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Visualization and flow velocity determination of molten polymers

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Abstract: The determination of flow vectors of molten polymer by dies and mold cavities filling is a topic of many investigations, realized by various measurement techniques. The non-direct measurements are based on temperature or pressure fields determination along the flow channels, where the sensors are usually positioned in the streaming polymers, leading to flow field disturbance. The Laser Doppler Velocimetry (LDV) is an efficient touchless measurement technique of streaming gases and liquids velocity, delivering directly values of the flow velocity and its direction. Principle and examples of LDV determined flow velocity distribution, are presented. The direct flow lines determination may be done by using markers with a plate-like form, scanning electron microscopy (SEM) observation of fractured product surface.

Keywords: molten polymer flow, velocity, non-direct methods, Laser Doppler Velocimetry, flow markers.

Wizualizacja oraz pomiar prędkości przepływu stopionych polimerów

Streszczenie: Pomiar wektorów prędkości przepływu stopionych polimerów przez dysze i kanały form wtryskowych stanowi przedmiot badań prowadzonych z zastosowaniem różnych technik. Pośrednie pomiary są oparte na ocenie pola wartości temperatury lub/i ciśnienia na długości kanału, przy czym czujniki z reguły są umieszczane bezpośrednio w przepływającym polimerze, co może prowadzić do zakłóceń przepływu. Laserowa ocena przepływu z wykorzystaniem efektu Dopplera w optyce (LDV) to bardzo efektywna bezstykowa metoda pomiaru przepływu gazów i cieczy, pozwalająca na określenie prędkości i kierunku przepływu. Opisano zasady dokonywania pomiarów oraz przykłady zastosowania LDV. Bezpośrednią ocenę charakteru przepływu umożliwia wykorzystanie markerów w postaci napełniaczy płytkowych w polimerze i następnie obserwacja przełomów wyrobu za pomocą skaningowej mikroskopii elektronowej (SEM).

Słowa kluczowe: stopiony polimer, przepływ, metody pośrednie, laserowa ocena przepływu z wykorzystaniem efektu Dopplera w optyce (LDV), markery przepływu.

The final properties of polymeric products fabricated by techniques, like injection molding, extrusion, compression molding and others, beside material selection and its composition, are significantly macromolecular orientation dependent. This effect is widely known and described in papers where as well the anisotropic effects

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are observed, as the flow dependent shrinkage and thermal stability. An interesting processing outcomes are an orientation caused anisotropy of mechanical properties [1, 2] like elastic modulus and elongation, anisotropy of the heat transfer [3, 4], as well as changes of thermal properties, like melting temperature by semi-crystalline polymers [5, 6], due to significant macromolecular orientation.

Consequently, to find how the physical properties are influenced by macromolecular orientation, and/or privileged positon of polymeric chains, several structure related investigations, like wide-angle X-ray scattering (WAXS), differential scanning calorimetry (DSC), scanning electron microscopy (SEM), atomic force microscopy (AFM) are habitually used. In this case a special procedure of sample preparation is needed. Most frequently techniques used for sample preparation, for structure determination are: micro cuts of microscopic samples, cutting of mg-like samples for DSC, as well as creation of special forms, like film with a micrometric thickness, or etching [7] for samples where the spherulitical morphology on the surface will be detected. The main task by all these investigation methods, is the observation of structure in the final products and in specially prepared samples. To complete the structure characteristic, especially in samples and products with larger thickness, special procedure like gradient dependent investigation has to be applied [8]. An alternative way allowing to follow the structure formation during polymers processing is the molten flow visualization, by means of optical studies of the flow lines. This methods are known since years, although due to certain restrictions and experimental concerns, still in development.

FLOW VISUALIZATION BY BIRKS AND BAGLEY

Bagley and Birks published in 1960 [9] the flow lines of molten polyethylene at the capillary entrance, using a new technique for visualization, of flow patterns below and above the critical value of shearing stress, leading to distorted stream of extruded molten polymer. It allowed to observe directly the melt flow above the capillary entrance, and to determine the rate at which the energy in the flow may be dissipated, resulting in a steady flow into the capillary (Fig. 1).

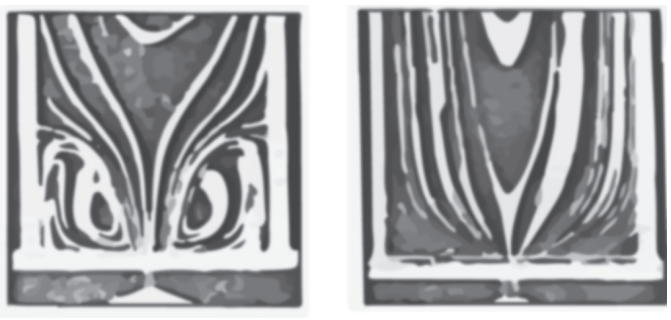

Fig. 1. The flow of molten LDPE at the inlet of the die [9]

FLOW MEASUREMENTS BY USE OF PRESSURE AND TEMPERATURE SENSORS

The usage of temperature and pressure sensors, positioned directly in the streaming polymer may, also serve to determine indirectly the molten flow velocity of polymers by passing the channels or the dies. Several restriction have to be taken into account by these measurements, particularly the ratio between measurements receptivity and short response time *vs*. mechanical resistance of sensors, sufficiently negligible distortion of the flow of molten polymers, due to the sensors placed directly in streaming melt, as well as appropriate thermal resistance of the measuring system. As a temperature gradient is normally observed in the molten polymer flow [10], to gain the knowledge of the entire temperature field on the cross section of the flow, the sensors must be displaced along the die/channel radius, thus special systems are needed in this case [11–13].

The positron emission particle tracking was applied by [14] to visualize the flow of a polymeric composite in twin-screw extruder, in realistic processing conditions. The residence time and residence time distribution in screw sections, like kneading part, conveing and reverse elements was compared with numerical particle tracking.

THE TOUCHLESS OPTICAL FLOW VELOCITY MEASUREMENTS

The arrangement and orientation of additives in form of fibers, by flow of polymeric composites inside the *orifice narrow channels*, was studied by means of visualization and numerical evaluation [15]. The determination of fiber angle variation along the flow channel, in different initial locations of the fiber, allows to conclude that by using of such method, a better understanding of flow induced anisotropy of physical properties of composite may be achieved.

The way to follow the homogenization process in the case of a composite of low density polyethylene with carbon black was published by Wong *et al*. [16–20], where also another observations obtained by this visualization technique are reported. The dynamic mixing in a *single screw extruder* was observed by using a window mounted directly in the extruder barrel. The time dependent mixing process and its fluctuation as a function of screw velocity, *i.e.* residence time were observed, where also the screw rotation speed, leading to the best mixing quality, was determined.

The quality of twin-screw extrusion mixing of polymer blends, composed of various ratio between polypropylene (PP) and polystyrene (PS) parts, was studied by [21]. The direct flow observation, using of a glass window incorporated in the twin-screw barrel, allows to realize the online visualization of the quality of mixing procedure of these two polymers.

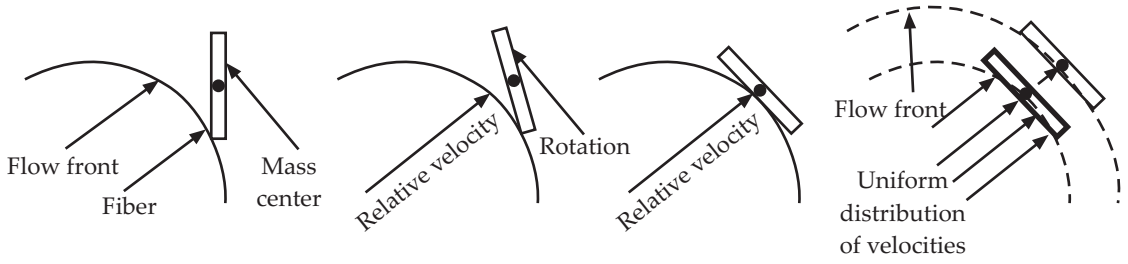

Fig. 2. Angular rate of rotation of single particle [25]

The direct visualization of molten polymer flow, by means of optical windows fixed in the injection mold, have also been published. Masato *et al*. [22] have studied the flow of polystyrene in an injection mold with low--friction mold surface coatings. Particularly, the flow of polymer in the thin-wall parts, causing the wall slip effect, has been observed by use of a mold with sapphire windows. A high-speed visualization during the filling phase, and the measurements of velocity distribution across the cavity thickness allowed to confirm the absence of the typical "fountain-flow" in this particular case. On the contrary, the importance of the adhesion at the polymer-mold interface was demonstrated by means of measurements of contact angle of molten polystyrene across the considered mold surface coatings, showing that the value of angle was inversely proportional to the melt flow resistance.

An interesting and valuable visualization of instabilities in polymer flow was proposed by Nabiałek [23], by investigations of polymer flow during cavity filling phase. In this case a dedicated injection mold which enables the observations and video recording of the *in situ* flow of the molten polymer, has been used. The direct monitoring of the streaming polymer inside the mold cavity in two planes was realized by means of a transparent sight-glasses, made of the glass ceramic Zerodur [24], characterized by the nearly zero coefficient of thermal expansion. To record the stream direction, a digital video camera has been employed, allowing to described the effect of polymer flow around the rectangular obstacle at the cavity, as well as the jetting phenomena.

A very simplified validation of particles orientation was proposed by Rajasekaran *et al*. [25], based on determination of natural fiber orientation during flow in transparent mold cavity, and by the numerical and experimental approach to evaluate the flow phenomena. A silicone resin, as a viscous fluid and natural filler – short coconut fibers with an average length of 2 mm, were applied in these investigations. The cavity filling steps during silicone/coconut fiber injection were recorded by a digital camera, and the numerical calculations were done by considering fluid elements and predicted angular velocity of laminar flow. The Authors described the shape parameter as a fiber curling factor, in the case when the coconut fibers are not enough stiff and revealed curled effect. The angle of coconut fibers orientation was validated by the assumption that the polymer flow front induce an impulse moment on short fiber and shears short fiber to rotate with respect to fiber geometrical center (Fig. 2).

The Rajasekaran researcher group found that fibers move with rotational and translation motion during filling of cavity, an effect in a good agreement with theory of flow. The numerically predicted orientation allow to conclude that the proposed model was validated with experimental data.

THE LASER DOPPLER VELOCIMETRY OF MOLTEN POLYMERS FLOW

The most effective way to determine the flow velocity of gases and liquid systems are the direct measurements by means of laser Doppler velocimetry (LDV), called also laser Doppler anemometry (LDA) [26]. This widely used technique in the case of flow measurements in turbine systems is based on the Doppler principle in optics. One of the main benefit of LDV is the possibility to realize the measurements of the flow velocity of gases and liquids touchless, *i.e.* only a laser beam is positioned in the stream, thus a principle of non-distortion of the flow is fulfilled. By the LDV measurements the velocity vector with its value, direction, turn and local position may be touchless determined, thus the LDV experiments may deliver significant information's necessary to design channels and dies in many industrial applications, allowing to optimize the flow pathway of practically all streaming mediums.

In 1964, LDV was employed for the first time by Yeh and Cummins [27] for measuring the velocity profiles of streaming water. Due to the relatively complex design of LDV measurement systems, particularly in the case of molten polymers flow determination where experiments with the use of thermally resistant optical systems should be used, its description may be found in few papers [28–32].

The setup and the way to realize the measurements of the molten polymer flow by LDV was published by [33]. Similarly as in [27–31] the Authors have used a slit die with a planar contraction of 14 : 1. The investigation of the velocity fields was performed in the steady state of flow. The optics of the LDV system was reliable for measurements of even very low velocities. To improve the signal

quality the TiO_2 tracer particles were introduced into the molten polymer. It was also demonstrated that the LDV allow to detect the velocity of polymer melts with an error of few percent, *i.e.* lower comparing with the measurements of the volume flow rate. The viscosity functions were obtained by measuring over a wide range of shear rates, using the velocity profile by fully developed flow in the slit die, checked additionally by measurements done with a capillary rheometer.

The Authors have concluded their research with a statement that the LDV setup is a powerful experimental tool to investigate the flow behavior of polymer melts, and its accuracy and high spatial and temporal resolution opens a way to get more quantitative insight into the flow of polymer melts, and to check the validity of certain model calculations.

The entry flow of low-density polyethylene was determined by laser Doppler velocimetry by Wassner *et al*. [34]. The measurements were realized by using an experimental extrusion set with a slit die, where the flow velocity was measured. This highly developed measurement technique was used also to determine the secondary flow of the polyolefins melts depending on its molecular structure [35].

The volumique defects appearing during extrusion were discussed in the paper published by Combeaud and other [36], based on the measurements of molten polystyrene flow. These experiments were realized simultaneously by birefringence and laser Doppler velocimetry. An analogous experimental set, *i.e*. instantaneously birefringence and LDV measurements were employed by Schubert and Muenstedt [37] for the determination of the stress and flow velocity distribution on the cross section of the channel.

In the papers from Hertel and Muenstedt [38, 39] the influence of the processing parameters on the secondary flow of polyethylene, measured by LDV by the three dimensional entrance flow of LDPE and LLDPE into a slit die, are presented.

Burghelea and others [40] have described an experimental study of the physical origin and the mechanisms of the sharkskin instability. Two polymers, *i.e*. a linear low density polyethylene (LLDPE) which exhibits sharkskin instability, and a low density polyethylene (LDPE) which does not show any instability over a broad range of flow rates, were investigated by combining the laser

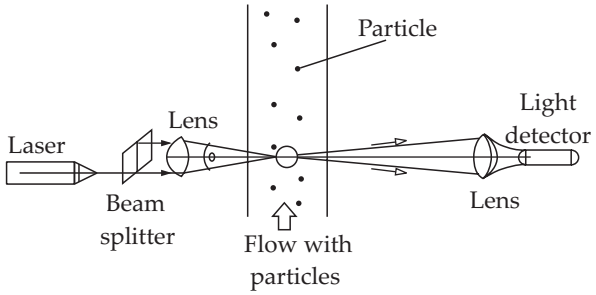

Fig. 3. The principle of laser Doppler velocimetry measurement technique [26–29, 33]

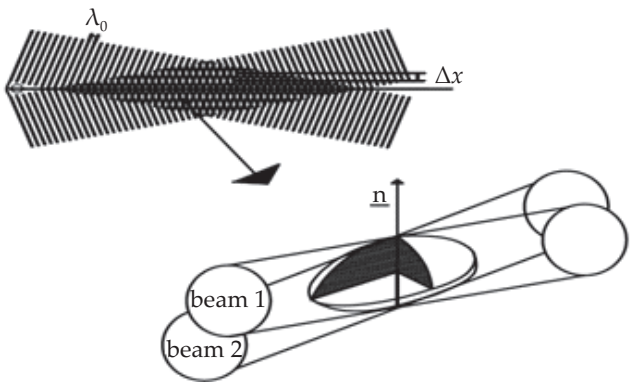

Fig. 4. The measurements volume created by focused laser beams with the interference lines

Doppler velocimetry (LDV) within a slit die with rheological measurements in both uniaxial extension and shear flow. The principle of the laser Doppler velocimetry measurement technique is presented on Fig. 3. The laser beam radiation is splattered on two beams, where by one of them, by passing through a Bragg cell, a defined shift of light beam frequency is achieved [26–29, 33].

The two beams are focused passing by the lens, and create a measurements volume with the dimension of some micrometers, which may vary depending on the angle between the beams focused by the lens, and the distance between the lens and the measured flow (Fig. 4).

In case of the LDV measurements in the flow of molten polymers, due to the relatively high temperature of the streaming medium, the distance between the optical system and the extrusion die may not be too short [28, 29, 33]. The creation of the back-scattered light is essential to fulfill the necessary conditions of the Doppler effect in case of measurements of streaming medium. Therefore, usually the micro particles, like for example TiO_2 [28, 29, 33], are dispersed in the liquid, in our case in the molten polymer.

Such LDV measurements are restricted due to some technical difficulties, like the necessity to use an extrusion die with transparent wall in a form of glass windows, resistant against the high temperature and assuring a tightens for the flow of molten polymers. Another significant condition is the achievement of a high transparency for the optical beam, even in application of molten polymer processing, *i.e.* the light refraction coefficient should be as low as possible. In this case the most favorable results [23, 28, 30] were completed by using the Zerodur [24], a special low thermal expansion glass ceramic for special optical applications. The Zerodur may relatively easily be formed mechanically, possessing the necessary thermal resistance and optical properties. As stated already above, the additional requirement in case of molten polymer flow determination is a high temperature resistance of the glass window, combined with the tightens of the extrusion/injection molding molten polymer flow.

As mentioned before the ability to perform the flow velocity measurements in a touchless mode, it means without introducing the sensors into the streaming medium, is the

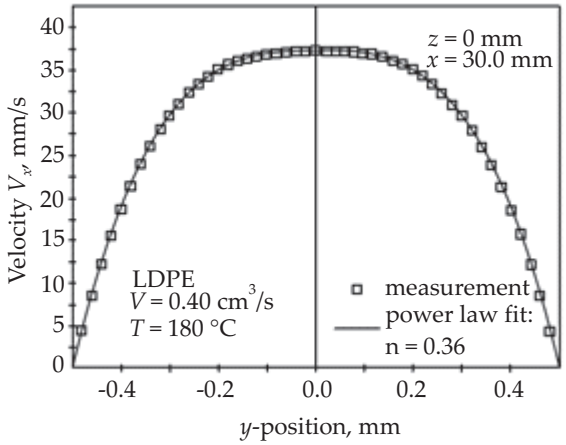

Fig. 5. The LDPE flow velocity distribution on the cross section of a die; comparison of LDV measurements with power low fit for n = 0.36; the measurements were realized across the section of a die at the positon of 30 mm after the entry from the reservoir to the die [39 similar results at 30–32]

main advantage of LDV. Another appreciated factors are the very small dimensions of the measurement volume, relatively to the cross section of the streaming medium. On this way relatively easy the flow velocity distribution on the cross section of the channel/die, may be measured and established [31]. This, in turn allows to measure the flow at the positions even very close to the die wall, where a significant shearing in the streaming molten polymer occurs (Fig. 5). On Fig. 5 the flow velocity on the cross section of the die is presented, at the position of 30 mm after the entry to the die from the reservoir. In this case a fully developed flow is characterized by relatively wide core section without shearing, and the near wall part where the shearing of the molten polymer is predominant.

By the LDV measurements the velocity vector is determined at the surface normal to the optical axis of the LDV system, thus, adjusting the LDV system also the velocity vectors in the direction perpendicular to the flow axis may be measured. Such determination of the velocity vectors in the radial directions (secondary flow) may particularly be important, when the flow reaches the high Reynolds values, what is usually not the case by molten polymers streaming.

THE MICROPLATE-LIKE ADDITIVES AS FLOW MARKERS

The usage of microplate-like additives, introduced into the molten polymer, serving as *flow lines markers* presents another relatively simple method employed to determine the flow of molten polymers. Talc and/or mica are the mostly used additives in this case. The first experimental stage by this technique is the preparation of composites of investigated thermoplastic material charged with a defined quantity of plate-like additives. On Fig. 6 the talc particles with a mean particle diameter of 3 μm, are

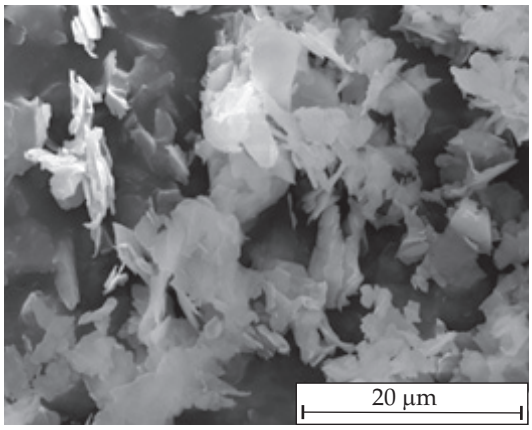

Fig. 6. SEM image of talc particles Naintzsch A10 (from Luzenac) [41]

presented [41]. Such method was already used by [42, 43] and various extrusion/injection molding processing conditions may be investigated by means of this technique. The flow lines estimation is performed by microscopic observations of the cross section of the extruded/injection molded products.

A comparable way for multiple cavity filling visualization procedure was chosen by Bociąga *et al*. [44, 45]. In these investigations also the talc-filled low density polyethylene was used for the flow lines visualization, in an injection mold with multiple cavities. The microscopic observations were performed on the cross-section of the runners and of the mold cavity using slices cut perpendicular to the flow direction. The observations were realized by means of optical microscopy with polarized transmitted light, and the allocation of the talc plates was analyzed by scanning electron microscopy (SEM). It was stated that the molten polymer does not fill all mold cavities at the same time, what probably may be due to changes of the direction of polymer flow in runners. According to previous experiments [41], we have found that 10 wt % is the best concentration of the plate-form markers, and mica as the marker is less convinced for such investigation, as the mica additives are hardly visible at the cross section of the polymeric products.

An observation of a characteristic molten polymer "fountain flow" during mold cavity filling, was published before [41]. It was shown that various forms and dimensional relations of the flow lines distribution exist, where independent on the filling conditions, a "fountain flow" effect was confirmed. Depending on the cavity thickness, a clear distinction of at least three flow lines orientation regions, on the cross section of the product, were observed.

At the skin layer, near the wall part of the flow, a direction oriented alignment of makers may prove a significant shear flow of molten polymer, existing in this channel zone. At the core of the flow, a perpendicular position of the markers, relatively to the filling flow direction, signify a "fountain flow" at the middle axis of the cavity (Fig. 7). At the region between the core and the near wall

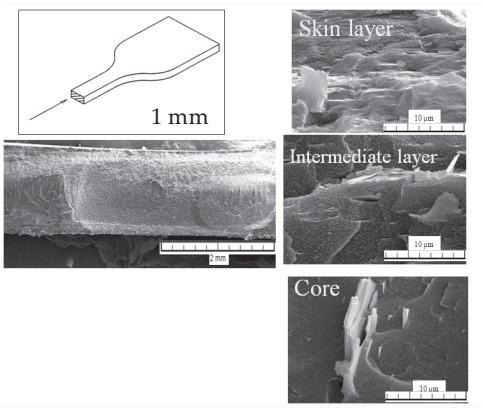

Fig. 7. SEM images of cross sections of a 1 mm thick injection molded part made of LDPE 10 wt % of talc [41]

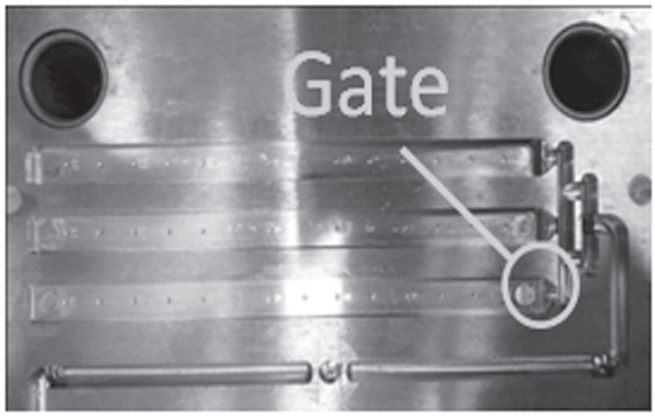

Fig. 8. Top view of injection mold cavity with localization of observed inlet gate with thickness of 1 mm

part, an intermediate alignment of the markers is well visible, an effect which approve a filling of the cavity by a fountain-like flow [41, 43–45].

In our previous investigations [46], the composites with the markers were considered to study the flow lines, and consequently the macromolecules positon by filling a mold cavity with metal inserts. In the view of growing production of the light constructions, used in automotive, aircraft and other industrial applications, such injection molding products, shaped with a strong adhesive bonding of metallic with polymeric parts, are gaining an importance today. Special requirements by growing technical applications of light-construction is the reason of the importance of new developments by such metal-polymer connections, achieved without any additional mechanical coupling, nor by the use of any technical glue.

MARKERS TYPE POLYPROPYLENE FLOW DETERMINATION AT THE CAVITY ENTRANCE

The polypropylene HP 500N (Basell Orlen Polyolefins, Plock, Poland, *MFR* = 12 g/10 min) was applied as matrix of the composites. Commercially available talc extra 10 (purchased from Aurum Chemicals, Katowice, Poland) was used as flow markers. The composites of PP with talc were produced by extrusion, using a ZAMAK twin-screw extruder, operating at the temperatures between 165 °C and 205 °C along the barrel, by screw rotation speed of 150 rpm.

The injection molding was realized by means of Engel HLS 80/20 injection molding machine, with the barrel temperature between 185 °C and 220 °C, operating by following processing parameters: hydraulic filling pressure 10 MPa, hydraulic packing pressure 7 MPa, packing time 4 s and cooling time 25 s. The polymer/metal connection parts were produced using a mold presented on Fig. 8.

On the microscopic observations of the cross section of injection molded part, the segment of the mold inlet may be seen (Fig. 9). The observed area is focused on the place where the cross section of the flow abruptly increases, gaining a double thickness comparing to the channel, causing a local disorientation of the polymer flow arrangement. In this particular area the flow lines of the melted polymer with plate-like particles may be seen. This area is particularly of interest because of well seen polymer flow lines with easily detected talc particles allocation. The brittle behavior of sample fracture allows us to localize the side view of the narrow edge on the top surface of platelet particles, as visible markers of macromolecules flow path during channel/cavity filling. Once again talc – as a flow marker was successfully used for quantification of orientation/disorientation of polymer structure in micro and macro-scale observation.

As it follows from Fig. 9, a significant change of the flow induced orientation of the marker, therefore of the macromolecules by passing the sharp edge of the cavity inlet, may be clearly observed. The primary flow direction alignment of the markers is strongly influenced by an extension of the flow cross section (Fig. 9 b) leading to an almost perpendicular flow regarding the main filling direction. Shortly after, an alignment of markers again in the main flow direction (Fig. 9 c) results in significant macromolecular orientation, as usually observed [46] in the case of alongside filled cavities.

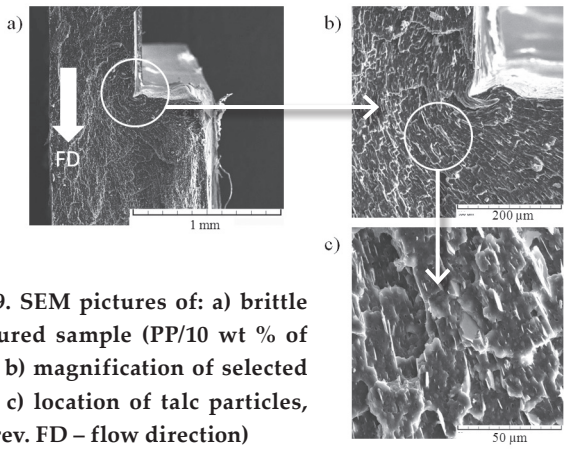

Fig. 9. SEM pictures of: a) brittle fractured sample (PP/10 wt % of talc), b) magnification of selected area, c) location of talc particles, (abbrev. FD – flow direction)

CONCLUSIONS

We have presented several methods of observation of mold cavity filling by thermoplastic polymers. Such investigations are now-a-days very important due to significant influence of orientation on the products properties. This dependence is particularly worth in a case if isotropy and/or anisotropy may be decisive by polymeric products applications.

Few methods of flow visualization have been presented and briefly discussed in this paper. The direct flow detection, what is the case by laser Doppler velocimetry, provides exact values of the velocity vectors at channels, cavities *etc*., thus may be very profitable for the designing of molten polymer flow, as well as may be applied for the verification of rheological models. From this point of view the LDV presents a very important technique, currently used for the designing of turbines, flow meters *etc*. The main difficult although is the high cost of such equipment and a long time necessary to establish the measurement system.

The microscopic observation of the cross section of polymeric products filled with flow markers, presents on the contrary a very simple, low cost and multipurpose universal method, delivering the information's about the flow lines distribution on the section of the streaming polymer. Such information's are in many cases sufficient for the validation of the mold cavity filling simulations, and/or prediction of the local positioning of the macromolecular structure, thus giving data's of possible structure/properties anisotropy.

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