

Friction Measurements with Picoliter Droplets Using Scanning Probe Microscopy

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Topographical and chemical defects on solid surfaces tend to pin three-phase contact lines of moving liquid drops. Our quantitative understanding of the pinning process is, however, still poor. Here we use an atomic force microscope to slide ≈ 100 pL droplets of water-glycerol mixtures over hydrophobic surfaces and measure friction forces. By using picoliter droplets, the sensitivity for detecting processes at the contact line is enhanced. We have found that only a region < 200 nm around the contact line contributes to friction. By imaging isolated nanospherical defects, we could quantify the force and energy dissipation when the front and rear of the droplet passes the defect and compare it to theory.

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Water drops sliding over surfaces is an everyday phenomenon that is relevant for many industrial applications. For example, water drops easily slide off feathers, or fur, that keep birds and mammals dry and warm. Important applications include printing, coating, and condensation and evaporation in heat exchangers. Still, our knowledge of the forces acting on moving drops is incomplete. Even for a simple wetting configuration, such as a tilted plate, it is not yet clear which friction forces limit the speed of sliding drops [1,2]. Drop friction is concentrated at the three-phase contact line, hereinafter referred to as the “contact line.” To analyze the influence of the contact line and the role of defects we measured the friction of small drops. The smaller the drop, the larger the relative contribution of the contact line to the friction force. Usually drop friction is measured with macroscopic drops of 1–50 μL volume applying the tilted plate method [3–6] or drop adhesion apparatus [7–11] or using a centrifuge [12–15].

Atomic force microscopy (AFM) provides the capacity for probing otherwise imperceptible interaction forces, typically within the nN to μN range [16–18], which yields crucial information about the mechanics governing liquid-solid contact lines. Here, we image a surface with 60–100 pL

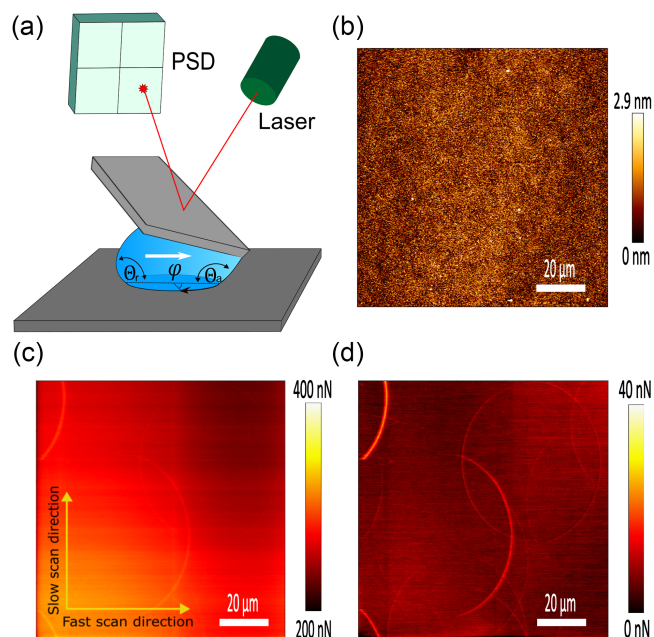


FIG. 1. (a) Schematic of droplet probe microscopy. The torque on the cantilever is caused by capillary forces acting at the contact line between droplet, air, and sample surface. This leads to a lateral deflection of the laser beam that is recorded with the quadrant position sensitive detector (PSD). (b) AFM image of a typical Teflon surface. (c) Representative $100 \times 100 \mu\text{m}^2$ friction image of a Teflon surface scanned with a 84 pL water-glycerol droplet. The fast scan direction (225 $\mu\text{m/s}$) was from left to right, the slow (two lines per second) from bottom to top. (d) Friction map after background subtraction.

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droplets using friction force microscopy [Fig. 1(a)]. The droplets were attached to the end of a tipless cantilever as described in [19,20].

The goal of the experiments is threefold. First, we want to experimentally determine the size of the region around the contact line that contributes to the drop friction and determines the contact angles. The associated length scale is referred to as the “peripheral thickness” [21,22].

To our knowledge there is no experimental measurement of this peripheral thickness. A theoretical lower limit is given by the range of interfacial forces between the solid-liquid and liquid-air interfaces [23–25], which is of the order of 10 nm. Another limit is given by the forces exerted by the surface tension of the liquid on the solid surface. The surface tension is balanced by an elastic response in the solid. Molecular dynamics simulation showed that for a rigid solid, this stress is typically distributed over a region of ≈ 5 nm width [24–26].

Our second goal was to better understand the influence of nanoscopic defects on drop friction. When a sliding drop encounters a defect, its motion is hindered by the local pinning of the contact line. The contact line is deflected until the force is strong enough to depin from the defect [11,27,28]. The work done to deflect the contact line is lost by viscous dissipation [29–32]. Here, we address the following question: what is the force and how much energy is dissipated for a contact line passing over a nanoscopic defect? The energy barriers posed by nanoscopic defects are much higher than thermal energy $k_B T$. In contrast, defects treated in molecular kinetic theory consider barriers on the order of $k_B T$ [32–34].

The third goal was to determine how much a certain segment of the contact line contributes to the total friction force. How strong is the force of a small, single defect when passing a drop’s contact line at different azimuthal angles φ ?

To measure friction forces with picodroplets, tipless AFM cantilevers (MikroMasch® CSC37 tipless, uncoated silica, 250 μm long, 35 μm wide, 2 μm thick, nominal normal spring constant 0.8 N/m) were coated with trimethoxyoctadecylsilane by chemical vapor deposition for 1 h in a dessicator, as described in [35]. Then, a hydrophilic spot of diameter 35 μm was formed at the lower end by removing the silane layer with a focused ion beam (FEI Nova 600 Nanolab) to act as a pinning site for the droplet. We used a mixture of 40% glycerol and 60% MilliQ water (18.2 MOhm/cm) to reduce evaporation. This mixture has a surface tension $\gamma = 69.7$ mN/m, a density $\rho = 1130$ kg/m³, and a viscosity of 3.8 mPa s [more details about sample preparation and the drop probe in Supplemental Material (SM), Sec. 1 [35]]. We measure friction forces by scanning with the droplet over the surface in friction mode AFM (Nanowizard IV JPK) [Fig. 1(a)]. The friction force of the droplet leads to a twist in the cantilever, causing a lateral deflection signal of the AFM photodetector. The scan speed was 225 $\mu\text{m/s}$ recording one

image took 8.5 min. As samples, we used indium tin oxide (ITO) coated glass slides (CEC020T Präzisions Glas & Optik GmbH, 60 $\times$ 25 mm², 0.175 mm thick) that were coated with Teflon by dip coating. The ITO coating reduces charging. Teflon films had a thickness of 60 nm and a roughness of ≈ 0.5 nm, measured with AFM over 100 $\times$ 100 μm^2 [Fig. 1(b)]. To characterize samples, advancing (Θ_a) and receding (Θ_r) contact angles were measured by inflation-deflation cycles using a sessile drop goniometer (OCA35, DataPhysics Instrument GmbH). For 40% glycerol in water, $\Theta_a = 114 \pm 1^\circ$ and $\Theta_r = 108 \pm 1^\circ$ (SM, Sec. 1 [35]). Friction force images obtained on a Teflon surface show ringlike structures with diameters of ≈ 60 μm on a strong background friction force of ≈ 350 nN [Fig. 1(c)] [37]. To better analyze these ringlike structures, we subtracted the background using a polynomial (degree 5) baseline fitting and median method [Fig. 1(d)]. We believe that these ringlike friction peaks are caused by small, randomly distributed defects on the Teflon surface. Every defect leads to one ring. Since the defects were much smaller than the droplet, the image reflects the shape of the contact line. These images verify that only a thin region around the contact line contributes to drop friction. Defects inside the droplet do not contribute. The weakest defects detectable were ≈ 1 nN above the background force and caused rings of ≈ 200 nm width. The width has been corroborated with different measurements with higher lateral resolution (and more than 512 pixels). These results set an upper limit for the size of the peripheral thickness. In contrast to friction forces measured with solid tips, calibration for experiments with droplets is more challenging. Here, we chose to use the background force as a reference [see SM [35] Sec. 2 Fig. S2] and assume that it can be described by the capillary force. The capillary force is obtained by integrating the lateral component of the surface tension around the contact line [38,39],

$$F = kw\gamma(\cos \Theta_r - \cos \Theta_a). \quad (1)$$

Here, w is the width of the contact area, γ is the surface tension of the liquid, and k is a shape factor. We used $k = 0.77$, as suggested for small drops with an almost circular contact line [40]. From the ringlike features we obtain the width of the droplet $w = 66$ μm [Fig. 1(d)]. With $\Theta_a = 114^\circ$ and $\Theta_r = 108^\circ$ we obtain $F = 346$ nN (SM, Secs. S2 and S3, Fig. S3 [35]). Our calibration is, however, only an order of magnitude. Considering an error of $\pm 2^\circ$ in the contact angles and ± 0.1 in the k factor the error in the force calibration was roughly $\pm 50\%$.

To ensure that liquid evaporation was not relevant in our measurements, we measured droplet mass via the resonance frequency of the cantilever at different stages of the experiment (SM Sec. 4 [35] [20,41,42]). Before starting the friction measurement in the example [Fig. 1(c)], the droplet had a volume of 84 pL. With every scan, the droplet lost

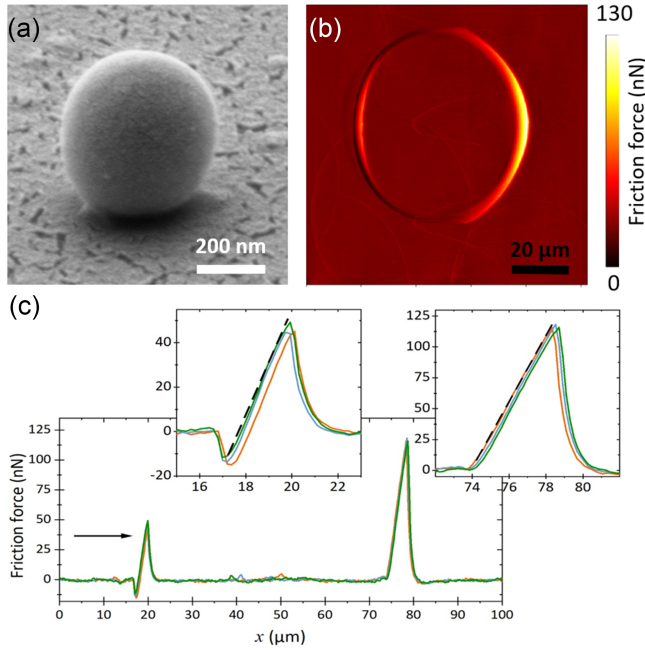


FIG. 2. Experimental results with a single nanosphere as a defect. (a) Scanning electron microscope (ZEISS GeminiSEM 560) image of a single embedded silica nanosphere on a Teflon surface. (b) Friction map recorded with a droplet of 84 pL glycerol-water. Scan area: $100 \times 100 \mu\text{m}^2$. A constant background was subtracted. (c) Friction force versus x position for three traces recorded in (b) at y positions of 51.8 (orange), 53.7 (blue), and 55.7 μm (green). The dashed lines in the insets illustrate the effective spring constant of the contact line pinned by the nanosphere.

between 0.8 and 2 pL, which was only 1%–3% of its volume.

To further understand the influence of defects on contact line friction, silica spheres with a nominal diameter of 500 nm (Bangs Lab.) were attached to flat Teflon surfaces [Figs. 2(a) and S1 [35]]. A representative two-dimensional image of the friction force after background subtraction is shown in Fig. 2(b). Again, ringlike features were observed. The peak force, however, was stronger than for the random defects on the bare Teflon surfaces. The diameter of the ringlike feature was 66 μm (in y direction) and corresponds to the width of the contact area of the droplet. Three cross-sectional traces taken near the center of the ringlike feature [Fig. 2(c)] illustrate the force exerted by the nanosphere on the droplet.

To interpret such force traces quantitatively, we derive a simple theoretical model. As a model defect we consider a single spherical particle of radius R , which is partially sintered to a planar substrate [Fig. 3(a)]. The degree of sintering is described by the opening angle β . The contact angle of the droplet on the substrate is Θ_a for the front and Θ_r for the back. To describe how such a defect influences drop motion, we consider a drop moving with its center over such a defect. The defect is supposed to be much

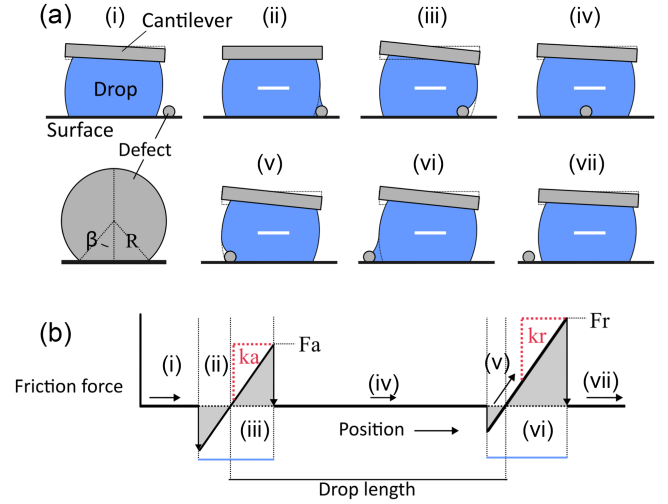


FIG. 3. (a) Schematic front view of the sequence of events when the drop passes a spherical defect. (b) Capillary friction force acting on the drop according to the linear spring model. Blue arrows indicate the width that was used as an upper limit for the peripheral thickness.

smaller than the drop size and the capillary length, $\kappa = \sqrt{\gamma/\rho g}$, where $g = 9.81 \text{ ms}^{-2}$ is the standard acceleration of gravity. In this case, the liquid surface appears planar on the length scale of the defect. Drop motion is assumed to be so slow that at any given drop position its shape is at equilibrium. Before the defect comes into contact with the front contact line, the friction force is determined by the advancing and receding contact angles of 114° and 108° , respectively, and Eq. (1) [Fig. 3(a), i]. As the drop approaches the defect, at some point it makes contact. The defect is wet and forms a meniscus that extends in front of the drop (ii). The meniscus pulls the drop toward the defect. As the drop continues to move, the meniscus becomes smaller. Eventually it disappears and there is no capillary force acting on the drop. With further movement of the drop, a meniscus is formed again, this time extending into the drop and pulling opposite to the direction of motion (iii). The front contact line is released from the defect when a certain force threshold, F_a , is exceeded. Once the defect is fully inside the drop, it no longer exerts any force on the drop (iv). When the back of the drop touches the defect, the same sequence of events occurs: the back surface of the liquid jumps forward and partially dewets the particle (v). After passing the point of zero force a rear meniscus is formed (vi) until a threshold force of F_r is reached and the drop is finally released from the defect (vii). Results of this model are confirmed by surface evolver simulations for a hemispherical defect (SM Sec. 7 [35,36]).

To estimate the effect of the spherical defect quantitatively, we use a linear spring model. Joanny and de Gennes found that the meniscus force exerted by a defect on a moving liquid front increases proportionally to the

extension of the meniscus [28]. Adopting their formalism to the front and rear of the contact line, the corresponding effective spring constants are [28,43]

$$k_{a/r} = \frac{\pi\gamma\sin^2\Theta_{a/r}}{\ln(L/D)}. \quad (2)$$

Here, the main parameter is the ratio between the size of the defect, D , and a large-scale cutoff length, L . Equation (2) was derived for a homogeneous and smooth substrate with a single flat patch of width D . Taking the width of the drop as the large scale cutoff and setting $D = 2R$, we get $k_a = 0.036$ N/m $\gamma = 0.0697$ N/m, $L = 66$ μm , $D = 440$ nm) on the advancing side ($\Theta_a = 114^\circ$) and $k_r = 0.039$ N/m on the receding side ($\Theta_r = 108^\circ$). Experimentally the spring constant is equal to the slope of the linear parts of the friction force plots recorded at $\varphi \approx 0$, as indicated by the black dashed lines in the insets in Fig. 2(c). The measured spring constants were $k_a = 0.027$ N/m on the advancing and $k_r = 0.031$ N/m on the receding side. Considering the error in the calibration, this is a good agreement.

Characteristic features of the friction traces [Fig. 2(c)] are peak forces measured when the defect passes the front and rear of the drop. The first peak is the force required to pull the front of the contact line over the defect (III $\rightarrow$ IV in Fig. 3(a)). We calculate this force in analogy to an expression derived by Scheludko & Nikolov [44]. They derived the maximal force required to move a spherical particle through a fluid interface. Applied to our situation the maximal force is $F_{\max} = 2\pi R\gamma\sin^2(\Theta_a^{\text{def}}/2)$. Here, Θ_a^{def} is the advancing contact angle on the spherical defect. To go from a full sphere to a spherical cap we assume that this maximal force is proportional to the length of the contact line on the sphere. We replace 2π by $2(\pi - \beta)$:

$$F_a = 2(\pi - \beta)R\gamma\sin^2\left(\frac{\Theta_a^{\text{def}}}{2}\right) \quad (3)$$

On the rear side, we consider the force required to move a sphere out of a liquid [43,44]. For this reason, the sine function is replaced by a cosine:

$$F_r = 2(\pi - \beta)R\gamma\cos^2\left(\frac{\Theta_r^{\text{def}}}{2}\right) \quad (4)$$

Here, Θ_r^{def} is the receding contact angle on the spherical defect. Usually F_a is not equal to F_r .

In experiments with the spherical silica defects, the ratio between the front and rear force peaks was ≈ 0.45 . From this ratio we can estimate the contact angle on the defect to be $\approx 70^\circ$ (S5 Fig. S4 [35]). With $\Theta_a^{\text{def}} = \Theta_r^{\text{def}} = 70^\circ$, $R = 220$ nm and $\beta = 35^\circ$ we estimate $F_a = 26$ nN and $F_r = 52$ nN (Eqs. (3) and (4)). Measured forces were

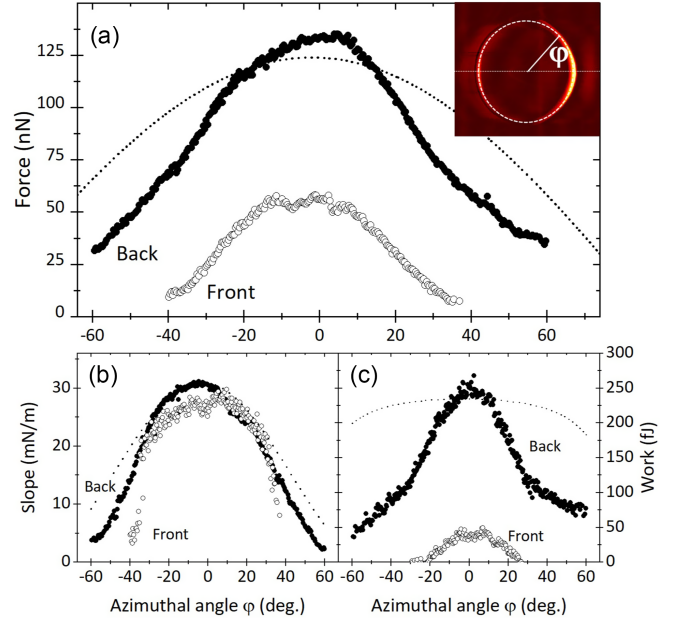


FIG. 4. (a) Peak force, (b) slope, and (c) work of the contact line of the drop when overcoming the defect. It is plotted versus azimuthal angle φ for the front and back of the drop. The dashed lines represent the cosine dependence (a) and the theoretically expected shape of the curves [(b) and (c), described in SM Sec. 6, Fig. S5]. They take into account that we detect only the x components of force and the extension of the meniscus. They were obtained by Eqs. (S1)–(S3) in SM with $\delta_{\max} = 2.8$ μm , a mean radius of the contact area $r = 33$ μm and $k_d = 0.031$ N/m.

roughly a factor two higher, which is still within the limits of the estimated error (Fig. 4).

The friction maps in Fig. 2(b) show that the forces exerted by the defect vary along the contact line. They depend on the azimuthal angle φ [Fig. 4(a)]. To quantify this angular dependence, we fitted an ellipse onto the force maps [inset, Fig. 4(a)]. We analyzed the force scans that crossed the ellipse at a specific angle φ . For $\varphi = 0$ the measured peak forces plotted in Fig. 4(a) correspond to F_a and F_r calculated in Eqs. (3) and (4). The peak force remained high up to $|\varphi| \approx 20^\circ$ and then decreased. For $|\varphi| > 60^\circ$ it was difficult to identify the peak force and subtract the corresponding background. For this reason, for $|\varphi| > 60^\circ$ we can only set an upper limit of $\approx 15\%$ of the central force. Since we measure the x component of the friction force, we would expect it to be proportional to $\cos\varphi$, as indicated by the dashed line (see SM Sec. 6, Fig. S5 [35]). The measured friction force decreased more steeply for high $|\varphi|$ than expected.

The slope of force peak also depended on the azimuthal angle φ [Fig. 4(b)]. To calculate the slope of the force peaks, we fitted the linear increase in scans of force versus x position, as indicated by dashed lines in Fig. 2(b). The slope for $\varphi = 0$ corresponds to the spring constant calculated in Eq. (2). Considering that we only detect the slope in

x direction, the experimental and theoretical results agree [dashed lines in Fig. 4(b)].

To estimate the effect of a defect on the apparent contact angles, we need to know how much work is required to move the contact line over the defect. To calculate this work, we integrate the area under the curve in 2(c) from the beginning of the force peak to its top. This work, W_x , decreased significantly for $|\varphi| \geq 20^\circ$ [Fig. 4(c)]. One reason for this decrease is the fact that W_x does not represent the total energy dissipated W . The total work is independent on φ , as shown by surface evolver simulations (SM Sec. 7, Figs. S6 and S7 [35]). However, like the force, this decrease was steeper than expected [dotted line in Fig. 4(c)].

We do not yet have a clear explanation for the measured steep decrease in force and work with azimuthal angle. One possibility is a premature rupture of the meniscus at high φ due to evaporation, causing the drop to detach. Because of the small size of our meniscus, evaporation may be much more effective than for larger systems. Another possibility is sliding electrification [45,46]. Since our defects are hydrophobic and electrically insulating, negative charges are deposited by the receding contact line. These could stabilize the meniscus at low φ . One possible artifact could be crosstalk between a twisting of the cantilever and a bending in the normal direction (SM Sec. 8, Figs. S8 and S9 [35]). If the drop passes the defect eccentrically, there will be a lateral capillary force F_y in the direction of the cantilever and not just perpendicular to it, F_x (SM Sec. 7, Fig. S7 [35]). This component increases with φ . However, the crosstalk should point in opposite directions for a defect passing left ($\varphi < 0^\circ$) or right from the center ($\varphi > 0^\circ$). Our force images were mirror symmetric indicating that crosstalk is negligible. In the theory, we neglected the eccentricity of the contact line and assumed a circular shape. Again, the error should be small.

The energy dissipated when passing a nanosphere was much lower than the energy dissipated when moving the drop on the homogeneous part of the surface. When moving the drop by $\Delta x = 66 \mu\text{m}$ (diameter of contact area) the energy dissipated was $wk\gamma\Delta x(\cos\Theta_r - \cos\Theta_a) = 26 \text{ pJ}$. In comparison, the additional work required to pass the defect of $\approx 0.3 \text{ pJ}$ is negligible. One would need 87 defects distributed across the contact area to make the work caused by defects similar to that induced by contact angle hysteresis of the smooth Teflon surface. Contact angle hysteresis on the homogeneous Teflon surface may be caused by the deposition of electric charges behind the drop [45,46].

We conclude that using AFM to measure friction forces of sliding picodroplets with a sensitivity better than 1 nN can be resolved. On apparently smooth Teflon surfaces, single nanoscopic defects causing friction forces of 2–5 nN were detected, on top of the friction of $\approx 350 \text{ nN}$ caused by the contact angle hysteresis. From such single nanoscopic defects we can deduce an upper limit for the size of the

peripheral thickness of 200 nm. The central parts of the drop with respect to the direction of motion ($\varphi \leq 50^\circ$) contribute most to defect-induced friction. Energy dissipated by passing a nanoscopic defect is orders of magnitude lower than the drop friction caused by contact angle hysteresis.

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Correction: The contact author information was missing and has now been inserted.