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Tip-related artifacts in scanning tunneling potentiometry

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We show that tip-related artifacts can produce artificially abrupt and/or nonmonotonic behavior in scanning-tunneling-potentiometry (STP) measurements of rough surfaces, and thus considerable care must be used in interpreting actual STP images. Low-noise STP measurements were made on several metal films, and confirm that such artifacts do in some cases exist. These tip artifacts are often more obvious in STP images than in the corresponding topographic images, and hence STP may provide a valuable means to detect the presence of tip artifacts in other scanning-tunneling-microscope experiments.

Over the past few years, a number of workers have developed methods to use a scanning tunneling microscope (STM) to measure spatial variations in the electrochemical potential in materials and devices in which lateral potential gradients exist.[1-4] This "scanning tunneling potentiometry" (STP) has been used to investigate electronic transport properties in materials with (apparently) nanometer spatial resolution and microvolt voltage sensitivities. A number of measured STP images exhibit spatially abrupt voltage drops, which were used to infer local transport properties at metal-film discontinuities,[1] grain boundaries,[4] and at semiconductor-junction interfaces.[2,3]

There has been, however, little discussion on the possible role of the STM probe tip in these measurements. In conventional scanning tunneling microscopy, it has long been appreciated that the imperfect and unknown structure of the probe tip can introduce serious complications into the interpretation of measured topographic images.[5] A blunt tip will artificially broaden measured topographic features and reduce corrugation amplitudes. So-called "multiple tips" can create "shadows" and repeated features in topographic images. These problems are particularly severe on samples with surface roughness comparable to the roughness of the probe tip.

In a previous paper[6] we noted that a blunt tip could produce artificially abrupt "steps" in measured STP images. In the first part of the present paper, we consider in some detail the nature of tip-related "artifacts" which may be present in STP measurements. It turns out that STP is particularly sensitive to tip artifacts. Imperfect probe tips could in some cases produce measured STP behavior which is quite different, quantitatively and qualitatively, from actual behavior. Furthermore, this artificial behavior is quite similar to actual behavior one might expect from highly resistive structures within a conducting sample. Hence, without a detailed knowledge of the actual probe-tip geometry, it may be very difficult to determine to what extent measured potentiometric features on rough samples represent actual transport phenomena within a sample or result merely from tip artifacts. We then consider STP measurements we have made of thin-film metal samples. In a number of cases, we find evidence that tip artifacts are in fact present, and in some cases these tip artifacts are much more obvious in the STP images than in the corresponding STM topographic images. Hence, STP may provide a way to detect and study tip-related artifacts in other STM measurements which might otherwise be undetected.

Figure 1 illustrates the physical origin of many tip-related artifacts in STP. In Fig. 1(a), we consider a conducting sample with a rough surface topography, in which a lateral current flows from left to right. This current creates a spatially varying potential on the sample surface. This is sketched in Fig. 1(b), assuming that there are no structures in the films (e.g., grain boundaries) which create abrupt voltage drops. We now assume that this sample is scanned with an STM probe tip which is not perfectly sharp. Most of the electron tunneling is localized to a position on the surface within a few Å of the point of closest approach between tip and sample, which we shall refer to as the tunneling location. As the tip is scanned a small distance across a narrow depression (valley) on the surface, the tunneling location may discontinuously jump from one side of the tip to the other. In this case, the measured potential would show a spatially abrupt jump, even though the actual potential varies

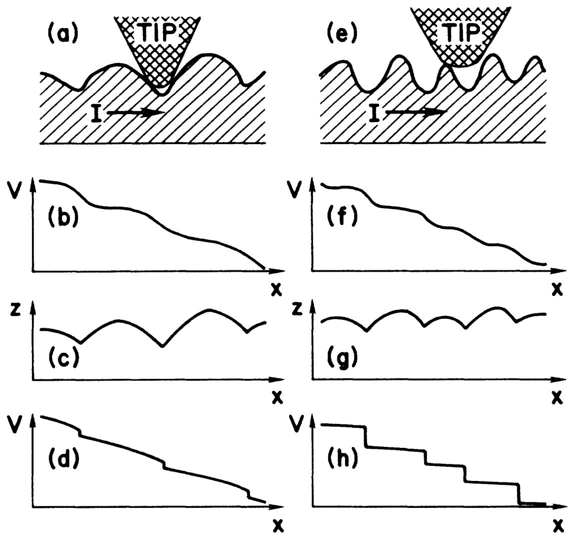

FIG. 1. Origin of measured signals as tip scans rough surfaces. (a) Cross section of sample and moderately sharp tip. (b) Actual potential vs position on surface. (c) Line scan of measured topography. (d) Line scan of measured potential. (e) Cross section of rough sample and blunt tip. (f) Actual potential vs position on surface. (g) Measured topography. (h) Measured potential.

smoothly with position. In Fig. 1(c) we sketch how a line scan of the *measured* sample topography might appear: Most of the topographic peaks are reproduced, but are slightly broadened by the tip and appear with somewhat reduced corrugation amplitude. These tip-related distortions in measured topographies are well known.[5] In Fig. 1(d) we sketch how the measured sample potential might appear. Close to a topographic peak, the measured potential is qualitatively quite similar to the actual potential. In the valleys, however, artificial abrupt drops in the voltage are present, which are spatially correlated with topographic minima.

In Fig. 1(e) we consider a more extreme case in which the sample surface is considerably rougher than the tip. The measured topographic peaks in Fig. 1(g) are considerably broadened and washed out, and their shape mainly reflects the shape of the tip rather than the sample. In Fig. 1(h) we see that the measured voltage consists of nearly flat terraces of constant potential separated by abrupt jumps. The flat terraces occur when the tip scans across a sharp peak on the sample: The tunneling location does not move very far on the sample even though the tip moves a large distance, and hence the measured voltage is nearly constant. Eventually, the tip will encounter another peak, and the tunneling location will discontinuously switch to another peak, causing an abrupt change in the measured potential.

Even more misleading behavior can result if the probe tip has multiple sharp asperities. In Fig. 2(a) we consider a double-tip scanning a rough surface. As the tip is scanned, the tunneling location can switch back and forth between the two asperities. The measured potential will appear to vary nonmonotonically and discontinuously with tip position, even if the actual potential in the sample varies smoothly and monotonically.

We now consider STP measurements we have made on several thin-metal-film samples which demonstrate the existence of some of these artifacts. In our custom-designed STM, the tip is scanned via a combined piezoelectric cross and tube assembly, while the sample is mounted on a Besocke-type[7] inertial walker. The apparatus and ac technique used to make the STP measurements has been described in detail elsewhere.[6] All measurements described here were taken in air, the STM operating with no vibration isolation and minimal electrical shielding, and using mechanically sharpened PtIr tunneling tips. Similar results were obtained with chemically etched tungsten tips.

Figures 2(b) and 2(c) show topographic and STP images simultaneously acquired from a 50-nm-thick AuPd alloy film deposited on an oxidized Si wafer substrate, with nominal current flow direction from left to right in the image shown. In the central part of the topographic image, we see hillocks of height ~2.5 nm and typical lateral dimension ~10 nm. In the corresponding STP im-

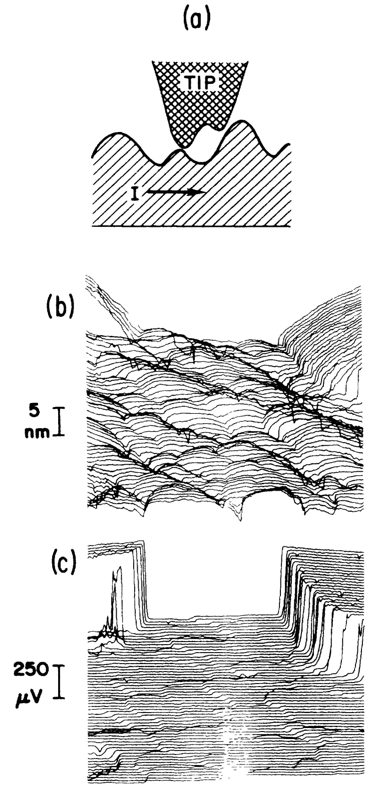

FIG. 2. (a) Schematic view of double-tip scanning rough surface. (b) Measured line scan of AuPd sample topography, covering roughly 60×60 nm^2 area. (c) Measured potential vs tip position for same area as in (b). AuPd film resistivity $\rho \sim 100$ $\mu\Omega$cm, residual-resistance ratio (RRR)~1.07, lateral ac current density $J_{\mathrm{ac}} \sim 2.5\times10^5$ A/cm^2 rms. In (c), the STP electronics were saturated by two large steps observed in upper corners of image, actual step heights larger than measured step height of 450 μV.

age we see flat terraces of nearly constant potential separated by abrupt jumps. The terraces correlate spatially with topographic hillocks, and the abrupt jumps generally occur at topographic valleys. This behavior is qualitatively similar to previous measurements on similar samples.[4,6] It may result from actual voltage drops in the sample due, for example, to highly resistive grain boundaries located at topographic minima,[4] or from tip-related artifacts, as described above, or some combination of both. Without a detailed knowledge of the actual tip shape it is difficult to say with certainty what this measured behavior represents.

In the upper corners of Figs. 2(b) and 2(c), we see quite different behavior: In the topographic image large mounds appear, while in the STP image large jumps in potential at least 450 μV high are observed. While it is possible that these large jumps are due to highly resistive structures in the film, a more likely explanation is that they result from a double-tip effect, as described above, in which the tunneling location discontinuously switches from one sharp asperity on the tip to another. In this case we can estimate the separation of the two asperities to be larger than $\sim$90 nm, simply by considering the size of the voltage jump and the average electric field in the sample.

Figures 3(a) and 3(b), taken from a different location on the same AuPd sample, offer more convincing evidence of a double-tip artifact. The topographic image shows hillocks similar to those in Fig. 2(b) and offers no particular indication that a double-tip effect may be present. However, the corresponding STP image is quite unusual: The voltage on much of the image appears to be near a certain value, while the rest of the image appears to be near a different, higher value. Many of the regions at the higher voltage appear as isolated plateaus. These isolated plateaus almost certainly result from a double-tip artifact: As the tip is scanned, the tunneling location switches back and forth between two widely separated parts of the tip (and the sample), causing the measured voltage to switch between two different levels. This conclusion has a significant impact on our interpretation of the *topographic* image shown in Fig. 3(a) as well: What appears to be an image of a particular region of the sample is in fact a *collage* of topographic features taken by two separate asperities on the tip, from very different parts of the sample. Here is a case where a tip-related artifact is much more obvious in an STP image than in the corresponding topographic image, and shows that performing STP may provide a means to detect tip artifacts in STM experiments of rough films. We note, for example, that a number of workers have used STM topographic measurements to draw quantitative and qualitative conclusions about the morphology of rough conducting films.[5,8] Much could be learned about the severity of tip artifacts in such measurements by simultaneously performing STP. As another example, we consider recent work by Gimzewski and co-workers,[9,10] who measured subnanometer spatial variations in the tunnel-current-induced light emission from silver films. While the abrupt, nm-scale variations in measured photons may well have been due to atomic-scale photon-emission sources on the surface, they could also have (in part) resulted from the discontinuous switching of the tunneling location on the sample as the tip was moved a small distance. One way to test for this latter possibility would be to simultaneously perform STP while measuring the emitted photon intensity. A discontinuous switch of the tunneling location from one part of the sample to another would generally be accompanied by an abrupt step in the measured potential.

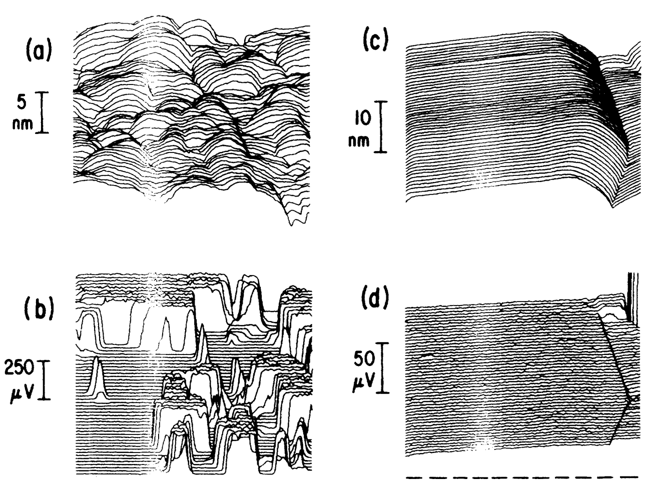

FIG. 3. (a) Measured topographic scan for different portion of AuPd film, covering 50×50 nm^2 area. (b) Measured potential for same area as in (a). (c) Measured topographic scan for epitaxial Au film, with $\rho\sim2.8\ \mu\Omega$ cm and $J_{ac}\sim6.5\times10^5$ A/cm^2. Scan covers a 100×100 nm^2 area. (d) Measured potential for same area. STP electronics not saturated in these scans.

Tip-related artifacts should, in general, be less severe on very flat samples. In this case the tunneling location will be confined to the particular part of the tip closest to the sample surface. Figures 3(c) and 3(d) were measured on a flat sample: a 100-nm-thick Au film epitaxially grown on a mica substrate. As shown in Fig. 3(c), this sample has regions which are extremely flat over large areas, but also exhibits some strong topographic features. In Fig. 3(d), we see that in regions corresponding to flat topographic areas, the sample potential varies quite uniformly, with a local voltage gradient quite close to the average gradient across the entire sample. On this flat surface, the tunneling location is localized to a well defined part of the tip, and the measured voltage accurately reflects the actual voltage on the sample surface. However, steps in the voltage are observed on the right-hand side of the image where strong topographic features are present.

Finally, we consider a specific case where actual variations of voltage on a rough sample surface can to some extent be distinguished from artificial behavior. The images shown in Fig. 4 were measured on a 10-nm-thick Au film which was grown on a liquid-nitrogen cooled, oxidized Si wafer substrate. The top-view grey-scale image of the measured sample topography in Fig. 4(a) shows that a characteristic kidney-bean shape roughly 10 nm across appears repeatedly over the image. This repeated image is a clear indication of a tip artifact: Each such shape was produced when a kidney-bean shaped part of the tip was scanned over a sharp protrusion on the sample surface.

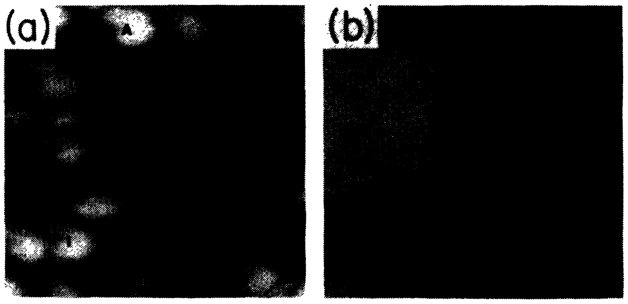

FIG. 4. (a) Measured topography of rough Au film, with $\rho \sim 14\ \mu\Omega\,\text{cm}$, RRR ~ 1.2, and $J_{ac} \sim 4.5 \times 10^5\ \text{A/cm}^2$. Scan covers $50 \times 50\ \text{nm}^2$ area, grey-scale range ~ 5 nm. Letters label various kidney-bean shaped features in topography (see text). (b) Measured STP image for same area: grey-scale range $\sim 100\ \mu\text{V}$.

We thus have a situation similar to that shown in Fig. 1(e), and hence we know that the measured potentiometric steps are artificially abrupt. However, since we see the *same* shape repeated over the surface, we also know that we *do not* have a multiple-tip effect, in which the tunneling location switches between widely separated asperities on the tip. Hence, we do know the approximate distance between the sharp protrusions on the surface responsible for the kidney beans, and we can approximate a locally averaged electric field between protrusions. For example, the average electric field between areas labeled *C* and *D* is approximately 40 V/cm, whereas it is less than 2 V/cm between *D* and *E*, and roughly 15 V/cm between *E* and *F*. Similar reasoning can be used to approximate the average electric field in other parts of the sample. We can thus conclude that the electric field in this sample is in fact highly nonuniform. A particularly large electric field does exist between areas *C* and *D*, and happens to occur across a particularly deep crevice in the measured topography. In this very thin granular film, it is quite plausible that this deep crevice and large electric field are due to a physical discontinuity in the film.

In summary, we have shown that tip-related effects can produce artificially abrupt steps and/or flat terraces in measured STP images of rough surfaces. These artificial features are qualitatively very similar to actual features one would expect from highly resistive structures in the films (e.g., grain boundaries, discontinuities), hence without a detailed knowledge of the actual tip shape, it is difficult to determine to what extent measured features represent actual as opposed to artificial behavior. We have, however, shown one example in which the approximate tip shape can be determined, allowing an approximate locally averaged electric field distribution to be determined within a sample. We have also considered several other images of rough metallic films in which tip artifacts can be identified. In many cases, tip artifacts are much more obvious in STP images than in the corresponding topographic images; hence STP may prove to be a general method to detect tip artifacts in STM measurements of thin conducting films.

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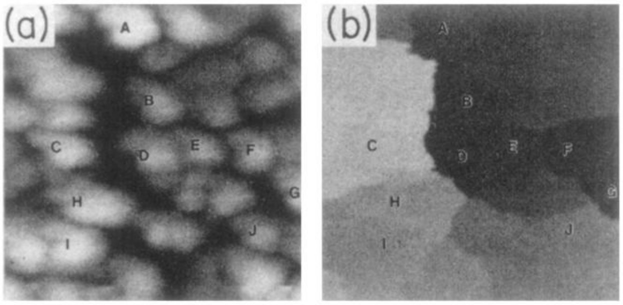

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