Calibration method of curvature distortion in step height measurement by atomic force microscopy

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Abstract. Atomic force microscopy (AFM) is widely used to characterize the surface topography in nanomaterials and biology research since the high resolution in 3 dimensions. Height measurement is important in the investigation of graphene thickness. Due to some AFMs use tube scanner, the curved scanning distortion will cause height measurement error. Step height standards calibrated by metrological AFM are used to calibrate the Z axis of conventional AFMs. However, for step structure with large width, the method specified in ISO 5436 is not suitable. The distortion of the curvature scanning is discussed for different samples, and for large step width the influence is significant. So the substrate correction method is proposed to eliminate the scanning curvature and the comparison with several other fitting correction methods is also discussed.

1. Introduction
Due to the 3-dimension characterization capability with high resolution, atomic force microscopes (AFMs) are commonly used to image the material and biological surface topography, and also some mechanical and electrical properties with special tips and modules[1,2]. The height measurement is an importance parameter for AFM in the measurement of the thickness of nano material like graphene [3,4]. The piezoelectric scanner in AFMs needs to be calibrated for the hysteresis for qualitative measurement of the height of nano dimensional structures. The calibration artefacts standards are traced to SI unit by metrological AFMs which can directly trace the measurement value to the laser wavelength by three interferometers in X, Y and Z axis of the scanner. Some AFM utilizing tube scanners has bow scanning distortion. This effect can be eliminated by AFMs with special scan heads or flexture stage scanning AFM [5,6]. However, the bow distortion in tube scanning AFM has to be corrected by fitting methods. The scanner calibration methods for height measurement have been studied, however the methods to correct the bow distortion has not been studied thoroughly. In this paper the influence of the bow distortion of tube scanner on step height is studied and some step height analysis methods are discussed. A substrate correction method is introduced to accurately measure the step height by correction the bow error.

2. Methods
2.1. Experimental materials
Step height standards artefacts with different height are measured with AFM (Dimension Icon, Veeco). The AFM scans the sample with a tube scanning head. The Z axis of AFM scanner is calibrated with standards. The artefacts are Cr coated on quartz substrate. The maximum scanning range of the AFM
is 90 μm in lateral direction. A serial of step height standards are measured to investigate the influence of the bow error on the height measurement.

2.2. Step height evaluation methods

The step height is usually defined according to ISO 5436 as shown in figure 1. The lateral length of measured step profile is at least 3 times the width of the step $W$. The height of the step is expressed as $h = (A+B)/2-C$, where A, B and C are the average height of the $w/3$ region of the profile. According to the ISO 5436, the width of the step is limited by the scanning range of AFM when using a standard to calibrate the step height value. For some samples the width is 100μm, larger than the scanning range of common AFMs, then only one sidewall of the step can be scanned in one scanning profile. The definition is not applicable for these samples. For the Si atomic layer height standards with single edge artefacts like terrace structure, different methods are proposed to evaluate the step height [7].

![Figure 1. Step height definition according to ISO 5436.](image)

2.2.1. Polynomial fitting method

For tube scanning AFM, the measured height is influenced by the bow error. The fitting methods proposed by Yang et al can be used to corrected bow error [8]. The image was processed line by line. First, a profile curve is obtained by averaging several consecutive lines, which significantly reduces the burr of the curves. Second, the Sobel edge detection algorithm was used to extract the edge of step of the curve, and then the curve is bisected by the edge (or as the complete step divided into three parts). The step part was shifted down successively according to a unit shifting distance to obtain the shifted curve (reasonable number of moving steps). For each move, 6 points around the edge were removed for the edge effect by the tip and the higher-order polynomial was fitted by the least square method. Then the residual of the shifted curve and the fitting curve was calculated, and the minimum value of the residual was taken to obtain the optimal shifted curve and the number of moving steps. Finally, the original curve minus the optimal shifted curve gives the step curve, which removes the bow error. The product of the number of steps and unit shifting distance is the height of the step.

2.2.2. Substrate subtraction method

For the bow error is caused by the instrument and not related to the sample, a substrate subtraction method is introduced here to eliminate the influence of the scanner bow error. The sample is scanned with AFM successively with the step is shifted a certain range laterally to scanning the left and right sidewall respectively. Then the two images of the sample are subtracted to obtain the final step height profile as shown in figure 2. Then the bow distortion can be eliminated by subtracting the two images. This method is direct and does not require complex programs. Here we used the SPIP software. Then the residual profile is the step difference as shown in figure 2. The height can be evaluated with standard methods from the corrected curve.

![Figure 2. Step height evaluation method for bow distorted image.](image)

3. Results and discussion

The bow error due to the tube scanning in AFM is demonstrated firstly. A 50 nm step height is
measured and the 3D topography is shown in figure 3. The distortion can be obviously seen and a section profile of the topography cross the step is also shown in figure 3.

**Figure 3.** Step height image of a 50 nm height sample.

The standard w/3 evaluation method is applied to the results and the height is 51.8 nm. However, due to the bow error of the scanner, the height is not accurate. The substrate is quadratic and with the polynomial fitting method, the substrate is removed from the raw data. The height is 45.7 nm. By shifting the sample laterally and get another image, the substrate can be eliminated and the height is 46.0 nm, consistent with the polynomial fitting method. 100 nm and 20 nm step height standards are also studied and the results demonstrated that the step heights calculated with the two methods are equivalent as shown in table 1.

| Method                  | W/3  | Polynomial Fitting | Subtracting Substrate |
|-------------------------|------|--------------------|-----------------------|
|                         | 23.1 | 17.7 nm            | 18.3 nm               |
|                         | 51.8 | 45.7 nm            | 46.0 nm               |
|                         | 91.2 | 84.5 nm            | 85.6 nm               |

The methods are also used for a 44 nm height sample with width larger than the scanning range of the AFM. The scanned result and the fitting process are shown in figure 4 and figure 5.

**Figure 4.** Scheme of shifting fitting correction method. (a) The original scanning profile. (b) The curve shifted by a certain distance. (c) The red curve fitted and the curve shifted by an optimal distance. (d) The adjusted step profile.

**Figure 5.** The relationship between the shifting distance and residual error.
As can be seen from figure 6, as the unit shifting distance increases, the step height fluctuates around the result measured by the minimum unit shifting distance, and the error is getting larger and larger. The error of unit shifting distance less than 0.9 nm will not exceed 0.2 nm, and the error will not exceed 0.025 nm when the unit shifting distance is less than 0.1 nm. Considering the error and operation time, 0.1 nm is selected as the unit shifting distance. The result is 43.4 nm.

Figure 6. The influence of the unit shifting distance during the polynomial fitting on the step height.

For the step sample wider than the AFM lateral scanning range, the two edges of the step are scanned respectively with AFM. Then the two images are subtracted with each other. The residual curve is shown in figure 7 and then the step is obtained by removing the bow error. Just like the shifting fitting method, the continuous averaging multiple lines, the middle section was shifted down or up according to certain unit shifting distance, and the polynomial of order 1 was fitted to calculate the residual of the shifted curve and the fitted curve. Finally the step profile curve minus the optimal curve gets the fine step. The result is 44.4 nm. The height can also be obtained by fitting the 3 step with linear fitting and then the difference is the height between steps.

Figure 7. Scheme of substrate removal correction method. (a) The original scanning profile of the left edge. (b) The curve of the right edge. (c) The profile of step with the bow error removed.

4. Conclusion
A method to eliminate the bow error of tube scanning AFM is introduced. The method is compared with the common standard W/3 methods and the polynomial fitting method by measuring some step height standards. The step height results of the method are consistent with the fitting method within 1 nm. The method is also applied to sample with step structure wider than the scanning range of AFM. This method provided a convenient way to correct the bow error and calibration method for AFM.
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