

New two-dimensional friction force apparatus design for measuring shear forces at the nanometer scale

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A device to study the friction of two molecularly smooth surfaces separated by an ultrathin liquid film is presented along with its design, calibration, and performance. The apparatus can move one of the surfaces and measure the friction force on the other one bidimensionally for both processes. A high mechanical impedance system (10^4 N/m) measures continuous friction forces where only stick-slip was previously observed. The frequency and travel distance of the movement can be varied over a wide range (frequency from 10^{-4} to 7 Hz and distance from 1 to 800 μm) to provide variations of the shear rate over seven orders of magnitude. The actual movement provided by piezoelectric bimorph drive can be affected by the friction forces and is measured by strain gauges. The friction forces are measured with an accuracy of $\pm 2\mu\text{N}$ with a capacitance sensor. The mechanical design prevents the surfaces from rolling under force. The apparatus is tested with hexadecane. The potential applications of this apparatus and its limitations are discussed. © 2001 American Institute of Physics. [DOI: 10.1063/1.1412860]

I. INTRODUCTION

Friction has been, together with adhesion, very important subjects for the advancement of civilization: Amonton's law was one of the first attempts to explain it in a simple way, but the reality is that friction is still poorly understood.¹ For example, the contributions of surface roughness^{2,3} and of molecular contribution^{1,4} to friction are not yet established. Due to its importance in industrial processes (space, nuclear, chemical, aircraft, storage disk industries) and because it is associated to wear, friction is the subject of much research.⁵

Tribology, the science that deals with the study of friction, wear, and lubrication of moving surfaces, has tried to predict the friction in many macroscopic processes, mostly from a mechanical point of view.⁶ One of many challenges of measuring friction is its reproducibility: a friction coefficient is not a defined parameter like density or dielectric constant. It is highly affected by numerous factors always difficult to control. It is in recent years that the field of nanotribology has attracted many researchers from very multidisciplinary backgrounds.⁵ At this size level it is difficult to understand what is the nature of the friction of layers or even single atoms. Which aspects of the behavior of thin films can be attributed to the confinement alone? When does a liquid thin film stop behaving like a continuous fluid? The molecular nature of friction is now a very passionate subject and there

is even more to know about how one can control the macroscopic effects.

Some authors^{2,3,7} have focused their studies on rough macroscopic surfaces (multicontacts) and some others^{1,8-13} on microscopic friction between molecularly smooth surfaces (single contacts), and some consensus has emerged that both approaches are complementary. Although much work has already been done, there is still a poor predictive capacity about the friction forces produced by any new system that was not previously tested. Microscopic friction has been studied by the friction force apparatus (FFA)^{1,8-13} derived from the surface force apparatus (SFA)¹⁴ and by the friction force microscope (FFM)^{15,16} derived from the atomic force microscope (AFM).¹⁷ The advantage of FFM is that it is a fairly sensitive method and it can image the different zones of a surface which is inhomogeneous from the point of view of friction.^{15,18} However, it does not give access to the geometry of the contact between the tip and the sample. The FFA gives access to the size and profile of the contact zone, and to the thickness of the lubricating film when present, which is very useful for understanding quantitatively the forces as a function of the various parameters. While the existing FFA have already provided much insight through the pioneering work of Israelachvili, Granick, and Klein, some of their features could be improved in order to investigate some new tribological phenomena. For example, transient effects may not be eliminated after a sliding of several tens of microns, and longer travel distances may be appropriate.

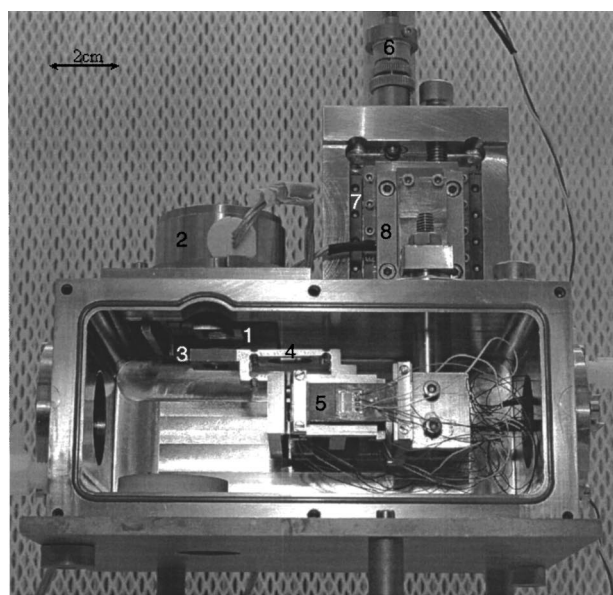
The existing apparatuses that have been constructed have either a short travel distance with a high force accuracy,¹¹⁻¹³ or a longer travel range with a lower force resolution.¹⁹ The control of the shear rate is also very important and the use of bimorph piezoelectric components gives

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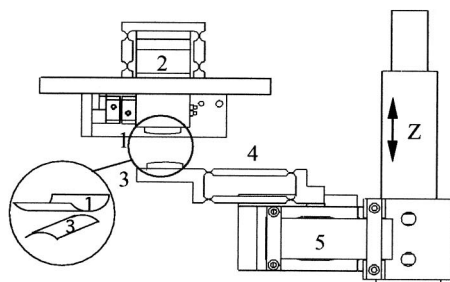
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(a)



(b)

FIG. 1. (a) Image of the apparatus. The micrometric unit 6 pushes the slide 7 that carries the piezotranslator 8; (b) upper 1 and lower 3 surfaces, friction force measuring device 2, normal force spring 4, bimorph piezoelectric drive 5.

limitations to this control because of their flexibility. The relationship between the film lubricant structure and the friction behavior is not clear and bidimensional friction may be necessary to investigate some possible anisotropic effects. The small mechanical impedance of the friction force measuring device often limits the possibilities of study.

We have therefore designed a prototype of a FFA which introduces new features and improved performances. The purpose of this article is to describe this apparatus as well as to prove its functionality.

The apparatus is based on the same interferometric technique as the classical SFA which allows accurate distance measurements. Our prototype combines: bidimensional measurement of the friction forces; an accurate X - Y (bidimensional) measurement of the bimorph lateral movement ($\pm 0.2 \mu\text{m}$) that provides a high control of the shear displacement and rate; a high sensitivity on the friction force ($\pm 2 \mu\text{N}$); a long travel range ($800 \mu\text{m}$); a high mechanical impedance of the measuring system (10^4 N/m); a design that prevents any rolling of one surface relative to the other. Therefore we can both drive and detect the friction bidimensionally.

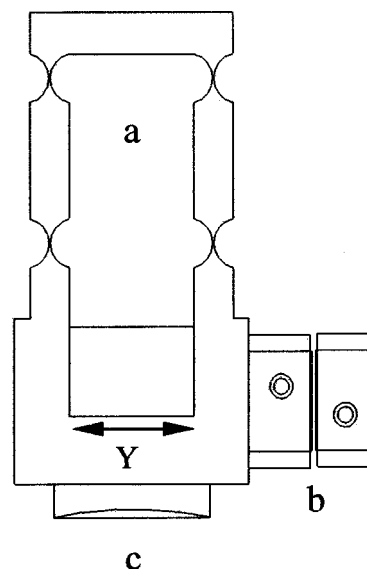


FIG. 2. (a) Spring with flexure hinges; (b) capacitance plates (Y movement); (c) cylindrical lens bearing one mica sheet.

II. PRINCIPLES OF OPERATION

As in the now classical SFA,^{1,8-14,20-22} the apparatus features two curved molecularly smooth mica surfaces which are horizontal, with a curvature of about 2 cm, arranged in a crossed cylinder geometry. The distance D between the surfaces is measured by multiple beam interferometry²² to within $\pm 1-2 \text{ \AA}$. D is controlled by a three-stage translation. The force normal to the surfaces, i.e., the load, is measured by a spring. All the springs of this apparatus are deformable parallelograms with flexure hinge²³ which ensure that only translation and no rotation of the surfaces occur during the movements.

The friction is produced between the two surfaces 1 and 3 of Fig. 1. The upper surface 1 is attached on a system 2 which measures the friction forces. It can only move horizontally. The lower surface 3 is attached by means of a spring 4 to a bimorph piezoelectric system 5 that generates a lateral movement to create friction. Strain gauges on the bimorph elements measure the actual lateral displacements. The bimorph system is attached to the vertical translation that controls the distance between the surfaces. A two-stage micrometric screw 6 drives a slide 7. The third stage consists in a piezotranslator 8. The load is measured by the spring 4 with a stiffness of 700 N/m .

All the pieces are made in 316L stainless steel except those of the bimorph system which are made of titanium alloy TA6V to reduce their mass.

A. Friction force measuring device based on capacitance plates

The friction force measuring device uses capacitance plates (Physik Instruments D-015.00 with the control electronics E-509.C3) that probe the displacement of a spring to which the upper surface is attached. When the lower surface slides against the upper, any friction force will deform the spring. The latter is a deformable parallelogram spring with flexure hinges shown in Fig. 2. One of the capacitance plates,

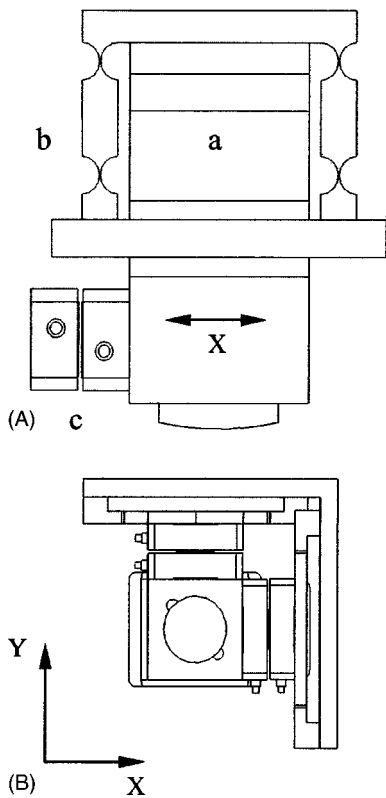


FIG. 3. A (a) Spring of Fig. 2 rotated by 90°; (b) second spring, perpendicular to the first one, resting on an immobile part of the apparatus; (c) capacitance plates (Y movement); B bottom view of the system shown in part A; the two pairs of capacitance plates probe the movements of the lens.

the “target,” is attached to the spring while the other the “probe,” is at a fixed position. Since errors in parallelism can influence the linearity and gain factor, some adjustment of the probe position is allowed by the design to obtain a parallel positioning of the plates at a distance of 15 μm.

While this force measuring device works on the same mechanical principle as one previously described,²⁴ the two-dimensional friction measurement device is more complicated and is shown in Fig. 3: two independent springs are assembled and each of them is used to measure the friction force in one direction by means of capacitance plates that are mounted accordingly.

The friction force measuring springs can be optimized to combine high sensitivity and wide range (0–100 mN). Higher force sensitivity can be attained by decreasing the spring constant, but this also lowers the friction measuring range. Here, the spring constants are about 10 000 N/m. The distance range of the capacitance plates was adjusted to 12.5–17.5 μm and the output voltage was 0–10 V. In these conditions, the friction force sensitivity is about 2 μN, which corresponds to a displacement of 0.2 nm. The mechanical coupling between the X and Y capacitance measurements is weak because of the high stiffness (10⁴ N/m) of the force measuring springs: when a large friction force, for example 1 mN, is measured in one direction, the resulting spring flexion is only 0.1 μm and this affects only slightly the other capacitance measurement (see Fig. 11). The electrical coupling between the X and Y capacitance measurements is sufficiently small so that it cannot be seen in the measurements: the

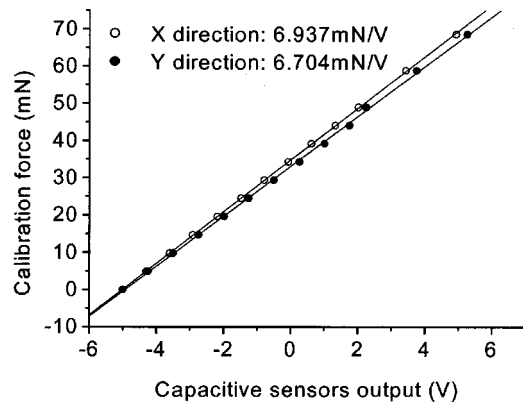


FIG. 4. Calibration of the capacitance plates for X and Y directions (temperature 24 °C, humidity: 35%).

friction force measured in one direction will not depend on whether or not it is measured in the orthogonal direction (not shown).

The plates capacitance was calibrated against known applied forces. Figure 4 shows the calibration curves of the capacitance plates for X and Y direction friction measurements. Compared with strain gauges, the capacitive plates not only have high natural frequency (the frequency bandwidth can be adjusted between 300 and 3000 Hz), but also high displacement sensitivity (0.1 nm) and therefore good force sensitivity.

This sensitivity, combined with temperature drift and mechanical vibrations, gives an accuracy on the friction forces of ±2 μN (shown in Fig. 5). The typical drift of the capacitance plates distance is 0.3 nm/min. In many cases, this drift can be partly corrected by measuring the friction corresponding to one direction, and immediately after, on the opposite. The alignment of the two springs in order to have perpendicular flexions is not perfect, and a friction on the X direction always produces a small signal in the Y direction. This will be discussed in the Sec. IV.

B. Piezoelectric bimorph drive

To produce friction, the lower surface is driven horizontally by a piezoelectric bimorph system. This surface is supported by a spring used to measure the normal forces (Fig. 6)

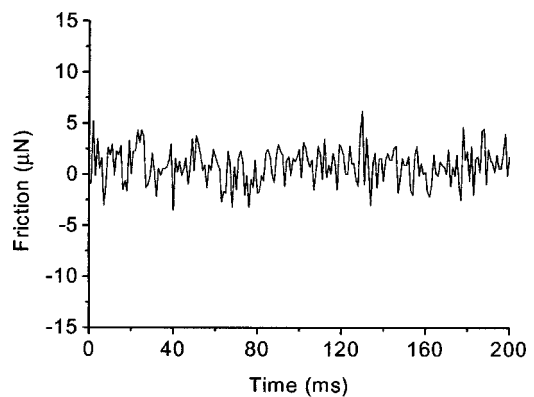


FIG. 5. Noise recorded in the absence of friction forces in the X direction. The noise in the Y direction is similar (not shown).

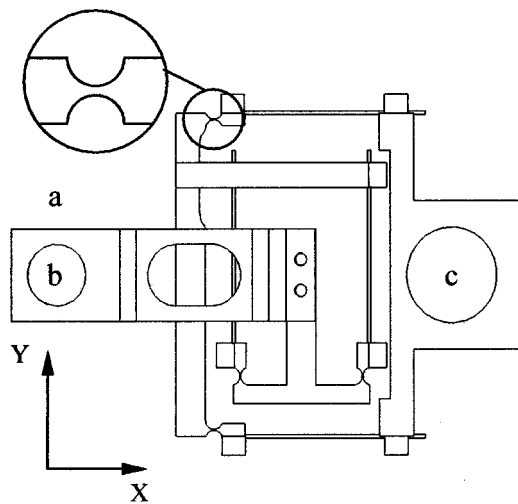


FIG. 6. Bidimensional bimorph piezoelectric drive. The movable ends of the bimorphs are attached to flexible parts in order to obtain the largest possible movement. (a) Spring for normal forces; (b) lens supporting the lower mica surface; (c) hole for attachment to a rod driven by the vertical translation. This bimorph system is attached to the vertical translations of the apparatus.

and the spring is driven in two horizontal directions by two pairs of bimorph piezoelectric elements that produce orthogonal displacements (Fig. 6).

The outer (inner) pair of bimorphs produce a displacement perpendicular (parallel) to the axis of the normal force spring. Bimorphs can produce much larger displacements than piezoelectric tubes for the same applied voltage^{11,19} and are often used as displacement transducers and force sensors.^{20,21,25} The bimorphs are not insulated and the immersion of the surfaces in a liquid is not presently possible, so a drop of the liquid to be studied must be introduced between the surfaces. The bimorphs are Philips (Ref. 0201462), with a size of $35 \times 12 \times 0.6$ mm and with a displacement of $935 \mu\text{m}$ for ± 150 V. In order to take advantage of this long travel range, we designed a flexible spring structure for the attachment of one end of the bimorph, and we adjusted the mounting position to get the largest free length L of the bimorph (the displacement is proportional to L^2). The bimorph strips were attached at one end to the vertical translations (those used to control the distance and the load between the surfaces), and at the other end to a flexible part, as indicated in Fig. 6. This way, total travel distances of $700 \mu\text{m}$ were made possible for ± 120 V. As the precision of the mounting is limited, the movement of the bimorph system is never perfectly parallel to the mica surfaces, and the angle between them cannot easily be adjusted below 0.003 rad. This can be entirely corrected to keep the distance and load constant by using the vertical translation piezotranslator (described in the next section) which is computer controlled.

The free length L of the piezoelectric bimorphs is 26 mm, giving a stiffness of 4000 N/m for each bimorph slider. The effective mass of the whole bimorph system is about 50 g, which determines the upper limit for dynamic measurements (corresponding to the resonance frequency of the drive) about 50 Hz.

The calibration curves of bimorph strips movement as a

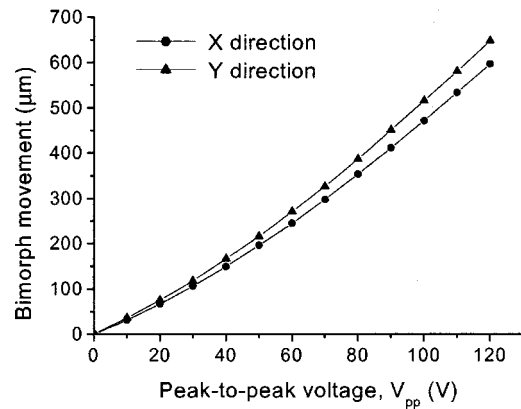


FIG. 7. Calibration of the bimorph displacement for the X and Y directions (temperature 24°C , humidity: 35%).

function of the input voltage (not shown) for the X and Y direction drive display the typical hysteresis curves expected from piezoelectric materials. When the tension increases from -120 to $+120$ V, the X and Y sliders move, respectively, by 650 and $700 \mu\text{m}$, which is more than 100 times larger than piezoelectric tubes for the same applied voltage.¹¹ Repeating the calibration for different electrical tensions, we can obtain the mechanical characteristics of the bimorphs in Fig. 7, which display some nonlinearity with the input voltage.

All the above calibrations are made with no friction force. When a friction force is acting on the bimorph, its displacement will be smaller because of bimorph flexibility. To improve the control on the displacements, resistance strain gauges were glued on each bimorph surface. When used with a strain gauge amplifier (Vishay Micromasures, model 2110B), the bimorph displacement sensitivity is about $0.2 \mu\text{m}$. From the calibration results showed in Fig. 8, the strain gauges output changed linearly with the bimorph movement with a coefficient of 0.153 and $0.184 \mu\text{m}/\text{mV}$, respectively, for X and Y direction drives. Furthermore, due to the flexible design of the attachment of the normal spring to the bimorph drives, this attachment elastically bends under a force with a stiffness constant of 9480 N/m. The actual movement of the normal force spring is equal to that of the movement of the bimorph as probed by the strain gauges minus the flexion of the attachment. The lateral movement of the normal spring is therefore controlled to $\pm 0.2 \mu\text{m}$.

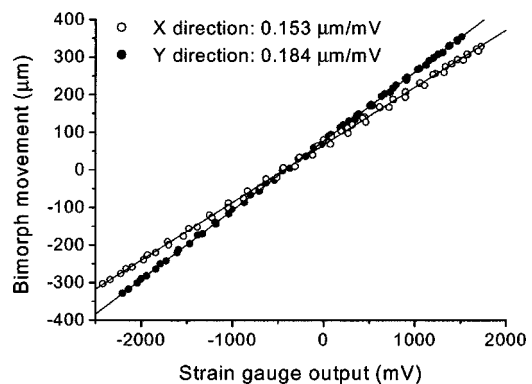


FIG. 8. Calibration of the strain gauges glued on the bimorphs for the X and Y directions (temperature 24°C , humidity: 35%).

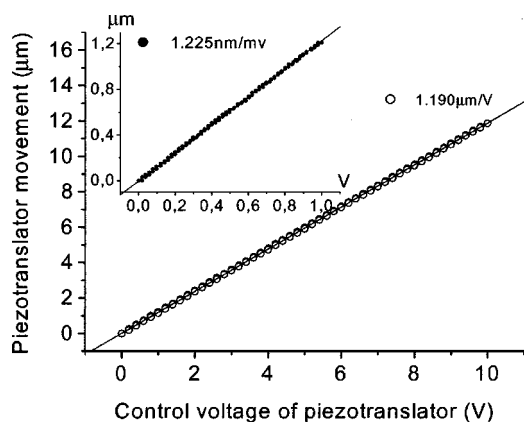


FIG. 9. Calibration of the piezomovement normal to the surfaces. The inset shows the precision of the movement.

In friction experiments, a triangular voltage input is often used to move the surfaces at constant speed. It produces constant velocity motion in one direction up to the turning point and then repeatedly in the reverse direction. This way, very low or high constant velocities can be achieved. The range of speeds attainable using a function generator that provides a 1–120 V peak–peak triangular signal (corresponding to 10–1400 μm total travel per cycle) is from 1 nm/s at a driving frequency of 10^{-4} Hz and 1 V peak–peak triangular signal to 10 mm/s at 7 Hz and 120 V. For a film thickness of 10 nm, these speeds correspond to shear rates that can be varied from 0.1 to 10^6 s^{-1} —a range of 7 orders of magnitude.

Needless to say, all kinds of periodic or nonperiodic movements can be programmed for the experiments and allow it to work as a nanorheometer to obtain dynamic modulus or to perform creep relaxation experiments.

C. Control of the distance between the surfaces and measurement of the load

The distance between the surfaces is controlled with a three-stage translation system which vertically drives the piezobimorph system shown in Fig. 1. A two-stage micrometric screw 6 (Microcontrole BM17.04N) provides 1 μm sensitivity on 6 mm travel range and 0.2 μm sensitivity on 300 μm (see Fig. 1). It drives a slide 7 mounted on a prestrained cylinder bearing. The third stage is a piezotranslator 8 (Physik-Instruments P-753) that has a repeatability of 1 nm over a travel range of 12 μm . It includes one pair of capacitance plates (Physik Instruments, mode D-015.00, range 6–18 μm) and control electronics (Physik Instruments, mode E-509.C3 and E-505.00) for closed loop operation. It provides a drift-free and hysteresis-free linear movement, high stiffness, and long-term position stability. Figure 9 shows a typical calibration of the piezotranslator movement. A more detailed plot (inset) shows that it can move linearly with 20 nm steps. It is calibrated prior to each experiment with the FEKO fringes, and its sensitivity is about 0.1 nm.

The load, i.e., the normal force between the surfaces is measured by the spring 4 with a stiffness of 700 N/m which relates the lower surface 3 to the bimorph system 5 (see Fig. 1). This spring is also a deformable parallelogram with flex-

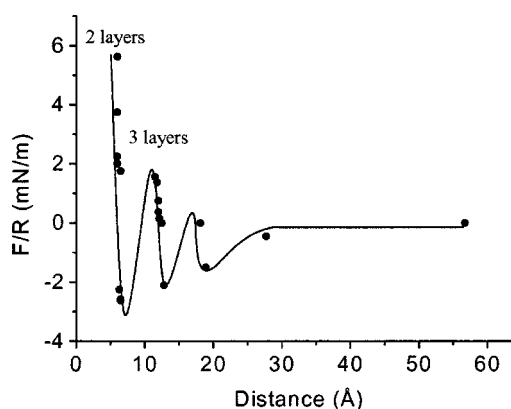


FIG. 10. Force/distance profile of mica surfaces across hexadecane (temperature 24 °C, dry atmosphere).

ure hinges which prevent the rolling of one surface on the other under large loads. Its flexion under a force is deduced from the difference between the displacement of its base and the actual change of distance between the surfaces measured interferometrically.

III. TEST OF THE APPARATUS WITH HEXADECANE

A. Normal force versus distance curve

The measurements of the interaction forces normal to the surfaces as a function of their distance give access to many of the properties of the film confined between the surfaces. This has been the subject of intense studies, in the last decades, aimed towards the understanding of the loss of bulk properties when a liquid is trapped between two surfaces at distances of a few molecular diameters. Many liquids have been shown to be structured in layers when confined between two surfaces. The normal force was oscillatory as a function of distance, with a spatial period that was equal to one of the molecular dimensions. Any information on the film structure is useful to analyze the friction forces and also to define in which conditions they are to be measured. Therefore, prior to friction measurements, the normal forces were measured as a function of distance in each experiment.

Force/distance profiles were measured between mica surfaces separated by hexadecane. 20 μl of hexadecane (Merck, gas chromatography grade, 99.5% purity) were introduced between the mica surfaces and equilibrated overnight with P_2O_5 in the air inside the apparatus to eliminate any trace of water between the surfaces that could affect the properties of the hexadecane film.²⁶ Figure 10 shows a typical normalized force/distance profile for hexadecane. The profile displays forces oscillating with distance, with a period of ≈ 0.45 nm (corresponding to the thickness of the molecule) showing that the confined hexadecane is structured in layers. Each maximum corresponds to a discrete number n of monolayers between the mica surfaces. This profile is similar to those previously reported for this liquid.²⁶ With hexadecane, it was difficult to bring the mica surfaces closer than a distance corresponding to two hexadecane monolayers and this prevented a reliable reference contact for zero distance. The latter was obtained at the end of an experiment by in-

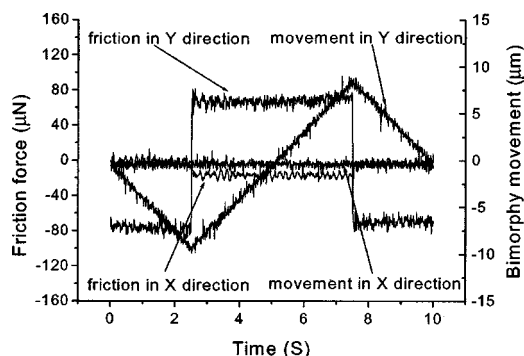


FIG. 11. Friction traces of hexadecane for Y movement only, with two layers between the surfaces; load $100 \mu\text{N}$, temperature 24°C . The X and Y movements are measured by the strain gauges.

serting a drop of water in the hexadecane which displaced for wettability reasons, and enabled it to make the two surfaces go into molecular contact in water.

B. Friction force measurement

Once the force/distance profile is known, we are able to position the surfaces at different distances relative to the layer structure of the liquid. The friction forces were measured for surface separations of two and three layers at various loads. Figure 11 shows one typical frictional trace for highly compressed hexadecane films ($100 \mu\text{N}$ load) of thickness about 0.9 nm at sliding velocities of $4 \mu\text{m/s}$. The displacement of the lower surface probed by the strain gauges is shown. It is not exactly proportional to the voltage input (ramp voltage, not shown) because of the hysteresis of the piezoelectric bimorphs and of their flexibility under the friction force which is the large square signal. The friction force in the Y direction also shows a nonzero signal. Three effects are responsible for this signal in the Y direction: (i) the two springs that probe the friction forces are not exactly orthogonal; (ii) the movement of the lower surface produced by the bending of the bimorphs under electrical tension is a translation along a parabola (if only the bimorphs X are actuated, the component in the Y direction will be $Y = 2X^2/L$, L being the free length of the bimorphs); (iii) when the distance changes between the two capacitance plates of the X friction, one of the plates of the Y direction is shifted laterally, as mentioned in Sec. I and this affects the measure of the capacitance. In any case, the computer controlled feedback system can be used to compensate for any kind of asymmetry.

In Fig. 11, the friction sliding takes place along the Y direction. The traces show that the friction force is about $70 \mu\text{N}$ at the load of $100 \mu\text{N}$. We could repeat these measurements with different loads ranging from -20 to $300 \mu\text{N}$. It was possible to use negative loads because of the oscillating nature of the force/distance profile in Fig. 10. Performing these measurements with two and three hexadecane layers between the surfaces yielded the curves shown in Fig. 12. We have limited the range of loads to $300 \mu\text{N}$ because above this value, surface damage easily occurred. This could be seen from the friction traces which showed an irreversible change of friction behavior, and, as the damage increased, it could be seen from the interference fringes.

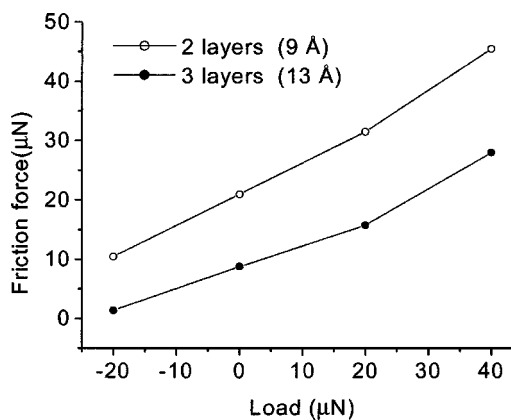


FIG. 12. Friction force as a function of load for two (closed circles) and three (open circles) hexadecane layers between the mica surfaces. Sliding speed: $35 \mu\text{m/s}$ (temperature 22.9°C).

The friction force varies approximately linearly with the load, which corresponds to Amontons's law. The slopes are equal to the friction coefficients. Such friction coefficients are represented in Fig. 13 for different sliding speeds for two and three hexadecane layers. As expected, the friction coefficient is larger for two than for three layers. The friction force decreases with the speed of sliding. This is the solid-like regime which occurs at medium speed ($0.3\text{--}20 \mu\text{m/s}$). In this regime, the chain molecules still have the time to make entanglements, but as the speed increases, they have less and less time.²⁷ This is in agreement with the values obtained for tetradecane¹ (friction coefficient $\mu \approx 1$) which differs by two CH_2 from hexadecane. However, our data differ from previous data²⁸ obtained from a similar system. The difference is not surprising considering that the experimental conditions were largely different from ours (loads were two orders of magnitude higher and stick-slip was observed).

IV. DISCUSSION

We have built a FFA. This apparatus provides long travel distances and a precise mapping of the surfaces. It studies the normal and friction forces of thin films, i.e., transient states of lubricant films under shear, asymmetric friction, viscosity measurements, etc. One of the particularities of our design is that any mechanical coupling or misalignment between lat-

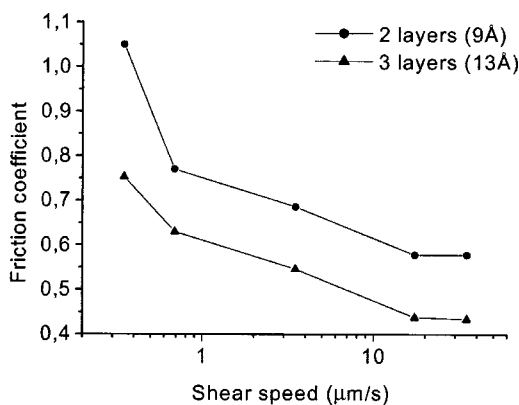


FIG. 13. Speed effect on the friction coefficients of hexadecane for two and three layers (temperature 22.9°C).

eral and vertical movements can be corrected allowing the use of long travel distances and maintaining constant the load and the distance between the surfaces. This correction is possible by means of the closed loop piezoelectric translator which is computer programmable by a feedback voltage tension. The large travel distance also obtains a large range of sliding speeds which make it possible to study a wide range of situations with different relaxation times.

The apparatus provides bidimensional friction forces measurements but, because it uses two systems of piezoelectric bimorph strips, it has a low mechanical resonance frequency. A FFA in which strain gauges are used to measure the friction forces in two perpendicular directions has been patented by Israelachvili.²⁹ Our apparatus allows work with a drop of liquid between the surfaces. It is also possible to fill up the box, keeping the surfaces immersed in the liquid by coating the bimorphs with an insulator.

The springs that measure the friction force are extremely stiff ($k = 10^4$ N/m) in contrast to most FFA. This compares with the FFA reported by Peachey *et al.*³⁰ in which a spring constant of $10^3 - 10^4$ N/m is used. In the present FFA, a friction force along the X direction will result in a small displacement of one of the capacitance plates, visible by the Y capacitance system [see Fig. 3(B)], but the signal will be sufficiently small so that it will not prevent reliable measurements in the Y direction. The high stiffness of the springs has the advantage that reduces the coupling between the X and Y friction force measurements. However with this high mechanical impedance, in most cases, the apparatus does not allow, stick-slip phenomena to be observed at the speeds used. This offers the possibility to measure continuous friction forces where it was previously only possible to measure stick-slip friction. Stick-slip can be due to the apparatus but is also influenced by the critical shear stress that a squeezed film can undergo without moving under load. The influence of the mechanical slip is highly diminished by our setup.

Another of the new and key features of this prototype is the mechanical control of the orientation of the surfaces during the movements: this is due to the design of the springs, and it prevents the surfaces from rolling onto each other, even under high loads.

This apparatus should be useful in investigating the effect of the order in the lubricant films on their tribological properties, and also on anisotropic friction that may occur in some situations like, for example, dry friction between the facets of crystalline solids.

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