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Both holographic and electron-beam recording in new carbazolyl – containing photoresists

Andrei Andries*, Valeriu Bivol, Okan Ersoy[a], Stefan Robu,
Serghei Sergeev, Lyudmila Vlad

Center of Optoelectronics, Academy of Sciences of Moldova
1 Academiei Str., MD-2028, Chisinau, Republic of Moldova
[a]Purdue University, School of Electrical Engineering/West Lafayette, IN 47907-1285, USA

ABSTRACT

Electron-beam and holographic recording of diffraction gratings was processed in the layers of poly-N-poxypropylcarbazole (PEPC) and co-polymers of carbazolylalkylmethacrylate with octylmethacrylate (CAM:OMA) containing additons of CHI_3. The dependence of the diffraction efficiency of planar gratings on the recording current was studied. The influence of post-effect and storage in the dark on the diffraction efficiency is considered. By chemical development technique the reflecting relief diffraction gratings are obtained with the diffraction efficiency of 25-30 %.

Keywords: Holography, electron beam, recording, carbazolyl, polymer, photoresist, grating, diffraction efficiency

1. INTRODUCTION

As is known carbazolyls polymers from vinylcarbazolyl with oktylmethacrylats, and also N-polyepoxypropylcarbazolyl, in addition containing electron-acceptor additives of a iodophorm type and others under the action of a ultra-violet irradiation are exposed to a photochemical transformations, that conducts at the end to deep photopolymer structurization. Structurization of layers is accompanied by change of coloration of the irradiated sites, their solubility in the organic solvents, increasing of mechanical durability and adhesives properties of a material. Is established, that spatial cross-linked is carried out by means of a free radical-cations arising as a result of occurrence carbazolyl nucleuses with molecules of iodophorm.

With the purpose of expansion of a class of used polymer materials in the area of photonics and photolithography by us were prepared, tested and investigated photopolymer layers from carbazolylalkylmethacrylates (CAM) with oktylmethacrylates (OMA) copolymers added with 0-10% of iodinophorm CHI_3 and other cross-linking agents.
Recently in the literature the more and more strengthened attention render to organic photothermoplastic carriers of the information being in most cases by donor-acceptor systems. The donor as a rule serves carbazolyl or other polymer containing π-electronic systems – carbazolyl nucleus and in quality of acceptor use various low molecule connections containing an electron-acceptor groups forming with carbazolyl nucleuses a complex with carry of charge.

2. EXPERIMENTS AND RESULTS

Carbazolylalkylmethacrylate (CAM) copolymers with octylmethacrylate (OMA) of the common formula containing about 60 mol % of carbazole links were obtained by a method of radical polymerization in a tholuen solution.

The synthesis of co-polymers CAM:OMA was processed according to radical mechanism in solutions of organic solvents (toluene, benzol) at presence of 1-2 mol.% of azobisizobutyronytril (AIBN) at 80°C during 6-8 hours. Co-

*e-mail: bivol@as.md, Tel: (+373 2) 73 80 87, Fax: (+373 2) 73 98 05

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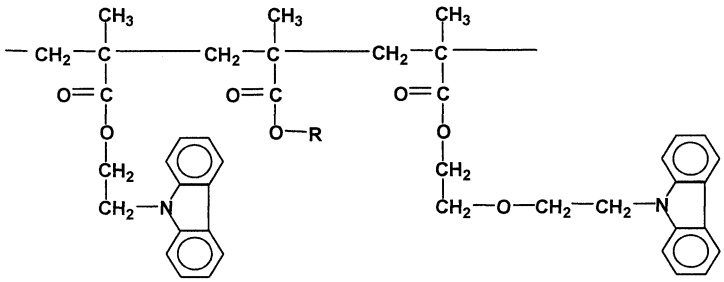
polymers CAM:OMA have been synthesized with various percentage of plasticizer OMA: 60:40 mol. % and 80:20 mol. %. The synthesis of the poly-N-epoxypropylcarbazole (PEPC) was processed by anion mechanism at presence of 2 % of potassium hydroxide at 120°C during 4 hours. Prepared co-polymers CAM:OMA and PEPC were re-precipitated from methanol and hexane, respectively.



For preparation of the samples the solutions of above indicated CAM and PEPC copolymers added with 0-10 % of iodinophorm CHI_3 or other cross-linking agents were prepared. The photopolymer layers were applied both on transparent poly(ethyleneterephthalate) films and on rigid substrates from an optical glass. The thickness of the samples ranged from 1,0 μm to 20,0 μm. The samples were dried up on air and then in a drying camber at $T^o \sim 40^oC$ within 24 hours.

2.1. Sensitometric Investigations

It is known that photo-chemical transformations in carbazol-containing polymers at the presence of structurizating agents like iodinophorm CHI_3 occur according to the ion-radical mechanism with formation at the issue of spatially – cross-linked structures, that allows to use them for registration of information. In the given part of work the sensitometric characteristics (photo-sensitivity, coefficient of visibility and other parameters) in dependence from the concentration of CHI_3 in a photopolymer layers and from their thickness, from the kind of radiation were investigated. The trial study was carried out on layers from PEPC taken as an analogue and from CAM:OMA copolymers. The layers were exposed to ultra-violet light with incident energy E = 10 - 20 mW/sm^2 and also to white light (mercury-quartz lamp PRK-4 and 500 W incandescent lamp as sources). The photo-structural transformations were observed visually as modification of colour of the layers and through losing of solubility of the irradiated areas. Quantitatively, the photo-structural modifications are well seen in the spectra of visible region.

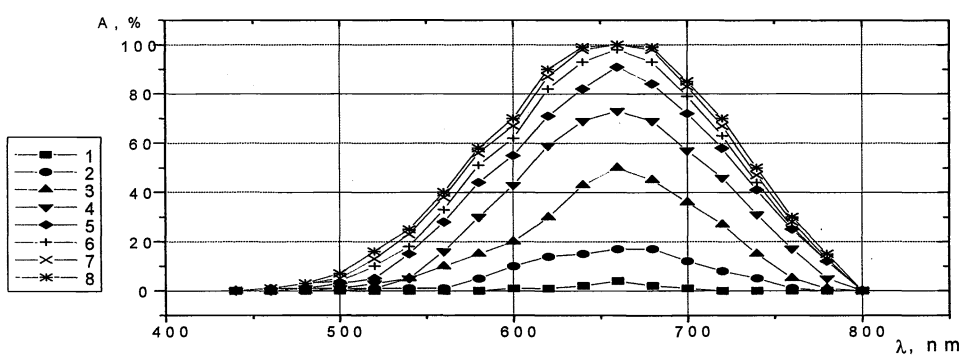


Fig. 1 Modification of the absorption intensity with the time of exposure of the layers:1 - 0 s; 2 - 5 s; 3 - 10 s; 4 - 15 s; 5 - 20 s; 6 - 25 s; 7 - 30 s; 8 - 40 s;

The maximum of visible absorption spectra (Fig.1) is observed in the interval 630-650 nm. As follows from Figure 1 in a defined time t = 30 s the intensity of absorption becomes constant and corresponds to a full photostructurization of the layers.

The process of a photo – structurisation of photorezists layers on the base of carbazolylalcjylmethacrylats copolymers containing threeiodinomethan (CHI_3) as the cross-linking agent, proceeds on the radical mechanism due to formation of complexes with carry of a charge (CCC) between carbazolyl nucleuses and CHI_3. CCC formation confirmed by occurrence of a new bend of absorption on 640-660 nm in visible area of a spectrum (Fig. 1). The complexes with carry of a charge being decomposed under action of ultra-violet radiation result in formation of free radicals, which are easily found out by electron–paramagnetic-resonant spectroscopy (EPR) (Fig. 2).

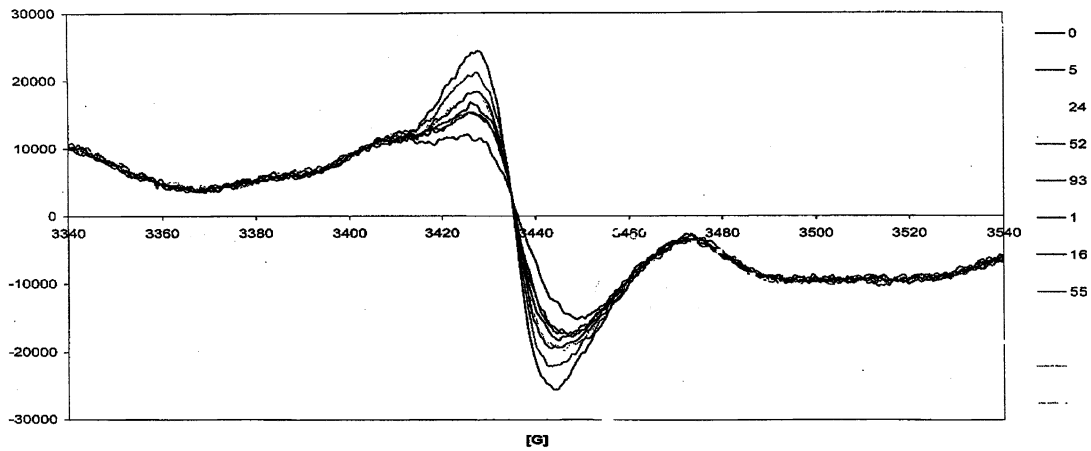


Fig.2 Electron–paramagnetic-resonant spectroscopy of CAM:OMA (80:20 mil%) layers

EPR spectra were measured on spectrumphotometer such as "BRUKER" at frequency 9,637 GHz in a range of 3350 – 3540 Gauss. Is shown, that at ultra-violet irradiation during 100 mines the concentration of free radicals grows almost in 2 times (Fig. 2 and Fig. 3). The switching off of a UV-source of radiation results in gradual reduction of concentration of free radicals (Fig. 3).

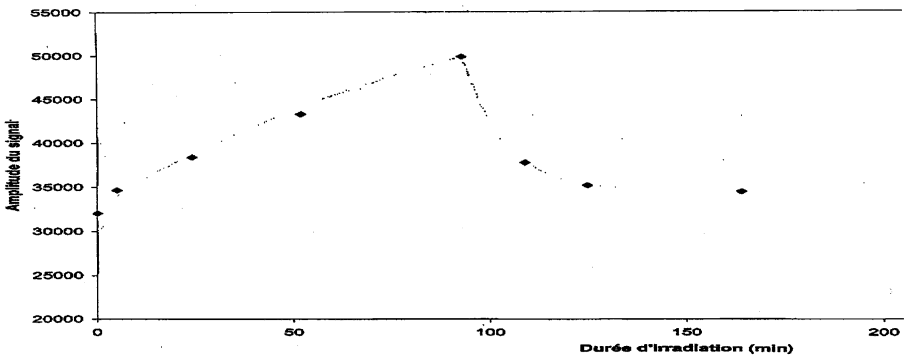


Fig.3 Dependence of concentration of free radical in CAM:OMA (80:20 mil%) layersafter switching on and switching off of a UV-source of radiation

The occurrence in IR-spectra of new bends of absorption at frequencies 803 $см^{-1}$ and 877 $см^{-1}$ entitles to consider, that structurization proceeds in positions 3 and 6 of a carbazolyl nucleus.

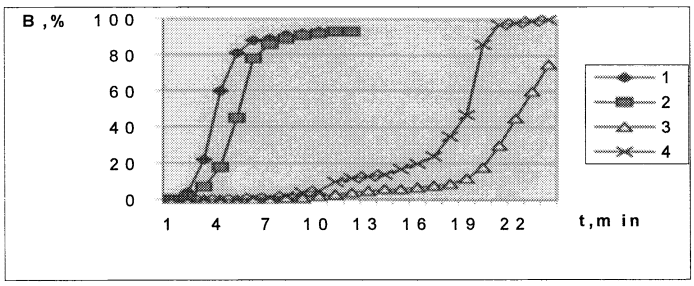


Fig. 4 Modification of the degree of photo-structurization *B* with time for PEPC layers (1,4) and CAM:OMA layers (2,3): 1,2 - exposure by white light; 3,4 - exposure by UV-light

In Figure 4 the dependency of modification of the intensity of absorption of the layers is shown during a photostructurization, correlating with the degree of *B* in time. By a criterion of evaluation of photo-sensitivity (*S*) we chose the ratio of the magnitude of incident energy to the time of saturation on the characteristic straight lines $B = f(t)$. As an outcome of frequent tests it was established that the optimum concentration of the structuruzation component CHI_3 in the PEPC and CAM:OMA PC-layers is 8-10%.

The investigation of the polymeric composition influence on structural photo-cross-linking in dependence on carbazole nucleus concentration (as CAM-1 and CAM-2) in polymers represents a great interest. There are investigated the process activation of layers photo-cross-linking with different additives (f.e. chloranyl etc.).

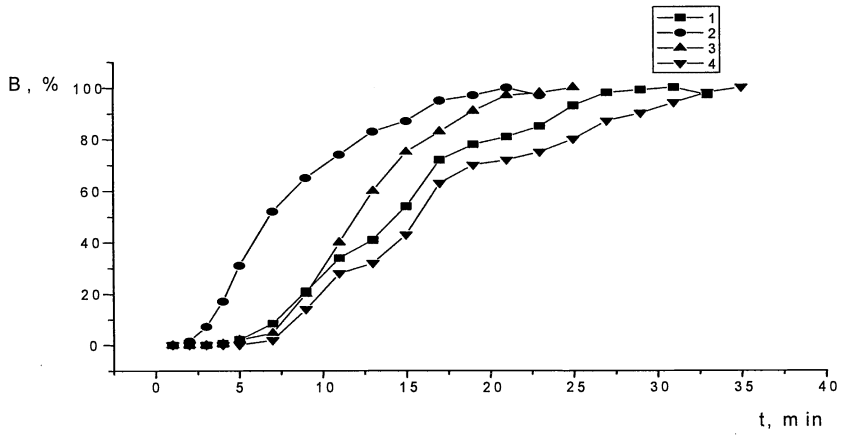


Fig.5 Dependence of photo-structurization of layers with the time of UV-irradiation:
1 – CAMC:OMA (60:40)+10%CHI_3+1%ClAn;
2 - CAMC:OMA (60:40)+10%CHI_3+2%ClAn;
3 - CAMC:OMA (60:40)+10%CHI_3+3%ClAn;
4- CAMC:OMA(60:40)+10%CHI_3 (without ClAn).

It was shown the layers photo-structuruzation acceleration in dependence on increasing of chloranyl (ClAn) concentration (1-2%) in polymeric compositions (CAM-1:CAM-2 (50:50mol%) was called as poly-CAM, CAM:OMA). The time of layers full photo- structuruzation regresses from 30-35 minutes to 18-20 minutes in the case of CAM:OMA with UV-irradiation. In the case of laser irradiation the photo- structuruzation time is about 3-5 min. The optimal concentration in photopolymer layers is about 2% as we can see from Fig.5.
With the following increasing of chloranyl concentration the photo-structuruzation process became less as we can see from Fig.6. It can be connected with carbasole nucleus isolation. Such effect we have observed in the case of OMA concentration increasing in the CAM:OMA copolymers.

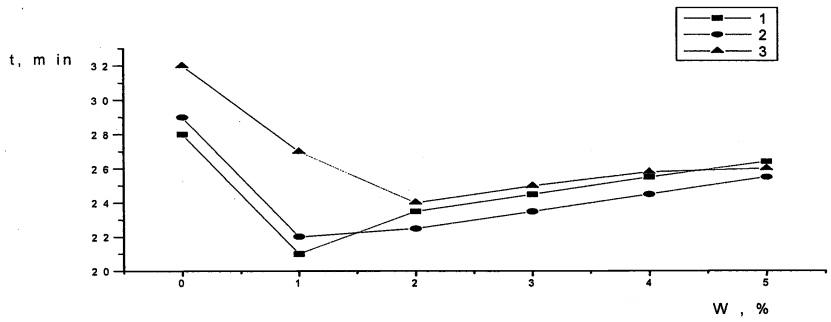


Fig.6 Dependence of the complete photo-structurization of the CAM-layers with different OMA concentration on the concentration of ClAn: 1 – poly-CAM; 2 – CAM:OMA (80:20); 3 - CAM:OMA (60:40).

The increasing of OMA plasticizer component concentration always results to the layers photo- structuruzation time increasing. The increasing of OMA molar concentration in copolymer layers from 0 to 40mol % results to the layers photo-structuruzation time increasing more than for 15 minutes.

2.2. Holography investigations

To obtain good holograms on the photopolymer layers an installation on the base of 1W CW Argon - Ion laser and 20 mW He-Cd laser was mounted. This installation allows to obtain good holographic scene with the depth of definition up to 15 sm. The laser power in single mode output was 200 mW on wavelength λ=0,49 μm for Argon-Ion laser and 15 mW on wavelength λ=0,42 μm for He-Cd laser.

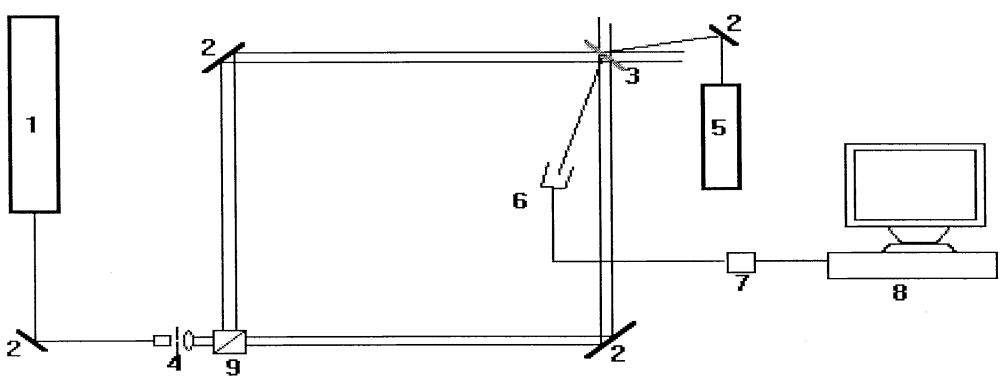


Fig.7 Optical set-up: 1 - He-Cd laser; 2 - mirrors; 3 - PC polymer layer; 4 - collimator system; 5 - He-Ne laser; 6 – photo-detector; 7 - optical power meter and transformation system; 8 - computer system; 9 - beam splitter.

Optical set-up of the experimental holographic installation is presented in Fig. 7 and consists of: 1 - He-Cd laser; 2 - mirrors; 3 - PC-polymer layer; 4 - collimator system; 5 - He-Ne laser; 6 – photo-detector; 7 - optical power meter and transformation system; 8 - computer system; 9 - beam splitter.

The experimental results on reflection hologram recording have shown that the resolution of the holographic mono-layer based on PEPC and CAM:OMA polymer layers is no less than 2000 mm^{-1}. The maximum of the diffraction efficiency of the reflection holograms is 1,5 % at the layer thickness of 5-6 μm. The following chemical treatment permits to increase the diffraction efficiency up to 20 %.

For the photoresists capable to photostructurization made from copolymers of carbazol-containig compositions with 4-10 % of cross-linking agents, the dependence of diffraction efficiency of registered diffraction gratings on both layer

thickness and the time of exposition was investigated. The registration of diffraction gratings was carried out at spatial frequencies 1500 mm^{-1}. The results of measurements are shown in a Table 1.

Tab. 1
λ=0,42 μm; W=7 mW; ν=1500 mm^{-1},
1,2,3,4 - PEPC samples; 5,6,7 - CAM:OMA samples

N_{sampl}	n, μm	$\eta_{1R} + \eta_{1'R}$, %	$\eta_{1T} + \eta_{1'T}$, %	t_{expos}, min	t_{treat}, s
1	3-4	0,63	0,81	5	30
2	3-4	1,8	8,1	10	30
3	8-9	0,78	2,6	7	30
4	8-9	2,1	18,3	15	30
5	5-6	4,5	2,0	5	30
6	5-6	20,0	10,0	7,5	30
7	5-6	7,7	1,0	10	30

It is seen from the table 1 that the diffraction efficiency of the PEPC-layers increases up to 20 % with increase of the layers thickness up to 9 microns and the exposition time up to 15 min.

For the layers capable to photostructurization made from copolymers of carbazol-containig compositions with 4-10 % of cross-linking agents and with1-2% of ClAn, the dependence of diffraction efficiency of registered diffraction gratings on the time of exposition was investigated. The registration of diffraction gratings was carried out at spatial frequencies 1500 mm^{-1}. The results of measurements are shown in a Table 2.

Tab. 2
1 – PEPC+8%CHI_3+1%HlAn ; 2, 3 – PEPC+8%CHI_3+2%HlAn ;
4, 5 – poly-CAM+8%CHI_3+2%HlAn ; 6 – CAM:OMA (80:20)+8%CHI_3+2%HlAn;
7 – CAM:OMA (60:40)+8%CHI_3+2%HlAn;
λ=0,42 μm; W=7 mW; ν=1500 mm^{-1},

$N_{sampl.}$	n, μm	$\eta_{1R} + \eta_{1'R}$, %	$\eta_{1T} + \eta_{1'T}$, %	t_{expos}, min	t_{treat}, s
1	5-6	1,5	2,7	4	10
2	5-6	1,8	3,5	4	10
3	5-6	2,2	15,3	8	10
4	5-6	5,4	3,1	4	10
5	5-6	23,0	12,0	8	10
6	5-6	19,6	10,3	8	10
7	5-6	16,3	8,2	8	30

It is seen from the table that the diffraction efficiency of the poly-CAM-layers with 2% of ClAn increases up to 23 % with the exposition time up to 8 min.

2.3. Electron Beam Information Recording

The diffraction gratings with constant period (1 μm) were recorded by electron beam in the column of a scanning electron microscope BS-300. At the accelerating voltage of 23 kV the electrons penetrated throughout the layer. The beam current determining the electron irradiation dose was varied in between 0.1 and 5.0 nA. The grating dimensions were about 400×600 square μm.

As a result of electron bombardment, planar amplitude gratings were formed due to electron-induced coloration in the layers. The diffraction efficiency (η) was measured in the first diffraction order at normal incidence of He-Ne laser beam (wavelength 0.6328 μm). The diffraction efficiency value for the planar gratings was determined as a ratio of the light intensities in the first and zero diffraction orders. The results of measurements were presented as dependencies $\eta(I)$ of

the diffraction efficiency on the recording current. The diffraction efficiency was daily measured during several days after the recording.

The planar gratings were used as a parent material for formation of relief gratings. The relief in PEPC and CAM:OMA (60:40 mol. %) layers was obtained by developing in an organic solvent CCl_4. To produce reflection gratings the samples were covered by aluminium. In this case the diffraction efficiency was measured in reflection mode as an intensity ratio of diffracted and normally incident light beams.

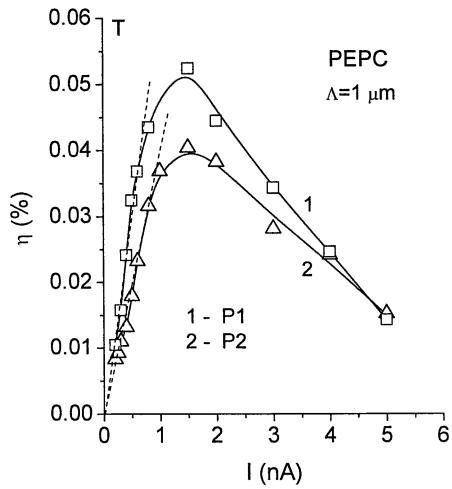


Fig. 8 The dependence of diffraction efficiency on recording current for gratings recorded in fresh (1) and stored for three months (2) PEPC layers. The data was obtained next day after recording.

In Fig. 8 the dependence of the grating diffraction efficiency on recording current is plotted using the measurements made after one day storage. As it is seen the $\eta(I)$ dependences for the two samples are similar. At the initial portion of the graph the diffraction efficiency steeply increases with the recording current and reaches a maximum in the interval from 1 to 2 nA. At $I > 2$ nA the values of η decrease at growing of the recording current. The initial portion of the $\eta(I)$ dependences can be fitted well by the straight line crossing the co-ordinate origin. After aging of the PEPC layer the diffraction efficiency was reduced (curve 2). At high recording currents the effect of aging on the recording was insignificant.

In Fig.9 the $\eta(I)$ dependencies for the gratings recorded in the layers of CAM:OMA co-polymers of the composition 80:20 mol.% (solid line) and PEPC (dashed line) are presented. An initial portion of linear growth of η with increasing of I was observed for both materials, with somewhat lower rate for PEPC. A distinguishing feature for the CAM:OMA is the increase of η with increasing of recording current at $I > 1$ nA resulting in rather high values of η. For samples with greater percentage of OMA plasticizer (up to 40%) the diffraction efficiency of gratings was much (4 to 5 times) lower (Fig. 10). At the same time the character of the $\eta(I)$ dependence remained the same (compare curves in Fig.9 and Fig. 10). In the co-polymer CAM:OMA layers the post- writing effect manifested itself in increasing of the diffraction efficiency of gratings formed at high recording currents.

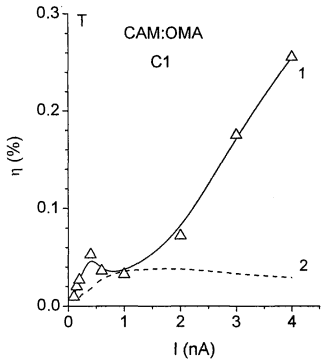


Fig. 9 Dependencies η(I) for gratings recorded in CAM:OMA layer of 80:20 mol % composition (1) and in PEPC layer (2).

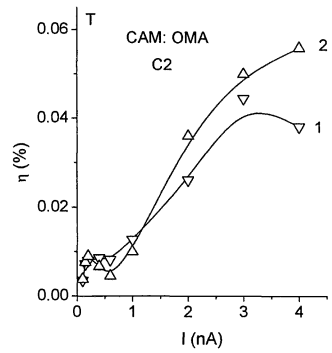


Fig. 10 Dependencies η(I) obtained one (1) and seven (2) days after recording for gratings in CAM:OMA layer of 60:40 mol.% composition

In Fig. 11 there are presented the *η(I)* dependencies for reflection relief gratings produced in PEPC (curve 1) and CAM:OMA (curve 2) layers at development time 20 s. The character of the *η(I)* dependencies was almost the same as for the planar] gratings. At low recording currents both curves showed growing portions, but at $I > 1$ nA the *η(I)* decayed in the first case and increased in the latter case. The maximal value of η for the gratings in PEPC layers was reached in the low recording current region and valued about 26 %. For gratings formed in co-polymer layers the η values greater than 30 % have been obtained in the high recording current region.

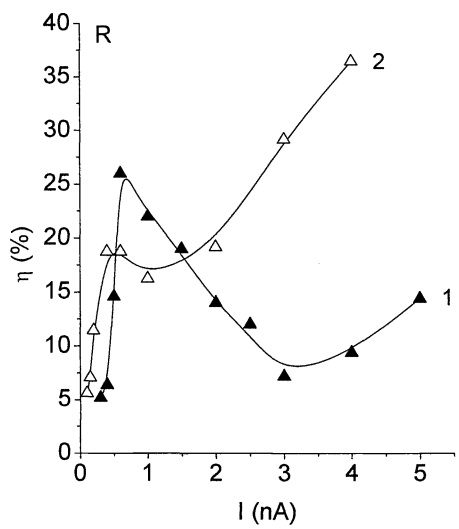


Fig. 11 Dependencies of the diffraction efficiency on recording current for reflection gratings formed in layers of PEPC (1) and 60:40 mol % CAM:OMA (2)

The η(I) dependence for the planar gratings in carbazol-containing polymer layers was sensitive both to the layer content and the sample pre-history and can to a certain extent serve as a characteristic of the material of a given class in view of application for electron-beam recording. It is because the planar grating represents the periodic structure with spatial modulation of optical characteristics of the media. It is formed as a result of processes (radiochemical reactions) in the polymer layer under action of the electron irradiation leading to the structural changes. The diffraction efficiency may serve as a measure of the induced alterations.

The most probable process induced by electron irradiation in the carbazol-containing polymer layer is a reaction of cross-linking of macromolecules [6,7] resulting in formation of three-dimensionally linked polymer. Under assumption

of the analogy with photochemical structure transformations the linking of macromolecules is provided by formation of complexes due to irradiation induced reaction between the carbazol macromolecules with CHI_3. Such process, as it has been experimentally observed, leads to coloration of the irradiated areas to a blue-green color due to structurization of polymer layers. The role of the carbazol rings in the cross-linking process is supported by greater color intensity and, as a consequence, higher diffraction efficiency of gratings in co-polymer CAM:OMA with higher CAM content.
The specific dose dependence of the diffraction efficiency in CAM:OMA co-polymers with various behavior in the regions of low and high recording currents indicate the possible difference of the recording mechanisms at low and high doses. A certain role in this difference plays the greater rigidity and thermodynamic stability of CAM:OMA macromolecules in comparison with those of PEPC.

The radiation induced linking of the polymer layer governs its behavior at chemical development of the relief as a negative resist. The recording current and development time determine the diffraction efficiency of a relief grating. The observed correlation between the $\eta(I)$ dependencies for the planar and relief gratings is conditioned by similar origin of both the electron-induced coloration and reducing of chemical dissolution rate in the considered polymer layers.

In PEPC and CAM:OMA layers was carried out the imposing of diffraction gratings generated by both electron - beam and holographic recording. Group of micro-gratings formed by electronic beam was registered at first at a variation of a recording current. Then the holographic grating, covering this group of electron micro-gratings, was registered perpendicular to the direction of a lines of electron micro-gratings. Before recording the PEPC layers were subjected to a preliminary irradiation by white light with use of standard luminescent lamp. The period of a holographic grating is 1 micron, and electron micro-gratings - 1 micron and 2 microns. Then the relief imposed gratings with the help of chemical processing in a solution of four-chloride carbon were generated.

The diffraction efficiency in the first order of diffraction of relief gratings was measured on transmission and reflection. The influence of imposing of electron beam recording on diffraction properties of a holographic grating was estimated under the relation of meanings of diffraction efficiency of imposed (η_s) and single (η) areas between micro-gratings: $k=\eta_s/\eta$.

Both for transmission (Fig. 12) and for reflecting (Fig. 13) gratings the value of k monotonously decreased by growth of a recording current, reaching the value less then 0.2 at I > 0,4 nA at imposition with electron micro-gratings with the period 1 micron (curves 1). The increasing of the period of electron micro-gratings weakened influence of imposing on diffraction efficiency of a holographic grating (curves 2).

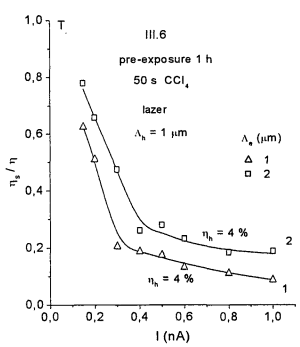


Fig. 12 Influence of imposing of electron beam recording on diffraction efficiency of transmission holographic gratings with the 1 μm (curve 1) and 2 μm (curve 2) periods. Duration of a preliminary exposition is 1 hour.

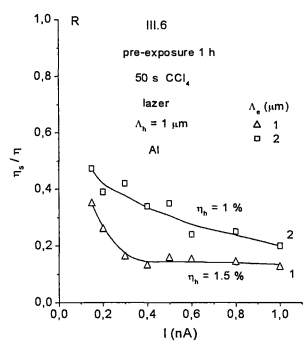


Fig.13 Influence of imposing of electron beam recording on diffraction efficiency of reflective holographic gratings. The period of micro- gratings is 1 μm (curve 1) and 2 μm (curve 2).

While with growth of a recording current on an initial site was observed of sharp reduction of value k, the diffraction efficiency of electron micro-gratings in transmission on the contrary appreciablly grew (Fig. 14), reaching approximately 12 % at I=0.4 nA for the period 1 μm (curve 1). For reflecting micro-gratings with the period 1 μm the increasing of the

diffraction efficiency with growth of a recording current was observed only at I > 0.4 nA (Fig.15, curve 1). Both for reflection and for transmission gratings were received more lower value of diffraction efficiency (curve 2 in a Fig. 14 and Fig. 15).

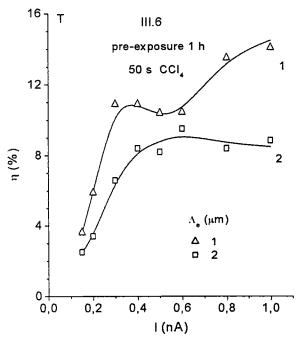


Fig.14 Dependences of the diffraction efficiency on recording current for the imposed transmission relief micro-gratings with the period of 1 μm (curve 1) and 2 μm (curve 2).

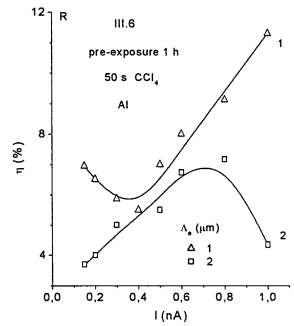


Fig.15 Dependences of the diffraction efficiency on recording current for the imposed reflection relief micro-gratings with a period of 1 μm (curve 1) and 2 μm (curve 2).

For reflecting and transmission micro-gratings the greatest value of the diffraction efficiency were received at I=0.8-1 nA and approximately coincided. Their values were 12-14 % and 7-8 % for gratings with the period 1 μm and 2 μm accordingly. Diffraction efficiency of a single holographic transmission grating was 4 %.

The increase of time of a preliminary exposition (Fig. 16) did not result in change of a course of dependences k (I). From comparison of a Fig. 12 and the Fig. 16 is visible, that the transition of a site of strong reduction of values k to smooth reduction of values k occurs approximately at the same value of a recording current. However at the greater time of a preliminary exposition the smaller values of diffraction efficiency of the imposed gratings are received. From a Fig. 16 it is visible, that the imposing caused approximately identical easing of diffraction efficiency of a holographic grating on transmission and reflection (curves 1 and 2).

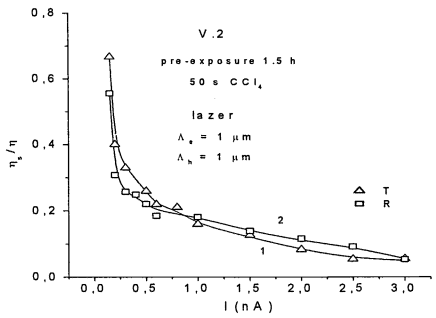


Fig. 16 Influence of imposing of electron beam recording on diffraction efficiency of holographic gratings in transmission (curve 1) and reflection (curve 2). Duration of a preliminary exposition is 1,5 hours.

The basic reason of mutual influence of the imposed relief gratings is the distortion of a profile of lines in units of their crossing. Confirmation of this is the smaller easing of diffraction efficiency of a holographic grating at imposing with e-micro-gratings of the greater period (2 μm). Varying a beam current it is possible to change a parity of diffraction efficiency of the imposed gratings received by methods of holographic and electron beam recording.

In the carried out experiment from positions of visualization of both types of gratings optimum it is possible to consider a rather low current of recording of micro-gratings near 0,3 nA and with period 2 μm. However considered experiment it

only episode as more complex task. First it is possible to vary as well a doze of a laser irradiation at holographic recording. Secondly essential role is played by the order of processing of the carrier of the information. The received results show that the holographic recording is at a loss on sites of e-micro-gratings. Therefore for the best visualization of the holographic imposed gratings it is preferable to registered them first of all. An additional homogeneous irradiation of resist is known procedure, which can render a positive role for recording of the information. At use of PEPC layers it has allowed essentially to improve quality of a surface of samples after chemical processing. It is necessary, however, to find out when better to carry out (if it the additional irradiation is necessary): up to or after recording of gratings.

To obtain a good holograms on the photoresist polymer layers an installation on the base of 1W CW Argon - Ion laser and 20 mW He-Cd laser was mounted. This installation allows to obtain good holographic scene with the depth of definition up to 15 sm. The laser power in single mode output was 200 mW on wavelength λ=0,49 μm for Argon-Ion laser and 15 mW on λ=0,42 μm for He-Cd laser.

The regimes of holographic recording were correlated with the e-beam writing to develop the procedure of conformation and superposition of the patterns formed by both techniques. A sample view of superimposed patterns on a PEPC layer is shown on Photo 1 and on a CAM:OMA layer is shown on Photo 2. The holographic diffraction grating of 1 μm period is oriented vertically, while the diffraction grating formed by e-beam recording is oriented horizontally, with 1μm period in the upper part and with 2 μm period at the bottom of the image. As it is seen, the relief depth is the same for both gratings. The e-beam record was made first, next the holographic grating was recorded. The relief superimposed gratings were prepared by 50 s etching.

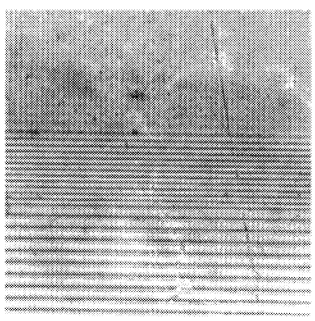


Photo 1

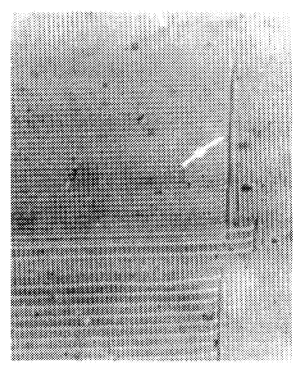


Photo 2

In the cycle of superimposed diffraction structure technology the regimes and conditions of holographic pattern processing were taken as a basis. Versatility of e-beam recording conditions allowed attaining proper choosing of the recording current for microgratings. Samples with superimposed e-written and holographic structures were developed in CCL_4 for 50 s. For this etching time the 25% of η for microgratings and 15% for holographic gratings were obtained. Non-trivial results were obtained for the effect of recording current and chemical development time on the η of the relief gratings in the samples. It was found that the recording current is determined by the development time chosen for the treatment of the holographic pattern.

3. CONCLUSION

1. The photopolymer layers capable to photostructurization from carbazol-containing compositions with 4-10 % of the cross-linking agents are offered. The contents of carbazol-containing copolymers have an influence at all photo-structurization process.

2. The process of a photo – structurisation of photorezists layers on the base of CAM:OMA copolymers containing threeiodinomethan (CHI_3) as the cross-linking agent, proceeds on the radical mechanism due to formation of complexes with carry of a charge between carbazolyl nucleuses and CHI_3.

3. The increasing of carbazole component in copolymers reduces the full photo-structurization time more than for 25%. The optimum concentration of structurization component CHI_3 in PEPC and CAM:OMA layers is 8-10%. The activator presence (chloranil – 1-2%) accelerates the photo-structurization process.

4. The diffraction efficiency of the registered holographic diffraction gratings on the base of PEPC and CAM:OMA layers with 4-10 % of cross-linking agents (CHI_3) and with1-2% of ClAn reaches 15% on reflection for PEPC samples and 23 % on transmission for CAM:OMA samples.

5. With the aid of electron-beam recording, the diffraction gratings are formed in fresh and stored polymer layers of PEPC and CAM:OMA.

6. The dose dependence of the diffraction efficiency of planar gratings is shown to be the material characteristic sensitive to variation of the molecular composition and some factors, such as storage and additional light exposure. The dose dependencies of PEPC and CAM:OMA are significantly different, with correlation only in the range of low recording currents, indicating the differences in the molecular structure.

7. The structure relaxation is clearly revealed in the dose dependence as post-writing changes in the diffraction efficiency of the planar gratings.

8. Due to correlation existing between the diffraction properties of gratings in planar and relief modifications the examination of dose dependencies of the planar (starting) gratings is helpful for optimization of the processing conditions of the relief (ultimate) gratings.

9. For reflection gratings formed in co-polymer layers the diffraction efficiency η values greater than 30 % have been obtained in the high recording current region.

10. The imposing of diffraction gratings generated by both electron - beam and holographic recording was obtain in PEPC and CAM:OMA layers.

11. For reflecting and transmission imposing electron-micro-gratings the greatest value of the diffraction efficiency were received at I=0.8-1 nA and approximately coincided. Their values were 12-14 % and 7-8 % for gratings with the period 1 μm and 2 μm accordingly. Diffraction efficiency of a single holographic transmission grating was 4 %.

12. The basic reason of mutual influence of the imposed relief gratings is the distortion of a profile of lines in units of their crossing. Varying a beam current it is possible to change a parity of diffraction efficiency of the imposed gratings received by methods of holographic and electron beam recording.
13. In the cycle of superimposed diffraction structure technology the regimes and conditions of holographic pattern processing were taken as a basis. For etching time for 50 s in CCL_4 the 25% of diffraction efficiency η for electron-beam microgratings and 15% for holographic gratings were obtained.

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