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Influence of patterned surface in the rheometry of simple and complex fluids

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The influence of the surface microgeometry in rheometry is investigated using the correlation between experiments and numerical simulations of the flow in plate–plate rotational geometry. The presence of micropillars or microchannels on the plates induces effective or apparent slip of the fluids at the walls generating a "dynamic hydrophobic surface". The end/edge effects and the gap error are first estimated for the plate–plate smooth geometry, the computational results being found consistent with the performed experiments. The comparison between the measured torques in smooth and microchannels patterned configurations are analyzed using the numerical simulations performed by the Fluent code for the Newtonian fluid and the Carreau model.

The present study demonstrates that hydrophobic effects can be induced at the walls, without the violation of the no-slip condition, by changing the local flow spectrum due to the presence of patterned surfaces at the solid boundary. The results confirm that computational rheometry is an useful tool not just to interpret the experimental data but to calculate the errors of the measurements, as well as to explore and model flow phenomenon as the apparent slip.

The applications of the paper are meant to develop novel testing procedures in rheometry and to design micro-patterned surfaces for the control of slip/adherence of simple and complex liquids in microfluidic devices.

1. Introduction

Microfluidics is today one of the most dynamic domain of study in engineering, especially in relation with novel Lab-on-a-Chip applications which involve the flows of simple and complex fluids [1–3]. One important subject of investigation in microfluidics is the prediction of the fluid behavior in the very vicinity of the walls. Is the fluid slipping or not to the wall? [4,5]; is the surface hydrophilic or hydrophobic in respect to a particular dynamic process? [6–8]; which is the most indicated pattern of the wall to induce hydrophobicity? [9–11], are inevitable questions whose answers would be an important impact in the design procedure of the new devices and applications [12]. Therefore, the control of wall adherence and degree of slipping become central topics for the fundamental studies in microfluidics. If the material surface and the fluid sample are well defined, in order to control the degree of fluid adherence to the walls then we have to design the proper micropattern which induces the desired phenomenon (i.e. total/partial adherence or slipping). The rheological bulk properties of liquids are experimentally determined using rheometers and special designed measurements procedures based on viscometric flows [13]. In all commercial rheometers (rotational or capillary), the solid surfaces in contact with tested liquids are normally smooth and the slip or perfect adherence of the samples to these surfaces are analyzed and interpreted for each type of measurement, mainly in relation with the microstructure and formulation of the liquid samples [14–17].

The goal of the present study is to investigate and analyze the influence of patterned surfaces in rheometry, to model the flow in plate and plate rotational geometry and to understand how the presence of microgeometries on the plate's surface induces the apparent slipping at the wall and creates "dynamic hydrophobic surfaces", even if the fluid is considered to adhere to the solid walls.

In 1975, professor Ken Walters published *Rheometry* [18], the first book dedicated exclusively to the measurements procedures of the rheological properties of non-Newtonian fluids. In Introduction, the author presented one main objective of this discipline (which is working in "tandem" with rheological modeling and numerical simulations [19,20]): "to determine the behavior of

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non-Newtonian liquids in a number of simple flow situation using suitable defined material functions" [18].

To fulfill this objective it is necessary not only to find general/universal solutions for the equation of motion, but also to impose proper boundary conditions for the working domain. However, in rheometry (especially for complex fluids) it is difficult to always put well posed boundary conditions, since the viscoelastic solutions might need "more boundary conditions than are supplied by the no-slip condition" [18].

In rotational rheometry (in particular plate–plate configuration) the boundary conditions are directly related to the fluid behavior at the walls (adherence or slip) and to the shape of the free surface at the edge of the plates (which is aprioric unknown). If the no-slip condition is normally accepted at the plates (considered to be perfectly smooth surfaces), it is almost impossible to control the boundary condition at the edge of the geometry.

Ken Walters inferred the importance of boundary conditions in rheometry and their direct connection to the quantification and interpretation of the experimental errors; in consequence, each of the chapters from *Rheometry* includes a paragraph dedicated to the analysis and discussion of *possible sources of errors*.

In the present paper we are mainly concerned with two categories of errors mentioned by Ken Walters in Ref. [18]: (i) edge and end effects, respectively (ii) instrument imperfections. Both are related with the fact that boundary of the flow domain is not precisely defined, and this is not referring only to the unknown free surface at the edge of the plates, but also with the deviation of the real geometry of the plates from the calculus geometry (due to misalignment, lack of parallelism, tilted axes).

We shall refer to these errors as end/edge effects and gap errors; we suppose that no-slip condition to solid walls holds and the free surface between the plates is cylindrical and in contact with the atmospheric pressure. End effects include inertia influence in a finite geometry (onset of secondary flows) and the error induced by the approximation of the real free surface of the sample at the edge with a cylindrical surface. Gap error is considered to be generated mainly by the non-parallelism of the plates, so the real gap is not constant along the surfaces. Since the goal of the work is to investigate the influence of pattern plates on the measurements in plate–plate geometry, it is compulsory to evaluate first the contribution of the end effects and gap error on the experiments performed for the smooth (commercial) plates, see Refs. [21–23].

The structure of the paper is the following: Section 2 is dedicated to the characterization of fluid samples and the measurements of the torques in simple shear for the smooth and patterned surfaces. A presentation of the surfaces microgeometry is also made. In Section 3 the gap error is analyzed in relation to a thin film (lubrication) analytical Stokes solution for rotational non-parallel surfaces. The results from numerical simulations of Newtonian and generalized Newtonian fluids (Carreau model) in plate–plate smooth and patterned configurations are shown in Section 4. Finally, in Section 5 the experimental and numerical results are analyzed and the conclusions of the work are presented.

2. Experimental

The investigations are performed with the Anton Paar Physica MC301 rheometer in controlled strain mode using the parallel plates configuration with diameters of 25 mm, respectively 50 mm, at constant temperature within a range from 10 °C to 25 °C. The reference values for the samples shear rheology are obtained using the cone and plate configuration (cone diameter of 50 mm and 1° angle). In experiments the upper plate was always the regular commercial stainless steel plate. Several lower plates of different materials and patterns have been tested: (i) current lower plate of the rheometer (PN-plate), (ii) perfectly smooth silicon plate (Si-plate), (iii) silicon plate with pillars pattern (Si-pillars), (iii) copper alloy plate with microchannels pattern (channels plate).

Silicon wafers of 76 mm in diameter were processed at the IMT Bucharest (National Institute for Research and Development in Microtechnologies) using photolithography and DRIE (Deep Reactive Ion Etching) techniques to produce surfaces with pillars pattern, see also Ref. [24]. The plates patterned with parallel microchannels were obtained by classical mechanical procedure, see Fig. 1.

The most difficult part of the experimental protocol was to obtain a working set-up with minimum alignment and parallelism errors of the lower plates, relative to the rotational upper geometry plate. First, the horizontal position of the original lower plate was adjusted and the calibration to some prescribed gaps between the plates was performed using the measurements in three points of normal force between the upper plates and the feeler gauges of 50 μm and 100 μm (nominal height) mounted on the lower plate. The zero gap corresponds to the position where the measured normal (axial) force against the upper plate is zero. Therefore, in our case the prescribed nominal gap of the rheometer indicates actually the lowest gap height between the plates.

Each manufactured lower plate is fixed on the rheometer lower plate and the described calibration was performed before starting a new measurement. However, this procedure did not eliminate the lack of parallelism between the tools and the misaligned plate–plate problem generated by the gap variation along the contact surfaces. Using a set of feeler gauges of 40 μm, 50 μm, 60 μm, 70 μm, respectively 150 μm and 160 μm, we measured a gap difference between opposite edges of the 25 mm plate diameter of approximate 10 μm (for all nominal gaps magnitude).

The samples used in experiments are two Newtonian liquids: Si-oil (silicone oil, with nominal viscosity of 0.4 Pa s at 20 °C) and En-oil (10W50 engine oil, with nominal viscosity of 0.4 Pa s at 10 °C and 0.275 Pa s at 20 °C), and a PIB-solution, a weakly elastic polymer solution of polyisobutylene with $M_w = 0.5$ mil. (from Sigma Aldrich) in En-oil, with zero shear viscosity of 1.55 Pa s at 10 °C and 0.9 Pa s at 20 °C.

The reference temperature for each test and fluid was fixed as function of the measured temperature of the patterned lower plate. This temperature cannot be strictly controlled by the Peltier system of the rheometer, in consequence the shown data are obtained at different temperatures.

The oscillatory shear test of PIB-solution is presented in Fig. 2. The measured data disclose two phenomena: (1) the decreasing of the measured viscosity with reducing the gap in plate–plate configuration (so called the gap error effect), and (2) the influence of the lower plate quality on the measured torque. The first phenomena is well known in simple shear rheology and was recently investigated and analyzed in relation to the shear rheometry at high rates [25–28], see also Refs. [21–23]. The influence of the plate quality (assuming the plate is smooth) is determined by the adherece of the fluid at the material surface and possible slip occurence. In Fig. 2 the measured differences in complex viscosity between the PN-plate and Si-plate are up to 20%, for the same value of the gap.

The samples (Newtonian liquids and PIB-solution) are not expected to exhibit significant slip at the walls of commercial plates. However, the measurements performed with perfect smooth Si-plates indicate possible presence of slip for PIB-solution in plate–plate geometry, the phenomenon which seems to be absent in the cone–plate configuration (where the recorded data are in the range of the experimental errors for the two tested smooth lower plates).

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