A Quick Guide to Better Viscosity Measurements of Highly Viscous Fluids

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Abstract:
The performance of tests with elastic liquids at high shear rates is cumbersome due to viscous dissipation heating, high normal forces, and - above all - edge fracture. This paper shows how such measurements can be improved and simplified over the conventional cone-plate technique by using a partitioned plate. For a polystyrene melt with zero shear viscosity 44.5 kPas at 190°C, steady state viscosities can be obtained up to 100 s^{-1}. For samples with twice the diameter of the sensing area of the tool, the strain beyond which disturbances can be noticed is about 2 - 3 times higher than for conventional cone-plate. As a consequence of the design, precise viscosity measurements can be made without knowing the exact radius of the sample and without well centring it. This geometry is ideal for quick and dirty loading. Drawbacks are that the tool requires regular cleaning of the ring gap, that it can only be fitted to rheometers with a non-displacing force measuring cell (force rebalance transducer), and that it is not suited to measure low viscous systems such as polymer solutions.

Zusammenfassung:
Es wird gezeigt, wie Spannversuche an elastischen Flüssigkeiten bei hohen Schergeschwindigkeiten durch Verwendung einer geteilten Platte in Kegel-Platte Geometrie verbessert und vereinfacht werden können. Für eine Polystyrolschmelze mit einer Schernullviskosität von 44.5 kPas bei 190°C können Gleichgewichtsviskositäten bis 100 s^{-1} gemessen werden. Für Proben mit dem doppelten Durchmesser der Messfläche kann die Scherung, bei der Strömungsinstabilitäten sichtbar werden, um einen Faktor 2 - 3 vergrössert werden. Das spezielle Design der Platte erlaubt es, mit einer schlecht zentrierten Probe, deren Radius man nicht kennt, präzise Messungen durchzuführen. Nachteilig ist, dass der Ringspalt regelmässig gereinigt werden muss, dass die geteilte Platte nur an Rheometern befestigt werden kann, die mit einer bewegungsfreien Kraftmesszelle ausgestattet sind und dass die Methode wegen des Ringspaltes nicht für niederviskose Systeme (wie Polymerlösungen) geeignet ist.

Résumé:
Il est montré comment des tests à haut taux de cisaillement avec des liquides élastiques peuvent être conduits plus facilement en utilisant une géométrie cône-plan dont le plan est partitionné. Pour un polystyrène à l´état fondu possédant une viscosité limite en cisaillement de 44.5 kPas à 190°C, les viscosités en régime stationnaire peuvent être déterminées jusqu'à 100 s^{-1}. Pour des échantillons dont le diamètre est le double de celui de la zone de mesure, la déformation à partir de laquelle des hétérogénéités d'écoulement apparaissent peut être augmentée d'un facteur 2 à 3. De plus, la conception spéciale du plateau partitionné permet de faire des expériences précises même si l'échantillon est mal centré ou si son rayon n'est pas connu de façon précise. Les désavantages de la méthode sont que la fente circulaire nécessite un nettoyage régulier, que l'outil ne peut être monté que sur des rhéomètres à capteur de force non mobile et qu'elle n'est pas applicable dans le cas de liquides de faible viscosité tels que des solutions de polymère.

Key Words: partition plate, cone-plate, polymer melt, edge fracture

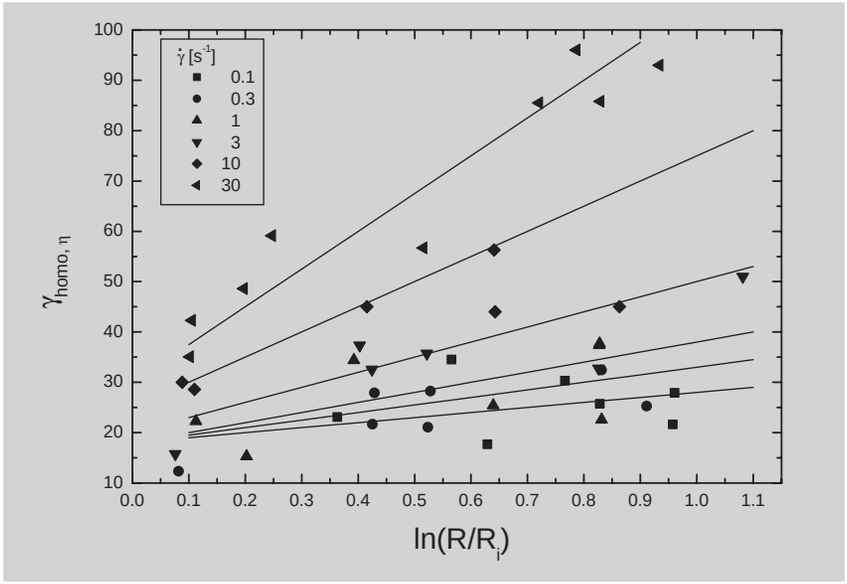

Figure 4: Strain $\gamma_{homo, \eta}$ beyond which distortions can be observed in the measured torque signal. The lines are guides to the eye.

The error bars show the standard deviation from averaging. The data for $\dot{\gamma}$ = 40, 60, 80, and 100 s^{-1} are single measurements. For this data, the error bars represent the uncertainty due to viscous dissipation heating [4]. For conventional cone-plate tests, 30 s^{-1} is about the maximum practical shear rate [5]. That paper shows that the viscosity error bars for conventional and partitioned plate are comparable. The dashed line in Fig. 3 is the linear start-up viscosity η^{o}(t) calculated from the relaxation time spectrum. For the experiments with $\dot{\gamma}$ = 40 s^{-1}, the normal force of 20 N was exceeded while the torque measurement was in progress. This is a physical problem of non-linear shear rheometry since torque scales as $\dot{\gamma}$, whereas normal force increases as $\dot{\gamma}^2$. For the same reason, at low shear rates, the normal force vanishes much quicker in the noise than the torque signal.

A measure for the damping effect of the melt outside the ring gap is the onset strain $\gamma_{homo, \eta}$ for seeing distortions in the viscosity signal, as shown in Fig. 4. This graph summarizes $\gamma_{homo, \eta}$ as function of shear rate and sample radius. "Distortion" is understood as either a sudden strong decrease of the viscosity as shown in Fig. 2 or the beginning of some undulations, probably an expression of the rim being pressed out of its circular shape. This undulations are small (order 100 Pas) and can only be seen under magnification. Fig. 4 shows two effects: First, $\gamma_{homo, \eta}$ at ln(R/R_i) = 0, i.e. at the position of the ring gap, increases with increasing shear rate. Second: With increasing shear rate the dependence of $\gamma_{homo, \eta}$ on radius becomes much stronger. Both effects are counterintuitive. It makes sense that $\gamma_{homo, \eta}$ increases with R since there is a wider span of damping melt between the rim and the gap. The other dependences can only be understood by the viscoelastic nature of the melt: If it is sheared quicker, it behaves more elastically. Thus, either a melt with oriented molecules has better damping properties or the stronger orientation delays the formation of rolls that finally consume the sample. Concerning the limit of stable flow at $\gamma_{homo, \eta}$, an interesting observation can be made: The torque signal can be quite smooth and stable, while the normal force signal is already perturbed. This is an expression of the very different sensitivity of the transducer in peripheral and axial direction for disturbances due to squeeze flow motion.

Several useful features of torque measurements with the partitioned plate tool have been mentioned so far. Of course, there is a price to be paid: The partitioned plate can only be fitted to rheometers where motor and transducer are separate units, since the sensing area of the stem must not move relative to the fixed annulus. The partitioned plate tool cannot be purchased, but the design is so simple that any workshop should be able to build it. The most challenging modification required on the rheometer is its conversion to electrical heating, since the annulus is too bulky to fit into a nitrogen convection oven.

To prevent friction there has to be a small gap between the torque measuring stem and the annulus fixed to the frame. Since there is always some melt penetrating that gap - particularly under the influence of high normal forces - regular cleaning is required. The gap also makes the method unsuitable for low viscous systems (like solutions) since the rapidly penetrating fluid consumes the sample and adds extra friction.

The experimental limit of the method in the case of the RMS800 is given by the maximum normal force and not by excessive torque. The maximum torque would allow even higher shear rates to be applied. If at high shear rates not only the steady state but also the overshoot of the viscosity is of interest, there arises the problem that the time to reach the maximum is of similar order as the time for the motor to accelerate to full speed (about 0.06 s). This has to be considered when analysing such data.

4 CONCLUSIONS

For viscosity measurements the partitioned plate tool offers the advantage that the measuring

range can be extended by up to a factor of three compared to conventional cone-and-plate rheometry. The onset of edge fracture (unbound decrease of the viscosity) is substantially delayed to higher strains if large samples are used. Since the edge is in the sample volume not connected to the transducer, a bad centring of the sample or virtually any size of the sample (as long as it is not too close to R_i) will have no influence on the results, thus quick and dirty loading is allowed. This splendid feature more than compensates for the effort to convert the rheometer. The axial compliance of the transducer, which is a critical issue in normal force measurements, is not a problem for viscosity measurements since the squeeze flow emerging from the "breathing" of the rheometer under high normal forces has little influence on the torque signal.

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