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TECHNICAL PAPER

Replication quality of micro structures in injection moulded thin wall parts using rapid tooling moulds

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Abstract Injection moulding of micro structured polymer parts is often limited due to the replication quality of the structured surfaces. To enhance the replication quality process parameters, e.g., pressure, temperature or injection velocity, are adapted. Here, the mould temperature is the most important factor. This paper investigates the influence of the mould temperature on the replication of micro structured surfaces using amorphous and semi-crystalline polymers. Using rapid tooling moulds and a dynamic tempering system allows mould temperatures about the solidification temperatures during injection and a sufficient cooling for save ejection of the part. The results reveal that for amorphous polymers the mould temperature should be above the glass transition temperature for high replication quality. For semi-crystalline polymers the high cooling velocity seems to inhibit the crystallization process and this leads to a sufficiently low viscosity to achieve high replication quality.

1 Introduction

A reduction of part dimensions causes an increasing cooling that affects the process and filling behaviour and also the replication quality of a micro part. In a conventional injection moulding process the mould surface temperature is far below the melt temperature. This leads to a high cooling velocity and results in a frozen layer close to the mould surface (Gornik 2004; Tom et al. 2006; Sha et al. 2007; Zhan and Lu 2008; Nguyen-Chung et al. 2011). Eder and Janeschitz-Kriegl (1997) found that the crystallization temperature decreases with increasing cooling rates, thus for a PP the crystallization temperature would be 0 °C when the cooling rate is about 270 K min^{-1} (Stern et al. 2005). In addition, the viscosity increases too which affects the filling behaviour negatively and thus the replication of micro-structured surfaces (Giboz et al. 2007; Meister and Drummer 2013a). To counteract this effect, different strategies were developed and investigated to modify and optimize the process parameters. An increasing injection velocity can also favour the transcription of surface structures in the mould (Yokoi et al. 2006; Attia et al. 2009; Kayano et al. 2011). Also an increasing pressure (Karl 1979; Moneke 2001; Attia and Alcock 2009; Rudolph et al. 2011) or a high shear rate (Stern et al. 2005; Janeschitz-Kriegl and Ratajski 2005; Zhu et al. 2006) can affect the crystallization process of the polymer melt: a higher pressure shifts the crystallization temperature to a higher value, whereas a higher shear rate favours the nucleation and thus the crystallization of the material. Zhu et al. (2006) stated that in the shear region the chain tend to be locked or frozen like a quasi-quenching. Notwithstanding, the most important process parameters that are discussed to influence the cavity filling are the temperatures of the mould and the melt, whereas the mould temperature appears to be the key parameter (Martyn et al. 2004; Giboz et al. 2007; Sha et al. 2007; Bekesi et al. 2010; Tosello et al. 2010; Meister and Drummer 2013b): in general, with increasing mould or melt temperature the filling behaviour is favoured and an increasing aspect ratio can be reached. Kim and Kim (2014) revealed that the temperature is the most important factor to influence the replication of micro structures due to the impact on the melt viscosity. In addition, using thermal low conductive

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mould materials (Schmiederer and Schmachtenberg 2006; Lurz et al. 2008) or a dynamic temperature control of the cavity (Walter et al. 1999; Giessauf et al. 2008; Drummer et al. 2011; Chen et al. 2013; Xie et al. 2013) can influence the cooling velocity of the melt.

2 Experimental

2.1 Materials

In the investigations different thermoplastic polymers were used: a polypropylene homopolymer (PP, 505P, Sabic Europe) as semicrystalline polymers and an amorphous polycarbonate (PC, Makrolon OD2015, Bayer MaterialScience AG) were investigated. Characteristic values of these materials are shown in Table 1.

These materials are used due to their solidification temperature (glass transition or crystallization temperature).

Table 1 Characteristics of the investigated materials

Parameter	Standard	PP	PC
Density[a] ρ (kg m^{-3})	ISO 1183	905	1,190
Melting temperature (10 K s^{-1}) (°C)	ISO 11357	159	–
Crystallization temperature (10 K s^{-1}) (°C)	ISO 11357	116	–
Glass transition temperature (10 K s^{-1}) (°C)	ISO 11357	15	144
Thermal conductivity[a] k (W m^{-1} K^{-1})	ISO 8302	0.22	0.2
Specific heat capacity[a] c_p (J kg^{-1} K^{-1})	ISO 11357	1,700	1,170

[a] Manufacturer's data

This allows for a mould temperature below and also above the solidification temperature.

Furthermore, the materials show a different shear thinning behaviour, Fig. 1. However, the PP material reveals already at lower shear rates (10^1 s^{-1}) a reduction of the viscosity, whereas the PC reveals a shear thinning at a shear rate of (10^3 s^{-1}). In addition, the shear thinning of the PP is more evident. The viscosity curves show also for the PC a more distinct dependence of the temperature as for the PP material. The difference of the viscous behaviour of the materials is more evident in a linear plot Fig. 1 (right).

2.2 Mould and specimen

The mould consists of a master mould and rapid tooling inserts: the master mould and rapid tooling inserts with the cavity. These cavity inserts are built up layer by layer from a steel powder using the rapid tooling process LaserCusing which was developed by Concept Laser GmbH. This manufacturing process allows for a complex design of cooling channels whereby an optimized tempering of the cavity can be ensured. The combination of insulation from the master mould and conformal cooling channels are conductive to particularly rapid temperature changes in the cavity. The micro-structured mould insert is shown Fig. 2, left. Figure 2 (right) demonstrates exemplarily the rapid tooling mould with cavity near cooling channels.

The injection moulded specimen is a thin wall plate with a thickness of 0.5 mm and a square-based shape with a length of 35 mm. On one side the plate is micro structured with two alternating types of a lamellar microstructure, Fig. 3.

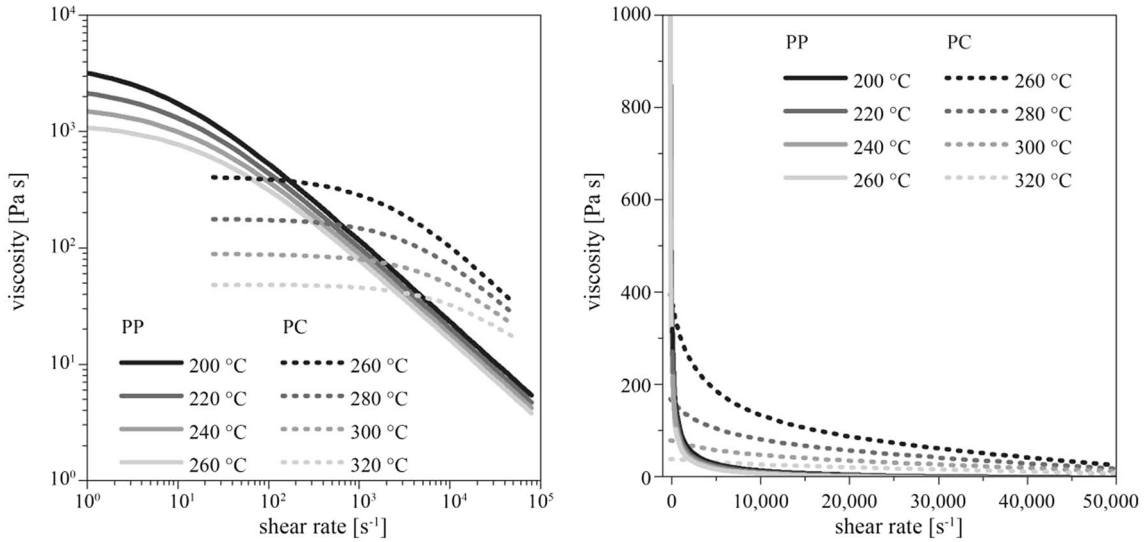

Fig. 1 Viscosity curves (*left* plotted logarithmically, *right* potted linearly) of the used materials (ISO 11443, manufacturer's data)

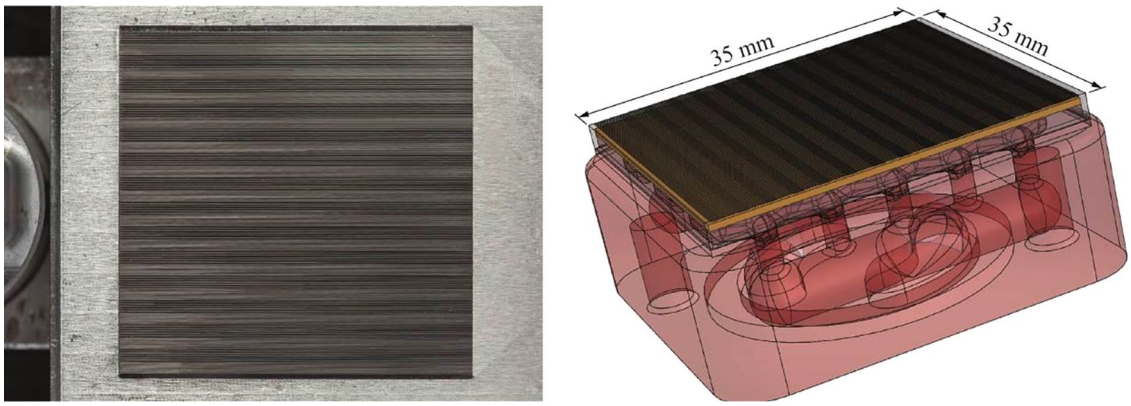

Fig. 2 Rapid Tooling mould insert with lamellar microstructures (*left* picture of the assembled insert, *right* CAD drawing of the insert with illustration of the cavity near cooling channel geometry)

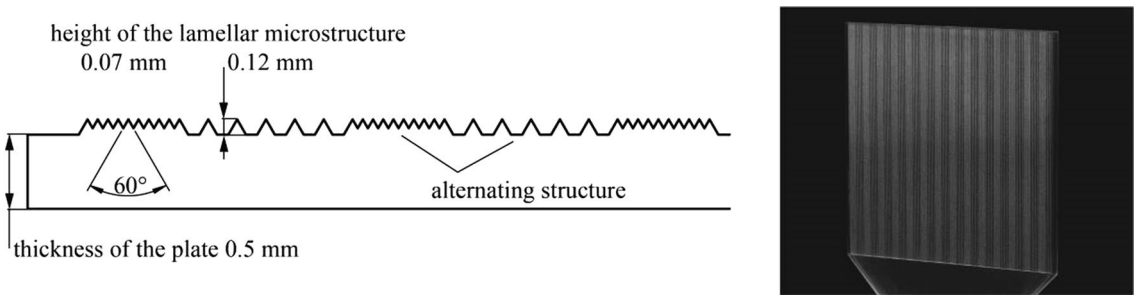

Fig. 3 Lamellar structured thin plate (*left* dimensions of structures and *right* injection moulded specimen)

2.3 Processing

For injection moulding an Arburg Allrounder 370U 700–30/30 injection moulding machine was utilized, equipped with a position controlled screw with a diameter of 15 mm. Relevant process parameters shows Table 2. To vary the mould temperature a variothermal process was realized. For tempering the cavity inserts a variothermal temperature control system (type: SWTS 200, Single Temperiertechnik GmbH) was used. The system employs water as the circulating fluid and has a heating and a cooling circuit-switching device. It allows a fluid temperature up to 200 °C. The mould is maintained at a constant temperature for the purpose of process stability, and only the temperature of cavity inserts is actively controlled. The combination of insulation from the master mould and conformal cooling channels conduces to particularly rapid temperature changes in the cavity. The mould temperature is measured by cavity near temperature sensors.

Table 2 Relevant process parameters

Parameter	PP	PC
Melt temperature (°C)	240	300
Injection velocity ($cm^3\ s^{-1}$)	18	18
Mould temperature[a] (°C)	40/160	80/160

[a] See Fig. 4

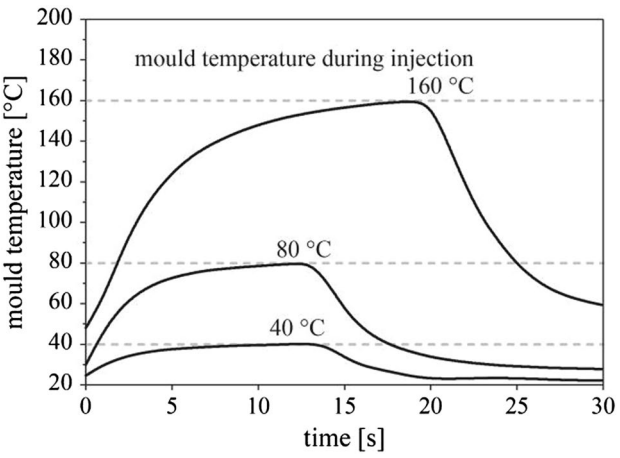

Fig. 4 Mould temperature of the variothermal injection moulding process with different mould temperature for injection moulding

In the investigations the lowest mould temperature of 40 °C for PP and 80 °C for PC was applied with a sufficient filling of the cavity. For the high value 160 °C was used. Hence, with the used variothermal tempering process, the temperature of the mould can be above the crystallisation temperature of the PP or rather above the glass transition temperature of the PC during the injection of the melt. Afterwards the mould and the melt is cooled down and a save ejection can be achieved. The curves of the temperature for the different mould temperatures during injection are shown in Fig. 4.

2.4 Analytic approach of the melt-mould contact temperature

The cooling velocity in the surface area of the part (area with direct contact between mould and part) can be determined only by approximation. Due to the local and rapid effect a safe measurement is restricted, especially in thin wall and micro injection moulding processes. However, in the literature several estimations were carried out (Fig. 5): Wuebken (1974) calculated the cooling behaviour for polycarbonate and polystyrene in plates with 3 and 2 mm respectively. Here, a cooling velocity in the surface area (0.2 mm distance to the mould) between 60 and 100 K s^{-1} can be determined. Hoffmann (2003) revealed the same dimensions for a polypropylene with a cooling velocity 35 K s^{-1} in a distance of 0.1 mm to the surface. Jungmeier (2010) has simulated the cooling of a 0.5 mm PA66 part and revealed a cooling velocity of more than 600 K s^{-1}.

These calculated or simulated cooling velocities are iterations of a particular cooling phase. Thus, the real maximum cooling velocity can be expected at 500 K s^{-1} or higher, especially in thin wall injection moulding processes. A distinct and accurate assessment of the occurring cooling velocity and the temperature in the surface area is currently not feasibly.

An analytical approach to calculate the contact temperature of the polymer melt with the cold mould surface is shown in Drummer et al. (2012). The contact temperature $T_{contact}$ is dependent on the temperature of the mould T_{mould}, the temperature of the polymer melt $T_{polymer}$ and the thermal diffusivity e:

$$T_{contact} = \frac{T_{mould} \cdot e_{mould} + T_{polymer} \cdot e_{polymer}}{e_{mould} \cdot e_{polymer}} \quad (1)$$

$$e = \sqrt{k \cdot \rho \cdot c_p} \quad (2)$$

with k the thermal conductivity, ρ the density and the specific heat capacity c_p of the materials. The values for the investigated polymers are shown in Table 1. For the mould material a density of 7,850 kg m^{-3}, a thermal conductivity of 29 W m^{-1} K^{-1} and a specific heat capacity of 460 J kg^{-1} K^{-1} was used.

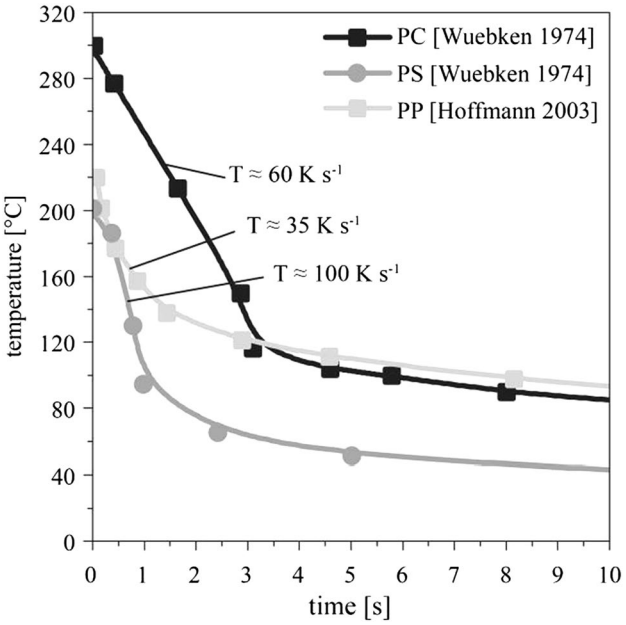

Fig. 5 Calculated cooling conditions for exemplarily three materials, according to Wuebken (1974) and Hoffmann (2003)

2.5 Characterization

The investigation of temperature dependent viscosity with overlapping shear rate of the material measurements with a rotational viscometer (ARES 2000, TA Instruments) were carried out with an oscillating deformation. An increasing complex viscosity n* gives information about the solidification of the material and the resistance to flow. To vary the shear rate of the material frequencies of 1 and 50 Hz were applied. A circular blank with a diameter of 25 mm with a thickness of 2 mm fixed between two parallel plates (diameter 25 mm) and heated up to a starting temperature of 200 °C (PP) and 260 °C (PC) and cooled down during measurement with a cooling rate of 2 K min^{-1}. The resulting shear rate is about 10^{-5} s^{-1} and 10^{-3} s^{-1} respectively. According to Zhao et al. (2003) the shear rate in micro injection moulding processes is significantly higher with rates between 10^4 s^{-1} and 10^7 s^{-1}. In addition, Jungmeier (2010) revealed a shear rate above 10^6 s^{-1} for micro injection moulding processes. Despite the applied low shear rates a temperature and shear rate dependent behaviour can be expected.

The cooling velocity affects the solidification (crystallization or glass transition) of the polymer melt. For this, high speed differential scanning calorimetric (DSC) measurements were carried out using a Flash-DSC 1 (Mettler-Toledo GmbH). It allows an investigation of the material behaviour using a weighted sample of approx. 10 µg at different cooling velocities between 10 K min^{-1} (0.17 K s^{-1};

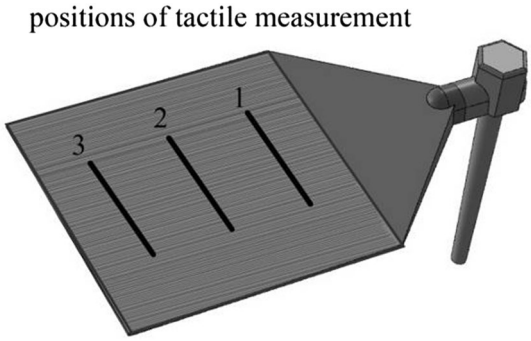

Fig. 6 Positions of the measuring section for tactile characterization of the height of the lamellar structures

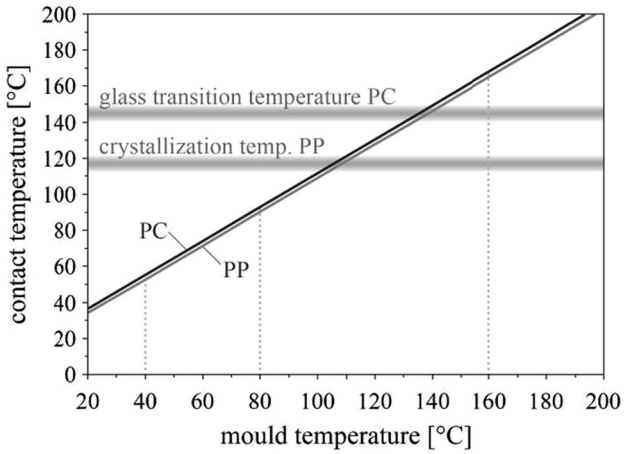

Fig. 7 Analytically calculated contact temperature in dependence of the mould temperature

cooling velocity for most standard DSC measurements) and 60,000 K min^{-1} (1,000 K s^{-1}). These high cooling rates correlate to typical process conditions (see Fig. 4).

An optical analysis of the replicated microstructures was carried out with a sub-µ computer tomograph (Fraunhofer IIS-EZRT, Erlangen). This allows a non-destructive 3D-analysis of the lamellae with a resolution up to 5 µm. Based on these measurements the cross section in the specimen centre can be investigated. A further characterization of the lamellae of the mould and the replicated specimens was carried out by SEM images (SEM Ultra Plus type, supplier: Zeiss). These investigations allow a precise investigation of the replication of the microstructures with a resolution up to 1 µm and better.

A tactile characterization was carried out using a Hommel-ETAMIC T1000 (Jenoptic AG) with a probe tip of 5 µm with a radius of 90°. On the specimens three measuring sections are investigated in dependence of distance to the ingate, Fig. 6.

3 Results and discussion

3.1 Analytical calculated contact temperature

Figure 7 shows the analytically calculated contact temperature of the polymers in dependence of the mould temperature. Due to the high thermal diffusivity of the metal mould the contact temperature approaches always a marginal higher value as the deployed mould temperature. Thus, the surface area of the part which has direct contact

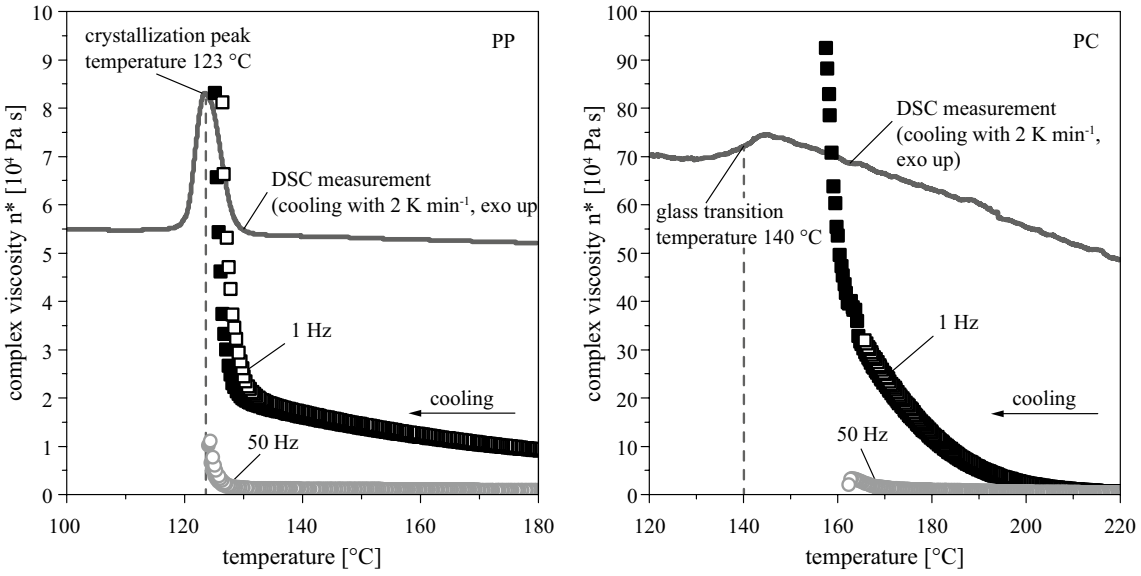

Fig. 8 Complex viscosity of PP an PC in dependence of temperature for two different oscillating frequencies supplemented to the DSC cooling curves (both with a cooling rate of 2 K min^{-1})

to the mould is cooled down immediately. This can affect the flow behaviour (due to an increasing viscosity) especially in thin mould elements and hence the replication of microstructures.

Consequently, to achieve a defined contact temperature the mould has to be tempered at this temperature. With the used dynamic tempering system the mould can be heated above the crystallization or the glass transition temperature, as mentioned above.

3.2 Rheological behaviour

In Fig. 8 can be seen the complex viscosity of the two materials. As expected, the materials reveal a low viscosity (between 200 and 3,500 Pa s) at higher temperatures which allows good flow behaviour. However, under real process the viscosity must be significant lower due to the overlapping shear thinning effect. The PP shows almost a constant viscosity up to a temperature with incipient crystallization of

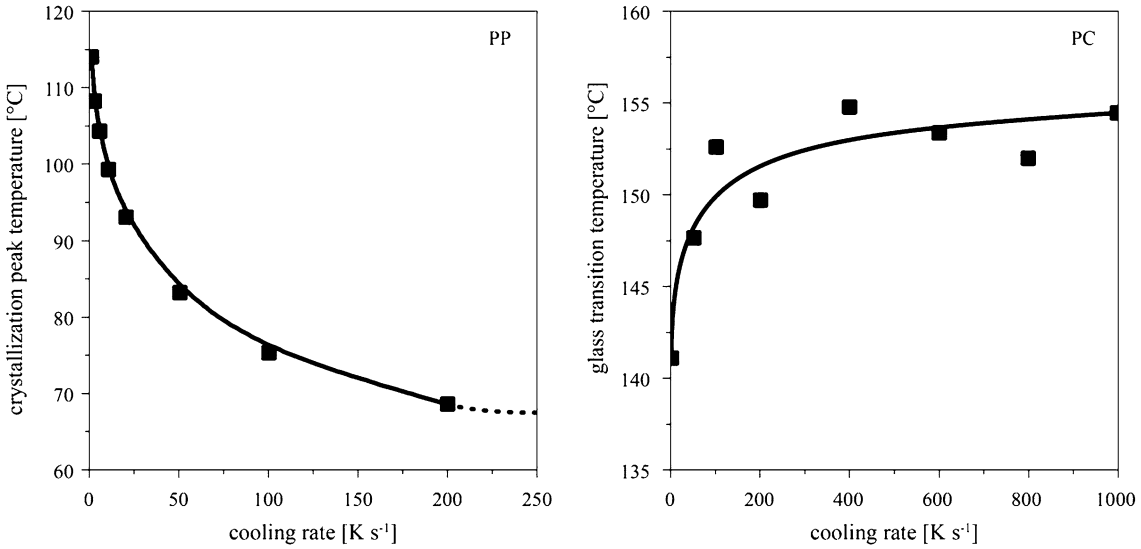

Fig. 9 Crystallization peak temperature (PP) and glass transition temperature (PC) in dependence of the cooling velocity

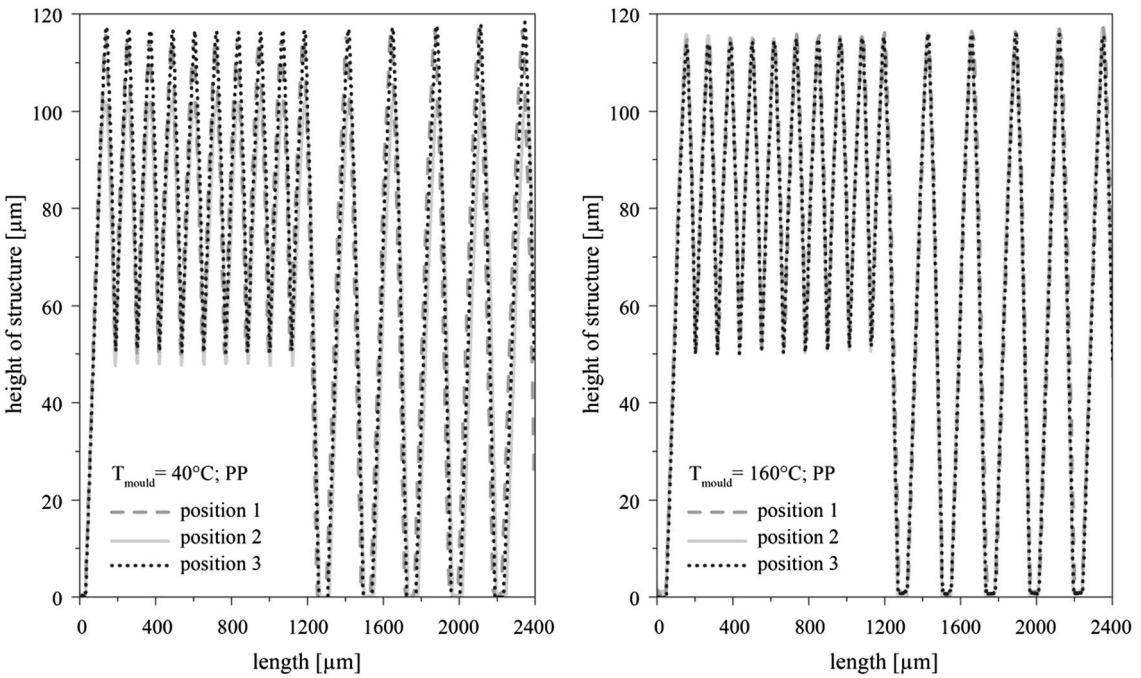

Fig. 10 Tactile measurements of the lamellae of the PP specimens in dependence of process conditions and position

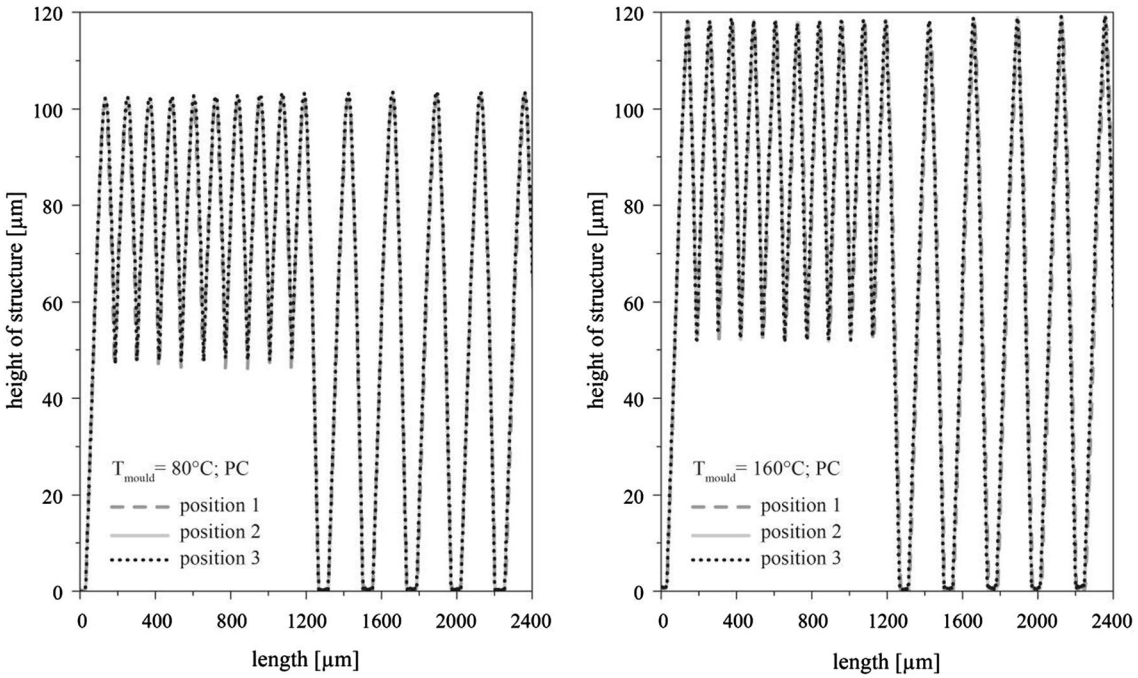

Fig. 11 Tactile measurements of the lamellae of the PC specimens in dependence of process conditions and position

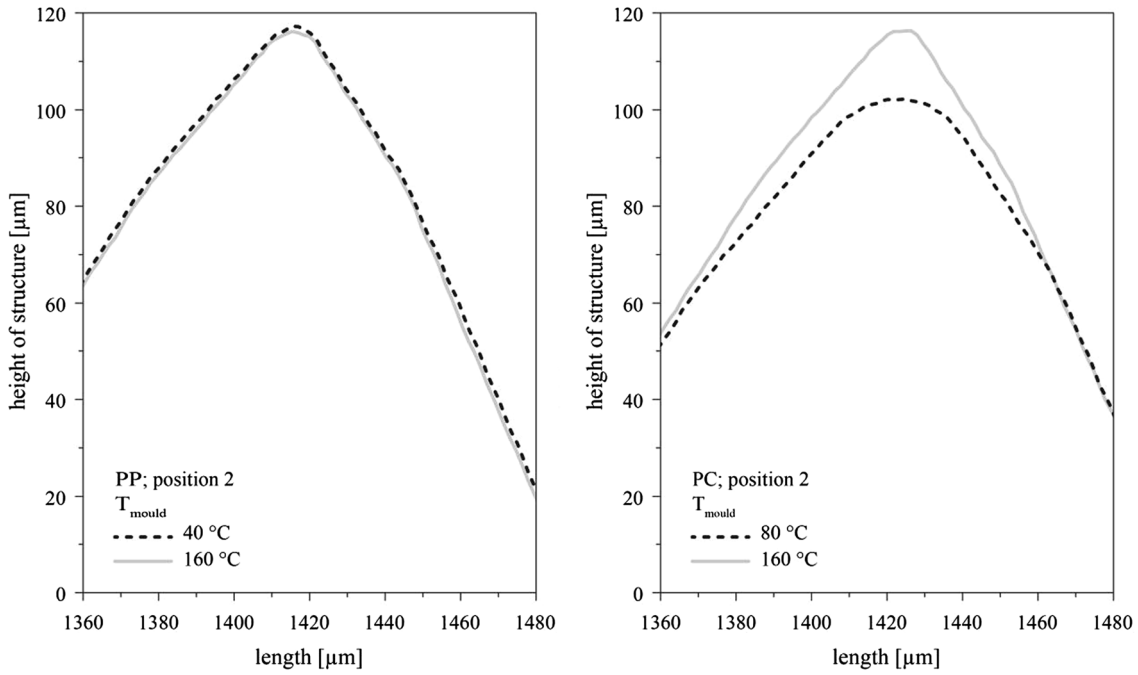

Fig. 12 Tactile measurements of the lamellae of the PC specimens in dependence of process conditions and position

the material below 130 °C. Recognizable is also the decreasing viscosity with higher shear rate of the material at a frequency of 50 Hz. This suggests that the higher shear leads to a significant shear thinning of the material which counteracts the increasing viscosity due to the crystallisation. This can be seen that rotational measurements with higher shear rates can be carried out up to the crystallization peak temperature whereas with lower shear rates the viscosity increases already

with the crystallization onset. In a real process the shear rate is significant higher which can result in a higher shift.

The shear thinning effect can also be found by the PC material. A higher shear rate of the material leads to a lower viscosity due to the shear thinning effect. Furthermore, at a low deformation rate (1 Hz) the material reveals an increasing viscosity below a temperature of approx. 195 °C. This temperature range might be defined as flow temperature or melting point equal to semi-crystalline polymers (Rudolph et al. 2009). Above this temperature the material exhibits a nearly constant low viscosity. A higher shear rate seems to shift this temperature range to a lower value which can be seen in a slight increasing viscosity at 165 °C or rather the subsequent abort of the measurement. Thus, below this temperature an ongoing measurement is not possible due to the fast increasing viscosity of the material which can be go along with the initiating glass transition of the material. However, DSC measurements at 2 K s^{-1} reveal a glass transition temperature at 140 °C with an onset at 147 °C. This is slight below the values obtained at standard cooling rates of 10 K s^{-1}. Furthermore, a significant increase of the viscosity occurs already approx. 20 K above the glass transition temperature.

In general, the semi-crystalline PP material reveals a lower viscosity even at lower temperatures below the used 160 °C whereas the amorphous PC material has an approximately ten times higher viscosity which increases already at the maximum applied mould temperature of 160 °C.

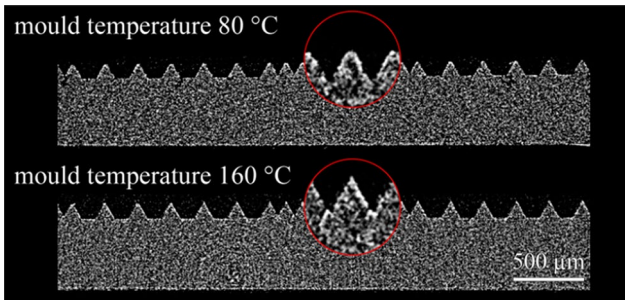

Fig. 13 CT-recording of PC specimens

3.3 Cooling behaviour

The cooling velocity affects the solidification of the material, i.e., the crystallization behaviour and the glass transition respectively. For the PP material (Fig. 9, left) a significant shift of the crystallization peak temperature is obviously with increasing cooling rate. For a cooling rate of 0.17 K s^{-1} (10 K min^{-1}) the crystallization peak reveals at 114 °C which correlates with the crystallization temperature range according to the manufacturer's data. An increasing cooling rate reduces the peak temperature. Already at a cooling rate of 200 K s^{-1} the peak temperature is below 70 °C. An increasing cooling rate suggests a further decreasing peak temperature, but there is no more a crystallization peak identifiable. Thus, a very high cooling rate seems to inhibit an immediately crystallization of PP, which decelerates the solidification of the material. Consequently, this effect allows for the material sufficient flow behaviour especially in the contact area. This effect can be intensified by the overlapping shear thinning of the material.

The glass transition temperature of the PC material in dependence of the cooling rate is shown in Fig. 7 (right). In contrast to the PP material, the PC is less affected by the cooling rate. The PC shows quite in contrast a slight

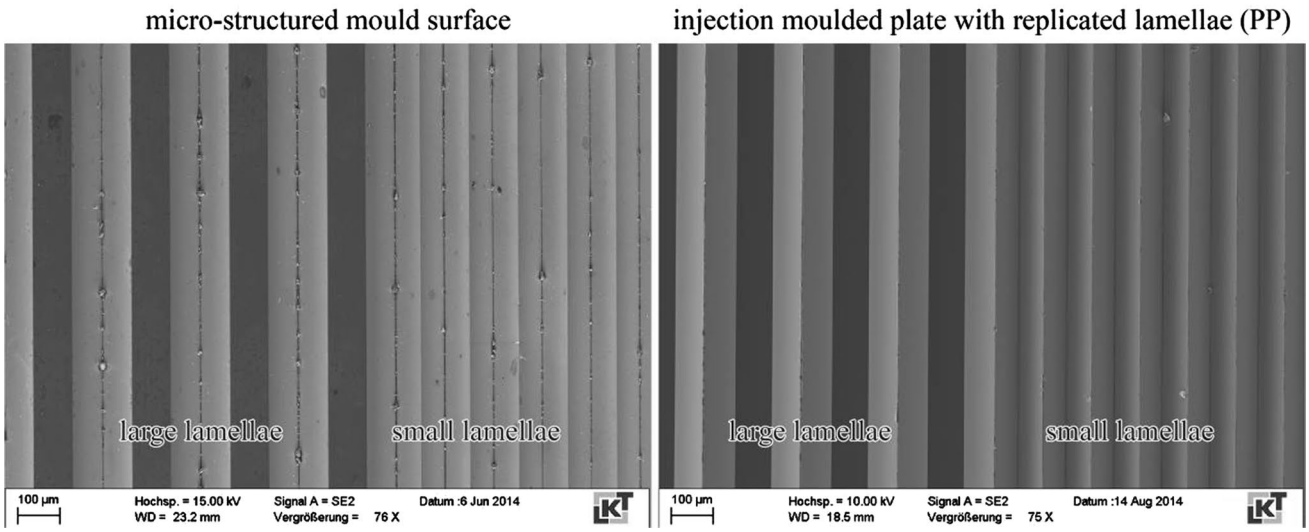

Fig. 14 SEM-recording of the lamellae of the mould (*left*) and an exemplary specimen (*left*)

increase of the glass transition temperature. This is at a low cooling rate (0.17 K s^{-1}) at approx. 145 °C and at higher cooling rates up to 1,000 K s^{-1} nearly constant at approx. 154 °C. This is according to Rudolph (2009) due to a reduced time for relocation processes of the molecular chains and a resulting increase of the free volume. Consequently, a fast cooling of the material impairs the flow behaviour. This effect occurs especially in the surface area of the part which has direct contact to the cold mould.

3.4 Tactile measurements

The tactile measurements allow a quantitative discussion of the replicated micro structures. Figure 10 shows the tactile measurements of the PP material in dependence of the process conditions and the distance to the ingate. A slight deviation of several micrometers is within the resolution of the testing method. The results show that the replication of the microstructure is not affected from the distance to the ingate and the flow length respectively. This is the same for both investigated process conditions. The height of the lamellar structures correlate with the dimensions of the mould, thus with the PP material a good replication of the microstructures is possible. This is relatively independent of the mould temperature.

For the PC material the measurements (Fig. 11) also reveal no difference between the investigated positions. However, the PC material reveals a significant influence of the applied mould temperature that means a degreasing mould temperature reduces the replication of microstructures. This is due to increasing viscosity with reaching the glass transition temperature range during melt cooling.

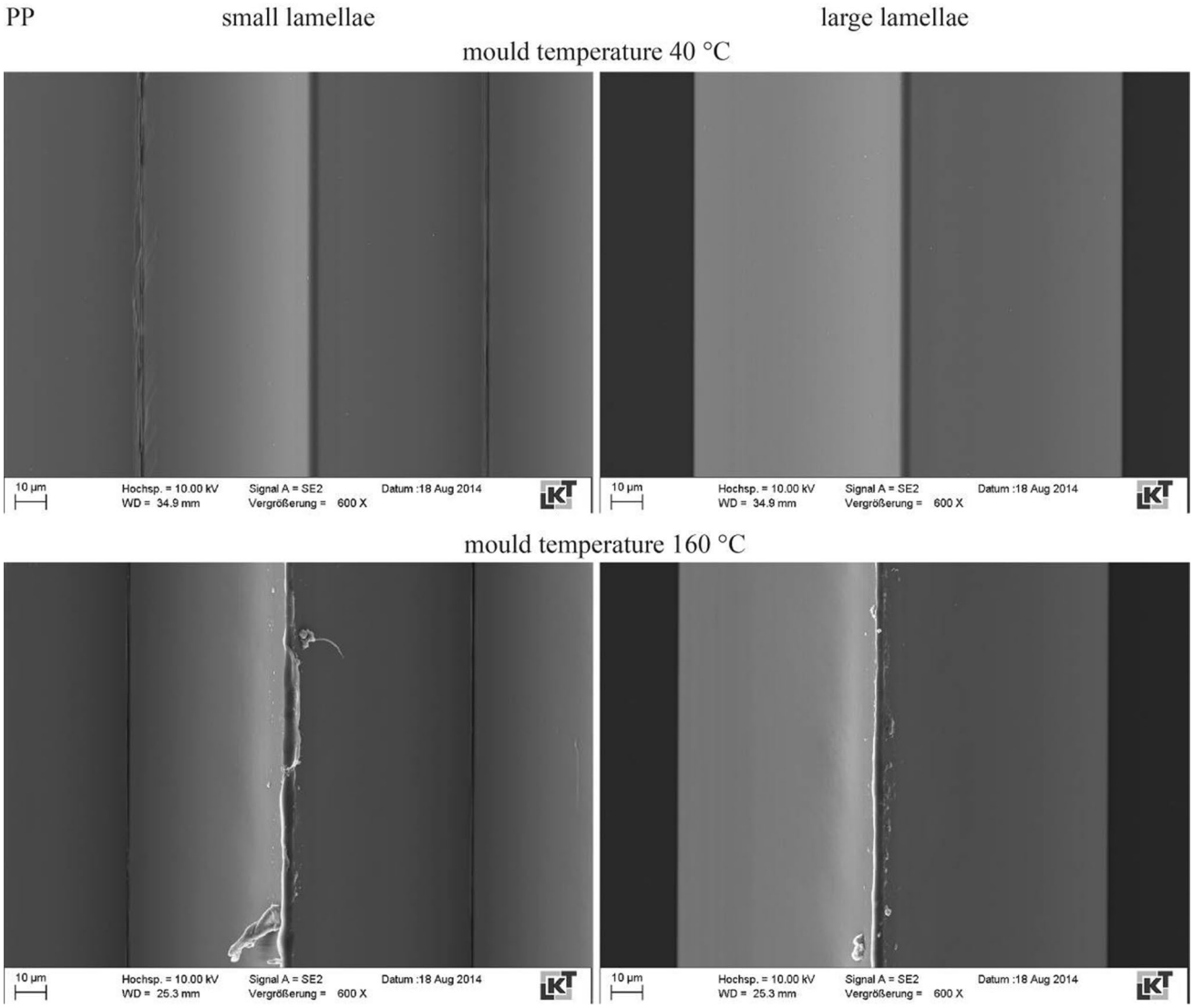

Fig. 15 SEM-recording of PP specimens with focus on the large and small lamellae in dependence of the process conditions

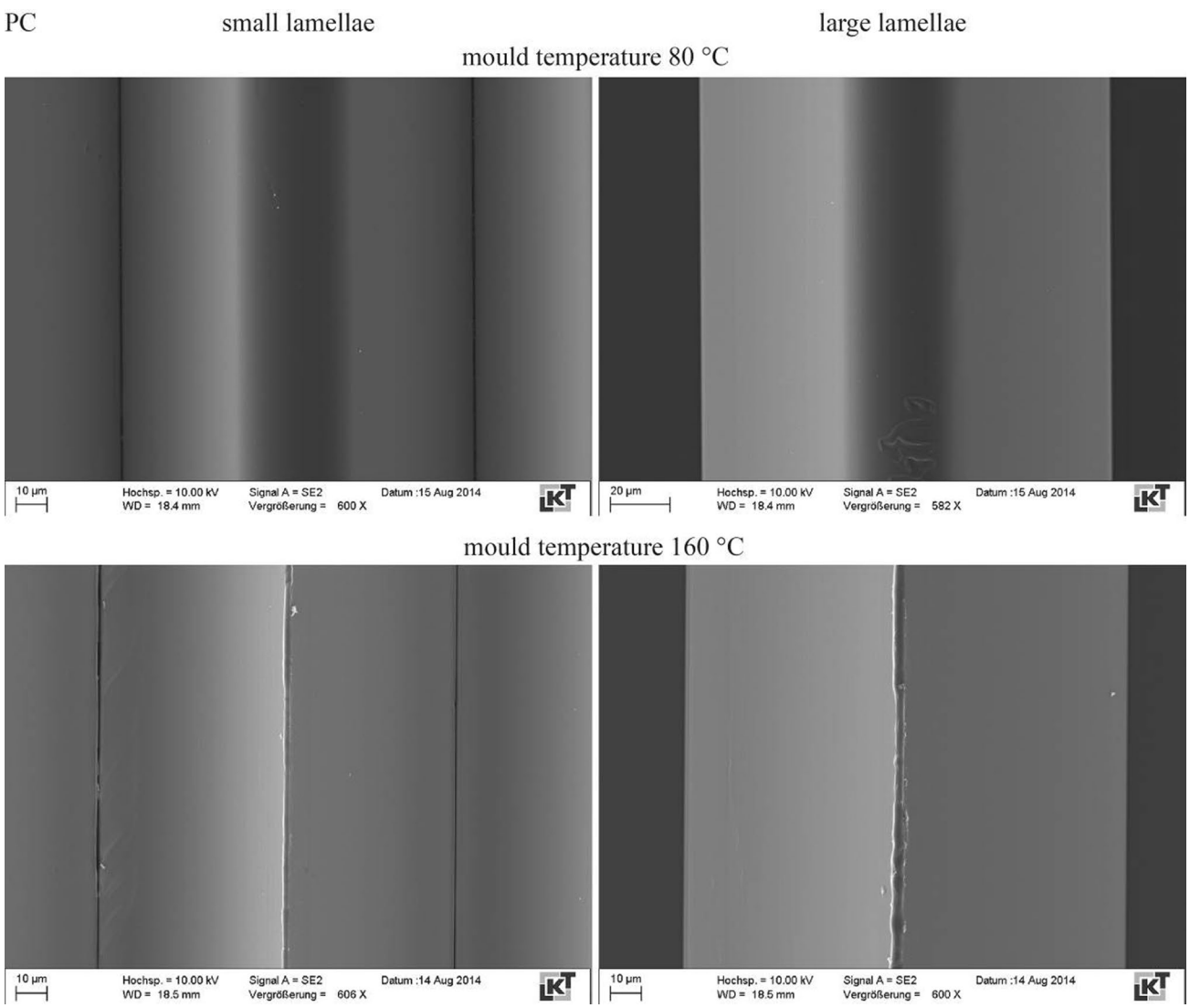

Fig. 16 SEM-recording of PC specimens with focus on the large and small lamellae in dependence of the process conditions

The different behaviour of PP and PC is compared in Fig. 12 on one exemplary lamellar peak. This emphasises the good microstructure replication of the PP material independent of the process conditions. Only the peak replication shows a slight difference which is better for a mould temperature of 160 °C. This can be also due to the deviation of the tactile measurement. The PC reveals a significant difference of the replication of the lamellar peak. While with a mould temperature of 80 °C a height of the lamellae reaches only 100 µm; a mould temperature of 160 °C allows a nearly complete replication of the lamellae.

3.5 Optical characterization

The PC specimens are exemplarily investigated with computer tomographic (CT) measurements. Figure 13 represents the different quality of the replication of the micro structure. The specimen injection moulded at 80 °C reveals rounded edges which is a result of an insufficient mould filling due to the fast cooling of the material. The increased mould temperature of 160 °C and the exceeding of the glass transition temperature favours the replication of the micro structures. This can be observed in a sharp moulded top of the lamellar micro structure which correlates with the tactile measurements.

The precise geometry of the lamellae in the mould surface is shown in Fig. 14 (left). However, small impurities can be found on the bottom of the lamellar peaks. This can be due to a contamination during preparation for SEM analysis. On Fig. 14 (right) a specimen (PP) with the replicated lamellar structures is shown. Besides the good replication is also the alternating lamellar structure of large and

small lamellae visible. The small impurities found on the mould surface cannot be identified on the specimen.

The replicated lamellae for the PP material are shown in Fig. 15. Generally, the replication of the small and the large lamellae is comparable. Furthermore, different process conditions (i.e., mould temperatures) have minor effect on the replication of the micro structures. Only a slight rounding on the peak of the lamellae of the specimen injection moulded at 40 °C can be found. However, the dimensions are in the range of microns, thus with tactile measurements (see Fig. 12) cannot resolve the differences. Futhermore, the specimen injection moulded at 160 °C reveal artefacts on the peaks. These can be small deformations occurring during the ejection of the part due the good replication and the resulting form closure.

For the PC material a significant higher dependence of the process conditions to the replication of the lamellae can be observed, Fig. 16. As already identified with tactile measurements a mould temperature of 80 °C leads to an insufficient replication of the mirco-structures. This is evident in the rounded edges of both the small and the large lamellae. The specimens injection moulded at 160 °C are replicated with sharp peaks. The small deformations in the peak are visible similar to the PP specimens which can be attributed to the form closure due to the good replication.

4 Conclusion

The results have revealed that the mould temperature is a key process parameter to achieve high replication quality of micro structured polymer parts. Analytical calculations have shown that the contact temperature (the temperature in the contact area of melt and mould) is slight above the mould temperature. Consequently, a fast cooling occur which increases the melt viscosity. For this, a rapid tooling mould with cavity near cooling channels and a dynamic tempering system allows a mould temperature above the solidification temperature during injection of the melt and a subsequently cooling of the part for save ejection.

Rotational viscosity measurements have revealed that a higher melt shear rate reduces the viscosity due to the shear thinning effect. For the semi-crystalline PP the solidification temperature (crystallization temperature) reduces slightly to a lower temperature thus higher shear rate supports the flow behaviour of semi-crystalline polymers. For the amorphous PC material a higher shear rate increases slightly the onset-temperature of the glass-transition temperature worsen the flow behaviour. A fast cooling of the melt, which occurs in the contact area particularly, inhibits the crystallization of semi-crystalline polymers, whereas for amorphous polymers a slight increase of the glass transition temperature was observed.

Consequently, the investigations to the replication of micro structured polymer parts allow for the following statements:

- Mould temperatures about the solidification temperatures (i.e., glass transition temperature or crystallization temperature) lead a sufficient low melt viscosity delaying the solidification of the melt which allows a sufficient high replication quality.
- For semi-crystalline polymers the fast cooling inhibits the crystallization process. As a consequence, the viscosity remains at a low value which allows a good replication also at a mould temperature below the solidification temperature. The shear rate of the melt favours this effect as it reduces the melt viscosity due to the shear thinning effect.
- For amorphous polymers the fast cooling increases slightly the glass transition temperature to a higher value. This impairs the replication with moult temperatures below the solidification temperature. A higher shear rate of the melt lead to a reduced viscosity, too, but also the glass transition temperature is shifted to a slight higher value. This lead to significant limited replication quality at low mould temperatures.
- Contrary to the expectation, no dependence of the replication quality of the flow length was observed.

Futher studies should investigate the influence of the mould temperature on other polymers with higher solidification temperatures. Also, the influence of the flow length to the replication quality should be investigated with specimens with longer flow paths.

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