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Friction mechanisms and interfacial slip at fluid–solid interfaces

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Abstract

We present series of experiments based on near field laser velocimetry, developed to characterize the friction mechanisms at fluid–solid interfaces. For polymers, entangled polymer melts are sheared against smooth solid surfaces, covered by surface attached polymer chains of the same chemical species, having a controlled surface density. Direct measurements of the interfacial velocity and of the shear force allow identification of the molecular mechanisms of friction. Depending on the value of the inverse of the shear rate experienced by the polymer compared to the reptation time, the transition between a regime of high and a regime of low friction observed when increasing the shear rate can be related to disentanglement or to the extraction of the surface chains from the bulk polymer. Surfaces with adjusted friction properties can thus be designed by choosing chain anchored length and surface density. For simple fluids, the direct measurements of the interfacial velocity show that, contrary to the usual hypothesis of hydrodynamics, a simple fluid can exhibit slip at the wall. Both the surface roughness and the strength of the fluid–solid interactions fix the amplitude of wall slip, acting in an antagonistic way.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

When a fluid is sheared between solid surfaces, it is usually assumed that the relative fluid–solid velocity is equal to zero. This is the so called non-slip boundary condition (NSBC) [1]. This hypothesis, supported by experimental evidence at macroscopic scales [2, 3], is not justified by strong physical arguments at microscopic scales, and much research has been devoted to the investigation of adhesion and friction of liquid molecules at solid interfaces. The pioneering work by Coulomb [4] concluded that the NSBC was true even at microscopic scales. But

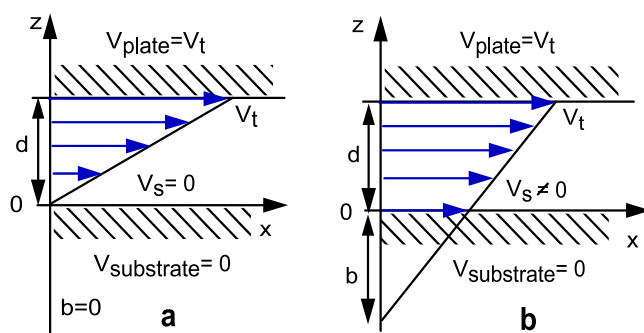


Figure 1. Schematic representation of a no-slip (a) or a slip (b) boundary condition for the fluid velocity at the solid wall. For the sake of simplicity, the top and bottom surfaces are assumed to be different and to lead to different boundary conditions for the flow velocity at the wall. Such a situation can be achieved experimentally by treating the two surfaces in a different manner, as will be detailed in the presentation of the experimental results.

this was relying on experiments with a poor resolution [2, 5]. Several indications, both from experiments [6, 7] and from molecular dynamic simulations [8, 9], suggest the possibility of slip at the wall, in the case of a weakly bonding surface [4–6, 8–16].

The situation is schematically presented in figure 1 for a simple shear flow. The extrapolation length of the velocity profile to zero, b , is usually admitted to be comparable to a few molecular sizes, either below the surface (positive b) or above the surface (negative b), depending on whether the fluid–solid interactions are weaker or stronger than the interactions within the liquid. In a simple fluid, it is usually admitted that the effect of the change in the strength of the interactions introduced by the presence of the interface cannot produce effects on a range much larger than the range of the interactions. The slip length, b , is thus expected to remain small, comparable to a molecular size (figure 1(a)). The situation could be quite different in complex fluids for which the range of interactions could be much larger (figure 1(b)).

In fact, b is directly related to the friction coefficient between the fluid and the surface. This can be easily seen by evaluating the friction force transmitted to the solid by the sheared fluid: the friction force can be estimated as the product of the interfacial velocity V_s by the friction coefficient, k , and also as the product of the fluid viscosity by the velocity gradient at the interface, i.e. $F_f = kV_s = \eta \partial V / \partial z|_{z=0} = \eta V_s / b$. The friction coefficient is thus inversely proportional to the slip length. Any direct information on the local velocity profile close to the solid wall can be translated in terms of surface friction, provided the viscosity close to the wall is known. If one suspects that the viscosity close to the wall could differ from the bulk one, the friction coefficient can be determined without any further assumption by simultaneous measurements of the friction force and of the local velocity V_s .

The above remark points out the interest of direct measurements of the fluid velocity at a fluid–solid interface. This is however a particularly difficult challenge if one wants to gain spatial resolutions from the wall ranging from micrometres down to molecular sizes. Conventional laser velocimetry techniques are far from such resolution. We have developed a near field laser velocimetry (NFLV) technique which allows increasing significantly the resolution compared to more conventional velocimetry technique [17] (even laser velocimetry). This technique has been used to characterize the friction at an interface between a polymer melt and a solid wall [18–23], and also to demonstrate that a simple fluid could exhibit significant slip on a smooth solid surface [24, 25]. Quite recently techniques based on surface force

apparatus (SFA) [26, 27] or drainage force measurements using a modified atomic force microscope (AFM) [28, 29] have been used and have confirmed that a simple fluid could slip at the wall. The resolution from the wall is better in those experiments than in NFLV, but the flow pattern is much more complicated than a simple shear flow, and the characterization of friction laws appears more difficult.

We review these results in the present paper. In the first part, we briefly introduce the principle of the NFLV technique and discuss its principal limitations. In the second part, we review the results obtained for a polymer melt sheared along a solid wall on which polymer chains are end grafted, in order to get a model situation representative of practical situations of polymer flows, for which polymer chains are most often surface anchored. These surface anchored chains deeply affect the fluid–wall friction. The complex observed friction behaviour will be rationalized in term of decoupling of the bulk fluid from the surface at high enough shear rate, due to either disentanglement between surface and bulk chains or to extraction of the surface chains from the bulk melt depending on the relative values of the longest relaxation time of the melt compared to the inverse of the shear rate experienced by the polymer. In the last part, the results obtained for a simple fluid (hexadecane) sheared along either oleophilic or oleophobic surfaces will be presented and compared with data available from SFA or AFM measurements.

2. Principle of the near field velocimetry technique

Up to recently, direct determinations of flow velocities close to a solid wall were scarce and generally obtained using small spheres as flow tracers, i.e. with a spatial resolution of the order of the diameter of the spheres, not better than 150 nm [1–3]. To investigate the boundary condition of the fluid velocity at the solid interface without perturbing the flow, tracers of molecular dimensions are needed. It is also essential to reduce the dimension of the observation volume in the direction normal to the interface, ideally down to a molecular size. We have developed an experiment aiming to fulfil these two requirements, first for polymer fluids [17, 18] and second for simple fluids [24, 25]. In both cases, the condition on the size of the tracers is matched by the use of fluorescent molecules chosen so that they easily photobleach under intense illumination. Photobleached probes are produced by a local intense illumination of the sample (writing period of the experiment) and act as flow tracers. The way they are transported by the flow is followed by monitoring the fluorescence intensity excited with an attenuated illumination so that no further photobleaching is produced during the reading period of the experiment. The confinement of the investigated volume close to the solid–fluid interface is achieved by the use of optical evanescent waves for reading the fluorescence and eventually also to produce the photobleached probes. This technique, based on fluorescence recovery after photobleaching and evanescent wave induced fluorescence, has been named NFLV. Detailed descriptions of the experimental set up and of the data analysis of the fluorescence recovery after photobleaching in order to extract the interfacial velocity are reported in [33] both for polymers and simple fluids.

In the case of polymers, the fluorescent probe is chemically attached to the extremities of a few chains in the fluid. The diffusion of the photobleached probe is then very slow, and the photobleached pattern printed in the sample essentially evolves with time due to convection by the flow. The re-entry of fluorescence can then be directly related to the average velocity in a slab of fluid close to the wall with a thickness comparable to the penetration depth of the evanescent wave used to read the fluorescence. A typical order of magnitude of this penetration depth, Λ , is in the range 30–100 nm, i.e. comparable to the size of a polymer chain. NFLV thus

gives direct access to the fluid velocity at the interface, averaged over a thickness comparable to a molecular size.

This is no longer the case for simple fluids. The fluorescent probe is chosen to be very similar to the molecules of the fluid, in order to avoid preferential interactions at the solid surface, and their diffusion is then fast, imposing modification of the experimental procedure. Keeping a simple shear geometry, the sample is sheared continuously. A photobleached line is printed through the sample, normal to the solid wall, and then the re-entry of fluorescence in the immediate vicinity of the wall is monitored, using an attenuated laser beam which unpins the surface exactly at the same place as the bleached line, but in evanescent wave geometry, so that only a thickness comparable to the penetration depth of the evanescent wave is illuminated. The kinetics of fluorescence recovery is then governed by both diffusion and convection of the photobleached probes. The detailed description of the scaling procedure of the fluorescence recovery curves which allow us to deduce the slip length, b , can be found in [33]. The effect of the fast diffusion is to blur out the bleached pattern, and to average the velocities over a distance from the wall which is larger than the penetration depth of the evanescent wave, and is comparable to the diffusion length during a time comparable to the time constant of the fluorescence recovery, typically in the micron range. The fluorescence recovery curves remain in any case sensitive to the boundary condition of the fluid velocity and allow us to quantify the slip length.

3. Friction and slip at polymer–solid interfaces

The NFLV technique briefly described above has been used to characterize the friction behaviour of polymer melts. In a first step high molecular weight polymers well in their Newtonian fluid regime have been investigated. In order to gain information on the molecular mechanisms of friction at such interfaces, and to be able to vary the molecular parameters in a controlled manner, model systems have been used. They were made of low polydispersity polydimethylsiloxane (PDMS) melts in contact with smooth silica surfaces, covered with controlled densities of end grafted PDMS chains of various molecular weights. The choice of surfaces covered with surface anchored chains was dictated by the fact that in any practical situation where a polymer melt is sheared along a solid surface the most probable situation is that corresponding to the spontaneous formation of a layer of surface anchored chains (even weak monomer–surface interactions result in strong polymer–surface interactions, as a polymer chain has on average \sqrt{N} monomers in contact with the surface, with N the polymerization index of the chain).

The important general result is that when the polymer melt is sheared against a surface on which polymer chains are strongly attached three different friction regimes are observed when increasing the shear rate, provided the surface chains are able to entangle with the bulk polymer. This non-trivial friction behaviour is illustrated in figure 2, which presents velocity measurements at an interface between silica and a PDMS melt, the silica being covered with chemically end grafted PDMS chains, at low surface coverage (see caption for details). In order to prevent slip at the top surface, the top surface is covered by longer end grafted PDMS chains, with an adjusted surface coverage.

Slip is always observed: the measured velocity, which is an average of the velocities within Λ from the surface, is larger than what would be obtained with a no-slip BC, shown as the lower dashed line in figure 2(a). At low top plate velocities the corresponding slip length is small ($b \sim 1 \mu\text{m}$) and constant (figure 2(b)), meaning that V_s is proportional to V_t . At intermediate V_t values, V_s increases more rapidly than linearly with V_t to finally reach a value close to V_t (quasi-plug flow; $V_s = V_t$ is the upper dashed line in figure 2(a)), at high shear rates.

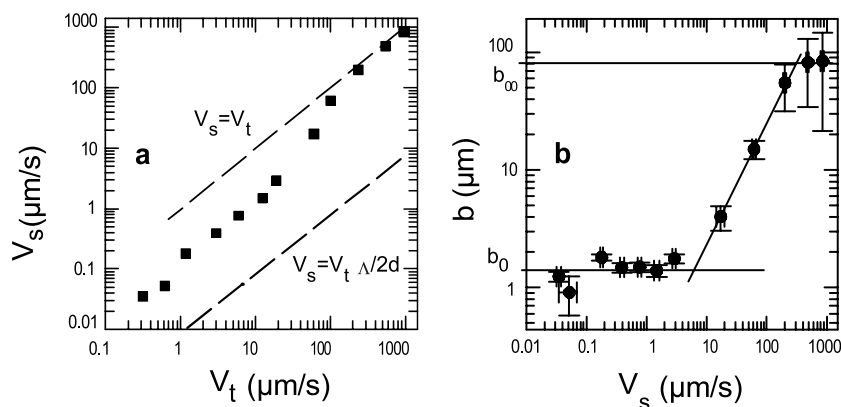


Figure 2. Typical results for (a) the slip velocity and (b) the slip length, for a PDMS melt (molecular weight $M_w = 970 \text{ kg mol}^{-1}$ ($M_w/M_n = 1.14$)), flowing against a silica surface covered with end grafted PDMS chains. The surface under investigation is covered with end grafted PDMS chains with $M_w = 96 \text{ kg mol}^{-1}$ ($M_w/M_n = 1.1$) and a surface density $\Sigma = 0.00025$ (the surface density Σ is related to the average number of grafted chains per unit surface, ν , through $\nu = \Sigma/a^2$, with the size of a monomer $a = 0.5 \text{ nm}$ for PDMS). The top surface is treated by grafting the same PDMS chains with a larger grafting density, in order to prevent slip at the top surface.

In the intermediate regime, b appears proportional to V_s (see the log–log plot of figure 2(b)). As b is inversely proportional to the friction coefficient, this is a non-linear friction regime, with a friction coefficient inversely proportional to the interfacial velocity. In the high shear rate regime V_s becomes again proportional to V_t and b is constant (linear friction regime), of the order of $100 \mu\text{m}$, implying a friction coefficient two orders of magnitude smaller than in the initial low shear rate regime. The striking feature is that, all through the intermediate regime, the shear rate experienced by the polymer, $\dot{\gamma} = (V_t - V_s)/d$ (d is the cell thickness), remains constant, a fact which has been confirmed by direct measurements of the shear stress transmitted by the polymer fluid to the prism. If the solid surface is covered with short PDMS chains having a molecular weight smaller than the average molecular weight between entanglements, only the low friction regime is observed, confirming that entanglements between surface anchored and bulk chains are indeed the source of the non-trivial friction behaviour in these systems [33, 37].

A molecular model describing how a weak density of polymer molecules end grafted on the solid surface (surface chains independent of each other) and entangled with the bulk polymer affect the BC for the velocity at the polymer–solid interface has been developed by de Gennes and co-workers [34–36] and quantitatively agrees with the NFLV results on PDMS for low surface densities of surface chains. This model takes into account the fact that, due to entanglements, the surface chains act as obstacles to the flow of the bulk chains. These entanglements produce a large friction between the melt and the surface at low shear rates, and suppress the huge slip at the wall expected for ideal surfaces [37]. Surface chains are not, however, rigid objects. Under the effect of the friction force, they can deform, and an elastic restoring force develops. In steady state, elastic and friction forces balance each other. Increasing the shear rate increases the friction force and the elongation of the surface chains. The initial high friction regime has to stop when the elongation of the surface chains becomes large enough so that the elongated chains are at the limit of disentangling from the melt. A distinct friction regime, called the marginal regime, is then encountered. The surface chains keep the limiting elongation corresponding to the limit of disentangling for a large range of slip velocities (i.e. a range of imposed V_t), because if elongated more the surface chains would

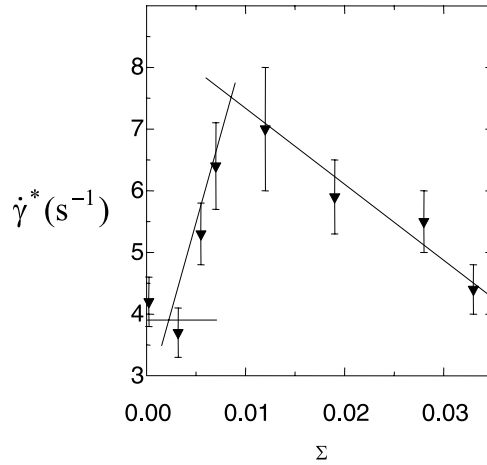


Figure 3. Evolution of the critical shear rate $\dot{\gamma}^*$ at the onset of the marginal regime as a function of the surface density in the grafted surface layer, Σ , for a PDMS melt with a molecular weight 970 kg mol^{-1} , and surface chains with a molecular weight 96 kg mol^{-1} . For small surface densities, $\dot{\gamma}^*$ increases linearly with Σ , indicating that the surface chains contribute to the friction independently of each other. Above a surface density $\Sigma = 0.01$, a collective behaviour of the surface chains shows up.

disentangle from the melt and the friction force would strongly decrease and would no longer balance the elastic restoring force. The surface chains would then re-entangle. In the marginal regime the shear force is thus fixed and independent of the slip velocity, implying a friction coefficient inversely proportional to V_s . The marginal regime ends when the frequency at which the surface chains are excited by the flow (entangling and disentangling from the melt) becomes larger than their relaxation frequency [36]. The surface chains then become fully disentangled, the melt dynamically decouples from the surface and the friction becomes constant and low, with a slip length b_∞ independent of V_s and comparable to what would be obtained on an ideal surface.

In all three friction regimes the shear force per surface chain (which is directly measured in the experiment) is always much smaller than the force necessary to break a hydrogen or a covalent bond [38]. The observed transition from low to high slip at the wall is thus not due to tearing out of the surface chains. For PDMS melts with lower molecular weights mixed with glass beads [39] a slip transition has been observed and attributed to desorption of the PDMS chains from the glass surface. This may not be contradictory. The low to high slip transition is in fact the signature of a dynamic decoupling between the surface and the bulk polymer. Depending on the physico-chemical conditions, this decoupling may take place through different modes: tearing off the surface chains under the shear stress, if the bonding force is weak, or elongation and disentanglement of surface and bulk chains when the bonding force is large. Only the disentanglement mechanism is associated with the existence of a marginal regime extending over several decades in slip velocities.

We have experimentally characterized situations with larger surface densities, as shown in figure 3, where the critical shear rate for the entrance in the marginal regime is reported as a function of the surface density of grafted chains. The first linear regime is characteristic of surface chains acting independently of each other for the friction. Above a given surface density, the critical shear rate decreases, indicating that the decoupling between the bulk polymer and the surface layer becomes easier than for lower surface densities. This can

qualitatively be related to the fact that for high enough surface densities, one expects that the bulk chains can no longer fully penetrate into the surface layer. The number of mutual entanglements should thus become a decreasing function of Σ , and the critical shear rate too. At present, we have however no detailed theoretical description, at the molecular level, of this high density regime, in which the surface chains are no longer independent of each other. The experiments and models described above give however precise lines of thought to design surfaces with adjusted friction properties.

The case of non-Newtonian polymer fluids has also been investigated experimentally, using NFLV, and appears more complicated [41]. A decoupling between the surface layer of surface anchored chains and the bulk polymer still exists, but can be accompanied by a non-stationary regime with stick–slip interfacial motion, as shown in figure 4 for another polymer (styrene–butadiene copolymer, SBR, for which the average distance between entanglements is smaller than for PDMS, so that for comparable molecular weights the reptation time, T_R , is much longer and the limit $\dot{\gamma}T_R \geq 1$ can be easily reached in the shear rate range of the experiment). We have as yet no full molecular explanation for such a behaviour, but it can be qualitatively understood assuming that for high enough shear rates, the bulk chains can no longer disentangle fast enough from the surface layer. In order to allow the slip motion, the surface chains need to be elongated and sufficiently deformed so that they are extracted from the bulk. Such a deformation corresponds to the initial transient in the constraint versus time curves. When the surface chains are extracted from the bulk, the interface can no longer sustain the constraint, and slip develops. If the top plate velocity is not large enough, however, deformation energy is relaxed during the initial slip motion, and the interfacial motion slows down, eventually enough to allow for a partial re-entry of the surface chains into the bulk, initiating the stick–slip behaviour. In figure 4(b), we show that in the absence of entanglements between the surface layer and the bulk at the beginning of the experiment, only the high slip, low friction regime develops. These experiments appear as first steps towards trying to understand friction at crosslinked elastomer–solid interfaces.

4. Case of simple fluids

The NFLV technique has been used to investigate the boundary condition of the flow velocity of hexadecane on surfaces having different wetting properties [24, 25, 41]. We summarize here the main results of these investigations.

Hexadecane has been chosen as a model of non-polar lubricant, with a relatively high surface energy ($\gamma_L = 27.4 \text{ mJ m}^{-2}$ at 20°C). The fluorescent probe was NBD-dihexadecylamine at 5 ppm. The solid surface was a disc of sapphire ($\alpha\text{-Al}_2\text{O}_3$, {001}), with an rms roughness of 0.4 nm, as measured by x-ray reflectivity. Sapphire has a high refractive index, leading to a small penetration depth of the evanescent wave (of the order of 35 nm) and the surface can be chemically modified. Four surfaces have been investigated:

- (a) the surface was cleaned by a UV–ozone procedure [42], yielding a high energy surface, totally wetted by hexadecane;
- (b) the surface was modified by grafting a monolayer of octadecyltrichlorosilane (OTS), following an adjusted procedure similar to that used for silica [43, 44], yielding an oleophobic surface ($\gamma_s = 21 \text{ mJ m}^{-2}$ for an optimal treatment);
- (c) a surface covered with a grafted layer of perfluorodecanetrichlorosilane (FDS) ($\gamma_s < 13 \text{ mJ m}^{-2}$) and
- (d) a surface covered by a layer of physically adsorbed stearic acid (octadecanoic acid) formed *in situ* by incubating the sapphire surface with a solution of stearic acid in hexadecane, inside the flow cell.

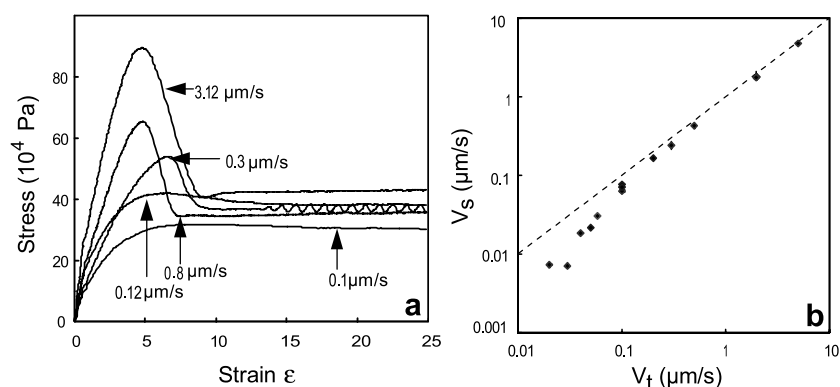


Figure 4. Typical results for (a) the shear stress as a function of the displacement of the top surface of the sample for different velocities of this top surface displacement. The polymer is an SBR melt with a weight average molecular weight 135 kg mol^{-1} . The surface under investigation bears an adsorbed layer of SBR molecules with a weight average molecular weight 50 kg mol^{-1} , and a surface density $\Sigma = 0.005$. A stick-slip oscillating regime clearly appears at intermediate shear rates. (b) Slip velocity as a function of top plate velocity for the same SBR melt as in (a), but in contact with a surface covered by a thin layer of PDMS. PDMS is incompatible with SBR and there are no entanglements between surface and bulk chains. High slip and a quasi-plug flow are observed for the whole range of shear velocities investigated (the dashed line corresponds to $V_s = V_t$ (plug flow)).

X-ray reflectivity analysis of the FDS layer suggested that the FDS molecules were not densely packed and that the monolayer was rather made of FDS islands. Typical rough data for the fluorescence recovery curves are reported in figure 5, for surfaces (a), (b) and (c), and for two different shear rates applied to the fluid. It is clear that the fluorescence recovery is faster on the OTS modified surface, and surprisingly enough it appears faster on the totally wetting bare sapphire surface than on the most oleophobic FDS one. This is the signature of a more efficient convection of the photobleached probes out of the illuminated volume for surfaces (a) and (b) than for surface (c). A quantitative analysis of the curves, in terms of diffusion and convection of the probes, can be conducted, as detailed in [33, 41]. The results in terms of slip lengths are reported in figure 6, along with the corresponding results obtained for surface (d), after various incubation times. Figure 6 clearly shows that the wetting properties of the surface (i.e. the strength of the interactions between the molecules of the fluid and the surface) are not the only parameter affecting the boundary condition of the flow at a solid wall: hexadecane slips at the wall on the totally wetting bare sapphire surface. The no-slip surface is the FDS grafted layer, for which the interactions between the fluid molecules and the solid are the weakest, but the roughness of that surface at molecular scales is important. The smooth OTS surface, with weak interactions and weak roughness, leads to high slip (slip length of the order of half a micrometre, much larger than a molecular size). For the progressively formed adsorbed stearic acid layer, the slip length evolves with time: slip first decreases, compared to the original bare smooth sapphire, and then progressively increases with time when the adsorbed layer becomes dense and smooth. These data clearly demonstrate that a simple fluid can develop noticeable slip at the wall, and that at least two factors deeply affect the amount of slip (i.e. the friction): the strength of the interactions between the fluid molecules and the solid, and the roughness of the surface, at molecular scales, both acting on the level of slip in an antagonistic way. We are presently working with other simple fluids, in order to test the generality of these behaviours. Independent experiments have however been developed, based on measurements

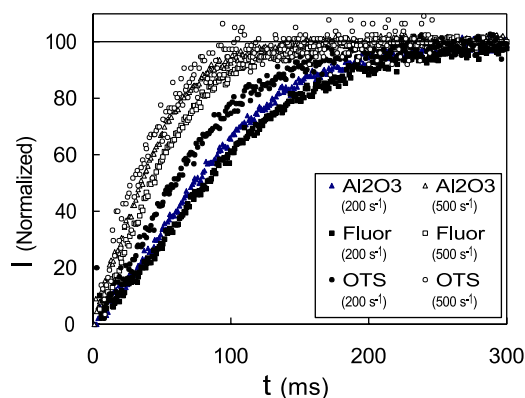


Figure 5. Normalized fluorescence recovery curves as a function of time for hexadecane sheared along surfaces (a), (b) and (c), and two different applied shear rates: 200 and 500 s^{-1} . The top surface is rendered rough, so that a no-slip BC is achieved at the top surface.

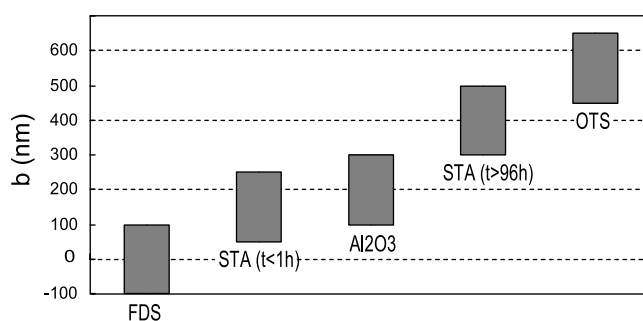


Figure 6. Summary of the measured slip lengths for hexadecane sheared over the four surfaces (a), (b), (c) and (d) (the shear rates used for such determinations are in the range 200–5000 s^{-1}).

of the drainage force between two close surfaces, either in an SFA [26, 27] or in a modified tip AFM [28, 29], leading to conclusions quite similar to ours as far as the existence of wall slip is concerned, for water, water–ethylene glycol mixtures and tetradecane. Slip at the wall thus seems to be a somewhat general feature for smooth surfaces. Our experiments do not give any evidence of a dependence of the slip length on the shear rate, contrary to what is found in [26], and the reason for this discrepancy is not fully understood yet. The flow pattern in both SFA and AFM experiments is however much more complicated than in our NFLV experiment. In both SFA and AFM, the shear rate depends on the distance from the singular line which appears in the centre of the apparatus, and an analysis in terms of shear rates may be delicate.

5. Conclusion

We have briefly reviewed recent results on the friction and flow behaviour at fluid–solid interfaces, obtained through direct measurements of the interfacial velocity, using NFLV techniques. These experiments clearly demonstrate that a fluid can exhibit flow with slip at the wall, and that the no slip boundary condition postulated in most hydrodynamics investigations may be far to be fulfilled. In the case of polymer fluids, this is not so surprising: entangled polymers have a mixed fluid and solid dynamic behaviour. The characterization of the

interfacial velocity as a function of the molecular parameters of the system has allowed us to fully identify the molecular mechanisms of friction at interfaces between a polymer melt and a surface bearing layers of surface anchored polymer chains. The understanding of these mechanisms can lead to the design of surfaces with adjusted friction properties, a quite important objective in a large number of practical situations. Extending the investigation towards crosslinked elastomers should allow one to make steps towards the understanding of solid friction. For simple fluids, we have demonstrated that the zero-velocity hypothesis for the interfacial velocity at a solid wall was not always satisfied. We have established that in the case of hexadecane, the strength of the fluid–solid interactions and the roughness of the surface at molecular scales were acting strongly on the level of wall slip, in an antagonistic way. We have, at present, no real physical explanation for such behaviour, even if it is predicted by molecular simulations. Systematic additional work needs to be done to try to demonstrate the generality of the phenomenon observed in the case of water and alkanes, and also to try to characterize what are the scales of roughness of the surface which are the most important to suppress wall slip. This is certainly of first importance for the development of micro-fluidic devices.

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