Use of Capacitance To Measure Surface Forces. 2. Application to the Study of Contact Mechanics

Peter Frantz,† Alexander Artsyukhovich, Robert W. Carpick,‡ and Miquel Salmeron*

Materials Sciences Division, Lawrence Berkeley National Laboratory, University of California, Berkeley, California 94720

Received March 10, 1997. In Final Form: August 25, 1997⊗

We performed capacitance measurements between silver layers on the back side of thin (<1 μm) mica substrates in surface force apparatus to determine the contact area with very high precision (<0.1% of the total area) and at a rate that is faster (∼1 kHz) than that in more traditional methods based on interferometry. To demonstrate the capabilities of the technique, we measured the adhesion and adhesion hysteresis between two mica surfaces. A peculiar discontinuous decrease in contact area, with decreasing load, between two dry mica surfaces is observed. We also studied the possible effect of shear forces on the value of the contact area due to adhesive forces, as in JKR theory.

Introduction

When two solid substrates are brought into contact with one another, interfacial adhesive forces play an important role in modifying the area of contact beyond the value expected from pure elastic deformation. Because of these forces, it is necessary to apply a negative load (pull-off force) to separate the surfaces. The accurate determination of the contact area between elastic solids is thus critical in determining their adhesive properties and for a more complete understanding of the mechanisms of friction[1−3] and fracture.[4−6] Yet it is widely recognized that the experimental contribution to this determination lags far behind the theories which have been developed. The difficulty in providing adequate experimental support lies primarily in the roughness of real surfaces and the fact that the actual area of contact, often the sum of several microscopic contacts (asperities), is not known with precision. A number of techniques was used to study contact mechanics in the past.[7−12]

The surface forces apparatus (SFA) was developed following the pioneering work of Tabor.[13,14] It provides well-defined, molecularly smooth contacts in a geometry that is ideal for the study of contact mechanics. In its most common form, the instrument relies on light interferometry between silver layers deposited on the back side of mica sheets.[15] These interference fringes of equal chromatic order (FECO) provide both a measurement of the separation between the surfaces and a cross-sectional image of the contact. This technique is now routinely used to determine the adhesion force between two surfaces by monitoring the growth of contact area with applied load.[16,17] Studies of contact shape[13,16,18] and adhesion hysteresis[5,9,10,19] have also been common.

Although the FECO method provides excellent resolution in the measurement of the distance of separation between the surfaces (with angstrom resolution[20]), the determination of the contact diameter is 10^3 times less accurate.[16] We also find that the contact edges of the interference fringes are often diffuse due to optical imperfections in the mica−glue−silica substrate, further limiting the resolution and reproducibility of the measurements. Furthermore, the time required to collect and analyze a single interference fringe, typically ∼30 s, forces the overall time required for a complete experiment to exceed several minutes. This precludes observation of rapid dynamic processes, such as some physicochemical surface reconstructions[5,9] and requires of the scientist an affinity for tedium.

Monitoring the electrical capacitance between the silver layers on the back side of the mica surfaces provides an easy and alternative method that overcomes many of these difficulties. In a previous article, we described the method in detail[21] as applied to the determination of the distance of separation. Here we describe how the method can also be used to measure changes in the contact area. Because of its superior accuracy, it allows us to study in greater detail how this area varies as a function of normal load, giving the adhesion and adhesion hysteresis. It allows us also to ascertain the role of lateral forces in modifying the contact area, which has fundamental implications for the interpretation of fracture mechanisms.[22] The capacitance method not only is simpler but also is much less demanding of the quality of the mica and silver coating. Because of

† Present address: The Aerospace Corporation, 2350 El Segundo Boulevard, El Segundo, CA 90245-4691.

‡ Also with the Department of Physics, University of California, Berkeley, CA 94720.

⊗ Abstract published in *Advance ACS Abstracts,* October 1, 1997.

(1) Israelachvili, J. N. In *Fundamentals of Friction: Macroscopic and Microscopic Processes*; Singer, I. L., Pollock, H. M., Eds.; Kluwer Academic Publishers: Dordrecht, 1992.

(2) Savkoor, A. R. In *Fundamentals of Friction: Macroscopic and Microscopic Processes*; Singer, I. L., Pollock, H. M. Eds.; Kluwer Academic Publishers: 1992.

(3) Bowden, F. P.; Tabor, D. *The Friction and Lubrication of Solids*; Clarendon Press: Oxford, 1950.

(4) Lawn, B. R. *Appl. Phys. Lett.* **1985**, *47*, 809.

(5) Israelachvili, J. N.; Berman, A. *Isr. J. Chem.* **1995**, *35*, 85.

(6) Maugis, D. *J. Colloid Interface Sci.* **1991**, *150*, 243.

(7) Johnson, K. L.; Kendall, K.; Roberts, A. D. *Proc. R. Soc. London, Sect. A* **1971**, *324*, 301.

(8) Hough, D. B.; Ottewill, R. H. *Prog. Colloid Polym. Sci.* **1983**, *68*, 101.

(9) Chaudhury, M. K.; Owen, M. J. *J. Phys. Chem.* **1993**, *97*, 5722.

(10) Chaudhury, M. K. *Mater. Sci., Eng. Rep.* **1996**, *R16*, 97.

(11) Parker, J. L.; Claesson, P. M. *Langmuir* **1994**, *10*, 635.

(12) Carpick, R. W.; Agrait, N.; Ogletree, D. F.; Salmeron, M. Submitted for publication.

(13) Tabor, D. *J. Colloid Interface Sci.* **1977**, *58*, 2.

(14) Tabor, D.; Winterton, R. H. *Proc. R. Soc. London, Sect. A* **1969**, *312*, 435.

(15) Israelachvili, J. N. *J. Colloid Interface Sci.* **1973**, *44*, 259.

(16) Horn, R. G.; Israelachvili, J. N.; Pribac, F. *J. Colloid Interface Sci.* **1987**, *115*, 480.

(17) Christenson, H. K. *J. Phys. Chem.* **1993**, *97*, 12034.

(18) Maugis, D.; Gauthier-Manuel, B. *J. Adhes. Sci. Technol.* **1994**, *8*, 1311.

(19) Scmitt, F.-J.; Yoshizawa, H.; Schmidt, A.; Duda, G.; Knoll, W.; Wegner, G.; Israelachvili, J. N. *Macromolecules* **1995**, *28*, 3401.

(20) Grunewald, T.; Helm, C. A. *Langmuir* **1996**, *12*, 3885.

(21) Frantz, P.; Agrait, N.; Salmeron, M. *Langmuir* **1996**, *12*, 3289.

(22) Johnson, K. L. Submitted to *Proc. R. Soc. London, Sect. A.*

S0743-7463(97)00265-5 CCC: $14.00

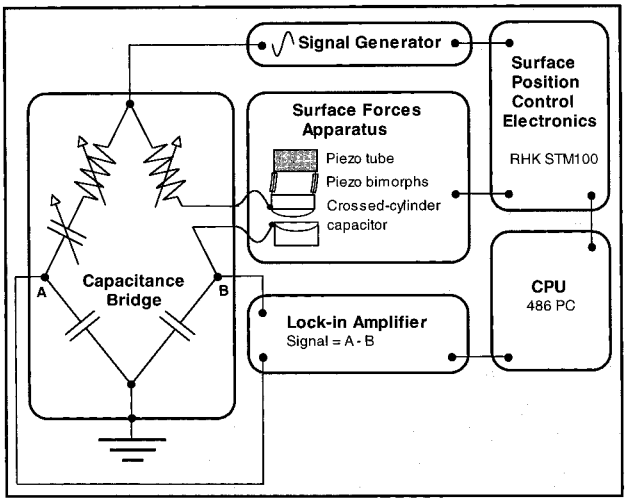

Figure 1. Schematic of the instrumental arrangement used to measure the capacitance between the silver layers on the backside of thin mica substrates in the surface forces apparatus.

its high signal-to-noise ratio, it allows study of rapid processes at a rate limited only by the response time of the electronics (10^{-3} s in the present configuration). An additional advantage of the capacitance method is that it lends itself naturally to dielectric spectroscopy studies of the material interposed between the mica sheets.[23] This study differs from previous studies[24,25] with its use of the capacitance method in conjunction with the FECO method for the purposes of calibration and comparison.

Design

A schematic diagram of the experimental setup is shown in Figure 1. The silver-coated mica sheets in the SFA form a capacitor in a bridge which contains a high-precision variable capacitor on the opposite arm. A signal generator sends a high-frequency sine wave voltage to the bridge. At the beginning of an experiment, the capacitance is balanced so that no potential difference exists between points A and B (see Figure 1). When capacitance between the crossed mica cylinders is changed, a difference in potential arises between points A and B.[26] The in- and out-of-phase components of this high-frequency signal are then collected by a two-channel lock-in amplifier and sent to a computer for analysis. A temporal resolution of 1 ms is easily achieved in the 20−100 kHz range.

A homemade SFA, described in detail elsewhere,[13] was used for these experiments. Unless otherwise noted, the experiments discussed below were conducted with mica glued to cylindrical-fused silica substrates of 1 cm radius of curvature and 1 cm diameter. A 50 nm layer of silver was sputter-deposited onto the back side of the mica, which was then affixed to the cylindrical silica lenses with a comparatively thick (100 μm) layer of compliant-thermo-setting epoxy, Epon 1004 (Shell Chemical Co.). The lower surface was supported by double cantilever leaf springs with a spring constant of 320 N/m. The chamber was purged and sealed with dry nitrogen and with P_2O_5 inside to act as an aggressive desiccant. In our apparatus, the support for the upper surface was modified to allow the controlled application of lateral forces. The arrangement is similar to the friction force device developed by Van Alsten and Granick[27] in that the substrate is suspended on two piezoelectric bimorphs.

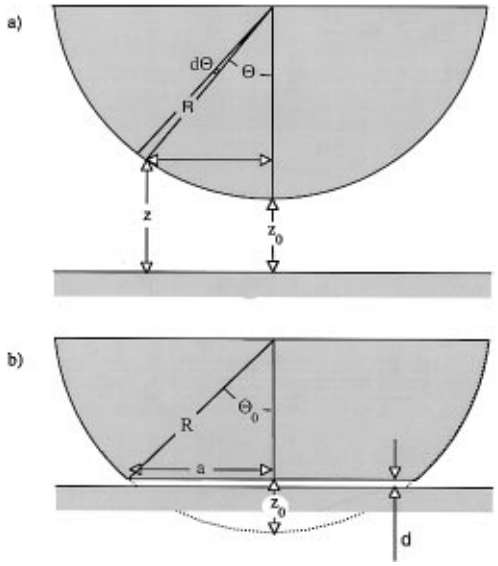

Figure 2. Illustration of the geometry used to calculate the capacitance between a flat and a sphere in close proximity, (a) before and (b) during contact. R is the radius of curvature of the mica surface, z_0 is the distance between the surfaces (during contact, $z_0 < 0$), a is the contact radius, and d is the total thickness of the mica between the silver electrodes when in contact. Typical values of these parameters are $R = 1$ cm, $d = 1\,\mu$m, and $a = 30\,\mu$m for loads in the 32 mN range. During such a contact, $C = 1-4$ pF.

The potential difference between A and B can be converted to capacitance units and then to values of the separation between the two mica surfaces or their contact area, using the appropriate calculations based on the particular geometry. Absolute calibration, however, is best obtained by the simultaneous measurement of the geometrical parameters at a few points using the FECO method.

Relation between Capacitance and Contact Area

The functional dependence of the capacitance on the distance and contact area between the mica surfaces can be numerically calculated for a known geometry. It is more instructive, however, to derive an approximate solution that can be easily obtained for the crossed cylinder configuration, which is equivalent to a sphere and a flat surface, when their separation is much smaller than the radius.[28] In these conditions, the field lines are parallel to each other and perpendicular to the two surfaces. The capacitance can then be obtained by simple integration of contributions of annular elements, as shown in Figure 2. The result is:

$$C \approx 2\pi\epsilon R \int_0^{\pi/2} \frac{\sin\theta\cos\theta\,\mathrm{d}\theta}{(1-\cos\theta)+z/R}$$

where R, z, θ, and ϵ are the radius of curvature of the lenses, their shortest distance of separation, the angular distance from the center of the contact, and the dielectric permittivity, respectively. The result of the integration is:

$$C = 2\pi\epsilon R\left[\log\frac{R}{z_o} + O(1)\right]$$

where $O(1)$ is a number of order 1 that depends on the

(23) Broekman, L.; Artsyukhovich, A.; Frantz, P.; Salmeron, M. To be submitted for publication.
(24) Parker, J. L.; Christenson, H. K.; Ninham, B. W. *Rev. Sci. Instrum.* **1989**, *60*, 3135.
(25) Tonck, A.; Georges, J. M.; Loubet, J. L. *J. Colloid Interface Sci.* **1988**, *126*, 150.
(26) Fortney, L. R. *Principles of Electromagnetics: Analog and Digital*; Harcourt Brace Jovanovich Publishers: Orlando, FL, 1987.
(27) Van Alsten, J.; Granick, S. *Phys. Rev Lett.* **1988**, *6*, 2570.
(28) Boyer, C.; Houze, F.; Tonck, A.; Loubet, J.-C.; Georges, J. M. *J. Phys. D* **1994**, *27*, 1504.

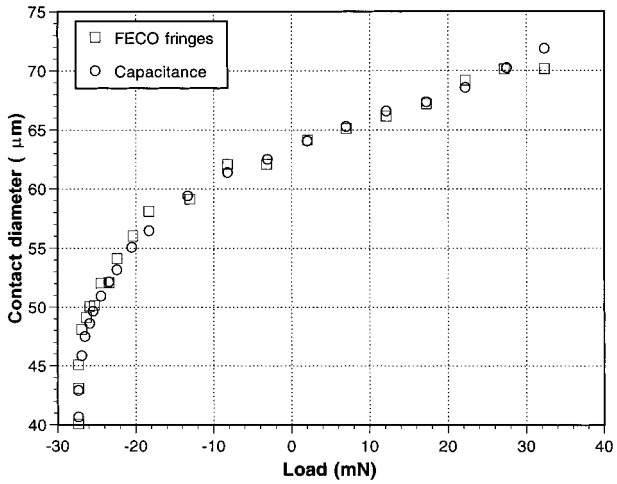

Figure 3. Contact diameter as a function of applied load measurements to compare capacitance and FECO method results. Squares represent optical measurements using the FECO method. Circles represent the square root of the measured capacitance, scaled to fit. Both measurements were performed simultaneously.

actual shape and extent of the electrodes far from the point of closest approach. This constant disappears in any application where only changes in C are measured. For example, the separation z_0 is linear in the inverse of the derivative of capacitance:

$$z_o = -\frac{2\pi\epsilon R}{(\partial C/\partial z)}$$

as shown in our previous paper.[21]

When a flat circular area is produced after elastic deformation due to contact, the capacitance can be decomposed into the contribution of the flat contact area, plus the contribution of the curved surfaces outside the contact. Using the same procedure as described above, this last contribution is easily calculated (again in the limit of $d/R \ll 1$) by integration, this time starting at θ_0 (the angle subtended by the contact radius a from the center of the sphere). The result is

$$C = C_{in} + C_{out} = \frac{\epsilon_{in}\pi a^2}{d} + 2\pi R\epsilon_{out}\left[\log\frac{R}{d} + O(1)\right]$$

where d is the distance of separation between the silver electrodes. We have assumed that the dielectric constant might be different inside and outside the contact area. Notice that the contribution from areas outside the contact is constant in the $d/R \ll 1$ approximation. Although in absolute terms, C_{out} is much larger than C_{in} (by about a factor of 10^3-10^4), the excellent sensitivity ($\delta C \approx 0.4$ fF) and low noise level in the measurement of C makes the determination of the changes in contact area perfectly feasible. For a contact of 60 μm diameter of mica sheets 1 μm thick, the capacitance contributed by the contact area is ~1 pF. Thus an area sensitivity better than 0.1% is achieved with this technique.

A comparison of the contact diameter as a function of applied load (during separation), as measured by the FECO method, and the square root of the capacitance (in arbitrary units), is shown in Figure 3. Both data were acquired simultaneously. We find that, after scaling the capacitance data, the results of the two methods of area measurement are almost indistinguishable from one another. However, although it is not shown in the figure, the optical data is significantly noisier than the capacitance data.

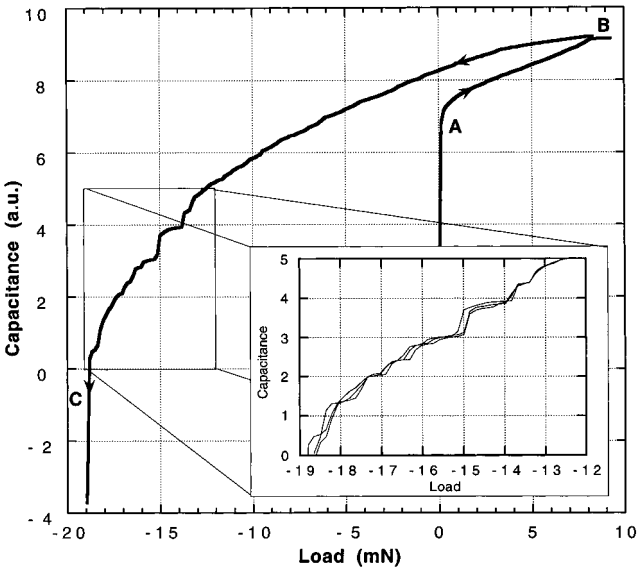

Figure 4. Capacitance data showing the changes in contact area with applied load as two mica surfaces are compressed (AB) and separated (BC) in a dry nitrogen atmosphere. The lower surface was supported by a double cantilever with spring constant of 320 N/m. The load was applied by attaching a magnet to cantilever and placing SFA in a homogeneous magnetic field. The load was cycled three times on the same contact, consuming a time of 2 min for all three cycles (shown in the inset).

Results

In this section we demonstrate the capabilities of this technique by applying it to a few examples where high precision in the measurement of the contact area is important. These include the dependence of contact area on applied forces, in both the normal and tangential directions.

Contact Area as a Function of Applied Load. The measurement of the change in area as a contact is loaded and unloaded is important in order to determine the adhesion energy. Figure 4 shows the data from an experiment where the measured capacitance is plotted against the normal force applied to the surfaces for a single load−unload cycle. Fresh mica substrates were prepared by cleaving the mica after it had been glued to the silica lens. A piece of adhesive tape was used to remove the top layer of mica, while all parts were enclosed in a nitrogen-purged atmosphere. Thus, the surface was never exposed to laboratory air. The load cycle began at point A where the surfaces had jumped into contact under the influence of their interfacial forces. The capacitance then grew with surface area, as the applied load was continuously increased. At point B, the load was reversed and the separation began. As the tensile force applied to the contact to overcome the adhesive forces was increased, we found that the separation proceeded in discrete steps instead of a continuous release, as shown in more detail in the inset of Figure 4. Finally, the surfaces jumped out of contact at point C. This cycle was repeated two more times, as shown in the inset of Figure 4, consuming a total of 5 min.

Small jumps in contact area during loading and unloading cycles were noticed in early experiments with mica cleavage.[29] Similar phenomena, sometimes called stick−slip adhesion[30] or peeling instability, are now scrutinized in adhesive tape peeling experiments.[31] It was shown that, depending on the pulling speed, the

(29) Deryagin, B. V.; Metsik, M. S. *Soviet Phys.-Solid State* **1960**, *1*, 1393.
(30) Israelachvili, J. N. *J. Vac. Sci. Technol., A* **1992**, *10*, 2901.
(31) Hong, D. C.; Yue, S. *Phys. Rev. Lett.* **1995**, *74*, 254.

system can exhibit irregular bifurcation of peeling speed, periodic stick−slip behavior, or, at high pulling speed, can enter a dynamic chaos regime. Although the jumps in the area of contact for dry mica during loading and unloading cycles appear similar to peeling instability, the fact that the sequence of jumps is reproducible in consecutive cycles, as shown in the inset of Figure 4, eliminates peeling instability as a cause.

We surmise that the steps are due to some domain structure of the mica surface, probably patches of different surface potential produced by cleavage, because we find that the steps appear at the same contact positions throughout repeated cycles (see inset of Figure 4). They appear at different values of applied load when the contact is moved to a different location. Also, they are more pronounced the cleaner the mica; step size and frequency are diminished when the surfaces are exposed to laboratory air for extended periods (several hours) and steps are completely absent when the atmosphere in the chamber is saturated with water or organic vapors (hexadecane, undecane).

It was long observed[29] that cleavage of mica in vacuum or dry atmosphere leaves complementary domains of the opposite charge on its surfaces. The positive charge is due to K^+ ions randomly shared between two surfaces.[32] The corresponding negative charge is supplied by negative Al ions in tetrahedral Si sites under the top surface oxygen layer. Cleavage results in electrostatic attraction of the cleaved surfaces, far stronger than van-der-Waals attraction.[32] A very wide range of domain sizes has been reported: ∼1 nm;[32] ∼1 mm;[29,32,33] <9 nm.[34] The jumps in contact area observed in our experiments correspond to charge domain sizes on the order of 10 μm. Although this length does not match the other measurements, it is possible that there could be multiple length scales if a domain superstructure exists.[32]

In another experiment we have assembled a monolayer of (3-aminopropyl)triethoxysilane in water on both mica surfaces. The surfaces were then rinsed in water and dried out in N_2. In contrast to the clean mica case, no steps were observed in loading and unloading cycles with these chemically modified surfaces. This observation again eliminates peeling instability as a cause of observed jumps, since a 0.9 nm self-assembled monolayer does not change significantly the elastic response of the system.

It has been argued[16,35] that the radius of the contact between the two mica surfaces should behave as predicted by the JKR relationship

$$a^3 = \frac{R}{K}[F + 6\pi R\gamma + \sqrt{12\pi R\gamma F + (6\pi R\gamma)^2}]$$

Since there are typically two unknown parameters in this relationship—K, the elastic modulus, and γ, the adhesion energy—it is necessary to fit both variables simultaneously. However, it is difficult to decide how such a model should be fitted to curves such as those in Figure 4. Since the origin of the steps is not known with certainty, it is not possible to prescribe the location along each plateau at which the fitted curve should cross. This uncertainty introduces substantial error into the analysis.

To evade these uncertainties, we performed a new experiment that eliminated the spontaneous jumps in separation. This was done by replacing the flexible double-cantilever leaf springs with a rigid stainless steel lever, with effective spring constant of ∼2 × 10^5 N/m. The piezoelectric tube to which one mica sample was attached (see Figure 1) was used to bring the surfaces into contact and to apply a load. The results are shown in Figure 5. The thin white line corresponds to the actual data during the unload cycle. The curve can be precisely fitted by the JKR formula, although one must bear in mind that the large spring constant of the rigid lever introduces a larger error in the absolute force measurement. The solid curve is a least-squares fit to the data, using K and γ as parameters with values of 10.4 GPa and 413 mJ/m^2. Excellent agreement between the data and the JKR expression is obtained under the conditions of this experiment. Once in contact, a change of normal load of 6 mN produced an increase of contact diameter from 56 to 92 μm. The corresponding change in capacitance was from 30 to 120 fF. It is worth noting here that, for experiments done in dry N_2 atmosphere, the adhesion of dry mica ranged between 300 and 500 mJ/m^2, much higher than the 60−150 mJ/m^2 range obtained for mica cleaved in laboratory air.[17]

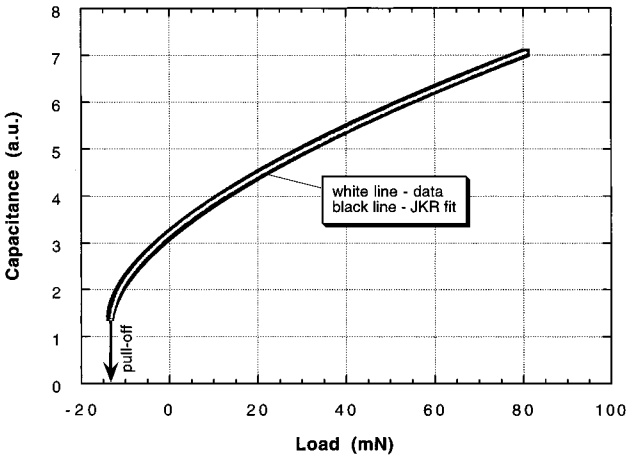

Figure 5. Capacitance data showing the changes in contact area with applied load as two mica surfaces are compressed and separated in a dry nitrogen atmosphere. The lower surface was supported by a stiff lever with spring constant of 2 × 10^5 N/m. The load was applied through expansion of a piezoelectric tube supporting one of the mica samples.

Adhesion Hysteresis. Adhesion hysteresis has recently been discussed as a possible explanation for the relationship between adhesion and friction, which our technique is able to investigate in detail. Adhesion hysteresis results primarily from nonequilibrium processes such as physicochemical reconstruction of the interface, as bonds or entanglements are formed, or viscoelastic bulk deformations when the contacting materials are not perfectly elastic.[1,9,19]

In addition to measuring the magnitude of hysteresis, it has been recognized that the time scales for these processes may also be of interest.[19] With our technique, hysteresis loops may be obtained by cycling the load up and down and avoiding the jump out of contact. One such experiment is shown in Figure 6, corresponding to a mica/mica contact, this time prepared by cleavage of the mica in laboratory air. The hysteresis is sufficently small that it would have been difficult to observe with the noisier and slower FECO method.[15] Here, as in Figure 4, the capacitance (∝contact area) is plotted against the cyclic load. The next unloading segment was continued (not shown in Figure 6) until the surfaces were completely separated to provide a measurement of the pull-off force. This unloading segment was calibrated using the FECO method and a JKR fit was done to evaluate an effective Young modulus of contact. With this information, two values of adhesion were calculated for the retraction

(32) Wan, K.-T.; Smith, D. T.; Lawn, B. R. *J. Am. Ceram. Soc.* **1992**, *75*, 667.
(33) McGuiggan, P. M; Israelachvili, J. N. *J. Mater. Res.* **1990**, *5*, 2232.
(34) Gerlach, R.; Polanski, G.; Rubahn, H.-G. *Surf. Sci.* **1996**, *352−354*, 485.
(35) Christenson, H. K. *Langmuir* **1996**, *12*, 1404.

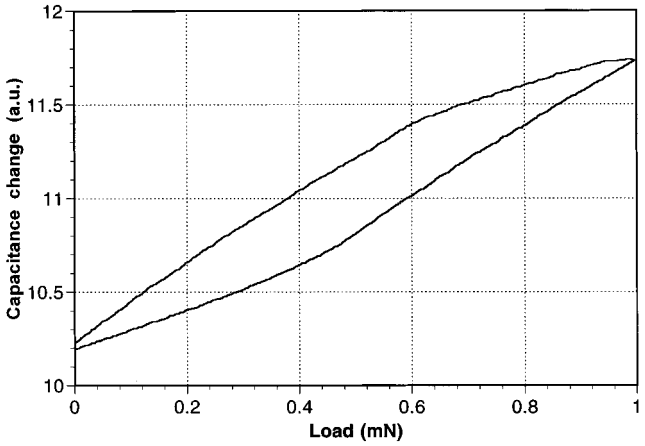

Figure 6. Hysteresis loop in the contact area (capacitance change) of mica surfaces cleaved in air, as the normal force is cyclically loaded and unloaded without pulling the surfaces out of contact.

(upper) and approach (lower) parts of adhesion loop in Figure 6. On approach, the adhesion energy is 184.7 mJ/m^2 and, on separation, 185.2 mJ/m^2. As pointed out above, one should notice the overall smaller value of γ, compared with that obtained previously (413 mJ/m^2), due to preparation in dry N_2 atmosphere.

For a perfectly elastic contact of two crystalline surfaces, such as the mica used in this experiment, there should be no adhesion hysteresis.[1] The small amount of hysteresis observed in this experiment is probably due to inelastic deformation of the glue, particularly near the edges of the contact. No energy was lost to the jump into contact, since these surfaces had been in contact for several minutes before initiation of the load cycle.

Dependence of Contact Area on the Presence of Lateral Forces. It is known that the results of the JKR theory for an adhesive contact of sphere on a plane can be explained in terms of radial crack propagation and fracture dynamics.[22] It is thus reasonable to inquire whether applying tangential load would influence crack propagation. There is experimental evidence[36] that the fracture dynamics of this kind of contact is influenced by tangential load. In an attempt to describe the interdependence between adhesion and friction, Johnson derived a formula for general stress release (G_c) in a mixed fracture mode, expressed through both adhesion stress release ($[G_c]_a$) and friction stress release ($[G_c]_f$) as:[22]

$$G_c = \sqrt{\{[G_c]_a + [G_c]_f\}^2 - 2\alpha[G_c]_a[G_c]_f}$$

with α as an interaction factor. We assume that $\alpha > 0$ for an interaction between adhesion and friction to exist. At $\alpha = 0$, the contact area follows regular JKR dependence on normal load and does not depend on tangential load. The interaction factor α can be determined experimentally from the decrease of the adhesive contact area with an increase in tangential load applied.[22]

To make that determination, the dependence of contact area ($\propto$capacitance) on tangential force (0−32 mN) applied to one contact lens along its axis was studied at different normal loads (from +3 to −3 mN). At no time during these experiments were the applied tangential forces large enough to overcome the critical shear stress between the mica surfaces. No changes in the contact area greater than 1% ($\Delta C \sim 1$ fF) were ever measured, which is consistent with a lack of change in surface area detected by the FECO method.[37] This result allows us to estimate an upper limit of the value of α for dry mica. We have made such an estimate for our experimental conditions. The elastic properties of contact were mainly ascribed to the thick ($\sim$100 μm) epoxy layer underneath the thin mica. Thus we have used the values of Young's modulus $E = 9$ GPa and Poisson ratio $\nu = 0.34$. By interpolation of the results reported in ref 22 to the values of dimensionless static friction force and normal pressure used in our experiment a decrease in area of less than 1% would correspond to $\alpha < 0.03$.

The common mechanism of the separation of surfaces out of adhesive contact is by "crack" propagation.[32] Because of the existence of the adhesion domains, the crack propagation in dry mica contacts is "erratic and irreproducible".[32] It is the existence of domains in adhesion of dry mica that has limited our ability to follow the changes in contact area caused by tangential stress to a 1% accuracy, despite the much better sensitivity of the capacitance technique. Another possible reason for the poor reproducibility of the tangential load experiments could be the micro damage (tearing) of mica caused by tangential stress as a consequence of the unusually high adhesion of dry mica in N_2 atmosphere. For future experiments, it seems reasonable to repeat contact area *vs* tangential load measurements with surfaces that have comparatively lower adhesion and do not exhibit domains in adhesion.

Summary

It has been shown that the capacitance method can give measurements of the contact area with unprecedented speed and resolution. A 0.1% accuracy of the contact area measurements could be achieved with a 1 ms temporal resolution.

Discrete jumps in the area of contact were observed during loading/unloading cycles with very clean mica in dry N_2 environments. The reproducibility of the jumps in consecutive cycles argues for the existence of adhesion domains due to electrostatic surface potential patches as the probable cause. The characteristic size of the observed domains in dry mica adhesion is $\sim$10 μm.

The adhesion of mica cleaved in laboratory air exhibited very small hysteresis: 184.7 mJ/m^2 on approach and 185.2 mJ/m^2 on retraction. No area changes larger than 1% of the total were detected under the application of lateral forces (0−32 mN) to dry mica contacts in the normal load range of +3 to −3 mN. This observation implies that the interaction factor α in Johnson's model,[22] representing the correlation between adhesion and friction, should be less than 0.03 for dry mica contacts.

Acknowledgment. This work is supported by the Lawrence Berkeley National Laboratory through the Director, Office of Energy Research, Basic Energy Science, Materials Science Division of the U.S. Department of Energy under Contract DE-AC03-76SF00098. R.W.C. acknowledges support from the Natural Sciences and Engineering Research Council of Canada.

LA9702650

(36) Savkoor, A. R.; Briggs, G. A. D. *Proc. R. Soc. London, Sect. A* **1977**, *356*, 103.

(37) Yoshizawa, H.; Chen, Y.-L.; Israelachvili, J. N. *J. Phys. Chem.* **1993**, *97*, 4128.