STM activity at the University of Basel

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We have built a scanning tunneling microscope (STM) with a design similar to that published by Binnig and Rohrer. An electromagnetic device called the "maggot" has been developed for nanometer-scale movement over a wide temperature range. So far we have studied the surface topography of a Pd(100) single crystal, of a glassy $Pd_{81}Si_{19}$ alloy, and of graphite. Finally, nanometer-scale structures have been produced with the STM, indicating its potential for high-resolution microfabrication.

1. Introduction

Our main reason for building an STM was to apply this new technique for surface imaging on an atomic scale in real space to materials which we are currently studying in our laboratory. These materials are metallic glasses, rapidly quenched nano- and micro-crystalline alloys, quasicrystals or crystalloids, graphite, and graphite intercalation compounds. The focus of our research with the STM is mainly on nonperiodic structures and on obtaining structural information going beyond that deduced from scattering experiments and conventional electron microscopy. However, we strongly believe that in addition to topographical studies this novel technique has great potential in spectroscopy and nanometer-scale lithography, particularly with respect to the materials mentioned above. In fact, the STM seems to be the only instrument capable of studying both the real-space structure and certain physical properties sensitive to tunneling spectroscopy. Therefore the STM is extremely well suited for investigating nanocrystals in a matrix.

In the following sections we describe our contribution to the development of the STM design and present characteristic experimental results of our recent research activities.

2. Experiment

Our first version of the STM in 1983 followed very closely the design of Binnig and Rohrer [1–3]. The tunneling unit is placed on a vibration-free working platform with a two-stage spring system and eddy current damping. We also started with the piezoelectric "louse" to move the sample but we finally designed a two-dimensional magnetic walker, the so-called "maggot" [4]. **Figure 1** shows the tunneling unit consisting of the maggot (left) and the rigid piezoelectric drive (right). The maggot consists of two linear motors which are formed by a coil (a) and permanent Co-Sm magnets (b). The coil is wound from foils of Cu with Kapton insulation. The triangular body of the maggot stands on three ball bearings (c) and is moved on the vibration-free platform made from macor or stainless steel. The other parts marked in Figure 1 are (d) the sample, (e) the tip, (f) the shielding tube for the wire connected to the tip, and (g) the piezoelectric drive tripod (*xy*- and *z*-drives). We have paid considerable attention to the rigidity of the tunneling unit and to the complete electromagnetic shielding of the wire

connected to the tip. This wire is brought into the tunneling unit through a metal cylinder, allowing free movement of the wire.

The maggot is propelled across the platform by the magnetic fields from the two coils which are powered by current pulses generated by discharging a capacitor. The pulses are triggered by a microcomputer. The controlled movement of the maggot is achieved by vector addition of the individual steps. The maggot can be moved in any direction of the plane parallel to the working platform by simply changing the discharge voltages. The solution of Maxwell's equations shows that the maximum of the field pushing or pulling the permanent magnets coincides with the point where the radial component of the field which tries to move the permanent magnets off-center vanishes. This force pushing the magnets off-center would cause a twisting of the maggot. Calculations of the magnetic force show that the area where this radial component is negligible and the force pushing or pulling the permanent magnets is within 15% of its maximum value is sufficient for a reasonably large working range of the maggot in our STM. In **Figure 2** the calculated lines of constant axial force in the coils are shown. A saddle point which is rather flat occurs in the middle of the coil and this yields a stable step size.

The average step size of the maggot is measured by optical microscopy and with the STM itself. These measurements agree very well. Movement down to 200 Å per step is reliable and reproducible. Step sizes down to 70 Å have been achieved but are not very reliable. The finish of the surface of the platform does not seem to be crucial. Our experience indicates that for stainless steel a rather rough surface (e.g., normal grinding) and for macor a polished surface is needed. The maggot is ultrahigh-vacuum (UHV) compatible and has the potential to operate from 4.2 K to 500 K.

We have tried to optimize the suspension in order to suppress all kinds of vibrations. This has been achieved by calculating the different eigenmodes of the two-stage spring system and also by checking the movement of the platform [5]. Our STM development occurred in two successive parts. First we designed a version working in the 10^{-7}-torr range. Our aim was to gain experience with the design and the operation of the STM. The suspension which we designed took care of the vibrations of the diffusion pump and of the building. No low-frequency vibrations have been detected in the feedback loop and no high-frequency vibrations have been observed in the tunneling current. Second, we built a UHV version equipped with an ion pump yielding a base pressure of 10^{-11} torr. In this version the whole suspension is put underneath the tunneling unit and is hence lowered into the region of the liquid nitrogen trap and titanium sublimation pump in order to give easy access to the sample and the tip. The tunneling unit itself is placed on a macor platform and all the wires lead to a connector made from macor underneath this platform. This enables us to simply unplug the tunneling unit from the suspension, which is left in the vacuum chamber for replacing the sample and for repairs.

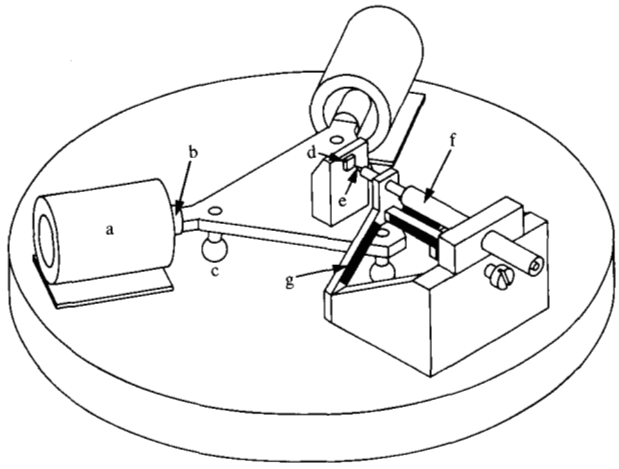

Figure 1

STM tunneling unit: (a) coils, (b) Co-Sm magnets, (c) ball bearings, (d) sample, (e) tip, (f) shielding tube, (g) piezoelectric drives (see text).

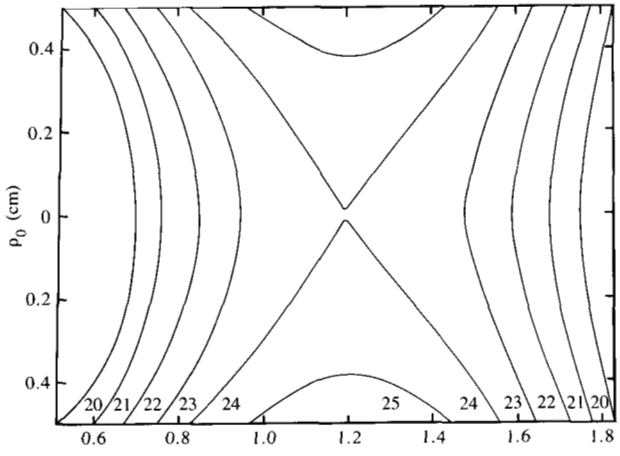

Figure 2

Calculated contours of constant axial force in units of 10^{-3} N/Å for the radial and axial positions of the magnet. ρ_0 is the displacement of the center of the magnet to the center of the coil in the radial direction. The x-axis shows the displacement $(z_c - z_0)$ of the center of the magnet z_c and the center of the coil z_0 in centimeters, the magnet and coil being 2 cm long.

The piezoelectric slabs of the tripod are driven by a symmetrical voltage of 0 to ±250 V. This voltage is generated by high-voltage amplifiers built at our institute with commercially available semiconductor components. A

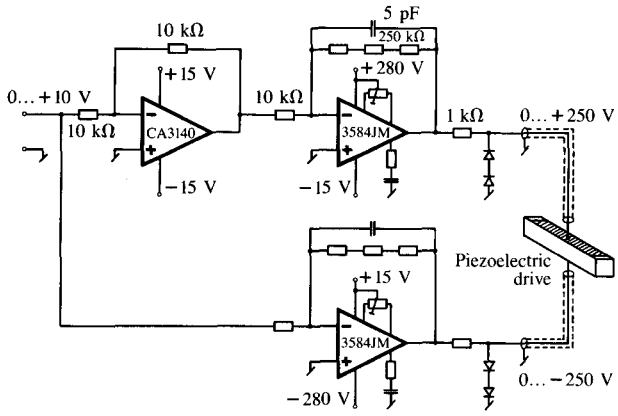

Figure 3

Circuit diagram of the high-voltage amplifiers designed at our institute.

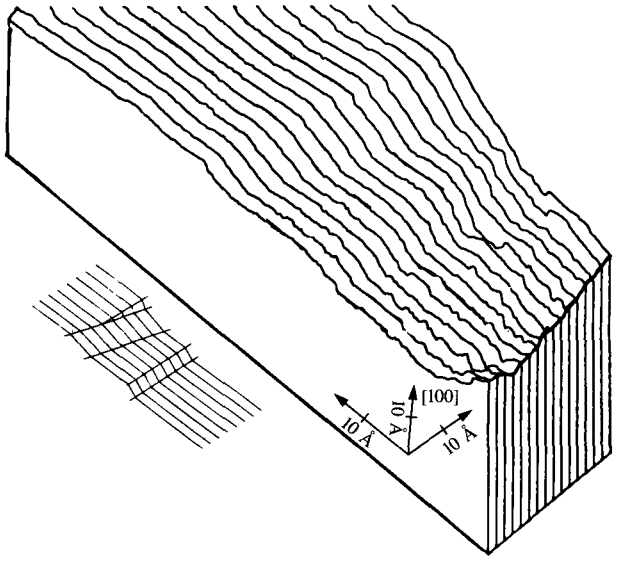

Figure 4

Mono- and diatomic steps on a Pd(100) surface.

schematic drawing of the electronics is shown in **Figure 3**. It turned out to be crucial to use well-stabilized ±280-V and ±15-V power supplies which are blocked by electrolytic condensers for the high-voltage amplifiers. Thus we have been able to achieve an overall root mean square (rms) noise level of 2.2 mV across the piezoelectric elements which results in an rms movement of the piezoelectric elements of roughly 0.02 Å in our case. The input voltage is supplied by a microcomputer which controls the movement of the *xy*-drive as well as the movement of the louse or maggot. For the regulation of the *z*-drive in the initial experiment we used a PID controller but we soon dropped the differential part because it led to instabilities.

3. Results and discussion

• *Surface topography of a Pd(100) single-crystal surface*

In order to check the performance and the resolution of our first STM we investigated the surface of a Pd single crystal [6,7]. The sample surface [3.7 degrees vicinal to (100)] had been sputter-cleaned and annealed several times outside the STM; no *in situ* surface preparation had been applied. The surface of the sample can be characterized by flat terrace-like regions coexisting with richly stepped areas [7]. On one of the flat parts of the surface we resolved mono- and diatomic steps which are shown in **Figure 4**, with the expected step heights of 3.5 Å and 7.0 Å, respectively. The inset of Figure 4 represents schematically the relative orientation of the steps, which are nonparallel to each other and seem to converge toward a point defect located outside the scanning area. Frequently we observed mono-atomic steps with curved contours. The detection of such nonperiodic features on nonideal surfaces illustrates the unique advantage of the STM technique, which is capable of imaging atomic arrangements without having to rely on crystal periodicity.

• *Surface topography of glassy* $Pd_{81}Si_{19}$

The palladium-rich Pd-Si alloys are among the first metals which have been prepared in the glassy state and their physical and chemical properties are now well known [8–10]. This archetypal glassy metal thus provides the opportunity to study an element both in its well-ordered crystalline state and in an amorphous structure. The glassy samples used in this investigation were prepared by the so-called splat-cooling technique [11]. In this procedure a droplet of the liquid alloy is held in a levitation coil. After the rf-power is turned off the droplet falls downward, interrupting a laser beam which triggers the acceleration of two carefully polished copper pistons. The pistons are accelerated towards each other and the droplet is squeezed between the pistons to a splat which is approximately 30 μm thick. The droplet is thus quenched from its liquid state to room temperature and the cooling rate of 10^6 degrees per second allows the generation of the noncrystalline structure.

We report on three sets of measurements. First we describe the topography of a freshly prepared sample as seen in the first non-UHV version of the STM. We then discuss our study of a sample which had been stored in air for several months. Finally we report on high-resolution measurements on a fresh sample in the UHV STM.

The topography of the optically completely featureless and shiny surface of $Pd_{81}Si_{19}$ observed by scanning tunneling microscopy at moderate resolution reveals two types of regions: droplet-like features and characteristic flat areas.

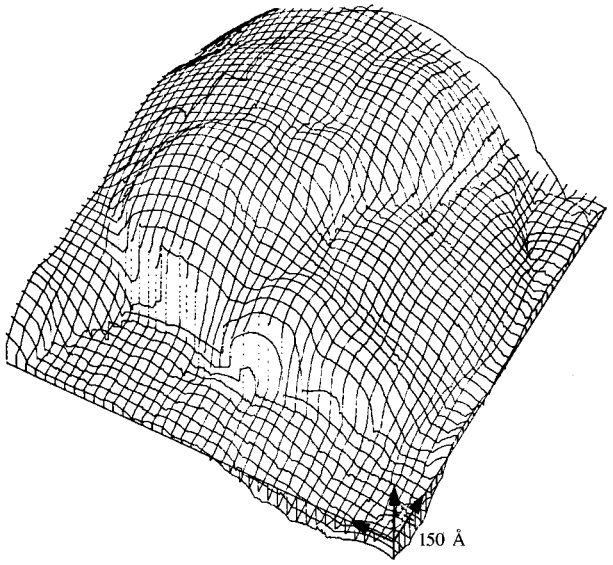

Figure 5

A droplet on the surface of glassy $Pd_{81}Si_{19}$.

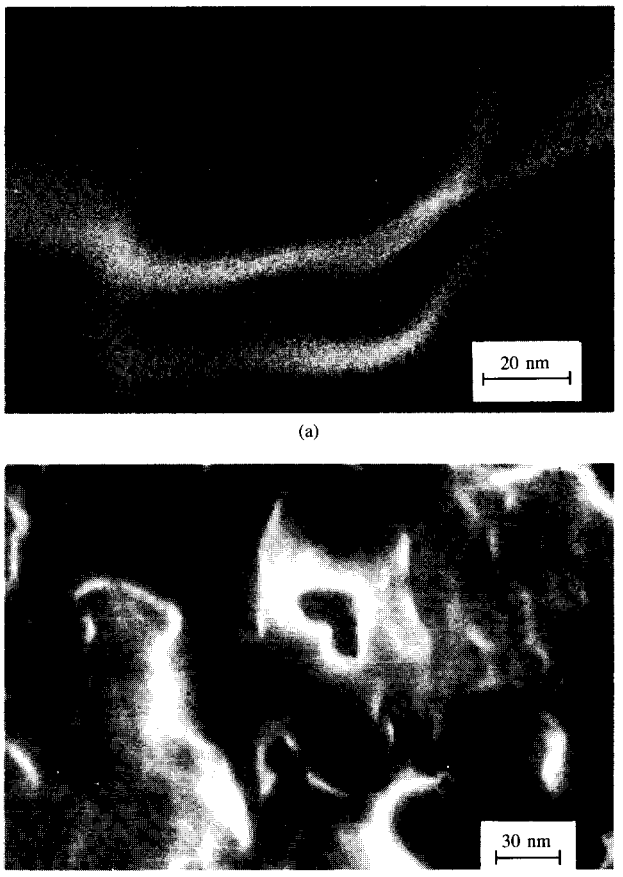

Figure 6

High-resolution SEM images of glassy $Pd_{81}Si_{19}$: (a) from the outer surface and (b) from the inner surface of a large droplet after electrochemical etching. Note the platelets as gross features in both images.

Figure 5 shows a two-dimensional scan over a typical "surface droplet." The stability of our STM is demonstrated by the minute mismatch which we attribute to thermal drift of the consecutively performed scans in the x and y directions. The curvature of the individual droplets is reminiscent of the splat-cooling process. Such aggregates of droplets are superimposed on the prevailing flat surface of the sample. A surface morphology of the characteristic flat areas which can be modeled by tiling a surface with disk-shaped objects (see, e.g., the STM images in [7] and [12]) was revealed at a resolution similar to that of Figure 5. In sharp contrast to the Pd surface, no aligned step structures or well-defined edges were observed and most of the contour lines were bent in random orientations. The STM thus gave the first indication of a hitherto unknown secondary structure of metallic glasses which is also discussed in the interpretation of other results [14].

The interpretation of these typical STM images is corroborated by high-resolution scanning electron microscopy (SEM) images. The resolution of high-magnification high-voltage SEM extends into the region of low-resolution STM, and thus a comparison of the gross features in the images obtained with both techniques should give similar results. We note that the details on such images will be rather different due to the different modes of image formation. **Figure 6(a)** displays a typical SEM image of $Pd_{81}Si_{19}$ which clearly shows the flattened droplet structure. The featureless surfaces are artifacts arising from insufficient contrast. Gentle electrochemical etching allowed us to view inside the material. From **Figure 6(b)** we see that the surface of a small droplet inside a metallic glass is made up of disk-shaped objects similar to those on the outer surface of the bulk material. We believe this secondary structure to arise from the rapid solidification process. This is discussed in detail elsewhere.

The absence of long-range order in the glassy state is reflected by the lack of organization of the surface topography. This seems to extend down to atomic-scale resolution, where we observed essentially structureless images such as that displayed in **Figure 7**, which should be compared with Figure 4. Areas of spikes such as those appearing on the left of Figure 7 are typical of these images. This feature arises from the sample and is not an artifact of the measurement because the local correlation is too high for this effect to be ascribed merely to electronic noise. We attribute the islands of spikes to nuclei of segregated and oxidized Si on the surface of the alloy. During the storage in

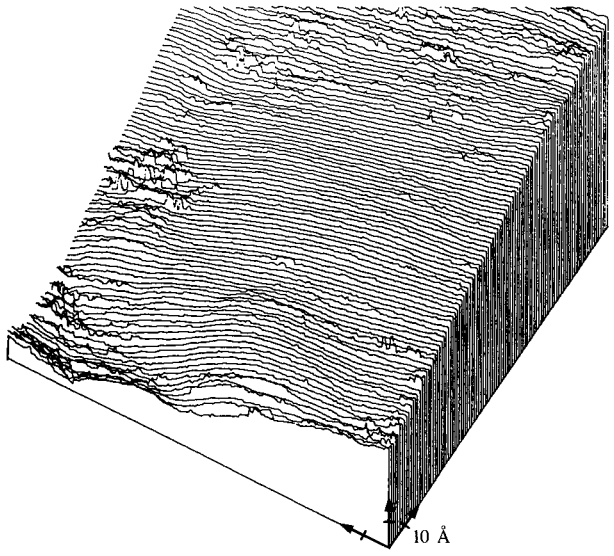

Figure 7

Typical flat area of glassy $Pd_{81}Si_{19}$ at high resolution.

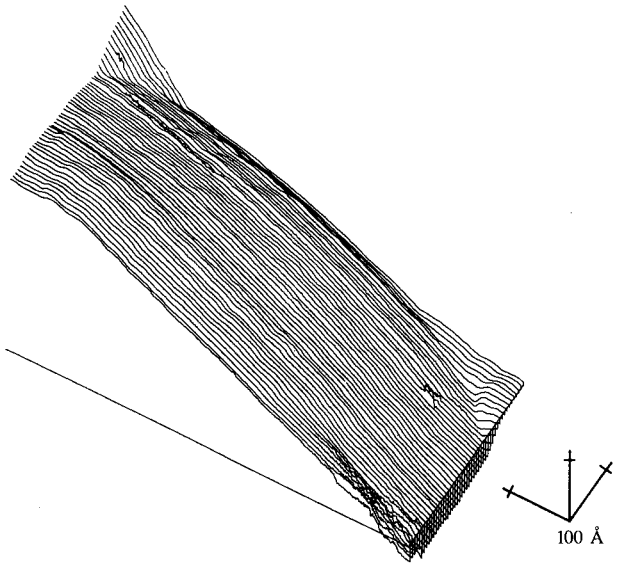

Figure 8

Surface waves on a glassy $Pd_{81}Si_{19}$ splat.

air these nuclei grew into a dense layer of silicon oxide which is known to cover aged surfaces of Pd-Si glasses [7]. Consequently we were unable to record any useful STM image from the aged sample mentioned above. We note here that results from photoelectron spectroscopy indicate the presence of a silicon oxide film on fresh samples as well as on aged material. The film thickness and surface compositions are different, however, in the two cases: The fresh sample was covered only by a few monolayers of silicon oxide (Si:O ratio below 1:2), whereas the aged sample exhibited a thick film with a Si:O ratio of 1:2.5.

Gentle surface cleaning in the UHV STM by heating the sample to a temperature well below the ordering temperature for the glass-to-crystalline transition enabled us to obtain better-resolved images than in the non-UHV instrument. The essentially smooth surface of a splat as displayed in **Figure 8** shows a wave-like structure with a periodicity of 35 Å and an amplitude of 3 Å. This structure may well represent a snapshot of frozen surface waves excited during the splat-cooling process on the liquid alloy drop. We discarded the view that the wave-like structure might have arisen as a template from the copper pistons of the splat-cooling apparatus. Much larger polishing marks on the copper pistons are usually not transferred onto the surfaces of splat-cooled surfaces since the liquid alloy does not wet the piston surface sufficiently. In a few cases, however, we observe transfer of features onto splats, but then the structures appear as sharp steps in the SEM and traces of copper can be located at these structures. The phenomenon of wave-like structures is currently being studied in more detail.

In general, STM provided valuable information on nonperiodic features of surfaces of bulk amorphous metallic glasses. We do not infer, however, that our images arise uniquely from the amorphous bulk structure of the specimen: Surfaces of bulk crystalline materials may exhibit similar features unless their surfaces are reconstructed. As an example, consider the surface of a well-crystallized Pd_2Si film prepared from evaporated Pd on a Si(100) wafer. The film has been exposed to air after preparation and then part of it has been sputtered and transferred through air into the STM. **Figure 9** shows (a) the surface of the sputtered Pd_2Si and (b) the Pd_2Si/Si interface. We observe a grain-like structure on the surface of the sputtered Pd_2Si film [Figure 9(a)]. A smaller, hillock-like structure has been found on part of the interface, but larger islands (>100 Å) also appear [Figure 9(b)]. The hillocks at the interface are most likely due to the Si substrate [13], while the larger structures are interpreted as Pd_2Si islands. X-ray diffraction gave evidence for the presence of Pd_2Si besides bulk Si at the interface.

Some features in Figure 9 are reminiscent of the $Pd_{81}Si_{19}$ images, although the frequent and typical surface droplet feature seems to be characteristic for rapidly quenched materials. The similarities between the two sets of images are in sharp contrast to the differences in the bulk structures of the specimen. We conclude that an STM surface analysis is not necessarily typical for the bulk structure of a material unless, with crystalline samples, UHV preparation procedures generate an atomically ordered reconstructed surface. In the case of the metallic glass the surface

morphology is seen as the consequence of the nonperiodic bulk structure, whereas the thin-film preparation and sample treatment result in disordered surfaces of bulk crystalline materials.

• *Nanometer-scale lithography*

During the SEM inspection of the glassy $Pd_{81}Si_{19}$ samples which had been used in the non-UHV STM, we found certain modifications of the areas which have been subjected to the STM investigation [12]. Traces of the STM scan lines have been observed on the fresh sample, as can be seen in **Figure 10**. The lines are approximately 20 Å wide, with a spacing of 160 Å. Tilting experiments and the absence of any deposits at the end points of the scan lines imply that the contrast is due to differences in the yield of secondary electrons rather than arising from a topography such as grooves or scratches. Attempts to deliberately scratch the surface of $Pd_{81}Si_{19}$ resulted in much larger topographies with deposited material at the end of the scratch, which was several hundred Å wide and in deformations along the edges. The lack of resolving power of high-energy high-resolution SEM becomes apparent from Figure 10. In the SEM image the surface appears structureless, whereas the corresponding STM scan clearly reveals the disk-like structure [12] discussed above. This height modulation of the "writing surface" is believed to be responsible for the irregular interruptions of the scan lines suggested by the SEM image.

Figure 10

SEM image of lines drawn with the STM on glassy $Pd_{81}Si_{19}$.

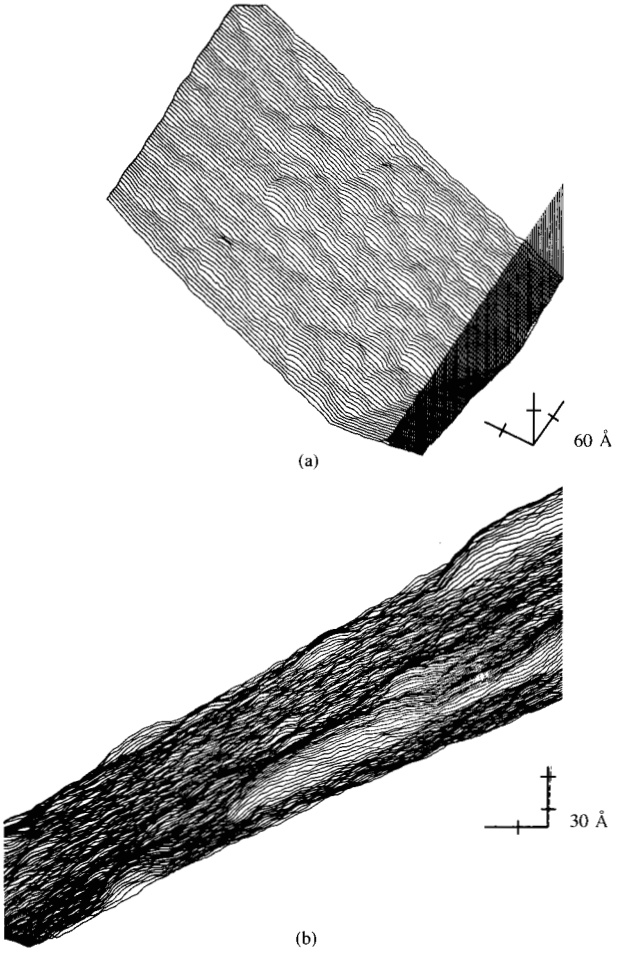

Figure 9

STM images from two different locations on the surface of a sputtered thin film of Pd_2Si on Si(100): (a) Pd_2Si, (b) Pd_2Si/Si interface.

To obtain information on the chemical nature of the surface on which we observed the lithographic effect, $Pd_{81}Si_{19}$ samples were studied by XPS before and after examination in the non-UHV STM. A multi-layer adsorbate of carbonyl groups containing hydrocarbons arising from diffusion pump oil was found to be the characteristic difference between the surfaces before and after the STM experiment. Upon heating in UHV the adsorbate layer was transformed into a carbonaceous deposit with accompanying desorption of water. Experiments with the SEM instrument, which operates under vacuum conditions similar to those of the non-UHV STM, revealed that after roughly six hours a "contamination layer" formed which was thick enough to allow line drawing with a 200-keV electron beam leading to a contrast similar to that shown in Figure 8. However, these lines drawn by the high-energy electron beam are strictly parallel to the viewing frame. The results from XPS and

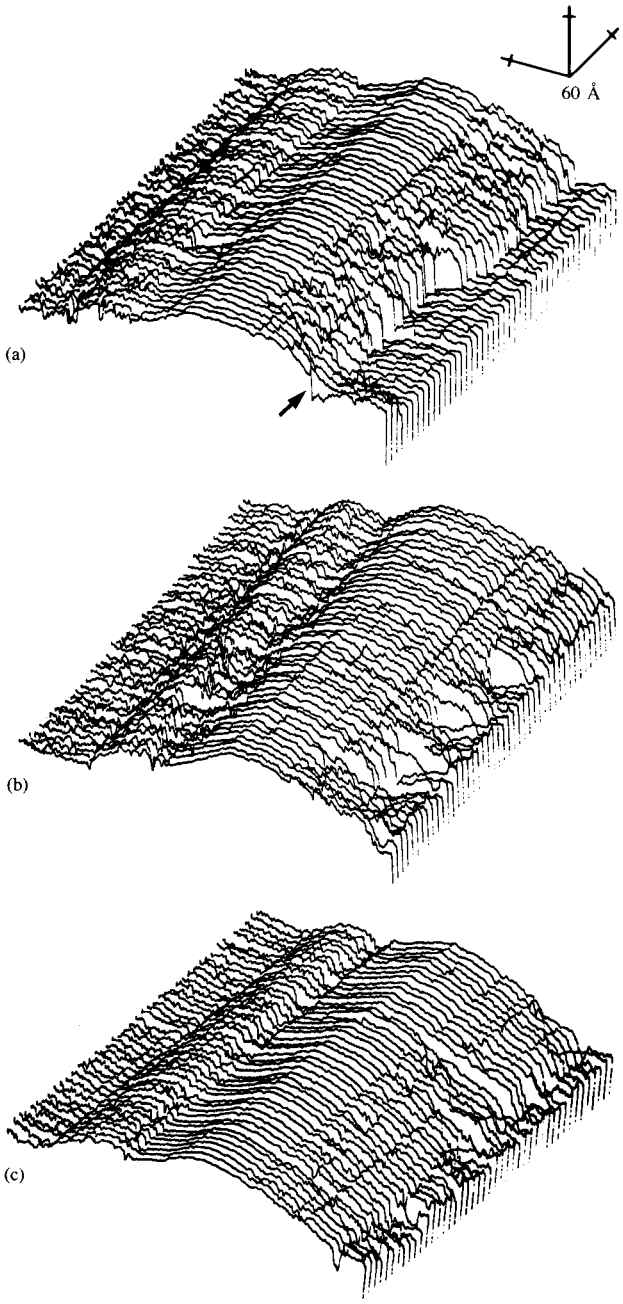

Figure 11

Scans over the same area of a graphite sample with different bias voltages: (a) The step on the right edge (see arrow) is clearly seen with 150 mV; (b) the step appears only at some places with 175 mV; (c) the step disappears with 250 mV.

SEM substantiate our assumption that a locally restricted chemical transformation of an adsorbate layer plays an important role in the STM lithographic process. The influence of the STM operating conditions and of the substrate on the nanometer-scale lithographic process is currently under investigation.

• *STM studies of graphite*

Graphite is well known for its anisotropy in chemical bonding, and its structure consists ideally of a stack of carbon sheets made up of planar hexagons with very weak van der Waals forces acting perpendicularly to the layer planes. In reality the infinite carbon planes are disrupted by defects frequently aligned along crystallographic main directions [15]. Cleaving therefore results in a flat surface with large terraces and well-oriented boundaries. STM images of several oriented pyrographites confirm this picture, which was derived from transmission electron microscopy (TEM) [16]. The height of the terraces varied between one and 20 interplanar distances ($n \times 3.35$ Å), information which would be difficult to obtain by conventional electron microscopy and which can be used to calibrate the piezoelectric drives of the STM. In general, the length of undisrupted terraces was considerably larger in hard pyrographite (containing many defects) than in soft highly oriented pyrolytic graphite (HOPG), which contains fewer defects. Terraces on HOPG exhibited frequently irregular prismatic faces which were not observed in pyrographite. These topographic observations reflect the number and degree of interaction of defects in the two types of graphite.

The weak interplanar forces allow us to estimate the upper limit of electrostatic forces between tip and surface in the STM setup. Above a certain critical voltage between tip and graphite surface one expects individual carbon layers to peel off from the crystal. This leads to a characteristic "open" surface morphology which is well known from cleaving experiments with the cleaving force perpendicular to the surface. In a sample of HOPG we observed this unfolded surface structure at locations of STM examination using a tip-to-surface voltage of approximately 200 mV. Under certain circumstances parts of the graphite surface are not rigidly attached to the crystal but may be moved around an axis parallel to defect lines (for example, like a page in a book). **Figure 11** demonstrates how—simply by varying the tip voltage—different topographies (here part of a step structure) can be generated by moving parts of the surface (which are attached to the bulk outside the imaging area) while scanning over the imaging area. Such experiments may give an impression of the magnitude of forces occurring during image formation in the STM.

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