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Determining the melt flow index of polypropylene:Vistalon 404

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Properties

ABSTRACT

Purpose: Experimental determination using the MFR and MVR methods to obtain the most uniform material, smooth, without structural defects.
Design/methodology/approach: The device used to determine the index MFR and MVR was MFlow extrusion plastometer, Zwick/Roel. The evaluation of obtained samples was carried out using a stereoscopic microscope, Zeiss, and a scanning electron microscope Supra 25, Zeiss. Determination of the chemical composition of materials was carried out by using IR spectrometer with Fourier transformation, SpectroLab.
Findings: One of the requirements for quality control of thermoplastics is determination of volume and mass flow rates. To ensure in industry that production is reproducible it's important to control this index.
Practical implications: The purpose of the determination of MFR and MVR is a quick and inexpensive knowledge about the basic properties of thermoplastics processing, which is the flow rate. Knowing the material's quality which is introduced into the manufacture makes the process parameters selection (injection molding, extrusion) much easier. It also reduces the time and minimizes the cost of the production setup.
Originality/value: The MFR and MVR were experimentally determined and they have been confirmed by microscopic research. The research were focused on observing the topography changes depending on temperature.
Keywords: Thermoplastics; Melt mass flow rate (MFR), Melt volume flow rate (MVR)

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1. Introduction

Currently, both the scientists and manufacturers want to get the materials with an optimal properties. Research into polymer materials are in progress for over 100 years. Currently, polymeric materials are used in many fields ranging from the aviation and electronics, to furniture and light industry. They occur very rarely in a pure form without additives such as fillers, stabilizers, plasticizers, dyes, or even other polymers. Continuous improvement and doping allows us to create new and more efficient products.

One of the requirements for quality control of thermoplastics is determination of volume and mass flow rates. To ensure in industry that production is reproducible we must control this index. MFlow extrusion plastometer can be used to determine the melt mass flow rate (MFR) and melt volume flow rate (MVR). Typical uses are in quality assurance and goods inwards checks.

The purpose of the determination of MFR and MVR index is up quick and inexpensive to know the basic properties of thermoplastics processing, which is the flow rate.

Knowledge of the quality of the material that it's introduced into the manufacture makes it much easier to select process parameters (injection molding, extrusion), reduces the time and minimizes the cost of production preparation.

2. Materials and methods

Research was conducted on polymeric material in granular form. The research covered preparation of a granular material (cleaning, drying, weighting), determination of the chemical composition of materials by using IR spectrometer with Fourier transformation, correct selection of the temperature and flow parameters of the material through the orifice, the insertion of the plastometer and generating appropriate segments.

To determine the optimum process temperature the material was subjected to careful analysis. Detailed research was carried out on a stereoscopic microscope and scanning electron microscope (SEM). Samples for stereoscopic microscope were not particularly prepared. Samples for SEM were respectively purified from fat and other surface contaminants. Then they were vapor deposited by metal (platinum) to reduce the loading effect and enhance the emission of secondary electrons, so that the resulting image was more pronounced.

The purpose of this research was to determine the melt mass flow rate (MFR) and melt volume flow rate (MVR) of polypropylene:Vistalon. The device used to determine the index MFR and MVR was MFlow extrusion plastometers, Zwick/Roel. The evaluation of obtained samples was carried out using a stereoscopic microscope, Zeiss, and a scanning electron microscope Supra 25, Zeiss.

The scope of the research included:

- identification and analysis of a polymeric material chemical composition by using an IR spectrometer with Fourier transformation, SpectroLab;
- preparing a polymeric material as a granulate to determine the melt mass flow rate and melt volume flow rate - removing the impurities, drying of the granulate, weighting;
- preparing MFlow extrusion plastometers to determine the MFR and MVR of the polymeric material;
- determine the MFR and MVR of the polymeric material with different temperatures and using different of weights;
- examination obtained samples of the polymeric materials with using a stereoscopic microscope, Zeiss, and a scanning electron microscope Supra 25, Zeiss.

All research was carried out for observing the topography changes depending on temperature. The purpose of this research was to experimental determination of the MFR and MVR, so as to obtain the most uniform material, smooth, without structural defects.

3. Results and discussion

The Fourier infrared spectroscopy (FTIR) method is one of analytical methods that allow a rapid diagnosis of small amount of material (<1 mg). Spectra of the test samples was performed by using a spectrometer IR with Fourier transformation, SpectroLab. The research was carried out with ATR (Attenuated Total Reflection).

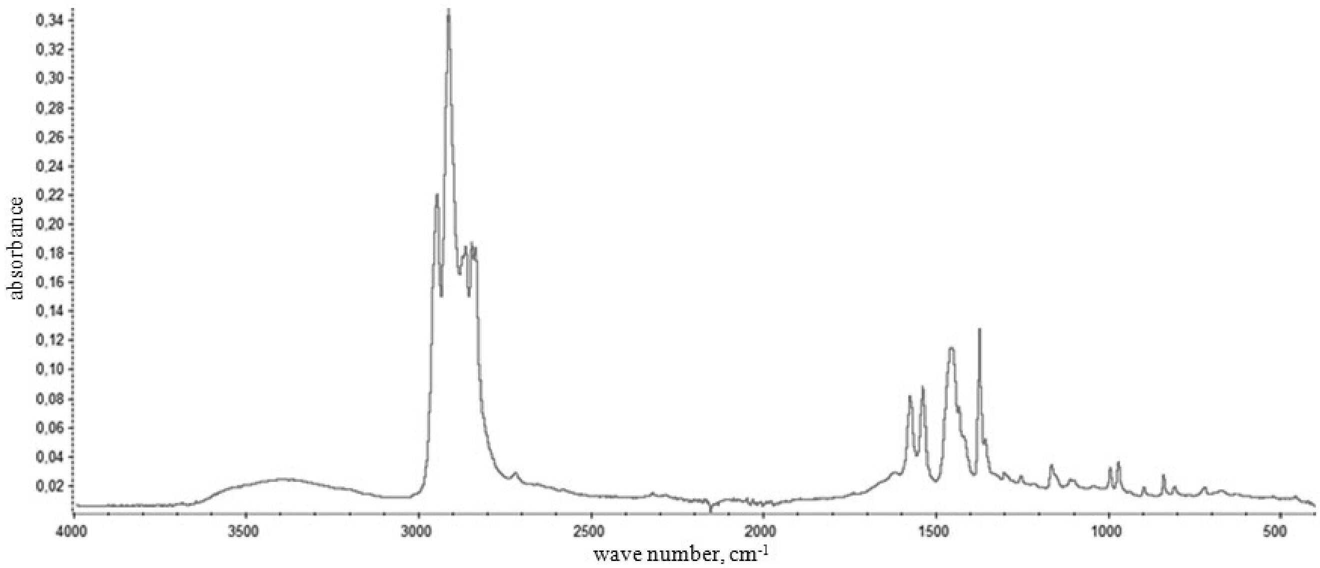

Fig. 1. The spectrum obtained on a FT-IR spectrometer with ATR for a sample of the material in granular form

In order to register the spectrum we have set the following parameters of the spectrometer:

- the number of scans of the spectrum of sample: 128,
- the number of scans of the spectrum background: 128,
- the resolution: 4,000,
- gain: 4.0,
- scanning speed 0.6329,
- diaphragm: 100.00.

The IR spectrum obtained from the test material was shown in Fig. 1. On registered spectrum at a wavenumber from 2700 to 3000 cm-1 was shows a band of stretching vibrations bond for C-H. In the range between 1300 to 1500 cm-1, there is CH deformation vibrations band. At a wavenumber below 1000 it can be seen C-C skeletal vibrations band.

Based on the data read from the wave spectrum and ratio analysis of elements and their bonds were identified that the material tested was polypropylene:Vistalon 404 (Fig. 2).

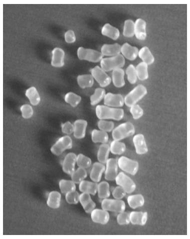

Fig. 2. The material in the form of a granulate used to determine the melt mass flow rate (MFR) and melt volume flow rate (MVR)

To determine the correct temperature flow, so as to obtain the appropriate research material properties should refer to EN ISO 1133, the data are presented in Table 1. On the basis of the flow temperature of pure polypropylene it is easier to choose the process parameters of the material test sample.

Table 1.
Flow temperature of pure polypropylene by using a base load - EN ISO 1133

Norm	Temperature [°C]
ISO 1873-2	230
ASTM D 1238	125
ASTM D 1238	230

Research was carried out on plastometer Mflow Zwick/Roell (Fig. 3), which is used in determining weight and volume flow rate. Research was carried out according to the existing standard.

The temperatures at which the material was investigated (polypropylene:Vistalon), are located in range from 170-180°C (Table 2).

During the research, the following parameters have been set:

- method of measuring the B - cutting segments dependent on the position of the piston - 52 mm, 5 segments being 5 mm on the scale of the piston - test carried out by using a piston with a load of 325 g, the material passed through the orifice properly and 5 segments were correctly obtained,
- pre-heating time - 300 s,
- active option cut-off episode after detecting vesicles in the material,

The mass of granules and the canal temperature of the test are shown in Table 2.

During the tests it was observed that:

- below 168°C the material arrears in the canal and does not pass through a orifice,
- at 170°C, flow time of the polymeric material through the die is quite long, but with additional weight (2.16 g) a lot of air bubbles has appeared in the material,
- material heated above 180°C flows through the orifice too fast, pulling up and no test bead form is created,
- the material is transparent thus it is very easy to observe any flaws in form of air bubbles (Fig. 4).

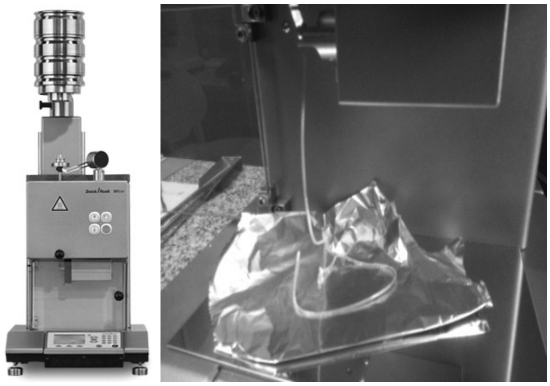

Fig. 3. Determination of the melt flow index of plasticized polymer material by using plastometer Mflow Zwick/Roell

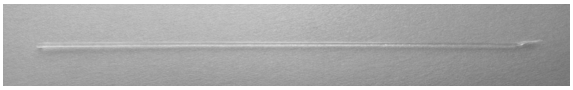

Fig. 4. A segment of the polymeric material (polypropylene:Vistalon) acquired by plastometer Mflow Zwick/Roell

Table 2.
Weight of granules polypropylene:Vistalon 404 and the temperature of the test channel

Mass [g]	Temperature [°C]	Comments
3.70	170	Long time of performed test (25 minutes)
4.08	175	-
3.30	180	Visible air bubbles

The test results for the polypropylene material are shown in Table 3, observations made during the test:

- The test material has both the highest mass (MFR) and volume (MVR) melt flow rate at 175°C and the lowest rates at 170°C,
- At 170°C only 3 segments were obtained because of too long heating duration (according to the polymeric material norm, it can't be heated in the test system longer than 25 minutes)
- The test took least time at 175°C.

Table 3.
Summary of the tests results for polypropylene:Vistalon material

Nr (temp.)	Index (section number)	MFR_n g/10min	$\overline{MFR}$ g/10min	MVR_n cm^3/10min	$\overline{MVR}$ cm^3/10min	$t(Start)_n$ s	$T(Ende)_n$ s
1 (170°C)	1	0.68	0.66	0.92	0.89	577.3	810.2
	2	0.65		0.88		810.2	1052.7
	3	0.65		0.88		1052.7	1295.2
2 (175°C)	1	1.22	1.234	1.65	1.67	300.2	345.9
	2	1.23		1.66		345.9	474.5
	3	1.24		1.68		474.5	601.7
	4	1.24		1.68		601.7	728.9
	5	1.24		1.68		728.9	856
3 (180°C)	1	0.94	0.96	1.28	1.298	625.2	793.4
	2	0.96		1.29		793.4	958.3
	3	0.97		1.31		958.3	1121.8
	4	0.96		1.30		1121.8	1286.5
	5	0.97		1.31		1286.5	1449.7

The sample of material obtained using MPlastometer has been tested using both stereoscopic light microscope (Figs. 5-7) and scanning electron microscope. During the follow-on light microscopy it has been noticed noticed that at 170°C there are transverse lines and places where you can see the refraction of light in the material, at a temperature of 175°C the material was smooth, uniform without any visible structural defects and air bubbles, while at highest temperature (180°C) the air bubbles were explicitly visible. On this basis, we can state that the material obtained at 170°C is most patchy of all received burrs. The research was also conducted on the scanning electron microscope (Figs. 8-10).

Tests were carried out over a long period of time, due to the desire to achieve a smooth and uniform material with no major structural defects, the observations have confirmed that there were no visible defects and impurities.

Small cracks and air bubbles were observed in the test samples, but the most uniform material was obtained at a temperature of 175°C.

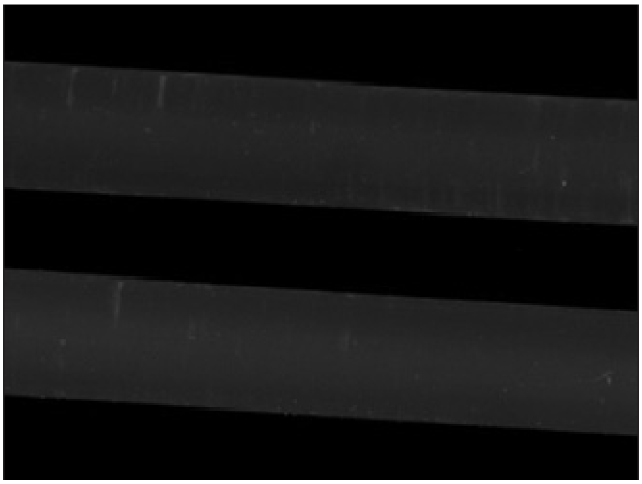

Fig. 5. Stereoscopic microscope image obtained from polypropylene:Vistalon 404 tested at 170°C (16x zoom)

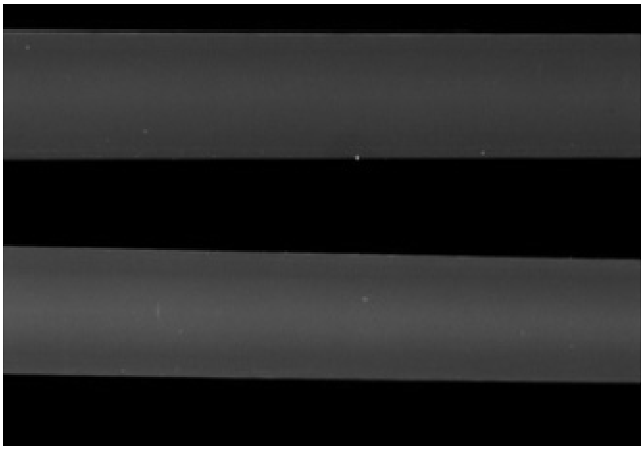

Fig. 6. Stereoscopic microscope image obtained from polypropylene:Vistalon 404 tested at 175°C (16x zoom)

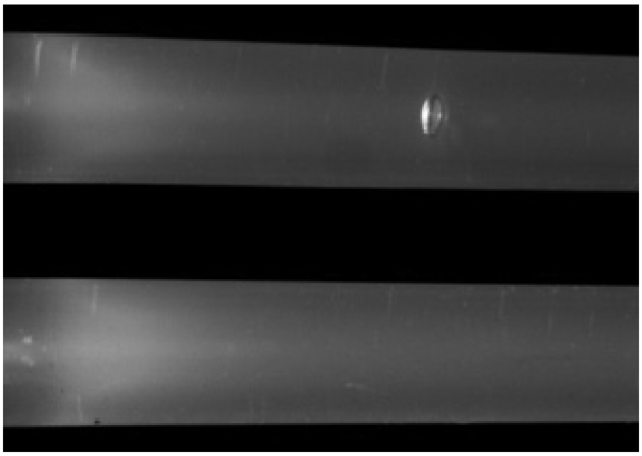

Fig. 7. Stereoscopic microscope image obtained from polypropylene:Vistalon 404 tested at 180°C (16x zoom)

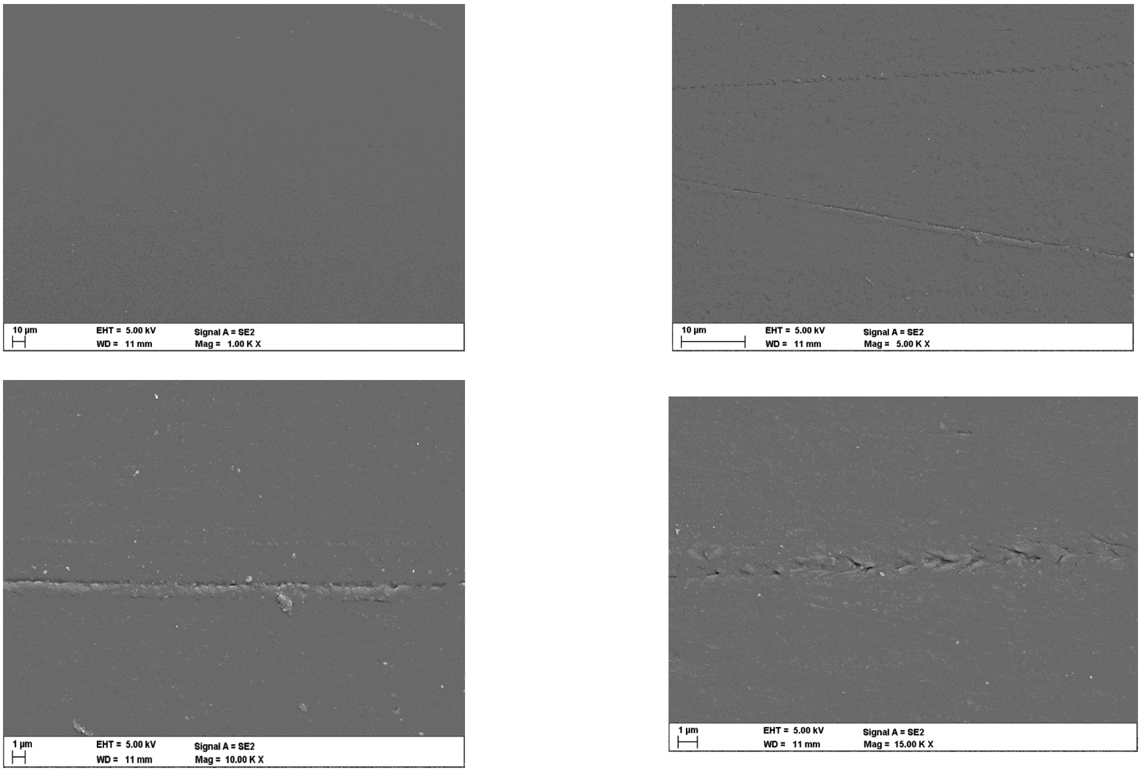

Fig. 8. Scanning electron microscope image obtained from polypropylene:Vistalon 404 tested at 170°C

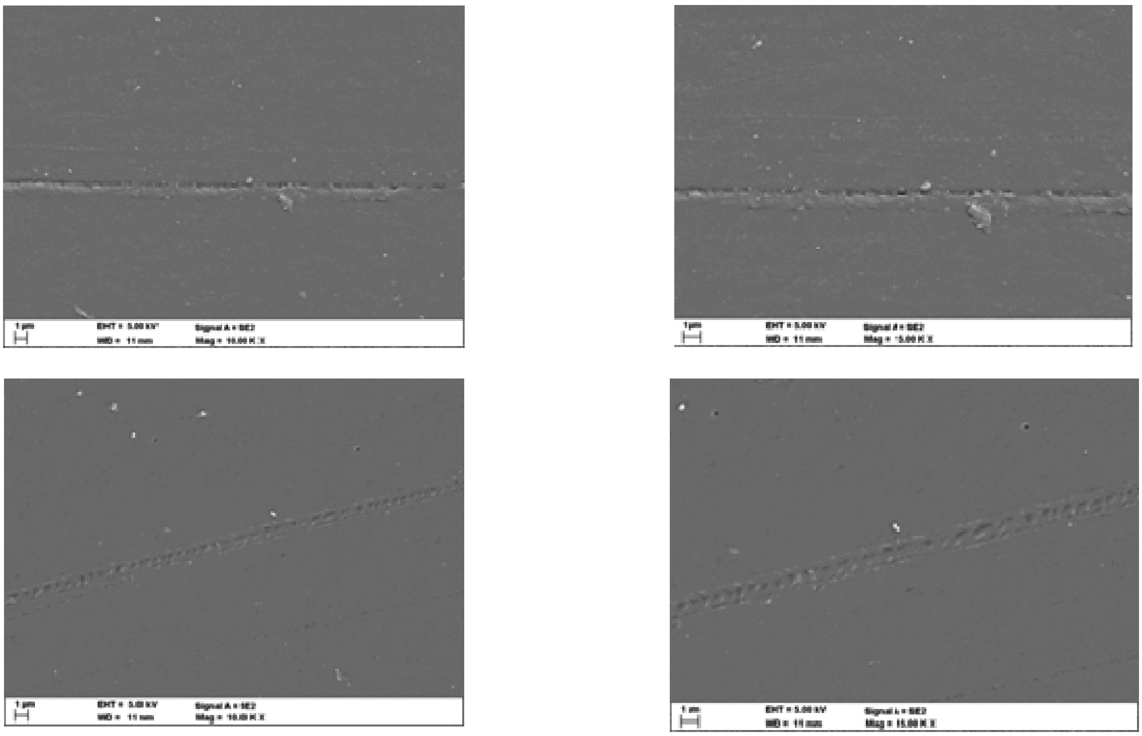

Fig. 9. Scanning electron microscope image obtained from polypropylene:Vistalon 404 tested at 175°C

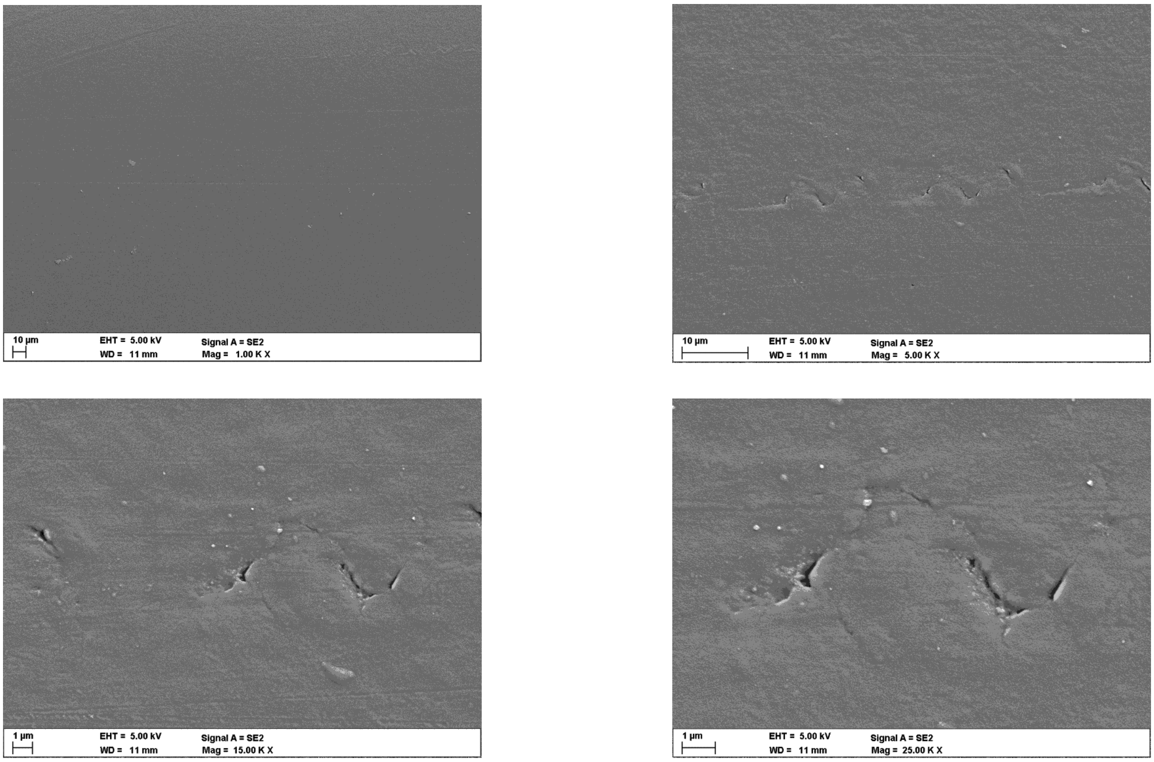

Fig. 10. Scanning electron microscope image obtained from polypropylene:Vistalon 404 tested at 180°C

4. Conclusions

During the test of the material polypropylene:Vistalon 404, it was easy to notice any material defects such as air bubbles, due to its transparency.

At a temperature of 170°C time of the polymeric material flow through the orifice was quite long, and with the additional weight (2.16 g) there was a lot of air bubbles in the material.

At temperatures below 168°C the material stack up in the test canal and did not pass through the orifice.

The granulate heated to a temperature above 180°C flowed through the orifice too fast and didn't get a form of bead for further studies.

Sample of polypropylene:Vistalon 404 obtained at 175°C had the most equal topography of the surface.

The temperature of the polymeric material and the pressure in the test canal significantly affect the value of the volumetric flow rate and weight of the sample.

Basing on microscopic examination it was found that the most equal topography of the sample's surface is from a polypropylene:Vistalon 404 obtained at 175°C with a 0.325 g weight of the piston.

The mass melt flow rate for this sample was MFR = 1.234, and the volumetric melt flow rate was MVR = 1.67.

In order to obtain a uniform bead, reduce time and minimize costs of production it is recommended to prepare this material in the molding temperature of 175°C.

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References

[1] A. Stolarzewicz, Methods for the synthesis of polymers and their characteristics, Publishing House University of Silesia, Katowice, 2005 (in Polish).

[2] D. Żuchowska, Structural polymers. Processing and properties, Publishing House Technical University of Wroclaw, Wrocław, 1993 (in Polish).

[3] W. Kucharczyk, W. Żurowski, Processing of plastics for mechanics, Publishing House Radom University of Technology, Radom, 2005 (in Polish).

[4] I. Hyla, Plastics, property - processing-application, Publishing House Silesian University of Technology, Gliwice, 2004 (in Polish).

[5] K. Kelar, D. Carpenters, Physical chemistry of polymers. Publishing House Poznan University of Technology, Poznań, 1997 (in Polish).
[6] J. Kijeński, waste polymeric material and a source of trouble Conference - Ministry of Economy, Warsaw, 2010.
[7] I. Gruin, Polymeric materials, Publishing House PWN, Warsaw, 2003.
[8] A. Proń, Synthetics metals, Knowledge and Life 2 (2001).
[9] W. Grellmann, S. Seidler, Polymer testing, Hanser Verlag, 2007.
[10] M. Żenkiewicz, P. Rytlewski, K. Moraczewski, M. Stepczyńska, T. Karasiewicz, R. Malinowski, W. Ostrowicki, Some effects of multiple injection moulding on selected properties of ABS, Journal of Achievements in Materials and Manufacturing Engineering 37/2 (2009) 361-368.
[11] A. Akinci, Mechanical and structural properties of polypropylene composites filled with graphite flakes, Archives of Materials Science and Engineering 35/2 (2009) 91-94.
[12] M. Rojek, J. Stabik, Mechanical and electrical properties of mined coal filled polyethylene and polyamide, Archives of Materials Science and Engineering 36/1 (2009) 34-40.
[13] J.D. Ferry, Viscoelastic Properties of Polymers, Publishing house Wiley, 2009.
[14] K. Van de Velde, P. Kiekens, Polymer testing. Thermoplastic polymers: overview of several properties and their consequences in flax fibre reinforced composites, 2001, 885-893.
[15] H. Shirakawa, Synthesis and characterization of highly conducting polyacetylene, Synthetic Metals 69 (1995) 3-8.
[16] T.A. Osswald. G. Menges, Materials science of polymers for engineers, Carl Hansen Verlag, Munich, 2003.
[17] G. Hartwig, Polymer properties at room and cryogenic temperatures, Springer, 1994.
[18] J. Stabik, A. Dybowska, M. Chomiak, Polymer composites filled with powders as polymer graded materials, Journal of Achievements in Materials and Manufacturing Engineering 43/1 (2010) 153-161.
[19] J. Weszka, M. Szindler, A. Śliwa B. Hajduk, J. Jurusik, Reconstruction of thin films polyazomethine based on microscopic images, Archives of Materials Science and Engineering 48/1 (2011) 40-48.
[20] J. Weszka, M.M. Szindler, M. Chwastek-Ogierman, M. Bruma, P. Jarka, B. Tomiczek, Surface morphology of thin films polyoxadiazoles, Journal of Achievements in Materials and Manufacturing Engineering 49/2 (2011) 224-232.