

DEVELOPMENT AND TESTING OF A NEW PRESSURE CELL FOR RHEOLOGICAL CHARACTERISATION OF POLYMER MELTS

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ABSTRACT:

A new pressure cell is described to measure the flow behaviour of polymer melts in dependence of temperature and pressure. Special attention is laid on the construction and functionality of the pressure cell. The pressure cell can be pressurized up to 120 bar and is heatable up to 260°C. As a measuring geometry a plate-plate-system is used which is capable of characterising high viscous fluids. First results with high viscous silicone oil show good agreement with known references in literature.

ZUSAMMENFASSUNG:

Es wird eine neuartige Druckmesszelle zur Messung der Materialeigenschaften von Kunststoffen in Abhängigkeit der Temperatur und des Druckes beschrieben. Besonderes Augenmerk ist hierbei auf die Konstruktion und Funktionsweise der Druckmesszelle gelegt. Die Druckmesszelle ist für Drücke bis 120 bar und Temperaturen bis 260°C ausgelegt. Als Messgeometrie wird ein Platte-Platte-System verwendet, womit es möglich ist, auch hochviskose Fluide rheologisch zu charakterisieren. Erste Ergebnisse mit einem höher viskosen Silikonöl zeigen gute Übereinstimmung mit bekannten Referenzlösungen.

RÉSUMÉ:

Une nouvelle cellule de pression est décrite dans le but de déterminer en fonction de la température et de la pression les propriétés du matériaux plastiques. Une attention particulière sera portée à la construction et au de fonctionnement de la cellule de pression. Cette cellule de pression peut être pressurée jusqu'à 120 bar et chauffée jusqu'à 260°C. L'utilisation du plaque-plaque-système pour la géométrie de mesure rend aussi possible la caractérisation des fluides très visqueux. Les premiers résultats obtenus à l'aide d'un fluide très visqueux (silicone pétrole) sont en bonne conformité avec les résultats tirés de la littérature.

KEY WORDS: rheometer, plate-plate-geometry, PDMS, temperature dependence, pressure dependence

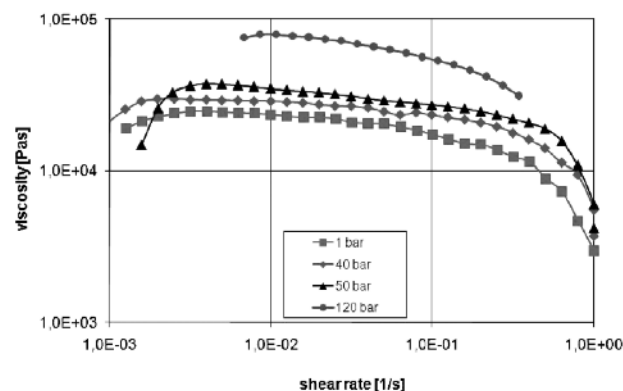
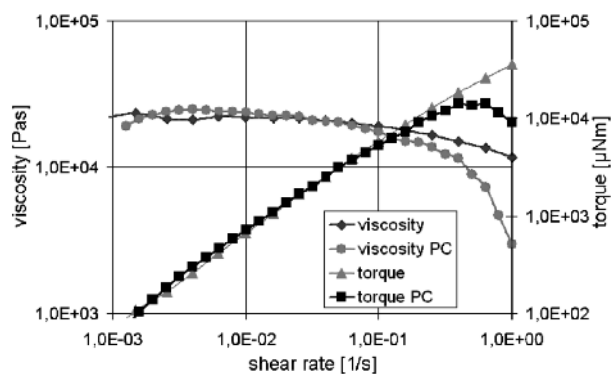
1 INTRODUCTION

Laminar flow processes are of great practical relevance in food technology, process engineering and the chemical industry. In many fluids used the viscosities are very high and often display non-Newtonian characteristics, such as shear thinning viscosity, elastic properties or normal stress differences. The material is frequently processed under a combination of high temperatures and great pressure. Thus, both temperature and pressure dependency is to be taken into account in the rheological characterization of polymer melts and high viscous silicon oils [1].

The material properties of the fluids are usually investigated under atmospheric conditions with regard to their viscous and viscoelastic properties using different equipment. In particular, the capillary rheometer and rotation rheometer, in which the fluid is pressed through a capillary or subjected to laminar flow [2], are used. For spe-

cial cases other designs like torque rheometer [3] and sliding plate rheometer [4–6] are available. Determining the dependencies of temperature, especially for the area of polymer melts is state of the art and easily possible with these rheometers.

Measuring the dependency of material properties on pressure is, however, possible only to a limited extent with the usual rheometers. There are studies in which polymer melts with dissolved gases, so-called super-critical fluids, have been investigated using capillary rheometers. The shear thinning viscosities of PDMS [7] and polystyrol [8, 9] revealed clear dependencies on the weight share of the carbon dioxide released. A disadvantage of this measuring method is that the fluid in the capillaries is exposed to a clearly higher pressure than the pressure at the end of the capillaries and thus affects the result [10]. This is not the case with rotation rheometers with concentric cylinder measuring systems [11, 12]. The development of such high-pressure Couette systems is so



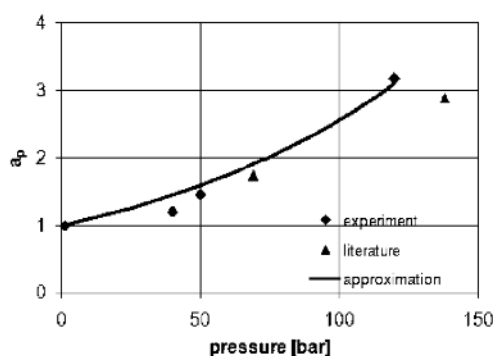
p [bar]	η_0 [Pa·s]
1	24000
40	28640
50	34770
120	76000

Figure 10 (left above): Comparative of torque and shear viscosity at 30°C under atmospheric pressure.

Figure 11 (right above): Pressure dependence of shear viscosity of PDMS at 30°C.

Figure 12 (below): Pressure shift factor for PDMS at 30°C, material coefficient $\beta = 0.0095$.

Table 1: Pressure dependence of zero shear viscosity



3 RESULTS

We use polydimethylsiloxane (PDMS) as a test fluid. This material is already fluid and highly viscous in ambient temperatures. Comparative measurements between the commercial rheometer and the PC under atmospheric pressure can be seen in Figure 10. For the plate-plate geometry with a diameter of 25 mm and a plate distance of 1 mm the difference at a temperature 30°C is hardly detectable between the torques measured in the range of $4 \cdot 10^{-3} \leq \dot{\gamma} \leq 0.1 \text{ s}^{-1}$. There are corresponding differences for the viscosity calculated in accordance with Equation 3. For higher angle velocities and so also for higher shear rates the agreement decreases: at $\dot{\gamma} = 0.25 \text{ s}^{-1}$ the deviation increases to more than 17 %, for $\dot{\gamma} = 0.63 \text{ s}^{-1}$ the difference amounts to as much as 46 %. The large deviations of viscosity at higher shear rates and the associated early reduction of the torque indicate that the gap is no longer completely filled. For the optical checking of correct gap setting the previously mentioned endoscope can be used in order to monitor the plate distance during a measurement online. The measurements shown here reproduce clearly the dependency of the viscosities on the shear rate in a range of over 2.5 decades. There is no correction of data for the plate-plate measuring geometry for higher shear rate.

The torque and the viscosity over the shear rate depending on the pressure can be seen in Figure 11. The torque and with it the viscosity increase with increased pressure. The values for zero shear viscosity under ambient pressure and at different pressure-levels are shown in Table 1. An exponential pressure function can be used to describe the pressure shift factor a_p

$$a_p(p) = \frac{\eta_o(p)}{\eta_o(p_o)} = e^{\beta(p-p_o)} \quad (4)$$

where β is a material-dependent pressure coefficient [12]. The best approximation of the experimental data succeed with $\beta = 0.0095$. In Figure 12 the factor is depicted above increasing pressure. This behaviour is confirmed by [11] where the pressure-dependent viscosity of PDMS, among other things, was measured with a falling ball rheometer. There results are also illustrated in Figure 12 and show good agreement with our experiments.

4 CONCLUSION

It has been possible to show that the newly developed and constructed pressure cell is suitable for measuring high-viscous fluids. The investigations show that the temperature and pressure inside the measuring cell are constant over any period of time. The use of magnetic coupling for contact-free transmission leads to a loss of precision in the case of low torques. The first investigations with the PC for the silicon oil PDMS show a high level of conformity in the measuring area investigated.

In addition to the pressure-dependent shear viscosities the viscoelastic parameters, such as storage and loss module, are useful for the rheological characterization of PDMS. In the next set of measurements the intention is to carry out oscillatory tests under pressure. A further goal is the investigation of plastics in the temperature range from 180°C. In particular, there are plans

for measurements of melts using foaming agents, which are required in the production of micro-foams.

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