on the photoinduced surface relief modulation in an acrylamide-based photopolymer system^{6,7}, developed at the Centre for Industrial and Engineering Optics, Dublin Institute of Technology. A maximum surface relief modulation in the order of 1.5 μm was achieved using recording times less than 20 s. The dependence of the photoinduced relief modulation on the spatial frequency, recording intensity and sample thickness are presented. These characteristics are compared with the reported data for other photopolymer systems.

Theory

Photopolymer holographic recording systems

The general composition of the photopolymer layer is a monomer, electron donor (initiator), photosensitizing dye and, optionally, a polymer binder. Upon illumination of the

photopolymer with non-uniform light of appropriate wavelength the sensitizing dye absorbs a photon and reacts with an electron donor to produce free radicals. These initiate polymerisation where the light was absorbed. The changes in the density and the molecular polarizability, which accompany the polymerisation, lead to change in the local photopolymer refractive index and a hologram is recorded. According to recently developed theoretical models⁸⁻¹¹ describing holographic recording in photopolymer systems an important process in hologram formation is the monomer diffusion from the dark to bright fringe areas. Our recent results reveal that the monomer diffusion in the acrylamide-based photopolymer system is comparatively fast and is characterised by diffusion constant of $D_0=1.6 \cdot 10^{-7} \text{ cm}^2/\text{s}$ ¹². If as believed the monomer diffusion is involved in the process of photoinduced surface relief formation then relatively short exposure times and high surface relief amplitude even at low spatial frequencies could be expected in our photopolymer system.

Experiment:

Materials

The photopolymer layer was prepared as described elsewhere⁵. The sample consisted of a polyvinyl alcohol binder, two monomers – acrylamide and N,N'-methylenebisacrylamide, the second one plays the role of a crosslinker, Erythrosin B sensitising dye and triethanolamine as an initiator. The investigated photopolymer system is water-soluble. After mixing of the components the solution was spread on glass plates with dimensions of 5x5 cm². The films were ready for use after drying for 24 hours at constant humidity of 70%.

In order to investigate the dependence of the surface relief modulation on the photopolymer film thickness different amounts of photopolymer solution were deposited on the same area of 5x5 cm². Layers of thickness from 25 to 100 μm were obtained following this procedure.

Experimental set-up

The photoinduced surface relief was inscribed by holographic recording of transmission diffraction gratings with spatial frequency in the range of 10 - 300 l/mm. The second harmonic of a Verdi 05 NdVO₄ laser at 532 nm was used to record the gratings. The recording intensity was varied in the range 1-10 mW/cm². In order to observe the photoinduced surface modulation at different stages of formation the exposure time was varied in the range 0.5 s to 90 s. The photopolymer surface after recording was profiled using white light interferometric (WLI) surface profiler *MicroXAM S/N 8038*.

The film thickness was determined after cutting the photopolymer layer with a sharp scalpel and measuring the dimensions of the cuts with the WLI profiler.

Results and discussion:

Volume phase grating and surface relief grating

It is well known from the literature⁵⁻¹¹ that during holographic recording in photopolymers a volume phase grating is formed due to non-uniform modulation of the refractive index of the material. We have observed that gratings of a spatial frequency above 300 l/mm and diffraction efficiency close to 100% do not show detectable phase shift modulation using the WLI technique. From previous investigations⁶ of surface relief in acrylamide-based material at spatial frequencies of recording up to 6.66 l/mm, it was established that the relief peaks

correspond to the light fringe areas. This would mean that the surface relief grating and the volume phase grating are not phase shifted. Based on this observation the phase shift measured at low spatial frequency is assumed to be mainly due to local change of the sample thickness. An analysis of the diffraction efficiency of the recorded gratings combined with the measurements of the surface relief modulation at different spatial frequencies of recording will give the opportunity to distinguish more precisely the contribution of both types – volume phase and surface relief gratings.

Surface relief modulation at different stages of recording

In order to trace the surface relief formation, the recording process was interrupted at different stages. The profile was measured after exposing the sample to a sinusoidal modulated intensity with a spatial frequency of 10 l/mm for varied time intervals between 0.5 s and 90 s. The recording intensity was 2.5 mW/cm². The surface profiles after recording for 0.5 s, 10 s, 30 s and 90 s are presented in Fig. 1. As can be seen from the figure a significant surface relief modulation of 250 nm is inscribed only 0.5 s after the beginning of the recording. The surface profile modulation increases up to 1.6 μm after 20 s of recording and

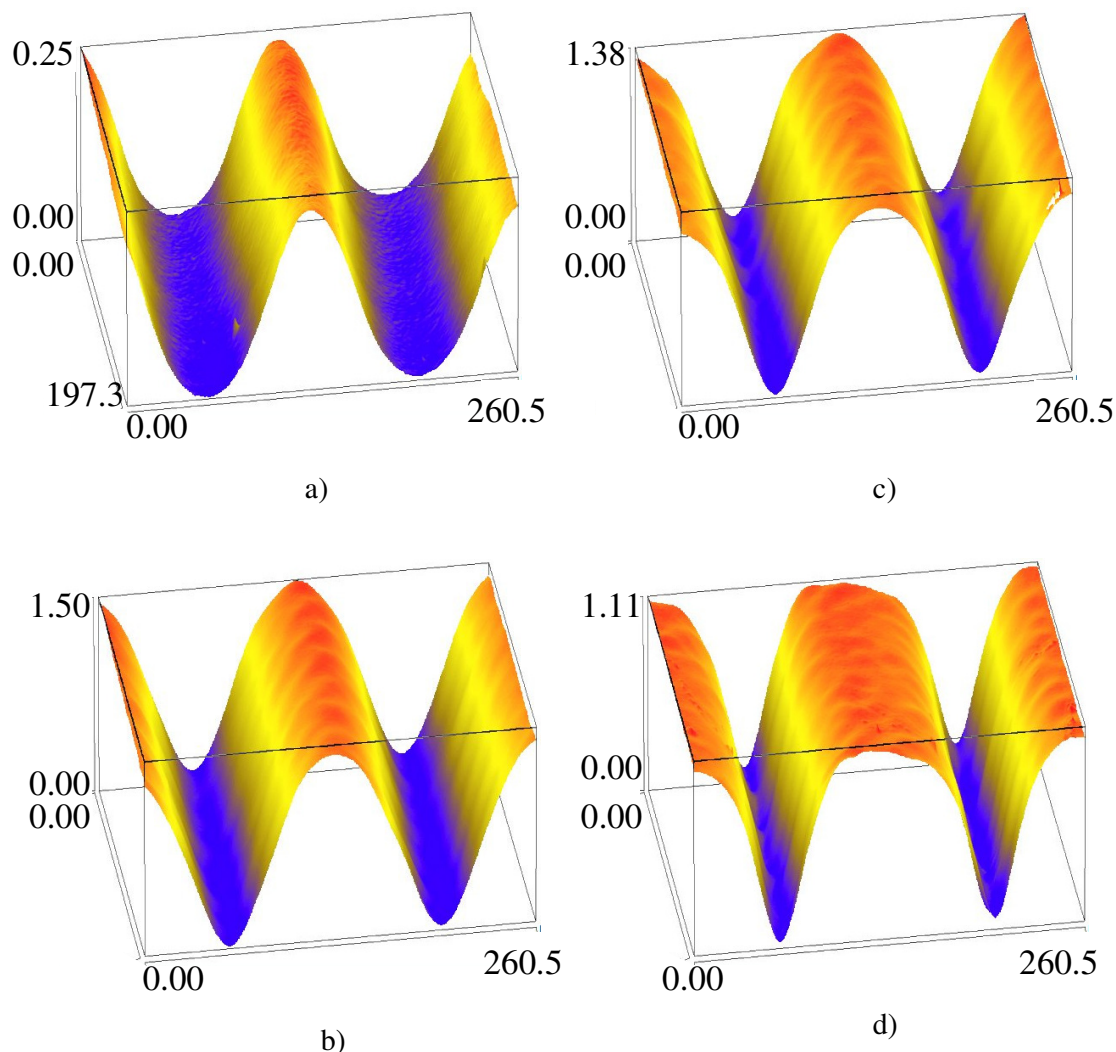


Fig. 1 Surface relief modulation in μm after recording at spatial frequency of 10 l/mm with intensity of 2.5mW/cm² for 0.5 s (a), 10 s (b), 30 s (c) and 90 s (d).

then decreases to 1.1 μm when the recording time is 90 s. The change in the amplitude of photoinduced surface relief is accompanied by pronounced change in the shape of the surface profile. It supports the hypothesis that diffusion of material is involved in the mechanism of formation of the observed periodical structure. It is noteworthy that the relatively short exposure times required in order to observe surface relief modulation above 1 μm in the investigated system. For comparison, the required time for recording in other photopolymers systems^{1,2} using similar intensity is in the order of couple of minutes.

Influence of post exposure

It was observed that post exposure using UV light leads to increase of surface amplitude. The results for gratings recorded at spatial frequency of 10 1/mm are presented in Fig. 2. As can be seen from the figure the post exposure leads to more than 30 % increase in the surface relief amplitude.

Dependence on the spatial frequency of recording

The dependence of the surface relief modulation on the spatial frequency of recording is presented in Fig.3. The recording time and intensity were 30 s and 2 mW/cm^2 respectively. The surface profile was measured immediately after the recording. As seen from the figure the increase in the spatial frequency of recording leads to decrease of the surface modulation amplitude. Similar observation has been reported for other photopolymerisable systems^{1,2,4}. It can be explained by the presence of surface tension forces or in other words by the tendency of the surface to adopt smaller surface area. There was no decrease in the surface profile amplitude with increasing the recording pattern fringe spacing up to 130 μm . This observation could readily be explained by the fact that the diffusion processes in

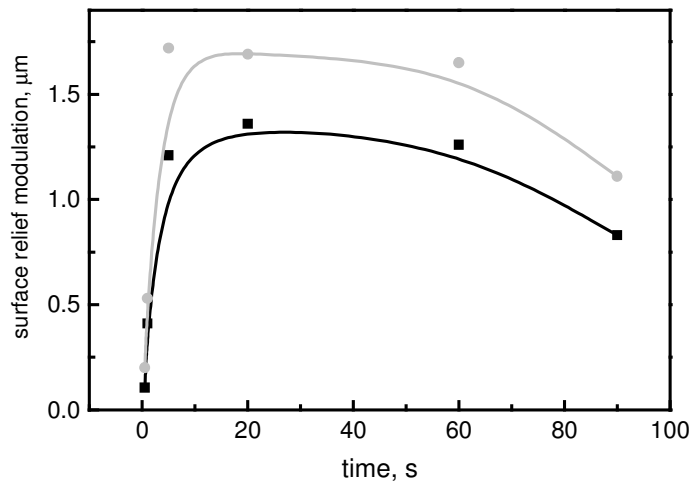


Fig. 2 Influence of the post exposure on the surface relief amplitude. The surface profile is measured immediately (black) and after UV light post exposure (grey). The measurements are carried out after using different time intervals of recording.

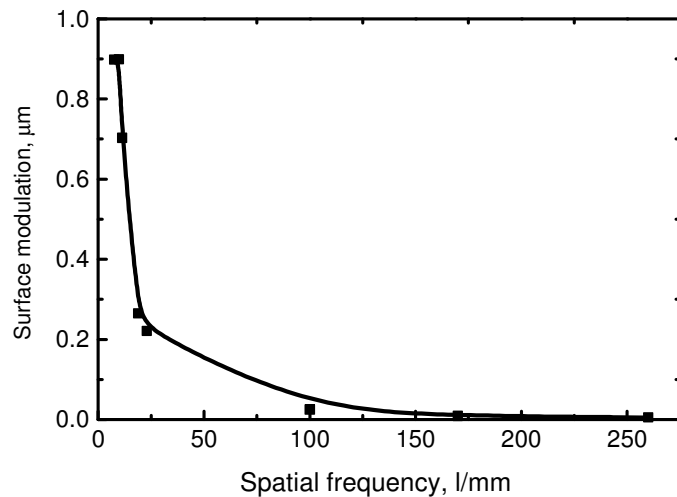


Fig. 2 Dependence of the surface relief amplitude on the recording spatial frequency.

the investigated photopolymer are fast and the material can easily be transported over large distances.

Dependence on the film thickness

We have carried out preliminary investigation of the influence of the photopolymer thickness on the amplitude of the photoinduced surface relief modulation after recording for 45 s with recording intensity of 6 mW/cm². Change in the sample thickness in the range of 25 to 75 μm does not lead to significant change in the surface profile. Further experiments with samples with much thinner layers obtained by spin coating technique are under progress.

Conclusions:

We have investigated the photoinduced surface relief modulation in acrylamide-based photopolymer system. It is observed that surface relief with spatial frequency of 10 l/mm and amplitude above 1 μm is inscribed using recording time intervals less than 20 s. The time required to achieve surface modulation is considerably shorter compared to other photopolymer systems^{1,2}. The surface relief amplitude decreases at higher spatial frequencies and it is not detectable above 300 l/mm. No decrease was observed at lower spatial frequencies up to 7 l/mm. Post exposure of the sample with UV light leads to more than 30 % increase in the initial surface relief amplitude regardless of the recording time between 0.5 s and 90 s. No dependence of surface relief amplitude on the sample thickness was observed in the range of 25-75 μm.

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