# Experimental Observation of Forces Acting during Scanning Tunneling Microscopy

U. Dürig, J. K. Gimzewski, and D. W. Pohl
*IBM Zurich Research Laboratory, 8803 Rüschlikon, Switzerland*
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Forces acting between tunnel tip and sample during scanning tunneling microscopy were explored experimentally. Force gradients were detected as resonance shifts of cantilever beams used as sample stage. Beam deflections caused by force variations were also recorded. We present data for tungsten tips and polycrystalline silver surfaces prepared under UHV conditions. Large positive force gradients were found for the range of tunnel distances investigated. Force maps show pronounced features correlating directly with topographic images.

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In addition to surface topographic data,[1] scanning tunneling microscopy (STM) can provide local information on electronic and chemical structure.[2] In this paper, we turn our attention to local tip-surface forces acting during normal tunneling operation in the STM. In contrast to tunneling, where only electrons with momentum $k_{\perp} \simeq k_F$, i.e., near the Fermi surface, contribute appreciably to the tunnel current,[3] interaction forces involve electrons in total $k$ space ($k \leq k_F$). The interaction forces between tip and sample, therefore, can give unique information complementary to conventional tunneling data.

Most experimental investigations of interaction forces between the surfaces of two macroscopic objects have been limited to an approximately planar geometry.[4-6] Residual roughness limits the minimum distance and resolution to $\geq 10$ Å. Tunneling under STM conditions, on the other hand, typically takes place at smaller gap widths, $S < 10$ Å. Only scant information is available in this range. For these reasons, it is of interest to explore the interaction forces between tip and sample, and correlate them to the tunnel properties observed in the usual STM mode.

The interaction between tip and sample was studied by use of a flexible cantilever beam as sample holder. Forces are detected by measurement of the deflection of the beam from its equilibrium position, whereas force gradients $\partial F/\partial z$ (where $z$ is the surface normal) cause a detectable shift in the resonance properties of the beam. This scheme is similar to the one described by Israelachvili and Tabor[5] to investigate long-range interaction forces between two parallel surfaces.

With the STM geometry, the interaction forces expected are orders of magnitude smaller than in planar arrangements. The difference is adequately compensated by the extreme distance sensitivity of the tunnel current. This sensitivity was recently exploited by Binnig, Quate, and Gerber[7] to sense subatomic beam deflections. They were induced by an independent stylus scanned across a surface (atomic force microscopy) under *isodynamic* conditions, that is, with force quantities as the control parameter.

In the present experiment, the tip of a normal STM is used both for tunneling and as a force probe. This permits high-resolution mapping of forces (force gradients) simultaneously with conventional STM images. In the present mode of operation, the tip is scanned through use of the tunnel gap spacing $s$ as a control parameter which can readily be adjusted by the tunneling parameters (barrier height, current, and voltage). Force maps obtained in this way can be directly correlated to surface topography and other data obtained in STM.

The experiments were conducted with a "pocketsize" STM[8] mounted in a UHV chamber ($p \leq 2\times 10^{-10}$ mbar) equipped with a sample-preparation chamber and transfer device. The sample substrates were thin cantilever beams (CB) made of steel, copper, and tantalum foils. Thick silver films ($\sim 2000$ Å) were condensed on the CB's, at $T = 300$ K, $p \simeq 10^{-9}$ mbar in the preparation chamber, and transferred to the STM chamber under UHV conditions. STM measurements were conducted by use of an *in situ* sputtered W tip sharpened by operation in the field-emission mode with a separate sample as anode.

A force gradient $C_i^* = \partial F/\partial z = \partial^2 U/\partial z^2$ (where $U$ is the interfacial interaction potential) influences the compliance of the combined tip-CB system. It also changes the CB bending curve for a given mode of excitation. Both effects result in a shift of the resonance frequency $\nu_R$.

A vibration amplitude of the CB between a tenth and a hundredth of an angstrom can be readily detected as a modulation in the tunnel current. Vibrations with such a small amplitude need not be excited externally; they exist because of thermal excitation even in macroscopic CB's. For instance, for the steel CB (length $l = 10$ mm, width $w = 1.5$ mm, thickness $t = 0.05$ mm, $T = 300$ K), we calculate a mean thermal amplitude of the lowest eigenmode ($\nu_R = 415$ Hz) of 0.25 Å and a compliance $C^* = 10$ N m$^{-1}$ (measured at the free end of the CB). This compliance is in magnitude comparable to values deduced from theoretical interaction potentials,[9] which served as a guideline in the planning of this experiment (see below).

In a first set of experiments, we concentrated upon the *spectrum of the tunnel current* $I_t$. Figure 1 shows two typical spectra measured with the tunnel tip near the end of the steel CB; $S(\nu)$ is the power density of $\ln I_t$ calibrated to the amplitude of the reference peak (see below). The spectra were measured sequentially at $V_t = 0.45$ V, $I_t = 2$ and 10 nA, respectively. The thermal excitation of the CB manifests itself as a broad peak around 650 Hz (830 Hz). The spikes are caused by residual vibrations and line frequency pickup. The 1200-Hz peak, however, is a control signal produced by modulation of the tip-sample gap width with an amplitude of 0.06 Å. The modulation was used to calibrate the spectrum and to determine tunnel barrier height ($\Phi \simeq 2-3$ eV) by phase sensitive detection.

Note that the STM control-loop cutoff frequency was lowered to about 10 Hz during spectra acquisition and hence cannot compensate the gap-width modulations of interest. The two spectra of Fig. 1 and other ones consistently show a positive frequency shift which increases when we go to higher tunnel currents or lower voltages, i.e., smaller gap width. This implies that the force gradient is positive for the whole tunnel range, a somewhat surprising finding which will be further discussed below.

The rms amplitudes of the thermal vibration were determined by integration of the spectra in Fig. 1 (spikes and background level removed). The resulting values of $0.19 \pm 0.02$ and $0.13 \pm 0.02$ Å for the 650- and 830-Hz peaks, respectively, compare well with amplitudes of 0.16 and 0.12 Å obtained from calculations taking into account the force gradient between tip and sample determined from the frequency shift.[4]

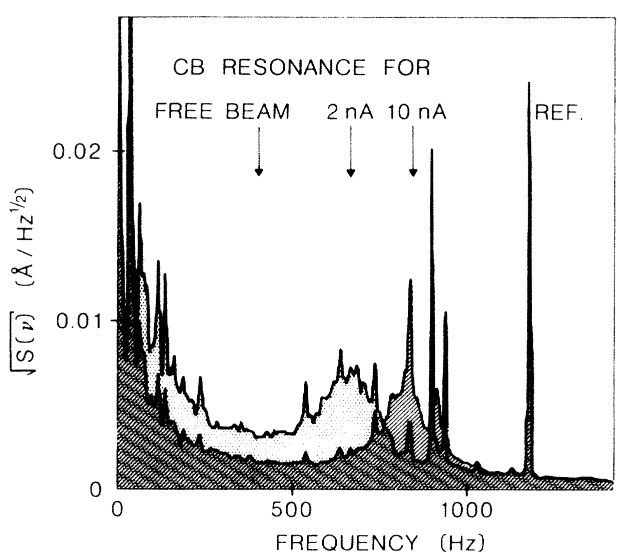


FIG. 1. Spectra of the tunnel current for $I_t = 2$ nA (dotted area) and 10 nA (hatched area) at $V_t = 0.45$ V.

The latter varies in a systematic way as a function of position $x_{\rm tip}$ along the beam. Figure 2 shows, for Ag surfaces, experimental values for the steel beam and for a fairly soft beam cut from a copper foil with dimensions $5.8 \times 1.15 \times 0.02$ mm$^3$, $\nu_R = 280$ Hz, $C^* = 1$ N/m. The observed increase of $\nu_R$ vs $x_{\rm tip}$ results from a decrease of the static compliance $C^*(x_{\rm tip})$, along the CB ($x_{\rm tip} = 0-l$). From the known data on the beams, $\nu_R(x_{\rm tip})$ can be calculated, leaving $C_i^*$ as the only fit parameter. The curves introduced in Fig. 2 suggest a $C_i^*$ between 15 and 50 N/m for both beams and the tunnel current-voltage range used.

We suspected that the scatter of data as well as the large width of the CB resonance are caused by local variations of the interaction potential on the sample surface and unavoidable drift in lateral position during data acquisition. To substantiate this idea, we took a series of STM images for different positions $x_{\rm tip}$ and values $I_t$, $V_t$ (Fig. 3). Above the base [$x_{\rm tip} = 0$, Fig. 3(a)], the position $z_{\rm tip}$ recorded in these scan images represents the topography of the surface in the usual way; individual crystallites with facets gently inclined towards each other separated by compact boundaries are clearly resolved. The topography closely resembles that previously reported for Ag films condensed at 300 K.[10] Above the free part of the CB, however, the value of $z_{\rm tip}$ is expected to be a superposition of topography and CB deflection at constant gap spacing.

Figures 3(b)–3(d) were obtained at $x_{\rm tip} = l$ for three different values of $V_t$ on the same image area. The

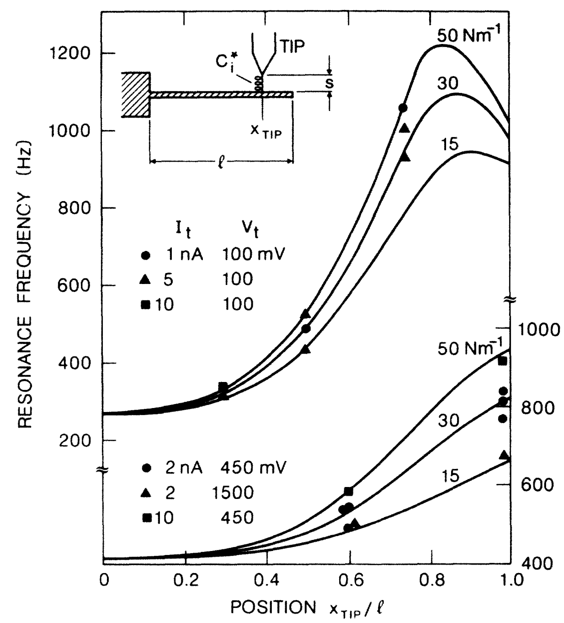


FIG. 2. Experimental and calculated CB resonance frequencies for various positions of the tunnel tip. Upper family of curves, for the copper, and lower, for the steel beam.

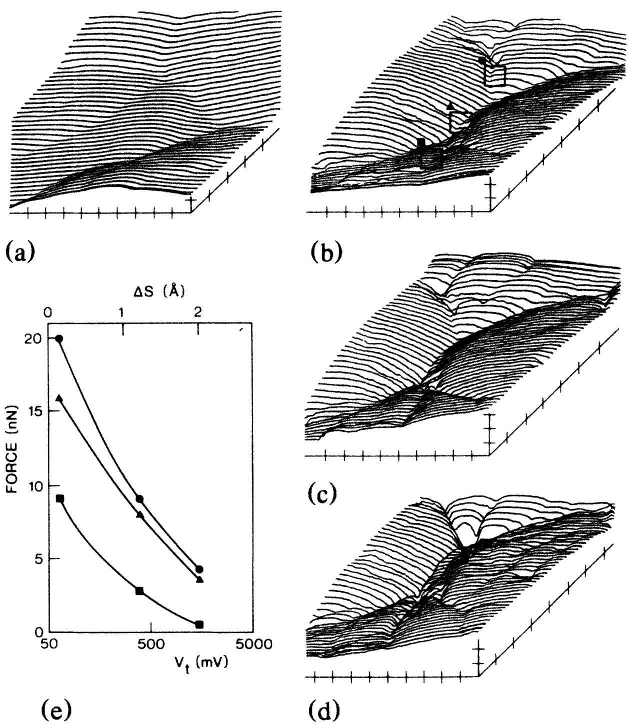


FIG. 3. STM graphs of polycrystalline Ag (a) at the base and (b)-(d) at the free end with (b) $V_t = 1.4$ V, (c) 0.45 V, and (d) 0.0063 V. Tick marks correspond to 50 Å. (e) Force variations as a function of $V_t$ for the three sites indicated in (b). The curves are a visual guide only.

grooving between the flat areas and grain boundaries increases dramatically when we lower $V_t$ from 1.4 to 0.063 V. No such increase in apparent grooving could be found for the same variation of $V_t$ on the base. We conclude that these deep valleys are caused by a strong change of forces in the vicinity of the silver grain boundaries. Figure 3(d) showing the largest grooving hence is essentially a *force map* with an estimated lateral resolution of $\sim 10$ Å.

Figure 3(e) shows the change of this additional force as a function of tunnel voltage for the three sites indicated in Fig. 3(b). The approximate scale on the top of the plot was determined from $\Delta s \approx (\Delta \ln V_t)/A\Phi^{1/2}$ ($A \sim 1$ Å$^{-1}$ eV$^{-1/2}$) and the measured value of $\Phi$.

One recognizes very similar behavior at the three sites with a force swing as large as 10 nN. The slope of the curves yields compliances between 50 and 150 N m$^{-1}$ which is broadly consistent with the above frequency-shift analysis. STM scans repeated at different positions, on different CB's, and with various values of $I_t$ and $V_t$ showed similar pseudotopographic features.

On the basis of our data and the assumption of an interaction radius comparable to the lateral resolution observed ($\sim 10$ Å), one estimates a pressure of $\sim 1$ GPa (10 kbar). Such pressures are close to the *macroscopic* plastic deformation limit. The respective *microscopic* limit is considerably larger[4]; indeed, we detected no topographic changes induced by repetitive scanning in our experiments. Soler *et al.*[11] recently pointed out that the interaction forces can induce significant deformations for soft material surfaces. In our case, however, the calculated elastic deformations amount to less than 0.1 Å.

Calculations based on a jellium model of the interaction between two metals[9] consistently predict attractive potentials with a minimum near one lattice constant and a depth comparable to the binding energy. The force gradients derived from these calculations, normalized to the area covered by one surface atom (approx. $3 \times 3$ Å$^2$), are of the order of $-0.1$ to $-0.5$ N m$^{-1}$ at distances $s$ of several angstroms. They change signs at approximately 1.5 Å between the jellium surfaces reaching values of about 5 N m$^{-1}$ near the potential minimum. The force gradients $C_l^*$ found in our experiments have values between $+15$ and $+50$ N/m. To interpret this result in terms of the above estimate one has to have a distance $s < 1.5$ Å and an interaction area of about 10 by 10 Å$^2$. While the latter requirement appears reasonable, the first is in contradiction to existing estimates of the tunnel distance $s$ during STM operation, typically 5-10 Å.[3] Actually, if the tunnel gap were 1.5 Å only, the barrier would practically collapse resulting in a low-resistance metallic point contact and extremely small response to distance modulation. Neither was the case in our measurements since $V_t/I_t = 10$-1000 MΩ and $\phi$, derived from the modulation signal, was about 3 eV as it should be. Also the distance variation of $>2$ Å for the different settings of $V_t$ and $I_t$, cf. Fig. 3(e), implies that at least some of the measurements were performed at a larger gap width.

Whether additional contributions to the interaction potential or a difference in the effective gap spacings for force and tunneling interactions are responsible for the discrepancy observed cannot be resolved at the present stage. Speculatively, the difference might be caused by a layer of adsorbed oxygen covering tip and sample surfaces after, say, an hour of experimentation at $10^{-10}$ mbar.

It is known from STM experiments in air and liquids that a sufficiently thin layer does not seriously modify the tunnel effect as compared to a vacuum environment. The interaction energy, on the other hand, would become a superposition of contributions from the interfaces of tip, adlayer, and sample resulting in significant differences from the above estimate. Our results suggest that interaction forces resulting from adsorbate layers could complicate the interpretation of STM data even under UHV conditions. Clearly, such effects are to be considered in all STM experiments.

In summary, we have shown how information on local interfacial interaction can be obtained from the tunnel spectrum; that the "Brownian" motion of a quite macroscopic cantilever beam can be readily observed in the tunnel current; that the interaction is stronger than expected, an indication of a difference between tunnel and force distance; and that, by proper variation of operating conditions, the STM picture can be made to represent either a topographic or a force map of the surface under investigation. This work also leads to the suggestion that tips equipped with tailor-made thin nonmetallic adlayers could be used to explore the repulsive region of interfacial interaction which might become of relevance for nanomechanical applications such as AFM.

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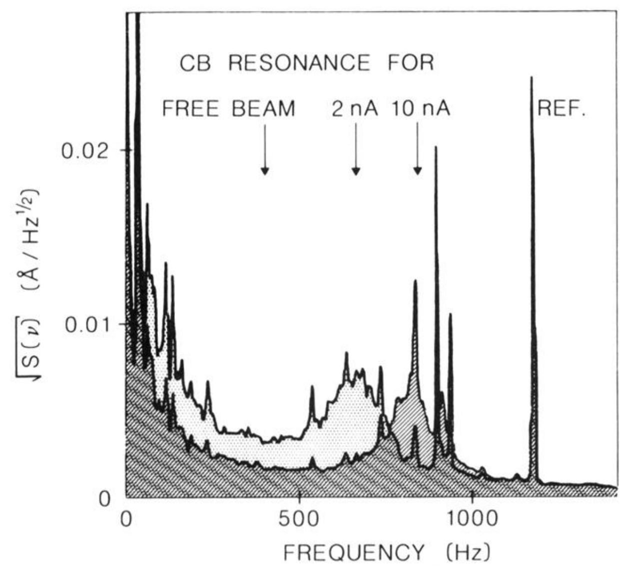


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