5th International Conference on Experimental Fluid Mechanics – ICEFM 2018 Munich
Munich, Germany, July 2-4, 2018

Narrow-Gap Rotational Rheometer

H. Dakhil, A. Wierschem*

Institute of Fluid Mechanics, Friedrich-Alexander-Universität Erlangen-Nürnberg (FAU), Erlangen, Germany

*andreas.wierschem@fau.de

Abstract

Rotational rheometers are frequently used to quantify viscoelastic material properties. The shear-rate range of rotational rheometers is usually confined to about 10^3 s^{-1}. The measurement window can be extended by reducing the gap width in the parallel-disk configuration. Yet, zero-gap errors and a superposition with elongation flow make it is difficult to reduce the gap width below about 100 µm. We present a modification for commercial rotational rheometers aligning the plates in the parallel-disk configuration perpendicular to the axis of rotation with a precision in parallelism of about 1 µm. This lowers the zero-gap error by a factor of about 25 and more and enables measurements at gap widths well below 100 µm.

1 Introduction

Rotational rheometers are the work horses of rheologists. They offer a wide dynamical range for measuring the viscoelastic properties of fluids and solids (Mezger 2011). Besides, they can be used to precisely set up, control and change shear flows, see e.g. the studies by Agudo et al. (2012, 2014, 2017, 2018). Important quantities to control or to measure are, among others, the shear stress and the shear rate. The wide range of shear stresses is due to the torque range of the apparatuses, which covers several orders of magnitude, and can be further extended using different geometrical configurations with varying size (Mezger 2011). The accessible shear rate is governed by the angular velocity, which again can be varied over several decimal orders, and by the gap width between moving surfaces that confine the samples. Any easy way to set up high shear rates is to use parallel plates at narrow distance. Yet, imperfections in the alignment of the plates and in determining the zero gap result in increasing errors when narrowing the gap, usually restricting the gap to widths of about 100 µm or larger.

Nevertheless, it is advantageous to decreases the gap width well below this limit. Improving the alignment of the plates considerably, it is possible to extend the measurement range of rotational rheometers from shear rates of about 10^3 s^{-1} to more than 10^5 s^{-1}. With decreasing gap width, thresholds for flow instabilities that may restrict the measurement window shift to higher shear rates (Dakhil and Wierschem 2014; Ewoldt et al. 2015). Furthermore, only very little amount of sample (of the order of 10 µl) is required and studies of low viscous samples at high shear rates are possible (Dakhil and Wierschem 2014). Also new applications become accessible: The effect of geometrical confinement can be studied (Luengo et al. 1997) and average linear viscoelastic properties of a large number of cells can be determined quantitatively in single experimental runs as shown by Dakhil et al. (2016) allowing to study the impact of diseases and of drugs on the mechanical cell behavior. Also the adhesion limit of cells on substrates (Dakhil et al. 2018) as well as their load limit (Kokkinos et al. 2016) can be studied in a low viscous environment. Here, we briefly show how the accessible gap width is restricted in standard rotational rheometers and we describe how to overcome this limitation.

2 Zero-gap error

In a parallel-disk configuration, the sample is placed in a gap between two horizontal plates. One disk is usually held at rest while the other one rotates around its central vertical axis, creating a linear shear flow. The shear rate increases linearly with distance from the axis of rotation having a maximum value at the radius of the smaller plate, R. The maximum shear rate $\dot{\gamma}_R$ is:

$$\dot{\gamma}_R = \frac{\Omega R}{h}, \tag{1}$$

where Ω and h indicate angular velocity and gap width, respectively. Hence, the shear-rate range can be increased easily by decreasing the gap width. Yet, the measured viscosity usually decreases with smaller gap width. Figure 1(a) shows an example for a Newtonian silicon oil. The decline of the viscosity values is usually due to a zero-gap error. This error is provoked by inclined plates and by squeeze flow when setting the zero level for the gap width (Davies and Stokes 2008) and results into an underestimation, Δh, of the real average gap width. Hence, while the rheometer determines the viscosity using the shear rate from equation (1), the real average shear rate $\dot{\gamma}_{Rreal}$ at the radius of the plate is:

$$\dot{\gamma}_{Rreal} = \frac{\Omega R}{h+\Delta h}, \tag{2}$$

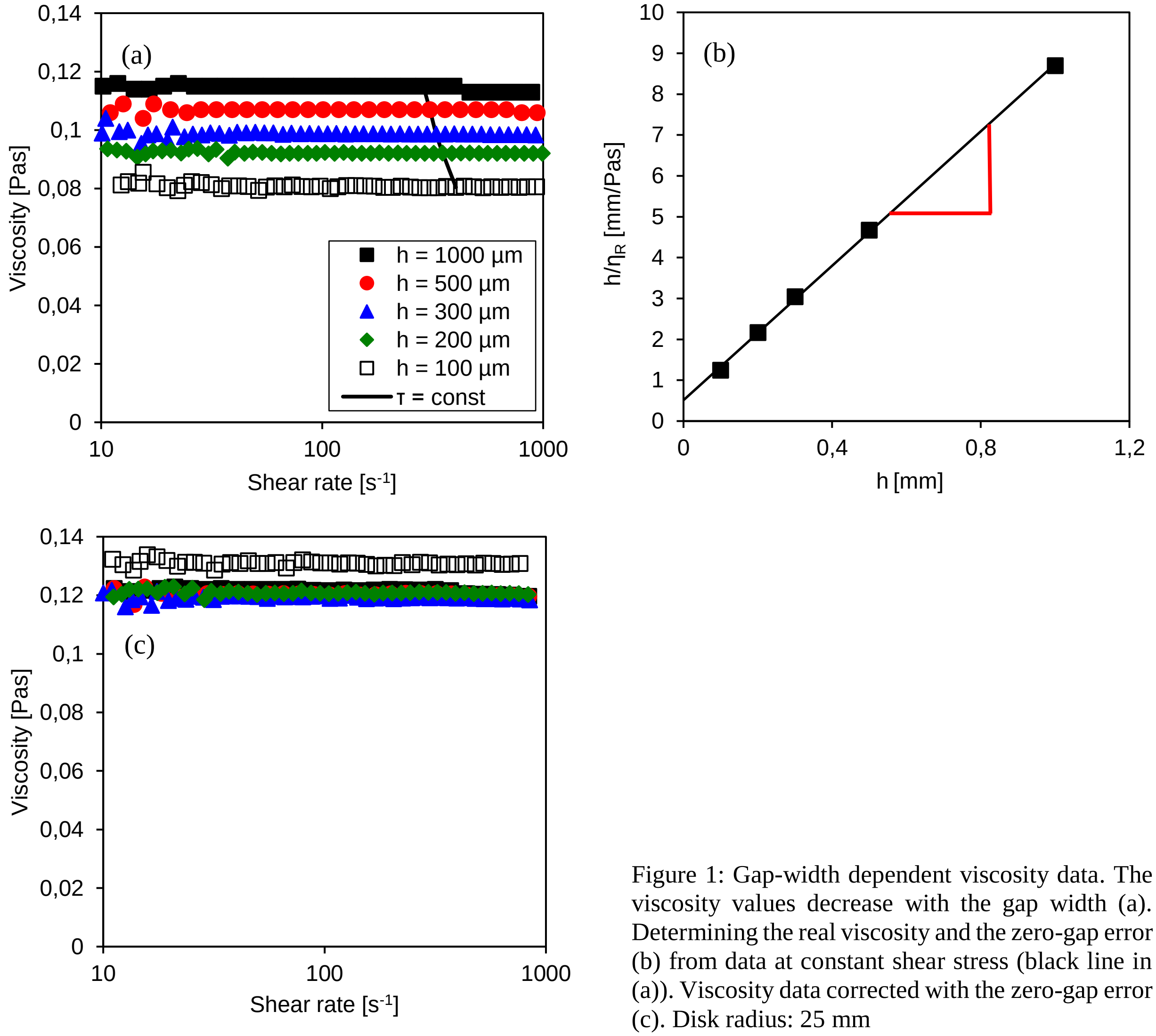

Figure 1: Gap-width dependent viscosity data. The viscosity values decrease with the gap width (a). Determining the real viscosity and the zero-gap error (b) from data at constant shear stress (black line in (a)). Viscosity data corrected with the zero-gap error (c). Disk radius: 25 mm

The shear stress τ_R remains unaffected by the zero-gap error. Hence,

$$\tau_R = \eta_R \dot{\gamma}_R = \eta_{Rreal} \dot{\gamma}_{Rreal}, \tag{3}$$

where η_R and η_{Rreal} indicate the viscosity data of the rheometer and the viscosity determined from the real average gap width. Expressing $\dot{\gamma}_{Rreal}$ by $\dot{\gamma}_R$, equation (3) can be brought into the following form (Davies and Stokes 2008):

$$\frac{h}{\eta_R} = \frac{h}{\eta_{Rreal}} + \frac{\Delta h}{\eta_{Rreal}}. \tag{4}$$

This is a linear equation between the parameters h and h/η_R. Hence, varying the gap width and detecting the viscosity data at constant shear stress, one can determine the real viscosity from the slope and the zero-gap error from the offset. Figure 1(b) shows the result for the data from Figure 1(a). Once the zero-gap error is determined, the shear rate and the viscosity can be corrected according to equations (2) and (3). The results are shown in Figure 1(c). It indicates that the correction works well. However, at gap widths of less than about three times the zero-gap error, we find even an overcorrection. It is supposed to be due to a contribution from elongation flows that is caused by the strong gap-width modulation during shear due to plate inclination (Andablo Reyes et al. 2010, 2011).

3 Narrow-gap rheometer

Zero-gap errors of about 25 µm or larger have been reported (Davies and Stokes 2005, 2008; Pipe et al. 2008). While the data at gap widths of about three or more times this error may be corrected, plate inclination and unevenness result in a superposition of elongation flow with the shear flow, which is difficult to approach (Andablo Reyes et al. 2011). To access gap widths well below 100 µm, the zero-gap error needs to be reduced drastically. To this end, we modified a commercial rotational rheometer as sketched in Figure 2. We used a UDS 200 rheometer from Physica. The plates have to be aligned not only parallel to each other but, above all, perpendicular to the axis of rotation. As disks we use two glass plates with an evenness of λ/4 or λ/10, where λ is the testing wavelength (633 nm). The fixed bottom plate has a diameter of 75 mm and the diameter of the rotating plate is 50 mm. The top plate is adhered to a measurement head of the rheometer with a diameter of 25 mm. To align the bottom plate perpendicular to the rotation axis, it is fixed to a tripod, which is mounted on the rheometer table. The tripod itself is aligned with three actuators. For disk alignment, the gap width is measured with a customized confocal interferometric sensor. The sensor is placed underneath the fixed glass plate on a traverse to measure the gap width at different locations.

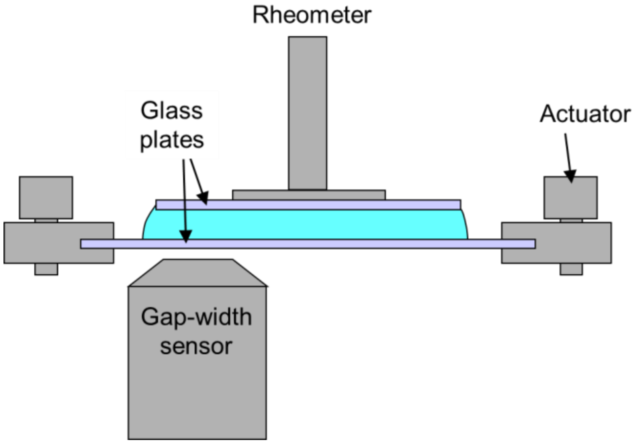

Figure 2: Sketch of the narrow-gap rheometer.

While the upper plate slowly rotates, the sensor detects the local gap width at three different locations close to the actuators. The lower plate is adjusted with the actuators to minimize deviations in the average local gap width between the three locations. Thereafter, the upper disk is dismantled from the rheometer head, aligned to the lower plate by placing it on its top and in this position glued again to the rheometer head. Alignment is checked again with the sensor to achieve undulations at each location with an overall deviation of less than ±1 µm. For further details on the alignment procedure, we refer to Dakhil and Wierschem (2014).

An example of the applicability of the device shows Figure 3. It is the same silicon oil as in Figure 1, yet at a different temperature. It shows a direct comparison between measurements with the narrow-gap device at gap widths of 1000 µm and 20 µm, the latter being a gap width well below the former zero-gap error. A dramatic decline of the viscosity values at reduced gap width like in commercial rheometers does not occur. Instead, both measurements differ only by about 3%. Hence, the deviation is less than the relative gap-width modulation at 20 µm gap width.

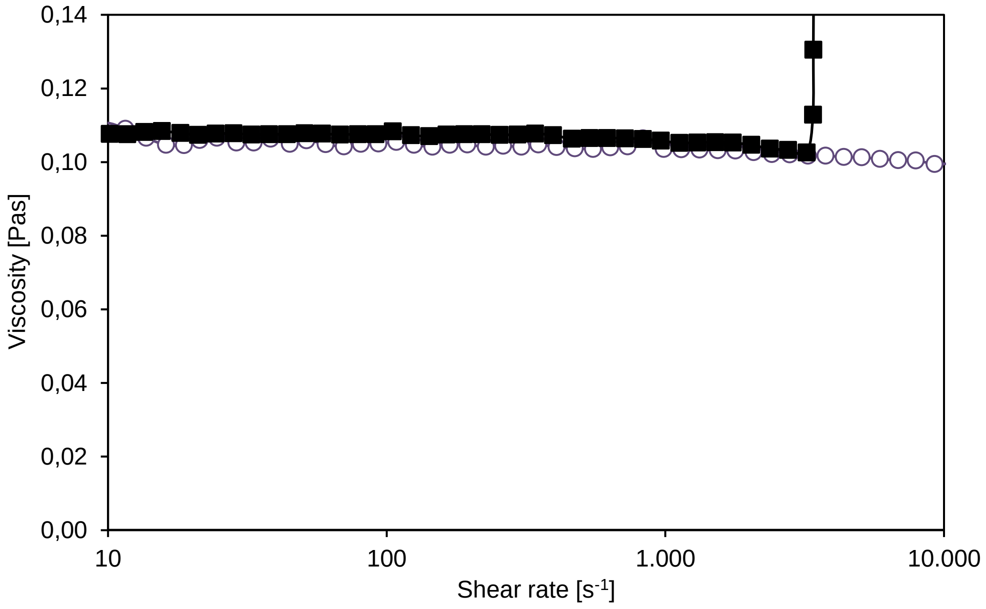

Figure 3: Measurement of the viscosity of a silicon oil with the narrow-gap device. Solid and open symbols indicate measurements at 1000 µm and at 20 µm, respectively. The steep increase of the large-gap-width data at the highest shear rate indicates the maximum rotational speed of the rheometer.

4 Conclusion

We modified a commercial rheometer to align the plates in a parallel-disk configuration perpendicular to the rotational axis. Parallelism is controlled with a precision of about 1 µm. This modification allows to overcome the significant error in the gap height while zeroing the device plates and enables extending the measurement window for shear rates to more than 10^5 s^{-1}.

References

Agudo JR, Dasilva S, and Wierschem A (2014) How do neighbors affect incipient particle motion in laminar shear flow? *Physics of Fluids* 26:053303

Agudo JR, Han J, Park J, Kwon S, Loekman S, Luzi G, Lichtenberger C, Delgado A, and Wierschem A (2018) Visually based characterization of the incipient particle motion in regular substrates: From laminar to turbulent conditions. *Journal of Visualized Experiments* 132:e57238

Agudo JR, Luzi G, Han J, Hwang M, Lee J, and Wierschem A (2017) Detection of granular motion using image processing with particular emphasis on rolling motion. *Review of Scientific Instruments* 88:051805

Agudo JR and Wierschem A (2012) Incipient motion of a single particle on regular substrates in laminar shear flow. *Physics of Fluids* 24:093302

Andablo Reyes EA, Vicente J, and Hidalgo-Alvarez JR (2010) A method for the estimation of the film thickness and plate tilt angle in thin film misaligned plate-plate rheometry. *Journal of Non-Newtonian Fluid Mechanics* 165:882-886

Andablo-Reyes E, de Vicente J, Hidalgo-Alvarez, JR (2011) On the nonparallelism effect in thin film plate-plate rheometry. *Journal of Rheology* 55:981-986

Dakhil H, Do H, Hübner H, and Wierschem A (2018) Measuring the adhesion limit of fibronectin for fibroblasts with a narrow-gap rotational rheometer. *Bioprocess and Biosystems Engineering* 41:353-358

Dakhil H, Gilbert DF, Malhotra D, Limmer A, Engelhardt H, Amtmann A, Hansmann J, Hübner H, Buchholz R, Friedrich O, and Wierschem A (2016) Measuring average rheological quantities of cell monolayers in the linear viscoelastic regime. *Rheologica Acta* 55:527-536

Dakhil H and Wierschem A (2014) Measuring low viscosities with a rotational rheometer in a thin-gap parallel-disk configuration. *Applied Rheology* 24:63795

Davies GA and Stokes JR (2005) On the gap error in parallel plate rheometry that arises from the presence of air when zeroing the gap. *Journal of Rheology* 49:919–922

Davies GA and Stokes JR (2008) Thin film and high shear rheology of multiphase complex fluids. *Journal of Non-Newtonian Fluid Mechanics* 148 73–87

Ewoldt RH, Johnston MT, Caretta LM (2015) Experimental challenge of shear rheology: How to avoid bad data. *Complex Fluids in Biological Systems* (Ed. Spagnolie SE), Springer, Berlin

Kokkinos D, Dakhil H, Wierschem A, Briesen H, and Braun A (2016) Deformation and rupture of Dunaliella salina at high shear rates without the use of thickeners. *Biorheology* 53:1-11

Luengo G, Schmitt FJ, Hill R, and Israelachvili J (1997) Thin film rheology and tribology of confined polymer melts: Contrasts with bulk properties. *Macromolecules* 30:2482-2494

Mezger TG (2011) The Rheology Handbook. *Vincentz Network*

Pipe CJ, Majmudar TS, and McKinley GH (2008) High shear rate viscometry. *Rheologica Acta* 47:621–642