Rheological Properties of Elastomer-Modified Polypropylene and Their Influence on the Formation of Flow Marks

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ABSTRACT: A premium appearance is essential for many polymeric products and for this reason surface defects associated with the manufacturing process, e.g. injection molding, are detrimental. In this study, the interest is focussed on defects arising during injection molding of such elastomer-modified polymers that are often used in the automotive sector to produce interior and exterior components. In particular, defects denoted as "flow marks" or "tiger stripes" were investigated. These defects appear on the surface of the injection-molded components, especially if long flow lengths are involved and consist of alternating glossy and dull bands. Here an attempt was made to elucidate to what extent some important rheological properties of the polymer melts influence the generation of such flow marks. Hence, the flow properties, mainly in shear; of three different grades of elastomer-modified polypropylene containing mineral fillers were correlated to their propensity for defect generation. In summary, it was noted that a higher melt elasticity, as reflected in pressure losses during flow through a capillary, the degree of die swell and to some extent the dynamic-mechanical behavior, leads to less severe flow marks or retards the formation of such defects. Elongational draw-down experiments also indicated a more stable flow in the case of the melt that exhibited the highest elasticity. Furthermore, subjecting the measured flow behavior to a Mooney analysis, the results obtained pointed to that wall-slip in itself is not a primary cause for the appearance of the type of flow marks studied here.
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Key words: flow marks; injection molding; polymer processing; rheological properties; surface defects; capillary rheometer

INTRODUCTION

Besides fulfilling requirements on functional properties, such as stiffness, strength, thermal resistance etc, polymeric components should have an aesthetically appealing appearance. This is important as the appearance has a direct bearing on the perceived quality impression. Colors and gloss levels should be carefully matched and should be uniform to ascertain the desired perceived quality, cf. Ref. 1. A premium appearance is of vital importance for many plastic products, not least within the automotive sector and applies to interior as well as exterior parts (like bumpers). Preferably, the required appearance should be achieved without an additional painting or any other surface treatment, as this adds to the complexity and cost of the manufacturing.

Recognizing the importance of the appearance, surface defects associated with the manufacturing, e.g. injection molding, of the components are clearly detrimental in this respect. Although, painting is possible way to reduce the visibility of such defects, it is should preferably be avoided and the defects should in the ideal case be eliminated by through a proper design of the manufacturing process combined with a careful material selection. In the case of injection molding, several types of defects can occur such as flow marks, sink marks, distortions, weld lines, hot spots etc, see for example.[2] In this study, the interest is focussed on a surface defect, often denoted as "flow marks" or "tiger stripes".[3–6] Such defects are visible as alternating glossy and dull bands on the surface of the injection-molded component, especially if long flow lengths in thin-walled molds are involved. A car bumper would be a typical example of such a product. The flow marks appear normally perpendicular to the flow direction and are often opposite in phase, which means that a glossy band on one side of the component corresponds to a dull band on the opposite side.

Most studies on flow marks on injection-molded compounds involve rubber-modified polymers or

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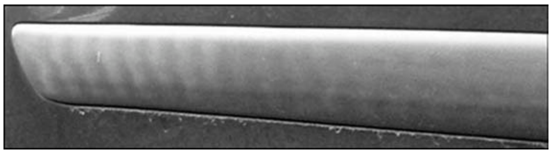

Figure 1 Detail of a car bumper showing flow marks.

polymer blends,[5,7,8] but the problem is encountered also with neat polymers.[9] Polypropylene (PP), containing elastomeric particles, is a typical material that typically has been used in several studies, to some extent this is related to its use in the automotive industry. A number of possible mechanisms underlying the appearance of the flow marks has been put forward, but the situation cannot be said to have been completely clarified, cf., Refs. 2, 10 and 11. Most approaches to explain the phenomenon appear to rely on a flow instability resulting in an unstable, "snake-like"[8] motion of the flow front, i. e. a disturbance of the fountain flow,[7,10–13] whereas others suggest a flow instability induced by a stick-slip flow at the wall of the mold[5,14] or a similar course of events.[9] The aim of this work is not to assess which reason is the most probable one; it suffices to assume that it is related to a flow instability. Figure 1 is a photograph of the flow marks on the surface of an injection-molded automobile component. The alternating glossy and dull bands are clearly visible. The glossy regions appear darker and the dull areas lighter.

For obvious reasons, the number and severity of these tiger stripes should be reduced or, preferably, such defects should be eliminated. It is known that the processing conditions during the molding are of importance in this context. For example, the tendencies for defects formation is reduced by an increased mold and/or melt temperature.[2,7] Furthermore, it has been reported that the problem is enhanced when increasing the injection rate.[5,7,13] Of course, it is expected that geometry of the mold and the location of the gates are of importance here, since those govern the flow during the mold filling. A serious and known problem encountered with flow marks is that their visibility can be enhanced during aging, i.e., as a result of exposure to ultraviolet light, temperature changes and outdoor conditions.[15,16]

In addition to the processing conditions, the composition of the polymer melt and its rheological behavior will influence the formation and the appearance of the flow marks. This will be discussed in more detail in the following. Here it might suffice to mention that some studies indicate that a high elasticity of the polymer melt would suppress the formation and the severity of the flow marks, cf., Refs. 12 and 13. This opens up for reducing the flow mark problem through modifications of the molecular mass distribution and using adequate additives.[3,12] There are also indications that a more pronounced extensional strain-hardening of the melts could lead to improvements in this respect.[4]

This study aims at highlighting the importance of some rheological properties of the polymer melts with regard to the extent of flow mark generation. Three different grades of elastomer-modified polypropylene containing mineral fillers were used. In production (using injection molding), two of them were noted to be prone to formation of the flow marks, whereas when using the third grade, the problem was significantly less pronounced. The flow properties of these three materials were evaluated in terms of viscous and elastic characteristics, wall-slip behavior and extensibility and hence correlated with their propensity for the defect generation.

EXPERIMENTAL

Materials

Three polypropylene (PP) grades used for injection molding of automotive components (both exterior and interior) were included in this study. They contained talc as filler and the talc content was typically 10–15 wt %. No detailed information on the content of elastomeric particles was available, but two of them were elastomer-modified and the amount of elastomeric particles was expected to be rather low. These three grades will be denoted A, B, and C in the following. The density of grade A was 1030 kg/$m^3$and its melt flow index (230°C/2.16 kg) 11 g/10 min. Grade B had a density of 990 kg/m^3 and a melt flow index (230°C/2.16 kg) of 16 g/10 min. The corresponding data for grade C were 1000 kg/m^3 and 18 g/10 min, respectively. When injection molding, components of grades A and B often exhibited clear flow marks (tiger stripes) whereas the problem was significantly reduced when employing grade C. This is exemplified by the photographs shown in Figure 2 in case of materials B and C. Material B exhibits clear flow marks whereas material C gives a quite homogeneous impression.

Experimental techniques

A Göttfert Rheograph 2002 (Germany) capillary viscometer was used to evaluate the rheological properties (in shear) of the PP-materials at a temperature of 190°C. Two sets of capillaries, one set having a length of 10 mm and the other set a length of 20 mm, were chosen. Each set included three different capillaries, with a flat entrance region, having a diameters of 0.5, 1, or 2 mm. The shear rates covered in the experiments were from 30 to 6500 s^{-1}. All experimental data were subjected to both the

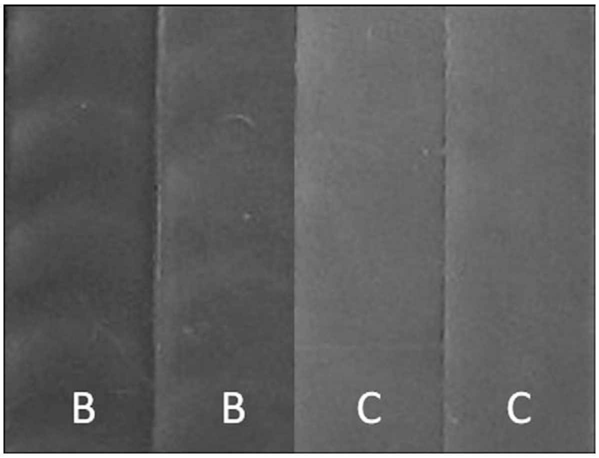

Figure 2 Photographs of injection-molded specimens (bars) of materials B and C. The specimens produced with material B exhibited clear flow marks.

Rabinowitsch correction[17] and the Bagley correction,[18] see also below.

Mooney analysis[19] of the rheological results was performed to establish whether or not a stick-slip flow took place and to evaluate the slip velocity during the flow. When dealing with nonNewtonian fluids, surface slip of the flowing melt (close to e. g. the wall of a channel) can occur and result in abnormally large velocity gradients in a thin layer adjacent to the wall.[20] The measured (average) fluid velocity v is the sum of the true velocity v_{true} and the slip velocity v_{slip}.

$$v_{\text{true}} = v - v_{\text{slip}} \tag{1}$$

where

$$v = \frac{Q}{\pi R^2} \tag{2}$$

here Q is the volume flow rate and R the radius of a cylindrical channel.

The apparent shear rate ($\dot{\gamma}$) at the wall of the channel can be expressed as

$$\dot{\gamma} = \frac{4Q}{\pi R^3} = \frac{4v}{R} \tag{3}$$

In case of a power-law fluid and rearranging eq. (1), the following relation is then obtained, cf., Ref. 21:

$$\frac{4Q}{\pi R^3} = \frac{4n}{3n+1}\left(\frac{\tau_w}{K}\right)^{1/n} + \frac{4v_{slip}}{R} \tag{4}$$

where τ is the shear stress at the wall of the channel, n the power-law exponent of the fluid and K the consistency in the power-law relation. For a constant wall shear stress, the slip velocity now can be obtained from the slope of the graph $\dot{\gamma}$ vs $1/R$. When determining the slip velocity using capillary viscometry, this requires the use of several capillaries of the same length, but with different diameters.

The extrudates were photographed with a digital camera, Pentax K200D, as they left the capillary exit to assess any signs of distortions or instabilities such as shark skin etc. These photos were also used to determine the degree of die swell and here the diameter of the extrudates was measured approximately 1.5 cm below the exit of the capillary.

A Rheometrics SR200 dynamic stress rheometer (USA), with a parallel plate configuration, was used to measure the dynamic-mechanical properties (storage modulus G', loss modulus G'', and mechanical loss factor tan δ) of the PP-materials. A stress sweep was performed in the stress range 200 to 500 Pa at a constant temperature of 190°C and with a frequency of 1 Hz.

The melting points (T_m) of the PP-grades were evaluated using differential scanning calorimetry (DSC7, PerkinElmer, USA equipped with an intracooler). Each material was first heated from 20 to 240°C at a heating rate of 10°C/min, then cooled at the same rate down to 20°C and finally reheated to 240°C, again using the same heating rate. The melting points of the different grades were determined using the second heating cycle (average values of two separate experiments).

The elongational properties of the melts at 190°C were evaluated using a capillary viscometer (CEAST Rheoscope, Italy) equipped with a "rheotens"-unit, i.e. a rotating disc that picked up the extruded fiber. The rotational velocity increased linearly and the velocity at which the fiber broke was determined. The draw-down ability (or the ultimate draw-down ratio) of the material was taken as the ratio between the tangential velocity of the disc at break and the velocity of the fiber exiting from the capillary. The capillary used had a diameter of 1 mm and a length of 10 mm. The acceleration of the disc was kept at 10 rpm/s and the velocity of the fiber when exiting the capillary die was 7.6 mm/s.

RESULTS AND DISCUSSION

Thermal analysis

Differential scanning calorimetry (DSC) was used to evaluate the melting points (T_m) of the three PP-grades A, B, and C. As previously described in the experimental section, a heating-cooling-heating cycle was performed for each material between 20 and 240°C. Figure 3 shows the endotherms for the three materials and Table I gives the corresponding melting points obtained from the second heating cycle.

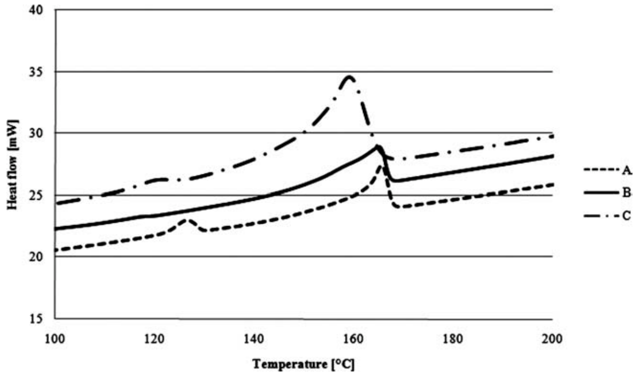

Figure 3 DSC endotherms for materials A, B, and C obtained from the second heating cycle.

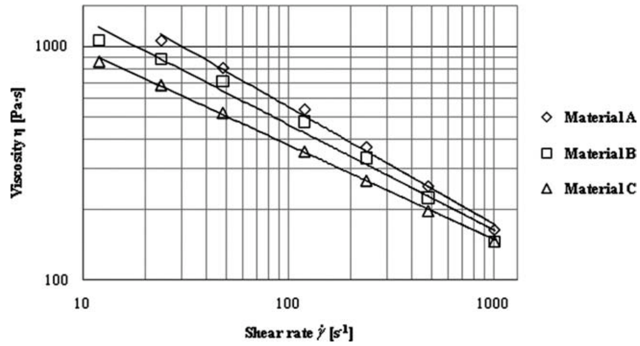

Figure 4 Viscosity versus shear rate at 190°C for materials A, B, and C. The viscosity curves were obtained using a capillary with the length 10 mm and the diameter 2 mm.

Grade C obviously exhibited a somewhat lower melting point than the other two materials. Note that in case of material A (and to some extent also for material C), a second peak, appeared in the range 125 to 130°C, which could indicate the presence of ethylene sequences.

TABLE I
Melting Points (T_m) for the Three Materials Used

Material	T_m (°C)
A	166
B	165
C	159

Rheological properties

Figure 4 shows the viscosity as a function of the shear rate at 190°C for the three melts. The curves were obtained using a capillary with a length of 20 mm and a diameter of 2 mm. No corrections were applied (to be discussed later) and these curves just give an overall view of the flow behavior. All three materials exhibited a similar level of the viscosity over the used shear rate region; with material A having a somewhat higher viscosity and material C the lowest. A similar behavior was observed when using capillaries of other dimensions. The viscosity levels as such do not appear to govern the formation of flow marks since it has been reported that PP-based polymers can exhibit a similar viscosity-shear rate dependence but differ markedly with regard to the intensity of the flow mark generation.[11]

Rabinowitsch correction

The melts obviously were shear-thinning and displayed a power-law behavior. In the following, the shear rate values were corrected using the conventional Rabinowitsch correction; i.e.:

$$\dot{\gamma}_t = \dot{\gamma}\left[\frac{1}{4}\left(3 + \frac{d\log\dot{\gamma}}{d\log\tau}\right)\right] \tag{5}$$

where $\dot{\gamma}_t$ is the true shear rate (at the wall of the capillary), $\dot{\gamma}$ the apparent shear rate, τ the shear stress (at the wall) and the quantity $d\log(\dot{\gamma})/d\log(\tau)$ is given by the slope of the straight line obtained when plotting $\log(\dot{\gamma})$ versus $\log(\tau)$. The apparent shear rate is given by eq. (3).

Bagley correction

As is well known, the shear stress to be determined from capillary viscometry must be corrected for inlet and outlet pressure losses during the flow through the capillary. This is known as the Bagley correction and these losses can be related to the elastic characteristics of the melt.[13] The procedure for evaluating the pressure losses is described in several textbooks, e.g.,[22] but briefly it involves measurements using capillaries of same diameter but having different lengths. The applied pressure is then plotted as a function of *L/R*, where *L* is the length of the capillary, at a constant shear rate. This is done for several shear rates. Extrapolating the lines to $L/R = 0$, the pressure losses are given as the scission with the pressure axis.

Figure 5 shows the pressure losses, in case of the materials used here, as a function of the true shear rate. In this case, a capillary set with a diameter of 1 mm was used but similar results were obtained also with the other capillaries. It is of interest to note that the pressure losses were significantly higher for material C than for the other two melts. This material is also the one that gave the least severe flow marks when injection-molded. This indicates that a more elastic melt should be beneficial with regard to the suppression of the flow defects. This is also supported by the results reported by Dharia.[13]

Figure 6 shows the viscosity as a function of the shear rate for material C. In this case, the shear rate

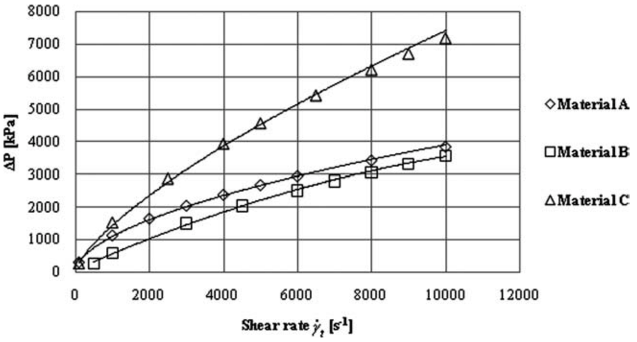

Figure 5 The pressure losses (ΔP) as a function of the true shear rate for the three materials investigated. The curves were obtained with a capillary of 1 mm in diameter.

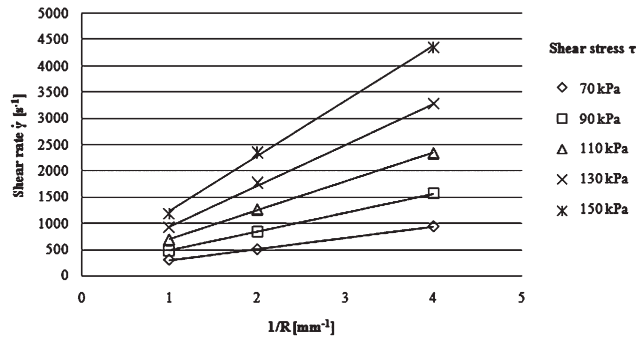

Figure 7 The apparent shear rate as a function of $1/R$ at different wall shear stresses for material C (Mooney plot) using a capillary length of 20 mm.

and the shear stress have been subjected to the Rabinowitsch and the Bagley corrections. It is however obvious that the viscosity curves depend somewhat on the diameter of the capillaries which might point to wall slippage during the measurements. To estimate the degree of wall-slip, the Mooney approach was employed here. The results shown in Figure 6 were obtained with a capillary length of 20 mm, but a similar behavior was noted also when the shorter capillaries were used.

Estimation of the slip velocity

The Money approach was used to estimate the wall-slip velocity, i.e. eq. (4). Figure 7 shows the apparent shear rate at different constant wall shear stresses as a function of $1/R$ in case of material C. The slip velocity was evaluated from the slope of the straight lines as described earlier. In this case the capillary length was 20 mm, but a similar pattern was also noted with the shorter capillaries.

Figure 8 summarizes the results of the slip velocity measurements for the three materials when using a capillary length of 10 mm (similar trends were obtained with the longer capillaries as well). Here, the slip velocity is shown as function of the wall shear stress. At low shear stresses, the slip velocity was low, or close to zero, and then increased as the shear stress increased. Materials A and B exhibited, throughout the shear stress region used, a considerably lower slip velocity than material C.

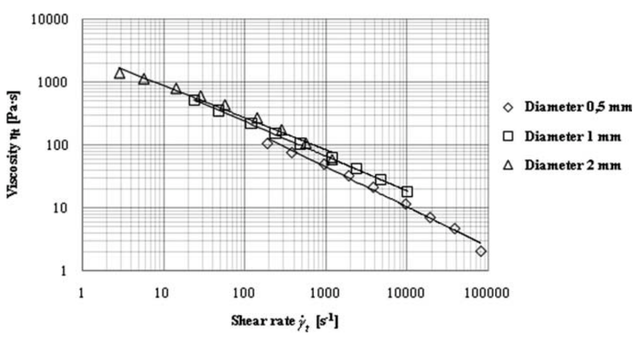

Figure 6 The viscosity versus the shear rate for material C after applying the Rabinowitsch and Bagley corrections. The viscosity curves were obtained using capillaries of different diameters, but with a constant length of 20 mm.

The degree of die swell

During the viscosity measurements, photos of the three investigated materials exiting the capillary were taken at different shear rates using different capillaries. The aim was to estimate the degree of die swell and assess any possible indication of flow instabilities, e.g. revealing itself in the form of shark skin on the surface of the extrudate. Two different plunger speeds (corresponding to different shear rates) were used in this study. The degree of die swell was taken as the ratio between the actual diameter of the extrudate (in a steady state) and the diameter of the capillary. Representative examples of the evaluated die swell are given in Table II below.

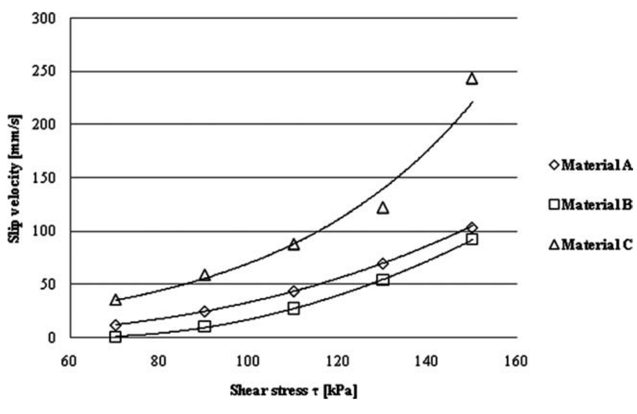

Figure 8 The slip velocity, as obtained from the Mooney analysis, versus the wall shear stress for the three PP-grades. The length of the capillaries used was 10 mm.

TABLE II
The Degree of Die Swell of the PP-Melts after Extrusion Through the Capillaries (about 1.5 Cm Below the Capillary Exit)

Apparent shear rate (s^{-1})	Capillary length and diameter (mm)	A	B	C
1500	10 and 0.5	1.25–1.3	No swelling	1.35–1.5
7700	10 and 0.5	–	1.6	1.8–2
190	20 and 1	1.2	No swelling	1.4–1.6
960	20 and 1	–	1.3	1.9

The obtained results clearly indicate that the die swell was more pronounced for material C, which was the polymer grade that was less prone to formation of flow marks during injection molding. This appears to be in line with the observations made by Chang.[14]

Only at the highest used shear rates, the extrudates showed some signs of shark skin and traces of visual flow instabilities. No significant differences between the materials could be said to exist in this respect.

Dynamic-mechanical properties

The dynamic-mechanical properties of the melts in shear oscillation were determined within the linear viscoelastic region. At a shear strain of 0.02 and at 190°C, the mechanical loss factors tan δ for materials A, B, and C were 1.58, 2.26, and 1.38, respectively. Again this points to that a more elastic character (lower loss factor) of the melt corresponds less severe flow marks. This is to some extent in agreement with the proposal by Brodil and Sehanobish[12] that a lower loss factor contributes to less defect generation. Furthermore it was noted that the ratio $G'/(G'')^2$ always was significantly higher in the case of material C. At low frequencies, a higher ratio can indicate a more pronounced elastic deformation, cf., Ref. 23.

Draw-down ability of the melts

The elongational behavior of the three PP-grades was assessed in terms of their ultimate draw-down ability. Material C exhibited quite a stable behavior with an average ultimate draw-down ratio of about 330. The other two melts (A and B) on the other hand displayed an instable behavior in the sense that the ultimate draw-down ratio varied considerably between different experimental trials; between 240 and 740.

Final remarks and conclusions

Most studies on flow marks or tiger stripes relate, as mentioned earlier, these defects to some kind of flow instabilities. In many cases, these flow disturbances are attributed to flow front instabilities,[7,8,10–13] but a slip-stick behavior may also be responsible[5,14] and there may also be other causes.[9] In reality, a slip-stick phenomenon can also result in a snake-like motion of the flow front. The reasons for the appearance of the flow marks are hence quite complex and there might not be one single cause; i.e. a combinations of different circumstances might produce this defect. In any case, a uniform and stable flow is considered to counteract the occurrence of the flow marks.

Several reports indicate that a higher melt elasticity can reduce the severity/appearance of the tiger stripes.[3,11–14] The melt elasticity was described or assessed in several different ways such as die-swell, pressure losses, normal stress differences, recoverable shear strain, etc. Brodil and Sehanodish,[12] examining thermoplastic polyolefins (TPO), showed that the rubber particles were elongated in the glossy region and were contracted in the dull ones. A similar observation was also reported by Patham et al.[11] A corresponding difference in molecular orientation is also expected for a neat polymer. In Ref. 12, it is argued that a fast relaxation of the rubber particles from its elongated shape, reducing the severity of the flow marks, is favored by decrease of the mechanical loss factor tan δ, i.e., by an increase of the elasticity of the melt. However, it is also pointed out that a lower tan δ of the system will result in more stable flow front, which can somewhat simplified be associated with a less flexible flow front. Chang,[14] who concentrated on the wall-slip hypothesis, noted that a higher die swell of the material, i.e., a higher melt elasticity, reduced the problem with the flow marks. Using materials containing high molecular mass fractions gave less apparent flow marks,[12,14] whereas adding fillers to the polymer reduced the die swell and consequently the severity of the flow defects increased.[14] The results obtained within this work appear to be in line with the above observations; a higher melt elasticity, as reflected in the pressure loss, die swell measurements and to some extent the dynamic-mechanical behavior, leads to less flow marks. Due to the higher elasticity, the higher normal forces, and the less flexible flow front would stabilize the flow in the channel. Furthermore, the draw-down experiments indicate a more stable extrudate (or flow) in the case of material C which exhibited the highest elasticity.

In the ideal case, the most straightforward way to circumvent the flow mark problem would be to ascertain that the flow front moved in a uniform and stationary manner. To some extent this may be achieved by adjusting the processing conditions or by choosing a polymer with adequate melt properties. However, considering the complexity of injection-molded components and the use of multigate cavities, this might in many cases not suffice. Now, if flow instabilities are at hand, the effects of them should be minimized. Assuming that orientation (on a molecular level or elongation of rubber particles) plays a major role for the appearance of the flow marks, relaxation of the deformed entities should provide a possible route. The usefulness of such an approach was, as indicated earlier, shown by Brodil and Sehanodish.[12] The importance of short relaxation times was also emphasized by Dharia[13] who mainly attributed the formation of the flow marks to a built-in stress difference between the skin layer of the components and its core region. A fast relaxation of this stress difference would reduce the severity of the defects. However, as noted by Patham et al.,[11] the relaxation characteristics must be carefully monitored. They observed that a too fast retardation of the elongated ethylene–propylene rubber particles in a PP-matrix, actually could enhance the difference in rubber deformation between the glossy and the dull regions, thus enhancing the flow marks.

A rather striking result of the present study is that illustrated by Figure 8. The Mooney analysis indicated that material C exhibited the highest slip velocity of the PP-grades. This was also the grade that was less prone to flow mark formation. Assuming that the results are also applicable to the injection molding case, this would point to that slip in itself is not a primary cause for the appearance of the type of flow marks studied here.

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