Atomic force microscope–force mapping and profiling on a sub 100-Å scale

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A modified version of the atomic force microscope is introduced that enables a precise measurement of the force between a tip and a sample over a tip-sample distance range of 30–150 Å. As an application, the force signal is used to maintain the tip-sample spacing constant, so that profiling can be achieved with a spatial resolution of 50 Å. A second scheme allows the simultaneous measurement of force and surface profile; this scheme has been used to obtain material-dependent information from surfaces of electronic materials.

I. INTRODUCTION: FORCE-SENSING ULTRAMICROSCOPY

Force measurement between surfaces separated by a fraction of a micron[1,2] and ultramicroscopy on the scale of a few angstroms[3,4] are both fields of active research. Recently, the atomic force microscope[5] as well as other methods[6,8] derived from tunneling microscopy[3] have combined force measurement with high spatial resolution. This opens the door to material examination involving a force type of interaction, and on a scale about three orders of magnitude smaller than typical optical resolution. All these techniques have a common thread: the force is sensed between an object and a fine tip.

We present a new technique for accurate measurement of the force between a tip and a material, as a function of the spacing between the tip and the material surface (Secs. II and III). It features a tip that is vibrated at close proximity to a surface, and optical heterodyne detection to accurately measure the vibration of the tip. This combination proves to be sensitive and flexible. It enables one to measure tip displacements over large distances and over a wide range of frequencies, which is a major advantage over the previous methods. As a result, the technique is applicable to noncontact profiling of electronic components on scales varying from tens of microns to a few tens of angstroms (Sec. IV). A second application is introduced, where material sensing and surface profiling are achieved simultaneously (Sec. V).

II. DESCRIPTION OF THE TECHNIQUE

An overall diagram of the technique is shown in Fig. 1. A tungsten tip at the end of a wire is mounted on a piezoelectric transducer. The transducer vibrates the tip at the resonance frequency of the wire, which acts as a cantilever. A laser heterodyne interferometer[9,10] accurately measures the amplitude of the ac vibration. Two modes of operation are considered:

(1) The gradient of the force between the tip and sample modifies the compliance of the lever, hence inducing a change in vibration amplitude due to the shift of the lever resonance. Knowing the lever characteristics, one can measure the vibration amplitude as a function of the tip-sample spacing in order to deduce the gradient of the force, and thus, deduce the force itself. The vibration amplitude of the lever also provides a feedback signal that allows the tip-sample spacing to be held constant for profiling applications.

(2) The long-range force between the tip and the sample is an attractive force, of the van der Waals type. For a lever having a small "spring constant," the force gradient of the interaction can be greater than the spring constant. When the tip is moved continuously toward the surface, the tip is suddenly attracted to the sample and comes to equilibrium at a position close to the potential minimum of the interaction. The force necessary to pull the tip away from the surface can then be measured to derive the peak strength of the attractive force, and thus to characterize the sample material.

Some advantages of optical detection over tunneling detection of the lever deflection have been clearly identified by McClelland *et al.*[8] It is more reliable and easier to implement, it is insensitive to the roughness of the lever, and it has a smaller sensitivity to thermal drifts. The laser interferometric probe used is described in Ref. 10. It can measure the amplitude and phase of ac displacements as small as 10^{-4} Å. In addition, it is totally insensitive to thermal drifts in the optics, an attribute of the heterodyne technique. Hence, the distance between the lever and the optical probe need not be accurately controlled. Furthermore, the optical detection scheme enables one to monitor the vibration of the lever even when it is excited into vibrations having amplitudes of several hundreds of angströms, which would prove extremely difficult by tunneling techniques.

III. FORCE MEASUREMENT AS A FUNCTION OF THE TIP-SAMPLE SPACING

The technique is run in the first mode of operation in order to map the force as a function of the tip-sample spacing d, varying between a few angstroms and a few hundreds of angstroms. The tip is vibrated at the lever resonance frequency and the vibration amplitude is recorded as a function of d. For large values of d, where the effect of the sample on the tip can be neglected, the tip vibration amplitude A as a function of the frequency ω is a Lorentzian of the form:

$$A = \frac{A_0(\omega_0/\omega)}{\sqrt{1 + Q^2(\omega/\omega_0 - \omega_0/\omega)^2}}, \tag{1}$$

where $\omega_0 = c\sqrt{k}$ is the resonance frequency, c is a function of the lever mass, k is the spring constant, A_0 is the amplitude at resonance, and Q is the quality factor ($Q \gg 1$). As the tip approaches the sample, van der Waals forces between sample and tip have to be taken into account. For small vibrational amplitudes, they cause an additional spring-type force

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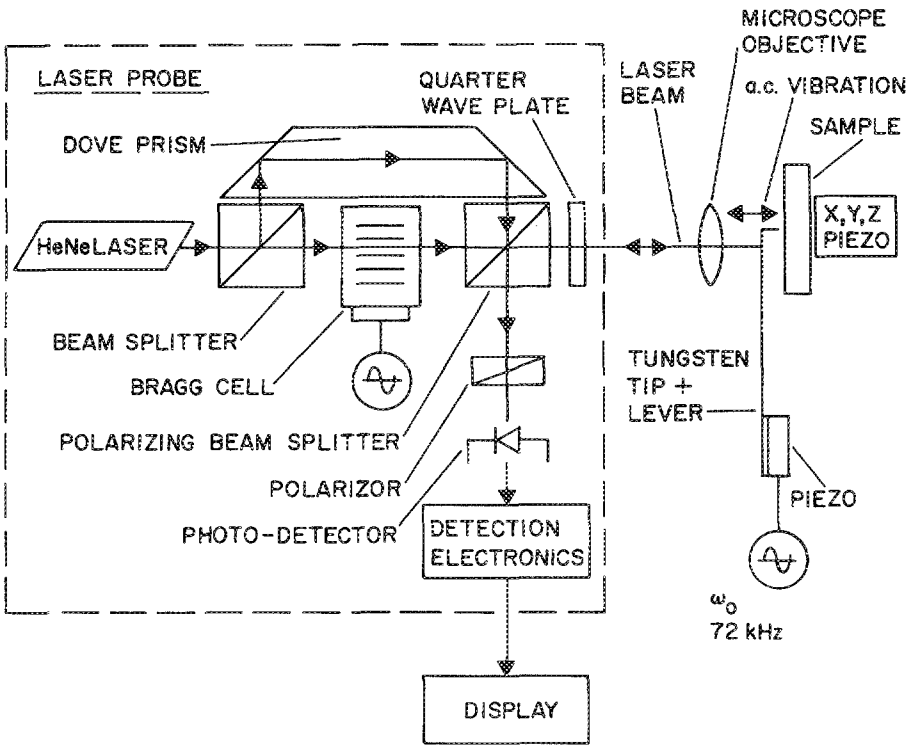

FIG. 1. Experimental setup for force derivative measurement as a function of tip-sample spacing.

f. Its derivative along the normal to the surface f' reduces the spring constant k, and shifts the resonance frequency to ω_0', given by $\omega_0' = c\sqrt{k - f'}$. The minus sign comes from the fact that f is increasingly attractive as the tip approaches the surface; thus, a decrease of the resonance frequency is observed. If we drive the lever at ω_0, we can measure the amplitude A' due to this effect and use Eq. (1) to calculate the new resonance ω_0'. From this, we derive

$$f' = k\left(\frac{1 - 2a^2 + \sqrt{4Q^2(a^2 - 1) + 1}}{2(Q^2 - a^2)}\right), \tag{2}$$

where $a = A_0/A'$. f is obtained by integrating f'. In this calculation, we make two assumptions. First, we suppose that the tip-surface interaction induces a frequency shift of the resonance, and not an energy transfer that would decrease the vibration amplitude. One can verify this assumption by vibrating the tip somewhat below its natural resonance. At a particular tip-sample spacing, the resonance peak is found again with a normalized amplitude of one. This verification also proves that the force between the tip and sample is increasingly attractive when the tip approaches the sample, with distances d varying between a few hundreds of angstroms and a few angstroms. Second, we assume that f' does not significantly affect the vibration mode of the lever, so that the equation $\omega_0' = c\sqrt{k - f'}$ is valid. From the previous test, and for tip-sample spacings where $f' \ll k$, we believe that the vibration mode is essentially unaffected since the amplitude at resonance is unchanged.

A. Minimum detectable f'

The basic measurement scheme described in this paper measures a force derivative f' by deriving a shift in the lever resonance frequency $\Delta\omega = \omega_0' - \omega_0$ from a change in the lever vibration amplitude driven at a constant frequency (in the previous calculation ω_0). The experiments described in this paper were all conducted in this mode. Clearly, in order to get the biggest change in lever vibration for a given change in resonance frequency $\Delta\omega$, one would work on the steepest portion of the A vs ω curve (Eq. 1). This is *not* at $\omega = \omega_0$. In the limit of a large Q, the maximum slope of the A vs ω curve occurs at $\omega_m \simeq \omega_0(1 \pm 1/\sqrt{8}Q)$ and at this point on the curve,

$$\frac{\partial A}{\partial \omega} = \frac{4A_0Q}{3\sqrt{3}\omega_0}.$$

A small f', i.e., $f' \ll k$, produces a frequency shift $\Delta\omega = \omega_0 f'/2k$, and thus an amplitude change ΔA given by

$$\Delta A = (2A_0Q/3\sqrt{3}k)f'. \tag{3}$$

The noise sources are optical and thermal. The optical noise is induced by the photons of the laser probe bouncing on the lever. Its effect can be neglected; the noise force due to a 1 mW beam is of the order of 10^{-19} N. The thermal noise brings a more serious limitation. The thermal energy excites the lever with a vibration of amplitude $A_T = \sqrt{2KT/k}$ and a vibration noise $N = \sqrt{4KTQB/k\omega_m}$, at a frequency ω_m and in a bandwidth B.[7,8] The smallest detectable force derivative, f'_m, is found by setting $\Delta A = N$ (and with the approximation $\omega_0 \simeq \omega_m$):

$$f'_m = \frac{1}{A_0}\sqrt{\frac{27kKTB}{Q\omega_0}}, \tag{4}$$

which is comparable to the value found previously.[8] Several parameters can be optimized in order to maximize the sensitivity: (a) Decreasing the lever stiffness k: the lower bound is limited by the maximum tolerable thermal noise vibration A_T, which varies as $k^{-1/2}$. (b) Increasing the quality factor Q: Q depends strongly on the quality of the mechanical con-

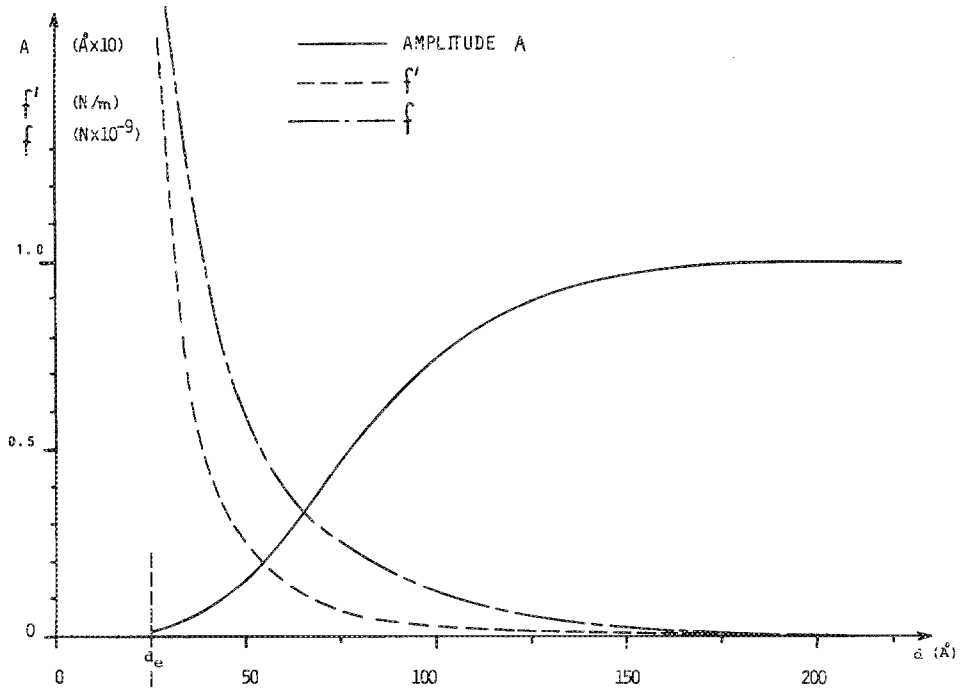

FIG. 2. Magnitude of the force derivative f' and force f between the sample and the tip, calculated from the measured vibration amplitude A as a function of the tip-sample spacing d; the force is attractive.

nection between the lever and the piezoelectric transducer. A value of 190 has been obtained experimentally. (c) Increasing the vibration amplitude A_0 and the natural resonance frequency ω_0: a main advantage of this technique over the previous experiment[8] is that one can increase A_0 and ω_0 to comparatively large values, since we only vibrate the small tip and not the while sample.

The size of the smallest detectable f' can be estimated with the following parameters: in order to obtain a small value of k and a large value of ω_0 simultaneously, one must resort to the smallest lever that could possibly be produced by micromechanics. A silicon lever, with dimensions $2\times0.5\times0.1$ μm, has a stiffness constant of 2.5 N/m, which corresponds to an acceptable thermal vibration of 0.6 Å, and a resonance frequency of 33 MHz. Considering a vibration amplitude A_0 of 10 Å and a quality factory Q of 200, minimum detectable force derivative f'_m in a bandwidth of 1 Hz is

$$f'_m = 3\times10^{-6}\text{N/m},$$

a value that is at least two orders of magnitude smaller than the values attained previously.[7,8]

B. Experimental results

The experiment was aimed at measuring the attractive force between a tungsten tip and a surface (silicon) over a wide range of tip-sample spacings. The lever/tip is made out of a tungsten rod, etched into a cone, having a length of 460 μ, a base diameter of 15 μ, and a final tip diameter of 0.1 μ. The last 40 μ of the cone are bent at 90°. The spring constant k, resonant frequency ω_0, and Q factor of the lever were 7.5 N/m, 72 kHz, and 190, respectively. For this tip, the calculated minimum detectable f' in a bandwidth of 1 Hz is $f'_m = 10^{-4}$ N/m. The experimental value was obtained by frequency modulating the driving signal (ω_0) by an amount $\delta\omega$. The smallest detectable frequency change $\delta\omega$ is then deduced from the smallest detectable amplitude change δA. The resulting experimental f'_m, given by $f'_m = 2k\delta\omega/\omega_0$ in the approximation of a large Q, was $f'_m = 1.5\times10^{-4}$ N/m. The smallest detectable force, or force change, depends on the shape of the curve f vs d, since the technique measures force gradients and not force. In the following situation, a force of the order of 10^{-11}N is measured. However, at tip-sample spacings of about 50 Å, force increments as small as 3×10^{-13} N can be detected.

Figure 2 shows the experimental curve of the amplitude A versus the tip-sample spacing d, when the tip is moved at constant speed toward the surface. The duration of the measurement is kept short, about 1 s, in order to minimize the thermal drifts. On the same axes we have computed the values of f' and f, deduced from A. f' varies from 0.01 to 2 N/m, while f varies from 10^{-11} to 1.5×10^{-9} N when the tip is brought from 180 to about 30 Å. The precision in the midrange is estimated to be $\pm$ 2%.

The origin on the x axis has been determined approximately in the following way: at the lower values of d, the experimental measurement ends at d_e, when $f'\simeq k$. The tip is then suddenly attracted to the surface by van der Waals forces, since the gradient f' is larger than the spring constant of the lever k. One then moves the lever by a positive distance D_v in order to apply a force $F_v = kD_v$ just large enough to pull the tip away from the surface. In the approximation of a narrow attractive potential well ($d_e \ll D_v$), F_v corresponds to the peak van der Waals attractive force. Experimentally, one finds $D_v = 860$ Å, and hence, $F_v = 6.4\times10^{-7}$ N. By extrapolating the values of f, plotted on a semilogarithmic scale to small values of d, one chooses the origin of the x axis at the point where the f curve reaches F_v. This corresponds to $d_e = 25 \pm 5$ Å.

The capability of the technique can be extended in two ways:

(1) In our experiment, the lever was driven at the resonant frequency ω_0. However, it could be any other frequen-

cy, ω_d, so we now have another parameter available. Particularly, one could choose ω_d so that the sensitivity of the measurement (and thus the accuracy) is maximum at any chosen point for A and f' on the curves of Fig. 2. The sensitivity is greatest when $\partial A/\partial f'$ is maximum, with A and ω_0' given by

$$A = A_0 \frac{\omega_0'}{\omega_d}\left[1 + Q^2\left(\frac{\omega_d}{\omega_0'} - \frac{\omega_0'}{\omega_d}\right)^2\right]^{-1/2}$$

and

$$\omega_0' = \omega_0\sqrt{1 - f'/k}$$

from previous relations. In the limit of a large Q, this occurs at

$$\omega_m' = \omega_0(1 \pm \sqrt{8}Q)\sqrt{1 - f'/k}\,.$$

By choosing $\omega_d = \omega_m'$, the change of amplitude δA for a small change of f' and $\delta f'$ is maximized and has the value

$$\delta A = \frac{\partial A}{\partial f'}\delta f' = \frac{2A_0 Q}{3\sqrt{3}(k - f')}\delta f'.$$

The minimum detectable $\delta f'$ is found by setting $\delta A = \mathrm{N}$. From this value of $\delta f'$, the signal-to-noise ratio which characterizes the accuracy with which one can measure f' is given by

$$\frac{\mathrm{S}}{\mathrm{N}} = \frac{f'}{\delta f'} = \frac{A_0 f'}{1 - f'/k}\sqrt{\frac{Q\omega_0}{27kKTB}}. \tag{5}$$

With the experimental tip, a precision of 0.1% can be obtained in a bandwidth of 1 Hz, for f' greater than 0.1 N/m.

(2) By using stiffer and weaker levers (that one can obtain by varying the lever length), it should be possible to precisely map f' and f over the distances $d < 30$ Å and $d > 180$ Å, respectively. These distances were not attained during this first experiment. We suggest this technique as a new method for mapping the force between a given tip and a surface over tip-surface spacings varying between 1 Å and several hundreds of angstroms, and with a precision of about 0.1%.

IV. PROFILING ON THE NANOMETER SCALE

Since the amplitude of vibration depends on the tip-surface spacing, one can use this signal in a feedback loop to maintain the tip at a given distance from the surface and scan the tip over the object in a manner similar to the scanning tunneling microscope (STM) or, more recently, to the thermal profiler.[11] The feedback signal applied to the piezoelectric transducer of the lever moves the tip in the direction normal to the surface in order to follow the profile of the surface. It is recorded as a function of the tip position, and displayed either in the form of line scans or of grey scale images.

Figure 3(a) displays the profile obtained over V-shaped grooves on a silicon wafer. The shape and size of the grooves agrees with the scanning electron micrograph of the same wafer, shown in Fig. 3(b). The time necessary to acquire the picture was about 2 min. The small ripples seen on the top and on the sides are real. They can be resolved into small steps composed of several atomic layers that are the result of the fabrication process (anisotropic etching). One of these

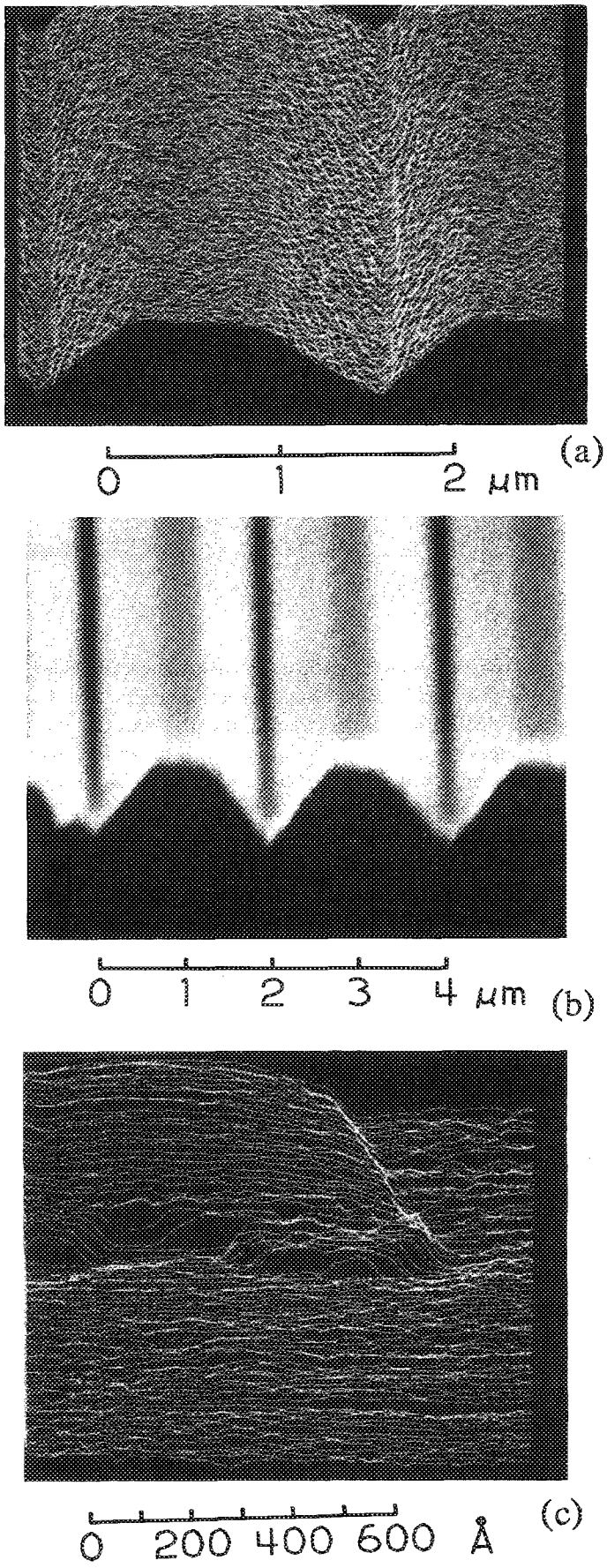

FIG. 3. (a) Profile over V-shaped grooves on a silicon wafer; (b) corresponding SEM image. (c) High-resolution profile of a small area on top of the V-groove; the step that is imaged has a height of 50 Å; the achieved spatial resolution is 50 Å.

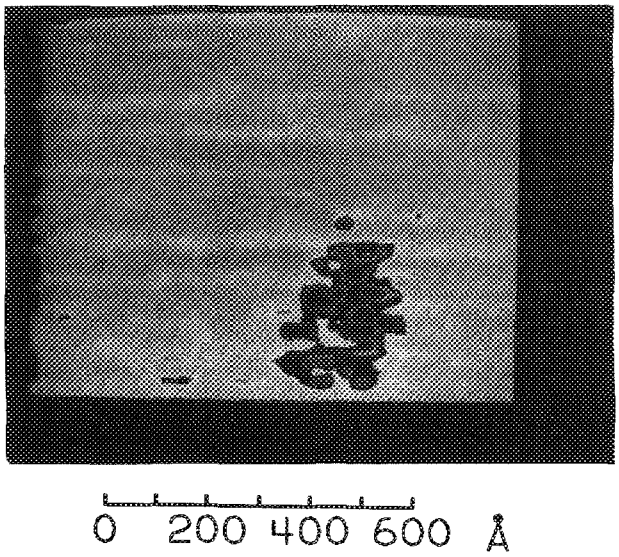

FIG. 4. Image of some contaminant on an atomically flat surface of graphite.

steps, shown in Fig. 3(c), is about 50 Å high. Some structures are observed, near the step, that are smaller than 100 Å. The steepest signal rise on the step is narrower than 20 Å. Another example is shown in Fig. 4, where some contaminant is detected on an atomically flat graphite surface. Features on the order of 100 Å are imaged in this example. In both cases, the tip-sample spacing was kept at approximately 50 Å. The present vertical stability amounts to about 10 Å. We expect to reduce this to within 1 Å with a better vibration isolation.

In this type of near-field microscopy, the spatial resolution is governed by the tip-sample spacing, i.e., 50 Å in this experiment. By using a stiffer lever in order to operate at closer distances to the sample, and still relying on the attractive van der Waal's force, one should achieve a spatial resolution better than 10 Å. With the present tip and with a very stiff lever corresponding to about 10^2 N/m, one could hope

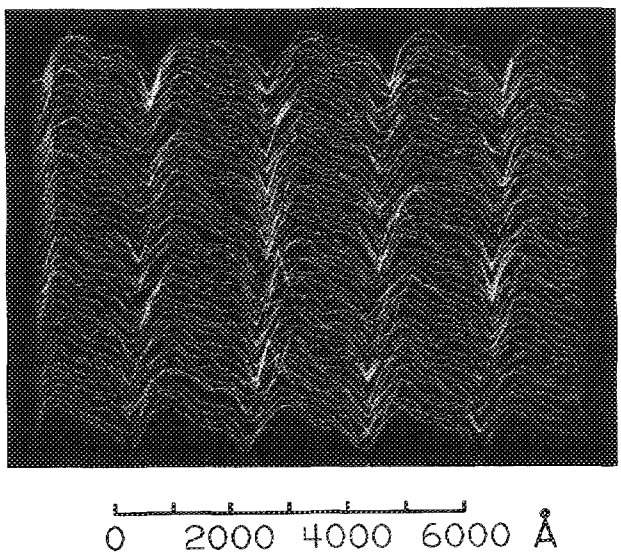

FIG. 5. Profile of a photoresist grating; linewidth = line spacing = 1000 Å, photoresist thickness = 900 Å; the strong difference between linewidths and line spacings results from the finite width of the tip ($\simeq$1000 Å).

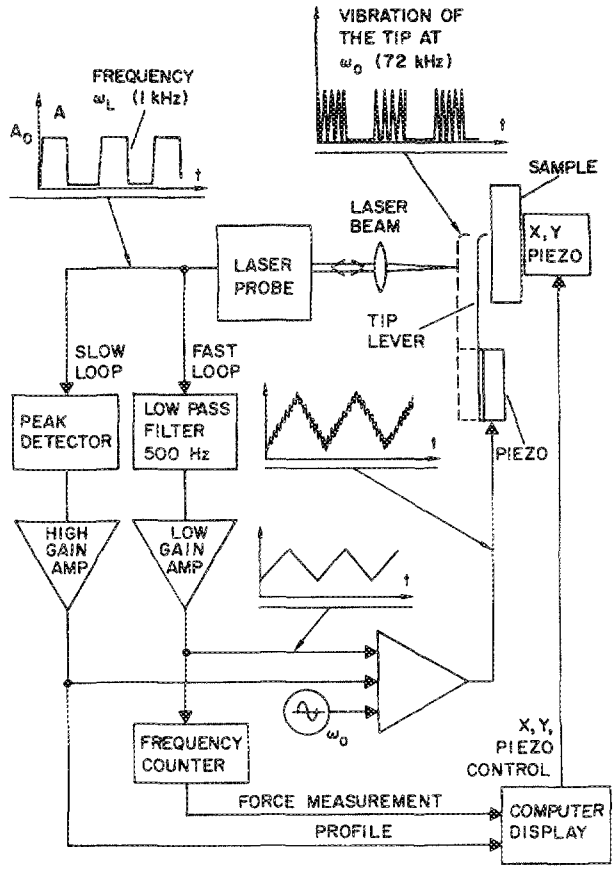

FIG. 6. Experimental setup for simultaneous mapping of the peak van der Waals force and surface profile.

to sense the strong electronic repulsive force at tip-sample distances within 1 or 2 Å from the surface, and thereby aim to achieve atomic resolution.

Another attractive feature of force microscopy lies in its applicability to the inspection of nonconducting surfaces. Figure 5 displays the profile of a photoresist grating deposited on silicon. The periodicity of the grating was 2000 Å and the thickness 900 Å. Small structures at the top of the lines are observable. However, the grooves themselves show no detail and appear narrower, since the tip end has a diameter comparable to the width of the grooves. A tip thinner than 500 Å over a length of 1000 Å or more at its end would enable one to resolve most of the structure at the bottom of the grooves.

V. MATERIAL SENSING AND IMAGING

Experiments have shown that the van der Waals force F_v defined in Sec. III varies significantly with the nature of the sample material. With the same tungsten tip, F_v was, respectively, 2.7×10^{-7}, 3.1×10^{-7}, and 1.8×10^{-6}N, on photoresist, silicon (nonetched), and graphite. Hence, by measuring F_v at every point on a sample, one can characterize the sample material with high spatial resolution.

We use the second mode of operation in this paper: the tip is vibrated at resonant frequency ω_0, with an amplitude A_0 of 3 Å, and is repeatedly moved toward and then away from the surface, at a low frequency of about 1 kHz, ω_1, over

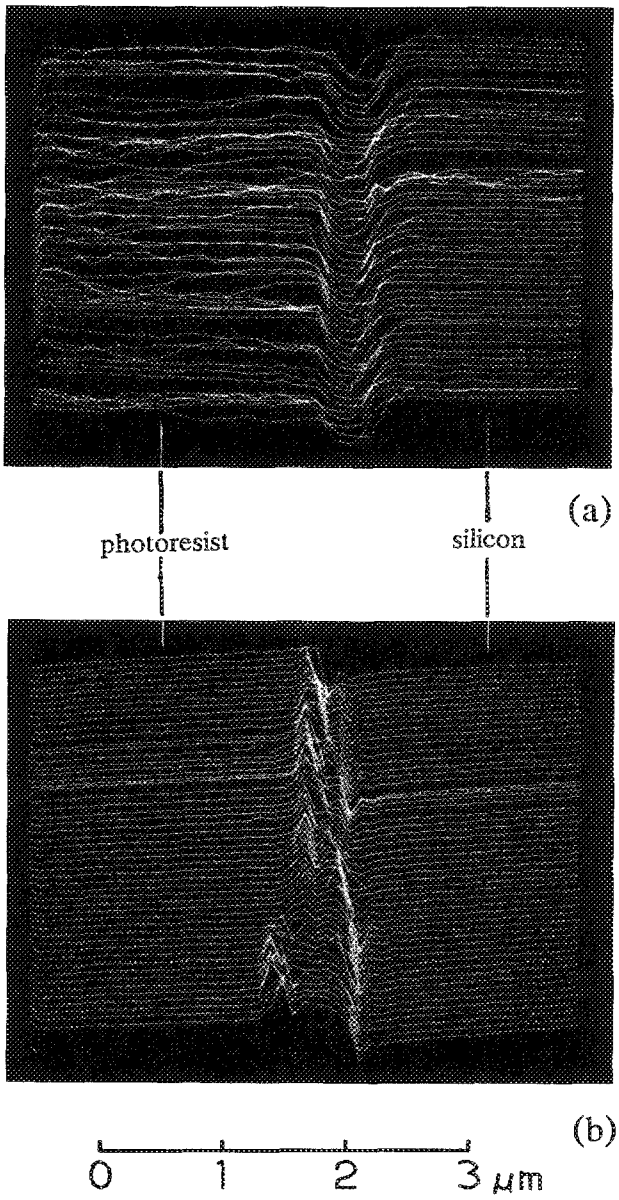

FIG. 7. Force measurement (a) and profile (b) over a silicon wafer partly covered with photoresist. The peak van der Waals force on the Si area is stronger and more uniform than on the photoresist, it is much weaker at the boundary.

a distance varying between 100 and 1000 Å, depending on the sample (see the experimental configuration in Fig. 6). The vibration amplitude at ω_0 changes abruptly from A_0, when the tip is far from the sample, to a value close to zero, when the tip comes in "close contact" with the sample. Hence, the modulation on the signal at ω_0 coming from the laser probe is almost a square wave at ω_1. Two feedback loops control the position of the tip. The first, a fast feedback loop, includes a low-pass filter with a cutoff frequency around 500 Hz. The square wave becomes a triangle wave, with fixed skew rates. The rise and fall times, however, are proportional to the distance necessary to pull the tip away from the surface D_v (see Sec. III), and hence to the force F_v. By measuring the frequency ω_1, one effectively measures the force F_v, which is material dependent. The second feedback loop, a slow feedback loop which responds to the peak value of lever vibration at ω_0, keeps the average tip-sample spacing at a constant value in order to follow the topography of the scanned sample. By this method, two independent signals are measured at each point on the sample to characterize both the material properties and the surface profile.

A. Experimental result

The measurement was conducted over a wafer of silicon partly covered with a 900-Å layer of photo resist. Figures 7(a) and 7(b), respectively, show the profile and the force measurements over the two regions, on an area of $4\times4\ \mu$. The force signal over the two regions differs in amplitude and regularity from the profile signal. In particular, over the photoresist region, the force signal appears more irregular. This suggests that the peak van der Waals force, F_v, is a function of the contact area between the tip and the sample; this area varies more readily in the case of the photoresist than with silicon, since it is a softer material. The boundary between the two regions is somewhat irregular, and extends over $0.5\ \mu$, which limits the practically achieved spatial resolution.

This mode of operation raises the question of the potential damage induced to the tip or sample, since it involves forces two orders of magnitude larger than those encountered in the first mode of operation. By scanning the same region twice, small changes of about 10% were noticeable in the photoresist area, but nothing was observed in the silicon. In order to ascertain the damage in silicon, small features of about 100 Å on the previous etched silicon wafer were profiled twice using the second mode of operation. No detectable change was observed. Hence, the technique appears to be nondestructive, at least for hard materials like silicon.

VI. CONCLUSION

We have described a technique than can precisely measure the force between two bodies separated by a distance ranging from 30 to 180 Å. We believe that it will be possible to extend the range to cover tip-sample separations from a few angstroms to several hundred angstroms.

Force measurement provides a new way of controlling the gap between a tip and surface. As an application, profiling is performed with a spatial resolution of 50 Å, which we expect to improve towards atomic resolution. Using a new control scheme, simultaneous measurement of the peak van der Waals force and profiling has been demonstrated. This added feature permits the characterization of materials on a submicron scale. We believe that with further refinement it should be possible to achieve spatial resolution in the nanometer range.

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[1]J.N. Israelachvili, *Intermolecular and Surface Forces* (Academic, London, 1985).
[2]D. Henderson, J. Colloid Interface Sci. (to be published).
[3]G. Binnig and H. Rohrer, Sci. Am. **253**, 50 (1985).
[4]D. W. Pohl, W. Denk, and U. Duerig, J. Appl. Phys. **59**, 3318 (1986).
[5]G. Binnig, C. F. Quate, and Ch. Gerber, Phys. Rev. Lett. **56**, 930 (1986).
[6]J. M. Soler, A. M. Baro, N. Garcia, and H. Rohrer, Phys. Rev. Lett. **57**, 444 (1986).
[7]U. Durig, J. K. Gimzewski, D. W. Pohl, and R. Schlittler, Phys. Rev. Lett. **57**, 2403 (1986).
[8]G. M. McClelland, R. Erlandsson, and S. Chiang, Review of Progress in Quantitative Non-Destructive Evaluation (Plenum, New York), Vol. 6 (to be published).
[9]D. Royer, E. Dieulesaint, and Y. Martin, in *Proceedings of IEEE Ultrasonics Symposium at San Francisco*, edited by B. R. McAvoy (IEEE, New York, 1985), p. 432.
[10]R. M. DeLaRue, R. F. Humphryes, I. M. Mason, and E. A. Ash, Proc. IEEE **119**, 117 (1972).
[11]C. C. Williams and H. K. Wickramasinghe, Appl. Phys. Lett. **49**, 1587 (1986).