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Tridimensional surface relief modulation of polymeric films

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1. Introduction

The continuous need of miniaturization and highly integrated functionality related also with the need to use ecological processing methods resulted in the last decade in new processing methods. As an example, electronic components require conducting traces smaller than 100 μm , micro optical components and structures are integrated in the volume of the material in order to develop MEMS and MOEMS components, increasing the efficiency of the photo-luminescent diodes and solar cells or in the field of organic micro-lasers and biomedical materials. In this idea, diffraction gratings are important components due to their broad range of applications in integrated optics. The present chapter presents some results about single step surface relief modulation of the polymeric films in order to create integrated structures in materials with known properties. The studied polymeric materials are photoresist and azopolymers. The problem of tridimensional surface relief modulation is presented from two points of view: (i) technical possibilities to obtain surface relief gratings in a single step process on polymeric materials; (ii) physic-chemical processes responsible for single step surface modulation under the action of light and time stability. A general accepted technique for micro and nanoscale processing technologies in microelectronics is lithography, which is the exposure of a photoresist to a light pattern followed by a developing stage. This is a two step method, which is time consuming, non-localized and in the second stage (developing stage) is using non-ecological solvents.

A single step patterning method is laser ablation, with the advantage of the high processing speed and the possibility of small or high areas patterning. However, this method is locally changing the material properties, and it is important in some applications to preserve the composition and structure of the host material. A single step processing technique able to create surface relief modulation preserving material properties was already demonstrated. For the first time pure laser induced periodic structures (without any ablation or any larger structure) of submicron size (period and amplitude 0.2 μm) were obtained on polymer surfaces poly(ethylene terephthalate), poly(butylene terephthalate) and polystyrene, by irradiation with one thousand pulses of the polarized beam of the excimer laser (193 and 248

nm) (Matthias et al. 1992). By using the fourth harmonic of Nd:YAG laser (266 nm) periodic structures were created on the surface of poly(ethylene terephthalate), polyimide type Kapton (Hiraoka & Sendova, 1994). Under the 514 nm laser beam from an argon laser the surface of an azoaromatic polymer was optically altered in order to induce a highly efficient diffraction grating (Rochon et al. 1995). A single step processing method for surface modulation has been developed in order to obtain submicron structures for special applications and was reported (Dyer et al., 1996), (Castex et al., 2002). There were obtained surface relief gratings on PMMA and on the polymeric materials based on carbazole chromophores in order to develop a plastic blue laser emitting around 400 nm (Castex et al. 2006),. Single step recording of sinusoidal surface grating in hybrid sol-gel glasses was reported (Pelissier et al., 1999) and also holographic patterning of acrylamide-based photopolymer surface (Naydenova et al. 2005). Generally, the materials were polymers commercially available or special applications were developed and not a unitary explanation was produced for this effect. Recently, at the SPIE Conference: Technologies for the New Millennium in Dresden, may 2009, we have observed a growing interest about polymeric thin film structuration for applications in microelectronics, but a low understanding of the phenomena involved in the structuration process. In the idea to contribute to a better understanding of the capabilities of the single step surface modulation under the action of UV laser radiation we present our results obtained on different materials like commercial polymers (respectively photoresists), but also on laboratory synthesized azopolymers. The study of surface induced structuration under the action of UV laser radiation on polymers with very well controlled structures gives us the possibility to propose also mechanism for the laser induced effects.

2. Polymeric materials used for surface relief modulation

An important part of our studies is devoted to surface structuration of azo-polymeric materials. These materials are based on polysiloxanic chains, modified with azobenzenic groups and nucleobases (Hurdud et al. 2007) (Fig. 1). The presence of the nucleobases in the side-chain, are justified by the potential applications in biology. Two types of applications are possible: immobilization and laser nano-manipulation of biomolecules on the film surfaces and directional cells growth on the nanostructured surfaces. Details concerning the polymers synthesis and characterization were previously reported (Hurdud et al., 2007).

Fig. 1. Chemical structure of the azo-polysiloxanes modified with nucleobases

During the photo-chromic studies of the azo-polysiloxanes modified with nucleobases, a mechanism concerning the possibility to generate a fluid phase under UV/VIS irradiation was proposed. This mechanism is based on the concept of *conformational instability* state. This special state is a consequence of the continuous azobenzene groups *trans-cis-trans* photo-isomerization processes, which may be accompanied, as a function of chemical structure, by strong dipole-moment fluctuations along the chain (i.e. in the case of azobenzene, the dipole-moment is 0.1 D for *trans* configuration and 3.5 D for the *cis* one (Shishido, et al. 1997). If the azobenzene group is connected in the polymeric main- or side-chain, these modifications will impose conformational changes on the entire polymeric chain level, accompanied by strong dipole-moment fluctuations along the chain. This continuous photo-isomerization motion of the azo-groups induces a conformational instability on the entire polymeric chain that hinders the phase stabilization in a solid state. The photoisomerisation kinetic curves are studied for some azopolymers (Enea et al. 2008 a), (Enea et al. 2008 b) (Fig. 2, 3). The high response speed of the azobenzenic groups at UV irradiation in the solid state comparing with the solution may be explained by the flexibility of the main chain with a polysiloxanic structure and by the amorphous film structure that assures a high free volume. The *cis-trans* azobenzenic groups' relaxation phenomena can take place thermal-activated only (in dark) but in this case the processes are much slower (in a time scale of days). Therefore, a big difference in the film surface response can be expected if the operational conditions are modified (presence or absence of visible light during UV irradiation).

Fig. 2. Photo-isomerization kinetic curves obtained in the solid state (film): the *trans-cis* isomerization process under UV irradiation and in the presence of the natural visible light; the *cis-trans* relaxation process in dark (azo-polysiloxane modified with thymine units)

Fig. 3. *Cis-trans* relaxation process at the dark, in solid state (azo-polysiloxane modified with thymine units)

The absorption spectrum for a great number of azopolysiloxanes evidenced a maximum for the absorption spectrum in a region around of a wavelength of 350 nm, but also a tendency for higher absorption maxima at wavelengths lower than 200 nm (Fig. 4).

Fig. 4. UV-VIS absorption spectrum of the azo-polysiloxane modified with thymine

From the category of commercially available polymers we have used photoresists. They are well known and widely used in microelectronics. For their surface structuring microelectronics industry developed standard now technologies of their processing based on their characteristic to change their properties upon exposure to light. But all these processing methods are two step methods, time consuming and involving solutions considered dangerous from the ecological point of view.

3. Irradiation conditions for submicrometric single step surface relief modulation

Generally the leading idea was to obtain diffractive optical elements with controlled pitch, usually with submicron values in materials with special applications. In order to obtain surface relief gratings interferometric or holographic methods were proposed. The single step surface relief modulation method consists in the exposure of the polymer film surface to a light field with a controlled distribution of the light intensity in the irradiation pattern. To obtain a surface structuration without phase changes and discontinuities in the materials characteristics the maximum light fluence in the irradiation spot has to be lower than the ablation threshold of the material. A possibility is to create an interference field, generally in UV laser light. The majority of polymers have a maximum of light absorption at about 190 nm and also at about 350 nm, and as a consequence surface processing at these wavelengths is very efficient. Also the low wavelength in the UV region is recommended to create structures in the hundred of nanometers range. The 193 nm radiation is a wavelength emitted by ArF excimer laser. The problem is that the excimer laser presents a low spatial and temporal coherence to obtain interferometric pattern at this wavelength. To compensate

the low coherence the interference pattern is produced between the diffracted beams of a phase mask (Dyer et al. 1996, Castex et al. 2002). The interference field pitch can be of the same order of magnitude or half as the pitch of the phase mask depending if interference take place between $0, \pm 1$ diffraction orders, respectively between $+1$ and -1 diffraction orders (Fig. 5). Unfortunately that type of interferometer introduces some inconvenient: difficulties to manipulation, necessity of a protection fused silica sheet, not a direct control of the interfering beams, secondary multiple beams. Those are overcome with an interferometer setup (Fig. 6) using as a beam splitter a diffraction grating. Laser radiation is diffracted and the beams are separated by an angle θ ; at the interference, according to the interferometer scheme, in case two beams at θ angle, we obtain an interferometric image with the pitch equal with the pitch of the diffraction grating, or two beams separated by 2θ angle, give us half the diffraction grating pitch.

Fig. 5. Talbot interferometric scheme

In case of a grating with $1\ \mu\text{m}$ pitch to obtain the interference pattern we use only the ± 1 and 0 diffraction orders. The beams are used in pair $(+1, 0)$ or $(-1, +1)$ that can be selected with a beam selector (S).

To superpose the diffracted beams a pair of kinematic mirrors (M1, M2) that can be tuned according with the desired interference pitch grating and miss-alignments compensation are used. To compensate the phase difference between the beams, in case of $(+1, 0)$ diffraction orders interference, we have used a fused silica plate introduced in the zero order beam pathway. The light source, of our setup, was an Nd:YAG laser working on his third harmonic at 355 nm and with a pulse length at FWHM of 5 ns, with a repetition frequency of 10Hz and a beam divergence of 0.6 mrad. The main advantage of the set-up is the possibility to know and to control the energy of the interfering beams and consequently the fluence of the laser field incident on the sample. For this, the laser energy was permanently monitored by an energy meter (PM).

Fig. 6. The interferometer scheme: Nd:YAG laser @ 355nm, WF-wavelength filter, PM-Energymeter, D - aperture, G-diffraction grating, M1,2 -kinematics mirrors, Q - fused silica phase compensator, S - beam selector, P - sample, LT - translation stage

4. Surface relief modulation of polymeric films

We have obtained very good surface relief gratings (SRG) with submicron pitch on commercial photoresist or PMMA foil (Apostol et al., 2006). Gratings inscription was realized in a single step (no developing stage) under the action of an exciter laser with emission at 193 nm wavelength, pulse length 7 ns. The obtained SRG were characterized through various investigations means like optical microscopy, atomic force microscopy (Fig. 7, 8), and also with scatterometry. The characterization of the gratings is necessary not only for insight in the creation process of gratings but also for checking the reproducibility and uniformity. The diffraction efficiencies of the various orders diffracted by the grating were measured and they were fitted to theoretical predictions corresponding to various structure models of the gratings. The fitting procedure was used to provide the parameters of the gratings, such as the width, the grating height, the pitch or shape factors, such as the wall angles for a trapezoidal structure (Logofatu et al., 2008).

Fig. 7. AFM image and profile of the surface relief grating with the pitch of 250 nm, modulation depth 130 nm, induced on commercial photoresist under the action of an interferometric field at the wavelength of 193 nm.

The modulation depth of the SRGs obtained in the process of the surface relief modulation under the action of a structured light field depends on the incident fluence/intensity but also on the subsequent irradiation pulses number. As a consequence the modulation depth was possible to be varied between 10 nm to hundreds of nanometers. The smooth profiles evidenced by the AFM investigation of the obtained SRG evidenced that the surface modulation process is not induced by laser ablation. This fact suggests that an inner reorganization process of the polymeric chains take place and is responsible for the surface modulation. Generally the value of the incident irradiation light field intensity was between $10^5 - 10^6$ W/cm². The complexity of the photoresist material makes difficult to analyse the mechanism of the surface relief modulation under the action of a light field.

Fig. 8. AFM image and profile of the 1 μm pitch SRG printed on commercial positive photoresist.

In order to have a better control on the photochromic properties of the studied materials but also because of their specified applications we have studied single step surface relief modulation of azopolymeric materials. Based on the structural changes induced in the polymeric materials by UV light we have studied the possibility to induce tridimensional

surface structuration under the UV laser radiation in azopolymeric films. Also analyses of the photochromic properties of the azopolymers evidenced an absorption maximum at around 350 nm. In our experiments we have used the third harmonic of the Nd:YAG laser, at 355 nm. A group of azopolymers studied from the point of view of the possibility to induce surface relief modulation under the action of laser radiation in a under ablation threshold regime are the azo-azopolysiloxane modified with nucleobases (Enea et al., 2008) Irradiation geometry schemes are given in Fig. 5 and 6. The irradiation beam is the Nd: YAG laser harmonic of 266 nm or 355 nm, pulse length 5 ns. By using a phase mask of 1 μm pitch we have formed on the sample surface an interference image with the period of the same order of magnitude as that of the phase mask. In the same set up it is possible to go to a lower pitch of the grating in the hundred of nanometer range. The surface relief gratings induced on the azopolisiloxane films were investigated with a Zeiss AXIO Imager microscope and an AFM. Laser induced effects on the material surface was studied as a function of incident laser fluence and number of laser pulses subsequent on the same place.

Fig. 9. Evolution of the surface relief formation for a fixed incident laser fluence ($8.4 \text{ mJ}/\text{cm}^2$) for 10 pulses (*left*) and 100 pulses (*right*) and AFM profile (*right*) on for an azo-polysiloxane modified with thymine units film.

In Fig. 9 the evolution of the surface relief formation is evidenced for fixed incident laser fluence and for two values of the number of incident laser pulses. It can be seen that for a lower number of pulses the structuration which results in SRG formation is not complete and for 100 subsequent pulses a very uniform line structure was obtained. The low fluence for which the structuration was observed evidences that the mechanism responsible for the surface relief gratings formation is an inner material reorganization and not the material ablation. In Fig. 10 the evolution of the relief surface structuration as a function of incident laser fluence for a fixed number of incident laser pulses is considered. For the fluence of $35 \text{ mJ}/\text{cm}^2$ and 15 pulses material organization is not complete and the SRG is not yet completely formed. It can be seen also that using high irradiation fluence ($196 \text{ mJ}/\text{cm}^2$) situated near but under the laser ablation threshold a good SRG is obtained, but also some damages can appear on the surface.

Fig. 10. SRG formation for 15 incident laser pulses with incident fluencies of 35 mJ/cm² (*left*) and 196 mJ/cm² (*right*) on azo-polysiloxane modified with thymine units film

For surface relief gratings formation understanding and also for technical applications the time evolution and stability of the induced surface structure is an important parameter and is a problem to be discussed. In case of single step surface relief formation on polymeric materials the process is connected with the photo chromic behavior of the materials. As it is known the UV light induced effects on the material structure are reversible under the action of visible light, but with different speeds. The time evolution of the SRG obtained on polymeric films under the action of the UV light has to be analyzed from two points of view (Apostol et al., 2009):

- surface relief evolution during the light treatment process;
- surface relief evolution in time after structuration (time stability)

Generally the studies about the trans-cis isomerisation degree were realized under the action of the classical UV lamps and the isomerisation time is of the order of hundreds of seconds. Irradiation of the sample surface with the interference field, having as a light source the high power pulsed laser beam, induces effects in a period of the order of magnitude of the laser pulse duration. In our case the laser pulse has 5 ns at the FWHM (full width half maximum) and we have obtained effects of surface structuration even at 1 irradiation pulse. The laser irradiation intensity is lower than the material ablation threshold, in order to avoid damages. Surface induced structure analyses were realised, as we have mentioned, with high resolution optical microscopy and atomic force microscopy (AFM). Both techniques are not adapted for analyses of the effects induced under the action of laser treatment in-situ and during the patterning process. To be able to analyse the time evolution (or the evolution with the number of irradiation pulses) of the surface induced relief it was used a sequential technique. Based on the property of high reproducibility of the effects induced under the action of laser radiation (if the laser-matter interaction is realised in reproducible conditions) there were realised a great number of samples (irradiations) in known conditions (incident laser fluence, interference field structure, film surface quality as composition and cleaning). Sequences of irradiations for a constant incident fluence and different number of irradiation pulses give us the possibility to analyse the time evolution of the surface structuring process. Samples were analyzed immediately after laser treatment and after a known time with optical microscopy and higher time delays with AFM.

Fig. 11. Surface relief modulation evolution from disorder to order on the surface of a photoresist film: sequence of AFM images and AFM profiles of the surface relief formation for an irradiation fluence of 4 mJ/cm^2 ($I = 0.8 \times 10^5 \text{ W/cm}^2$) as a function of the irradiation pulses number (from up to down): for 20 pulses: surface structuration appears; for 50-100 pulses the modulation depth is 10-15 nm and a more regular organization of the surface relief and for 400 pulses the modulation depth is about 90 nm and SRG is formed

The induced surface profile time evolution during the multipulse irradiation has evidenced the evolution of the surface structuration from disorder to order, up to a sinusoidal profile corresponding to the surface grating formation (Fig. 11). If we compare the profile of the interference pattern calculated for the phase mask we have used in the experiments with the induced surface relief grating profile visualised with an AFM they are completely similar. (Apostol et al., 2009).

The surface structuration evolution is similar for the photoresist films and for the azopolymer films. The structuration time depends also on the incident laser fluence/intensity from 1 pulse (5 ns) to up to 500 pulses of 5 ns each. If we consider that the surface volume structuration effect is due to the trans-cis isomerisation effect we can consider that under the action of laser radiation the isomerisation time is much less than for the classical UV lamps. Taking into account the fast response of the material at the UV irradiation it can be considered that the surface relief formation is due to a spontaneous reorientation of the molecules due to the conformational changes as a result of the isomerisation process. It is considered that in this case the surface relief formation effect is reversible under the action of visible light or at the temperatures higher than the vitrification

temperatures of the material. The relaxation time reported is generally of the same order of magnitude with the isomerisation time. But in case of surface structuring under the action of laser radiation we have obtained for some of the studied materials very good time stability. In case of the photoresist irradiation we have obtained gratings lasting in good conditions as modulation depth and pitch for more than two years. Only if mechanically damages like scratches are produced the grating is damaged.

Fig. 12. Surface relief grating relaxation, after 24 h from the irradiation of the film of azopolysiloxane modified with azophenol (95-98)%; Irradiation conditions: Fluence = 17 mJ/cm², Intensity = 3.5 x 10⁷ W/cm²

Also in case of an azopolymer film surface structuration it is possible to observe the surface modulation evolution from disorder (Fig. 10, left) to order (Fig. 10, right). In case of surface relief structuration of azopolymer films the stability of the induced SRG depends on the type of polymer. To analyze the time stability of the induced structures on the surface of azo-polysiloxane modified with thymine units films the samples were analyzed also after a month, taking into account that the *cis-trans* relaxation curves under the visible light and in dark indicate relaxation times from 500 s to hours. The samples were kept at the normal ambient (summer) temperature. The microscope analyses evidenced the same structure without damage, so their time stability can be reported (Enea et al., 2008). In case of a sample of polysiloxane modified with cu azophenol (substitution degree 95-98%) the time evolution of the structured surface was monitored up to 30 hours from the irradiation time. In Fig. 12 can be seen the microscope images of the grating induced under the action of laser radiation at 355 nm at 15 min. after irradiation moment and after 24h (Apostol et al. 2009). A sequence of microscope images is presenting the evolution of the decay of the contrast in a grating which is disappearing from the surface in about 24 hours (Fig.11.). The host material is also polysiloxane modified with cu azophenol. It was selected in photos a region with small defects, to have a spatial reference to recognize the analyzed region. The sample was kept at the normal room temperature (about 23-26°C). It can be observed that the line contrast is reduced up to the complete disappearance of the lines after 27 hours (Fig.13.). In case of films of azo-polyimide, with rigid main chain and azo-polysiloxane modified with thymine with flexible main chain the surface structure was induced under the action of 1laser pulse (5 ns)

up to 500 pulses. The microscope image was realized after 15 minutes from irradiation and the AFM analyses after more than 3 month (Fig. 12.).

Fig. 13. Time decay of the surface relief grating in a film of azopolysiloxane modified with azophenol (95-98) %; Irradiation conditions: Fluence = 17 mJ/cm², Intensity = 3.5 x 10⁷ W/cm²

Fig. 14. Microscope and AFM images of the surface relief gratings on films of azo-polyimide (upper row) and azo-polysiloxane modified with thymine (lower row). The microscope images are registered 15 min. after irradiation time, the AFM images and profiles are registered after more than three month after irradiation time; irradiation conditions: fluence = 8.4 mJ/cm² and 100 irradiation pulses.

AFM profiles of the surface relief induced under the action of an interference field with a medium fluence of 8.4 mJ/cm² and 100 subsequent laser pulses are similar for both azo-polymers, with rigid and flexible main chain (Sava et al. 2008). The depth of the induced

structure is about 90 nm for the azo-polymer film and 100 - 110 nm for the azopolyimide film (Fig. 14.). The difference is made by the evolution of the structure with the number of incident laser pulses, respectively irradiation time. After only 10 irradiation pulses the height of the "hills" formed on the surface of azo-polyimide was half from the height of the profiles induced on the azo-polysiloxane films. This fact could be the result of the rigid main chain of the azo-polyimide for which the molecular reorganization is slower. For both azo-polymers the AFM analyse was realized at about 3 month from the irradiation moment. The samples were preserved during this time at ambient temperatures between 23 - 35 °C at daily light. This indicates that the surface structuration was stable for a rather long time.

5. Conclusions

Two classes of polymeric films were analyzed from the point of view of the capability to induce single step surface relief modulation in the form of SRGs under the action of a UV interference field having as a light source pulsed laser radiation at 193 nm or 355 nm wavelength: photoresists and azopolymers. The incident laser fluence was lower than the ablation threshold of the material and the transversal profile of the induced structures has a continuous shape, without phase changes.

There were obtained SRGs with a pitch of 250 nm and 1 μ m, depending on the irradiation set-up. The modulation depth was between 10 nm and 800 nm, depending on the incident fluence/intensity and the number of subsequent incident pulses. The surface relief modulation time is of the order of laser pulse duration (5 -7 ns). There were obtained surface relief gratings with sinusoidal profile on photoresist films. The obtained surface relief gratings had very good time stability from the point of view of the pitch and modulation depth. In case of the azopolymers the time stability of the SRG depends on the specific composition. For azopolysiloxane modified with azophenol (95-98) % the surface induced gratings begins to decay after 1-2 hours from the irradiation moment up to a complete loss of the structuration after 24 hour. A stable structure was obtained on the surface of films of azo-polyimide and azo-polysiloxane modified with thymine films. The surface structuration was monitored 3 month after irradiation and a good contrast of the surface relief structuration was observed. In case of azopolymers the single step surface relief modulation under the action of a light field is considered to be the consequences of the photo-induced conformational changes in the molecular chain. More generally the property of a polymeric material to have different configurations as a function of external stimuli (laser light in this case) offers the possibility to obtain surface relief structures in functional surface coatings with applications in biophysics, pharmaceuticals, electronics and optoelectronics.

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Polymer Thin Films

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This book provides a timely overview of a current state of knowledge of the use of polymer thin film for important technological applications. Polymer thin film book covers the scientific principles and technologies that are necessary to implement the use of polymer electronic device. A wide-ranging and definitive coverage of this emerging field is provided for both academic and practicing scientists. The book is intended to enable readers with a specific background, e.g. polymer nanotechnology, to become acquainted with other specialist aspects of this multidisciplinary field. Part A of the book covers the fundamental of the key aspect related to the development and improvement of polymer thin film technology and part B covers more advanced aspects of the technology are dealt with nano-polymer layer which provide an up-to-date survey of current research directions in the area of polymer thin film and its application skills.

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