

Quality Analysis of Periodical Microstructures, Created By Using High Frequency Vibration Excitation

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Published: 1 June 2014

Abstract: Periodical microstructure, which is being developed in this experiment is the main part of the sensor, whose purpose is biosensing of processes, such as analysis of concentration of micro particles in biological environment. The main part of sensor is diffraction grating, produced by the method of hot imprint. Therefore paper deals with the methodology for analysis and comparison of the quality of microstructures, created with and without high frequency excitation. Three types of measurements are performed: measurement of diffraction efficiency, optical microscopy and atomic force microscopy, in order to analyze the quality of replica. New generation vibroactive pad, whose fundament is stack type piezoactuator, is used to generate high frequency longitudinal vibrations. Thus preheated until glass-liquid transition state polymer, is forced to flow and the quality of microstructure is being enhanced. Both microstructures (with and without vibration excitation) are created on the surface of polycarbonate, using the same temperature, pressure and time modes. During the experiment periodic lamellar microstructure, whose period is 4 μm is being analyzed. Analysis of diffraction measurements was performed by using laser and photodiode BPW-34. The relative diffraction efficiency of second maxima of nickel mold is 10 %, of microstructure impressed, by using ultrasonic vibration diffraction efficiency is 9.27 % of microstructure, created without vibration excitation is equal to 4.2 %. Optical microscopy is employed in order to obtain images of magnified surface view, thus allowing detect defects like bubbles of residual gas and distortions, while atomic force microscopy is performed in order to find out surface parameters like period, depth, surface roughness and obtain profile view of created microstructure.

This research was funded by a grant (No. MIP-026/2014) from the Research Council of Lithuania.

Keywords: periodical microstructure; Atomic force microscopy; diffraction efficiency; vibroactive pad; high frequency excitation; hot imprint.

1. Introduction

Periodical microstructure, which is being developed in this experiment is the main part of the sensor, whose purpose is biosensing of processes, such as analysis of concentration of micro particles in biological environment. Hot imprint is the process, used in order to reproduce microstructures, because of several advantages over other replication techniques, this process is chosen as the most suitable [1]. It is not possible to create high quality microstructure, by only knowing basic parameters like: preheating temperature, pressure and time, because some other factors should be taken into account. One of these factors is technology of high frequency excitation during the process, which enables to enhance surface quality, i.e. decreases surface roughness, allows avoid distortions and other possible defects. Novel vibroactive pad, whose fundament is stack type piezoactuator, generating longitudinal vibrations is employed during the process of replication [2]. In pursuance to employ all the potential of high frequency phenomenon, operating parameters (frequency of excitation and voltage), under which pad reaches first resonant mode were found. Under these regimes higher amplitudes of vibrations are obtained, this forces preheated liquid polymer to flow, thereby filling empty gaps between master mold and microstructure [3-8].

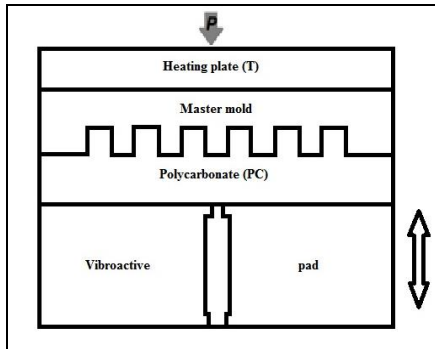
The purpose of this paper is to determine how the quality parameters change, when newly designed vibroactive pad is used as supplementary measure. When speaking about the specimens, investigated, they are as following: nickel matrix of master mold, which was used in order to imprint microstructure; microstructure, created on the surface of polycarbonate without the usage of high frequency excitation and microstructure, created with high frequency excitation. Ultrasonic excitation is the only one qualitative variable during the process of production. Other process parameters: temperature (148 °C); frequency of excitation (12.910 kHz), pressure (4 Atm) and pressing time (10 s) were held constant. Three types of measurements are performed: measurement of diffractive efficiency; optical microscopy and atomic force microscopy.

2. Experimental setup

Illustration of experiment is shown in Figure 1. There are three major steps in the experiment. During the first step heating plate, with master mold attached to it, is preheated till 148 °C (what corresponds to glass transition temperature of polycarbonate). After the preheating, heating plate with master mold starts to move towards the polycarbonate, which is placed on vibroactive pad (pad is vibrating at frequency of 12.910 kHz, what corresponds to first vibration mode of it). As the heating plate reaches the polycarbonate, it durates 0.5 s, till the pressure reaches nominal value (in case of experiment 4 Atm), and later nominal pressure is being held for 10 seconds. The third step – demolding. During this

step, heating plate with master mold is being lifted up. Thus process consists of three steps: heating, pressing and demolding.

Figure 1. Experimental scheme of hot imprint process with ultrasonic excitation [8]



3. Examination techniques, used to define the quality

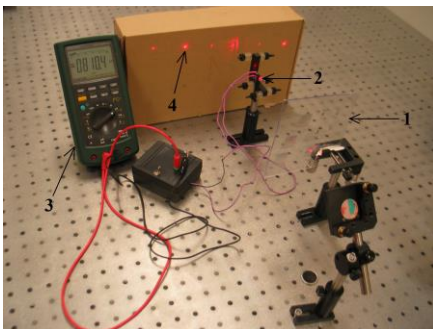
Measurement of diffracton efficiency was performed by using laser ($\lambda = 632.8 \text{ nm}$) and photodiode BPW-34 (Figure 2.). The whole scheme is connected with tester. The diffractive efficiency is being calculated by using the formula:

$$SE_{i,j} = \frac{I_{i,j}}{I_j} \quad (1), \text{ where}$$

$$I_j = \sum_i I_{i,j} \quad (2)$$

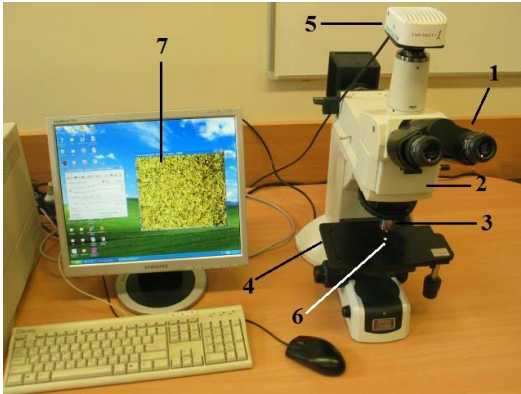
Explanation of the formula: first of all sum (I_j) of all currents (different diffraction angles) is being calculated, then particular current, obtained at particular maxima, is being divided from this sum and the relative value in percents is thus obtained. Before the measurement it was theoretically determined, that diffractive grating, whose period is $4 \mu\text{m}$ and depth 150 nm , has 6 maximas.

Figure 2. Diffractometer and measuring scheme (sample (1); photodiode (2); tester (3) and distribution of maximas (4)



Optical microscopy was used for analysis of microstructure. It was performed by using optical microscope NICON Eclipse LV 150 (Figure 3.), same objects were examined as well. The main purpose of this investigation is to spot, compare and calculate visible defects, in order to determine which one of microstructures is of better quality.

Figure 3. Optical microscope Nikon Eclipse LV 150. Trinocular tube (1); LV-UEPI2 Illuminator (2); CFI LU plan floor objectives (3); stage (4); CCD camera (5); specimen (6); view of specimen (7).

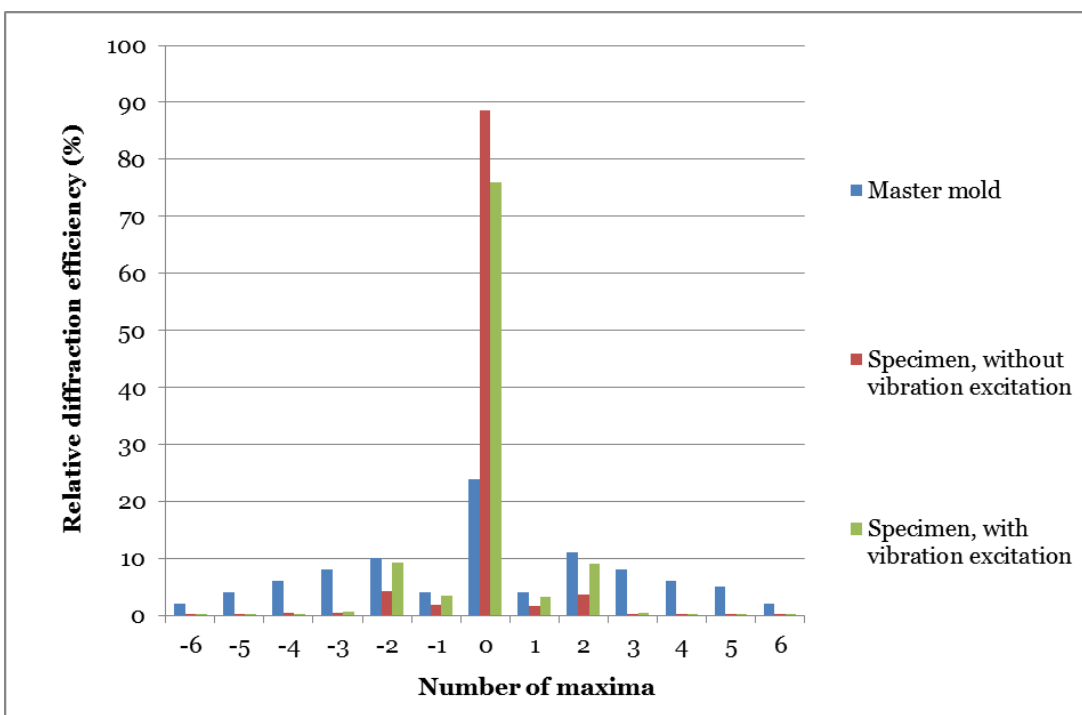


Atomic Force Microscopy is employed in order to find surface view by microscope NANOTOP-206. Investigation of this type is able to show profile of surface view of produced microstructure and show average depth of grating, this is the matter of concern, since the purpose is to replicate the master mold as precisely as possible.

4. Results and discussions

Results of measurement of diffraction efficiency (Figure 4) were analysed. After the measurement values of diffraction efficiencies were calculated, thus determining which one of them has better diffraction efficiency. In first and second maximas values of relative diffraction efficiencies are higher in the specimen, made with vibration excitation (3.5 % compared to 1.8 % in first maxima and 9.27 % compared to 4.27 % in second maxima).

Figure 4. Dependency of diffraction efficiency on the specimen



Magnified views of the polycarbonate's surface produced under the same conditions are presented in Figures 5 – 7. The main purpose of the comparison between the microstructures is to calculate visible defects and present the better case.

Figure 5. Master mold

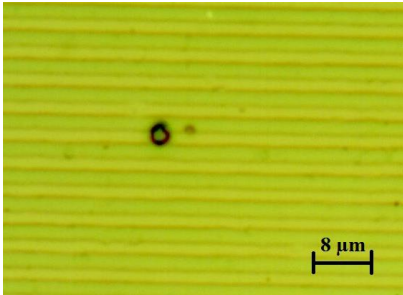


Figure 6. Microstructure made without excitation

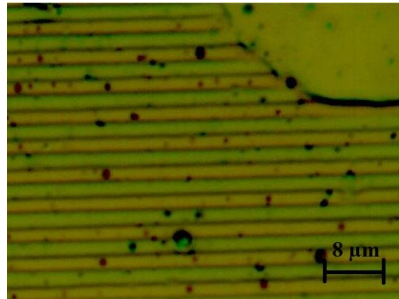
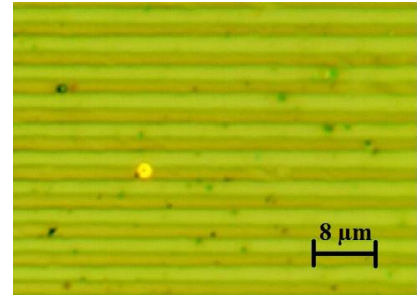


Figure 7. Microstructure, made with excitation



After the calculation of defects in images (per area of $2400 \mu\text{m}^2$), it was determined, that the specimen, made without vibration excitation has 51 visible defect, while specimen, made with vibration excitation has 34 visible defects (or 1.5 times less).

During the investigation with Atomic Force Microscope main concern was to find out how exact master mold is being impressed on the surface of polycarbonate. To show this, profile view images of master mold, replicas created with and without high frequency excitation are shown and compared.

Figure 8. Master mold

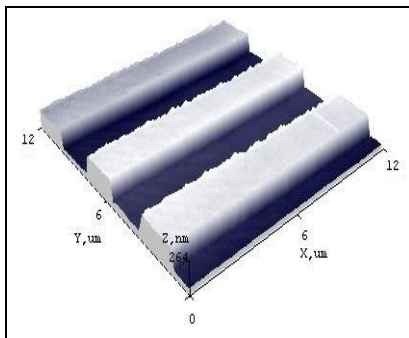


Figure 9. Microstructure made without excitation

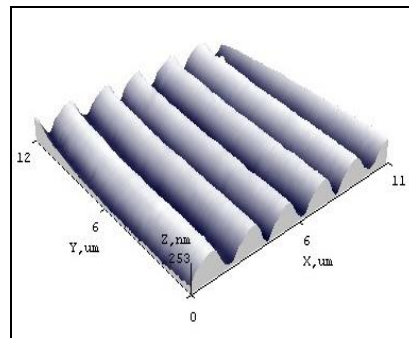
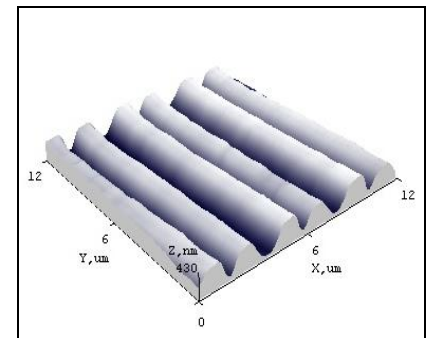


Figure 10. Microstructure, made with excitation



From the images, obtained after the examination with atomic force microscope, can be stated, that high frequency excitation allowed to obtain more similar to master mold microstructure. Average depth is the parameter which confirms this similarity. Average depth of master mold is 155.52 nm, depth of microstructure, made without vibration excitation is equal to 110.26 nm. The depth of microstructure, made with vibration excitation is 135.12 nm.

5. Conclusions

Measurement of diffraction efficiency shows, that most important maximas: first (3.5 %) and second (9.27 %) in replica, made with vibration excitation are higher than in specimen without vibration excitation (respectively: 1.8 % first maxima and 4.2 % second maxima).

After this investigation, by using optical microscope, number of visible defects was counted. The number of visible defects in specimen, made without vibration excitation is 51, whereas high frequency excitation decreased this number till 34, i.e. 1.5 times less.

Atomic force microscopy revealed average surface depth and showed profile view of microstructure. Average surface depth of master mold is 155.52 nm, microstructure, made without vibration excitation has average surface depth of 110.26 nm (difference between microstructure and master mold is 29 %) and microstructure, made with vibration excitation has average surface depth of 135.12 nm (difference between microstructure and master mold is 14 %).

Acknowledgments

This research was funded by a grant (No. MIP-026/2014) from the Research Council of Lithuania.

References and Notes

1. Lee, L.J. et al. Design and fabrication of CD-like microfluidic platforms for diagnostics: Polymer based micro fabrication.– *Biomedical Microdevice*, 2001, 3(4), p. 339-351.
2. Šakalys, R.; Palevičius, A.; Janušas, G. Vibroactive pad improvement using stack type piezoactuator.– *Vibroengineering PROCEDIA*. ISSN 2345-0533, 2013, vol. 2, p. 109-112.3.
3. Benatar, A; Gutowski, T.G. Ultrasonic Welding of PEEK Graphite APC-2 Composites.– *Polymer Engineering and Science*, vol. 29, no. 23, 1989, p. 1705-1721.
4. Nonhof, C.J; , Luiten, G.A; Estimates for process conditions during the ultrasonic welding of thermoplastics. – *Polymer Engineering and Science*, 36, 1996, p. 1177-1183.
5. Liu, S.J. et al. Development of Weldability Diagrams for Ultrasonic Welding of Thermoplastics in Far-Field.– *Plastic Rubber Composite Processing and Applications*, vol.27, no. 6, 1998, p.279-286.
6. Liu, S. J.; Chang, I.T.; Hung S.W. Factors affecting the joint strength of ultrasonically welded polypropylene composites. – *Polymer Composites*, 22, 1, 2001, p. 132-141.
7. Narijauskaitė, B.; Palevičius, A.; Narmontas, P.; Ragulskis, M.; Janušas, G. High-frequency excitation for thermal imprint of microstructures into a polymer.– *Experimental Techniques*. Malden : Wiley-Blackwell Publishing. ISSN 0732-8818, 2012, vol. X, iss. X, p. 1-7.
8. Šakalys, R.; Janušas, G.; Palevičius, A. Vibroactive Pad for Replication of Microstructure and Its Experimental Analysis. *Proceedings of 19th International Conference. Mechanika 2014*. ISSN 1822-2951, 2014, p. 225-227.

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