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Examining the surface of the polyamide nanocomposite using optical profilometry

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In this paper the results of neat polyamide 6 (PA6), processed matrix and polyamide 6 reinforced with montmorillonite 3 wt% (PA6/MMT), surface examination are presented. Examination of surface of the samples was performed using optical profilometry, which allows to measure their physical properties in non-invasive method. Based on the analysis of the radiation intensity of the reflected light from the surface the Fourier Transform was performed, to allow the analysis of images obtained in the frequency domain.

Keywords: polyamide nanocomposite, montmorillonite, optical profilometry, Fourier Transform images, surface examination

I. INTRODUCTION

In real surfaces can be distinguished characteristics more or less visualized periodicity and anisotropy. Cracks appear in most real surfaces (e.g. .: steel surfaces subjected to mechanical processing - cutting, turning, etc.). They can result from improper use of the item, or may be a remnant of the process of machining. If the size of the surface features are larger than 1 μm then optical profilometry becomes useful for characterization [1,2,3].

Analysis of images obtained with an optical profilometer could determine the shape of surface, the level of pollution, and in some cases the stage where processing has been stopped. Yet, only Fourier Transform allow the evaluation of the processes of creating surface and subsurface areas of the tested samples.

In the second chapter of this article tested materials and applied research method are presented. The results and their discussion are in the third chapter, and a summary closes this paper.

II. MATERIALS AND METHODS

The profilometer used (Figure 1), which is an equipment of laboratory of the Institute of Physics, Cracow University of Technology, is designed based on a photodetector, a laser diode driver's XY table OPTEL Opole, and fiber optic probe. The laser diode produces a light beam having a wavelength of 650 nm. Optical profilometry measurement was made on samples of polyamide 6 (PA6) - engineering polymer produced by Azoty Tarnow with trade name Tarnamid® T27 and Tarnamid® T30. The T27 is a variety of medium viscosity for the production of injection molded products having high strength requirements, including thin-walled, for the manufacture of monofilament, veins, bristles and fibers and for the preparation of granules modified by compounding.

The T30 is a variation a high viscosity in the preparation of extrusion casings for the meat industry, from sleeve film and flat single and multilayer pipes of small diameter, plates rods, profiles and injection molded articles thick [4]. For both varieties of PA6 samples were tested at three stages of production: unprocessed pellets, processed matrix and PA6/MMT nanocomposite.Montmorillonite (MMT) is a filler which is a layered silicate from bentonite family. Sodium montmorillonite, manufactured by the Italian company Laviosa Chimica Mineraria S.P.A., modified low content of quaternary ammonium salt (dimethyl dihydrogenated tallow ammonium) of the trade name Dellite® 72T.

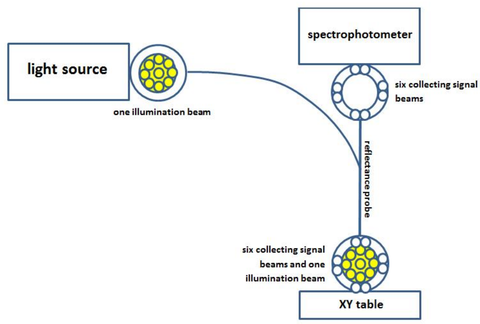

FIG. 1. (Color online) Schematic diagram of profilometer

TAB. 1. The selected processing conditions for polyamide-6/montmorillonite nanocompositesusing mini-technological line

Twin co-rotating screw extruder								
Flow rate (%)	Rotational speed (1/min)	Heating zones						
0.3	240	1	2	3	4	5	6	Die
Temperature (°C)		245	245	245	250	255	250	260
Atmospheric venting		–	–	–	–	Yes	–	–
Length of the zones (mm)		80	60	60	64	60	76	23
L/D		5.00	3.75	3.75	4.00	3.75	4.75	–
Cooling tank								
Length of cooling surface (mm)		1,500						
Tank volume (dm3)		27						
Height of bath (mm)		1,081						
Water temperature (°C)		18						
Granulators								
Size of pellets (mm)		1						
Rotational speed (1/s)		12						

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Before the preparation of nanocomposites, materials were dried in a laboratory vacuum oven. Polyamide was dried at 80°C for 3 hours. PA6 and PA6/MMT nanocomposite samples were prepared using a mini process line (twin co-rotating screw extruder Thermo Scientific Rheomex PTW 16/25 XL, cooling tank of ZAMAK and pelletizer ZAMAK G-16/325). The materials were processed at the processing temperatures shown in Table 1, at 240 rpm rotation of the screws. The sample bars in shape of plates were made using a laboratory injection molding machine ZAMAK WT 12.

For each surface sample area of 5 x 5 mm has been scanned. A measuring step of the intensity of light reflected from the sample was 0.05 mm. The program that was used to archive received data from the spectrophotometer was a 3D Scanner v 2.5.

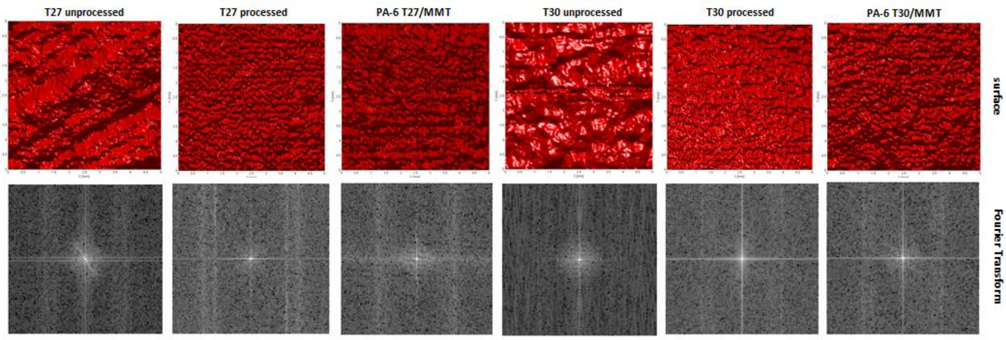

FIG. 2. (Color online) Visualization of the surface and Fourier Transform images for polyamide 6 T27 and T30 in three stages of production.

III. RESULTS AND DISCUSSION

The result of the measurement profilometry for the samples is shown in Figure 2. Pictures show the variation of the surface structure of test samples. For unprocessed samples Tarnamid® T27 and T30 could be distinguished areas where the pellets are not fully deliquesced. Clear folds on the surface and observed cavity allow to distinguish the unmolded samples from the rest. A variety of low molecular weight polyamide T27 shows a greater diversity of surface, than high molecular variant of the T30. This means that although the same conditions of processing, Tarnamid® T27 rapidly undergoes processing. Visible holes on the surface Tarnamid® T30 were formed as a result of contamination of the sample at these sites. For processed samples, a significant reduction of the folds of the two surfaces, was seen. It could be due to orientation of the chains of the polymer during high temperature processing. The area of PA6 T30 as opposed to the variety of low molecular weight shows a rarer variation surface which indicates the correct selection of processing conditions for this variety. Only at the stage of doping of polyamide 6, 3 wt% content of nanofiller, it is no possible to distinguish profilometry image of the sample containing the matrix T27 from the image of the sample containing the matrix T30. In the images shown in Figure 2, there is a similar arrangement of folds. Using optical profilometry the degree of homogenization of the filler within the test area was estimated and is at satisfactory level.

Analysis of Fourier Transforms obtained profiles allowed us evaluated the processes of creating surface and subsurface areas of polyamide 6 Tarnamid® T27 and T30. As it is apparent from Figure 2, processing operations indicate the random-periodic nature of a surface formation. In the case of unprocessed Tarnamid® T27 revealed the processes of the periodic nature. Yet, periodicity disappears after moulding. This is a proof of the disappearance of temporary factors and that the processing increases the 'randomness' of the process and results in the formation of isotropic surface in terms of the physical properties of the surface. Strong dispersion of the reflected light allows the assessment of the tested surface as a lambertian, and thus having characteristics of an ideal diffuser.

IV. CONCLUSIONS

Using optical profilometry the degree of homogenization of the filler within the test area was estimated and is at satisfactory level. Analysis of Fourier Transforms obtained profiles allowed us evaluated the processes of creating surface and subsurface areas both types of polyamide 6: Tarnamid® T27 and T30.

Test samples are very sensitive to any contaminants that begin to be significantly visible only during optical surface profilometry. This demonstrates the enormous sensitivity of the method used.

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