

UNIVERSITY OF LATVIA
INSTITUTE OF SOLID STATE PHYSICS



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**HOLOGRAPHIC RECORDING AND SURFACE
RELIEF GRATING FORMATION IN
AZOBENZENE COMPOUNDS**

SUMMARY OF DOCTORAL THESIS

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Subfield of Solid State Physics

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IEGULDĪJUMS TAVĀ NĀKOTNĒ

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Introduction

Azo-compounds containing photosensitive chromophore groups are investigated due to their possible application in the optoelectronics and photonics industry. Resists made from azo-benzene compounds and polymeric chains can be used for direct holographic recording. This means that surface relief grating forms during the recording and no post-treatment grating is required.

In direct recording, resists on an organic azo-compounds base under laser illumination molecular alignment perpendicular to the light electric field vector take place, fast trans-cis isomerisation occurs, as a result there are changes in the material absorption coefficient, photoinduced birefringence and dichroism appears, and the required condition appears for surface relief grating formation during holographic recording.

Scientists proposed several explanations for surface relief grating formation mechanisms, but there are flaws and contradictions to the experimental results. Until now, there is not one certain model explaining all the phenomena for surface relief grating formation.

In this work different materials were studied: functional polymers, where a functional azo-compound is covalently attached to the polymeric chain; azo-benzene containing low-weight organic glasses and “guest-host” systems.

Surface relief gratings in the azo-compound were inscribed below material glass transition temperature by laser radiation with a wavelength located in the material absorption zone. The depth of the grating reached several hundreds of nanometers, but it strongly depends on the recording beam parameters and material properties.

The purpose of this work is to develop and investigate material, which can be used for direct holographic recording; observe fundamental processes in materials to explain the surface relief grating formation mechanism, which could promote the practical usage of a direct recording resist.

The doctoral thesis was carried out at the Institute of Solid State Physics of the University of Latvia from 2010 to 2018. The work contains an introduction, nine chapters, a conclusion and a reference list. The work is written on 153 pages.

The aims and tasks of the work

The aim of this work was the development of a material for holographic recording possessing the following characteristics:

- Photosensitivity of $\lambda = 532 \text{ nm}$ is $1\text{-}5 \text{ J/nm}^2$
- Cheap raw materials for synthesis
- Easy synthesis of the material

In order to fulfill these aims, the following tasks were set:

- 1) Synthesize resist for direct holographic recording with good photosensitivity at a wavelength of $\lambda = 532$ nm;
- 2) Investigate the process of holographic recording in organic azo-compounds depending on different recording parameters – intensity, polarization of the recording beams, grating period, thickness of the resist;
- 3) Explain the experimentally obtained results;
- 4) To increase the photosensitivity of a material by optimizing recording conditions and modifying synthesis;
- 5) To explain the surface relief formation mechanism.

Performance of the work was organized by the following method: firstly, material was synthesized or purchased; using this material, a thin film was developed. The thin film's optical properties were investigated – optical spectra, photoinduced dichroism and birefringence and a holographic recording was performed. The obtained data were processed and based on the results, the content of the material was modified or a different compound was investigated.

Scientific Novelty of the work

- Azo-epoxy resist synthesis was developed and holographic recording parameters were optimized for this compound;
- Suggested non-holographic, controlled self-enhancement process for surface relief grating;
- Functional polymers, low-molecular organic glasses and „guest-host” systems on azo-benzene base were investigated and compared;
- Indirect method for mass transport direction determination was developed;
- Surface relief grating formation mechanism was suggested.

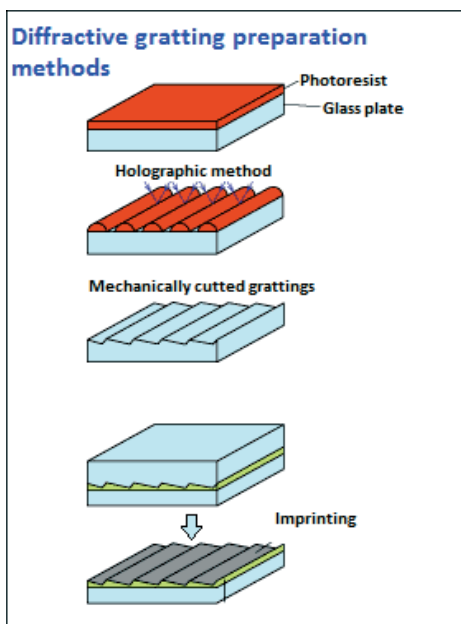
Thesis

1. Mass transport direction can be obtained indirectly, by investigating changes in diffraction efficiency monotonicity during holographic recording.
2. Mass transport during surface relief grating formation occurs in the materials possessing photoinduced birefringence phenomena.
3. Azo-epoxy AAB:BADGE compound is a promising material for direct holographic recording due to easy synthesis and good photosensitivity.

1. Literature review

1.1. Diffraction grating

A diffraction grating is a collection of reflecting (or transmitting) elements separated by a distance comparable to the wavelength of light under study. It may be thought of as a collection of diffracting elements, such as a pattern of transparent slits (or apertures) in an opaque screen, or a collection of reflecting grooves on a substrate. Physical explanation of the diffraction grating is light refractive coefficients periodic spatial variation. Electromagnetic wave after interaction to diffractive grating obtains electric field amplitude and phase or both determinate by diffractive grating light refractive coefficients periodic spatial variation



Pic.1.1: Preparation methods of diffractive gratings

Grating can be transmission or reflection in dependence on operating principle. Reflection grating is grating, which is pressed on reflective surface. Transmission grating - grating which is pressed on transmission surface.

Grating can be divided in amplitude (periodical amplitude variation) and phase (periodical phase variation) grating.

Based on preparation methods grating can be divided into 3 forms (pic. 1.1.): gratings obtained in mechanical cutting way. Characteristic of this grating is triangle profile. Gratings can be prepared by holographic method. Grating's profile is sinusoidal repeating interference pattern of recording light. Mechanical and holographic diffractive gratings can be multiplied by imprint method [1-3].

1.2. Holography

In holography light diffraction and interference properties are used to record picture of object in photosensitive material and reconstruct a picture in three dimensions. For this process coherent light source is required. The simplest hologram is recording of interference patterns produced by two laser beams crossed on photosensitive material.

1.3. Materials

1.3.1. Azobenzene compounds

Azobenzene is stable compounds. They are studied as small molecules added to some molecular structure or build into amorphous, crystalline, liquid crystal structure, self-assembled monolayers and superlattices, sol-gel silica glasses, and various biomaterials. A number of small molecules incorporating azobenzene have been synthesized, including crown ethers, cyclodextrins, proteins such as bacteriorhodopsin, and three-dimensional (3-D) polycyclics such as cubane and adamantane. Typically, azo chromophores are embedded in a solid matrix for studies and devices. As a result, matrix effects are inescapable: the behavior of the chromophore is altered due to the matrix, and in turn, the chromophore alters the matrix. Although either could be viewed as a nuisance, both are in fact useful: the chromophore can be used as a probe of the matrix (free volume, polarizability, mobility, etc.), and when the matrix couples to chromophore motion, molecular motions can be translated to larger length scales. Thus, the incorporation strategy is critical to exploiting azobenzene's unique behavior [4].

1.3.2. "Guest-host" system

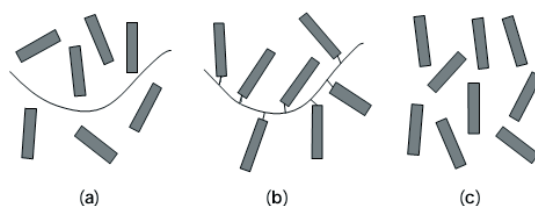
Receptors or host molecules in chemistry is a molecules or supramolecules who can selectively distinguish and bound guest molecules (pic. 1.3.2.1.). The bound between guest and host molecules is not covalent, molecules in the system is not attached to each other. The bound between guest

and host molecules usually is hydrogen bound, ionic bound, Van der Waals force or hydrophobic interaction.

It is important to optimize “guest-host” system for its practical usage. There are different factors affecting guest-host interaction: size, shape of the molecules, polarity, charge, hydrophobicity and hydrophilicity [5-6].

In order to use photosensitive properties of azo-benzene, molecule should be interposed between matrix molecules. The easiest way is azo-benzene interposing between polymeric chains. It requires only mixing up both components [7].

An azobenzene photochromatic molecules photochemical excitation can initiate reversible structural changes. If such molecules are inserted into host system, guest and host molecules can be reversibly bounded and make a host molecule photoswitchable [5]. This process in most guest-host systems is ineffective.



Pic. 1.3.2.1. Azo-benzene containing types of materials, a) “guest-host” system; b) azo-benzene containing functional polymers; c) azo-benzene containing low molecular weight organic glasses [4]

There are several disadvantages of guest-host system: guest and host should be soluble in one solvent; macroscopic phase separation; separation of chromophors; ineffective photochromatic switching, etc. [6].

1.3.3. Azo-benzene containing functional polymers

In azo-benzene functional polymers azo-compound is covalently attached to polymeric chain. In order to bound azo-benzene to polymeric chain two methods can be used: azo-compound can be added to polymer after polymerization reaction [8] or bound azo-benzene to monomer before polymerization reaction by free radical polymerization method [9-10].

Schematic functional polymer structure can be seen in picture 1.3.2.1.b. If azo-benzene is covalently attached to the main polymeric chain, it can be

moved together with azo-benzene chromaphoric molecule in the presence of electric field. Photoinduced processes are observed in solids and liquids [11].

The main disadvantages of functional polymers are difficult and expensive synthesis, because monomers can influence polymerization process - inhibit or stop it; there are small yield of pure compound, so post-purification is required. Azo-compound can change structure of monomer making polymerization process impossible [12].

1.3.4. Azo-benzene containing low-weight molecular organic glasses

Azo-benzene containing low-molecular organic glasses (pic. 1.3.2.1.c) are relatively new materials for application in optics. They are used as molecular crystals and polymer matrices.

Azo-benzene containing low-molecular organic glasses functional molecules are massive with defined size, with no tend to crystallization.

Glasses are simple in preparation; they can be obtained by sputtering method or from concentrated solvent. Obtained material is homogeneous and reproducible. Optical properties of such material can be observed more easily [13].

Azo-benzene containing low-molecular organic glasses molecules are weakly bounded to each other, so the motion is not inhibited and molecules can be easily moved in the presence of light electric field [14].

1.3.5. Azo-epoxy compounds

Epoxy can be obtained if epoxy resins are cured with hardener at appropriate conditions – increased temperature, applied electromagnetic radiation. Mechanical properties of epoxy strongly depend on epoxy segments elasticity and density.

Epoxy can shrink; shrinkage coefficient depends on epoxy content. Shrinkage creates internal stress in the material. It can be reduced by hardener agents.

Properties of epoxy depend on thermal treatment conditions. If temperature does not exceed glass transition temperature T_g , monomers can't move and hardening reaction stops. If temperature is increased, reaction can go on and epoxy is hardened.

Epoxy possesses good electric properties and adding metallic practical making epoxy a conductor. In general, epoxies are known for their excellent adhesion, chemical and heat resistance, good-to-excellent mechanical properties and very good electrical insulating properties. Different properties

allow using epoxy in many fields. More important are coating for painting, wires, auto and sea technologies; different material in construction.

As hardener amines, acids, acidic anhydrides, phenols, alcohols and triols can be used [15-16].

If we use azo-benzene with amino group, like 4-aminoazobenzene, it becomes possible to covalently attach azo-benzene to epoxy resin. At increased temperature oxirane ring opening reaction takes place and amino group attaches to the epoxy resin molecule.

The length of polymer chain depends on many parameters:

- Solvent
- Epoxy and aminoazo-benzene molar concentration: $c_{azo}:c_{amino}$
- Temperature
- Number of oxirane ring in one epoxy molecule
- Number of amino groups in aminoazo-benzene compound.

Azo-epoxy can be applied on glass surface and resist for direct holographic recording can be produced. Synthesis of such resist is easy, cheap and no high toxicity materials are used.

2. Experimental

2.1. Holographic recording

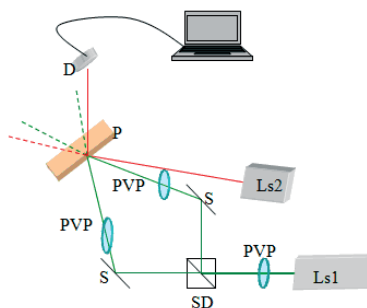
Holographic recording was performed using classic two-beam experimental set-up. It is based on two beam interference recording on a surface of a sample (pic. 2.1.1.).

By Glen-Taylor polarization cube laser beam was split into two equal intensities recording beams. Half-wave on quarter-wave polarization plate allowed controlling polarization state of recording beam. Using mirrors beams were diverted to the sample and crossed on the surface of the sample.

Produced interference pattern formed grating on the surface or in a volume of the sample in dependence on material-light interaction.

Diffraction efficiently of recorded grating was measured in transmission mode behind the grating (pic.2.1.1) of in reflection mode – before the grating.

For diffraction efficiency read-out low intensity diode laser beam was used. Reading beam wavelength had low intensity and low absorption in the material, thus it didn't effect holographic surface relief grating formation [17-20].



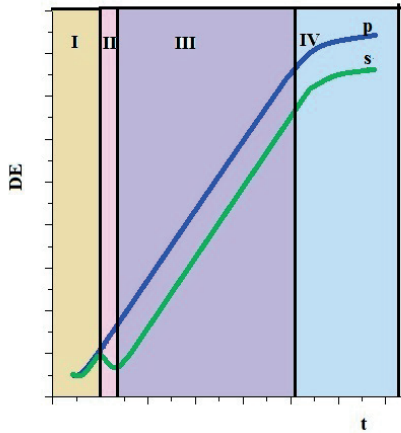
Pic. 2.1.1. Holographic recording setup

2.2. Determination of mass transport direction using diffraction efficiency curve of reading beam

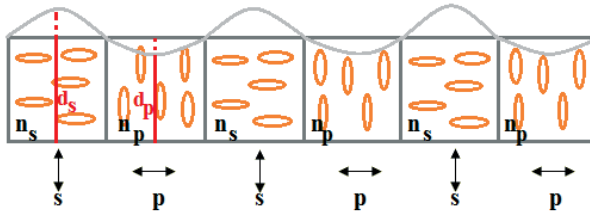
Direction of mass transport during holographic recording can be determinate indirectly, analyzing diffraction efficiency of reading beam in the beginning of the recording. Recording should be performed by $+45/-45$ recording beams producing on the surface of the sample.

On pic. 2.2.1. schematic DE dependence on time is shown. In area I changes in DE occurs in the first seconds of recording (typically 1-60 s, in dependence on photosensitivity of material), while there is no mass transport yet. When polarized light illuminates photosensitive material *trans-cis*

isomerization process takes place and molecules align perpendicularly to light electric field direction (pic. 2.2.2).



Pic. 2.2.1 Schematic curves of diffraction efficiency read-out by *p*- and *s*-polarized reading beams in reflection mode



Pic. 2.2.2. Azo-molecules alignment in grating in the presence of light electric field produced by +45/-45 recording beams

Photoinduced birefringence is an inherent property for photosensitive material for holographic recording, thus refractive index n_s and n_p values became different during the recording. Typically, organic materials and all materials studied in this work possess positive photoinduced birefringence, meaning:

$$n_p > n_s, \text{ where} \quad (2.2.1)$$

n_s – refractive index in the “*s*” polarization area (pic.2.2.2) or ordinary beam, n_p – refractive index in the “*p*” polarization area (pic.2.2.2) or extraordinary beam.

Diffraction efficiency is product of interference of waves which make different optical path nd in the media. In the area I difference in n_s and n_p values appears leading to optical path $n_s d_s$ un $n_p d_s$ difference, thus phase shipment appears and DE can be observed. There is no difference for *s*- and *p*-polarized reading beam, thus DE for both polarization is equal.

Parameters in area I:

n_s and n_p – variable;

d_s and d_p – const.

Difference in DE curves appears in *II* area, when mass transport takes place and surface relief grating starts forming. DE curves start to change for *s*- and *p*-polarization. Since photoinduced birefringence is positive $n_p > n_s$ and in the beginning:

$$n_p d_p > n_s d_s. \quad (2.2.2)$$

If reading beam is *p*-polarized, n_p regarding to reading beam have value of refractive index n_s , since writing and reading beam is perpendicular to each other. In “*s*” area reading and whitening beams are parallel and corresponding refractive index is n_p . In this case inequality (2.2.2) changes to opposite:

$$n_p d_p < n_s d_s. \quad (2.2.3)$$

Changes takes place due to change of point of reference.

Knowing that photoinduced birefringence value changes very rapidly, its maximal value is reached in *I* area. In *II* area changes of photoinduced birefringence are neglectable, thus DE changes determine surface relief grating formation.

Parameters in area II:

n_s and n_p – const;

d_s and d_p – variable.

If DE increases and slope of the curve is positive in *II* area, surface relief grating and volume polarization grating (refractive index changes) cases phase shift in one direction and d_s becomes greater than d_p . This case corresponds to *p*-polarization read-out and inequality 2.2.3.

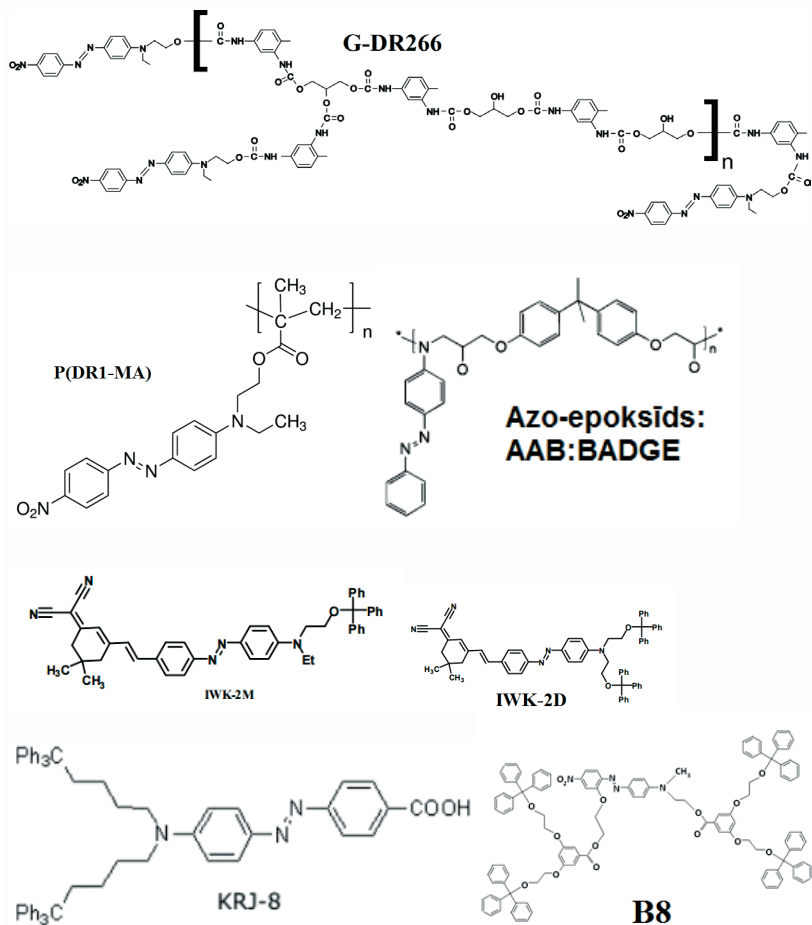
If slope of DE curve is negative, surface relief grating shifts phase in opposite direction than refractive index changes. Corresponding inequality – 2.2.2. Read-out is made by *s*-polarized beam.

For material with negative photoinduced birefringence like, chalcogenides, opposite DE curves in *II* area are observed – read-out made by *p*-polarized beam gives curve with negative slope, but *s*-polarized beam gives DE curve with positive slope [21-22].

In *III* area DE increases due to surface relief grating formation process, where d_s increases and d_p decreases. For some materials surface relief grating formation process is very effective and $d_p = 0$.

In the *IV* area saturations of DE is observed, maximal DE value is reached. Further process depends on material photoinduced properties. Maximal value for *s*-polarized beam is slightly bigger due to additional DE from volume polarization grating.

2.3. Studied materials



Pic. 2.3.1. Chemical structure of studied materials

In this thesis different materials were studied: functional polymers where azo-compound is covalently attached to polymeric matrix: DR-266 (pic. 2.3.1.); similar by compound and structure DR-342 but greater quantity of azo-compounds; commercially available P(DR1-MA) (pic.2.3.1.). Low-molecular organic glasses: AAB:BADGE, IWK-2M; IWK-2D, KRJ-8, B8 (pic.2.3.1.); “guest-host” system – azo-dye and gelatin mixture; DR1-PMMA.

3. Holographic gratings formation in functional polymers and low-molecular organic glasses

3.1. Kinetic

Curves of diffraction efficiency give all the information about surface relief formation process. It can be seen when grating forms, when grating reaches the maximal value and when erasing starts.

On the pictures 3.1.1. and 3.1.2. holographic recording curves in Poly(Disperse Red 1 – methacrylate) films are shown. Diffraction efficiency curves in transmission and reflection modes differs (pic. 3.1.1.). In the reflection mode diffraction efficiency depends on film reflection coefficient, thus values in refraction mode is lower due to low refractive coefficient in organic compounds. The value of refractive coefficients is approximately 10%. It is important that diffraction coefficient in refractive mode is measured by shorter wavelength - $\lambda_1 = 532$ nm, while in transmission mode wavelength $\lambda_2 = 660$ nm is used. Diffraction efficiency according to Kogelnik formula for sinusoidal gratings has inverse proportionality to wavelength [23]:

$$\eta = \sin^2 \left(\frac{\pi \Delta n d}{\lambda \cos \varphi} \right), \quad (3.1.1.)$$

where:

η – diffractive efficiency;

Δn – modulation of refraction coefficient of light at measured wavelength;

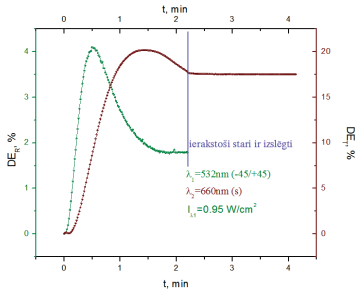
d – thickness of the sample in transmission mode/ height of surface relief grating in reflection mode;

λ – wavelength;

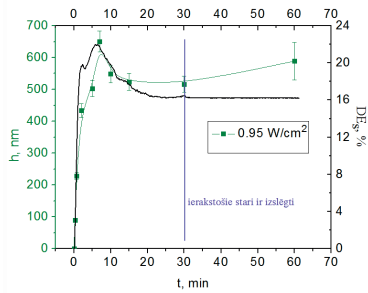
φ – an angle between grating vector and the beam.

[17; 24].

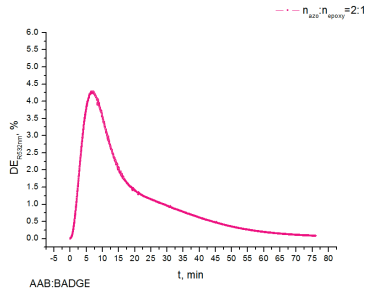
Holographic recording in AAB:BADGE films was studied in reflection mode (pic.3.1.3.), because volume grating formation in this material was not a research subject. Similar to P(DR1-MA) films, diffraction efficiency in AAB:BADGE films reaching maximal value indicates the depth of surface relief grating approximately $\lambda_1/2 = 250$ nm. This statement corresponds to AFM measurements data. Further decrement of DE indicates formation of surface relief grating, according to Kogelnig formula (3.1.1.). Monotonic decrement of DE is observed until maximal depth of surface relief grating is reached. For AAB:BADGE it is $h_{max} = 500$ nm for film thickness $d = 500$ nm.



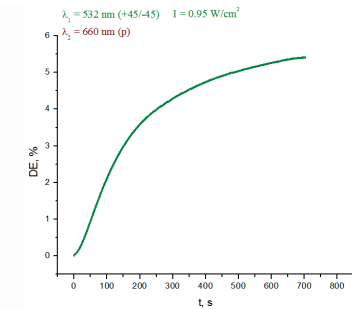
Pic. 3.1.1. Curves of diffraction efficiency in reflection mode (green line) and transmission mode (red line), and thickness of the sample $d = 250$ nm, sample: Poly(DRI-MA)



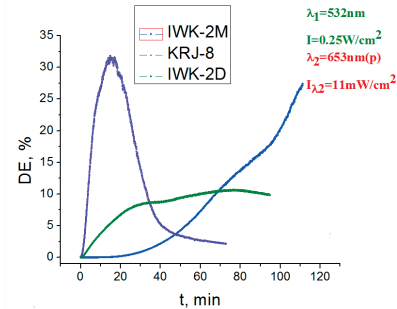
Pic. 3.1.2. Curve of diffraction efficiency in transmission mode and corresponding height of surface relief formation modulation h ; thickness of the sample $d = 500$ nm, sample: Poly(DRI-MA)



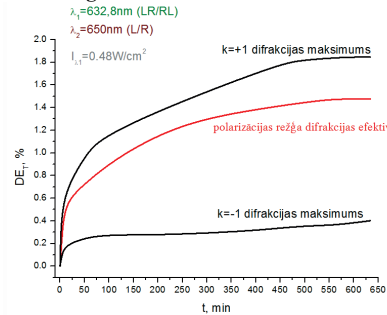
Pic. 3.1.3. Holographic recording in AAB:BADGE film by intensity $I = 0.90$ W/cm². Read-out made by recording beam in reflection mode



Pic. 3.1.5. Holographic recording in B8 film; intensity $I = 0.95$ W/cm², read-out made by reading beam in reflection mode



Pic. 3.1.4. Holographic recording in low-weight organic glasses IWK-2M, KRJ-8 and IWK-2D at equal conditions; read-out made by recording beam in transmission mode



Pic. 3.1.6. Holographic recording in D-GL266 film; intensity $I = 0.96$ W/cm², λ₁ = 532 nm (R-L), λ₂ = 650 nm (L/R), I₂ = 0.1 mW; read-out made by reading beam in transmission mode. ±1 maxima was measured (black lines); red line – calculated diffraction efficiency of polarization grating

In low-weight organic glasses curves of holographic recording is similar, but formation of surface relief grating proceeds with different velocities (pic. 3.1.4.). The best result was obtained in KRJ-8 films, and this material showed the best result between studied material within this thesis. The maximal depth of surface relief grating which can be obtained in KRJ-8 films is $h_{max} = 750$ nm (according to AFM data). Kinetics of holographic recording corresponds to Kogelnic formula (3.1.1.). The depth of surface relief grating in IWK-2W and IWK-2D films reach $h_{max} \approx 600$ nm, what is a good result, but it is required great expositions and long time to record such grating. Hardness of IWK-2W and IWK-2D film is much higher than KRJ-8, thus glass transition temperature is higher. There is higher tension in the volume of the material and greater doses of energy are required [25].

There is slightly different chemical structure of B8 low-weight organic glass. Azobounds are screened by benzene rings, thus interaction between molecules is week. Still B8 does not reaches KRJ-8 photosensitivity (pic. 3.1.5.). The maximal depth of surface relief grating is $h_{max} \approx 400$ nm [14].

Surface relief formation in D-GL266 and D-GL342 functional polymers is ineffective; however, volume polarization grating formation processes with high efficiency. Thus in this material research object was volume polarization grating formation (pic. 3.1.6.).

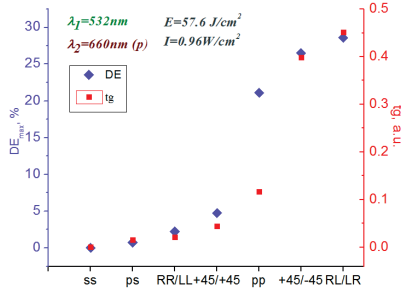
3.2. Dependence of polarization state

The most effective polarization states of recording beams are *RL/LR* and *+45/-45*. In the case of *pp* polarized recording beams the are intensity modulation on the surface and light electrical field vector is perpendicular to the grating lines. It helps to transport mass toward unenlightened areas, but efficiently of lower than using *RL/LR* or *+45/-45* polarized beams.

RR/LL and *+45/+45* polarized recording beams does not form noteworthy surface relief gratings, because vector of light electric field is directed in the angle toward grating lines, thus mass transport can't be effective.

Ss polarization state almost does not form surface relief grating. By *ss* polarization obtained depth of surface relief grating typically does not exceed 5 nm. In this case vector of electric field is parallel to the grating lines and mass movement is ineffective between illuminated and non-illuminated areas.

The experimental results showed *RL/LR* polarization state forms the best surface relief grating, nevertheless for further experiments *-45/+45* polarization state was used, because *RL/LR* polarization is technically harder to obtain, thus error given by inequality of polarization state should be considered. *+45/-45* gives slightly worse results, but polarization state can be installed correctly and results was repetitive.



Pic. 3.2.1. Velocity of surface relief grating formation *tga* and maximal diffraction efficiency in poly(Disperse Red 1 – methacrylate) films using different polarization states of recording beams

In all functional polymers and low-weight organic glasses dependence on polarization state of recording beams were similar – the best results were obtained by +45/-45 and *RL/LR* polarization state, slightly worse results were obtained by *pp* polarized beams. *RR/LL* and +45/+45 polarization state formed surface relief grating, but efficiency was weak. Intensity modulation produced by *ss* and *ps* polarization state forms very weak surface relief grating (pic. 3.2.1.).

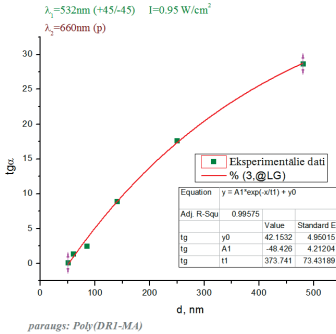
3.3. Dependence on the thickness of the sample

Velocity of surface relief formation *tga* has exponential dependence on thickness of the sample (pic. 3.3.1.). When surface relief grating forms, surface area increases due to sinusoidal grating formation. Surface tension ε_v of the film depends on tension coefficient σ of the material and surface area S [26]:

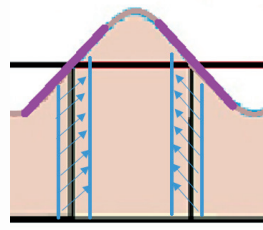
$$\varepsilon_v = \sigma S. \quad (3.3.1)$$

If surface area S increases surface tension increases as well in accordance to 3.3.1. Work done by external force (force of electric field vector of the laser beam) increases in order to surface relief grating continues formation.

Surface relief formation process is volume effect (pic. 3.3.2.). Mass transport process starts, when incident light penetrates the material. The deeper light penetrates the material, the greater light-material interaction area is. Material is pushed upwards, forming surface relief grating. Let's compare this process to squeezing elastic pipe filled with liquid – material moving upwards, forming surface structure. If thickness of the sample is greater than depth of



Pic. 3.3.1. Surface relief grating formation velocity dependence on thickness of the sample d .

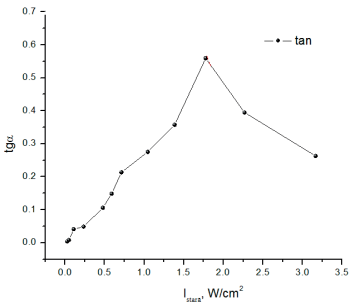


Pic. 3.3.2. Schematic representation of surface relief formation in the volume of the film

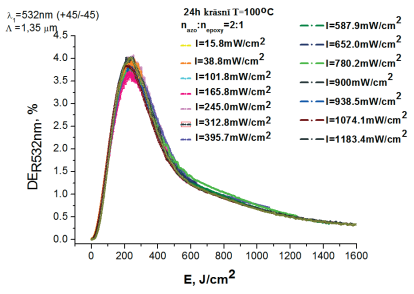
light penetration, further increment of film thickness does not affect surface relief grating formation process.

3.4. Dependence on recording beams intensity

Intensity of recording beam define velocity of surface relief grating formation. The greater intensity of the recording beam, the greater electric field vector gradient within half-period is produced and dielectric process can proceed faster (pic. 3.4.1.).



Pic. 3.4.1. Velocity of surface relief grating formation dependence on recording beams intensity in P(DR1-MA) film with thickness $d = 64$ nm



Pic. 3.4.2. Diffraction efficiency dependence on the exposure. Recording is performed by recording beams with different intensity in AAB:BADGE film

At lower intensities, for poli(disperse red 1 – methacrylate) film these intensities are $I < 2\text{W}/\text{cm}^2$, form grating faster, if intensity increases. Slight divergence from linearity could be result of sample thickness inequality what could reach $\Delta h = \pm 5\text{nm}$.

When intensity exceeds $I = 2.0\text{ W}/\text{cm}^2$, surface relief grating formation decreases. It could be explained by heating effects, it softens the material and inhibit grating formation.

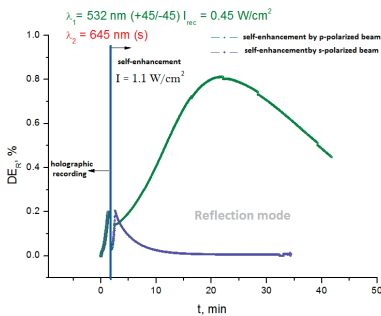
In others functional polymers and low-weight organic glasses intensity of recording beam dependence on surface relief grating formation velocity is similar to P(DR1-MA). One exception is AAB:BADGE films, where no dependence on intensity was observed in the range of $I = 15.8 - 1183.4\text{ mW}/\text{cm}^2$ (pic. 3.4.2.). It is very convenient in practical use, because recording have no dependence on intensity and it can be know how deep grating can be obtained if defined exposure is received.

3.5. Self-enhancement of surface relief grating

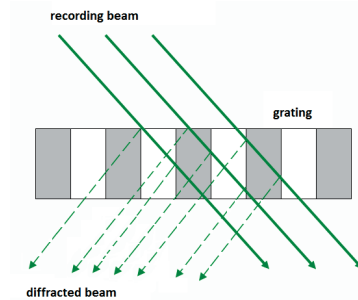
In azoepoxy compounds surface relief grating self-enhancement process has observed (pic. 3.5.1.) [27].

In self-enhancement process it is important to set correct parameters for enhancement beam.

If polarization state of enhancement beam is p regarding to grating vector, further grating recording takes place.



Pic. 3.5.1. Self-enhancement of surface relief grating using s and p polarized beams.



Pic. 3.5.2. Path of recording and diffracted beam in the grating

When grating is illuminated by one of recording beam, diffracted beam of -1^{st} order goes the way second recording beam should go (pic. 3.5.2.). As a result, interference pattern is produced and further grating recording becomes possible.

If polarization state of enhancement beam is p , interference pattern produced by enhancement and diffracted beam corresponds to p illuminated areas in $+45/-45$ polarized beams (both recording beams) and recording can proceed, like pp polarized two recording beams.

If enhancement beam has s polarization state, initial grating erases. In this case interference pattern is produced by enhancement and diffracted beam, but s polarization illuminated areas exchange with p polarization illuminated areas in $+45/-45$ polarization states of recording beams. It is known mass transport occurs from p polarized areas to s polarized areas [2.2. chapter]. In self-enhancement process mass transports takes place to opposite direction and grating erases. Since ss polarization state of recording beam does not form noticeable surface relief grating, further recording was not observed.

3.6. Application of low-weight molecular glasses

Within this work in KRJ-8 film pixel hologram – holographic sticker was recorded in corporation with G.Liberts' Innovative Microscopy Centre, University of Daugavpils (pic. 3.6.1.).



Pic. 3.6.1. Photo of surface relief-phase grating. Recorded in KRJ-8 film without etching post-treatment. Recording duration – 15h, diameter – 13.5mm.

This hologram was recorded by the image-matrix method without the operation of chemical etching. The diameter of the hologram is 13.5 mm, and the image is made up of frames size 160×120 mm. To modulate the colors used in the pixels of the hologram grating period of 900 to 1300 nm. The height of the relief hologram elements is 150–200nm. Such height defines a good visual brightness and can be replicated using standard techniques used in the

application of holography. The parameters of the manufacturing process of this hologram demonstrate the importance of reducing the energy of formation of the surface relief for the non-etching photoresist. This holographic image was recorded during 15h. Moreover, fabrication of such hologram by the traditional photoresist with the chemical etching process takes about 1 h.

Recorded hologram shows potential application of low-weight molecular organic glasses in holography. Material should be modified to increase photosensitivity [25].

3.7. Summary

Within this work different materials functional polymers and low-weight molecular organic glasses were studied. Each material has pros and cons.

Functional polymer P(DR1-MA) is commercially available material. In this material surface relief grating formation processed with high efficiency, height of obtained grating easily exceeded thickness of the film. Recording photosensitivity was $0.187 \text{ J/nm}^2\text{cm}^2$ what is one of the best result in this thesis. The cons are expressivity of material due to difficult synthesis. Samples thicker than 500nm could not be obtained. Altering of the material was observed – after long usage material lost its properties, films was not optically homogenous and surface relief grating formation process significantly slowed down, probably, due to function polymer chemical decomposition.

Low-weight molecular organic glass KRJ-8 is the most efficient material for surface relief grating formation studied in this work. Photosensitivity reaches $0.145 \text{ J/nm}^2\text{cm}^2$. Films were optically homogenous and there was no adhesion from glass plate. The cons of the material are difficult synthesis; post purifying is required [28]. There is no evidence of repetitive synthesis, thus in each synthesis slightly different material can be obtained, although significant differences in holographic recording for different synthesis was not observed.

In low-weight molecular organic glasses IWK-2M and IWK-2D surface relief grating formation was observed, depth of grating is high but required exposition are to big – photosensitivity is $5-6 \text{ J/nm}^2\text{cm}^2$. Synthesis of these materials, just like KRJ-8 synthesis, is difficult. Films had good optical quality, but low efficiency and high expenses does not make these materials perspectives for applications in holographic recording.

In low-weight molecular organic glass B8 recording photosensitivity reaches $\sim 0.5 \text{ J/nm}^2\text{cm}^2$, what is a good result. The cons are the same as to all low-weight molecular organic glasses.

In functional polymers on polyurethane base G-DR266 and G-DR266 surface relief grating forms with weak efficiency, but recording photosensitivity reaches $200 \text{ J/nm}^2\text{cm}^2$. Nevertheless good polarization volume grating and high values of photoinduced birefringence can be obtained in these materials. The

cons are low photosensitivity, non-repetitive synthesis. Chemical structure of material was not experimentally studied.

In azo-epoxy AAB:BADGE film photosensitivity reaches $0,91\text{J}/\text{cm}^2\text{nm}$, but this value can be increase at least 2 times by adding photosensitive azo-dyes, which increases recording beams absorption or by physical post-recording surface relief grating enhancent. The main con is insufficient photosensitivity.

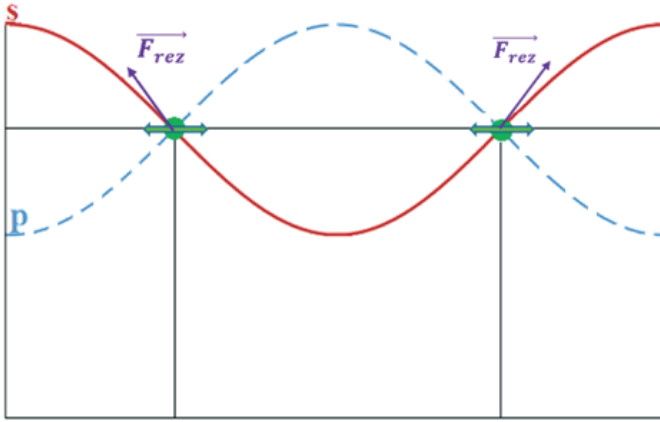
4. Surface relief formation mechanism

For surface relief grating formation dielectric materials was used; molecules posses dipole momentum. If such material is illuminated by interference pattern, where polarization modulation is present, dielectroforesis process takes place. This is driving force in surface relief grating formation [29]:

$$F(t) = (m(t) \cdot \nabla)E(t), \text{ where} \quad (8.1)$$

$m(t)$ – dipole momentum with time dependence

$E(t)$ – electric field with time dependence [29-31].



Pic. 8.3. Polarization modulation on the surface obtained by +45/-45 polarized recording beams

If polarization modulation on the surface of the sample is present (for +45/-45 and RL/LR polarized recording beams) on every surface point photoinduced driving force is applied. This point is dielectric partical with dipole momentum (molecule) [32;33]. Force this particle is experincing is described by 8.1. equation.

P and s polarization electric fields E is applied on the dielectric particle. According to Coulomb law electric field is inversely proportional to the dielectric permeability of the environment:

$$E \sim \frac{1}{\varepsilon}, \text{ where} \quad (8.2)$$

ε - dielectric permeability.

Dielectric permeability is proportional to light refraction coefficient:

$$\varepsilon \sim n^2, \text{ where} \quad (8.3)$$

n – light refraction coefficient [34].

According to the previous results it is known azopolymers have positive birefringence, meaning:

$$n_p > n_s. \quad (8.4)$$

Combining 8.2, 8.3, and 8.4, can be obtained:

$$E_p < E_s, \text{ where} \quad (8.5)$$

E_p – p -polarization electric field;

E_s – s - polarization electric field.

Driving force is proportional to electric field, thus particle will move toward s -polarization intensity maxima where maximal force will be obtained. This corresponds to experiments.

Surface relief formation is inhibited by surface tension depending on free surface energy.

5. “Guest-host” system

“Guest-host” system has significant difference in comparison to functional polymers in chemical content and efficiency. Azocompound and polymer are mixed in these materials and there are no covalent bound between them. Due to difference in chemical content there is difference in efficiency of holographic recording. Explanation for this is following: electric field interacts only to azocompounds, but polymer matrix does not move and does not participate in mass transport.

There are some polymer, where electrochemical reactions takes place under light illumination, as a result polymer chain shrinks or expands. This process can be observed in studied azo-gelatin films [20;35].

Summary

In azogelatin films weak surface relief formation was observed. Maximal depths of surface relief grating did not exceed $h_{max} = 25$ nm. The mechanism of surface relief formation in azogelatin films is simple – gelatin is swelling under light illumination, because surface relief grating is observed only if polarization state of recording beams with intensity modulation is choose.

During holographic recording in the volume of azogelatin films volume grating forms due to azomolecules isomerization process and bleaching of the gelatin.

In azogelatin films good photoinduced dichroism was observed, but photoinduced birefringence values were low.

Azogelatin films are easy to prepare, used materials are not toxic, are cheap and widely available. For sample preparation chemical laboratories are not required, they can be produced in home conditions.

High value of diffraction efficiency can't be obtained in azogelatin films, but photosensitivity can be significantly increased by providing acid environment in the film.

Conclusion

In this thesis, the experimental results of several materials for surface relief grating and volume grating holographic recording were gathered. The grating can be used in diffractive optical elements production. In the thesis, the synthesis of materials and the production of thin films are described; recording kinetics are discussed and its correspondence to a depth of surface relief grating in functional polymers, low-molecular organic glasses and “guest-host” system on azo-compound bases; the observed polarization state of recording beams influence on grating formation process. How thin film thickness influences surface relief grating depth is described, and it is shown that there are optimal film thicknesses for the grating with maximal depth which can be obtained. In the thesis it is shown that it is possible to determine mass transport direction using diffraction efficiency with different polarization states curves of a read-out beam in transmission mode. For this method it is not necessary to create an experimental set-up for mass transport direction determination or use extra materials. Surface relief grating formation with different periods and different intensities of recording beams were studied.

In order to understand surface relief grating formation mechanisms, photoinduced processes like photoinduced birefringence and photoinduced dichroism were studied. Surface relief grating formation’s dependence on different material properties like surface free energy, tension, direction of photoinduced birefringence and material chemical structure is explained.

In the studied material, photosensitivity does not meet the requirements for commercial holographic resists, still it is shown that the studied materials are suitable for art hologram production. Photosensitivity of a material can be increased by physical enhancement methods, thus further study in this field is required.

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