

Selection of Photopolymer for Microrelief Formation by Two-Stage Cold Stamping Method

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Microrelief formation by embossing the master matrix (with the period of 3.5 μm and depth of 1 μm) on photopolymer coatings and its application for large areas embossing technology has been investigated. The method embodies the direct contact microrelief cold stamping and two stages of UV curing processes. The purpose of the initial (stage I) UV curing is to obtain the coating of such hardness that periodical structure with the precise geometry could be embossed. The aim of the final (stage II) UV curing is to stabilize the mechanical properties of photopolymer coating and to fix the geometry of embossed microrelief. The investigations were performed using three types of acrylate based commercial photopolymers. The wetting and mechanical properties, surface topography of photopolymer coatings, diffraction efficiency and geometry of embossed periodical structures have been investigated in order to find the effective durations for both UV curing stages and define parameters of replication process. The quality of replicated microrelief was found to be in great dependence on the photopolymer type, its wetting properties, hardness and surface structure of the coatings and parameters of replication process.

Keywords: cold stamping, photopolymer, UV curing, periodical structure, mechanical properties, optical properties.

1. INTRODUCTION

Diffraction gratings are widely used in different areas of applications. Among of all the material used, polymer gratings are of great interest for their low cost and easy fabrication. Various methods can be used to produce microrelief replications in polymeric materials (hot or cold stamping, moulding, lithography, contact printing, etc.) [1]. An application of such gratings includes very sensitive humidity and temperature sensors, fine measurement systems, 3D image display systems [2, 3], flexographic plates [4], microoptical structures, high density data storage, other information processing applications and other devices used in electronics, optoelectronics, microelectromechanical systems [5–7].

A microrelief formation in photopolymers with subsequent UV curing became a well-accepted technology with widespread use. The major reasons for this development are the considerable economic and ecological advantages over traditional polymerization methods and the unique features: solvent-free compositions, high curing speed, low temperature processing, production of a highly resistant cross-linked polymer, high quality of the products obtained and low energy consumption due to the operation at ambient temperature, which make UV curing process attractive for industrial applications [8].

Photopolymers due to their favourable advantages, such as simple preparation, self developing capability, response for visible light, high diffraction efficiency, energetic sensitivity, spatial frequency, signal-to-noise and dry processing are the most promising with a view to the formation of diffraction gratings on their layers [9, 10].

Acrylic photopolymers perhaps are the most popular radically polymerizing commercially available systems that show good response for optical applications [11]. The curing time of these photopolymers is dependent upon the dose intensity and light wavelength of the UV light. Initiation of polymerization requires precise matching of product and UV light source [3].

A technology of periodical structure replication when the relief geometry of microstructured master matrix is transferred to the surface of photopolymer layer by direct contact cold stamping is used.

The method of the microrelief formation in large area of photopolymer layer was proposed [12]. In this case photopolymerization of the layer is performed in two-stages: first UV-precuring is carried out before microrelief embossing and second – after embossing process. Thus, the method composes of 4 steps: photopolymer coating, its UV-precuring (stage I), microrelief embossing and the final UV curing (stage II).

A main advantage of the method over the conventional thermal curing is a reduced influence of the temperature on geometrical parameters of formed microrelief [13] as well as a higher quality of the replication [5, 6]. Another advantage is the possibility to use the method for the continuous mass replication process. The aim of this study was to select photopolymer for relief replication process by two stages UV curing and investigate the influence of photopolymer nature and parameters of UV-curing stages on the quality of replications.

2. EXPERIMENTAL

2.1. Materials

Three types of acrylic commercial photopolymers (PHP) were used for the investigations:

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PHP I – polyfunctional monomer trimethylolpropane ethoxylate (14/3 EO/OH) triacrylate (TMPETA) with initiator 2,2-dimethoxy-2-phenylacetophenone (DMPA) (*ALDRICH*, Germany) which chemical structures (Fig. 1) and main properties (Table 1) are presented.

Also, two commercially available photopolymer compositions EXC90300 (PHP II) and EXC90307 (PHP III) from *ARETS GRAPHICS*, Belgium, were used for these purposes. Their viscosity is presented in Table 2.

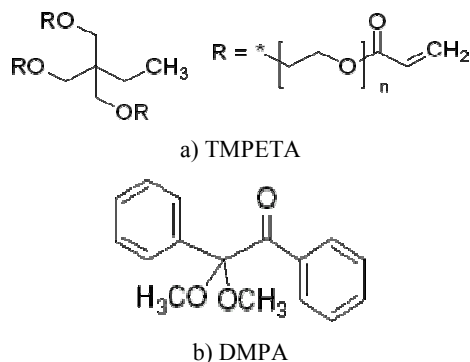


Fig. 1. Chemical structures of PHP I components

Table 1. The main properties of TMPETA and DMPA

Property	Component	
	TMPETA	DMPA
Dynamic viscosity η , mPa·s (at 25 °C)	40–80	–
Refraction index n_D^{20}	1.471	–
Boiling point T , °C	157	–
Flash Point T , °C	113	–
Density ρ , g/ml (at 25 °C)	1.12	–
Molecular weight M_n , g/mol	912	256.3
Melting point T , °C	–	64–67
Solubility in triacrylate monomer g/100 g solution (at 20 °C)	–	25

Table 2. Viscosity of investigated photopolymers

Photopolymers	Kinematic viscosity ν (at 21 °C), SUS
EXC90300	45–55
EXC90307	35–45

2.2. Photopolymer coating and periodical structures formation

Polyethylene terephthalate (PET) film with thickness of 100 μm was used as a substrate for photopolymer coating. Before photopolymer coating a surface of PET film was treated using oxygen plasma (radio frequency power of $RF = 13.56$ MHz, pressure of $p = 0.3$ W/cm² and treatment duration of $\tau = 30$ s) [14]. Deposition of a photopolymer layer was performed by anilox rolls with the surface engravings which volume was used to transport a precisely determined quantity of photopolymer in order to form a layer of 5 μm in thickness. The schematic view of photopolymer layer and microrelief formation process is presented in Fig. 2.

After photopolymer layer coating UV curing was performed for initial layer hardening. After that cold

stamping was implemented and periodical structure was formed on the surface of photopolymer coating. Applying II stage UV curing the coating with constant mechanical properties was obtained and embossed microrelief with steady geometry was attained.

The durations of I stage UV curing were 5 s–60 s. The relief features were transferred to the precured photopolymer coating by mechanical embossing with microstructured nickel matrix with the period of 3.5 μm and depth of 1 μm [15–17]. The embossing was performed at pressure of $p = (0.5 \pm 0.02)$ MPa and duration of $\tau = 3$ s.

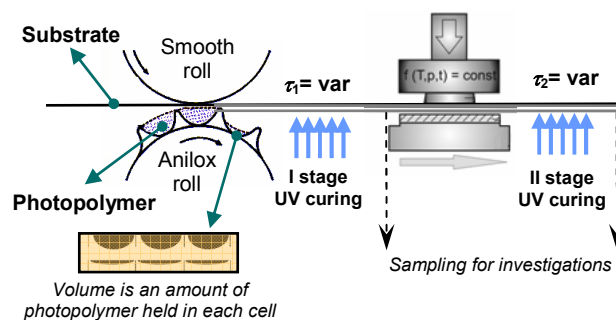


Fig. 2. Schematic view of photopolymer layer formation using two stages of UV curing

The samples for investigations were taken after coating formation and UV-precuring and after periodical structure replication and final UV curing stage.

2.3. Photopolymer layer properties investigation methods

As periodical structure was embossed using nickel master matrix, it was important to investigate the photopolymers ability to wet a nickel layer. A drop of each photopolymer of controlled volume of 10 μl was placed on the smooth Ni substrate and Ni matrix with periodical structure. The measurements of the contact angles were conducted at room temperature (22 °C) after 10 s of the drop placement. The measurements were performed in five different points of each substrate and the mean value was calculated.

In order to investigate the influence of UV curing duration on the mechanical properties of the photopolymer layer a constant (when normal force $P_n = 100$ mN and scratch speed 10 mm/min) and progressive load scratch tests were performed. The scratch resistance of the coating was measured under controlled conditions using an original construction KTU FEI Micro-Scratch Tester.

During progressive load micro-scratch testing diamond indenter moved linearly along the sample. A constant scratch speed of 10 mm/min and a speed of load increment of 100 mN/min were used. As the sample was moved laterally, the normal force was increased up to 120 mN and a scratch of 10 mm length was formed. During the scratch test normal and tangential forces were recorded as a measure of the “scratch resistance” of the coating.

A shape of the scratch was registered by scanning electron microscopy (SEM). SEM images were obtained by emission scanning electron microscope JEOL JSM-IC25S.

2.4. Investigation of surface morphology and microrelief

The morphology of photopolymer layer surface and replicated periodical structure were investigated by atomic force microscope (AFM) NANOTOP-206 with a silicon cantilever operating in contact mode (cantilever force constant 3 N/m).

An image processing and analysis of the AFM data were performed using standard software “Surface View” version 2.0 [18, 19]. Root-mean-square roughness R_q and arithmetic mean roughness R_a were used due to the most universal reported measurement of surface roughness and the ease of determination and calculation. The image roughness calculation obtained from the inbuilt software is based on finding a median surface level for the image and then evaluating the standard deviation within $N \times N$ range. For this three-dimensional $N \times N$ image of data heights $z(x, y)$ discrete approximations to roughness R_q and R_a are given by:

$$R_q = \sqrt{\frac{1}{N^2} \sum_{i=1}^N \sum_{j=1}^N (z_{ij} - z_{av})^2}, \quad (1)$$

$$R_a = \frac{1}{N} \sum_{i=1}^N \sum_{j=1}^N |z_{ij} - z_{cp}|, \quad (2)$$

where i and j are pixel locations on the AFM image; z_{ij} is height value at i and j locations; z_{av} is a mean value of height within the given area; z_{cp} is a value of height from the center plane; N is the number of data points in the image.

The quality and variation of replicated periodical structure geometry was evaluated by the non-destructive optical laser control method. Diffraction efficiency of embossed microrelief was measured experimentally by He-Ne laser with power of 30 mW and wavelength of 632.8 nm. The measurements were performed in ten different points of each replication and the mean value was calculated. The measured relative diffraction efficiency is defined as the ratio of the particular wavelength intensity of the light reflected by the grating in the given diffraction order $I(\theta)$ and the intensity of the coating material reflectance $I(0)$ [8, 20].

3. RESULTS AND DISCUSSION

The wetting of a nickel layer by photopolymer is very important for the replication process. It may be proposed that in the case of good surface wetting by photopolymer the geometry of replicated periodical structure is obtained defective and varied from that of master matrix. The results of the internal contact angle measurements of the nickel surface wetting before and after the replication of the photopolymeric grating are presented in Fig. 3.

It can be seen that the Ni matrix with periodic microrelief shows higher values of contact angle and indicates the decrease of wettability of photopolymers in comparison with smooth Ni layer. It is twice lower for PHP I and PHP II on smooth Ni surface. While for PHP III the influence of surface roughness on wettability is negligible.

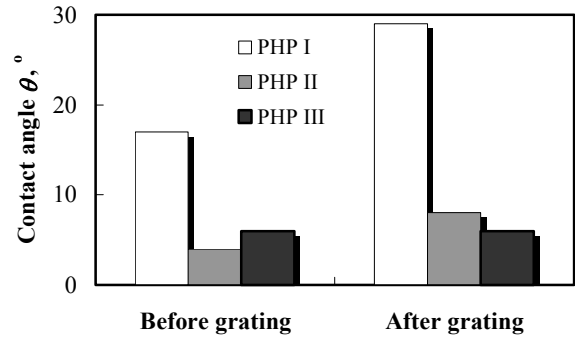


Fig. 3. The variation of contact angle vs photopolymer type and Ni surface roughness

As 3–5 times higher values of contact angle are characteristic to PHP I, the photopolymer distinguishes worst wetting properties with Ni surface that indicates on the lower adhesion interaction between the photopolymer and Ni layer, compared to those of PHP II and PHP III.

As a hardness of the coatings has critical influence on microrelief embossing process it was important to determine mechanical properties changes depending on UV precuring duration. Due to those scratch tests at constant load of 100 mN was performed. Typical SEM micrographs of obtained scratch groove views are presented in Fig. 4.

Thus, after 20 s of UV curing the layer exhibits minimal susceptibility to the mechanically induced surface damage (e. g. high scratch resistance). The decrease of the scratch width with the increase of UV curing duration indicates on the increasing hardness of photopolymer layer and decrease of possibility to emboss a periodical structure.

In order to determine the effective UV precuring durations for each of photopolymer compositions the character of coatings mechanical properties change on the dependence on UV precuring duration were investigated. The variation of shear force during scratch test is presented in Fig. 5.

For all compositions the increase of shear force with increasing UV curing duration was found. The shear resistance of the coatings considerably increases up to 5 mN–9 mN even for short curing duration of 10 s. When the duration increases up to 50 s the shear force in the range of 15 mN–20 mN was obtained. The largest increase was observed for PHP I. With the further increase of curing duration (up to 60 s) there are no more changes observed for all photopolymer coatings as a result of the formation of tight polymer network and stabilization of mechanical properties of the coatings.

In the cases of all investigated photopolymers an increase in shear force is observed up to 50 s of curing, but the characters of obtained curves are distinct. The shear force plateau in the range of 10 s–20 s of UV curing for PHP II and of 20 s–30 s for PHP III of curing duration was observed. Finally the shear force increases steeply again and reaches 17 mN and 16 mN at 50 s of curing duration. And after that a plateau of the force is observed again.

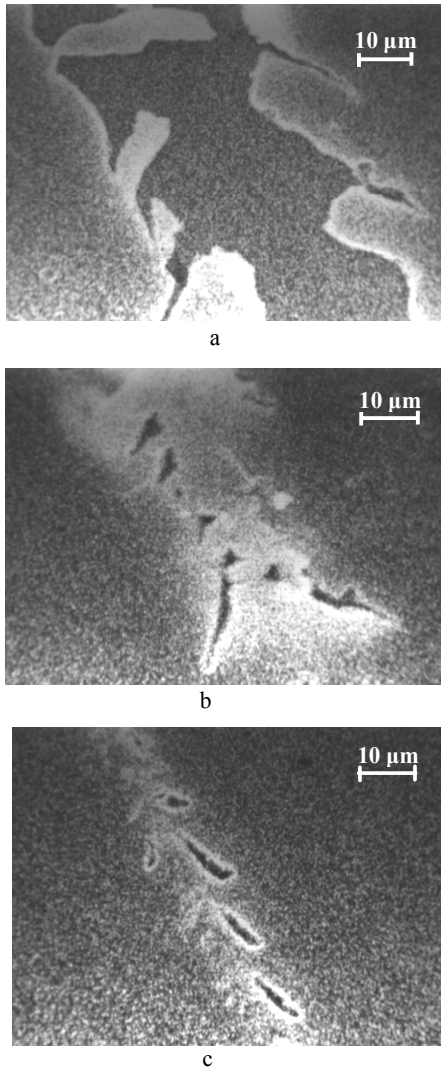


Fig. 4. SEM images of scratch groove ($P_n = 100$ mN) for PHP I coating cured at different durations: a – 5 s; b – 10 s; c – 20 s

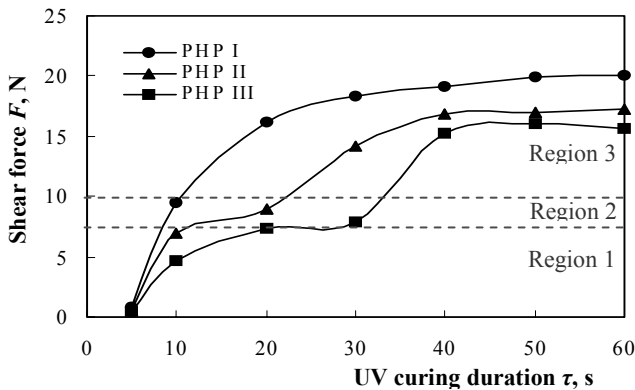


Fig. 5. The scratch resistance of polymeric layers vs UV curing duration and photopolymer type

Thus, the UV curing directly affects the stability and mechanical behaviour of photopolymer coatings despite the photopolymer type [21, 22]. The fastest response of shear force and the highest scratch resistance is characteristic to PHP I coating.

As the purpose of precuring stage is to obtain a coating with plastic properties suitable for embossing the microrelief with high quality it was important to

investigate the variation of photopolymer behaviour in dependence of UV curing. As it can be seen from Fig. 5, viscoelastic, plastic and brittle properties are characteristic for all investigated photopolymer coatings. Due to the viscoelasticity of photopolymer layers Region 1 (when the shear force is in the range of 0 mN – 7.5 mN) reflects poor mechanical properties of the coatings and bad conditions for the embossing of periodical structures. In region 2, when the shear force is in the range of 7.5 mN – 10 mN, photopolymer coatings show plastic behaviour. Thus, this region shows the best conditions for the embossing periodical structures with required geometrical parameters. And UV precuring durations in the region for the PHP I, PHP II and PHP III were determined to be 10 s, 20 s and 30 s, respectively. Thus, it may be assumed that the in the region 2 the suitable conditions for periodical structure replication on the layers of photopolymers is obtained.

The obtained results show that in the region 3, when shear force is higher than 10 mN, the photopolymer layers become brittle, have low deformability and crack during embossing process. The microrelief formation on such layers becomes impossible.

As the stabilization of shear force was observed at 50 s it was selected as the value of UV curing duration necessary to obtain embossed periodical structures with fixed geometry. This duration can be assumed as overall curing duration of the replication process that embody both initial and final stages of UV curing durations. It is already known that effective UV-precuring durations are 10 s for PHP I, 20 s for PHP II and 30 s for PHP III [23]. Thus, the final effective UV curing duration for the photopolymers will be 40 s, 30 s and 20 s, respectively.

The investigations by AFM of photopolymer coatings surface topography were performed (Fig. 6), because coated surface structure has influence on geometry of replicated microrelief. The coating morphologies for investigated photopolymers display quite different images. As can be seen from Fig. 6, a, the layer of PHP I is smooth and even, while for the other two photopolymer coatings irregularities and higher roughness were observed (Fig. 6, b, c). The differences of the coatings morphologies can be referred not only to the differences in the supermolecular structure but on the photopolymer wetting of matrix properties also.

Statistical parameters of the obtained surface roughness are presented in Table 3.

Table 3. Statistical parameters R_a and R_q of photopolymer layers vs photopolymer type

Photopolymer type	Coating	
	R_a , nm	R_q , nm
PHP I	3.4	4.2
PHP II	9.8	15.2
PHP III	8.0	10.3

Generally, R_a and R_q will be similar, if there are no large deviations from the mean surface level. A negligible difference between R_a and R_q ($\Delta = 0.8$ nm) for the PHP I suggest that obtained image basically is smooth.

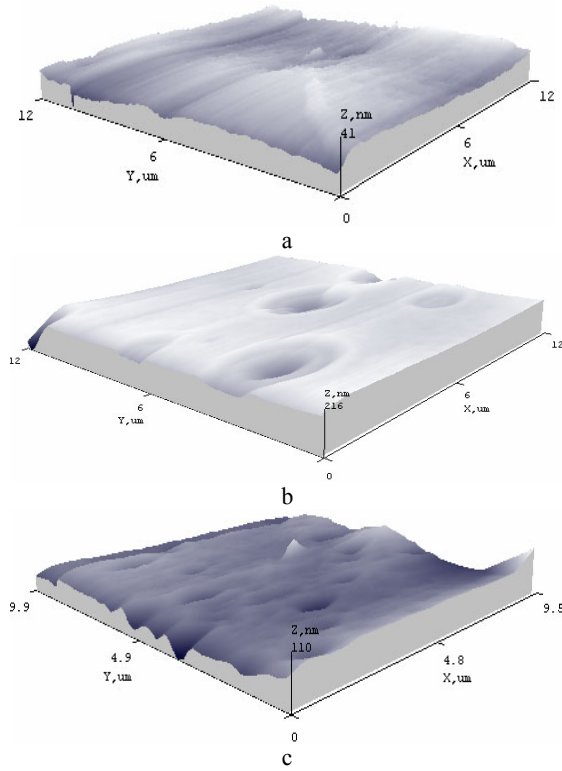


Fig. 6. AFM images and profiles of photopolymer coatings surface structure vs photopolymer type: a – PHP I; b – PHP II; c – PHP III

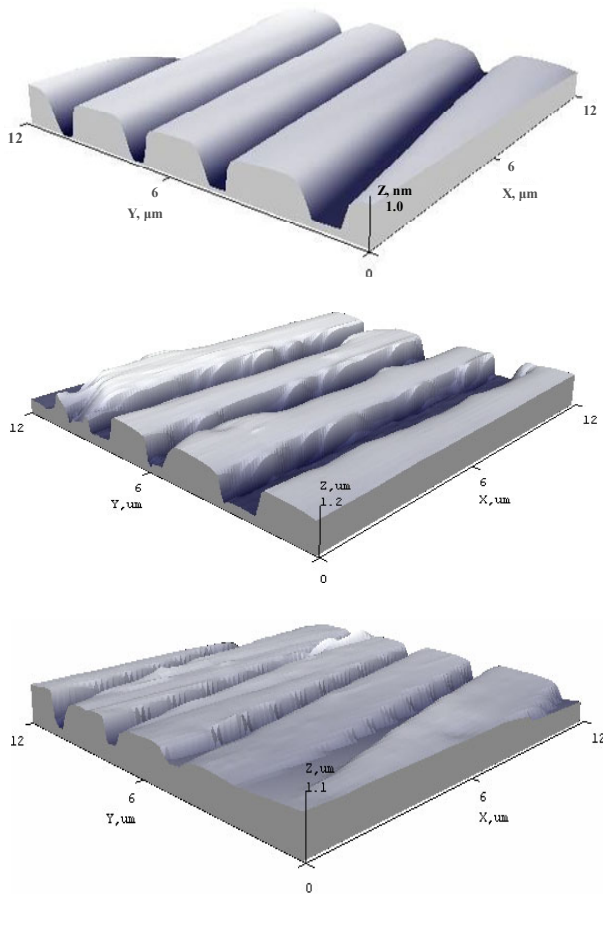


Fig. 7. AFM images and profiles of embossed periodical structures vs photopolymer type and effective initial UV curing duration τ , s: a – PHP I (10 s); b – PHP II (20 s); c – PHP III (30 s)

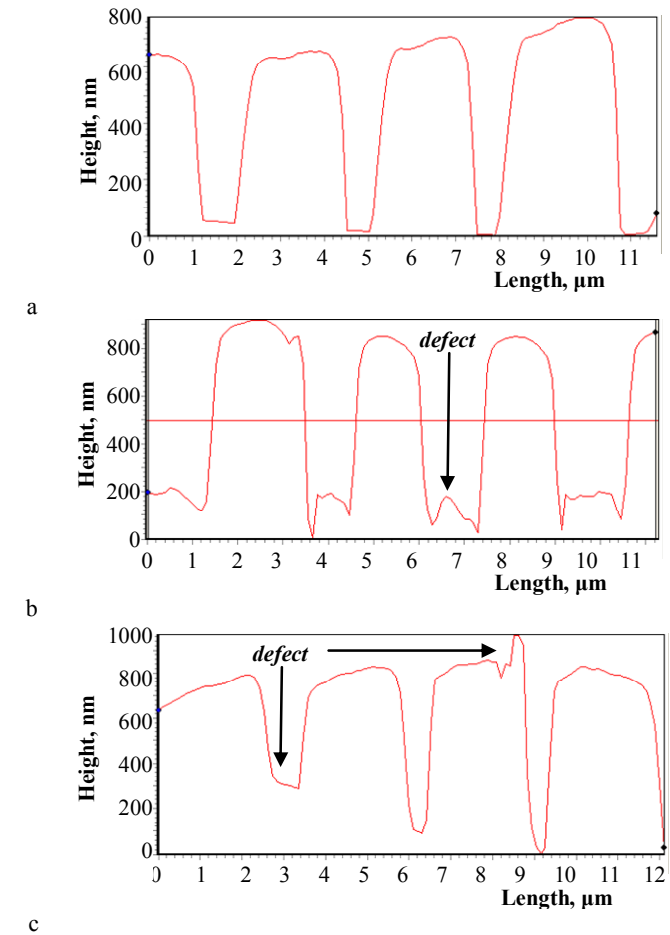
In the case of PHP II and PHP III the parameters R_a and R_q have higher differences ($\Delta = 5.4$ nm for the PHP II and $\Delta = 2.3$ nm for the PHP III) that show the presence of larger irregularities in formed surfaces.

The typical surface topography and geometry of embossed periodical structures in photopolymer coatings cured with effective initial UV curing durations are presented in Fig. 7.

As the geometry of replicated periodical structure depends on a roughness of the coatings, the replication with the most precise geometrical parameters was obtained on the smooth and free from the cavities surface of PHP I coating (Fig. 7, a). In the cases of PHP II and PHP III periodical structures were embossed on uneven layers and the geometry of replicated microrelief was obtained varied from that of the main matrix and defective (Fig. 7, b, c).

The phenomenon can be attributed to higher hydrophilicity of these two photopolymers (Fig. 3). It results in increased adhesion with the matrix and cohesive failure of the photopolymer with embossed microstructure.

One of the main quality parameters of replicated periodical structure is the diffraction efficiency. The optical properties of photopolymers and their layers, the geometry of periodical structure (depth, period, deviation angle), the wave length and UV curing duration influence on the changes of the light intensity distribution of the diffraction pattern and determine a quality of an embossed periodical structure [24].



The influence of photopolymer type on the diffraction efficiency of replicated structures is presented in Fig. 8. The highest efficiencies are obtained for PHP II and PHP III in “0” order and the values are 2 times and 2.5 times higher than that of PHP I, respectively. For “-1” and “1” maximums a significant decrease of efficiency can be observed – 63 % and 47 % for PHP II and 71 % and 59 % for PHP III, respectively. When in the case of PHP I for “-1” and “1” maximums diffraction efficiency increases at 10 % and 30 %, respectively. A significant decrease of efficiency at “-2” and “2” maximums can be observed for all investigated photopolymers (60 % and 40 % for PHP I, 87 % and 74 % for PHP II, 99 % and 83 % for PHP III, respectively). Especially it is an evident in the case of “-3” and “3” diffraction orders.

Thus, the efficiencies of PHP II and PHP III are highest in “0” order, when in the case of PHP I the highest values are obtained at “-1” and “1” maximums. It has significant importance for the practical application when periodical structure is used as dispersive element because “0” maximum has no influence for the dispersion.

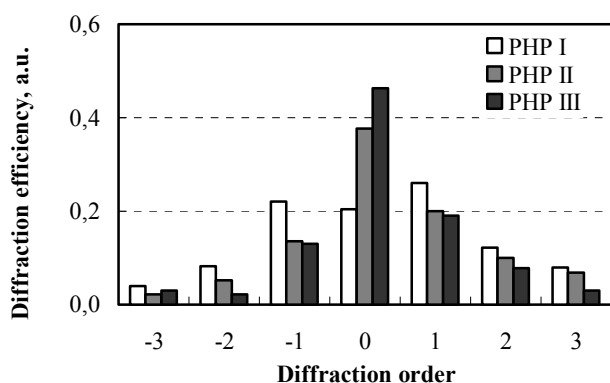


Fig. 8. Relative diffraction efficiency of replicated periodical structures vs photopolymer type

The defectiveness and roughness of PHP II and PHP III coatings may result in lower diffraction efficiency of replicas and impede the formation of structures with good optical properties.

4. CONCLUSIONS

The quality of embossed microrelief has substantial influence on photopolymer type, its ability to wet surface of stamping matrix, hardness and surface structure of the coatings and replication parameters.

AFM investigation confirms that during the replication process both depth and period of the structures is decreased for the photopolymers that distinguish good wetting properties and irregular surface structure of the coatings.

Both the geometry and diffraction efficiency of embossed periodical structures depend upon photopolymer type and determine the quality of obtained microrelief. Defectiveness and roughness of PHP II and PHP III coatings cause low diffraction efficiency and dispersion of replicas and impede the formation of microrelief with high quality.

UV curing duration has significant influence on mechanical properties of the coatings. With the increasing curing duration the increase of photopolymer coating

scratch force needed to scratch the surface is observed. Thus, the mechanical properties are improved.

The results of the study revealed that PHP I is the most effective photopolymer for replication of microrelief on its coating when periodical structures with the highest quality are embossed using defined parameters of replication process.

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