

Surface relief grating recording in azo polymer films

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Abstract. In this work holographic recording of surface relief grating in organic Poly(Disperse Red 1 – methacrylate) thin films was studied. In this compound azo dye is chemically bonded to monomers making mass transport very efficient in the presence of optical field gradient. Sample preparation was performed using different solvents and coating methods. In the obtained samples holographic recording was performed by solid-state diode pump laser with 532 nm wavelength. Polarization grating and surface relief grating formation was studied by diode laser with 660 nm wavelength. Diffraction efficiency's dependence on read-out laser beam polarization state was investigated. The depth of surface relief grating was measured by AFM and studied in dependence on recording beam intensity, exposure and thickness of the sample.

1. Introduction

In this work surface relief grating formation in azo polymer films was studied. Azo compound moieties can be aligned in the presence of electric field of light radiation. Due to that process photoinduced birefringence and dichroism appears. If we use interference pattern of two +45/-45 orthogonally linearly polarized beams resulting electric field vector has polarization modulation on the surface of the thin film, and surface relief grating (SRG) can be recorded. Holographic recording of SRG is particularly interesting due to its possible applications in optoelectronics, data storage and telecommunications [1, 2, 3].

We studied holographic recording in (Disperse Red 1 - methacrylate) thin films where well known PMMA polymer is used only methyl group is substituted by Disperse Red 1 azo-dye. SRG formation in these films is fast and doesn't require great exposures. The depth of obtained SRG exceeds the thickness of film, although SRG formation process strongly depends on intensity and thickness of the film.

2. Experimental

2.1. Sample preparation



Research was made using commercially available material Poly(Disperse Red 1 – methacrylate) – (P(DR1-MA)) purchased at Sigma-Aldrich. The chemical structure of used material is shown in figure 1. Maximal absorption of P(DR1-MA) polymer is at $\lambda_{max} = 467$ nm. For sample preparation P(DR1-MA) was dissolved in toluene or trichloroethylene. In order to obtain films with different thickness solvents with different concentration ($1.0 \pm 0.2\%$, $2.0 \pm 0.4\%$, $5.0 \pm 1\%$, $7.0 \pm 1.4\%$, $10.0 \pm 2.0\%$) were prepared. Solvents were purified using PTFE filters with pore size of $0.45 \mu\text{m}$. Material was applied on substrate by spin-coating method or using applicator with thickness $120 \mu\text{m}$. Before applying glass substrate was cleaned in dichromate - sulphuric acid solution. Films were dried in ambient conditions.

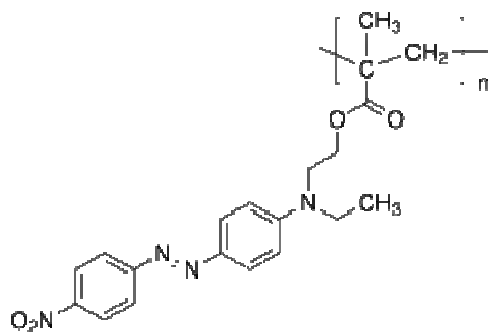


Figure 1. Chemical structure of Poly(Disperse Red 1 - methacrylate)

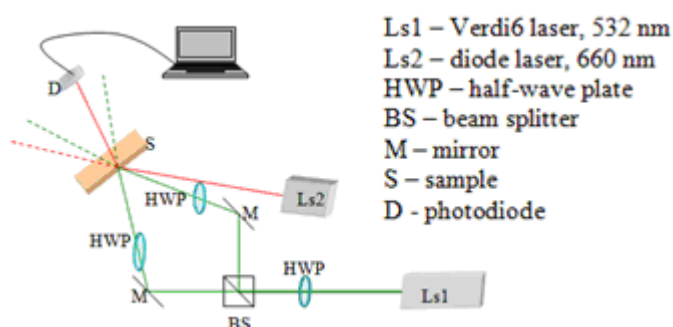


Figure 2. Experimental set-up

2.2. Experimental set-up

Holographic recording was performed on experimental set-up shown in Fig. 2. For recording solid-state diode-pumped laser Verdi-6 (Ls1) with wavelength $\lambda_1 = 532$ nm was used. It was split into two equal intensities beams by beam splitter (BS). Using mirrors (M) beams were deflected toward a sample (S). Half-wave plates (HWP) were used in order to set polarization state. The angle between recording beams was $\theta = 30^\circ$, thus grating period was $\Lambda = 1 \mu\text{m}$ according to Bragg's law. Read-out was made using diode laser with wavelength $\lambda_2 = 660$ nm. The laser was placed at Bragg's angle and intensity of diffracted beam of the first order was measured by photodiode (D) which was connected to PC.

Polarization state of recording beams was set to orthogonally linear - $+45/-45$. In this case on the surface of the film there is polarization modulation, but no intensity modulation [4].

3. Results and discussion

A kinetics of SRG formation and diffraction efficiency (DE) of recording with intensity $I = 0.95$ W/cm^2 in film with thickness $d = 490$ nm is shown in Fig. 3. As long as the depth of SRG does not exceed the thickness of the film, DE and the depth of SRG increase linearly. The speed of SRG formation reaches its maximal value in the first seconds of recording and strongly depends on intensity of recording beams. If in the first $t = 15$ s at intensity $I = 0.95$ W/cm^2 the average speed is $u = 7.8$ nm/s, then at intensity $I = 1.86$ W/cm^2 it is $u = 12.3$ nm/s. Average speed was evaluated using formula: $u = (h_t - h_{0s}) / t$, where h_t – the depth of SRG after $t = 15$ s recording. Further SRG formation is slowed down by surface tension of the film and SRG forms slower. When maximal value is reached, low decrement of the depth of SRG is observed due to high surface tension and then saturation is reached. DE curve corresponds to SRG depth changes with a slight shift (figure 3). The shift between h and DE curves is caused by the phase shift between volume and surface relief gratings due to read-out was made with s-polarized probe beam [5]. Thus, the main contribution in the value of DE gives SRG, while volume grating is

implicit.

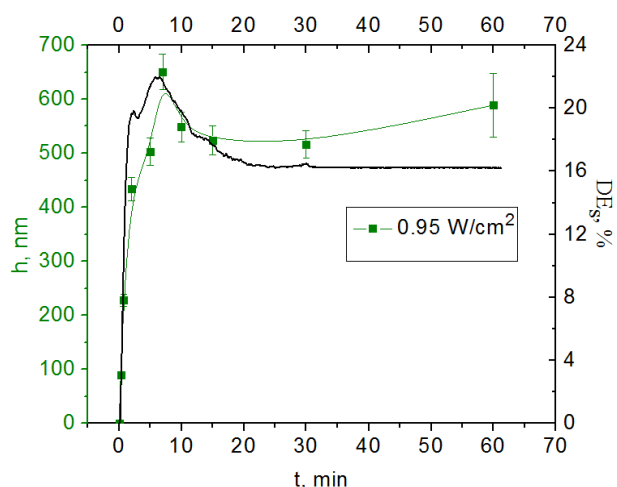


Figure 3. Diffraction efficiency and kinetics of SRG formation in film with a thickness of $d = 490$ nm

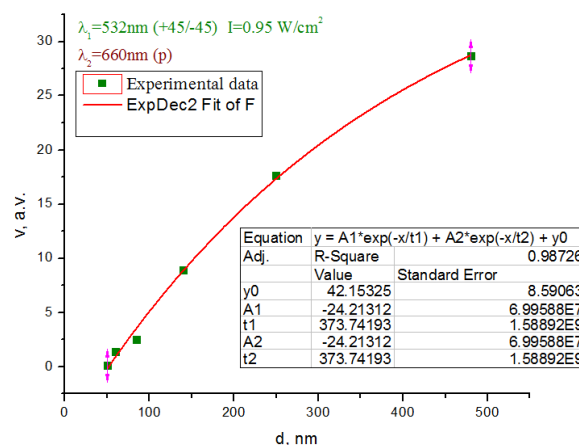


Figure 4. Recording velocity's dependence on thickness of the sample.

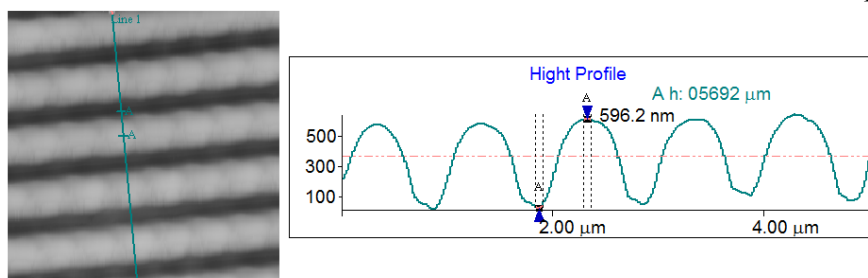


Figure 5. Profile of SRG recorded by intensity of $I = 0.95$ W/cm²; $t = 3$ min; $d = 500$ nm

The depth of SRG exceed the thickness of the sample 1.3 times, but ratio $h/\Lambda = 0.65$. Obtained result shows a very efficient and fast SRG formation during holographic recording. Chemically attached azo-dye to the main polymer chain provides effective mass transport, since azo-dye moiety is moving along with polymeric chain. Glass transition temperature of P(DR1-MA) is low – $T_g = 82^\circ$ C, thus there is low viscosity and deep SRG can be recorded. Gratings stay permanent after holographic recording is done.

The formation of SRG strongly depends on thickness of the P(DR1-MA) film (figure 4). If we want to discuss the velocity of grating formation, we have to measure a tangent of linear part of diffraction efficiency curve: $tg\alpha = v$ [4]. The velocity increases according to exponential decay function shown in figure 4. The saturation of the curve is expected at $d = 1.5$ μm, due to high absorption. The radiation can not penetrate the bottom layer of the film and velocity of SRG formation does not increase for thicker films.

The profile of obtained SRG by intensity $I = 0.95$ W/cm² recorded for $t = 3$ min in the film with the thickness of $d = 500$ nm is shown in figure 5. The depth h of SRG exceed the thickness of the film 1.2 times, but ratio $h:d$ strongly depends on the thickness d . In this case, ratio $h:d$ is limited by period of grating. Using thinner films ($d = 120$ nm), $h:d = 2$.

4. Conclusions

In this work experimental study of holographic recording in Poly(Disperse Red 1 - methacrylate) thin films is present. We observed efficient surface relief formation in this material. We showed that formation process strongly depends on the thickness of the film itself as well on the intensity of recording beams. The study of SRG formation kinetics showed that the fastest SRG formation takes place in the first seconds of recording, when the average speed reaches 12.3 nm/s. The depth of SRG

exceeds the thickness of the sample 1.3 times, but the value strongly depends on the thickness of the sample.

In Poly(Disperse Red 1 - methacrylate) thin film all the material applied on the glass substrate forms SRG making material usage maximal. The depth of SRG is limited by grating period and surface tension.

Acknowledgement

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