



Thermo-Rheological Investigation and Modeling of the Shear Viscosity of Polypropylene above the Melting Temperature

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(Received 13 May 2013; accepted 3 December 2013)

Abstract

The increasing use of polymeric materials in the daily life, leads to challenges in the processing industry to deliver high performance materials with affordable terms. However, new processing techniques lead to high costs. In order to reduce processing costs it is necessary to understand the non-Newtonian behavior of the polymers in their molten state to be able to simulate the processes before the construction of the plants starts. Here the shear thinning behavior of the viscosity of polymeric melts is essential. Thus, this paper deals with the experimental investigation of the thermo-rheological behavior of the viscosity of one of the most used polymers (Polypropylene) over a wide range of temperatures and shear rates. Furthermore, a modeling approach of the viscosity via a generalized non-Newtonian law combined with an Arrhenius model is done.

Keywords: Polymer melt, Polypropylene, thermo-rheological modeling, generalized Newtonian fluid, Cross model, shear viscosity, shear thinning behavior, rotational rheometer, high-pressure capillary rheometer,

1. Introduction

Polymer processing techniques are divided into two fields. The first is the polymeric material production techniques e.g. deriving polymeric ingredients from by-products formed during the refining process of crude oil. However, this field is a chemical motivated field where the polymeric materials are investigated in their macro- and microscopic ingredients. In case of Polypropylene (PP) the macromolecules are set together by monomer propylene. The long molecule structures of PP are made of olefin chains and the small molecule structures are broken olefin structures and are bounded together by covalent bonding mechanisms. However, it is well known that the physical and mechanical properties of polymers are strongly influenced by the size or length of the chains. E.g. increasing the chain length leads to

enhancement of the melting temperature point due to more joints between the chains [1,2]. The chemical bonding mechanism and the ingredients of PP are shown in the chemical structural equation in Fig. 1.

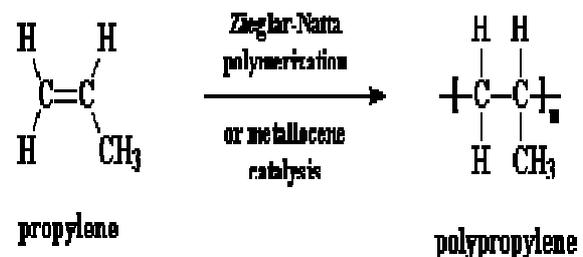


Fig. 1. Polymerization of propylene to polypropylene[3].

The melting point of polypropylene ranges from 160°C to 171°C, for perfectly isotactic PP the melting point is 171°C and for commercial isotactic PP it ranges from 160°C to 166°C. However, these melting points are an advantage

of PP in comparison with the Polycarbonates family, where the melting point is in the region of 240°C. The properties and the fields of application of PP are given in Table 1.

Table 1,
Benefits and Areas of Application of PP [1-3].

Usage benefits of PP	Areas of application
<ul style="list-style-type: none"> • act as an excellent insulator • very low moisture absorption (water resistant) • the surface structure is antibacterial • color miscible • non-toxic • low density • high toughness • stiffness and elastic flexibility at high temperatures 	<ul style="list-style-type: none"> • Electric industry • Microchip industry • clinical use • home facilities • packaging industry • toys industry • security devices in automobile and aircraft industry • lightweight technologies

Independent of all pros and cons of the chemical and physical properties of PP the material characteristic needs to be understood. Therefore the second field is the knowledge about the thermo-rheological properties of PP. This is very important for the process engineers. Producing usual PP components such as mentioned in table 1 need to be understood in such a way that the preliminaries experiments of the processes are bounded due to the reduction of cost and time. The rheological quantities are introduced in order to characterize the rheological behavior of PP as all other non-Newtonian fluids or respectively as non-Newtonian polymer melts [4-6,8,9]. However, a lot of complex phenomena are investigated in the field of non-Newtonian polymers such as: shear thinning, elastic and relaxation, elongation hardening and softening and normal stress differences behavior, refer to [20,21]. These phenomena take place in the context of simulation procedures of complex flow simulation of such processing techniques. The main information indeed is the knowledge about the shear viscosity since it is possible for small deformation rates to take the information about the elongation viscosity with the so-called Trouton-Ratio [7]. Furthermore, it is possible to introduce a time parameter with the help of a generalized Newtonian Model. It is possible to introduce big changes of the so-called zero shear rate viscosity by varying the temperature. In polymer processing this can help to save energy, since low pressure gradients in engineering

apertures are needed. Therefore, this paper deals with the thermo-rheological investigation of the shear viscosity of PP in a temperature region of 170°C to 300°C and a shear rate of 0.001 s^{-1} to 1000 s^{-1} . These regions are usually used in processing engineering.

2. Experimental Investigation

The experimental work was done with two different types of rheometer systems. The first system is specialized for low shear rates and gives little error oscillations during the moment measurements. This system is the AR-G2 rotational rheometer from the TA-Instruments company as shown in figure 2. For controlling the friction of the system during the rotation, the use of the TA-Instruments calibration software is preferred. For the preparation of the specimens the use of the heating oven and the camera was helpful. However, the preparation is done via setting the desired measurement temperature slowly, for example setting the temperature above 170°C means heating the specimen till 50°C under nitrogen atmosphere (10 L/min) with a waiting time of 10 min, then increasing the temperature by $\Delta T = 10^\circ\text{C}$ and waiting for 1 min at each of the steps till 170°C. By increasing the measurement temperature e.g. above 200°C, it is preferred to increase the waiting time, this also helps as a

drying process of the polymer specimen. In the case of Polypropylene the drying process needs to be increased at the very beginning of the

measurement processes for high temperatures. This leads to a longer waiting time at a temperature of 50°C.

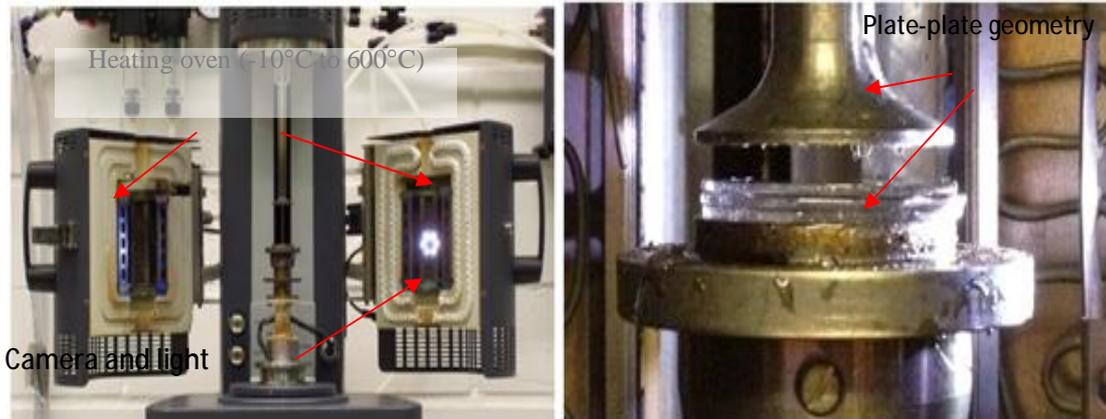


Fig. 2. AR-G2 (TA-Instruments) Rotational Rheometer with the used Plate-Plate Geometry.

In this work the so-called plate-plate geometry is used to introduce a very homogeneous specimen form, as shown in figure 3. Here some measurements were benchmarked with the cone-plate geometry. All benchmark measurements showed good agreements. The measurement of the viscosity is obtained from the given angle movement velocity Ω with

$$\dot{\gamma} = \frac{\Omega r}{h} \quad \dots(1)$$

and the measured moment M with the shear stress τ and the given shear viscosity can be calculated with

$$h = \frac{2M}{\rho r^3 \dot{\gamma}} \quad \dots(2)$$

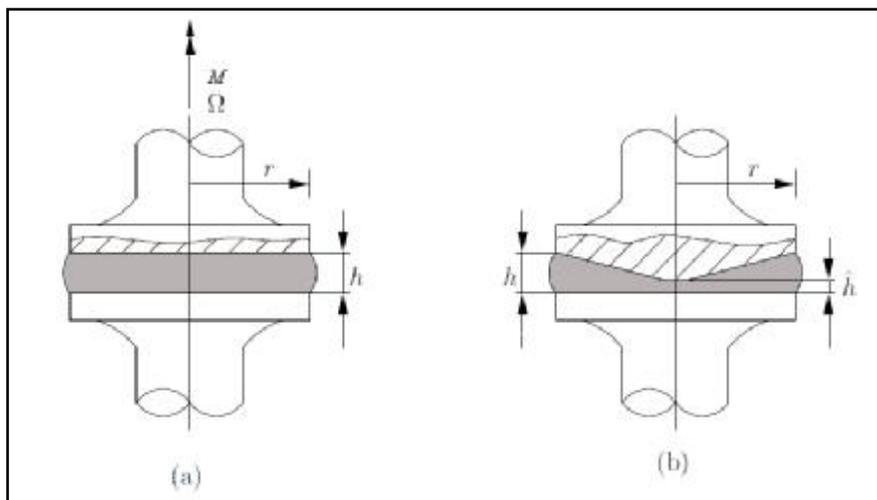


Fig. 3. Plate-Plate (a) and Cone-Plate (b) System, where M is Measured and Ω is the Given Angle Movement of the Upper Plates (r Radius of the Plate and h is the Height of the Specimen, Note $\hat{h} \ll 1$), Refer to [7].

In case of the high shear rates viscosity measurements the use of the high-pressure

capillary rheometer is important. Figure 4 shows the principle of the measurements.

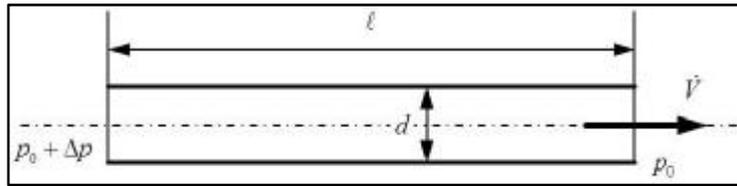


Fig. 4. Sketch of a Capillary Rheometer.

The fluid is pressed with a piston-cylinder system through a capillary tube. The difference of the pressure Δp between inlet and outlet of the tube is measured in connection with the volume flux \dot{V} , see therefore figure 4 and 5. These quantities are used to calculate two new variables, a weighted flow rate

$$q = \frac{32}{\rho d^3} \dot{V} \quad \dots(3)$$

and the wall shear stress

$$t_w = \frac{\Delta p d}{4 l} \quad \dots(4)$$

The correlation between the flow rate and the wall shear rate under stationary condition in a tube results from the balance of momentum and reads

$$q = \frac{4}{t_w} \int_0^{t_w} t^2 \dot{\gamma}(t) dt \quad \dots(5)$$

Integration by parts leads to the real shear rate at the wall

$$\dot{\gamma}_w = \dot{\gamma}\left(\frac{d}{2}\right) = \frac{1}{4} \left[3 + \frac{d(\log q)}{d(\log t_w)} \right] q \quad \dots(6)$$

This relation is called Rabinowitsch-Weissenberg-correction [19]. Afterwards the shear viscosity is calculated by

$$h = \frac{t_w}{\dot{\gamma}_w} \quad \dots(7)$$

The used apparatus is shown in figure 5. Around the specimen cylinder is attached a heating oven for temperatures between 20°C and 400°C. Furthermore, at both ends of the cylinder pressure and temperature sensors are located.

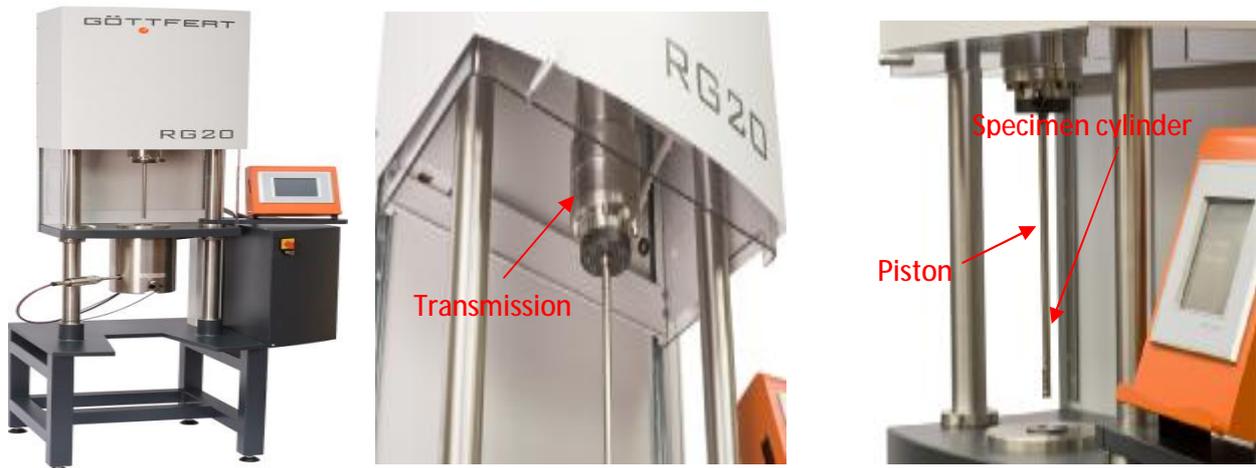


Fig. 5. Rheograph 20 (RG20 - Göttfert) High-Pressure Capillary Rheometer.

For each rheometer a minimum of 3 experiments are made for the same temperature. The results are shown in Figure 6 and 7, where the viscosity (measured for low and high shear rates) is shown over the shear rate for a given

temperature. The measurements are noted with R for rotational rheometer and K for high-pressure capillary rheometer. As shown in the figures there is a good agreement between multiple independent measurements at the same

temperature. Furthermore, the measured viscosities are obtained more successfully in the transition domain of the rotational rheometer compared with the capillary rheometer. It is also shown that the capillary rheometer has some troubles to measure the viscosity at low shear rates due to oscillations. Additionally in the case

of a temperature above 280°C and 300°C the viscosity starts to be low and the measurement via a rotational rheometer has its limitation, because here the polymer melt starts to flow out of the geometry. This leads to a decrease in the viscosity measurements as shown in Figure 7.

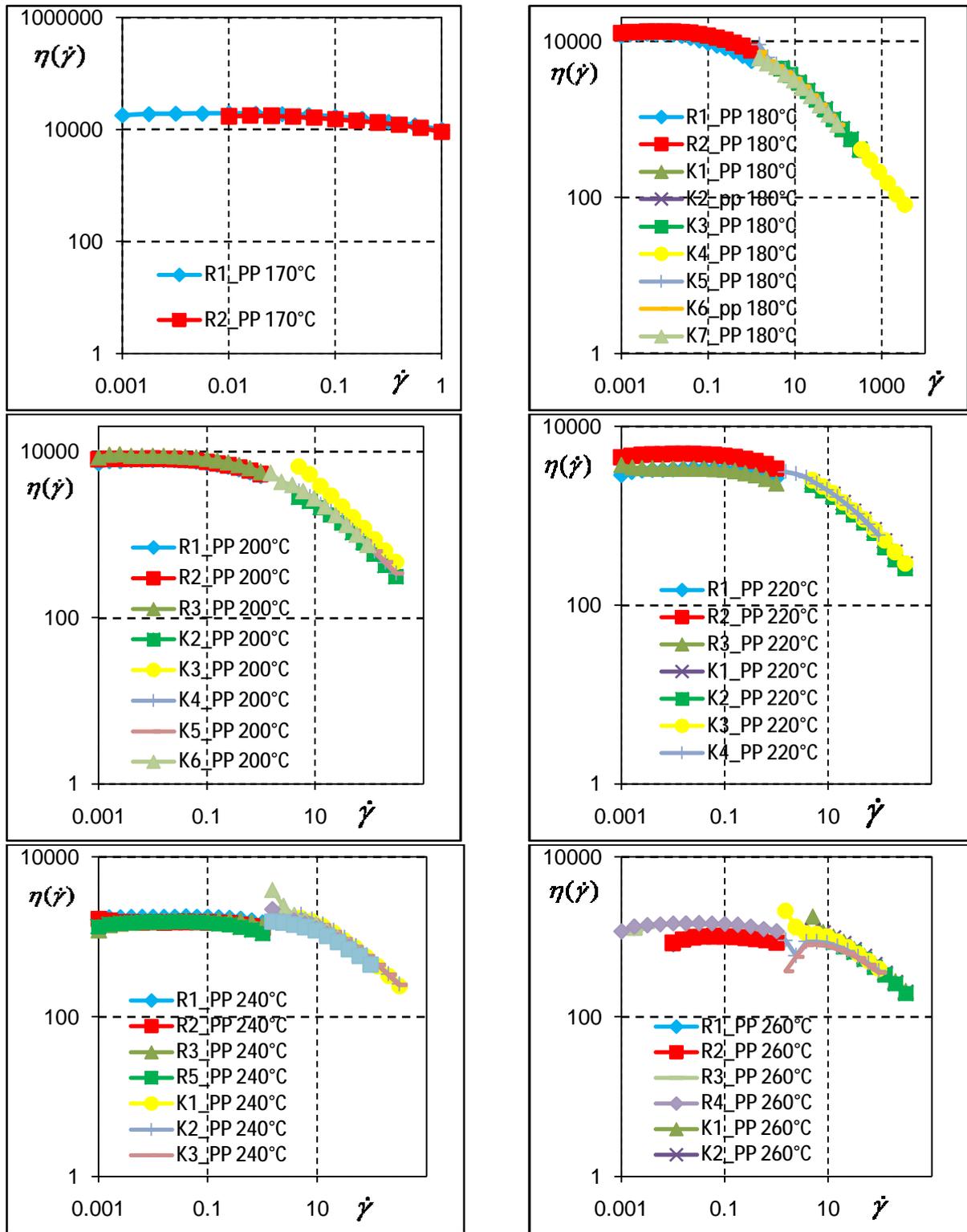


Fig. 6. Measurements of the Shear Viscosity of PP at a Temperature Region of 170°C to 260°C.

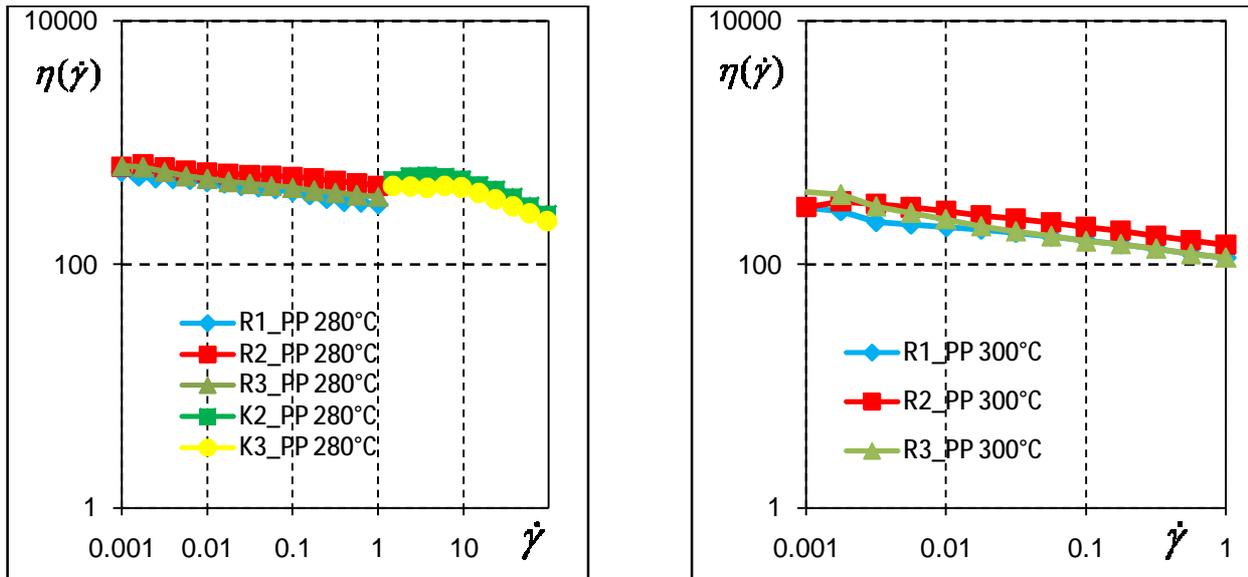


Fig. 7. Measurements of the Shear Viscosity of PP at a Temperature Region of 280°C and 300°C.

3. Modeling Approaches

Beside the experimental investigation, it is well known that the increasing use of Computational Fluid Dynamic packages (CFD) are used to understand the complex polymer melt flow in complex mechanical and processing devices which needs some usable material modeling techniques. For this aim it is essential to model the behavior of the polymeric material precisely. However, commercial CFD packages deliver only some linear models (e.g. the Newtonian Model: a linear viscose body model for viscose fluids [11,12]). For incompressible fluid flow simulations all CFD packages start from the continuity and momentum equation [7]

$$\nabla \cdot \mathbf{v} = 0, \quad \dots(8)$$

$$r \left(\frac{\partial}{\partial t} \mathbf{v} + \nabla \cdot (\mathbf{v} \otimes \mathbf{v}) \right) = \nabla \cdot \mathbf{S} + \mathbf{f}. \quad \dots(9)$$

Here \mathbf{v} is the Eulerian velocity field, r denotes the fluid density, \mathbf{S} is the Cauchy stress tensor and \mathbf{f} are body forces. In order to simulate the accurate material behavior the stress tensor \mathbf{S} needs to be modeled. Furthermore, to introduce the shear dependency in the material model in order to model the experimental observed shear thinning behavior it is possible to use a so-called Cross model. The Cross viscosity model is a good generalization of the constant Newtonian viscosity. Doing so leads to the well known structure of the Cauchy stress tensor \mathbf{S} for the incompressible fluids, refer to [8,10,13,14]

$$\mathbf{S} = -p\mathbf{1} + \mathbf{T}. \quad \dots(10)$$

Here, p denotes the static pressure and \mathbf{T} is the viscose stress tensor and can be written as

$$\mathbf{T} = 2h(\mathbf{g} \otimes \Theta) \mathbf{D}, \quad \dots(11)$$

where the viscosity depends on the shear rate \mathbf{g} and the temperature Θ . Furthermore, $\mathbf{D} = 1/2(\nabla \mathbf{v} + (\nabla \mathbf{v})^T)$ is the symmetric part of the gradient of the Eulerian velocity field $\nabla \mathbf{v}$. However, to reproduce the material behavior $h(\mathbf{g} \otimes \Theta)$ needs to be fitted on the measurements. In this work the generalized Cross model [16,17] with an Arrhenius type temperature dependency was used

$$h(\mathbf{g} \otimes \Theta) = \frac{h_0(\Theta)}{1 + (I(\Theta) \mathbf{g})^n} \quad \wedge \quad \mathbf{g} = \sqrt{\mathbf{D} \cdot \mathbf{D}}, \quad \dots(12)$$

the zero shear rate viscosity $h_0(\Theta)$ is given by the exponential Arrhenius model

$$h_0(\Theta) = h_R(\Theta_R) \text{Exp} \left[\frac{E_0}{R_0} \left(\frac{1}{\Theta + 273.15 \text{ K}} - \frac{1}{\Theta_R + 273.15 \text{ K}} \right) \right]. \quad \dots(13)$$

In eq. (13) $h_R(\Theta_R)$ is a reference viscosity at a reference temperature. However, the reference values in this work are set at the minimum measurable temperature with the experimental setup at $\Theta_R = 170^\circ\text{C}$ ($h_R(\Theta_R) = 19000 \text{ Pa}\cdot\text{s}$).

Furthermore, $E_0 = 70000 \text{ kJ/mol}$ and $R_0 = 8.314 \text{ J/(molK)}$ are the activation energy of PP and the universal gas constant. The time function $I(\Theta)$ is modeled by the same type of Arrhenius model with the reference value $I_R(\Theta_R) = 0.85 \text{ s}^{-1}$, see [15-18].

$$I(\Theta) = I_R(\Theta_R) \text{Exp} \left[\frac{E_0}{R_0} \left(\frac{1}{\Theta + 273.15 \text{ K}} - \frac{1}{\Theta_R + 273.15 \text{ K}} \right) \right] \dots(14)$$

In case of fitting the Cross Model without the Arrhenius models of eq. (13) and (14) the results are shown in fig. 8 and the parameters are given in Table 2.

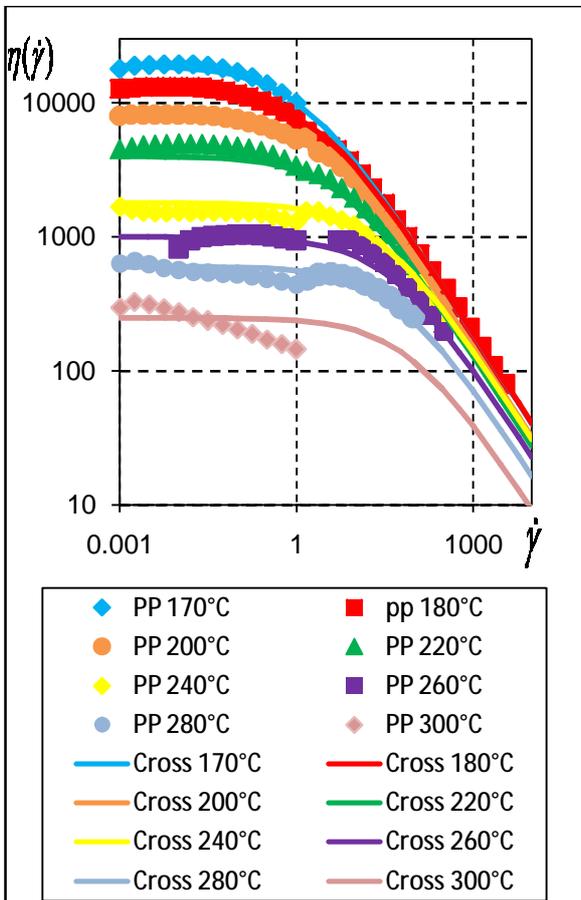


Fig. 8. Averaged Experimental Results (Symbols) and Fitted Lines with the Cross Model on Each Temperature Step without Using the Arrhenius Models.

Table 2, Parameters of the Cross Model Obtained for Each Temperature Step. Note that the * Noted Parameters are Estimated due to the Lack in the Measurements, since High Shear Measurements were not done for All Temperatures.

Θ	h_0	l	n
170	19000	0.85	0.68*
180	12500	0.45	0.68
200	8000	0.33	0.68
220	4000	0.15	0.68
240	1800	0.038	0.68
260	1000	0.025	0.68
280	600	0.019	0.68*
300	250	0.012*	0.68*

Both the zero shear viscosity and the time constant depend highly on the temperature. The exponent n is constant. Using the Arrhenius models leads to a good agreement with the Cross parameters in Table 2, this fact allows to use the viscosity model given in eq. 12, as shown in table 3 and Figure 9.

Table 3, Parameters of the Cross using the Arrhenius Models.

Θ	h_0	l	l/h_0	n
170	19000	0.85	4.474e-5	0.68
180	12493	0.5589	4.474e-5	0.68
200	5696.16	0.25483	4.474e-5	0.68
220	2767.99	0.12383	4.474e-5	0.68
240	1422.91	0.06366	4.474e-5	0.68
260	768.901	0.0344	4.474e-5	0.68
280	434.404	0.01943	4.473e-5	0.68
300	255.402	0.01143	4.475e-5	0.68

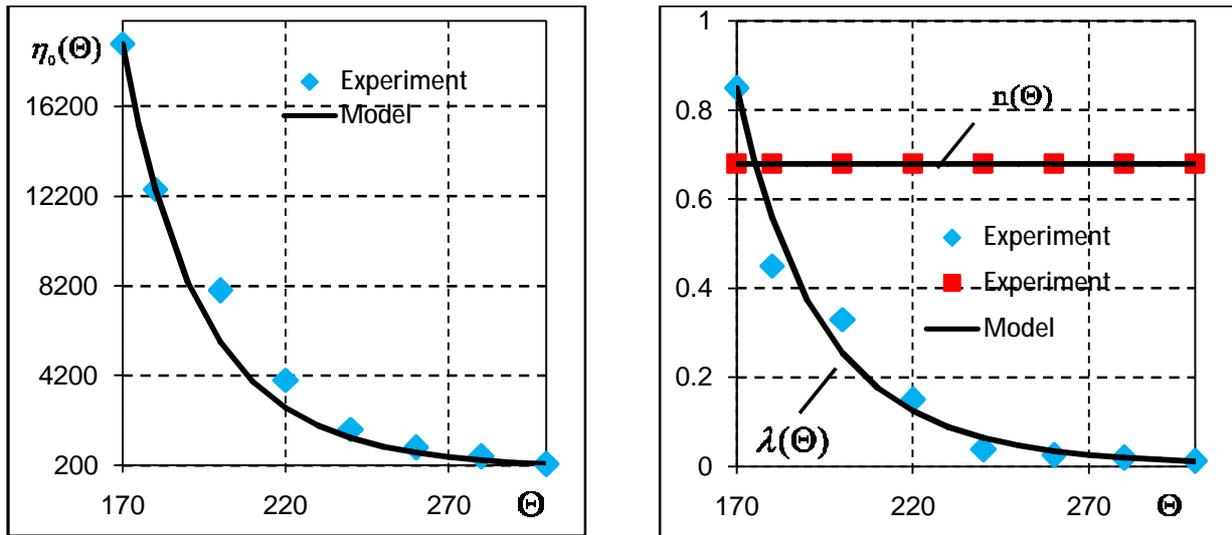


Fig. 9. The Arrhenius Models used for Fitting the Zero Shear Rate Viscosity and the Time Parameter *l* of the Cross Model.

A deeper analysis of the experimental data in table 3 shows, that the quotient of the time function and the zero viscosity for each temperature is constant. Then it is possible to rewrite the Cross model (12) in the form

$$\frac{h(\dot{\gamma}, \Theta)}{h_0(\Theta)} = \frac{1}{1 + (C \dot{\gamma} h_0(\Theta))^n} \quad \dots(15)$$

$$C = \frac{l(\Theta)}{h_0(\Theta)} = 4.474 \cdot 10^{-5} = const.$$

With this relation the shear behavior of PP for all melt temperatures can be described by a master viscosity function [18] in dependence of the product of shear rate and the related temperature dependent zero viscosity plotted black in Figure 10.

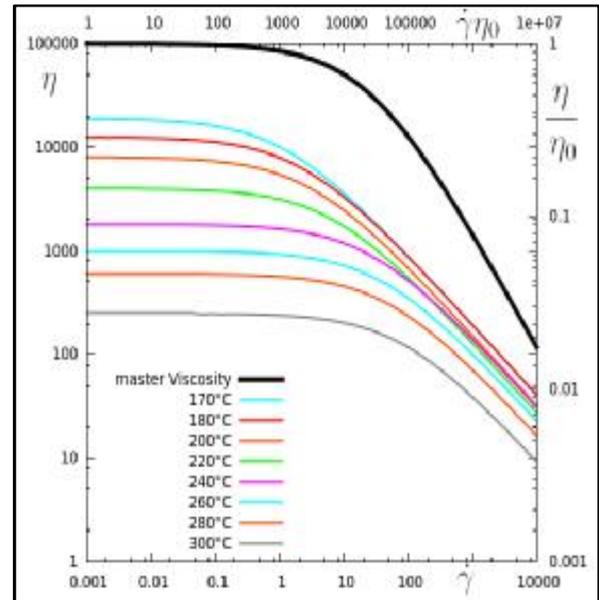


Fig. 10. The Master Viscosity Curve (Black Line) of the Measured Temperature and Shear Dependent viscosity. Note that here the Cross Model Curves where used from Figure 6.

As shown in figure 10 the mastered Cross model is able to reproduce the viscosity function in the whole temperature interval with one constant, see therefore table 3. This approach allows the modeling and simulation of isothermal production processes with a dimensionless parameter set. Furthermore it deliver first information and predictions of the thermo rheological process simulation.

4. Discussion and Conclusions

In order to characterize the thermo-rheological behavior of polymer melts in a wide area of shear rates it is necessary to use a multi set of experimental stations, e.g. the rotational rheometer and the high-pressure capillary rheometer. Furthermore, it is important to make the measurements in a nitrogen atmosphere in order to avoid oxidation processes of the polymer molecules. In this work the use of 10 L/min volumetric flow rate of nitrogen shows good results and it was not necessary to increase this rate. For high temperatures the high-pressure capillary rheometer was not able to measure the shear viscosity due to the fact of low zero shear rate viscosity. However, it is very hard for the rotational rheometer to get better results, due to the fact of drifting phenomena out of the plate-plate geometry. It is also supposed that Polymer melts show the same softening e.g. shear thinning behavior for high shear rates even at different temperatures.

In order to characterize the thermo-rheological behavior it is important to understand the so-called preloads techniques of the rotational rheometer to introduce a stationary shear flow in the polymer sample. Doing so leads to better results at the very beginning of the measurements or in e.g. at very small shear rates. Furthermore, for measuring the gap between the rotational rheometer and the capillary rheometer it is important to know that the capillary rheometer measurements takes incredible experimental time and the fluctuation of the zero shear rate viscosity. Due to these facts it is more useful to start the capillary measurements in a shear rate region above 5[1/s]. By increasing the shear rate, the capillary rheometer starts to return good results without oscillating.

The modeling approaches for isothermal and non-isothermal processes can be used to provide a close representation of the measurements. In fact it is possible to use the mastered Cross model both for isothermal and non-isothermal processes. In

case of using the Arrhenius model for the temperature dependent zero viscosity it is important, beside the continuity- and momentum equation (8) and (9), to take the energy equation into account.

It is well known that polymeric melt shows also so-called elongation hardening and softening behavior. Furthermore, it is known that such melts are highly elastic and this needs to be modeled via a viscoelastic Maxwell-like model. Here, the shear viscosity modeling would be implemented in the viscoelastic model without change. Therefore equation (11) would be extended with the elastic contributions. It is also important to model the real relaxation time, because the time constant in equation (12) and (14) is only mimicking a fluid own time where the shear thinning behavior starts to occur. In this way it is not a real relaxation time. However, this would be included in future work.

Acknowledgments

The authors would like to thank M. Eigenbrod and S. Descher (University of Kassel) for the careful experimental investigation of PP at the high-pressure capillary rheometer and the helpful work in the laboratory. Furthermore, the authors thank the German research Foundation (DFG) and the University of Bagdad for the financial support.

List of Symbols

$\dot{\gamma}, \dot{\gamma}_w$	Shear rate [s^{-1}]
r	The radius of the plate-plate geometry and the sample [m]
Ω	Angle velocity of the plate-plate geometry [radius/s]
h	Hight of the sample [m]
$\eta, \eta(\dot{\gamma})$	Viscosity and the viscosity function [Pas]
M	Measured moment [Nm]
q	Weighted flow rate [s^{-1}]
\dot{V}	flow rate (volume flux) [m^3/s]
d	Diameter of the tube of the capillary rheometer [m]
t, t_w	Shear stress and wall shear stress [N/m^2]
Δp	Pressure gradient [bar]
l	Length of the tube of the capillary rheometer [m]
ρ	Density of the fluid [kg/m^3]

\mathbf{v}	Euler velocity field [m/s]
\mathbf{S}	Cauchy Stress tensor [N/m ²]
\mathbf{f}	Specific volume forces [Nm ³ /Kg]
$\mathbf{1}$	Unity Tensor
p	Pressure [bar]
\mathbf{T}	Extra Viscous Stress tensor [N/m ²]
\mathbf{D}	Rate of deformation tensor [s ⁻¹]
Θ	Temperature [°C]
$h_0(\Theta)$	Zero shear rate viscosity
$h_R(\Theta_R)$	Reference viscosity
$I(\Theta)$	Time parameter function [s]
$I_R(\Theta_R)$	Reference time parameter
E_0	activation energy constant of PP [kJ/mol]
R_0	the universal gas constant [J/mol K]
Θ_R	Reference temperature

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التحقيق الحرارية الريولوجية ونمذجة لزوجة ألقص من ألبولي بروبيلين فوق درجة الأانصهار

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الخلاصة

يؤدي الاستعمال المتزايد للمواد البوليمرية في الحياة اليومية، إلى تحديات كثير في مراحل التصنيع لتقديم مواد عالية الأداء ضمن شروط معقولة. ومع هذا، فإن تقنيات المعالجة الجديدة تؤدي إلى ارتفاع التكاليف. ومن أجل الحد من الارتفاع في تكاليف الإنتاج، من الضروري فهم السلوك غير النيوتوني للبوليمرات في حالتها المنصهرة لتكون قادرة على محاكاة العمليات الإنتاجية قبل البدء بالخطط الإنتاجية. وفي هذه الحالة فإن سلوك قوى القص لطبقة البوليمر الرقيقة المنصهرة هو أمر أساسي. وبالتالي فإن هذه البحث يتناول دراسة السلوك الحرارية الريولوجي للزوجة احد البوليمرات الأكثر استعمالاً (البولي بروبيلين) على نطاق واسع من درجات الحرارة ومعدلات القص. علاوة على ذلك، يتم إتباع موديل رياضي للزوجة من خلال القانون الحالة غير النيوتونية إضافة الى موديل أرينيوس .