SIB61
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A good practice guide on crystalline step height standards

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1. Introduction

Atomic step heights have a large potential to serve as high quality transfer standards for the calibration of atomic force microscopes (AFM), as the atomic lattice spacing has been measured accurately using X-ray diffraction (XRD) or X-ray interferometry. The sub-nanometer calibration of AFMs is important for nanoscale roughness measurements, and for nanoparticle sizing of diameters less than 5 nm. This report presents a good practice guide on the use of crystalline step height standards for AFM calibration at a sub-nanometer level.

2. Crystalline step standards

A crystalline step height standard provides well-defined single atomic steps over a micrometer-sized area. This makes it possible not only to calibrate the z-axis of the microscope from the crystalline lattice, but also, the characterization of nonlinearities introduced by the positioning system of the AFM. Figure 1 shows an example of nonlinearities caused by the positioning system of an AFM when measuring a flat Si wafer. As can be seen, although the sample under test is supposed to be flat, the nonlinearities of the AFM and possible tilting of the sample when placed on the stage of the AFM introduce changes in height that can be comparable to the crystalline lattice when scanning over several micrometers.

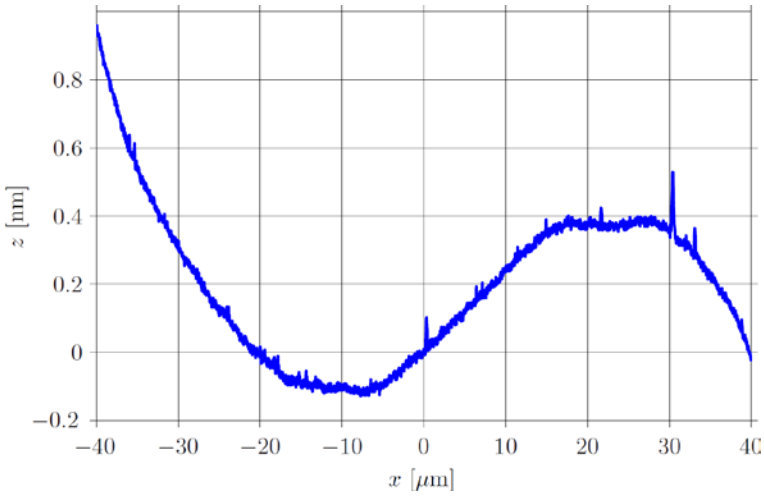
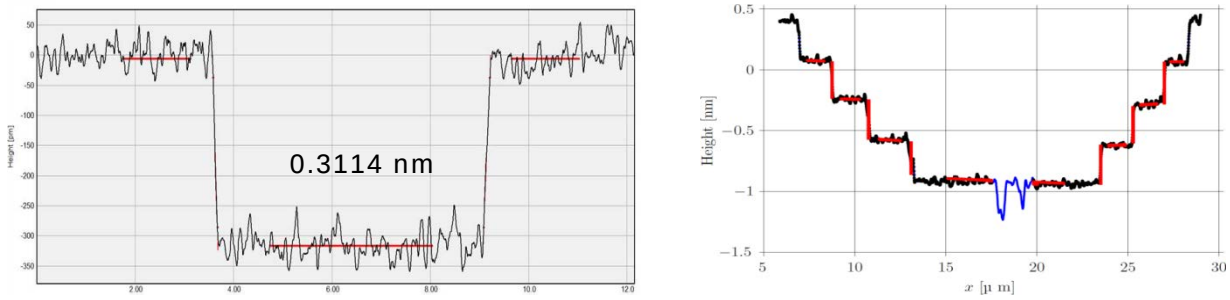


Figure 1 – Example of nonlinearities introduced by the positioning system of an AFM.

3. AFM calibration using crystalline step standard

Crystalline structures have the potential to be used as calibration standards based on their lattice parameters. A method for calibrating AFMs using Si(111) single steps is described in what follows. This is an offline method in analogy with the international standards for determining a symmetric step height (ISO 5436), see figure 2. The ISO method requires a flat plateau on each side of the step. The direct implementation of this standard is not possible in this case as the crystalline step standard is made of Si(111) single steps that form an amphitheater structure.



(a) *(b)*

Figure 2 – Single step measurements, (a) according to ISO 5436, (b) calibration method based on a crystalline step standard.

As can be seen in panel (a), the noise contaminating a single step measurement makes it difficult to measure the step height with a low uncertainty, that is, with an uncertainty that is at least ten times smaller than the measured quantity. Apart from using averaging procedures for reducing the influence of noise, a very effective way of reducing the uncertainty on the measured step height is to fit a model to

the measurement data. In this case, the lattice of a crystalline step standard provides the possibility of defining a model with very well-defined properties:

- Atomic step heights are invariant, and thus, all the measured steps must have the same height.
- Flat plateaus between steps. This means that any tilting in the measured profile comes from incorrect leveling of the sample, contamination of the sample (e.g., oxide layers) or nonlinearities of the microscope.

These two well-defined constraints can be used to correct the measured data as illustrated in figure 3.

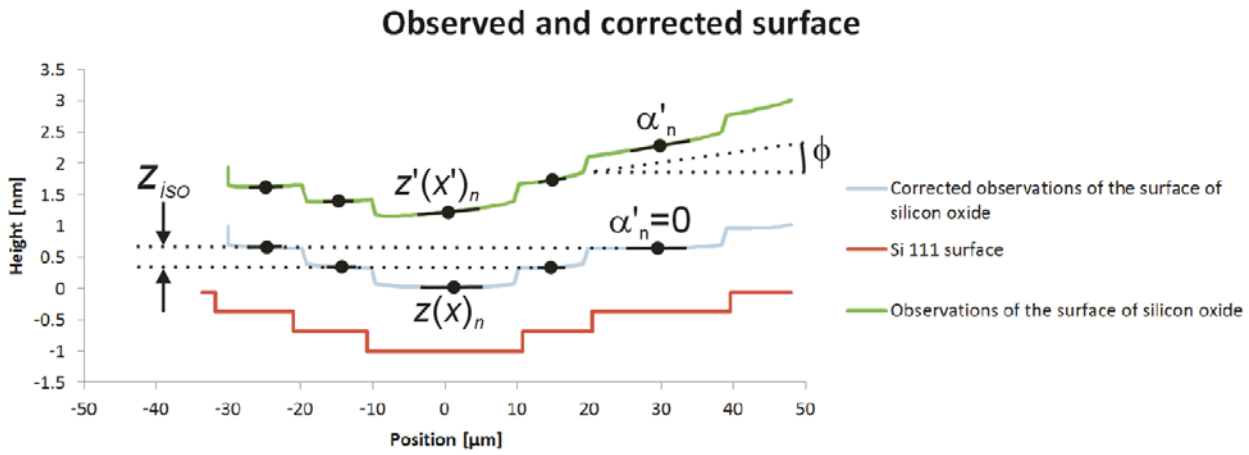


Figure 3– The lattice of the crystalline step standard makes it possible to correct the measured surface with well-defined constraints.

The correction algorithm must first detect the atomic steps in the measured profile and then identify the middle points between each pair of steps. Figure 4 shows this for a staircase profile.

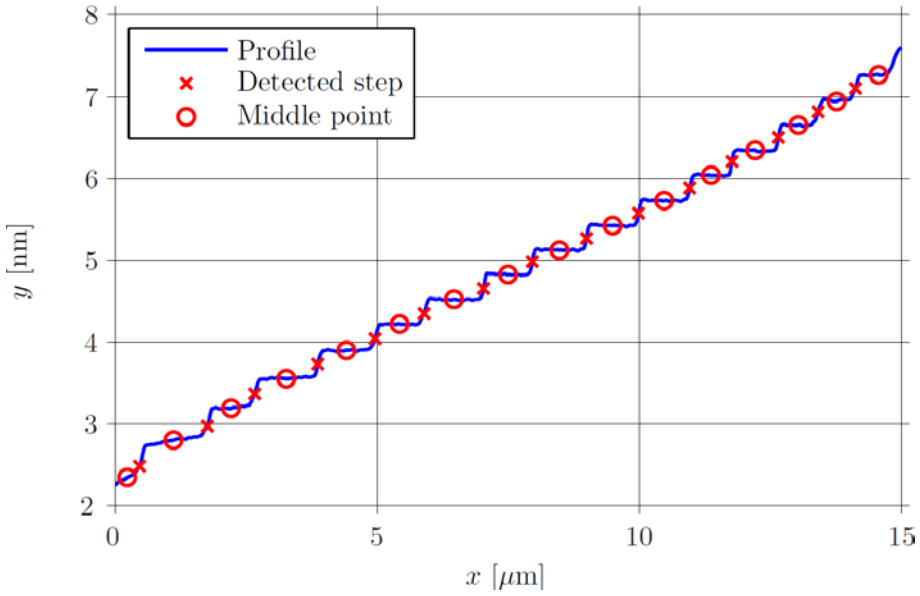


Figure 4 – Example of a staircase profile.

The steps can be detected by means of an edge detection algorithm that uses the derivative of a Gaussian kernel as an optimal edge detector [1], see figure 5. The detection algorithm can be adjusted depending on the quality of the measured profiles, for instance, by tuning the width of the Gaussian kernel or the threshold used to localize the maxima/minima of the convolved signal.

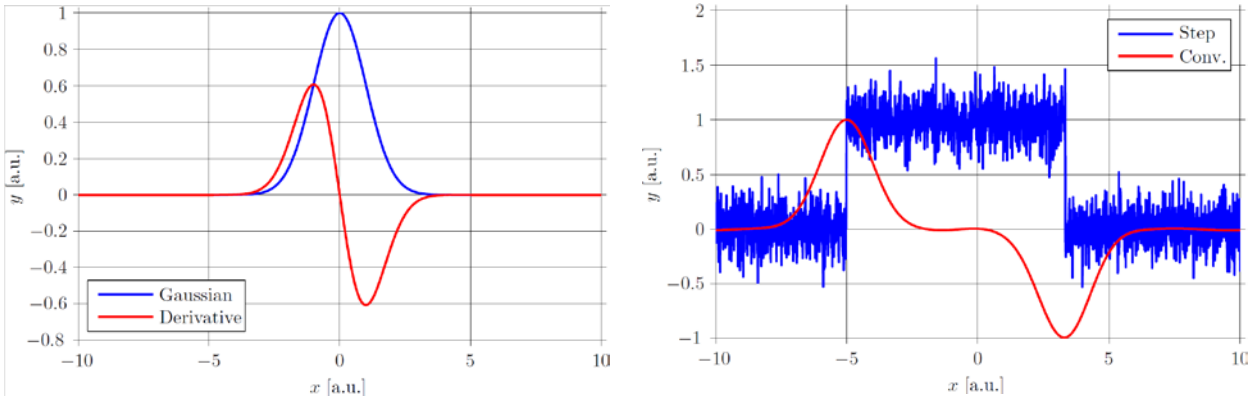


Figure 5 – Convolving an edge with the derivative of a Gaussian kernel yields maxima/minima at step edges. On the left hand side, a Gaussian kernel (in blue) and its corresponding derivative function (in red). On the right hand side, a step function contaminated with noise (in blue) and the result of convolving the noisy step with the derivative of a Gaussian kernel normalized to unity (in red).

The middle points between the steps are determined by fitting a line to 50% of the data points located at the central part of the plateaus. The slope of these fitting lines is also used to compensate for nonlinearities.

Once the steps are detected and the middle points together with their slopes are estimated, this information can be used as input parameters of a least-squares optimization algorithm that determines the actual step height of the sample with lowest uncertainty and fulfilling the constraints imposed by the crystalline step standard stated previously. This least- squares optimization algorithm can be summarized as follows:

- Nomenclature: the main variables of the algorithm are denoted as (see ref. [2] for further details)
  - $\boldsymbol{\zeta}$: the (exact) values of the quantities being measured directly or known a priori, for example, physical constants.
  - $\boldsymbol{z}$: actual values obtained by measurement or tables that estimate the values of $\boldsymbol{\zeta}$.
  - $\boldsymbol{\Sigma}$: covariance matrix associated with $\boldsymbol{z}$.
  - $\boldsymbol{\beta}$: quantities for which no prior information is available.
  - x'' and z'': x and z axis coordinates of the AFM.
- The relevant quantities $\boldsymbol{\zeta}$ for which prior information is available are identified: The height h of the steps in the measured standard, the heights z'' of the identified step middle points, and the slopes a'' of these steps. The heights and slopes refer to the coordinate system (x'', z'') of the AFM.
- Best estimates $\boldsymbol{z}$ and associated covariance matrix $\boldsymbol{\Sigma}$ are assigned to the quantities $\boldsymbol{\zeta}$.

- The relevant quantities $\boldsymbol{\beta}$ for which no prior information is available are identified: The parameters $\boldsymbol{A}$ in a transform $x'=T(\boldsymbol{A}, x'')$ used to correct for non-linearity in the x''-axis of the AFM; the parameter B in the transform $z''=Bz'$ used to calibrate the z''-axis of the AFM, the tilt ϕ of the step standard relative to the transformed coordinate system (x', z'), the height z_0 of the lowest step of the standard in a coordinate system (x, z) in which the steps are vertical to the z-axis.
- The constraints $\boldsymbol{f}(\boldsymbol{\beta},\boldsymbol{\zeta}) = \boldsymbol{0}$ between the quantities $\boldsymbol{\beta}$ and $\boldsymbol{\zeta}$ are formulated: First transform the middle points x'', the heights z'' and the slopes a'' to corrected middle points x', heights z' and slopes a' in the coordinate system (x', z'). Then transform the middle points x', heights z' and slopes a' to heights z and slopes a in a coordinate system (x, z) rotated an angle $-\phi$ relative to the coordinate system (x', z'). The heights z should then be equal to $z_0 + i\,h$, where $i = 0$ for the lowest step, $i = 1$ for the two neighboring steps, etc.
- Best estimates of the quantities $\boldsymbol{\beta}$ and $\boldsymbol{\zeta}$ are found by minimizing the chi-square $\chi^2 = (\boldsymbol{z}-\boldsymbol{\zeta})^t\boldsymbol{\Sigma}^{-1}(\boldsymbol{z}-\boldsymbol{\zeta})$ subject to the constraints $\boldsymbol{f}(\boldsymbol{\beta},\boldsymbol{\zeta}) = \boldsymbol{0}$ as described in ref.[2].

4. Conclusion

The guide show that a few symmetric step (amphitheater) heights can be used to calibrate the height sensitivity in the sub-nanometre range in a robust and reliable way quantifying an upper limit for the influence of possible sub-nanometre hysteresis, creep, tip-sample interaction and other sub-nanometre influence parameters. This assessment is typical done over a small segment of the lateral scanned profile. It will typical be found that e.g. for a small segment of the scanned profile compared to the maximum scan range the sub-nanometre nonlinearities are small for the microscope used. However, for large segments of the scanned profile compared to the maximum scan range, the influence of nonlinearities and sub-nanometre hysteresis could to be larger and other influence parameters such as crosstalk between lateral and vertical scanning could be significant. It will typical be found that e.g. these sub-nanometre nonlinearities and crosstalk are difficult to deduce from the parameters found on larger steps, gratings and flat surfaces and they would have been very difficult – if not impossible - to measure without a symmetric (amphitheater) step height.

5. References

[1] J. Canny, “A computational approach to edge detection”, IEEE Transactions on pattern analysis and machine intelligence, Vol. PAMI-8 (6), 1986.

[2] L. Nielsen, “Evaluation of measurements by the method of least squares”, in Algorithms for Approximation IV, J. Levesley, I.J. Anderson, J.C. Mason (Eds), University of Huddersfield, 2002, 170-186.

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