z calibration of the atomic force microscope by means of a pyramidal tip

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I. INTRODUCTION

The atomic force microscope (AFM), developed in 1986 by Binnig and co-workers,1 is a very promising new tool for investigating surfaces with high spatial resolution. Unlike its predecessor, the scanning tunneling microscope (STM), the AFM measuring is not restricted to conductive or semiconductive surfaces. This opens up to the study of a large variety of new areas—especially those involving conductors,2 nonconductors,3 and molecular films4—to an extent that has since been limited by the instrument itself (self-imaging) is presented. The self-imaging is accomplished by scanning the probe tip across a sharper tip on the surface. By using a pyramidal probe tip with a very well-defined aspect ratio, this technique provides an excellent z-calibration standard for the atomic force microscope.

II. BASIC IDEA

In this letter, we introduce a new way to measure the tip profile and to apply this for a direct z calibration of the AFM. The idea is to scan the probe tip which has a well-defined aspect ratio across a sharp tip on the sample with a larger aspect ratio. By doing this, the probe tip itself is imaged (self-imaging) and from the well-defined aspect ratio it is now possible to absolute calibrate the z axis from the scan range regime, is to scan a grid (possibly a double grid) with an appropriate lattice constant which can be manufactured, e.g., by means of e-beam lithography or holographically and calibrated using diffraction of laser light. There is, however, still a lack of a well-suited standard for the z calibration of the instrument.
FIG. 1. SEM micrograph showing the InP surface after processing by means of reactive ion etching. The surface has a high density of ≈100-nm-wide free-standing columns.

rate for (111) planes which self-terminates when the volume of the removed Si is bound by (111) planes. This procedure provides a nearly perfect pyramidal tip (well defined aspect ratio=\(v/2\)) with a well-defined orientation with respect to the cantilever. Cantilevers of this type are now commercially available.15

Figure 1 displays a scanning electron microscope (SEM) micrograph of an InP surface with a high density of free-standing columns with vertical sidewalls (sharp tips). The typical dimensions of the columns are laterally of the order ≈100 nm and vertically of the order ≈500 nm. This sample was prepared by means of reactive ion etching (RIE), which is a well-established plasma etching technique for fabricating small semiconductor devices and structures.16 In the present investigation, a parallel-plate reactor with a 13.6 MHz rf excitation applied to the bottom electrode (cathode) and gases let in through a shower head of the top electrode provided the plasma. A methane-based plasma (mixture of CH\(_4\)/H\(_2\)) was used for the etching.16 During etching in this type of plasma, a polymeric film is deposited on the surface with a thickness which increases with an increase in the concentration of CH\(_4\) and a decrease in rf power.16 This polymeric film acts as an etch mask decreasing the etch rate dramatically in areas covered with the film. By choosing a suitable set of etching parameters, it is possible to obtain a micromasking effect leading to the surface shown in Fig. 1. In this case, the rf power was 100 W with 15 sccm CH\(_4\) and 50 sccm H\(_2\) at 15 mTorr for 72 min.

III. RESULTS AND DISCUSSION

The AFM measurements were performed with a fully automated commercial instrument.17 The force detection part of the instrument combines the microfabricated cantilever15 with a beam-deflection sensor.18 Figure 2 shows a constant force topograph obtained when scanning an area of 2400×2520 nm\(^2\) on the sample (Fig. 1) with a pyramidal probe tip. The topograph clearly reveals that with this sample we in fact are able to image the pyramidal probe tip as described above. For the calibration of the x and y axis, we have used a grid made holographically with a lattice constant of 500±1 nm.

Figure 3 presents a top view of the probe tip. A cross section of the tip through the inserted line is shown in the two insets. It appears that there is a certain limitation, determined by the finite size of the columns, to the degree of detail with which we can study the very end of the probe tip. It is possible, however, to find an upper limit for the radius of curvature of the probe tip. This is found to be less than 400 Å in agreement with SEM investigations.14

From the linear part of the tip profile, determined by the (111) plane in bulk Si, an absolute calibration of the z axis can be achieved (provided that the x and y axes are calibrated) due to the well-defined aspect ratio. There is one problem which must be considered first. If the probe tip is not tilted with respect to the surface normal, the measured aspect ratio \(a^*\) is simply determined by the aspect ratio of the (111) plane [\(\tan(\Theta_0)\) cf. Fig. 4] and the z-calibration factor \(C_z\), i.e.,

\[
a^* = \frac{\Delta Z^*}{\Delta X} = \frac{\tan(\Theta_0)}{C_z} \frac{v/2}{C_z} = \frac{v}{C_z}.
\]
where the * indicates that the z axis is not calibrated. In general the probe tip is tilted an angle $\Theta$ with respect to the surface normal which will influence the measured aspect ratio (see Fig. 4). This problem can be eliminated by measuring the aspect ratio for two opposite sides (index 1 and 2) of the pyramidal probe tip, whereby two equations with two unknowns, the z-calibration factor $C_z$ and the angle $\Theta$, are obtained. With the notation in Fig. 4 we get the following expressions:

$$\begin{align*}
\alpha_1 &= C_z \alpha_1^* = \tan(\Theta_0 - \Theta), \\
\alpha_2 &= C_z \alpha_2^* = \tan(\Theta_0 + \Theta),
\end{align*}$$

where $\alpha_i = \Delta Z_i / \Delta X_i (i = 1, 2)$ is the aspect ratio after calibration of the z axis. Using the addition formulas, (2a) and (2b) can be rewritten as a quadratic equation in $\tan(\Theta)(=x)$:

$$\begin{align*}
\left(\frac{\alpha_1^*}{\alpha_2^*} - 1\right)x_0x^2 + \left(\frac{\alpha_1^*}{\alpha_2^*} \left(x_0^2 + 1\right) + x_0^2 + 1\right)x \\
+ \left(\frac{\alpha_1^*}{\alpha_2^*} - 1\right)x_0 = 0,
\end{align*}$$

where $x_0 = \tan(\Theta_0)$.

Inserting the measured aspect ratios (see insets in Fig. 3) of $\alpha_1^* = 1.41$ and $\alpha_2^* = 3.36$ into Eq. (3) results in the solution $\Theta = 11.4^\circ \pm 1.0^\circ$. Now, Eq. (2a) results in the calibration factor $C_z = 0.68 \pm 0.04$. This kind of calibration can of course be performed for different $(x,y)$ values, thus giving an $(x,y)$ mapping of the z response. Such a mapping is of course extremely useful for making a complete image correction.

In summary, we have presented a new technique for imaging the profile of an AFM tip. When using a probe tip with a well-defined aspect ratio, this technique provides a new method to calibrate the z axis in a regime which is very important for many technological applications such as measurements of surface roughness, etc. The ease by which this calibration is performed is also appealing, as the AFM technique spreads out to a wide community of users.

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