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Recent advances in the surface forces apparatus (SFA) technique

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Abstract
The surface forces apparatus (SFA) has been used for many years to measure the physical forces between surfaces, such as van der Waals (including Casimir) and electrostatic forces in vapors and liquids, adhesion and capillary forces, forces due to surface and liquid structure (e.g. solvation and hydration forces), polymer, steric and hydrophobic interactions, bio-specific interactions as well as friction and lubrication forces. Here we describe recent developments in the SFA technique, specifically the SFA 2000, its simplicity of operation and its extension into new areas of measurement of both static and dynamic forces as well as both normal and lateral (shear and friction) forces. The main reason for the greater simplicity of the SFA 2000 is that it operates on one central simple-cantilever spring to generate both coarse and fine motions over a total range of seven orders of magnitude (from millimeters to ångstroms). In addition, the SFA 2000 is more spacious and modulated so that new attachments and extra parts can easily be fitted for performing more extended types of experiments (e.g. extended strain friction experiments and higher rate dynamic experiments) as well as traditionally non-SFA type experiments (e.g. scanning probe microscopy and atomic force microscopy) and for studying different types of systems.

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

1.1. Before the surface forces apparatus (SFA)

In 1952, Overbeek and Sparnaay were able to measure long-range attractive forces between highly polished glass and quartz plates, but measurements at small distances (<20 nm) were difficult due to dust particles or surface roughness [1]. Distances between the surfaces were determined using Newton interference colors and the forces were determined by measuring the bending of a stiff spring that one surface was mounted on. In 1964, Derjaguin and co-workers used two crossed polarized metal wires (Pt pairs or Au pairs) to measure the potential barrier between the wires in electrolyte solutions [2]. They found that a repulsive force of non-electrostatic origin existed between the surfaces at high electrolyte concentrations and they were able to calculate the Hamaker’s constant for the molecular attraction of metallic filaments in water.

1.2. Early versions of the SFA

In 1969, Tabor and Winterton described the first apparatus where forces between surfaces could be measured for separations as low as 5–30 nm with a 3 Å distance resolution [3]. In this apparatus they used the method of multiple beam interferometry (MBI) for the first time to measure jump distances between molecularly smooth surfaces of mica in air to determine normal and retarded van der Waals forces. In this ‘jump method’, one surface is held by a spring, while the movement of the other surface can be controlled by using a piezoelectric transducer. Israelachvili and Tabor then extended this method by using the ‘jump method’ to measure forces in the range from 1.5 to 20 nm, and also used what they called the ‘resonance method’ in the range from 10 to 130 nm [4].

In the resonance method, the surfaces were held in a low pressure vapor environment. One surface was vibrated at a known frequency while the oscillations due to the van der Waals forces on the opposite surface were measured with a piezoelectric bimorph strain gauge. At this time they also showed the usefulness of the method for measuring forces in air between monolayers deposited on mica surfaces.

1.2.1. SFA Mk I. A new apparatus, later dubbed the Mark I, was described by Israelachvili and Adams as a way to measure the forces between surfaces immersed in liquids [5]. In this apparatus the mica surfaces were moved toward and apart from each other using a motor-driven micrometer and a piezoelectric crystal resulting in a range of control from the micrometer to the ångstrom level. As with the earlier versions, the distance between the surfaces was determined using MBI and the forces between the surfaces were measured by mounting the lower surface on a cantilever spring with a known spring constant.

1.2.2. SFA Mk II. The Mark II was developed as an improved version of the Mk I with extra attachments to enable more interfacial phenomena to be studied [6, 7]. Some of the improvements to the Mk II over the Mk I included a double-cantilever spring for increased strength which also prevented the surfaces from rotating as normal forces were applied. A significant attachment that was added to the Mk II was the ‘friction device’ which allowed the upper surface to be moved in the lateral direction using a motor-driven micrometer while the frictional forces were measured with strain gauges located on springs attached to the upper surface.

1.2.3. SFA Mk III. As the SFA continued to be used for increasingly complex systems, it was found that the Mk II needed to be improved. It needed to be more stable to thermal drifts, have a stiffer spring or a variable stiffness spring, have a more linear and larger range of motion and it needed to be easier to clean. The Mk III was developed and tested during the period 1985–1989 and then described by Israelachvili and McGuiggan [8]. In this new version the apparatus was made more compact than previous versions, which was better in systems where the surfaces needed to be completely immersed in liquids. In the Mk III, the control mechanism of the surfaces was confined to an upper chamber which was sealed by Teflon bellows from the lower chamber in which the surfaces and bathing solutions were kept. Thus, the ‘distance control chamber’ was less likely to degrade due to materials used in various experiments. The control chamber held a complex translation assembly that was very stable and linear. Also, the range of the distance control was extended to cover the ångstrom to the millimeter range using four levels of control (see table 1). As with all SFAs, the distance between the surfaces was measured using MBI.

As the SFA became increasingly used for measuring friction, a new attachment was developed for the Mk III which was called the bimorph slider [9]. This attachment will be described in more detail below in reference to the SFA 2000.

1.2.4. Other versions of the SFA. The SFA technique was also extended by other research groups. A significant advance was the introduction of the ‘SFA Mark PI’ for measuring very weak, long-ranged interactions (\(F/R\)) down to \(\mu N m^{-1}\)) between charged surfaces employing a magnetic drive mechanism [13]. In this apparatus, one of the surfaces is driven by two nanomover microstepping motors linked by a differential spring mechanism and the other surface is suspended by a weak cantilever spring connected to a permanent magnet which is driven by passing a current through an external coil.

Parker et al [10] developed an apparatus based on the Mk I with a cylindrically shaped chamber and a single seal on top which was later called the Mk IV. The goal of this device was to perform as well as the Mk I but at the same time be simpler to produce, clean and use. The main changes compared with the Mk I are the use of a flexure hinge device as the differential spring instead of a leaf spring and the use of a diaphragm to seal off the mechanical mechanism instead of O-rings. Klein [11, 12] developed a version that was somewhat simpler than the Mk II at the expense of control of the surfaces. Klein was more concerned about the difficulties in removing adsorbed materials from the surfaces of the liquid-containing chamber and tried to focus on minimizing the number of components that would come into contact with the experimental bathing...
solutions. His solution chamber was made of glass which could easily be cleaned overnight in sulphachromic acid.

Tonck and co-workers [14] developed an apparatus where they are able to simultaneously measure the forces and rheological properties of liquid films between opaque surfaces. They used one capacitance displacement transducer, C1, to measure the displacement between a flat surface and the base of the force-measuring cantilever spring and one, C2, to measure the displacement between the flat surface and the spherical surface at the end of the cantilever spring. Below separations of about 5 nm the roughness of their alumina surfaces began to affect the measured forces between the surfaces.

Sonntag and co-workers [15] developed an apparatus similar to Derjaguin’s [2] where they measured the forces between two crossed quartz filaments. In their apparatus, one filament was attached to a micrometer screw and a piezoelectric column with movement control of about 1 nm. The other filament was attached to a transducer that was used to measure the forces between the two filaments.

Heuberger and co-workers [16, 17] have developed what they call the ‘extended surface forces apparatus’ (eSFA) which takes advantage of fast spectral correlation interferometry for a fast and precise analysis of the interference spectra that give the distance between and shapes of the surfaces, as well as the refractive index of the material between them (or deposited on them). Their method also includes remote control of the SFA.

1.3. Some limitations of the existing SFAs

While the Mk III is an extremely stable and functional apparatus, there are a few drawbacks which are addressed in the design of the SFA 2000. These drawbacks and their causes are listed below.

(1) The two parts which make up the main translation stage are very difficult to machine and have very little tolerance. The stringent tolerance is important for maintaining the perfectly vertical and linear motion of the lower surface.

(2) There are many parts in the Mk III due to the design of the sealed translation stage. This makes it more difficult to fully assemble the apparatus to perform experiments.

(3) The Mk III has very little room for adding new attachments. While the small volume of the chamber is handy for experiments where the apparatus must be filled with a liquid, it does not allow for the easy addition of extra parts.

The SFA 2000 was designed to address these drawbacks and will be described below.

2. SFA 2000

The concepts behind the operation of the SFA 2000 are similar to the Mk II and III, but the 2000 apparatus is designed to have fewer parts and to be easier to produce, assemble, clean and operate, as well as to be able to accept numerous attachments while still performing as well as the previous versions. Although the SFA 2000 is described here for the first time, it has already been utilized for studying various types of tribological (including both dry and lubricated) and rheological systems [18] and materials such as nanoparticles [19–21], polymers [22], hydrocarbons [23, 24], polysaccharides [25], biological materials [26, 27], self-assembled monolayers [28], dye molecules [29, 30] and metal thin films [31].

2.1. The basic unit for normal and adhesion force measurements

The main improvement of the SFA 2000 is the use of one central single-cantilever spring which is used for generating both coarse and fine motions over a total range of seven orders of magnitude (from millimeters to ångstroms). A schematic of the basic SFA 2000 is shown in figure 1. The main components are the micrometers, the main stage containing the central single-cantilever spring, the lower disk holder and the upper disk holder.

There are four main controls for the distance between the surfaces. The controls and their specifications are listed in table 1. Three move the lower surface and one moves the upper surface. The coarse and medium controls are manipulated by hand using the differential micrometer. The fine control is manipulated by using a motor which drives a fine micrometer which then acts against a helical ‘coil’ spring. The extra-fine control is manipulated by changing the voltage across the inner and outer walls of a piezoelectric tube that supports the upper surface. More specific details of the ways the surfaces move in response to the distance controls is covered in the following text.

The stainless steel main stage, shown in figures 1 and 3, contains a single-cantilever spring that allows for two kinds of pivoting motions about the point P (figure 3). When a normal force acts on the end of the spring (pivot point B) using the coarse and medium control differential micrometer, the spring deflects in the ‘bending mode’: the spring bends at point P (pivot point A) and the lower surface moves by approximately the same distance upward that the micrometer shaft moves downward. More specifically it will move by \( L \tan \theta \approx L \theta \), where \( L \approx 4 \) cm is the horizontal distance between pivot points A and B, which is the same as the distance between pivot point A and the surfaces.

<table>
<thead>
<tr>
<th>Level of control</th>
<th>Type of control</th>
<th>Surface moved</th>
<th>Positional accuracy (Å)</th>
<th>Total range of movement (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coarse</td>
<td>Differential micrometer</td>
<td>Lower</td>
<td>2000</td>
<td>2000 (2 mm)</td>
</tr>
<tr>
<td>Medium</td>
<td>Differential micrometer</td>
<td>Lower</td>
<td>500</td>
<td>200</td>
</tr>
<tr>
<td>Fine</td>
<td>Differential spring mechanism</td>
<td>Lower</td>
<td>2</td>
<td>10</td>
</tr>
<tr>
<td>Extra fine</td>
<td>Piezoelectric tube</td>
<td>Upper</td>
<td>&lt;1</td>
<td>1</td>
</tr>
</tbody>
</table>

Table 1. SFA 2000 distance controls and their specifications.
Figure 1. (a) The SFA 2000 for measuring forces between two molecularly smooth surfaces. The figure shows a section through the center of the apparatus. Four different distance control mechanisms are shown and listed in table 1: coarse micrometer, medium micrometer, fine micrometer and extra-fine (piezoelectric tube) control. A new ‘universal disk mount’ has been designed for use with both the upper and lower disks. These new disk supports can accommodate both cylindrical disks and a new type of rectangular dove-tailed disk, shown here. The dove-tail disks can be mounted in the SFA 2000 with the main chamber filled with solution, making them ideal for the transfer of bilayer- or protein-coated surfaces into the apparatus without exposure to air. Teflon O-rings outside the main chamber are used to seal both the upper clamp and the front plate (not shown) when the screws for these components are tightened into place. The pivoting mechanism of the single-cantilever spring colored in light green is described in figure 3. (b) Top view of the main stage and lower disk holder. Removal of the motor, fine micrometer, coil spring and anti-backlash spring and screws I, II and III (which bolt the main stage to the upper chamber wall) allows for the main stage and attachment base (green) to be rotated anti clockwise about screw IV for easy access to the lower surface (dove-tailed disk holder). A photograph of the apparatus is shown in figure 2.

In contrast, the motor-controlled ‘fine control’ micrometer compresses a helical spring which presses against the top of the main stage. The far right part of the main stage remains in contact with the ‘coarse control’ micrometer while the cantilever spring buckles by a very small amount, $\Delta D$, as shown in the figure. The tips of the fine micrometer and differential micrometer are precisely positioned in the horizontal center line that also passes through the single cantilever (the pivot point) and the surfaces, so that while the attachment does rotate, its motion at the surfaces is (almost)
Figure 2. (a) Photograph of SFA 2000 with optics stand, closed and ready for experiments. The mirror, shown in the lower right, is normally placed below the entrance window for the light, as shown in (b). (b) Schematic of SFA setup showing the light path in a typical experiment (here shown with the bimorph slider attachment—section 2.2). The optical (FECO) fringes used to visualize the surfaces during experiments are described in appendix A.

Figure 3. Schematic showing the main stage and the single-cantilever spring. When a force is applied by the coarse control micrometer the pivot point (P) is at the center of the cantilever which is in the bending mode. The surface moves by $L\theta$ where $L$ is the distance between A and the surfaces. When the force is applied by the fine control, the pivot point is at point B and the cantilever is in the buckling mode. The surfaces move by $\sim2\Delta D$ which is typically much smaller than $L\theta$. 
perfectly vertical, thereby ensuring that the surfaces move normally to each other. Before each experiment the height of the spring mount is adjusted so that the lower disk is parallel to the upper disk at contact. The displacement, $\Delta D$, is determined by the ratio of the stiffness of the helical spring to the spring constant of the cantilever spring and results in a displacement of the surfaces equal to $\sim 2\Delta D$. This ratio of spring stiffness is chosen so that the fine control is 1000 times more sensitive than the coarse control. The combination of the bending and buckling modes allows for a controlled range of motion from ångstroms to millimeters. Strictly speaking the lower surface actually tilts a little when moving up in the bending mode, but this tilt can be minimized by positioning the surfaces to be parallel at contact.

The lower surface is connected to the attachments base under the main stage via a double-cantilever ‘force spring’. A double-cantilever spring is used to prevent the surface from twisting when a force is applied. The choice of the spring constant depends on the system being measured and the type of measurements desired. The larger the forces are expected to be, the stiffer the spring should be. For example, in a system where there are strong repulsive forces present, it would take a high force to bring the surfaces together. Since $F = k \Delta D$, the higher the spring constant, the smaller the $\Delta D$ needed to apply the necessary force.

Normal forces between the surfaces are measured by moving the surfaces at the base of the double-cantilever ‘force springs’ by a distance $\Delta D_{\text{applied}}$ using the differential micrometer, motor-driven fine micrometer and/or piezo tube. The actual distance that the surfaces move relative to each other, $\Delta D_{\text{meas}}$, is measured by MBI. The changed force $\Delta F$ between the surfaces, when they come to rest at a separation $D$, is therefore

$$\Delta F(D) = k(\Delta D_{\text{applied}} - \Delta D_{\text{meas}}),$$

where $k$ is the spring constant.

When $\partial F(D)/\partial D > k$, there is a mechanical instability and the lower surface will jump either toward or away from the upper surface to the next stable region during approach or separation, respectively. Thus, one can see that, while a weaker spring is more sensitive, it may also result in more inaccessible regions of a complex force profile because of the increased range between instabilities. However, the spring stiffness can be changed during experiments (see section 2.7) to build the full force profile.

Adhesion forces are measured from the force that it takes to separate surfaces from adhesive contact by measuring the distance that they jump apart from contact, $\Delta D_{\text{jump}}$ (figure 4), then using the following equation:

$$F_{\text{adhesion}} = k \times \Delta D_{\text{jump}}.$$  

The spring constant, $k$, is measured (calibrated) before or after the experiment by placing small weights of mass, $m$, on the lower disk holder and measuring the displacement, $\Delta d$, of the spring using a graduated traveling microscope. The graduated traveling microscope is not part of the apparatus, but a separate microscope that is located outside the apparatus, for example, just outside the front window (figure 2). The spring constant is then given by $k = mg/d$ (N m$^{-1}$), where ‘$g$’ is the acceleration of gravity. The spring constant of the force-measuring spring, $k$, typically varies from 30 to $5 \times 10^5$ N m$^{-1}$. The maximum stiffness that can be attained, by fully clamping the spring or using a rigid support, is approximately $1.5 \times 10^5$ N m$^{-1}$.

The measured force between the curved cylindrically curved surfaces, normalized by their radius, $F_{\text{curved}}(D)/R$, is directly related to the interaction energy per unit area between two flat surfaces, $W_{\text{flat}}$, by the Derjaguin approximation [32].

$$W_{\text{flat}}(D) = \frac{F_{\text{curved}}(D)}{2\pi R} = \frac{F_{\text{curved}}(D)}{2\pi R},$$

when $R_1 = R_2 = R$.

2.2. Measuring shear (friction and lubrication) forces

The surfaces can be sheared past each other by using a motor-driven micrometer to move the upper surface in the lateral direction, or by using a piezoelectric bimorph slider, as shown in figure 5. The motorized friction device has a reversible dc motor, $M$, that can be switched on or off or driven in reverse at different constant or variable speeds using a dc power supply or function generator. At point $M$ in figure 5(a) the motor presses against two parallel cantilever springs (double-cantilever spring configuration) which drives the friction device including the upper disk smoothly into or out of the paper with respect to the lower disk. When using a function generator, a square wave voltage function must be used to shear the upper surfaces back and forth at a constant velocity, $\pm V$. When using the piezoelectric bimorph slider,
the bimorph plates bend in proportion to the voltage applied to them. Any thermal drift of the piezo-material of the bimorph is minimized in the same way as for the semi-conductive strain gauges by utilizing temperature-controlled experimental rooms and by the thermally well-sealed thick steel chamber walls all around the SFA 2000 (see below). Thus as a constantly increasing voltage is applied, the bimorph bends at a constant rate. To shear the surfaces back and forth at a constant velocity, a triangular wave function must be applied. The bimorphs are designed with a flexure point in the middle which causes the surfaces to displace linearly as voltages are applied.

When using the friction device or bimorph slider, two vertical double-cantilever springs with four semiconductor or resistance strain gauges are attached symmetrically to oppositely bending arms of the springs thus forming the four arms of a Wheatstone bridge strain gauge system. The resistance (foil) strain gauges were from Vishay Micro-Measurements (typically 350 Ω gauges such as N2A-13-T028K-350); the semiconductor gauges were custom made temperature-compensated gauges from Micron Instruments, Simi Valley, California (typically 350–500 Ω gauges, temperature compensation: 10–60 °C). All semiconductor gauges have temperature-compensated resistors built into the circuit and adjusted/tested before being shipped. The experimental rooms are also temperature-controlled to ±0.1–0.2 °C in addition to the SFA 2000 being thermally well-sealed by 12.5 mm thick steel walls all around. When a lateral force is applied to the upper surface the strain gauges are used to measure the deflection with a signal conditioning amplifier (Vishay Measurements, 2300), which outputs the signal to either a computer data acquisition system or a chart recorder.

The friction device springs can be calibrated at the end of the experiment by hanging small weights from both sides of the device. The voltage signal is then recorded and calibrated against the weight. The motions of the upper surface need to be calibrated against the voltage applied to the motor or the bimorphs as described in figure 5. When used in situ during experiments, the laser beam of the Keyence position detector is reflected off a small reflective silicon wafer piece attached to the end of the bimorph slider. When a triangular function
Figure 6. 3D/XYZ translation stages and sensors. A bimorph slider with an additional piezoelectric stack links parts for simultaneous X and Y translations of the lower surface in the horizontal plane. Translation in the vertical, Z-direction, is generated by the piezoelectric tube supporting the upper surface. The upper surface is suspended from four vertical wire springs and four horizontal cantilever springs, each mounted in a square/cross configuration, each with one or two strain gauges on them, allowing for deflections and hence the forces experienced by the upper surface to be measured in the X, Y and Z directions.

is applied to the bimorph slider, the displacement is measured. To calibrate the displacement of the upper motor, the friction device is held vertically \textit{ex situ}, and a disk with a reflective piece attached to it is suspended from the upper disk mount spring. The displacement is then measured with the Keyence position detector as a function of the voltage applied to the motor.

The bimorph slider (figure 5(b)) operates in a similar fashion to previous bimorph sliders as described in [9], but has a longer range compared with previous ones due to the greater space of the SFA 2000, allowing for much longer bimorphs. Thus, for active bimorph of 50–75 mm, the lateral movement of the surfaces can be varied from about 5 to 10 $\mu$m V$^{-1}$. However, since bimorphs become increasingly non-linear at voltages exceeding 40 V (when deviations from linearity begin to exceed 1%), the calibration of the bimorphs is recommended if the applied displacements are large and need to be known to high accuracy. Bimorphs can be also used in liquids or ‘under water’ after suitably coating their surfaces and all the wiring connections with a suitable Humiseal\textsuperscript{TM} or Polydimethylsiloxane (PDMS) coating. This usually increases their stiffness by 10–15% and reduces their sensitivity to movement by a similar amount. The coating (PDMS, SYLGARD\textsuperscript{®} 184 Silicone elastomer curing agent) acts as an electrical insulator as well as a protective layer preventing exposure of the bimorph slider to potential harmful chemicals. Since PDMS is resistant to a wide range of liquids, including electrolytes, oils and certain solvents, the coated friction slider extends the capabilities of SFA 2000 to measure friction forces under liquids. In addition, since PDMS has relatively low modulus, the maximum range of movement of the bimorph is not compromised.

2.3. Measuring forces in three orthogonal directions (3D or XYZ attachments)

A variety of XYZ translation stages (scanners) and detectors (scanners) shown in figures 6 and 7 allow for generating relative movement of surfaces, and independently measuring the resulting forces, in three orthogonal directions: X, Y and Z. Measurement of adhesion, friction and molecular ordering in three directions is made possible by moving one of the surfaces in some arbitrary direction in the X-Y plane and/or the upper surface in the $\pm$Z-direction. Thus, circular or some other type of non-linear motion, i.e. other than back-and-forth motion, can be induced. Likewise, a force response on the upper surface along any spatial direction (not necessarily in the direction of the applied motion) can be simultaneously measured.

In figure 6, motion in the Z-direction of the upper surface is controlled by applying voltages across the inner and outer walls of the piezoelectric tube, with a linear range of $\sim 1 \mu$m. The lower surface is supported by a double-cantilever spring used for measuring the normal forces between the surfaces, and the spring is connected to an X-Y translation stage composed of a bimorph slider and a piezo stack. The expansion and contraction of the piezo stack under a certain voltage make the
Figure 7. Single unit allowing for 3D translation and sensing. The upper surface is moved in the horizontal (X–Y) plane by two piezo stacks whose motion is amplified mechanically 10-fold by the vertical levers and frictionless cross-spring flexure pivots. Motion in the Z-directions and force sensing is as in figure 6. Mechanical amplification of the piezo-stack motion allows for large displacements using low voltages (50–100 V), where similar displacements of unamplified stacks require 500–1000 V which are invariably accompanied by large hysteresis. There are two places where four vertical wire springs (black and orange) act as the four legs of a square table, allowing for motion in any direction in the horizontal (X–Y) plane. The top springs (two shown in black) move the blue movable stage relative to the gray round base. The movable stage is further connected to two green driving levers (one in the X-direction and one in Y-direction) via horizontal wire springs (also in black). The pink leaf springs (see top left inset) attached to the horizontal wire springs are designed to minimize elastic restoring forces and cross-talk when the blue and green parts are displaced relative to each other, for example, into or out of the paper in the top left inset. The four vertical lower springs (orange) are similarly configured, as described in figure 6.

lower surface travel in the X-direction which is orthogonal to the bending direction of the bimorph slider in the Y-direction, with a maximum linear travel of at least 100 µm (0.1 mm). Non-linear motion in some arbitrary direction in the X-Y plane, e.g. circular or elliptical, can thus be accomplished by simultaneously applying signals at different voltages and phases to the bimorph slider and the piezo stack.

A 3D force sensor, supporting the upper surface in figure 6, can measure the force in any spatial direction. This 3D force sensor has been used successfully in various studies of thin film adhesion, friction and lubrication [33, 34].

Figure 7 describes a single, compact 3D force-measuring unit that is particularly suitable for measurements in liquids, especially aqueous salt solutions, where contact of bimorphs or piezoelectric elements with such solutions is undesirable. However, it is possible to coat bimorph strips with a soft, chemically inert film of Humiseal™ or PDMS that prevents chemical degradation and loss of electrical insulation for at least 24 h in 1M NaCl solution (see section 2.2).

The XYZ translation stage of figure 7 can also be used as an atomic force microscopy (AFM) scanner, which is described in section 2.5 and figure 9.

2.4. High-speed attachment

The rotating high-speed disk attachment (figure 8) provides a much longer range and higher sliding speeds than previous
friction devices. When using this attachment, the lower flat surface is a transparent or opaque disk connected to a miniature dc motor (A2520, Maxon precision motors, Inc., MA), allowing for sliding velocities up to around 15 m s\(^{-1}\), i.e. about 6 orders of magnitude faster than current piezoelectric bimorph sliders. This attachment requires a sophisticated position and adjustment system for the rotating disk consisting of numerous adjustable springs and weights, shown in figure 8(b). When using an opaque surface as the bottom surface, a modification of the optical arrangement is required for observing the fringes of equal chromatic order (FECO) in reflected light rather than in transmitted light [35]. Using this method, reflection FECO appears as dark bands on a bright background as the result of destructive interference. The multi-matrix method (MMM) enables conversion of these wavelengths to separation distances [36].

2.5. 3D displacement and force sensing probe attachment

A miniature 3D displacement and force sensing attachment (figure 9) has been designed to measure forces in any spatial direction [33] suitable for both SFA and AFM measurements in the same experiment. Both the scanner and force sensor have been designed so as to minimize unwanted non-linear contributions of rotation, tilting and shear when a probe (tip) is pressed or scanned across the opposing surface, and the sensor springs deflect. This attachment therefore offers the ability to scan and measure the forces on a probe tip in three orthogonal directions, linearly and simultaneously [33]. The sensitivity of the cantilever foil springs (strain gauges) can be readily varied by \textit{in situ} adjustment of the tensions on the foils. Our mesoscale prototype has been optimized for equal sensitivities in all three directions, and can measure distance deflections as low as 5 nm. The spring constant of this prototype usually is between 100 and 1000 N m\(^{-1}\). A finite element modeling and linear beam theory show that a micro-scale device should function with similar characteristics to current AFM probes [33]. In principle, it should be feasible to scale the device from a macroscale tribometer/indenter to a (mesoscale) SFA to a nanoscale AFM.

The choice of using a resistive method for sensing/detecting force and/or displacement makes this device less sensitive than the beam-bouncing method, but it does make it possible to measure forces and displacement in 3D and also to build compact arrays of independent sensors, thus giving no limit to the number of sensors that can be used simultaneously, something that cannot be currently achieved with the beam-bouncing method.

2.6. Bimorph vibrator attachment

Figure 10 shows the bimorph vibrator attachment. It allows for the top surface to be vibrated vertically using the piezo tube while measuring—for example, with a lock-in amplifier—the
amplitude and phase of the vibrations induced in the lower surface. This attachment is useful for measuring rheological and visco-elastic properties of fluids and thin fluid films near or between two surfaces. The motions of the bimorph vibrator are similar to those of the bimorph slider (figure 5) but its oscillatory motions are normal rather than lateral. This means that the liquid film experiences squeeze flow rather than shear flow, so that the viscous forces are much larger and the response much more sensitive than in the case of the slider, i.e. the viscosities of thick, including effectively bulk, liquid films can be measured, which are not easily accessible in lateral sliding. For experiments with the bimorph totally immersed in a liquid
such as water, it is possible to chemically and electrically seal the bimorph surfaces and its attached wires with a suitable coating (see section 2.2). The bimorph vibrator has provided some important results on the viscosity of thin fluid films and the location of slipping planes [37–39].

2.7. Variable normal force-measuring springs

Often when measuring forces in the SFA, the stiffness of the force-measuring cantilever spring used in the experiment has to be chosen carefully to suit the specific system being investigated. Springs having a high spring constant are typically best suited for measuring large and long-range forces while a low spring constant is often necessary to resolve weaker and more subtle forces. Because many systems investigated with the SFA involve a combination of strong and weak forces [20, 40, 41], it is often difficult to fully characterize all the forces in a system using a single, fixed stiffness spring. Traditionally, changing the spring stiffness was an involved process that entailed stopping the experiment, disassembling the apparatus, reassembling the apparatus with a new set of cantilever springs and repeating the experiment from the beginning. For this reason, a variable stiffness spring, shown schematically in figure 11, was developed which allows the experimenter to controllably alter the stiffness of the force-measuring spring in situ without halting the experiment.

The variable stiffness spring attachment works on the simple principle that the stiffness $k$ of an end-clamped cantilever varies with its active length $L$ as $k \propto L^{-3}$. Consequently, decreasing the $L$ by a factor of 10 will lead to a 1000-fold increase in the stiffness, allowing the experimenter to drastically alter the spring stiffness by simply adjusting the active spring length using a variable clamping mechanism.

In the variable stiffness spring attachment, the lower surface is held at the end of a double-cantilever spring which in turn is held between a spring-loaded ‘adjustable sliding clamp’. The adjustable sliding clamp is supported by a dove-
tail translational stage which moves along a central screw that is connected to an external wheel via a gear mechanism. Turning the external wheel allows the sliding clamp to be moved forward or backward, decreasing or increasing the cantilever spring length and stiffness. Once the desired cantilever spring length is achieved, the wheel is retracted which disengages the gear mechanism allowing forces to be measured.

2.8. Constant-force (balance) attachment

The new balance attachment (figure 12) can be used to measure very weak forces which may take a long time to reach equilibrium, as well as drive surfaces together at constant force.

The idea behind this attachment grew out of the realization that, since it is practically impossible to totally prevent thermal drifts, one must devise a method that can still reliably measure surface forces even in the presence of drifts. It was also appreciated that the applied force could not be produced via the displacement or deflection of a spring element. Figure 12 shows a schematic drawing of a ‘constant-force balance’ that in principle meets all the operational requirements of a constant-force transducer, near zero stiffness, frictionless motion in one direction only with robustness in all other directions, no vibrations, insensitivity to thermal drift and very high force sensitivity. The principle of this balance is as follows. First, imagine that the balance arm is pivoted by a frictionless knife-edge pivot. The center of mass of the whole balance in liquid is then adjusted with small screws so that it is located at the pivot point. The balance therefore has little or no restoring force when displaced by a small amount (i.e. $K = 0$), and in principle it should remain at whatever angle it is left at. If the left arm supports one of the two surfaces and the right arm has a magnet attached to it, then by applying a magnetic field on the magnet a force will be felt on the other side, trying to displace the surface up or down. If, over the distance $\Delta D$ the magnetic field gradient remains uniform (which is easily achieved over the micrometer to millimeter distances of interest in surface force studies), then it is clear that any thermal drifts of the pivoting point relative to the upper or lower surfaces will have no effect on the applied force on the lower surface.

In practice, it is not possible to construct a frictionless knife-edge pivot. The closest one can approach a totally frictionless pivot is to use a combination of metal leaf springs that provide frictionless rotation about some axis. The ‘cross-spring flexure pivot’ is one such design. This type of miniature pivot was successfully used in an early friction attachment to the SFA [42] and has been used again in the 3D scanner of figure 9. But this type of pivot produces a small restoring force when it rotates, that is it has a small but finite elastic stiffness or torque. However, by designing the balance so that the center of mass is located above the pivot (cf figure 12) this introduces a new (opposing) torque into the rotation due to gravitational or buoyancy forces. This opposing torque can be made to offset exactly the torque of the flexure pivot.

Put in mathematical terms, referring to figure 12, if $m$ is the (displaced) weight of the balance whose center of mass is at a distance $h$ above the pivot point, then when the balance rotates through an angle $\theta$ there will be a ‘gravitational’
torque of magnitude $mgh \sin \theta \approx mgh\theta$ acting to rotate the balance further. On the other hand, the flexure pivot itself will have developed a restoring torque of magnitude $\tau \theta$ acting in opposition to the gravitational torque. Thus, if $mgh = \tau$ the two effects cancel out, and the balance will behave as a frictionless zero-stiffness balance. This condition can be expressed as $h = \tau/mg$.

The prototype balance (figure 12(b)) is made of aluminum (anodized) and weighs about $m \approx 20$ gm. The pivot is a commercial flexure pivot (Bendix Corp., part number 5006-600) of torque constant $\tau = 10^{-4}$ N m$^{-1}$, and is mounted at a height $h = \tau/mg \approx 5$ mm below the center of mass of the balance. Small horizontal and vertical adjusting screws, which can be manipulated from outside the SFA chamber, are used to obtain an overall restoring torque of zero. Our preliminary tests indicate that, when suspended in liquid, this system is highly robust and vibration free, and can be adjusted to have an effective stiffness as low as $K = 10^{-3}$ N m$^{-1}$. This is 4 orders of magnitude lower than the current lowest spring stiffness of $\sim 10$ N m$^{-1}$, allowing for forces to be measured in the sub-pico-newton regime over short time periods, and in the 0.1 N regime over longer times even in the presence of large thermal drifts.

3. Concluding remarks

In this paper we have reviewed the history behind the SFA including the changes involved in each generation of the apparatus and related apparatuses, and the reasons for these developments. The SFA technique can now be used to measure both normal and lateral forces between surfaces in liquids with a distance resolution of less than 1 Å. Recently introduced attachments, many of which were described here, allow for dynamic (non-equilibrium) measurements to be made in situ, and for extending the range and scope of surface force measurements to new surfaces and systems, including electrochemical and biological systems. In addition, incorporation of other techniques, such as x-ray scattering [43], IR spectroscopy, fluorescence microscopy [44] and AFM [33], allows for different measurements to be made on a sample at the same time.

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Appendix A. Multiple beam interferometry

MBI is used in the SFA to determine the distance between the surfaces as well as the shape of the surface [42, 45]. For a typical SFA experiment a pair of transparent surfaces (e.g. freshly cleaved mica) is used as the surface substrate, which are coated on the back with a highly reflective layer (e.g. silver) for providing a good interfering pattern between the reflecting surfaces. The surfaces are glued onto a cylindrical shaped glass disk and mounted in a cross-cylindrical configuration, allowing for different measurements to be made on a sample at the same time.

which simulates a sphere-on-flat geometry. When white light is directed normal to the surfaces the light is reflected back and forth between the silver layers and the transmitted light near the closest contact point between the surfaces creates Newton’s rings, as can be seen through a microscope objective. The transmitted light corresponds to a particular set of discrete wavelengths that is made visible by a spectrometer and are the so-called fringes of equal chromatic order (FECO). An example of the FECO between mica surfaces in air is shown in figure 13. In this appendix we will only consider this setup with three-layer (e.g. mica-medium-mica) interferometry, which is the most commonly used setup for SFA experiments. Derivation of the distance measurements for other cases can be found in [42].

When the surfaces are in contact with no medium between them, the interference fringes depend on the mica thickness. Assuming that the mica thickness, $T$, is the same for both surfaces, $T$, can be determined by the wavelength of the $n$th order fringe, $\lambda_n^0$, by the relation $T = n\lambda_n^0/4\mu_{\text{mica}}$, where $\mu_{\text{mica}}$ is the refractive index of mica at $\lambda_n^0$. The fringe order, $n$, is determined by

$$n = \frac{\lambda_n^0}{\lambda_{n-1}^0 - \lambda_n^0},$$

(A.1)

where $\lambda_{n-1}^0$ is the next fringe at higher wavelength. It can be shown that when the surfaces are separated by a distance $D$, the amount that the $n$th fringe shifts by depends on the refractive index of the medium in the gap between the surfaces, and the

\[\text{Figure 13. FECO of mica surfaces in air (a) in adhesive contact (} D = 0\text{) and (b) separated by } D \approx 9.9 \text{ nm. Note that the odd order fringes appear narrower than the even order fringes. In (a), the contacting parts of the surfaces flatten due to elastic deformations of the mica and glue. Each fringe appears as a doublet with a } y\text{- and a } x\text{-component due to the birefringence of mica. The vertical lines at the edges of the pictures are the green and yellow Hg calibration lines.}\]
original contact positions (because these are related to \( n \) and the interferometer thickness) \([42, 46]\).

\[
\tan(2\pi \mu D / \lambda_D^{\mu}) = \frac{2\mu \sin[\pi(1 - \lambda_D^{\mu} / \lambda_D^{\mu})/\lambda_D^{\mu}] / (1 + \mu^2) \cos[\pi(1 - \lambda_D^{\mu} / \lambda_D^{\mu})/\lambda_D^{\mu}] / (1 - \lambda_D^{\mu} / \lambda_D^{\mu}) \pm (\mu^2 - 1)}. 
\]

(A.2)

The sign is taken when \( n \) is odd (standing wave with node in the center) and \(-\) is taken when \( n \) is even (standing wave with anti-node in the center). At the separation distance \( D \), the \( n \)th fringe changes wavelength from \( \lambda_n^{\mu} \) to \( \lambda_{n+1}^{\mu} \). The effective refractive index is denoted as \( \bar{\mu} = \mu_{\text{mica}} / \mu \) where \( \mu_{\text{mica}} \) is the refractive index of mica and \( \mu \) is the refractive index of the medium between the two surfaces at \( \lambda_D^{\mu} \). If the positions of three consecutive fringes are known, then an iterative process can be used to determine the thickness and refractive index of the film between the surfaces. When the surfaces are separated by a distance which results in a fringe shift equal to \( \lambda_n^{\mu} \), it is easy to show that the film thickness is calculated by

\[
D = \frac{\lambda^{\mu}_{n+1} - \lambda^{\mu}_n}{2\mu}. 
\]

When the distance between the surfaces is small \((D \lessgtr 30 \text{nm})\), we can use Taylor series expansions to find approximate expressions for the trigonometric functions in equation (A.2):

\[
D = \begin{cases} 
  n F_{\text{m}} (\lambda_D^{\mu} - \lambda_D^{\mu}) / (2\mu_{\text{mica}}), & \text{for odd } n, \\
  n F_{\text{m}} (\lambda_D^{\mu} - \lambda_D^{\mu}) / (2\mu^{2}), & \text{for even } n,
\end{cases}
\]

(A.3)

where \( F_{\text{m}} \) is a correction factor which takes into account the refractive index dispersion and phase change of the reflection at the silvered surfaces. For light with wavelength \( \lambda \gtrsim 500 \text{ nm} \), the correction factor is \( F_{\text{m}} \approx 1.024 + 1/n \). Note that the film thickness determined from the even order fringes depends on \( \mu \), the refractive index of the medium between the mica surfaces, but that the film thickness determined from the odd order fringes does not. Therefore, if we know the wavelength shift of two consecutive fringes we can determine an approximate value for the refractive index of the medium:

\[
\mu = \mu_{\text{mica}} \sqrt{\frac{(n - 1) F_{\text{m}}}{\lambda_D^{\mu} - \lambda_D^{\mu}}}, \quad \text{for odd } n.
\]

(A.4)

Equation (A.3) can also be used to explain why odd fringes look different than even fringes for small \( D \) as shown in figure 13. For the odd fringes the shift in the wavelength is proportional to \( 2\mu_{\text{mica}} / n F_{\text{m}} \) while the shift in wavelength for the even fringes is proportional to \( 2\mu^2 / n F_{\text{m}} \mu_{\text{mica}} \). Thus if \( \mu < \mu_{\text{mica}} \), the shift in the wavelength of the odd fringe will be greater than the shift in the even fringe. This case is most pronounced for mica surfaces in contact in air \((\mu = 1)\). Comparing the difference in wavelength \( \Delta \lambda \) between light interfering in the contact region and light interfering at a spot just outside the contact region, we see that \( \Delta \lambda_{\text{odd}} > \Delta \lambda_{\text{even}} \). Therefore, the edges of the odd fringes appear sharp while the edges of the even fringes appear rounded as seen in figure 13(a). If \( \mu = \mu_{\text{mica}} \), the even and odd fringes will have the same shape. Finally, if \( \mu > \mu_{\text{mica}} \), the shift in the wavelength of the even fringe will be greater than that of the odd.

As shown in figure 13, the FEOC appear as doublets when the surfaces are mica. Mica is a birefringent material which means that it has two indices of refraction in the plane perpendicular to the incident light (mica also has a third optical axis, \( \alpha \)-component, which is parallel to the incident light and thus not observed in SFA experiments). The optical path with the lower refractive index results in the lower wavelength fringe, the \( \beta \)-component, and the higher refractive index results in the higher wavelength \( \gamma \)-component. Typical values for the refractive index of reddish or brownish mica are

\[
\mu_{\beta} = 1.5794 + 4.76 \times 10^{4}/\lambda^2 (\text{Å}^2),
\]

\[
\mu_{\gamma} = 1.5846 + 4.76 \times 10^{4}/\lambda^2 (\text{Å}^2).
\]

(A.5)

When the optical axes of the two mica surfaces are perfectly aligned, the shift between the \( \beta \)- and \( \gamma \)-components of the fringe will be maximized. Conversely, when the optical axes are at right angles to each other, there will be no shift between the \( \beta \)- and \( \gamma \)-components of the fringes. The mica acts as a polarizer for the light going through the surfaces, and if great care is taken to align the surfaces in a known orientation, it is possible to gain insight into the molecular alignment between the surfaces.

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