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Dr.-Ing. Christopher Fischer[1]; Dr.-Ing. Benoit Merle[2]; Prof. Dr.-Ing. Dietmar Drummer[1]
[1]Institute of Polymer Technology (LKT), Friedrich-Alexander University Erlangen-Nuernberg
[2]Materials Science & Engineering, Institute I, Friedrich-Alexander University Erlangen-Nuernberg

Achieving homogeneous properties for thin-walled components through dynamic temperature control

During injection molding of semi-crystalline thermoplastics, an influence by means of the mold temperature and thus on the cooling behavior of the plastic melt can have a significant effect on the resulting component properties. Within the scope of the present investigations, the influence of the mold temperature on the formation of internal and mechanical properties over the flow path length is investigated with the aim to produce components with homogeneous properties over the entire component geometry. The results show that an increase in the mold temperature leads to a significant homogenization of the component properties over both the component cross-section and the flow path length especially by improving crystalline and mechanical properties far from gate.

Realisierung homogener Eigenschaften bei Dünnwandbauteilen mittels dynamischer Temperierung

Bei der Spritzgießverarbeitung teilkristalliner Thermoplaste kann sich eine Beeinflussung der Werkzeugtemperatur und somit des Abkühlverhaltens der Kunststoffschmelze signifikant auf resultierende Bauteileigenschaften auswirken. Im Rahmen der vorliegenden Untersuchungen wird der Einfluss der Werkzeugtemperatur auf die Ausbildung innerer Bauteileigenschaften und mechanischer Eigenschaften über die Fließweglänge erforscht. Ziel ist die Herstellung von Bauteilen mit homogenen Eigenschaften über die gesamte Bauteilgeometrie. Die Untersuchungen zeigen, dass eine Erhöhung der Werkzeugtemperatur sowohl über dem Bauteilquerschnitt als auch über die Fließweglänge zu einer Homogenisierung der Bauteileigenschaften führt, was insbesondere über eine Verbesserung kristalliner und mechanischer Eigenschaften in angussfernen Bauteilbereichen erreicht wird.

Achieving homogeneous properties for thin-walled components through dynamic temperature controls

C. Fischer, B. Merle, D. Drummer

1 INTRODUCTION

In recent years, components in the field of micro technology have gained increasing relevance, whereby the demand for product miniaturizing is substantially driven by the micro electro-mechanical systems (MEMS) industry [1]. A particular role, especially for applications in biotechnology as well as medical technology, is played by components made of plastic, which typically have lower manufacturing costs compared to other materials [2].

Due to the small component's cross-sections, high cooling velocities of the polymer melt occur in micro and thin-wall injection molding. This can result in a premature solidification of the melt during the filling process and, therefore, in limitations in complete cavity filling. To attain a complete cavity filling of fine structures, an adaption of the process settings, e.g. by means of an increase in injection velocity, in mold temperature and melt temperature has been established. Especially an increase in mold temperature leads to a reduction in the polymer melt's cooling velocity what can result in more distinct internal component properties (e.g. higher degree of crystallization, coarse spherulitic structures, development of a more stable crystal modification, etc.) [3, 4]. By means of a defined adjustment of the internal component properties, external component properties, such as mechanical, optical and tribological properties, can also be influenced. For example, regarding semi-crystalline thermoplastics it was shown in [5] that an increase in the degree of crystallinity leads to an increase in stiffness and strength as well as a decrease in the elongation at break. Moreover, in [6] an increase in the mechanical property profile by about 100 % was achieved by influencing the crystal modification of the polymer. Furthermore, oriented component areas also lead to higher tensile moduli, higher yield stresses as well as a reduction in the elongation at break, which, for example, could be shown in [7]. Regarding the tribological behavior, for example Jungmeier [8] and could show a decrease in wear behavior when changing the internal properties by influencing the component cooling behavior.

A defined influence of the internal properties due to a reduction in cooling velocity of the polymer melt (for example by increasing the mold temperature or by using cavity materials with a low thermal conductivity) can contribute to an increase in the mechanical and tribological properties for injection molded semi-crystalline micro and thin-walled components [9, 10].

Nevertheless, present investigations only focus on concentrically located component properties while especially for thin-walled components, due to high changes of shear conditions over the flow path length, inhomogeneous component properties can be assumed. In particular, it is expected that, depending on local volume flows and the interaction of locally different shear heating and cooling speeds, differences in the crystalline and oriented microstructure must result. How internal properties develop as a function of component geometry and what influence this has on local component properties is still largely unexplored. Therefore, the objective of the present study is to show the influence of the mold temperature on the potential to manufacture thin-walled components that attain improved and homogeneous mechanical properties over the entire component geometry and, therefore, allow for a safe component design.

2 DYNAMIC TEMPERING

An increase in mold temperature can be realized by dynamic tempering of the mold. A schematic diagram showing the temperature time distribution during dynamic tempering is shown in Figure 1.

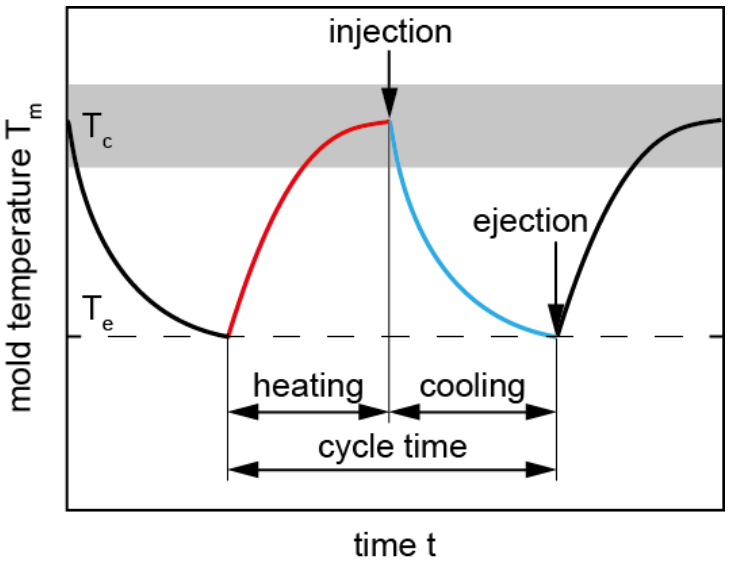


Figure 1: Mold temperature for dynamic temperature control based on [7]

During dynamic tempering the mold is getting initially heated into the material specific solidification temperature range where the melt is getting injected. Subsequently, after the cavity filling, the mold is getting cooled below the ejection temperature where the component can be ejected. Due to the high mold temperature during the injection phase lower cooling rates of the melt are

achieved what does not only enable a complete component filling but also an influence of the internal component properties [8].

Currently, different dynamic tempering systems are used in industry. Here, the heating often is realized by fluids (for example oil, water, gaseous CO_2) or electrically (for example induction, infrared, ceramic heaters). Novel water-based approaches realize short cycle times using cavity near temperature channels. By this means, plastic gears with increased tribological properties could have been manufactured [11]. Nevertheless, the relatively low maximum mold temperature of approximately 180 °C can be seen as a disadvantage. One of the most established proceedings is described by dynamic tempering using oil. Here, the low heat capacity in comparison to water is one of the main disadvantages. CO_2 tempering systems realize the heating by means of gaseous CO_2 [12]. Heating by using induction can lead to limitations regarding a homogeneous temperature distribution especially for surfaces that are not flat. Furthermore, overheating can occur when using external inductors [11]. Usually, infrared heaters also lead to an inhomogeneous heating, since undercuts normally can't get irradiated equally. Moreover, due to the reflective steal surface the efficiency is relatively low so that special coatings often have to be used [13]. Ceramic heaters typically show a short heating time and can reach maximum mold temperatures above 300 °C.

The cooling of dynamic tempering concepts normally is achieved by using liquid mediums. Here, the most established proceedings realize the cooling by means of water or by means of oil. Achievable cooling rates regarding water in combination with cavity-near cooling channels are located in the range of 10 to 18 K/s [9, 14]. Comparing oil with water, normally lower cooling rates are achieved due to the lower heat capacity. Next to water and oil, liquid CO_2 can be used as a cooling medium [12]. Here, maximum cooling rates of up to 30 K/s were achieved.

3 EXPERIMENTAL

3.1 Dynamic Mold and Tempering Concept

The used mold concept has been developed by the companies Ypsomed GmbH and gwk Gesellschaft Wärme Kältetechnik mbH in collaboration with the Institute of Polymer Technology in Erlangen (LKT). The heating is realized by segmented heating ceramics, Figure 2. Four heating ceramics are arranged directly below the cavity area on each ejector or nozzle side, that are controlled by thermal sensors. The temperature can be set separately within a temperature range of 25 to 300 °C. By this means, a homogeneous temperature distribution along the flow path length can be achieved of ± 1 °C. The temperature is controlled by an integrat evolution 40 temperature control unit from gwk. This allows adjusting the heating power in order to be able to

determine a defined heating rate. The tool is cooled by water. The corresponding cooling channels are located below the heating ceramics. The water flow rate can be adjusted separately via the temperature control unit for the nozzle and ejector side in order to synchronize the resulting cooling velocities.

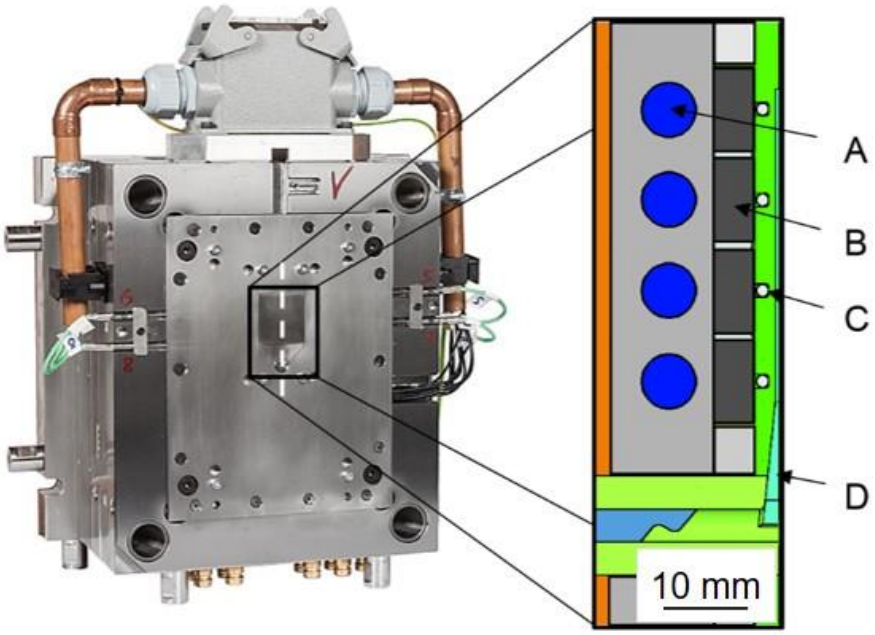


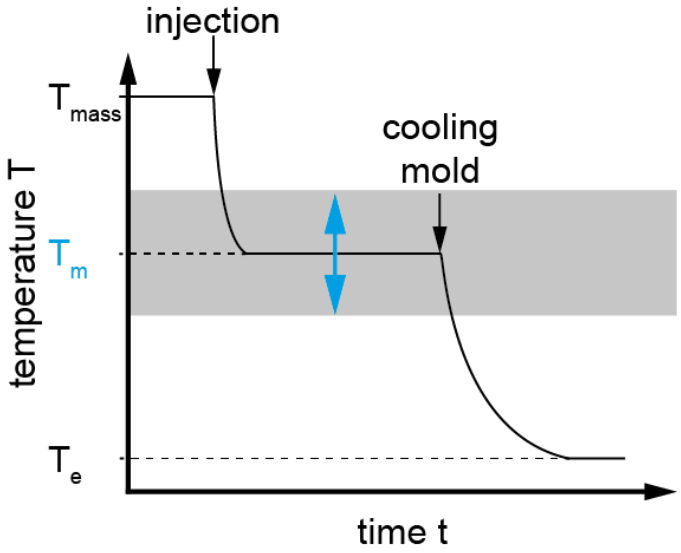
Figure 2: Dynamically temperature-controlled mold concept with heating ceramics and CAD section representing the cavity area (A: cooling channel, B: heating ceramics, C: thermal sensors, D: cavity)

3.2 Material, Specimens and Processing

For the present tests a non-nucleated PA12 (Grilamid L 20 G, EMS-Grivory) was used. As specimens 38 x 35 mm^2 (L X W) rectangular test samples with a component thickness of 500 µm were injection-molded using an Allrounder 370 U from Arburg with a screw diameter of 15 mm. Relevant injection molding parameters and the temperature curves investigated for homogenization are listed in Figure 3.

To investigate the influence of the mold temperature, a constant temperature distribution over the complete cavity area had to be realized. For this purpose, the four heating ceramics (at each the nozzle and ejector side) were adjusted to a homogeneous temperature level with a deviation along the flow path length of ± 1 °C. Temperatures of 60 °C were chosen in order to be able to estimate component properties which are based on the recommended mold temperature of the material manufacturer. The high mold temperature of 150 °C was selected in the crystallization temperature range of PA 12 on the basis of

internally performed DSC measurements. To reach the ejection temperature, a water temperature of approximately 10 °C was used.



parameter	value		
mold temperature T_m [°C]	60	100	150
mass temperature T_{mass} [°C]	250		
injection velocity v_{in} [mm s^{-1}]	100		
injection time t_{in} [s]	0.2		
isothermal holding time t_H [s]	5		
holding pressure p_N [bar]	1400		
ejection temperature T_e [°C]	25		

Figure 3: Used injection molding parameters

3.3 Analytical Methods

3.3.1 Morphology and Degree of Crystallization

For the observation of the morphology, a 10 µm thin section along the flow direction was taken from the center of the injection-molded components (see dashed line, Figure 4) and viewed at 45° linearly polarized transmitted light using an Axio Imager.M2 from Zeiss. To assess the degree of crystallization, X-ray diffraction measurements were carried out at the Department of Polymer Engineering from the University Bayreuth using a Seifert ID 3000 diffractometer system (40 kV, 30 mA, KαCu). For this purpose, the degree of crystallization was measured over the entire thickness of the component. The respective evaluation positions for determining the morphology as well as the degree of crystallization are close to the gate, concentric and far from the gate, Figure 4.

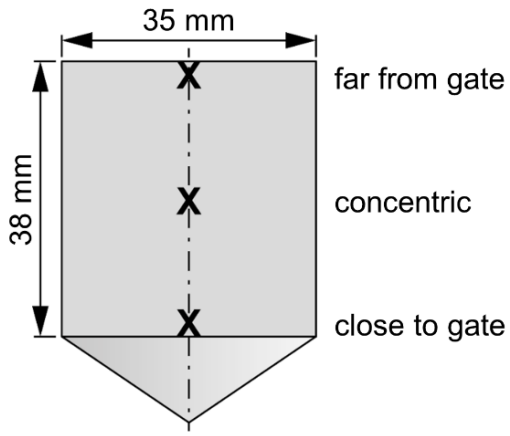


Figure 4: Positions for determining the internal properties

3.3.2 Mechanical Properties

For determining mechanical properties, tensile tests were performed to evaluate the respective modulus of elasticity, yield stress, elongation as well as the elongation at break. Therefore, tensile bars were prepared transversely to the flow direction (close to gate, concentric and far from gate) as well as longitudinal to the flow direction (concentric). For the tensile bar geometry, a 1 to 8 scaling based on the campus tensile bar according to DIN EN ISO 527 was used. Moreover, the test samples were dried at 23 °C using a vacuum oven until a humidity of less than 0,2 % was reached. The tests were carried out according to DIN EN ISO 527 on 5 tensile bars using an Instron 5948 MicroTester tensile tester at standardized climate (23 °C, 50 % relative humidity).

To evaluate the mechanical properties over the component's cross section, nanoindentation was performed using a nanoindenter G200 system (KLA-Tencor, Milpitas, CA, USA), equipped with a diamond Berkovich tip (Synton MDP, Switzerland) based on knowledge from a previous publication [15]. The embedded specimens were cut longitudinal to the flow direction, see dashed line in Figure 4. The cutting surface of the embedded specimens was polished with different polishing agents up to 0.1 µm grit size. Measurements were performed at specimens that had been conditioned at 23 °C and 50 % humidity. An oblique indentation array was used in order to measure the variation of the indentation modulus of elasticity E_{IT} (IT: Indentation Testing) across the cross-section of the polymer's core area on one component at a time. The data was evaluated using the standard Oliver-Pharr method. The spacing between the indents was set as 20 µm in order to avoid any overlapping of the plastic zones. The tests were run in load control mode: the force was ramped over 15 s up to a maximal value of 5 mN. Following a holding segment of 10 s, unloading was performed at the same rate as the initial loading. Finally, the indentation array

was imaged by optical light microscopy in order to measure the distance of every indent to the surface of the cross-section.

4 RESULTS

4.1 Morphology and Degree of Crystallization

Figure 5 shows the morphologies of the produced components at different mold temperatures. The component's edge area, which is in direct contact to the cavity wall, can be seen on top. For a clear magnified illustration, the components are only depicted up to the middle region.

The component edge layers of the components injection-molded at 60 to 150 °C show a strongly distinct oriented edge structure at positions close to the gate, which decreases over the flow path length. The high flow front speed with simultaneous high cooling of the plastic melt during the injection time is regarded as the cause for the formation of the strongly distinct oriented layers. The high shear and elongation stresses, especially in the component areas near the cavity wall, lead to an alignment of the molecular chains, whereby a simultaneously high undercooling of the melt and a resulting significant increase in viscosity leads to the fact that oriented molecular chains can no longer relax. Therefore, the primary cause for the most distinct oriented layers near the gate can be seen as a grown oriented area over the injection time. Due to the decrease of the volume flow along the flow path length, the oriented skin layer thickness is getting reduced at areas far from the gate. For this reason and due to the swelling flow during injection molding, which results in reduced shear stresses in the area of the immediate end of the flow path length, there are less to no distinct oriented skin layers in the component areas far from the gate. An increase in the mold temperature leads to a lower viscosity due to less cooling of the melt during the injection process. At the same time, the component edge areas facing the core area are able to relax due to the higher temperature. As a result, an increase in mold temperature leads to the formation of a smaller oriented skin layer thickness. Quantitatively, the concentric orientation edge layer to core ratio decreases from 18 % at 60 °C to 15 % at 100 °C and to 11 % at 150 °C.

Regarding the component's core areas, the formation of slightly coarser structures can be seen when increasing the mold temperature. This can be attributed to the lower nucleation rate at a lower supercooling, which typically results in a coarser structure, since the spherulites have more time and place to grow.

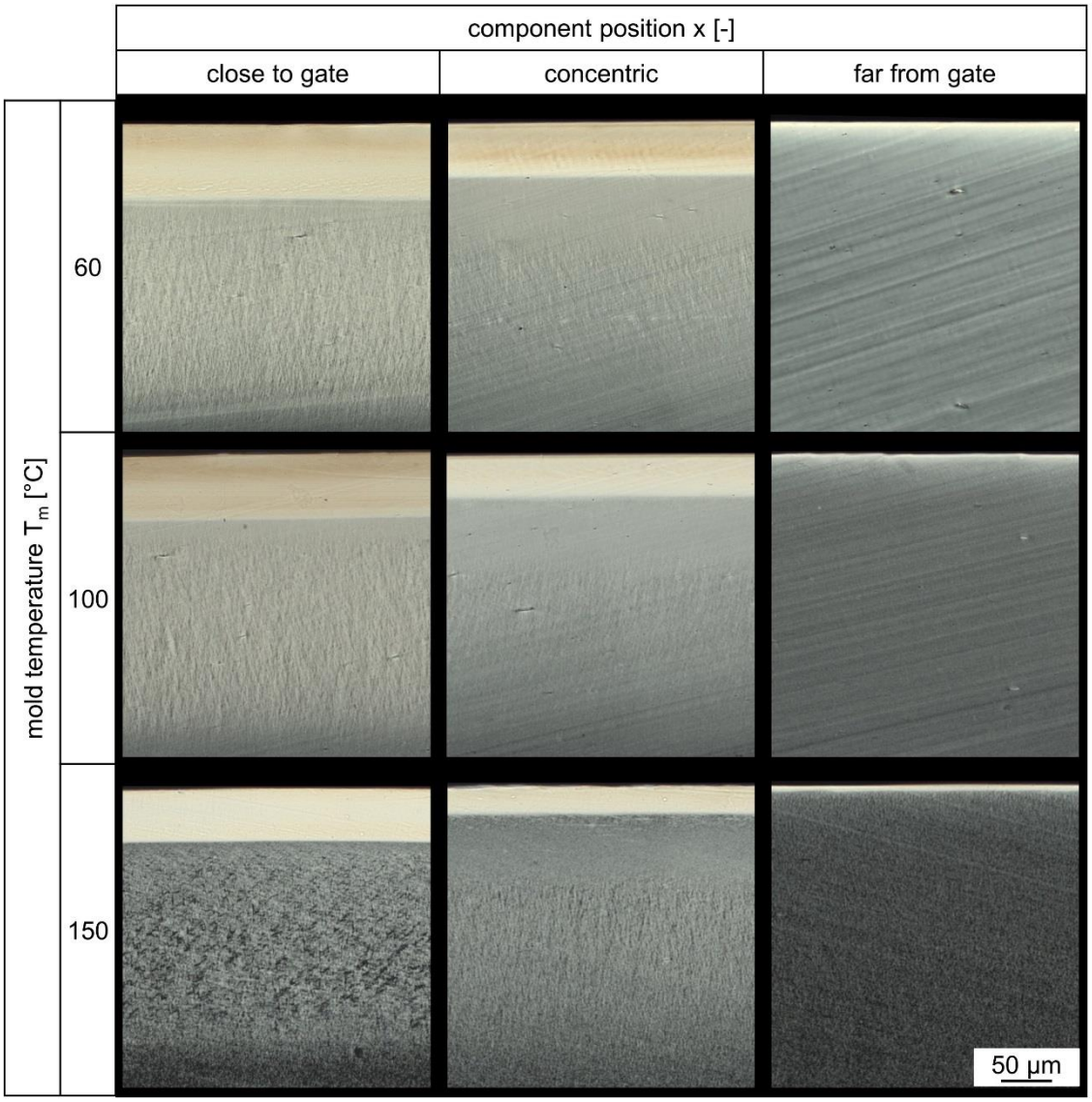


Figure 5: Morphology of the injection molded plates depending on the mold temperatures 60, 100 and 150 °C

Figure 6 shows the results of the X-ray diffraction measurements to determine the degree of crystallization. It can be seen that the highest degrees of crystallization are being measured at component areas close to the gate. Here, it is assumed that the comparatively high shear and elongation stresses in the component's skin area lead to a highly pronounced shear-induced crystallization. Since it is known that shear-induced component zones are even formed at high temperatures due to high shear-induced nucleation, there is also a clear deviation between the degree of crystallization far from gate and close to it at 150 °C. However, the differences are less pronounced during the injection phase than at lower mold temperatures due to the lower shear loads caused by relaxation effects and lower viscosities during the injection phase. The low

shear load far from the gate leads to a primarily temperature- and pressure-influenced (and less a shear-influenced) nucleation and crystallization, which leads to locally lower degrees of crystallization.

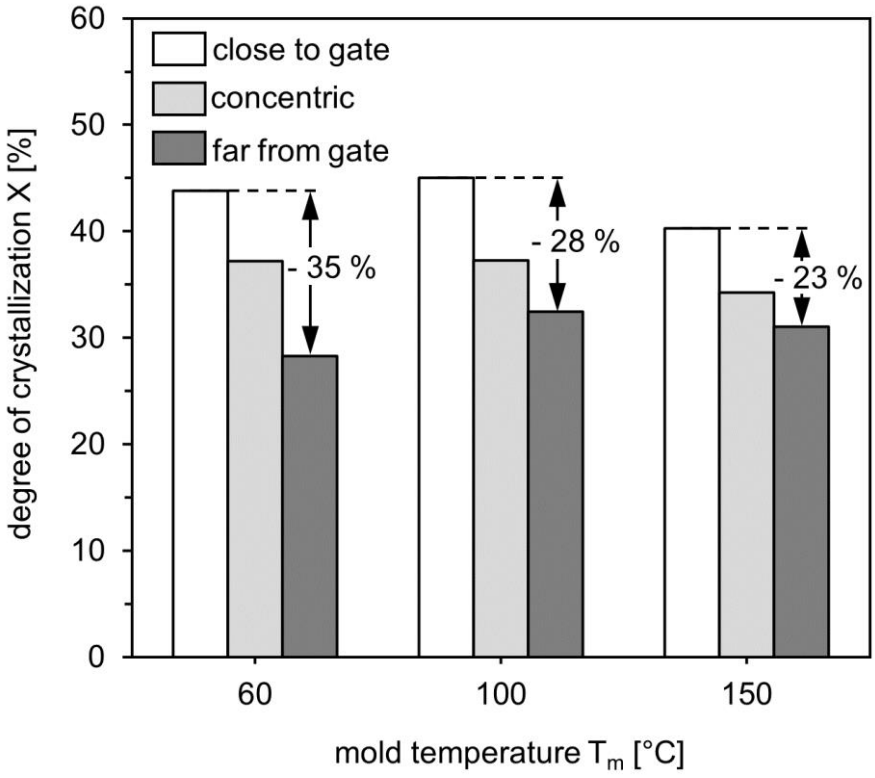


Figure 6: Degree of crystallization at different component positions depending on the mold temperature

4.2 Mechanical Properties

Figure 7 shows the results of the performed tensile tests. It can be shown that especially low mold temperatures, as recommended by material manufacturers, lead to significantly inhomogeneous properties over the component's flow path length, which does not sufficiently enable a safe component design for thin-walled components.

At a mold temperature of 60 °C, high stiffnesses and strengths are measured especially in the component area close to the gate, which is primarily attributed to the stiffness-increasing oriented skin layer. This contains a high proportion of directed polymer chains due to shear and elongation, which leads to a significant increase in stiffness and strength. A decrease in the oriented skin layer thickness and the degree of crystallization with increasing flow path length leads to a significant reduction in the mechanical properties. In component positions far from the gate, the mechanical properties are primarily determined by the structural and crystalline properties present in the core structure. Since

there are no clear orientations this core structure primarily equals the whole component cross-section. Here, the weakly defined morphology and crystalline properties lead to a significant reduction in component stiffness. With regard to the crystal modifications, at a mold temperature of 60 °C the formation of the γ' crystal modification can be assumed, shown in previous publications [16]. Since it is known that the thermally less stable mesophases are often associated with lower component stiffnesses, it is assumed that the γ' crystal modification primarily contributes to the reduction in stiffness-determining, whereby a precise assignment of the stiffness-decisive internal properties is not clearly possible due to changes in the crystalline as well as structural properties.

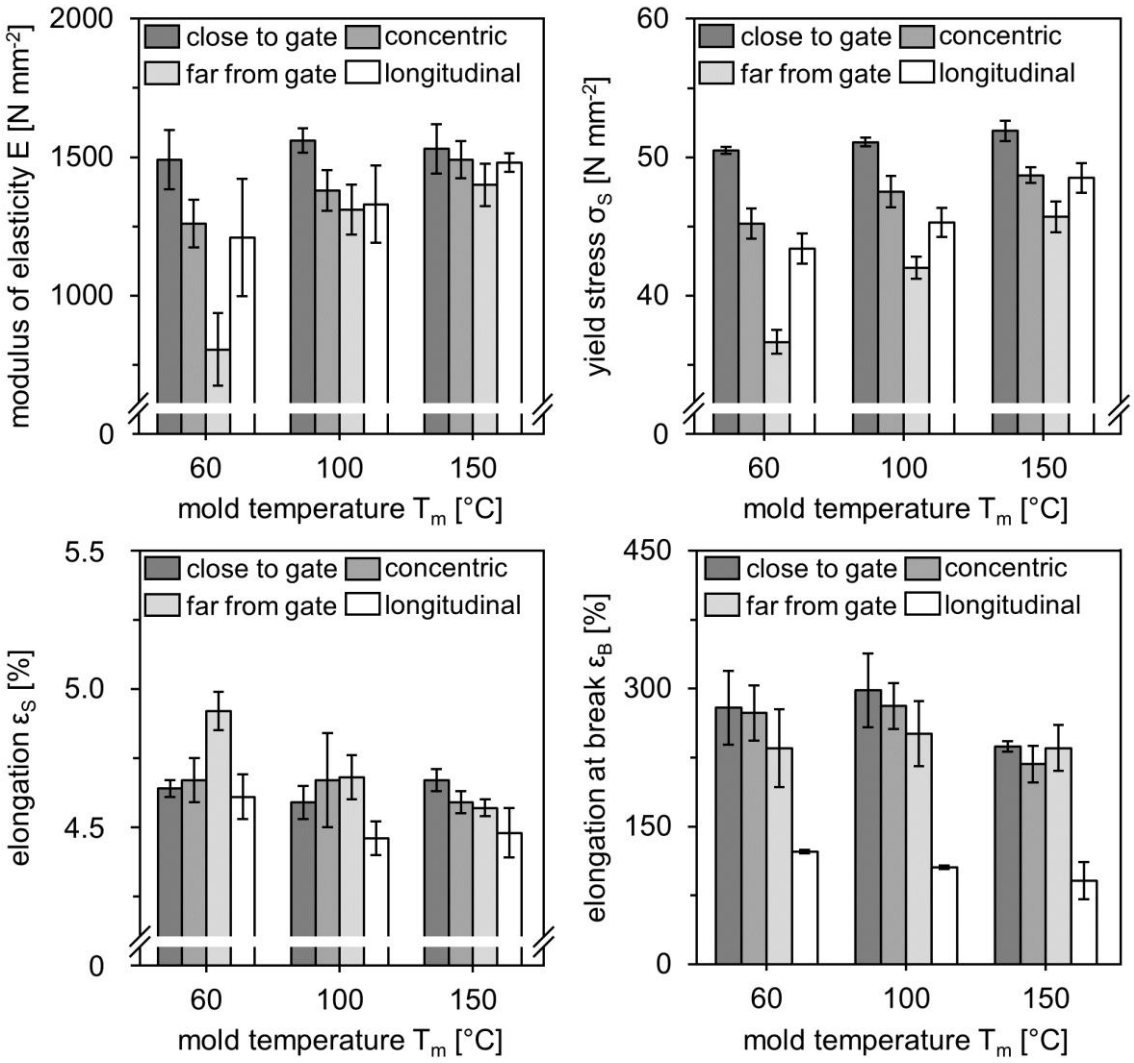


Figure 7: Tensile test results with regard to different component positions and mold temperatures

An increase in mold temperature leads to a reduction in the oriented skin layer thickness and to a more distinct crystalline structure in the core area of the sample. Furthermore, the formation of a primarily γ-dominant crystal modification at tool temperatures of 100 °C can be assumed [16]. All in all, a clear homogenization of the mechanical properties along the flow path length, for example by increasing the modulus of elasticity as well as the yield stress at positions far from the gate, results, what is reflected in a more homogeneous property distribution when considering the mechanical properties. In particular, the areas of the component that are far from the gate achieve an increase in the stiffness and strength parameters.

To estimate the mechanical properties over the component's cross-section the indentation modulus of elasticity E_{IT} was measured by nanoindentation. The results are depicted in Figure 8.

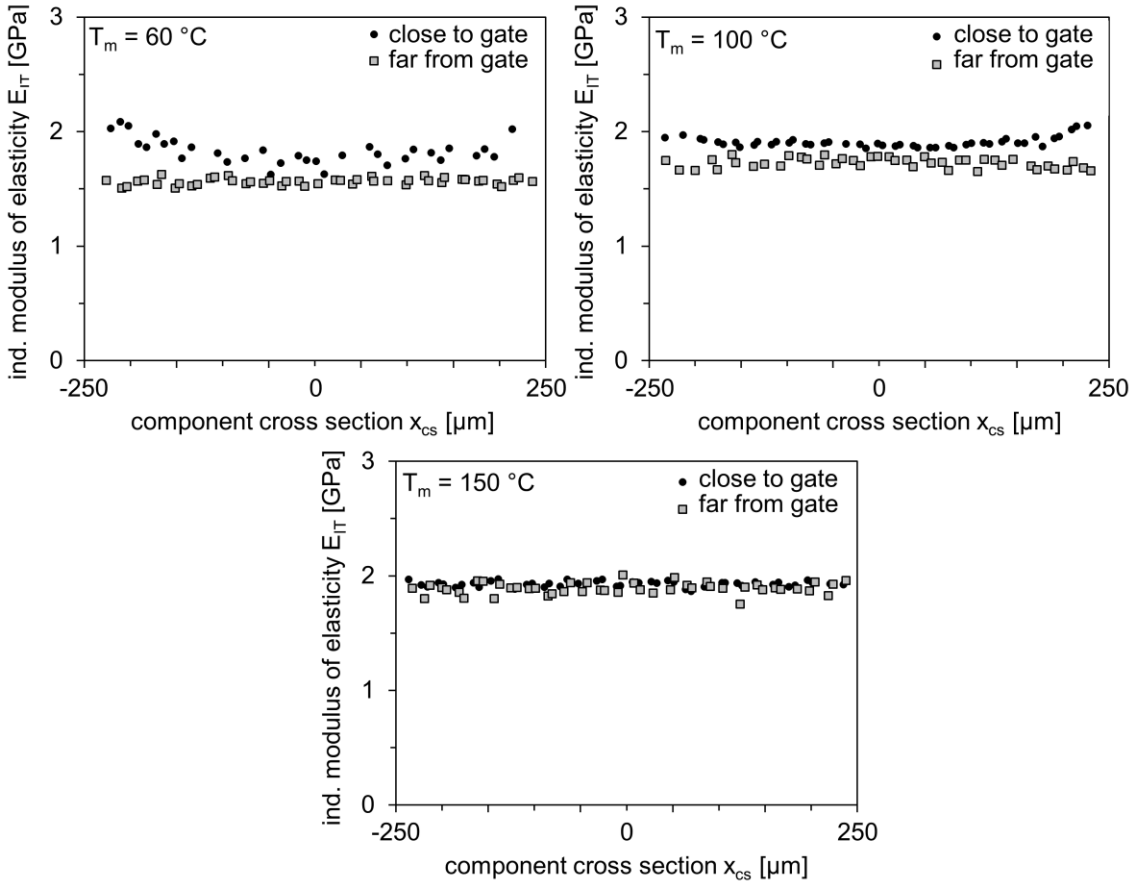


Figure 8: Nanoindentation results with regard to different component positions and mold temperatures

A mold temperature of 60 °C results in significantly different mechanical properties depending on the component's flow path length as well as the cross-section. In the skin-near areas close to the gate an approx. 30 % higher elastic

modulus is noticeable compared to the properties far from the gate. The differences decrease in the direction of the component core area, whereby also here higher mechanical properties are measured compared to positions far from the gate. The areas far from the gate show relatively homogeneous elastic modulus over the entire specimen cross-section. It is assumed that the high elastic modulus in the component's edge areas close to the gate can be correlated with the shear affected oriented skin layer, as explained for the mechanical tensile tests.

Increasing the mold temperature to 100 °C also leads to a comparatively inhomogeneous property profile in the areas close to the gate, whereby the highest mechanical characteristics are again measured in the edge areas. Again, the elastic moduli close to the gate are slightly higher than the values far from the gate. Due to an increase in the mechanical properties of the elastic modulus at regions far from gate a more homogeneous component property profile results with regard to the flow path length. When considering the mechanical characteristics for the mold temperature of 150 °C, there are no significant differences in the measured elastic moduli over the flow path length and over the specimen's cross-section. Here, it is assumed that the more distinct internal component properties, especially in the component's core area at positions close to the gate as well as the entire cross-section far from the gate, lead to an increase in the elastic modulus. As the crystallization could take place at a high temperature and, therefore, a distinct internal property profile over the entire component's cross-sections has been developed, there is a homogeneous elastic modulus with no clear differences close to the gate. Therefore, a homogeneous property profile could have been achieved especially due to a change of the crystalline properties.

5 SUMMARY

Within the scope of the present investigations, a dynamic tempering concept with segmented temperature-controlled heating ceramics was used to produce thin-walled components with specifically homogenized component properties. For this purpose, thin-walled plates made of PA 12 were injection molded. Components were produced conventionally at 60 °C as well as with increased mold temperatures of 100 and 150 °C. Finally, the morphology, the degree of crystallization as well as mechanical properties were investigated over the component's flow path length.

At a relatively low temperature of 60 °C, which is within the temperature range specified by the material manufacturer, clear inhomogeneous internal and mechanical properties are measured over the flow path length. Here, especially at positions far from the gate a drastic drop in stiffness and strength exists. With increasing mold temperature, it is possible to achieve a homogenization in internal properties. Consequently, the resulting mechanical component

properties also show a clear homogenization over the entire component geometry and especially at component areas far from the gate. Here, a significant increase in stiffness and strength is being measured. Due to the low volume flow at these positions, the orientated layers are less pronounced. Therefore, the crystalline properties and less the orientations show the most important influence on the development of the mechanical properties at positions far from the gate. Finally, for the used PA 12 the production of thin-walled components with temperatures given by the material supplier is not recommended. Here, especially for providing a save component design a higher mold temperature in the range of the stable γ-modification and, therefore, above 100 °C is recommended.

In further investigations, other semi-crystalline thermoplastics with different crystallization kinetics should be investigated with regard to their local properties. Furthermore, to realize a complete reduction of the oriented skin layer, temperatures higher than the crystallization temperature range should be used as mold temperatures to investigate if a complete homogenization and isotropic component behavior can be achieved.

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Autor / author:
Dr.-Ing. Christopher Fischer
Prof. Dr.-Ing. Dietmar Drummer
Lehrstuhl für Kunststofftechnik
Friedrich-Alexander Universität
Am Weichselgarten 9
91054 Erlangen

E-Mail: drummer@lkt.uni-erlangen.de
Webseite: www.lkt.uni-erlangen.de
Tel.: +49 (0)9131/8529700
Fax: +49 (0)9131/8529709

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E-Mail: info@hanser.de

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Dr.-Ing. Eva Bittmann
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