



# Effect of acoustic excitation on fiber-reinforced polypropylene and the influence on melt viscosity

Michael Heinrich<sup>1</sup> · Ricardo Decker<sup>2</sup> · Paul Reindel<sup>2</sup> · Katja Böttcher<sup>2</sup> · Isabelle Roth-Panke<sup>2</sup> · Lothar Kroll<sup>2</sup>

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## Abstract

The paper describes a novel technological approach to influencing the rheological properties of thermoplastic materials exposed to acoustic energy. The flow behavior of polypropylene with different mass percentages of glass fibers is investigated in a parallel plate rheometer under high-frequency longitudinal excitation. The influence of oscillation frequency on the melt viscosity is explained by means of shear thinning criteria. The dependence of the oscillation shape using sinusoidal excitation on shear thinning as a function of different fiber reinforcement percentages is also investigated. A phenomenological view describes the mutually influencing parameters with regard to different material compositions and different excitation frequencies over the time course of the rheometric measurement. Interacting relationships are analyzed and discussed and the potential of the actuator system to influence the plastic melt is worked out. Based on this, a technological approach follows which describes the transfer of an oscillating mold surface to plastics processing methods, which, especially in the case of energy-intensive injection molding technology, leads to the expectation of possible resource efficiency in energy and material.

**Keywords** Piezoceramic actuator · Acoustic excitation · Fiber-reinforced plastic · Melt viscosity

## 1 Introduction

The anisotropic property potential of fiber-reinforced plastic composites allows the force-flow-compatible exploitation of the extraordinary material potential. However, the load path-oriented design of injection-molded structural components with short-fiber reinforcement requires the targeted manipulation of the flow behavior of the thermoplastic melt during the injection molding process, since the fiber orientation is dependent on the orientation of the melt flow and the viscosity.

Today, advanced technologies in the plastics industry are increasingly working at their performance limits, as both economic and ecological constraints are accompanied by ever-narrower process windows. This also applies to plastics processes, such as the classic injection molding process with conventional thermoplastics, which is largely mastered in terms of

material processing and process control. However, such processes are very energy-intensive, since cyclical operation requires a continuous energy input with simultaneous high waste heat during heating and cooling of the plasticizing unit and the injection mold [1]. As a result, the injection molding process in particular exhibits considerable efficiency weaknesses, which can be counteracted, e.g., by combining two manufacturing processes [2], by supporting intelligent optimization algorithms [3] or by using functional tool systems in the process [4].

One possible approach to reducing overall energy consumption is to actively influence the melt flow in order to improve flowability in the injection mold and reduce energy-intensive process parameters such as pressure and temperature. Therefore, the present study describes the active acoustic excitation of the polymer melt for the targeted change of the viscosity on the basis of special ceramic piezo elements made of PZT (lead zirconate titanate).

The coupling of acoustic energy into processes in plastics processing is already being implemented in many different areas. For example, ultrasonic welding is used to join two plastic components together [5] or to emboss microstructures into a polymer substrate [6]. The manipulation of process parameters that influence the melt flow while injection

✉ Michael Heinrich  
michael.heinrich@fh-zwickau.de

<sup>1</sup> Department for Polymer Technology and Processing, University of Applied Science Zwickau, 08056 Zwickau, Germany

<sup>2</sup> Institute of Lightweight Structures, Chemnitz University of Technology, 09107 Chemnitz, Germany

molding, and in particular the active improvement of the flowability of the polymer melt by oscillating support (e.g., by shakers or ultrasound) during the process has already been the subject of numerous scientific investigations [7, 8]. The aim is both to improve the mechanical properties of the injection-molded structure and to optimize the injection molding process [9]. For example, manufacturing studies on microinjection-molded tensile test specimens made of CNT-filled polypropylene compounds show that an improvement in global stiffness is achieved by, among other things, increasing the injection speed, which is attributable to the orientation of the CNTs in a pronounced shear zone [10, 11]. Moreover, it has been shown that a superimposition of vibrations during the injection process causes a homogenization of the fiber-reinforced plastic compound as well as an increase in material density. In addition, the rheology of polymers depends not only on temperature and pressure but also on the vibration frequency and amplitude. Thus, among other things, speed-sensitive processes that depend on viscosity and relaxation kinetics can be influenced, such as nucleation and crystal growth, blending, and orientation. In particular, the introduction of ultrasonic energy into the polymer melt superimposes an oscillating shear stress on it, which lowers the viscosity and increases the flow rate [12]. The induction of friction between the polymer molecules also heats the melt locally and thus improves its flowability [13]. In addition, resource-efficient production is aimed for, which is achieved, among other things, by minimum energy input while complying with quality requirements [14, 15].

Martins and Covas investigated the influence of ultrasonic energy on the viscosity of polypropylene in a capillary rheometer. The results show that a reduction in viscosity only occurs at low shear rates when exposed to ultrasound at 20 kHz. However, a phenomenological consideration of the effect based on the analysis of different types of plastics with a variation of the excitation frequency was not carried out [16].

This article discusses the in situ control and manipulation of the rheological properties of the melt and thus the reduction of its viscosity in order to be transferred to injection molding applications by reducing the internal shear resistance and the frictional resistance between melt and mold wall. For this purpose, the viscosity was determined with a parallel plate rheometer in a shear rate controlled oscillating experiment under the influence of acoustic movement through the lower plate expanded with a piezoceramic actuator. As a result, the piezoelectric longitudinal (d33) working mode and its effect on the viscosity of polypropylene were proven.

Due to a local influence on the viscosity and thus the fiber architecture in the lightweight structure, a large number of process parameters influence each another. Mastering these complex processing-structure-property relationships requires the use of methods of artificial intelligence (AI). In addition, such technical system expansions can have the potential to

uncover new fields of research [17] under the influence of artificial intelligence (AI).

## 2 Experiments

### 2.1 Materials and samples

Two different polymers, Moplen HP501H and HP500V (LyondellBasell, Rotterdam, Netherland), were used for the experiments. Both types are widely and commonly used industrial polypropylene (PP) homopolymers. They differ in their molecular weight and thus in mechanical and thermal properties as well as viscosity. The melt flow indices (230 °C, 2.16 kg) are 2.1 g/10 min for Moplen HP501H and 120 g/10 min for Moplen HP500V. At 230 °C, the zero shear viscosity of the two types are 5050 Pas (HP501H) and 95 Pas (HP500V).

The determination of the molar mass of the two investigated polypropylenes was performed via high-temperature size exclusion chromatography (HT-SEC) measurements (PL\_GPC220, Polymer Laboratories, Church Stretton, UK) coupled with MALS (DAWN Heleos-II, Wyatt Technology Corporation, Santa Barbara, CA USA), using two GPC separation columns of 300-mm length, 7.5-mm diameter, and 13- $\mu$ m particle size (PLgel\_Olexis, Agilent Technologies, Santa Barbara, CA USA). 1,2,4-Trichlorobenzene stabilized with 0.1% butylated hydroxytoluene was used as eluent. The measuring and dissolution temperature was set to  $T = 150$  °C and a flow rate of  $v = 1.0$  ml/min. Table 1 contains the results of the molecular weight analysis. The number average molar mass  $M_n$ , the mass average molar mass  $M_w$ , and the polydispersity  $M_w/M_n$  were determined in a double determination.

As expected, the materials show significant differences in molecular weight. Similar to the zero shear viscosity, the molar mass of the HP500V is clearly lower than the molar mass of HP501H. However, the differences in the polydispersity are minor.

In addition to the dependence on the molecular weight of the polymers, the dependence of the acoustic-based manipulation of the flowability on the filler content is analyzed. For this purpose, the two base materials, Moplen HP500V and Moplen HP501H, were blended with two different mass

**Table 1** Molar mass distribution

Material	$M_n$ [g/mol]	$M_w$ [g/mol]	$M_w/M_n$
HP500V	39600	166000	4.19
	38400	166000	4.32
HP501H	111000	384000	3.46
	111000	383000	3.45

fractions of glass fibers (GF). Thus, fiber-reinforced polypropylene compounds with 40 wt% and 60 wt% of glass fibers were produced.

For the rheological tests, suitable samples of the polymers were manufactured by microinjection molding. The sample size has to correspond to the rheological measuring geometry. Thus, the samples are discs with diameter of 20 mm and a height of 1.6 mm. Depending on the fiber mass fraction, the mass of a sample is between 0.45 and 0.74 g.

In summary, six different materials were tested, a low-viscosity and a high-viscosity propylene, each containing 0 wt%, 40 wt%, and 60 wt% glass fibers respectively.

## 2.2 Setup and equipment

The complex viscosity of the investigated thermoplastic polymers was measured with a rotational rheometer (HAAKE MARS III, Thermofisher Scientific, Waltham, MA USA) equipped with parallel plates and modified measuring geometries. A piezoelectric actuator module was added to the stationary lower plate. The module consists of a circuit board and a piezoceramic disc. The circuit board serves as an electrical insulation layer to the rheometer plate and connects the actuator's electrodes to the piezo controller (HVPZT-Amplifier E508.00, PI Ceramic GmbH, Lederhose, Germany) via temperature-resistant wires. The rotating upper plate was also modified with an insulation layer. During the viscosity measurement, the thermoplastic sample was placed between the insulation plate of the upper rheometer plate and the piezoceramic disc of the actuator module. This test setup is shown in Fig. 1.

The piezo ceramic actuator disc consists of the modified lead zirconate titanate PIC300 (PI Ceramic GmbH). This ferroelectrically hard PZT has a very high Curie temperature of 370 °C and is applicable for operation temperatures up to 250 °C permanent and 300 °C temporary. Thus, the use under temperature loads inside the heating chamber is possible and the direct contact with the hot polymer melt does not thermally damage the ceramic material. The actuator has a diameter of 25 mm and a thickness of 250 µm. Circular electrodes with 20-mm diameter are placed concentric on both sides. In this configuration, the actuator oscillates in thickness direction based on the longitudinal (d33) working mode.

The circuit board on the lower plate and the insulation layer on the upper plate are from a hydrocarbon ceramic laminate (Rogers 4350B, Rogers Corporation, Chandler, AZ USA) with a thickness of 1.5 mm. The circuit board has a diameter of 30.4 mm and is equipped with golden electrodes for the connection between the piezo ceramic actuator and the wires. The upper insulation plate has diameter of 20 mm and thus the same dimension as the rheometer plates.

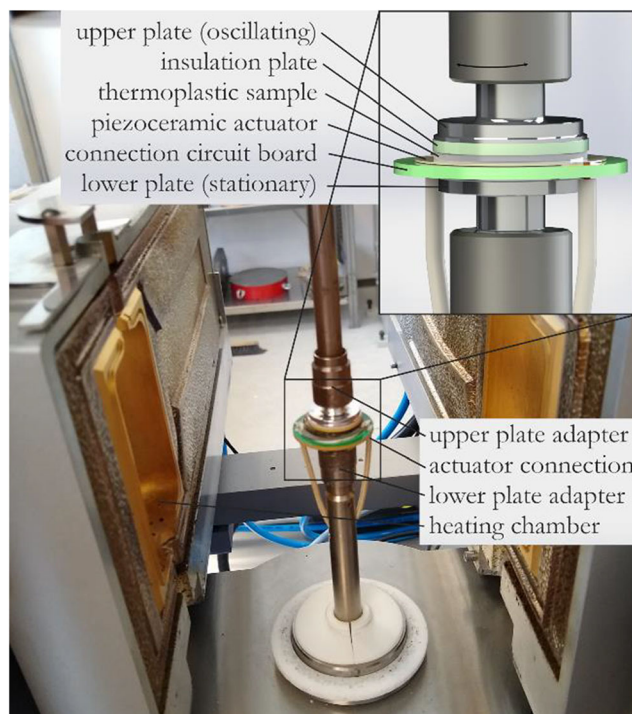


Fig. 1 Test setup of piezo adapted parallel plate rheometer

## 2.3 Test procedure

In order to determine the influence of the excitation through the piezoceramic actuator on the melt flow behavior, the complex viscosity  $|\eta^*|$  of the melt was examined.

In a first preliminary test, frequency and amplitude tests without piezo-active excitations were performed to determine the test parameters. The amplitude test was used to determine the linear viscoelastic range in order to perform the following experiments without influencing the internal sample structure. With the thus determined value for the deflection  $\Phi$ , an angular frequency  $\omega$  from the zero-shear viscosity plateau was selected after the frequency test had been performed, at which no change in viscosity occurred as a function of frequency.

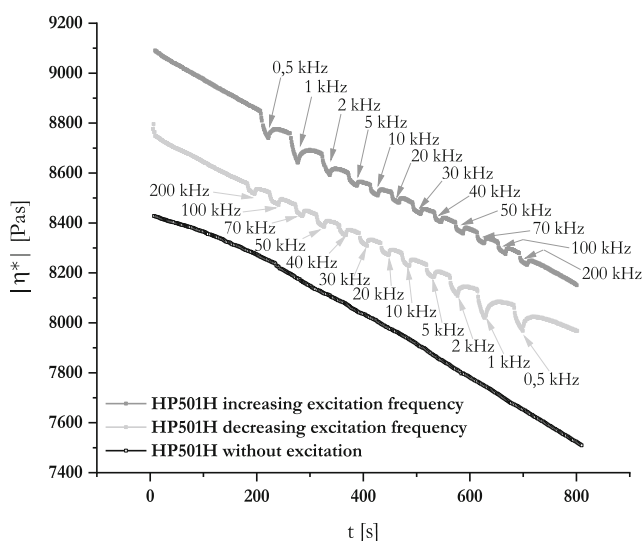
In order to determine the thermo-rheological behavior of the PP melt over the test duration of approx. 800 s, a second preliminary test was performed. For this purpose, reference curves of all investigated materials were recorded. The tests were done using the complete test setup and test conditions, but without the piezo-active high-frequency excitation. It could be seen that both polypropylene types underwent thermally induced degradation over time due to the influence of temperature. Subsequent to the reference measurements, first sinusoidal vibrations were applied by the piezoelectric actuator to the polymer melt during the viscosity measurement. The oscillation frequencies of the piezoelectric actuator were sequentially varied between 0.5 and 200 kHz. After a short excitation that resulted in a viscosity decrease, the excitation was stopped until the viscosity curve rose to a base level. Then

the next excitation frequency was applied. The measurements were performed with increasing and decreasing frequency steps. Figure 2 exemplarily shows a reference measurement without excitation and two measurements with piezo-active excitation of Moplen HP501H without glass fibers.

The reference curve runs parallel to the baseline of the others so that an additional damage to the polymer chains by the high-frequency excitation can be excluded. The differences in the viscosity level between the reference curves and the curves with high-frequency excitation are caused by typical experimental fluctuations. The viscosity decrease at each excitation frequency is in the same scale, irrespective of the start time of the excitation. For the exemplary curves in Fig. 2, at 1 kHz, the viscosity decreases by 1.1% if the excitation is started after 250 s and by 1.2% if the excitation is started after 610 s.

In addition, the heating of the polymer melt due to the energy input of the piezo-active excitation was investigated. The melt temperature was measured with a thermocouple inside the measuring gap. Short excitations did not lead to a rise of the melt temperature. Excitations longer than 15 s caused an increasing temperature, which lead to an additional decrease of the melt viscosity.

Based on the preliminary tests, the test procedure for the determination of the influence of the piezo-active excitations was defined. All test series were performed with a deformation preset in oscillation with a constant amplitude of  $\Phi = 0.1$  rad in the linear viscoelastic region of the polypropylene melt and a constant angular frequency of  $\omega = 6.2832$  rad/s at a measuring temperature of  $T = 230$  °C  $\pm$  0.5 K. The measuring gap height was set to 1.3 mm. For acoustic excitation of the polymer melt, the sinusoidal voltage amplitude was set to 500 V, which lead to a theoretical nominal displacement of the piezoceramic actuator of 77.5 nm. Furthermore, twelve



**Fig. 2** Complex viscosity of Moplen HP501H over the testing duration with and without acoustic excitation

oscillation frequencies between 0.5 and 200 kHz (cf. Fig. 2) were defined. Each sample was excited in all oscillation frequencies in ascending order. The acoustic excitation did not cause additional damage to the polymer. Hence, this procedure can reduce the experimental effort and the influence of sample fluctuation. Each frequency was applied for 15 s to prevent an increase in temperature. Between two frequencies, the excitation was interrupted until the viscosity curve rose to a base level.

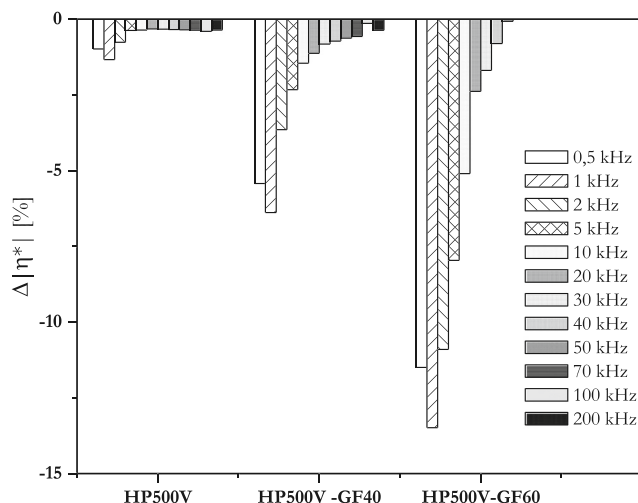
### 3 Results

To allow comparability of the different materials, the change in complex viscosity caused by the high-frequency excitation of the piezo actuators was calculated as a percentage of the baseline. Due to this procedure, the thermally induced polymer chain degradation or the different initial viscosities had no effect on the evaluation.

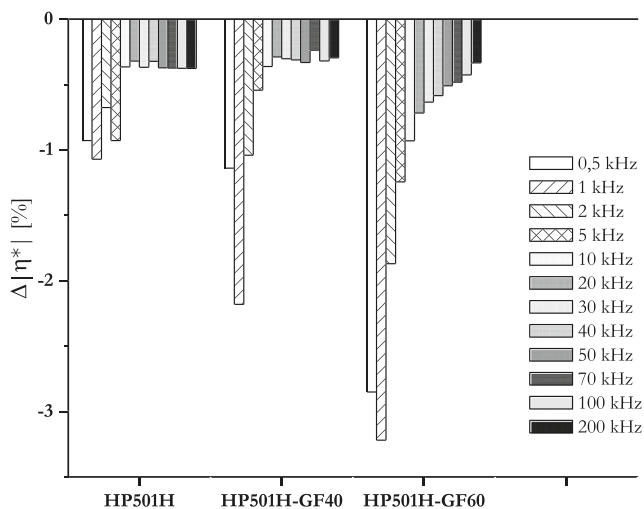
#### 3.1 Low-viscosity Moplen HP500V

The acoustic excitation of the low-viscosity polymer melt leads to a clear decrease of the melt viscosity. The magnitude of reduction depends on the excitation frequency and the glass fiber mass fraction. Figure 3 shows the results for Moplen HP500V without fibers (HP500V), with 40 wt% glass fibers (HP500V-GF40) and with 60 wt% glass fibers (HP500V-GF60) as a function of excitation frequency.

The viscosity reduction shows a clear dependence on the fiber content of the material. Without fiber reinforcement, the maximum decrease is 1.34%. In contrast, the viscosity decreases by up to 13.49% at a fiber mass fraction of 60 wt%.



**Fig. 3** Viscosity reduction of neat and glass fiber-reinforced Moplen HP500V as a function of the excitation frequency



**Fig. 4** Viscosity reduction of neat and glass fiber–reinforced Moplen HP501H as a function of the excitation frequency

Increasing the excitation frequency to 1 kHz leads to the maximum values of viscosity reduction. Further increasing up to 200 kHz results in a significant decrease of the viscosity reduction. At 200 kHz, a maximum reduction of 0.36% can be measured for the neat polymer.

### 3.2 High-viscosity Moplen HP501H

Similar to Moplen HP500V, the melt viscosity of high-viscosity Moplen HP501H can be reduced by piezo-active excitation. It also shows dependencies on fiber mass fraction and excitation frequency. The results for Moplen HP501H without fibers (HP501H), with 40 wt% glass fibers (HP501H-GF40) and with 60 wt% glass fibers (HP501H-

GF60) as a function of the excitation frequency are shown in Fig. 4.

The Moplen HP501H also shows the rising melt viscosity decrease with increasing fiber mass fraction of the materials. However, the effect is less intense than to HP500V. At 60 wt% glass fibers and an oscillating frequency of 1 kHz, a viscosity reduction of 3.22% occurs.

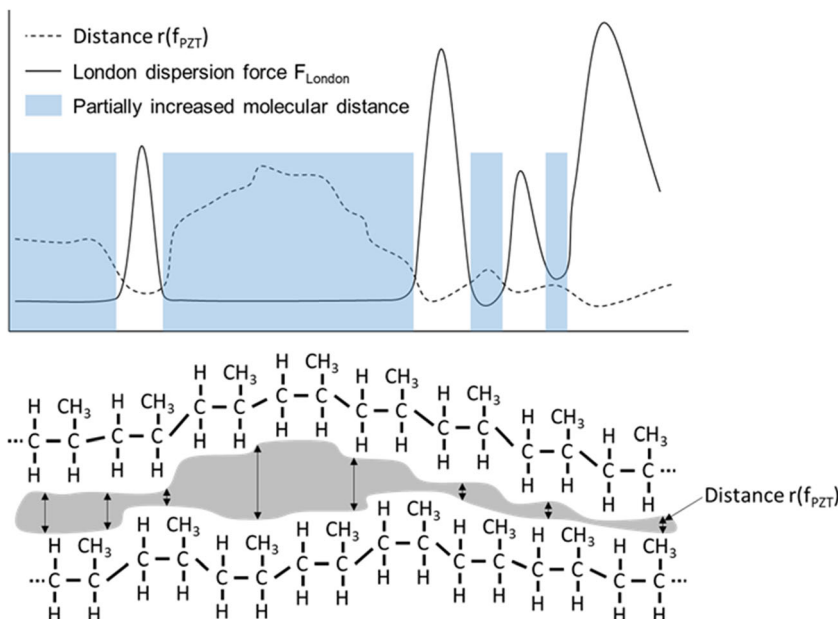
The dependence on the excitation frequency is also clearly visible. A maximum reduction of 0.38% at 200 kHz can be measured for the neat polymer.

## 4 Discussion

As shown in Figs. 3 and 4, the melt viscosity of thermoplastic polymers can be controlled by excitation with micro vibrations for unfilled and glass fiber–reinforced polypropylene types. During the short excitations, no change in the melt temperature can be measured. Thus, the decrease in melt viscosity is due to the acoustic excitation and not due to thermal influences. It is assumed that the oscillation of the polymer chains induced by the piezoelectric excitation of the actuator leads to a partially increased distance  $r(f_{PZT})$  between the polymer chains, as shown in Fig. 5. This counteracts the formation of stable intermolecular forces. Therefore, the London dispersion force  $F_{London}$  between the polypropylene polymer chains, which strongly depends on the molecular distance (cf. Eq. 1), is reduced.

$$F_{London} \sim \frac{1}{r(f_{PZT})^6} \tag{1}$$

**Fig. 5** Intermolecular interaction (London dispersion forces) and their molecular distance from each other as a function of acoustic excitation



**Table 2** Reduction in melt viscosity at an excitation frequency of 1 kHz

Material	$\Delta \eta^* $ [%] HP500V	$\Delta \eta^* $ [%] HP501H
Neat	-1.34	-1.07
GF40	-6.38	-2.18
GF60	-13.49	-3.22

All investigated materials show the best results in the frequency range between 0.5 and 2 kHz. The highest reduction in all cases occurs at 1 kHz (cf. Table 2). Thus, it can be assumed that resonances in the test samples occur and result in the higher viscosity drop.

The lower effect at higher frequencies can also be explained by the inertia of the molecular chains and the intermolecular friction. With faster oscillations of the piezoelectric actuator, the sliding of the molecular chains becomes increasingly difficult.

As a reason for the greater effect on the low-viscosity polymer, it can be assumed that the shorter molecular chains can better follow the acceleration of the oscillations. This could be due to the lower inertia of the shorter molecular chains because of the lower molecular weight and the lower intermolecular friction. In contrast, the longer molecular chains of the high-viscosity polymer have a higher inertia, higher intermolecular friction, and more entanglements. This counteracts the influence of the micro vibrations.

Commercially available glass fibers are coated with a sizing that must be compatible with a wide range of plastics. However, earlier investigations [18] show that, especially for polypropylene, the sizing and the adhesion promoters used in

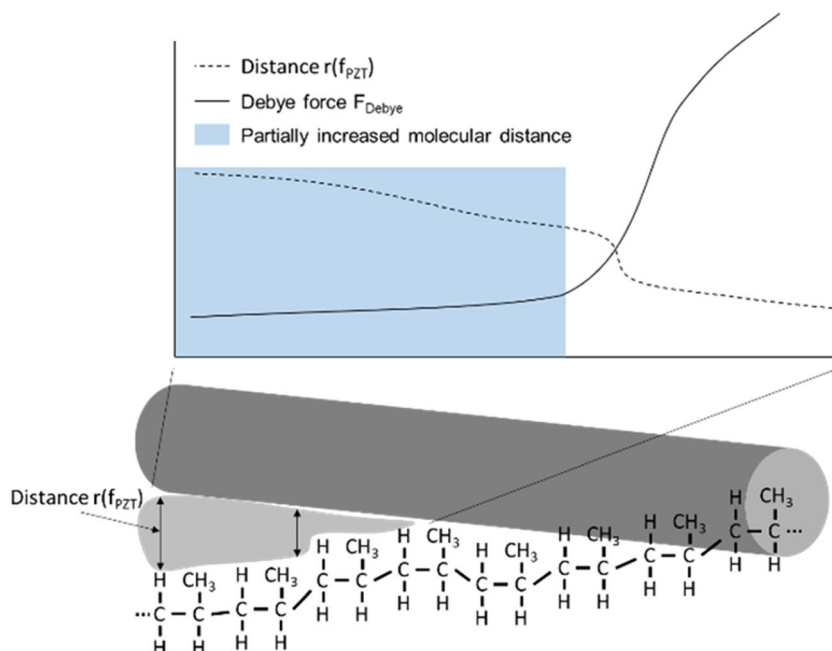
it must be adapted for good fiber-matrix adhesion. If the acoustic excitation now acts on the interface between glass fiber and polypropylene melt, the weak bonds can be loosened and the friction between the fibers and the polymer is reduced. Thus, the melt viscosity decreases. This process is reversible since commercially treated glass fibers do not form true covalent bonds between the fiber surface and polypropylene.

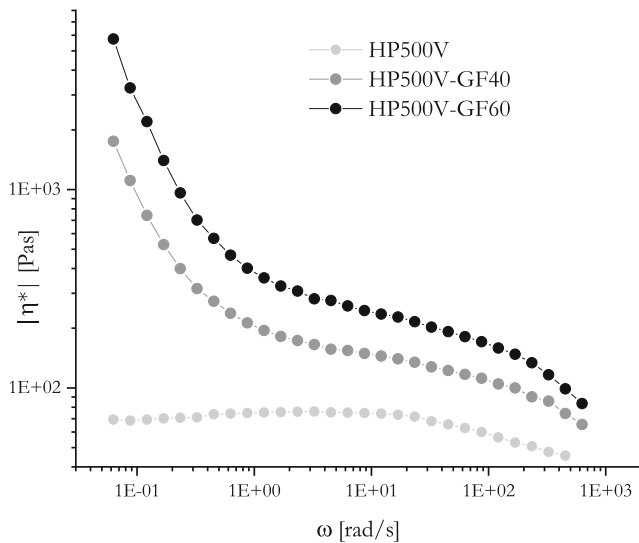
With increasing fiber mass fractions, the melt viscosity increases due to the growing interactions at the increasing interface area between the fibers and the polymer melt. Thus, when the acoustic excitation reduces the friction on the contact surface between the fibers and the polymer, the viscosity reduction increases with higher fiber contents.

It is assumed that in addition to the influence on the London dispersion force analogous to the neat polypropylene, the acoustic excitation takes also effect to the intermolecular Debye forces between the polypropylene polymer chains and the glass fiber surfaces, as shown in Fig. 6. As a result, the decrease in viscosity of the fiber-reinforced materials is higher compared to the neat polymers.

Furthermore, the acoustic excitation takes effect to both material parts, the polymer melt and the glass fibers. In contrast to the viscoelastic polymer melt, the glass fibers are solids and show lower mechanical damping to the induced oscillation. Thus, it can be assumed that the influence of the excitation on the glass fibers is higher than the influence on the polymer melt. Therefore, in total, more oscillation energy is coupled into the fiber-reinforced material. This leads to the increasing viscosity drop with increasing glass fiber fractions.

The manufacturing of the test samples by microinjection molding leads to a predominant orientation of the glass fibers in the direction of the melt flow. This alignment of the fibers

**Fig. 6** Intermolecular interaction of Debye forces and their molecular distance between glass fiber and PP as a function of acoustic excitation



**Fig. 7** Viscosity curves of Moplen HP500V with different mass fractions of glass fibers as a function of shear rate

parallel to the shear plane minimizes the influence of the fiber orientation on the viscosity function.

Thus, the fiber-filled melt behaves like a spherical suspension. The non-flowing glass fibers primarily lead to an increase in viscosity over the entire viscosity function, which rises with increasing concentration. Compared to unfilled melts, the course of the viscosity function changes significantly only at low angular velocities. At high angular velocities, the shear-thinning behavior is the same as for the base polymer. In the range of high shear rates, the filled plastic types show a pronounced structure-viscous flow behavior. This means that the viscosity drops sharply in the range of high angular velocities as a function of fiber content [19].

Figure 7 shows the shear rate dependent viscosity curves of HP500V with different mass fractions of glass fibers.

Since the solids do not experience any change in velocity in the flow in the measuring gap and only flow with the melt, the gap width for the pure liquid is reduced by a fixed value depending on the fiber mass content. This increases the actual, effective shear rate between the plates of the spherical suspension. With pseudoplastic media, such as fiber-filled polymer melts, the increasing shear rate results in a decrease in viscosity [20].

Based on this and the assumption of resonances in the oscillation of the glass fibers due to the acoustic excitation, it can be assumed that this effect of increasing the effective shear rate is increased by the acoustic excitation. The effective flow cross-section in the shear zones between the glass fibers is reduced by the oscillating glass fibers. This leads to an increasing effective shear rate of the polymer melt and results in decreasing viscosity.

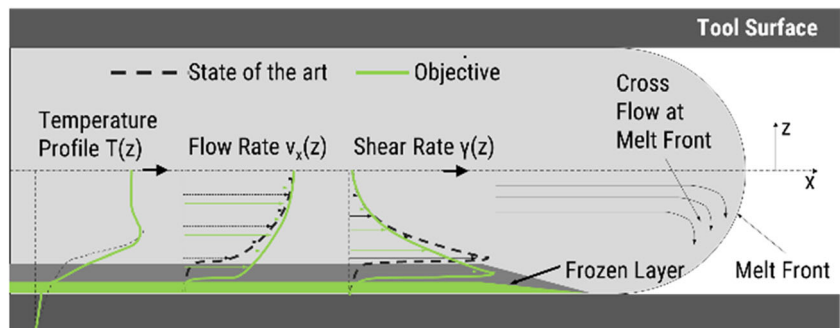
### 5 Conclusion and outlook

The experiments in this work demonstrate the possibility to manipulate the melt viscosity of neat and fiber-reinforced polypropylene. The excitation with high-frequency micro vibrations by piezoceramic actuators leads to significant viscosity reductions of up to 13.5%. The best results can be achieved with a low-viscosity polymer and high mass fractions of glass fibers. The most effective oscillation frequency is 1 kHz. The results further show that no additional material degradation occurs due to the short-term acoustic excitation. After excitation, the melt viscosity restores to the baseline.

The lower effect at higher frequencies can be explained by the inertia of the molecular chains and the intermolecular friction. It can be assumed that the effect on high-viscosity polymers is less intense than on low-viscosity polymers due to the higher inertia of the molecular chains and the higher intermolecular friction of the high-viscosity polymer. The increasing effect at higher percentages of glass fibers in fiber-reinforced polypropylene can be explained by the additional effect of loosened bonds and the decreased friction between the fibers and the polymer melt due to the acoustic excitation.

Based on the results of this work, further experiments are planned to investigate the influence of micro vibration on the fiber orientation of fiber-reinforced thermoplastic polymers. In this context, the possibility of in situ manipulation of the fiber orientation will be researched. For this purpose, a broad spectrum of polymers with different molecular structures and weights as well as different material compositions and filling degrees will be analyzed. In order to improve the effect of micro vibrations on

**Fig. 8** Melt profile in injection molding process



polymer melts, the use of other working modes of piezo ceramic actuators, such as shear modes or modified longitudinal and transversal modes, is a subject of further research. In addition, the influence of different non-sinusoidal oscillation forms, e.g., square waves or sawtooth waves, will be investigated.

Another objective is the integration of piezo ceramic actuators into injection molding tools for in situ control of the melt flow and fiber orientation during the injection molding process. The shear-thinning effect demonstrated in the paper also allows an energy-optimized shift in the temperature profile  $T(z)$  in the injection molding cavity as well as an increase in the flow rate  $v_x(z)$  as a result of the narrowing of the layer frozen on the mold wall. This also leads to a shift of the shear rate profile  $\dot{\gamma}(z)$  towards the cavity wall. The energy-efficient influence of such injection molding parameters is predicted by the authors and is shown in Fig. 8.

This complex task with its large number of mutually influencing process parameters requires new approaches for process control systems. In this context, artificial intelligence solutions will be used for the development of novel energy and resource efficient production processes and, for the first time, novel piezoelectric transducer arrays will be used in the injection molding cavity to fully exploit the lightweight construction potential of short-fiber-reinforced lightweight structures under mass production conditions.

The positive influence of in situ acoustic excitation on the viscosity of the melt also allows the technology presented in the paper to be used in microinjection molding. Since the volume-to-surface ratio of microinjected components shifts towards the surface, the already low heat capacity of the melt is quickly dissipated into the mold. A small reduction in viscosity of just a few percent already allows an expansion of the process limits, which goes hand in hand with an optimized mold filling process.

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**Author contribution** The corresponding author M. Heinrich and R. Decker have been responsible for the writing, internal review, and editing of this paper as well as for methodology and analysis, the rheology tests, set up designs, and validation of all the obtained raw data. K. Böttcher and P. Reindel have been responsible for the measurement and evaluation of the data. For supervision, funding acquisition, and project administration, L. Kroll and I. Roth-Panke have been responsible. All authors have read and agreed to the published version of the manuscript.

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**Data availability** The authors confirms that the data supporting the findings of this study are available within the paper. The raw data that support the findings of this study are available from the corresponding author M. Heinrich upon a reasonable request.

**Code availability** Not applicable.

## Declarations

**Consents** The authors consent for participation and publication.

**Conflict of interest** The authors declare no competing interests.

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