Compact, combined scanning tunneling/force microscope

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We have built a combined scanning tunneling and scanning force microscope. Owing to the compact design of the instrument with Nomarski type of interferometry for lever deflection sensing, we achieved excellent stability with a total rms noise of 0.03-0.04 Å in a frequency bandwidth of 0.01 Hz–2 kHz and a spectral noise density of $2.0 \times 10^{-4}$ Å/$\sqrt{\text{Hz}}$ at higher frequencies (> 2 kHz) using cantilevers with compliances of ~150 N m$^{-1}$. Simultaneous measurement of constant current contours, the acting forces, and the system compliance allows separation of sample topography from electronic and elastic effects.

I. INTRODUCTION

Combination of scanning tunneling microscopy (STM) and force microscopy (FM) for simultaneous operation is most desirable for a large variety of studies on the atomic or nanometer scale. A combined STM/FM allows investigation of mixed conducting and insulating structures such as partially oxidized metal and semiconductor surfaces by a force-controlled tip position on the insulating parts while retaining the STM capabilities on the conductive parts. In general, forces can provide the desired homogeneous control interaction for STM imaging of objects with strongly inhomogeneous electronic properties. Other approaches to deal with insulating regions are microwave operation of STM$^{1,2}$ and laser frequency mixing.$^{3}$

In the latter,$^{4}$ as in most force methods, the nonmonotonic distance dependence of the interactions restricts their use as control interactions—the feedback input—to limited distance regimes. In the case of pressure-sensitive materials, such as many biological molecules, it is very useful to know what effect the tip has on the object during the imaging process and to estimate the effect of elastic deformations on STM imaging, in particular on images such as "workfunction" profiles.$^{5,6}$ Finally, as we show below, combined STM/FM imaging can be used in STM to separate topography from electronic and elastic effects.

Early imaging methods involving tunneling and forces are summarized in Ref. 6. Pioneering work in this direction are the adhesion force studies of Dürrig et al.$^{7,8}$ Combinations of force and tunneling microscopy on graphite have been reported by Mate et al.$^{9}$ and Sugawara et al.$^{10}$ and on various substrates covered with organic matter by Salmeron et al.$^{11}$ and Specht et al.$^{12}$ In this paper we present a STM/FM combination based on the force microscopy method conceived by Schönenberger and Alvarado.$^{13}$ In order to guard against instabilities caused by possible large negative force gradients in the tunneling regime due to tip-sample interaction, it is necessary to use a rather stiff cantilever with a force constant larger than the force gradient of the interaction potential. This, however, requires a very stable and precise level deflection readout to partly compensate for the loss in force sensitivity due to the stiff cantilevers.

First results of simultaneous recording of constant current contours, the acting force, and the system compliance on highly oriented pyrolytic graphite (HOPG) are presented.

II. INSTRUMENTAL

Our miniaturized combined STM/FM is sketched in Fig. 1. The deflection of the force sensing cantilever is measured by means of the phase shift of two orthogonally polarized light beams reflected off the cantilever. This technique, known as Nomarski interferometry, minimizes fluctuations of the optical path length caused by air movement or thermal drift. The reference and the sensing beam correspond here to the two orthogonal polarization states [linearly polarized $p$ state with in-plane polarization with respect to the Wollaston prism (1) and the $s$ state with perpendicular polarization]. We use a commercially available laser diode$^{14}$ and a lens system to collimate the laser light. The light is led off axis through a Wollaston prism (1) in order to split the two polarization states of the laser light. An additional lens allows the two laser beams to be focused on the back of the cantilever. The reference and the sensing beams are focused on the root and the tip of the cantilever, respectively. The backreflected beams, now containing the information on the cantilever deflection inherent in their relative phase shift, again pass the lens and the Wollaston prism (1). In order to analyze the polarization states and hence the phase shift of the two beams emerging from the Wollaston prism (1), both beams pass another Wollaston prism (2) [rotated 45° with respect to (1)]. This
Wollaston prism acts as a polarizing device to produce interference between the reference and the sensing beam. The intensity of the two resulting interference patterns is measured by two photodiodes and converted into electrical signals \( A \) and \( B \). By shifting the Wollaston prism (1) perpendicular to the light path the phase between the two light beams can be shifted \( (2\pi \approx 10 \mu m) \). With this phase compensator the phase is adjusted for maximum light intensity contrast.\(^{15}\) In order to achieve high stability at low frequencies we built a very small and rigid instrument. This was done with an off-axis setup of the interferometer in which nonpolarizing beam splitters and optical isolators were omitted. The entire interferometer (including laser diode, photodiode, and cantilever sensor) has a length of \( \approx 50 \text{ mm} \) (Fig. 2).

The cantilevers with integrated tips are microfabricated from a single-crystalline silicon wafer and have a length of \( 200-300 \mu m \).\(^{16}\) The cantilever compliances are \( f_L = 100-150 \text{ N m}^{-1} \) with resonance frequencies of the order of \( \approx 200 \text{ kHz} \). Although the silicon material is heavily p doped, the cantilevers were coated with \( \approx 150 \text{ Å} \) of Au or Pt to avoid oxidation on the cantilever tip. Furthermore, we implemented a novel design of a current-to-voltage (\( I-V \)) converter with a superior bandwidth (>1 MHz) and signal-to-noise ratio to measure the electronic tunneling current.\(^{17}\) The \( I-V \) converter (gain \( 10^6 \text{ V A}^{-1} \)), equipped with a 100 kHz low-pass filter (first order), allowed STM experiments down to tunneling currents of \( I_t \approx 5 \text{ pA} \).

A typical noise spectrum and a dc oscilloscope trace of the interferometer signal of the free cantilever are shown in Fig. 3. The noise spectrum of the unfiltered interferometer signal (a) shows an increase of only one order of magnitude in the power spectrum density (PSD) over the entire frequency range of interest (0.01 Hz–100 kHz). The marked increase below 5 Hz is artificial and due to the finite window size of the signal analyzer whereas the peaks at 50 and 150 Hz are believed to be due to electrical pickup of the laser diode driver. At high frequencies (>1 kHz) the PSD is dominated by a white noise level of \( <2.0 \times 10^{-4} \text{ Å/}\sqrt{\text{Hz}} \). This is a factor of 2 larger than the sum of the spectrum noise density calculated from the thermal noise of the cantilever \( (3 \times 10^{-5} \text{ Å/}\sqrt{\text{Hz}}) \) and the shot noise of the photodiode \( (6 \times 10^{-5} \text{ Å/}\sqrt{\text{Hz}}) \). The lower trace is 2 kHz low pass filtered (first order) and shows a peak-to-peak noise of \( <0.1 \text{ Å} \) (rms noise: 0.03–0.04 Å) in a dc-2 kHz bandwidth for an acquisition time of over 10 s.

All STM/FM data presented are unfiltered raw data.

### III. EXPERIMENT

When scanning in the constant current mode, the difference signal \( dz = u_Z - u_L \), where \( u_Z \) and \( u_L \) denote the \( z \) excursions of the piezodrive and the cantilever, respectively, is equivalent to a conventional STM constant current image while \( u_L \) displays the acting force. The latter is henceforth referred to as the force image. Concurrent measurements of the total compliance \( f_Z = dF/du_Z = f_L \delta L/SZ \) were performed by modulating the sample position by \( \delta Z = 1-2 \text{ Å} \) in the \( z \) direction at a frequency of 1–3 kHz while measuring the cantilever response \( \delta L \). The
modulation frequency has to be larger than the cutoff frequency of the feedback loop but sufficiently smaller than the cantilever resonance frequency in order that the transfer function of the system remain unaffected by resonance frequency shifts and quality factor changes.

We used the combined STM/FM system to study the surface of HOPG. The graphite sample was freshly cleaved and imaged in air. Figure 4(a) presents a constant current STM image \( \delta z = u_T - u_L \). The simultaneously recorded force, obtained from \( F = u_f f_L \), where the cantilever compliance \( f_L = 130 \text{ N m}^{-1} \), is shown in Fig. 4(b). The average net force on the cantilever of \( -6.5 \times 10^{-9} \text{ N} \) was attractive. This is two orders of magnitude smaller than the repulsive forces reported necessary for obtaining STM images in previous works.\(^9,10\) We attribute this to oxidized or contaminated tips in those experiments. White areas correspond to (a) large \( \delta z \) or high “topography” in a conventional STM image and (b) stronger attractive force. The corrugation is \( \pm 1.0 \text{ Å} \) for \( \delta z \) and \( \pm 0.21 \text{ Å} \) for \( u_L \), where the latter corresponds to a force corrugation of \( \pm 2.7 \times 10^{-9} \text{ N} \). The measured total compliance \( f_T = dF/du_T \) of \( +23 \text{ N m}^{-1} \) shows no variation over the entire image. The positive value suggests partial compensation of the overall attractive force by a repulsive interaction between the foremost part of the tip and the sample surface. We favor contact through a mediating layer like water or a tip oxide, since a real tunnel gap should exist at the imaging parameter of \( +250 \text{ mV} \) for the sample bias voltage and a tunneling current of \( 200 \text{ pA} \). The total excursion \( u_T \) of the sample in a constant current scan consists of the sum of the sample surface corrugation \( \Delta z \), and the total distortion \( u_T \). The latter includes the cantilever deflection \( u_L \), change of tunnel gap which is described by an elastic medium with compliance \( f_f \) and sample distortion. \( u_T \) is given by \( u_T = u_T f_L / f_E \). We therefore obtain for the sample surface corrugation \( \Delta z = u_T - u_T - u_T f_L / f_E \). Thus measuring \( u_T, u_L \) and \( f_T \), simultaneously in a constant current scan, we can separate the sample topography from changes in the tunnel gap and sample distortion. Indeed, we obtain \( \Delta z = 0 \) within the 0.1 Å accuracy of the experiment in Fig. 4.

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REFERENCES

15. For an exact theoretical treatment please refer to Ref. 13.
18. The tip radius was estimated from the measured graphite compliance $f_s$ and calculated graphite layer distortions as a function of the tip radius in G. Overney, D. Tománek, W. Zhong, Z. Sun, H. Miyazaki, and S. D. Mahanti, J. Phys. C (to be published).