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Measuring Low Viscosities and High Shear Rates with a Rotational Rheometer in a Thin-gap Parallel-disk Configuration

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Abstract:
We modify a commercial rheometer so that the disks are aligned perpendicular to the axis of rotation with a precision in parallelism of about 1 μm independent of the rheometer reading. This leads to decrease the zero-gap error by a factor of 25 and more. It enables samples to be studied at gap widths well below the absolute error of commercial rheometers. At gap widths of 20 μm, the modification allows the measurement range to be extended to shear rates up to 10^5 s^{-1} enabling to measure low viscosities such as that of solvents or water and of dilute polymer solutions. The measurements are restricted mainly by the torque resolution at low shear rates and by inertia at high shear rates.

Key words:
thin-gap rheometry, parallel-disk configuration, low viscous liquids, high shear rates, polymer solutions

1 INTRODUCTION

Commercial rotational rheometers are used to measure a wide range of viscosities at shear rates up to about 10^3 s^{-1}. Low viscosities may be determined with a double-gap cylindrical system. The parallel-disk configuration is usually employed for higher viscosities at gap widths of about 1 mm. Yet, it has the advantage to select the shear-rate range by adjusting the gap width. At gap widths below about 100 μm, however, it suffers from errors in determining the zero point. They are caused by viscous resistance to squeeze flow during zeroing and by unevenness and small inclination angles of the plates [1–4]. For the zero-gap error effective values of about 25 μm or larger have been reported [3, 5, 6]. While the data may be corrected for the zero-gap error, plate inclination and unevenness result in a superposition of elongation flow with the shear flow, which is difficult to access [7]. At low torques, apart from the rheometer resolution, precision is further reduced by contact line forces, which result in a constant torque offset [8]. A value 20 times larger than the manufacturer's specification has been used as a practical low-torque limit [9, 10]. At high shear rates, the measurement range is restricted due to viscous heating, inertial deviations from viscosimetric flow and radial migration due to centrifugal forces or normal stress differences that may overcome surface tension forces [1, 2, 11–14].

Working at thin gaps offers a number of advantages for commercial rheometers. It extends, for instance, the working range to higher shear rates. Reducing the gap width by about two orders of magnitude increases the maximum shear rate accordingly while thresholds for flow instabilities are shifted to higher shear rates. This enables to carry out measurements at shear rates up to 10^5 s^{-1} of viscosities below 1 mPa·s. Also the required amount of sample is drastically reduced, making it attractive for expensive or rare samples. In our setup, about 40 μl are required at a gap width of 20 μm. Thin gaps, in principle, also allow for better temperature control at high shear rates. Furthermore, it enables to study the effect of geometrical confinement [15] and the rheology of biological cells [16].

Several groups have built piezoelectric devices to carry out oscillatory studies at gap widths below 100 μm [17–20]. At small amplitudes frequencies up to the kHz region can be explored. Granick and co-workers developed a shear apparatus for oscillatory studies that works at gap widths down to the sub micrometer range [21, 22]. McKinley and his group reached these gap widths

viscosity function of the aqueous 0.5 wt.% xanthan solution at gap widths of 1000 μm, 100 μm and 20 μm. The open symbols indicate data at a torque below 10 μNm (at low shear rates) and data at a Reynolds number beyond 10 (at high shear rates). In this case, the Reynolds number was defined with the measured viscosity. For the poly(ethylene oxide) solution, at a gap width of 100 μm this Reynolds number limit is reached at a shear rate below 10^4 s^{-1}. Reducing the gap width to 20 μm moves this limit to a shear rate of about 10^5 s^{-1}. The minimum shear rate due to the low-torque limit does not depend on the gap width. Yet, as discussed before, at lower shear rates data scatter at 20 μm gap width is larger than at 100 μm.

For the xanthan gum solution, at a gap width of 1000 μm a Reynolds number of 10 is reached at a shear rate below 10^3 s^{-1} as shows Figure 4(b). This limit is reached at gap widths of 100 μm and 20 μm at shear rates of about 10^4 s^{-1} and 10^5 s^{-1}, respectively. The data obtained for different gap widths nicely overlap at high shear rates. At high shear rates strong deviations from the power law is observed and a plateau at high shear rates seems to be reached. Measuring the disk-surface temperature on the gap side after maintaining the shear rate at 10^4 s^{-1} for 100 s did not show any deviation from ambient temperature. Increasing the shear rate to 10^5 s^{-1} like in Figure 4 resulted in a temperature increase of 0.2 K for the poly(ethylene oxide) and xanthan samples. Finally, we remark that the zero-shear viscosity could not be determined at a gap width of 1000 μm for the poly(ethylene oxide) solution.

4 CONCLUSIONS

We modified a commercial rheometer to align the plates in a parallel-disk configuration perpendicular to the rotational axis. Parallelity 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. This enables to measure low viscosities with a parallel-disk configuration. The measurement range is restricted at low shear rates by the torque limit of the rheometer and at high shear rates by inertia. At gap widths of about 20 μm, deviations from viscosimetric flow in thin gaps are shifted to higher shear rates, enabling shear rates to be covered up to about 10^5 s^{-1}.

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