

Application of the Light-Lever Technique to the Study of Colloidal Forces

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Force measurements in a variety of colloidal systems have been conducted using a new device which utilizes the light-lever technique employed in atomic force microscopy. The systems studied include electrostatic double-layer forces, hydrophobic interactions, liquid crystal structures, and polymer adsorption forces. The new device has some clear advantages over adaptation of an imaging atomic force microscope for force measurements.

Introduction

Measurement of the interaction forces between surfaces in aqueous solutions is of central importance for the understanding of fundamental processes, such as the stability of colloidal solutions, froth flotation, and clay swelling. The forces operating in these systems are typically electrostatic/electrodynamic in origin, although there is a substantial body of evidence in support of additional forces due to direct surface-solvent interactions (i.e., the so-called solvation forces).¹

The first accurate, direct measurements of forces between macroscopic solid surfaces immersed in aqueous electrolytes were reported in 1978 by Israelachvili and Adams² using a technique based on the use of muscovite mica, a material originally suggested by Debye. The separation distance between these molecularly smooth crystals could be accurately measured using interferometry and the force obtained by measurement of the deflection of a spring. Although problems were encountered, the forces in some cases³ were found to be in complete agreement with the theory of Derjaguin, Landau, Verwey, and Overbeek (DLVO).

Although the "surface forces apparatus" (SFA) technique has been successfully applied to the detailed study of surface interactions, it is limited by the requirements that the substrates are (i) composed of thin (μm) sheets, (ii) molecularly smooth on both faces over a relatively large area of several square centimeters, and (iii) semitransparent. So far, the technique using mica substrates has been extended to include silica⁴ and alumina,⁵ which can be produced in this form. However, these substrates are difficult to handle and the method is most effective with mica substrates. Partly to overcome this limitation, but also to improve the simplicity of data acquisition, a new technique was recently developed using an atomic force microscope.

The atomic force microscope (AFM) or scanning probe microscope (SPM) was developed⁶ following the dramatic appearance of the scanning tunneling microscope, and

both owe their development to the availability of improved piezoelectric devices, digital signal processing, and extended PC storage. The AFM device has the advantage of being able to image nonconducting surfaces to high resolution in aqueous solution, which enables the study of a wide range of solid-liquid interfaces under real conditions. A topographic image of the surface is obtained by monitoring the vertical movement of a piezo required to maintain a constant spring deflection, as the tip of the spring is scanned across the surface also by the piezo. This information is stored on the computer with the relative position and then used to generate a three-dimensional image of the surface.

In 1991, a commercial AFM device (Digital Instruments, Nanoscope II) was adapted to detect the spring deflection resulting from the interaction of a fine colloidal particle attached to the stylus with a flat substrate of the same material, immersed in a range of aqueous electrolyte solutions.⁷ Using this technique, colloidal forces were measured directly for the first time. The results obtained using a silica glass colloid and flat were found to be in good agreement with the DLVO theory down to surface separations of about 3–4 nm. The change in decay lengths with added electrolyte also agreed with theory.⁸ In addition, the surface electrostatic potentials extracted from the DLVO theoretical fits are consistent with values obtained using other techniques, such as microelectrophoresis. As observed in earlier studies using other techniques, the forces were found to be strongly repulsive at short range, rather than attractive as expected from the effect of the van der Waals component of DLVO theory. These repulsive forces are thought to be caused by the solvation of the silica surfaces due to the hydrogen bonding between the surface silanol groups and adjacent water layers. The range expected for this type of interaction is a point of some controversy but appears to be up to 1–2 nm per surface.⁹

The versatility of this new colloid probe technique is well illustrated by many of the studies since 1991, using colloidal materials such as ZnS,¹⁰ polystyrene,¹¹ polypropylene,¹² alumina,¹³ titania,¹⁴ and gold.¹⁵ A further

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advantage of the AFM technique is the ability to study the interaction between two different surfaces, of relevance to heterocoagulation.¹⁰

Although commercial AFM instruments have been successfully adapted for the measurement of colloidal forces, they have been designed primarily for surface imaging and recently there has been some criticism of the technique based on the digital control of the piezo and subsequent oscillations of the cantilever leading to an apparent hysteresis in the force curve.¹⁶ In this paper, we describe a light-lever device similar to the Digital Instruments AFM, operated by software designed to give almost complete control of surface separation and monitor surface forces under both very slow and very fast interaction conditions. It is anticipated that this control will provide a means to more fully investigate errors associated with the technique and develop methods to minimize or avoid them. Using this device, we have investigated a wide variety of colloidal interactions.

Materials and Methods

The force of interaction between two surfaces is determined using reflected laser light to detect the deflection of a spring. The reflected light is directed onto a split or quadrant photodiode via a mirror. Any movement of the spring results in the light spot moving across the face of the photodiode and leads to a voltage change. The movement of the spot is monitored by the difference in voltage between the two halves of the split diode, A and B, and is normalized by the total signal giving the unitless value $(A - B)/(A + B)$. The optimum linear response is obtained for a rectangular laser spot in the center of the split diode, as illustrated in Figure 1. Experiments are usually conducted with $(A - B)/(A + B)$ values of between +0.1 and -0.1 (i.e., in the linear response region). Analysis has shown that while the gap between the diode elements may affect sensitivity, it has no effect on the linearity of response. Forces as small as 0.1 nN can routinely be detected with this method.

The distance between the surfaces was controlled by two means. A coarse adjustment can be made using the micrometers that support the light-lever assembly head, illustrated in Figure 2. The spring can be raised or lowered in relation to the substrate in this manner. With a little practice, changes in separation of as little as 100 nm can be made. Fine separation control was obtained by displacement of the substrate which was attached to a piezoelectric tube. The piezoelectric tube was expanded and contracted by application of a dc voltage ranging from -500 to +500 V. This gave a distance range of about 2000 nm for the piezo used here. The instrument can be fitted with other piezo tubes giving smaller ranges for precision or larger ranges of more than 6000 nm. The voltage supplied to the piezo tube can be controlled manually or by computer, thereby controlling surface separation. In practice, the micrometers are used to bring the surfaces within range of the piezo extension and all measurements were conducted using the piezo to control separation. The piezo tube was calibrated by an optical interference technique (FECO) using silvered mica sheets in the Mk IV surface forces apparatus. Typical results are shown in Figure 3 which demonstrate the linearity of the piezo crystal over this range. Our results indicate that the degree of expansion of the piezo tube is unaffected by the rate at which the voltage is supplied over the time scales used in this work. A small degree of piezo hysteresis is seen. This does not affect the force curves produced as the approach and separation runs are scaled separately. Any data effected by hysteresis occurs at the top of the compliance region upon separation and can be eliminated.

The light-lever assembly has several noteworthy features. The cantilever spring can be mounted in a commercial Nanoscope

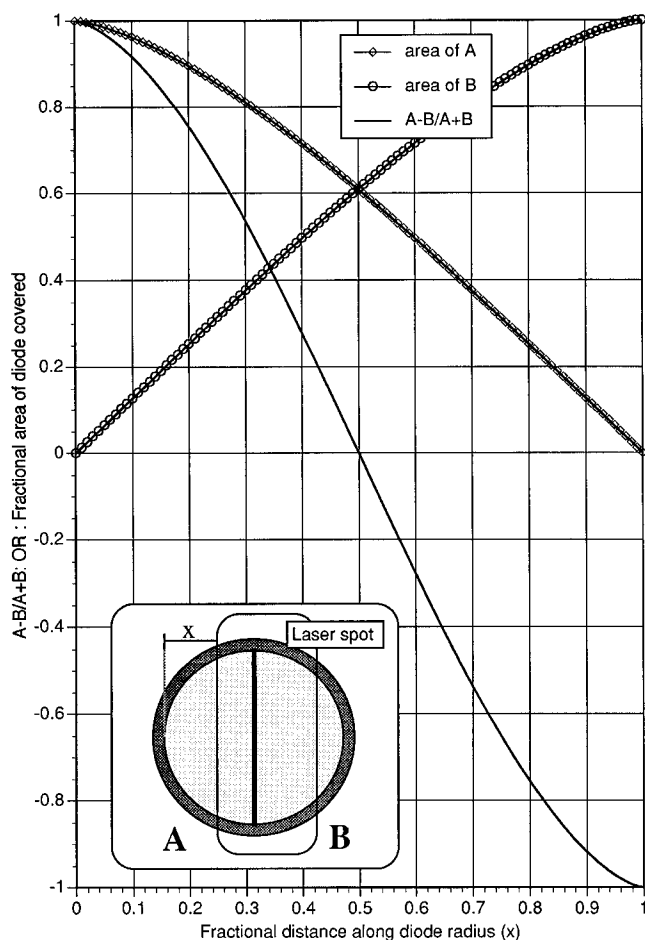


Figure 1. Calculated split diode response for a rectangular laser light spot versus position on the diode. The voltage (A and B) generated by each element is proportional to the area covered by the light. The experimentally important parameter $(A - B)/(A + B)$ is shown to vary linearly when the light spot is centrally located. In practice, experiments are conducted with $(A - B)/(A + B)$ values between -0.1 and +0.1.

fluid or air cell or, alternatively, in specially constructed cells.¹⁷ Both commercial and custom-made springs can be used. The cell was positioned using a kinematic mount to ensure that no movement of the cell occurs once clamped in place. The photodiode was fixed in an XY translator which enabled optimum light capture. Also, the diode was mounted on a swing arm so that it could be moved to enable adjustment of the mirror to view the laser spot position on the cantilever. This simplified the alignment of the laser spot on the tip of the cantilever and also allowed a clear view of the substrate. The photodiode housing was designed to accommodate a variety of photodiodes. The diode can be changed quickly and easily allowing for comparison of different photodiodes and selection of the most suitable diode for the application. Up to four photodiodes can be hard wired into the computer at any one time.

The laser diode is operated by a current supply developed by Bradley et al.¹⁸ to minimize fluctuations in light intensity and is controlled manually. Typically, the current to the laser was raised until any further increase produced no further increase in voltage at the photodiode and then increased a further 10%. This ensured that the photodiode response was proportional to the area of the photodiode covered and insensitive to any fluctuations in light intensity.

The photodiode signals were digitized (16 bit) via an analog breakout box using a MacADIOS II/16 card installed in a Macintosh Quadra 700. Piezo control was either manual or via a 16-bit daughterboard. Typically one bit corresponds to a voltage

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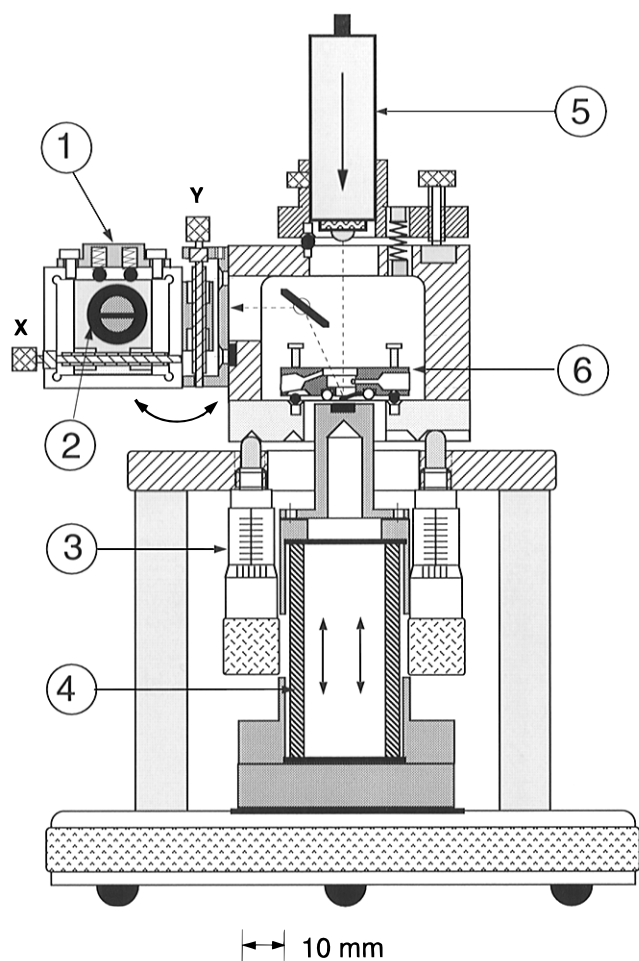


Figure 2. Light-lever instrument for force evaluation (LLIFE), indicating (1) miniature *XY* translation stage, (2) split photodiode, (3) micrometer, (4) piezoelectric tube, (5) laser, and (6) cantilever mounted in a fluid cell.

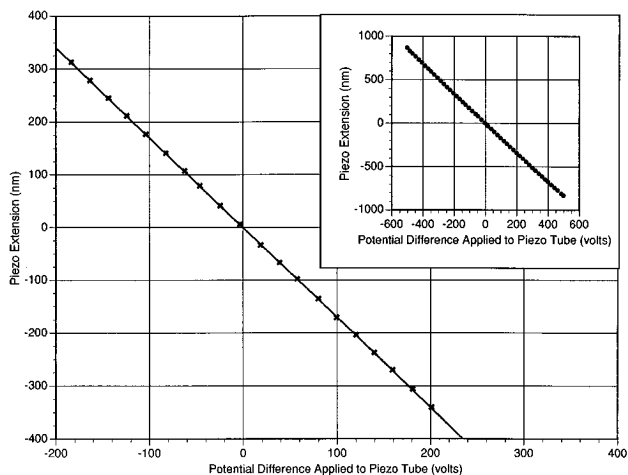


Figure 3. Piezo extension as a function of applied voltage. The extension was measured by an optical interference (FECO) technique, using silvered mica crystals.

change of 15 mV or a piezo extension of approximately 30 pm. The 16-bit control of the piezo eliminates artifacts due to digitization, as described by Siedle and Butt.¹⁶ Instrument control was via a front control panel using Superscope IIE software, as illustrated in Figure 4. The front panel displayed the piezo output, the diode input channels A and B, and the calculated $(A - B)/(A + B)$ wave form.

This arrangement enabled user control over a range of ramping wave forms, ramping rates, signal digitization, data manipulation, and data storage. Full ramps could be applied in less than

50 ms depending on the number of data points captured. Long-time runs were limited only by instrumental drift, which could be substantially reduced by good thermal control.

The stored diode signal versus distance data was converted to a force curve in the usual manner.⁸ The diode signal versus distance curve can be divided into three zones. At large separations, where there is no surface interaction, the diode signal is constant. This serves as an indicator of zero force. The next zone is that in which the surfaces are interacting and essentially contains the force versus distance information desired. The final zone is the compliance region, where the probe is in hard contact with the substrate and spring deflection is a direct response to the movement of the substrate generated by the piezo tube. This linear region serves to calibrate the diode signal $(A - B)/(A + B)$ in terms of the deflection of the spring. In addition, the deflection of the spring affects the separation of the surfaces and the distance data derived from the piezo voltage must be modified to account for this. Knowledge of the spring constant gives the magnitude of the force for any degree of deflection. An acceptable diode response must exhibit both a constant diode signal at large separations and a linear change in diode signal with distance in the compliance region.

The results presented here were obtained with a minimum of temperature and vibration control. It is anticipated that with a simple vibration isolation arrangement mounted in a temperature control cabinet an improvement in stability would be obtained, but this was found to be unnecessary for systems studied in this work.

Interaction forces between a silica flat and a silica sphere (Polysciences, 20 μm diameter) were measured in water and aqueous solutions of NaCl (AR grade, roasted at 500 $^{\circ}\text{C}$ for 5 h). The flat and sphere were plasma treated (0.65 Torr H_2O , 0.10 Torr Ar, 10 W, 45 s) to ensure cleanliness and complete hydroxylation of the surface. The interaction force between silica surfaces in solutions containing a range of concentrations of the cationic surfactant cetylpyridinium chloride (CPC) was measured in a background of 0.1 M NaCl. All water used in these experiments was passed through a coarse lamb's-wool filter, a reverse osmosis membrane, and an activated charcoal column before single distillation and storage in a laminar flow cabinet.

The sphere was fixed to a Nanoscope Si_3N_4 cantilever of a spring constant of 0.18, 0.035, or 0.21 N/m using Epikote resin and following the method developed by Ducker et al.⁸ Spring constants of a number of cantilevers from each wafer were determined using the method of Cleveland et al.,¹⁹ and the average spring constant was used to scale the forces. The substrate was attached to a square stub of magnetic stainless steel using a small amount of partially polymerized Araldite.

The effect of the nonionic water-soluble polymer poly(vinylpyrrolidone) (PVP) and a lamellar AOT phase on the interaction forces between silica surfaces was also studied. The polymer sample was of a special grade, supplied by Aldrich, with an average molecular mass of 40 000 g/mol and was used without further purification. The pH measurements revealed that the polymer solutions were acidic. A titration with NaOH demonstrated typical strong-acid/strong-base behavior indicating that the acidity arose from an impurity and was not due to the presence of acid groups on the polymer. Indeed, strong acid may have been used to quench polymerization in the manufacture of the polymer, as polymerization proceeds under alkaline conditions.²⁰ The aqueous lamellar phase consisted of 15% AOT and 1.9% NaNO_3 by weight. The sample was allowed to equilibrate at 25 $^{\circ}\text{C}$ with frequent shaking over a period of about 10 days.

Results and Analysis

Forces measured between a plasma-treated silica glass colloid and a flat surface immersed in aqueous 0.001 M NaCl solution using the light-lever technique are shown in Figure 5. The upper and lower solid lines were calculated using a numerical solution to the Poisson–

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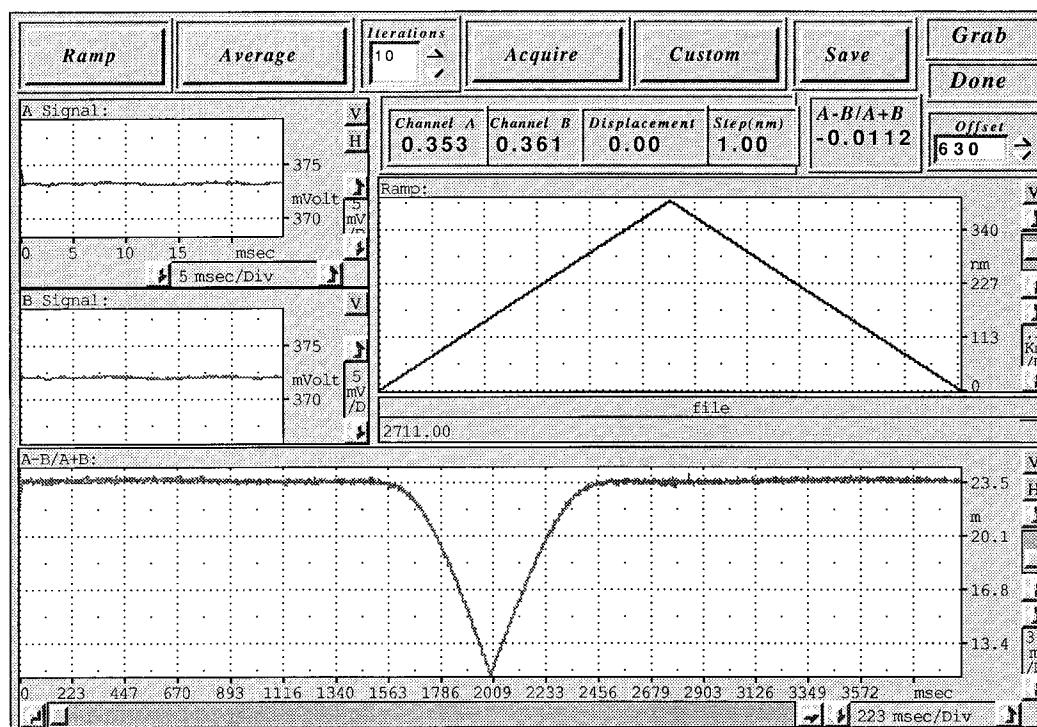


Figure 4. Interactive front panel employed to control the Superscope IIE software which was used to operate the instrument.

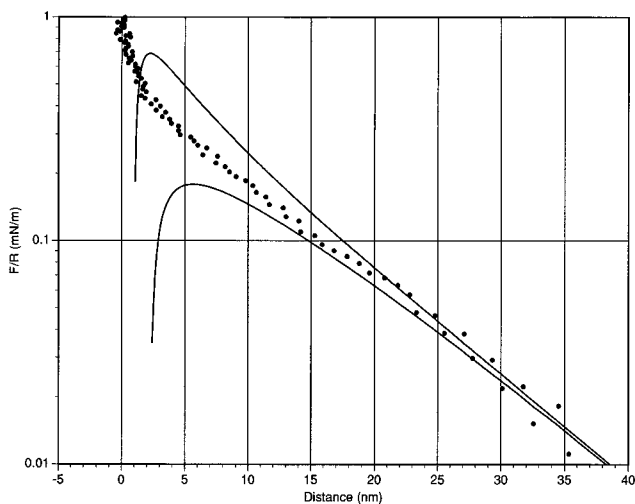


Figure 5. Interaction between silica surfaces in 0.001 M NaCl aqueous solution. The solid lines shown are calculated theoretical curves for a potential of -26 mV, a Debye length of 9.6 nm, and a Hamaker constant of -0.85×10^{-20} J, with boundary conditions of constant charge (upper curve) and constant potential (lower curve).

Boltzmann equation²¹ for boundary conditions of constant surface charge (upper) and constant surface potential (lower). The attractive van der Waals force was included as a nonretarded interaction with a Hamaker constant of 0.85×10^{-20} J. The observed forces correspond closely to the expected Debye length for this solution of 9.6 nm and appear to regulate in between the constant charge and potential conditions. As observed in earlier studies, the surfaces did not jump into primary minimum contact but there was a repulsive force extending about 1.5 nm per surface.⁹ The surface potential detected for these surfaces was unusually low at -26 mV, and this produces significantly lower forces, which demonstrates the sensitivity and precision of the technique. The low potential

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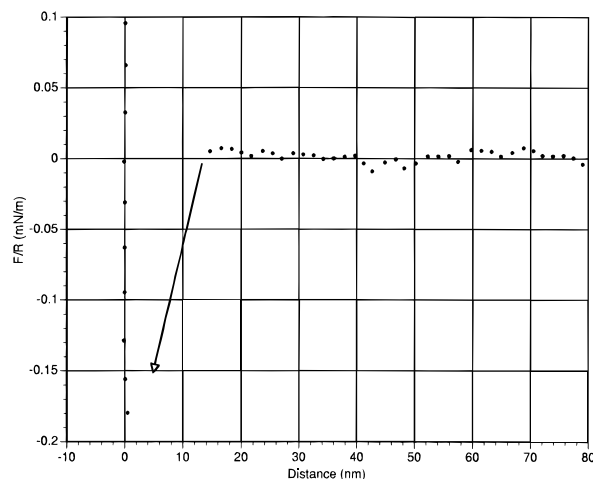


Figure 6. Interaction on approach between silica surfaces immersed in an aqueous solution of 3.0×10^{-6} M CPC and 0.1 M NaCl. No interaction forces were observed prior to the sudden onset of a spring instability (shown by an arrow) at which the surfaces jumped into adhesive contact.

can be attributed to the use of a soda glass sphere which contains calcium and sodium ions.

Silica surfaces immersed in solutions of 3.0×10^{-6} M CPC in 0.1 M NaCl adsorbed a submonolayer of the cationic surfactant, resulting in a mildly hydrophobic surface. The interaction is characterized by a jump into contact from approximately 15 nm, as seen in Figure 6. Any electrostatic DLVO component of the force is screened by the high background salt concentration.

When the concentration of CPC was increased to 5.0×10^{-6} M in 0.1 M NaCl, a more complete monolayer was apparently adsorbed at each silica surface, rendering them highly hydrophobic and producing a substantial increase in jump distance (see Figure 7). The jump distance varied from run to run and ranged between 30 and 50 nm and was much larger than the calculated (retarded) van der Waals interaction shown. The van der Waals forces were

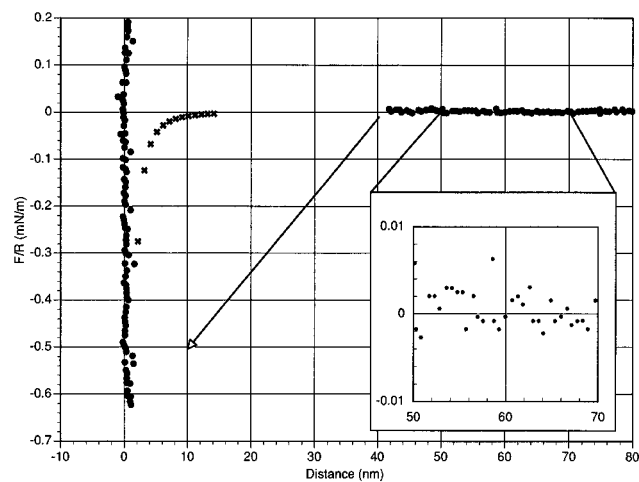


Figure 7. Interaction on approach between silica surfaces immersed in an aqueous solution of 5.0×10^{-6} M CPC and 0.1 M NaCl. No interaction forces were observed prior to the sudden jump into contact from approximately 42 nm, due to the gradient of the attractive force rapidly exceeding the spring constant. The calculated retarded van der Waals interaction for silica/water/silica is shown for comparison (small crosses).

calculated using the Lifshitz equation with a spectral model for water and soda glass.²²

The mechanism of the long-range hydrophobic interaction is currently unknown and a source of much debate.^{23–28} No interaction was observed at separations greater than the spring instability point, indicated by an arrow. This feature suggests a mechanism which involves either a single catastrophic event or an avalanche effect. Whereby, a small perturbation gives rise to further changes leading to a sudden inward jump. Other workers have observed a more gradual onset of the interaction.^{29,30} The difference may be due to the smaller radius of the probe used in this work. Processes involving instabilities in the separating medium or cavitation as the surfaces approach may behave in this way and be more obvious when a small probe is used. The variation seen in the jump distance from run to run is also consistent with such a mechanism. By comparison, these effects will essentially be averaged for less highly curved surfaces and hence give a more gradual onset of the force and a more reproducible interaction. If indeed, the mechanism proves to be related to fluid instability and depends on curvature, the implications for the manifestation of the force on a molecular level are far-reaching. The differences observed between the long-range macroscopic hydrophobic interaction and the short-range molecular interaction may be related to the nature of this fluid instability.

Also shown as an inset in Figure 7 is an expanded region of the base line showing the "noise" in the measurement of force/radius. Analysis of the base line data at separations between 50 and 80 nm revealed a peak to peak noise of less than $10 \mu\text{N/m}$ and a standard deviation of only 2.2

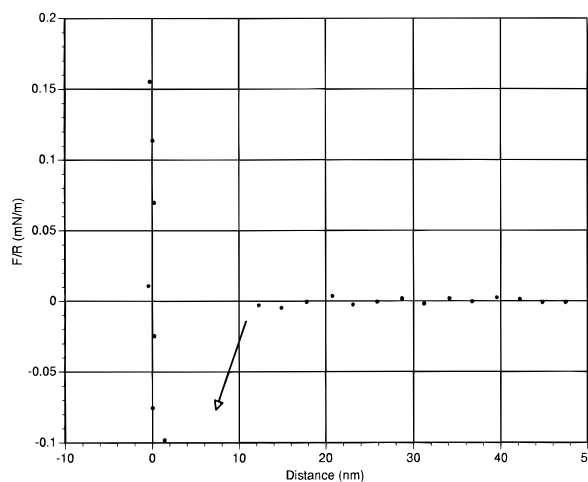


Figure 8. Interaction on approach between silica surfaces immersed in an aqueous solution of 1.0×10^{-5} M CPC and 0.1 M NaCl. No interaction forces were observed prior to the sudden onset of a spring instability (shown by an arrow) at which the surfaces jumped into adhesive contact.

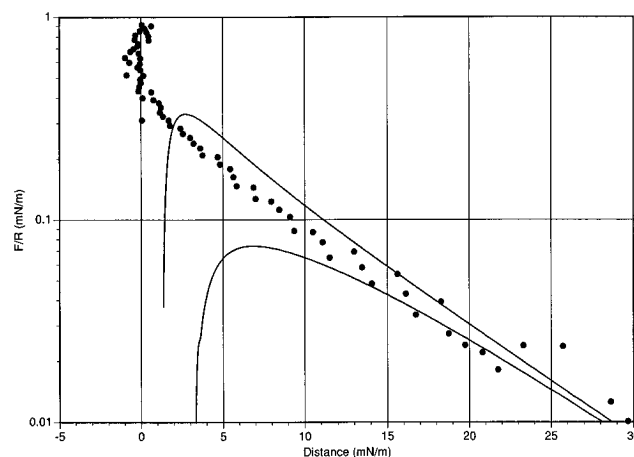


Figure 9. Interaction between silica surfaces immersed in 1.0×10^{-3} M CPC aqueous solution. The solid lines shown are calculated theoretical curves for a potential of +18 mV, a Debye length of 8.5 nm, and a Hamaker constant of -0.85×10^{-20} J, with boundary conditions of constant charge (upper curve) and constant potential (lower curve).

$\mu\text{N/m}$. This compares favorably with the accepted noise level of $5 \mu\text{N/m}$ quoted for the SFA. It is anticipated that further development will reduce the noise even further.

An increase in the CPC concentration to 1.0×10^{-5} M in 0.1 M NaCl resulted in a partially formed bilayer and a reduction in hydrophobicity, as seen in Figure 8. Qualitatively, this interaction is similar to that seen in the submonolayer case, with a moderate hydrophobic attraction leading to a jump into contact from around 12 nm.

At the critical micelle concentration (cmc) of about 1.0×10^{-3} M, a surface aggregate was formed at each silica surface resulting in overall electrostatic repulsion. Until recently it was believed that a bilayer was formed on silica surfaces around the cmc; however, recent work by Manne³¹ has shown that micelle-like aggregates may be formed. The interaction can be fitted with a potential of +18 mV and a Debye length of 8.5 nm (see Figure 9). This potential corresponds to an area per charge of 105 nm^2 . With an estimated CPC molecule head group area of about 0.4 nm^2 , the dissociation of chloride ions was found to be very low at about 0.4%. The interaction curves obtained on

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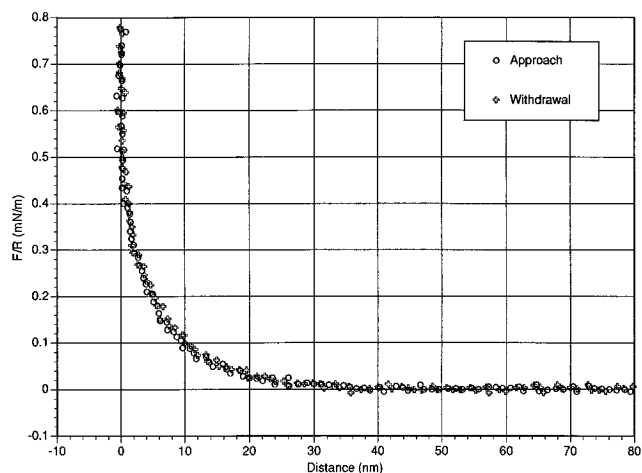


Figure 10. Interaction between silica surfaces immersed in 1.0×10^{-3} M CPC aqueous solution. The values shown were collected on approach and withdrawal, demonstrating the precision and reversibility of the instrument.

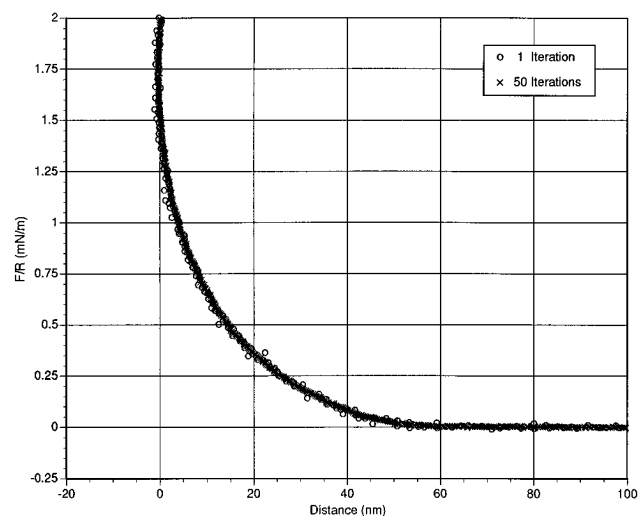


Figure 11. Interaction between silica surfaces in 0.1% (w/w) PVP and 0.015 M $\text{Ca}(\text{NO}_3)_2$ in aqueous solution. The values shown are for an average of 50 runs at a rate of 1000 nm/s and a single run at a rate of 33 nm/s.

approach and retraction were found to be almost identical, as illustrated in Figure 10, which indicates the reversible nature of the surface aggregate compression. This result also serves to indicate the precision of the instrument and demonstrates that piezo hysteresis did not present a problem in this system.

One of the advantages of the light-lever technique is the ability to vary the rate of measurement over a very wide range. Related to this is the ease with which repeat runs can be conducted. In Figure 11 the average of 50 force runs is given for these solution conditions and compared to the results of a single approach. Averaging can be used to improve accuracy if there are no thermal drifts in the position of the surfaces during measurements. In this case the surfaces were ramped together at a rate of 1000 nm/s for the 50-iterations curve and 33 nm/s for the single approach. The comparison indicates that the rate of approach did not affect the polymer adsorption density or configuration. The high linearity of the compliance region for these experiments suggests that the PVP was easily desorbed from the silica surfaces as

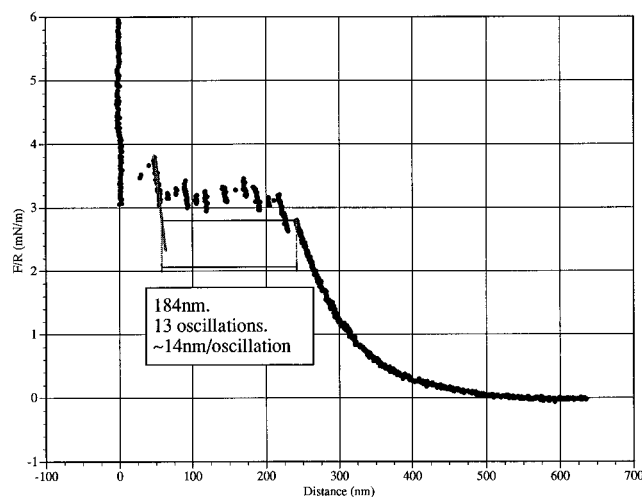


Figure 12. Interaction between silica surfaces immersed in an aqueous lamellar phase consisting of 15% AOT and 1.9% NaNO_3 by weight, at a ramp rate of 0.83 nm/s. Approximately 14 nm oscillations were clearly detected against a repulsive background force.

they were forced together and that readsorption occurred fairly rapidly within at least 1 s.

In a further study to test instrument versatility, we examined the interaction between silica surfaces immersed in a lamellar AOT phase. Oscillations in the force were detected from separations of about 250 nm (see Figure 12). Each oscillation was separated by a discontinuity of 14 nm periodicity, where the probe apparently pushed through each lamellar layer. This agrees well with the 14.7 nm periodicity seen in X-ray diffraction studies.³² This interaction was measured over a period of nearly 1 h, demonstrating the ability to measure forces over long-time intervals using this technique.

Conclusions

The successful application of atomic force microscopy to a diverse range of colloid and surface phenomena has led to a renewed interest in the basic light-lever method for colloid force measurements. We have demonstrated in this work that a relatively simple, versatile device, based on a commercial AFM but without imaging capability, can be used to obtain detailed, precise information not only on concentrated surfactant systems, requiring wide ranging ramp rates, but also on relatively simple double-layer forces. We have also shown that very precise force data can be obtained by averaging over many repeat scans when thermal and electrical drift were almost completely absent during the period of measurement.

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