Nanoscale caliper for direct measurement of scanning force microscopy probes

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We show the possibility to measure the effective tip shape and the lateral resolution of a scanning force microscopy (SFM) probe on the nanometer-scale directly from SFM images of SiC(0001). On this surface there are grooves 10–100-nm-wide related to cleavage planes. The SFM tip penetrates the groove but does not reach the bottom since its side walls touch both rims. The width of the narrowest groove resolved is the lateral resolution. The apparent topography across a groove yields directly the tip radius of curvature in excellent agreement with the values estimated from scanning electron micrographs. © 1997 American Institute of Physics. [S0003-6951(97)00433-6]

The characterization of the actual tip apex geometry is a crucial issue in scanning force microscopy (SFM). When the actual size of the sample features becomes comparable or smaller than the effective curvature of the tip apex, the apparent topography might lead to artifacts which depend on the probe.\textsuperscript{1} This frequently occurs in the mesoscopic range (1–100 nm) which is relevant to the study of thin film growth, quantum dots, supermolecular and biological structures, nanotribology.\textsuperscript{2} The information on the tip structure is encrypted in the apparent topography, and despite the intrinsic nonlinear nature of the mechanism of contrast it is possible to reconstruct the actual tip geometry from the \textit{a priori} knowledge of the sample features. Vice versa, the reconstruction of the true topography, albeit with some restrictions, is possible once the tip shape is known.\textsuperscript{3–5} Usually, the tip shape is imaged by scanning electron microscopy (SEM), with the limitations due to the intrinsic SEM resolution, and the artifacts arising from electrostatic charge which builds up on scarcely conductive tip apices. Furthermore, the effective contact area between tip and sample in a SFM experiment might differ because of surface forces, contamination, and wear. Another fundamental issue is the definition of the lateral resolution of a SFM probe which has been raised by Bustamante and Keller (BK).\textsuperscript{1} The resolution would be the smallest distance between two sharp features which can be resolved above the instrumental noise level. The apparent SFM topography of rough substrates (e.g., SrTiO\textsubscript{3})\textsuperscript{6} and needlelike outgrowths on Si (Ref. 7) shows the quality of the tip apex, but does not measure directly the lateral resolution.

In this letter, we propose a straightforward method to measure directly the tip geometry at the apex from SFM images with an accuracy comparable to scanning electron micrographs, but with no need to access electron microscopy data. It relies on a peculiar type of nanometer-scale defect on the SiC(0001) surface, whose size measures the lateral resolution of the probe.

We used a substrate from a commercial 6H-SiC(0001) Si-terminated 1" wafer, (CREE Inc., Durham, NC), which has been etched on hydrofluoric acid 5% for 1 min, degassed in high-vacuum (10\textsuperscript{−6} mbar) for several hours, then brought to air for the SFM measurements. This surface is very hard and stable in time, and is suitable for being imaged by contact mode SFM both in ultrahigh-vacuum and ambient conditions. The current SFM imaging has been performed in air. The SFM image in Fig. 1(a) shows the morphology of a 5 \textmu m\times 5 \textmu m area. Although the surface is smooth (rms roughness is \textsim 1 nm on the 5 \textmu m scale length) it is crossed by a dense network of grooves, which are angularly distributed along specific crystallographic directions as it appears from the pattern in the two-dimensional (2D) power spectrum of the topography [inset Fig. 1(a)]. The most frequently occurring lines form a 30° relative angle and are parallel to macroscopic wafer cleavage directions [1100] and [1120].\textsuperscript{8} The grooves are typically a few tens-nm-wide and their apparent depth ranges a few nm [see detail in Fig. 1(b)]. It has been shown that cracks develop along the corresponding hexagonal lattice planes due to mechanical stress, and propagate a few hundred nm from the surface into the bulk.\textsuperscript{9} Therefore, the SFM tip does not reach the bottom but it penetrates deep into the groove until its walls touch the opposite rims.

Such a double contact situation is suitable both for “imaging” the tip shape and for assessing the lateral resolution of the probe, as it has been discussed earlier by Keller.\textsuperscript{3} In general, the apparent topography is the envelope of inverted tips touching the surface at a single point. Since the SiC basal plane (0001) and the (hk10) planes form an angle of 90°, the actual curvature of the surface near the edge is smaller than the tip apex curvature. Thus, inside the well the surface edge probes the tip resulting into an apparent topography which is the inverted tip image as depicted in Fig. 2. In the case of a parabolic tip shape, the onset of the double contact makes the topographical profile to exhibit a cusplike
minimum as the tip is retracted back off. Beyond the minimum, the topography trace is the other inverted half of the tip projected along the scan direction. The tip radius of curvature $R$, the apparent minimum depth $D_z$, the width $d$, and height difference $D_h$ between the rims are related by

$$R = \frac{d^2}{2(2D_z + \Delta h + 2\sqrt{D_z} \sqrt{\Delta z + \Delta h})}.$$  \hspace{1cm} (1)

Equation (1) still holds if the groove has smoother edges, provided that the magnitude of the local slope is larger than $d/2R$, or equivalently for a step-wise slope if the terrace width $\Delta x \leq (2R\Delta z)^{1/2}$. Thus, each groove is a caliper which measures the effective width of the tip along a given direction, while the topography minimum yields the height from the apex. In principle, it is possible to reconstruct the full 3D tip apex structure by exploiting the large number of different projections directly from the topographical profiles across grooves at different orientation. Here we work out two cases using a 1D analysis.

The SEM micrographs in Fig. 3 show the projection of two tips on the $xz$ plane, where $x$ is the fast scan direction which is almost normal to the groove we selected and $z$ the SFM height coordinate. Figure 3(a) shows a Si ultralever (Park Scientific Instruments, Sunnyvale, CA) probe with a parabolic shape, and a radius of curvature $R = 25–30$ nm measured at the apex from the SEM micrograph in Fig. 3(a). The second is a SiN$_3$ pyramidal tip (Topometrix Inc., Santa
Clara, CA) with 1:2 aspect ratio and $R \approx 80 \pm 120$ nm [Fig. 3(b)]. The uncertainty depends on the thresholding of the white levels in the micrograph, and might be due to the outcome of electrostatic charge build up during the SEM imaging.

Starting from $2 \mu m \times 2 \mu m$ SFM images with 400 pixel per line, we took section profiles across a groove and normal to it. The fluctuations along these profiles range between 0.4 and 1 nm, due to the intrinsic features and the noise level. Thus, the relative error is lower if one takes the largest (30–80 nm) and deepest groove (6–10 nm) for the analysis. Representative results for the two tips are shown in Fig. 4. The absence of flat smooth bottom supports the situation depicted in Fig. 2, and the sharper tip yields higher aspect ratio traces. For each profile, we take the surface baseline as the mean height measured to the left and right side of the groove. We measure the depth at the minimum $\Delta z$ with respect to the baseline, and the width $d$ as the horizontal distance between the intersection points of the well with the baseline. If the height difference $\Delta h$ between the left and right mean height values is less than the rms fluctuation, we neglect it in Eq. (1). We estimate the radius of curvature $R$ from a statistical average over at least 10 profiles, and the error by propagation of the mean absolute errors on $d$ and $\Delta z$. We find $R = 24 \pm 5$ nm and $R = 93 \pm 25$ nm for the two tips in Fig. 3, respectively, in excellent agreement with the estimate made on SEM micrographs. As a crosscheck of our method, the width of the narrowest line which can be resolved in Fig. 1(b) is $d \approx 11$ nm, which implies $R \approx 20$ nm for a noise level of 0.5 nm. This is the lateral resolution of the probe in Fig. 3(a) according to BK definition.

In conclusion, we have shown that the effective tip shape and the lateral resolution can be directly measured from SFM images of the SiC(0001) surface. Such observations are straightforward on the micron scan range, which is sufficient for obtaining an accuracy of $\approx 20\%$ on $R$. The relative error can be lowered by acquiring images at higher resolution. Our results suggests that this method might be a promising standard for direct measurements of ultranarrow SFM probes, such as carbon-grown tips and nanotubes.

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