A new capacitive sensor for displacement measurement in a surface force apparatus

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Abstract. We present a new capacitive sensor for displacement measurement in a Surface Forces Apparatus (SFA) which allows dynamical measurements in the range of 0 – 100 Hz. This sensor measures the relative displacement between two macroscopic opaque surfaces over periods of time ranging from milliseconds to in principle an indefinite period, at a very low price and down to atomic resolution. It consists of a plane capacitor, a high frequency oscillator, and a high sensitivity frequency to voltage conversion. We use this sensor to study the nanorheological properties of dodecane confined between glass surfaces.

PACS numbers: 07.05.Fb,07.10.Cm,68.35.Gy,68.45.Gd,83.85.-c,83.85.Jn

Keywords : surface forces apparatus, capacitive sensor, nanorheology

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1. Introduction

The surface-force apparatus developed by Tabor and Winterton (1969) and further refined by Israelachvili and Adams (1978), Klein (1983) and Parker et al. (1989,) has proven useful for the measurement of colloidal forces between atomically smooth transparent surfaces in liquid and gases at molecular scale. In these classical apparatus the distance between the surfaces is measured by the interferometry of white light fringes (fringes of equal chromatic order, FECO). This technique allows to measure steady or slowly varying distances, with a resolution of a few 0.1 nm. Chan et al (1985) has employed videocameras to record rapidly changing position of the surfaces with a time resolution about 0.5 s, during the drainage of a fluid out of the contact region. Recently, this interferometric method has been improved (Grunewaled (1996)) by using expensive high speed video treatments. The first method proposed for doing a dynamical measurement was to use a piezoelectric bimorph (Van Alsten (1988), Israelachvili (1989), Peachey (1991)), which has also the advantage of allowing opaque surfaces to be used. Although Parker (1992) showed that a bimorph can be used to take a measurement from less than a tenth of a second to several minutes, these devices are unsuitable for measurements that take place over many minutes or hours. Furthermore, the single-cantilever construction of the bimorph implies that a displacement of his head results also in an angular rotation. The resulting shear motion makes it unsuitable for the measurement of adhesive forces. Recently the use of a capacitor dilatometry attachment for the conventional surface-force apparatus has been proposed by Stewart (1992) for statics measurements with a resolution of 0.1 nm. A. Tonck et al. (1988, 1989) described a surface forces apparatus in which they used capacitors to obtain both the distance and the interaction force between a sphere and a plane. This apparatus is suitable for non transparent surfaces and dynamical study of confined liquids.

In this paper, we propose a new method for measuring displacement at the nanometer scale in Surface Force Apparatus, based on a capacitor included in an oscillator. Unlike the capacitive sensor proposed by Steward (2000) or Franz et al. (1996,1997), our method for the capacitance measurement is a low cost method which does not require the use of a lock-in amplifier, without loss in resolution or dynamic performances. From the point of view of surface forces measurements, the method has the advantages of being linear on a large scale, of allowing measurements between non transparent surfaces, and is suitable for dynamical measurements. When used in conjunction with an interferometric technique (Crassous in preparation) for the purpose of calibration, this sensor can be used to perform nanorheological measurements or contact forces measurements between the surfaces.

2. The device
2.1. The surface force apparatus

A schematic diagram of the surface forces apparatus is given in figure 1. This apparatus has several features which distinguish it from the common SFA. First of all, the surfaces are not necessary transparent, since the SFA does not use the FECO technique. The surfaces are usually a sphere and a plane. The plane surface is mounted on the left-hand double-cantilever $L_1$ of stiffness $2950 \text{ Nm}^{-1}$. An optical interferometer measures the deflection of $L_1$ to obtain directly the force measurements. The sphere is mounted on the right-hand double-cantilever $L_2$ and can be moved in the direction normal to the plane. The cantilever $L_2$ prevents the rolling of the surfaces. The sphere motion is controlled by three actuators. The first one is a motorized microscrew driven by a stepping motor. It allows a displacement of 30 nm to 5 cm and is used for a rough positioning of the sphere. The second actuator is a piezoelectric actuator which allows a continuous approach of the two surfaces with a velocity range of 0.1 to 100 nm.s$^{-1}$. The last piezoelectric actuator is designed to add a small sinusoidal motion in to study the dynamic behavior of the sphere-plane interactions. The relative displacement between the sphere and the plane, $h$, is determined by the capacitive sensor described in this article. Finally, in order to calibrate the capacitive sensor, a permanent magnet mounted on the cantilever $L_1$ is located in the magnetic field gradient produced by a little coil of copper wire. This setup allows to calibrate the sensor over a large range of displacement (1 µm).

All these devices are controlled by a Hewlett Packard VXI 743 computer equipped with a E1421A 16 channels A/D and D/A converters.

A more complete description of this apparatus will be given in a forthcoming publication.

2.2. The capacitor sensor

The measurement capacitor consists of two duraluminium discs with a radius $R = 3 \times 10^{-2} \text{ m}$ and a thickness 1 mm. The typical distance between the plates is typically $d = 90 \mu\text{m}$ and the surfaces have been polished to have a roughness smaller than the distance between the two plates of the capacitor. One plate is fixed on the cantilever supporting the plane $L_1$, and the other on the cantilever supporting the sphere $L_2$ so that when the surfaces are brought together, the plates of the capacitor do also. Parallel alignment of the plates is obtained with a mechanical ball-and-socket joint which is rigidly screwed after the plates have been pushed in contact to obtain the parallelism. The terminals of the capacitor plates are connected to the oscillator with thin copper leads whose compliance is much higher than the one of the cantilever. To decrease the viscous drag induced by the air flow between the plates of the capacitor, some holes are drilled in the moving plate. The weight of the capacitor, which is important for the resonant frequency of our surface force apparatus is roughly $m \sim 12 \text{ g}$.

The capacitance $C$ of this sensor is typically 300 pF and its serial resistance about $1 \Omega$. 

In order to measure the capacitance variations of this sensor, we include it in an oscillator. We use a Clapp oscillator containing two fixed capacitor $C_1, C_2$, the variable capacitor $C$, and an inductance $L$ (figure 2). The Clapp oscillator is known to have a good stability and to be easy to build (Audouin et al (1991)). Neglecting the leads capacitances and straight capacitances, the frequency of the oscillations of the Clapp oscillator is:

$$f = \frac{1}{2\pi \sqrt{\frac{1}{L} + \frac{1}{C_1} + \frac{1}{C_2} + \frac{1}{C}}}$$  \hspace{1cm} (1)

This formula can be used to have an estimation of the nominal frequency and of the sensitivity of the sensor. Using the typical values $C = 2.5 \times 10^{-14}/(d + h)$, $C_1 = C_2 = 220\, \text{pF}$ and $L = 2.2\, \mu\text{H}$, we have a typical frequency of 12 MHz and a typical sensitivity of the order of 1 Hz/Å. This shows that in order to have a precision of 0.05 nm on the displacement measurement, we have to read frequency variations of 0.5 Hz. For this purpose, we use a Hewlett Packard HP53132A counter which reaches this precision with an acquisition time of less than 0.1 s. We emphasize that reading one part in $10^7$ is really easy in a frequency measurement but is difficult and expensive in voltage measurements.

2.3. Static performances

2.3.1. Linearity The conventional way to make a distance measurement with this device is first to fix the distance between the capacitor’s plates at a distance comprised between 50µm and 100µm and to calibrate the sensitivity for small displacements around this distance. Indeed we do not use equation (1) to determine the sensitivity of the sensor, since this latter depends slightly on the angular parallelism of the capacitors plates. In order to calibrate the sensor, we use an interferometer, which is mounted on our SFA (Schonenberger (1989)) and allows to perform easy calibrations. The detailed procedure is as follows: the Rhs cantilever $L_2$ is fixed, a force is applied on the cantilever $L_1$ by the mean of the coil/magnet system. The deflection of $L_1$ results in a displacement $x$ of the sensor’s plate, fixed on $L_1$, as well as of the mirror. The interferometer gives access to the absolute value of $x$, and the calibration is done by plotting $f(x)$, the frequency of the oscillator as a function of $x$. In order to reduce the noise (see paragraph 2.3.3), we usually integrate the frequency signal over a time of 1 s. Fig. 3 shows the calibration over a displacement range of 80 nm. One can see that the capacitive sensor is very linear. The typical maximum deviation to linearity over this scale is lower than 1% of the total excursion range. The measured sensitivity is: $7.70\, \text{Hz.Å}^{-1}$. This is closed to the estimated value deduce from equation (1) but take into account all the straight capacitances.

2.3.2. Influence of stray capacitance The capacitance measuring circuit is in fact sensitive to stray capacitance between the upper sensing electrode and ground. Therefore the value $C$ of the capacitance in equation (1) includes not only the sensor
A new capacitive sensor

capacitance, but also the value of stray capacitances, the larger of which is the capacitance of the screen cable connecting the sensor to the circuit. The order of magnitude of those stray capacitances can be estimated by increasing the distance between the capacitor electrodes up to the point where it does not affect anymore the frequency of the oscillator. The overall value of the stray capacitance can be as large as 50\(\text{pF}\).

During the typical time of an experiment in a SFA (typically 30 mn) and with the environment conditions required by the SFA itself (the SFA is located in a separated closed room where nobody enters during an experiment; signal acquisition and experiment control are performed from another room), it turns out that the overall stray capacitance does not change significantly except for smooth drifts which cannot be distinguished from the thermal drift of the measuring circuit itself (see hereafter). Significant change of the stray capacitance occur usually other large period of time (one day) or when a change is made on the sensor (tuning of the distance or orientation of the electrodes, change in the location of the oscillator). Therefore, the sensitivity of the sensor is periodically calibrated with the interferometer, in order to take in account the changes in sensitivity induced by the modification of the value of the stray capacitance.

2.3.3. Noise and drift

Without any displacement imposed on the cantilevers, we can measure the noise and the thermal drift in a typical situation. Those quantities will limit the static performance of our apparatus and the thermal drift must be corrected to obtain an accurate measurement of the relative displacement of the surfaces. Figure 4 shows a typical record of the signal given by the counter converted in displacement. The noise is less 0.1 nm peak to peak. This is due to the mechanical vibrations on the cantilever \(L_1\). With a simple plexiglass cover over the entire instrument and without any temperature control we find a drift rate smaller than 0.01 nm/s. This drift is of the same order as the drift reported in other articles (Schonenberger (1989)).

It is worthwhile to inquire about the electrostatic attractive forces between the charged plates of this plane capacitor. In general the force is given by:

\[
F = -\varepsilon_0 < V^2 > S/d^2
\]  

(2)

where \( < V^2 > \) is the average of the square voltage between the capacitor's plates, \( S \) the plates area and \( \varepsilon_0 \) the dielectric permittivity of vacuum. The force is \( \approx 1.38 \mu\text{N} \) for typical values \( d = 90 \mu\text{m} \) and \( < V > = 3.5 \text{ V} \). This force is nearly constant over one experiment since the relative displacement of the plates is always much smaller than \( d \).

2.4. Dynamical measurements

A piezoelectric crystal is used to add a sinusoidal motion (Tonck (1988)) of small amplitude on cantilever \(L_2\) in order to determine the dynamic behaviour of the sphere-plane interaction. The distance between the surfaces is thus \( h \) with \( h \) being the sum of two components:

\[
h = h_{dc}(t) + h_{ac}\cos(i\omega t)
\]  

(3)
where \( h_{dc}(t) \) is a slowly varying function of time \((0.01 < \dot{h}_{dc} < 100 \text{ nm/s})\). This results in a modulation of the frequency of the capacitive sensor. This harmonic frequency modulation cannot be read with the counter when \( \omega/2\pi \) is larger than 1 Hz. We built a high-resolution frequency to voltage converter to read this distance modulation between the surfaces. The diagram of this converter is drawn on figure 5. The principle of the operation is as follows: the high frequency signal (frequency \( f \)) is multiplied by a reference signal generated by a stable function generator HP31320A (frequency \( f_{ref} \)). The output signal is a combination of signals at \( f - f_{ref} \) and higher frequency signals. It is first passed through a low-pass filter then directed to a frequency to voltage converter built with a digital phase-lock-loop with a range of \( 5 \times 10^3 \) Hz and a sensitivity of \( 5 \times 10^{-4} \text{ V.Hz}^{-1} \). This frequency-shift technique allows to obtain a high sensitivity in the conversion which could not be directly obtained with a phase lock loop. The final sensitivity on the ac displacement is \( 5 \times 10^{-3} \text{ V.nm}^{-1} \).

The output voltage is connected on a digital two-phase lock-in amplifier (Standford Research System SR830 DSP Lock In Amplifier) whose reference signal is the signal used to drive the piezoelectric element (see figure 8).

The dynamical response of the displacement sensor can be obtained with the same procedure as the static calibration. A white noise excitation containing all the frequencies in the range 0 – 100 Hz is applied on the cantilever \( L_1 \) by the mean of the coil/magnet system. The frequency response of the capacitive sensor is calibrated by the frequency response of the interferometer mounted on \( L_1 \), whose response is flat in amplitude and frequency. This also allows a dynamical calibration of the displacement sensor.

The electrical noise of the capacitive sensor converted in distance is less than \( 1 \text{ pm.Hz}^{-1/2} \) in the range 0-100 Hz, except in the range 49 – 51 Hz, where the noise of the electronics is bigger than a few \( \text{ pm.Hz}^{-1/2} \). Since the dynamic experiments are usually made at a given frequency, this frequency must be chosen not to close to the line frequency.

The mechanical noise on the displacement sensor (figure 9) is due to the mechanical vibrations on the cantilever \( L_1 \) and are much more important than the electronic noise.

3. Application to surface forces measurement

3.1. Experimental system

In this experiments, we use a sphere of 2.7 mm in diameter and a plane made of Pyrex. The surfaces are washed in an ultrasound bath with distilled water and a detergent soap for more than an hour. The surfaces are then rinsed with propanol purified at 99%. Finally the surfaces are passed in a flame in order to burn out the last amount of pollution and to flatten the surfaces. The total roughness of this surfaces measured by an atomic force microscope is less than 0.3 nm rms on a 1 \( \mu \text{m}^2 \) square. The surfaces are quickly mounted on the apparatus.
A small drop of an organic liquid: n-dodecane obtained from Acros Organics, is placed between the surfaces. Dodecane is a simple, Newtonian, non-polar liquid. The length of this molecule obtained by X-ray diffraction is tabulated as 1.74 nm. The liquid has a purity better than 99%. The viscosity of the liquid is given as 1.35 mPl in the handbook at 25°C. The experiments are carried out at ambient temperature, i.e. 25°C. The apparatus is placed in a plexiglass box which reduces sound vibrations, and contains some desiccant (silicagel) to dry the atmosphere and prevent the dissolution of water in dodecane.

3.2. Dynamical measurements: a surface forces apparatus used as a nanorheometer

The experiment starts with the surfaces being separated by a distance of 500 nm. A voltage increasing linearly in time is applied to one of the piezoelectric actuator, so that the sphere moves toward the plane at constant speed. In the same time, we impose a small oscillation of the sphere \( h_{ac} \cos(\omega t) \), with \( h_{ac} = 0.80 \) nm at a frequency \( \omega/2\pi = 64 \) Hz. In the lubrication approximation, the viscous force between the two surfaces gives the well-known expression (Georges (1993)):

\[
F = \frac{6\pi\eta R^2}{h_T} \frac{dh_T}{dt} \tag{4}
\]

In our experiments, the viscous force on \( L_1 \) is:

\[
F = \frac{6\pi\eta \omega R^2}{h} h_{ac} \cos(\omega t + \pi/2) \tag{5}
\]

We can read the displacement \( x_{ac} \cos(\omega t + \psi) \) induced on the cantilever \( L_1 \) by the viscous flow between the sphere and the plane. Since 64 Hz, the value of the excitation frequency, is above the resonant frequency of the cantilever \( L_1 \), the real viscous force \( f_{ac} \cos(\omega t + \phi) \) is obtained by multiplying \( k x_{ac} \cos(\omega t + \psi) \) by the mechanical transfer function of the mass-cantilever system.

First of all, we find that the measured force is out of phase the displacement excitation \( (\phi = \pi/2) \) which means that a purely dissipative force is measured. On figure 8, the inverse of the damping coefficient \( f_{ac}/h_{ac} \) is plotted as a function of the distance \( h \) between the surfaces. This curve clearly shows the a good agreement of the lubrication theory for all the distances greater than 6 nm which corresponds to 5 molecular length of dodecane. The origin \( h = 0 \) is obtained by the linear extrapolation of the best linear fit of the experimental data and the origin of the axis \( h_{ac}/f_{ac} \). The slope of this curve combined with equation (3), gives the viscosity of dodecane. We find \( \eta = 1.37 \times 10^{-3} \) Pl which can be compared to the tabulated value (see Handbook) \( \eta = 1.35 \times 10^{-3} \) Pl at 25°C. This result agrees well those of Tonck et al. (1989) on the same liquid.

4. Conclusion

We have presented a new capacitive sensor for surface forces measurements allowing both static and dynamic measurements between non-transparent surfaces. This sensor
A new capacitive sensor does not need any lock-in amplifier for the statics measurements and has a very low cost. In the static regime, this sensor has a sensitivity of 0.1 nm with an integration time of 1 s. In the dynamic regime, combined with a frequency to voltage converter, this sensor has a sensitivity better than 1 pm Hz$^{-1/2}$.

We have used this sensor for surface force measurements between pyrex surfaces separated by a small meniscus of dodecane. Away from the contact between the surfaces, we have confirmed previous results showing that the bulk viscosity of the liquid is not affected by the confinement. At a distance between the surfaces smaller than a few molecular length, the dissipation increases.

Acknowledgments

We thank J.P. Zaygel for his help in electronics design, C. Cottin-Bizone for his experimental help. We have benefited from discussions with J.-L. Loubet and A. Tonck. We are happy to thank J.-M. Combes for technical help. This work has been supported by the Region Rhônes-Alpes contact number 98B0316 and the Délégation Générale de l'Armement.

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**Figure 1.** Horizontal section of the surface forces apparatus with the capacitor stage. The sphere is moved horizontally by a stepping motor allowing large displacements (a step is 30 nm and the displacement range is larger than 1 cm). A piezoelectric crystal controls the approach of the two surfaces at constant velocities. A second piezoelectric crystal adds a small oscillatory motion to the sphere. This allows to study the dynamic behavior of the sphere-plane interactions. The deflection $x$ of the first cantilever $C_1$ of stiffness $k$ measures the force exerted on the plane by the sphere. (a): schematic diagram. (b): mechanical diagram.

**Figure 2.** Diagram of the electronic oscillator.

**Figure 3.** •: Measured frequency $f$ (Hz) of the oscillator as a function of the relative displacement between the surfaces measured by an interferometric method. The displacement is imposed by the coil/magnet system. The full line represents the best linear fits of the data. This shows the very good linearity in the common measure range of a surface forces apparatus.

**Figure 4.** Measured frequency $f$ (Hz) of the oscillator for a fixed capacitor value.

**Figure 5.** Diagram of the electronics of the frequency to voltage conversion. The function generator used to provide a fixed frequency oscillatory signal is a Hewlett Packard HP313120A function generator.

**Figure 6.** Schematic diagram of the dynamical measurements.

**Figure 7.** Vibration spectrum measured on the capacitive sensor. The 50 Hz signal is large and the other main peaks are some vibration peaks observed in the environment. Two important peaks have been indexed on this figure: a: the resonant peak of the force cantilever, b: the resonant peak of the anti-vibration device.
Figure 8. Plot of the inverse of the damping coefficient $h_{ac}/f_{ac}$ as a function of the displacement $h_{dc}$, for dodecane at 25°C. $h_0 = 0.3$ nm for $h_{dc} > 6$ nm. The arrows indicate the inward (→) and outward (←) approach: no hysteresis in the dynamical response has been observed. The dotted line is the best linear fit of the data.
oscillator frequency to voltage converter
optical sensor

\[ x_{ac} \cos(\omega t + \psi) \]

\[ h_{ac} \cos(\omega t) \]

sinusoidal signal

lock-in amplifier

gpib

k

ac

\[ \cos(\omega t) \]
