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ORCID® iDs	Perawat Boonpuek - https://orcid.org/0000-0001-8815-6920

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Determination of the Radii of Coated and Uncoated Silicon AFM Sharp Tips using Height Calibration Standard Grating and Nonlinear Regression Function

Perawat Boonpuek *[1], Jonathan R. Felts ‡[2]

Address: [1]School of Manufacturing Engineering, Institute of Engineering, Suranaree University of Technology, 111 University Avenue, Muang, Nakhon Ratchasima 30000, Thailand and [2]Advanced Manufacturing Laboratory, Department of Mechanical Engineering, College of Engineering, Texas A&M University, College Station, Texas, 77843, USA

Email: Perawat Boonpuek – perawat@sut.ac.th

* Corresponding author

‡ Equal contributors

Abstract

AFM sharp tips have been used to characterize nanostructures and quantify the mechanical properties of the materials in several areas of research. The analytical results can show unpredicted errors if we do not know the exact values of the AFM tip radius. There are many techniques of in-situ measurements for determining the actual AFM tip radius but limited to uncoated tips. This paper presents an alternative and simple method to determine the radii of coated tips and the uncoated tip. The Pt-coated, Cr-Au coated, and Si uncoated tips were used to scan on the calibration standard grating in AFM contact mode with sub-nano loading to get the curved scan

profile of the edge corner of the grating structure. The data points of the curved profile of each tip were fitted with the nonlinear regression function to estimate the curvature radius of the tip. The results show that the estimated radius of the coated tips is in the range of nominal values provided by the tip manufacturer while the estimated radius of the Si uncoated tip is bigger than the nominal radius due to tip blunting during the scan. However, this method yields an accurate estimate of the tip radius with the low value of root mean squared error from curve fitting results.

Keywords

AFM tip calibration; nonlinear regression curve fitting

Introduction

Atomic force microscopy (AFM) with the sharp tip is typically used to characterize nanostructured materials for example graphene, carbon nanotube, nanoscale semiconductor, bio-materials, chemical molecules and measure some mechanical properties such as surface stiffness, adhesion, friction, electrostatics, electrowetting, etc.[1-4] In contact mode scanning, a contact area between the AFM tip and the sample, which depends on the size of the tip radius, defines how accurately the AFM tip determines those properties and the shape of fabricated micro- and nanostructures. The contact radius of the tip is a key variable for calculating the stiffness and Young's modulus of the material by fitting force curves with contact mechanics models and extracting the adhesion and friction forces.[5,6] If we do not know the exact value of the tip radius, the sample image with an observation of scanning frequency and the calculation results are not accurate. This indicates that the measurement results are strongly dependent on the geometry of the AFM tip. [7, 8].

The AFM coated tips for example a Pt-coated tip (HQ:NSC18/Pt, its radius: < 30 nm for Electrical, Force Modulation AFM Probe) and a Cr-Au coated tip (HQ:NSC16/Cr-Au, its radius: < 35 nm for Tapping Mode AFM Probe with Long AFM Cantilever) produced by MikroMasch.[9] have estimated nominal tip radii lower than 30 - 35 nm. But that is not the true radius of the tip. Therefore, the AFM users need to determine the actual tip radius before using it to probe micro-and nanostructures and measure the mechanical properties of the sample. From previous studies, Tip shape and radius at the sharp end could be measured using a tip characterizer and tip qualification or standard calibration grating. One of those found out the tip geometry by using a well-known sharp-edged silicon structure which includes height patterns with a certified pitch on a nanostructure plate. Three types of AFM tips (silicon nitride, silicon, and high aspect ratio tips) attached to the AFM tip holder in the AFM machine scanned through the calibration pattern and simultaneously the AFM measurement signal showed the tip path profile as a real geometry of a fabricated microstructure. The tip radius was obtained from the curvature radius of the curve profile at the top corner of a grating structure (a fabricated square column). Then, the tip radius was estimated by fitting a drawn circle in computer software fitted to the AFM tip scanned curve file.[10] Thereby, the obtained tip radius directly depends on the manually drawn circle which can vary the tip curve profile with line points on the fixed line of such circle, leading to some deviation of the tip curve profile radius. Another similar characterization method for the tip shape determination is by scanning AFM images of the tip in linewidth across a test characterizer to get a scanline profile of the tip for comparison with the result from their proposed geometry model of the tip profile that was calculated by using a tip slope angle as the tip scanned on the grating structure. Then, the tip cone angle incorporated with the tip slope angle gave the scanline profile of the tip with its slope angle for tip radius calculation. But their geometry-based method of the tip profile relied on the tip slope angle which varied upon different types of tips and the sample.[11] From the noncontact scanning method, the observation of force curve signal amplitude proposed by C. Maragliano et al, 2015 showed that the smaller sharp tip (small tip radius), revealed the smaller value of free amplitude transition of the attractive to repulsive force curve than the tip with a larger radius. The capacitive tip was also used to determine and evaluate tip radius through geometric relationship equations and then both results methods were compared with the nominal values of the tip radius given by manufacturers. According to the curve fitting result, both methods provide a reliable tip radius estimate.[12] However, this approach is not related to a physical aspect of AFM tip under contact scanning measurement in which the tip end shape needs to be extracted. For non-sharp tips, determining tip geometry if there is a lack of accurate knowledge on the real geometry of the characterizer pattern is difficult. Because the AFM scanline profile of the flared or spherical end of the tip goes offset from the actual vertical and sloped sidewalls of the characterizer (a calibration grating) when the tip moves through the corner edge. To achieve an accurate estimation of the radius, the tip scanned profile reconstruction method has been developed for fitting the measured points to the morphological filter surface of the characterizer.[13] Yet, this reconstruction technique may not be applicable for a very sharp tip that can make a smaller difference in the scanned profile offset at the corner of the characterizer.

A method for characterizing the blunt tip was also demonstrated. Nanoindentation techniques using four blunt tips on the AFM cantilever were performed in a normal loading process on a soft PVC sheet for 30 nm depth to obtain sufficient data of force curves. The blunted tips were imaged by SEM immediately after running the nanoindentation process in the AFM program and then each tip radius was determined by fitting the force loading curves with the Hertz model equation using the indentation depth and the reduced modulus.[14] Real-time measurement of tip radius based on analysis of power spectral density (PSD) of the topography of a surface of ultra-nanocrystalline diamond (UNCD) to detect the changes in tip radius during tapping mode with progressive scans. For each scan, the frequency data in relation to the power spectral density were collected and plotted to observe the tip blunting behavior under the same scanning condition. The quantitative determination of the tip radius compared to transmission electron microscopy (TEM) images of the tips showed corresponding results between the blunted tip and the PSD curve, concluding that the blunter tip produced a shorter frequency with low PSD value than the sharp tip.[8] However, the aforementioned measurements as long as reviewed are theoretical approaches with the lack of consideration of using the measured point-to-point data of tip position on the scanline profile in the coordinate system (x, y) for calculations of the exact tip radius after the scan. In addition, those studies are limited in the use of uncoated tips.

Here we present the actual measurement of the radius of coated and uncoated AFM sharp tips using contact scan mode with sub-nano normal load on a height calibration standard grating. After that, the round corner segment on the scanline profile of the tip apex obtained was taken to determine the tip radius with a nonlinear regression method. This method fits the arc curve to the measured point-to point data of tip position in coordinate system (x, y), allowing us to obtain the exact value of the tip radius.

AFM Tips and Calibration Standard Grating

Three types of AFM sharp tips used in this measurement method are as follows: a Pt-coated tip (HQ:NSC18/Pt, nominal radius: < 30 nm for Electrical, Force Modulation

AFM Probe)(see Figure 1a), a Cr-Au coated tip (HQ:NSC16/Cr-Au, nominal radius: < 35 nm for Tapping Mode AFM) (see Figure 1b), and a silicon uncoated tip (HQ:CSC17/No Al, nominal radius: < 8 nm for regular force contact mode) (see Figure 1c), supplied by MikroMasch. The full cone angle for all tips is 40^o which is characterized by MikroMasch.[9] The tip end shape is like a hemisphere with specific radius. For the coated tip, the tip radius includes the coated material. Figure 1 shows SEM images of three different probe tips manufactured by MikroMasch.

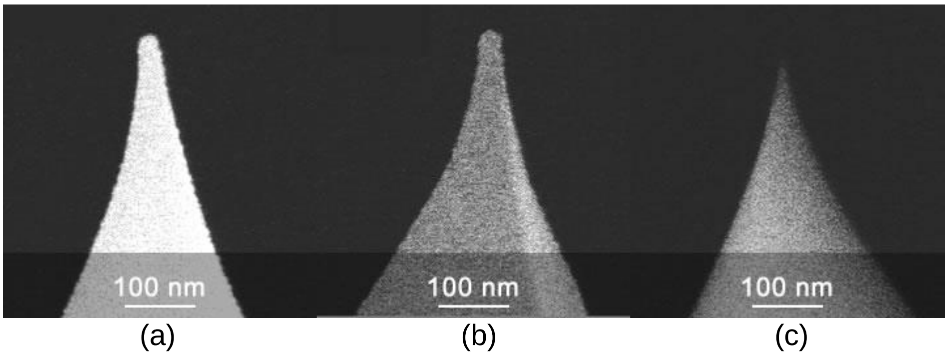

Figure 1 SEM images of coated tips and uncoated one; (a) the Pt-coated tip, (b) the Cr-Au coated tip, (c) the silicon uncoated tip [9]

The calibration standard grating used in this experiment is the HS-20MG Height Calibration standard (supplied by Budget Sensor) with an external size of 1mm x1mm and an inner size of 500 μm x 500 μm.[15] The rectangular grate height pattern is made of silicon oxide (SiO_2) with the height of 20 nm, grate distance = 2 μm, and pitch distance = 5 μm attached on top of the silicon-based substrate, which allows more space for these three AFM tips to sweep along the grate height geometry.

In our experiment, we used the inner standard grating area having the line grate pitch of 5 μm because the line grate pattern is easy to use the AFM tip to scan through each linewidth within a small scan area of 10 μm x 10 μm.

Determination of tip radii

Direct measurement of tip radii with tip scanning on height calibration grating

The linewidth scanning processes in contact mode of the AFM using each of the selected sharp tips with an applied constant normal load of 0.01 nN perpendicular to the calibration grating on a horizontal plane of the AFM workpiece holder were carried out under room temperature and 40% humidity (standard living room humidity). The scanning frequency of the tip is 0.1 Hz, which is appropriate for this experiment. The tips move from right to left across two pristine grating structures within the scan size of 10 μm x10 μm. Note, the calibration grating specimen was cleaned with DI water and dried with nitrogen gas before actual experiment. For each tip, we performed step-to-step linewidth scan pass (without repeating at the same scanline) in the contact mode with very light loading (0.01 nN) until it completed such defined scan area at a single time, thus tip wear effect is neglected. Noise effect is also unconsidered because the measurement was conducted inside a hood of the AFM machine, MFP-3D origin Asylum Research, with noise filtration system.

Considering the geometry of a fabricated nanostructure and the scanline signal obtained from the AFM, Figure 2 shows a schematic of the scanning direction of the AFM tip against a stationary grate height structure. Since the grating pattern is very small, the height of the calibration structure (20 nm) is obviously about the same level as the tip radius, except for the Si uncoated tip (<8 nm tip radius) and is steeper than the half-cone angle of the tip (α). So, the steep profile of the AFM scanline signal defined with the steep angle (β) represents the scan path of the apex of the tip sliding along the vertical sidewall of the grate height (a calibration column). Importantly, the curvature profile of the AFM signal scanline at the edge corner of the scanned calibration structure represents the tip radius (R) as shown in Figure 2. This analysis of the tip scanline profile is consistent with the previous study.[10] After all scanning processes are done, AFM height images taken from the AFM MFP-3D program are compared in three scanned images due to scanning with three different tips (Pt-coated tip, Cr-Au coated tip, and Si uncoated tip, see more detail in Supporting Information S1). The cross-section linewidth (red line) is used to extract the AFM signal scan profile for each tip that moved over two nanostructured heights of the calibration grate. Then, we plotted all those scanline profiles on the same graph (see Figure 3), showing that the scanline profiles correspond to the real geometry of the calibration grating as imaged by SEM. [16]

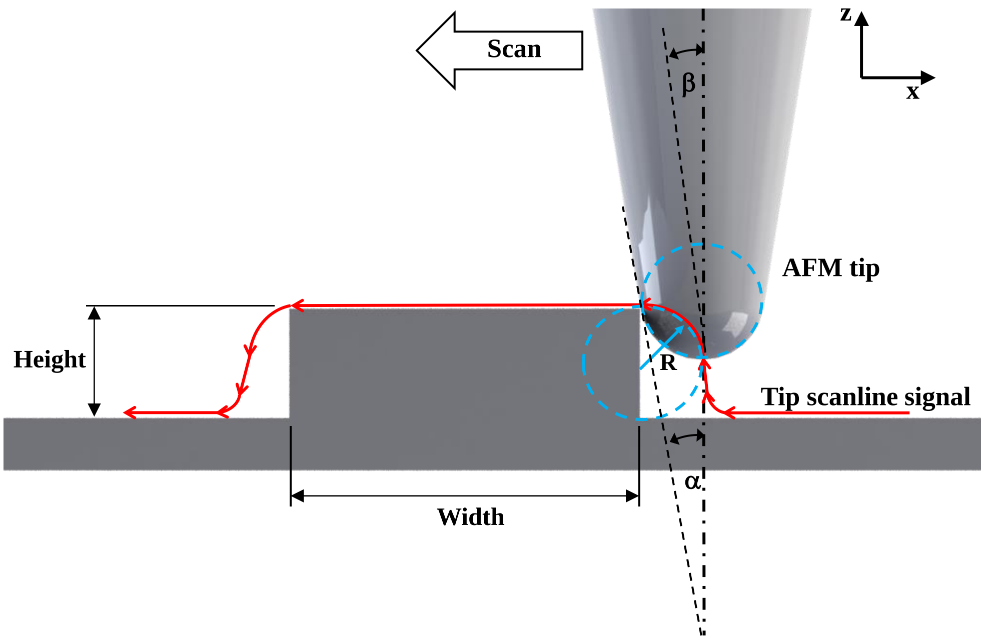

Figure 2 A side-view schematic of the scanned line profile of the tip along a calibration grating

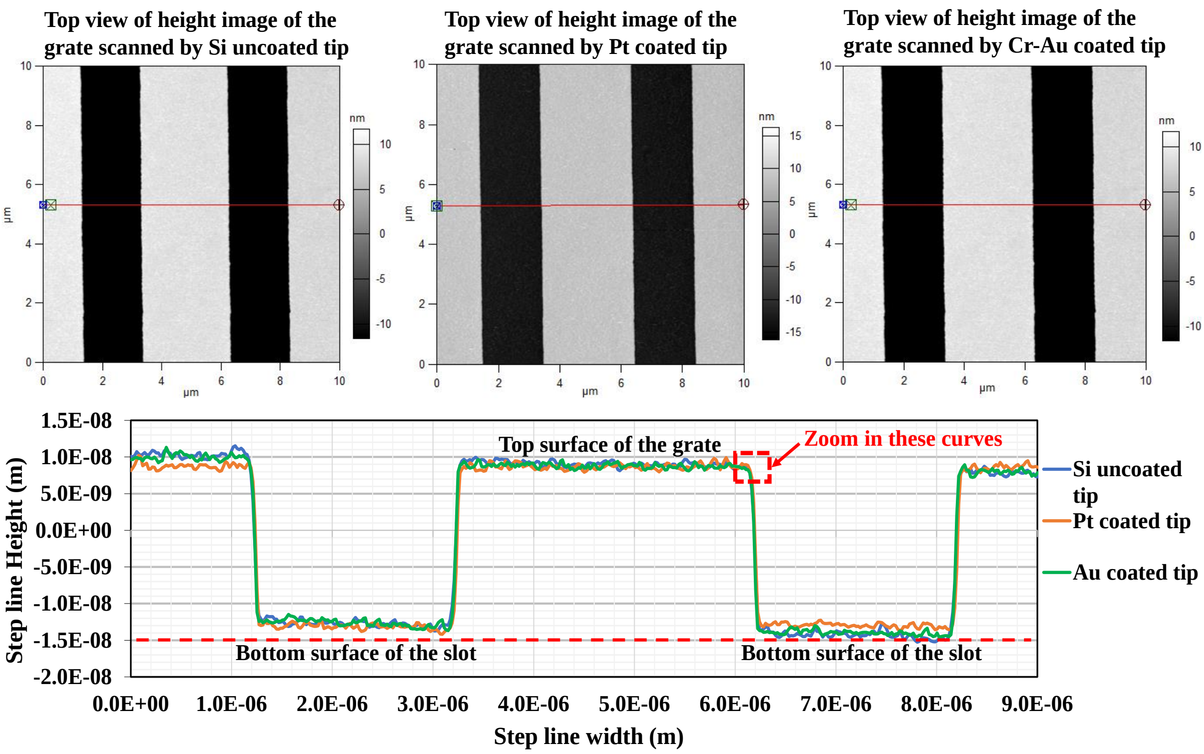

Figure 3 The height images (top view) obtained from scanning results using three tips (on the upper row) compared to the AFM signal scan profiles of the tips (side view) (on the lower row) extracted from the cross-section linewidth (red line)

The lines on the lower graph are created owing to connections of the measured point-to-point data of the AFM tip position when the tip slides along to the real geometry of the calibration grate. The height of the lines indicates the height of the grate. At this point, we are interested in tracing curved profiles at the edge corner of the height grate, thus zooming in that area and scanning each tip again at smaller scan area covering the grate corner with the same scanning process and load condition. The scanning result in terms of height images for all tips (see Supporting Information S2) shows realistic perspective in accordance with the analytical model of tip-sidewall contact in the sliding motion as denoted in Figure 2 above. Namely, brighter area means that the tip apex physically touched the grate's curved surface of the corner edge. Whereas the dark area represents inability of the tip to reach the bottom corner of the grating structure (a 20 nm SiO_2 column,[15]) because the tip cone angle is not parallel to the tip cone surface. The scanline profiles of all tips are extracted from the heigh images and compared on the same plot to ascertain the arc section for determining the tip radii with curve fitting method (see Figure 4). We take data points of tip position from each curve as marked with the red dash line (Figure 4), and then determine the radii of the tip by fitting the arc curves using nonlinear regression function in the next section.

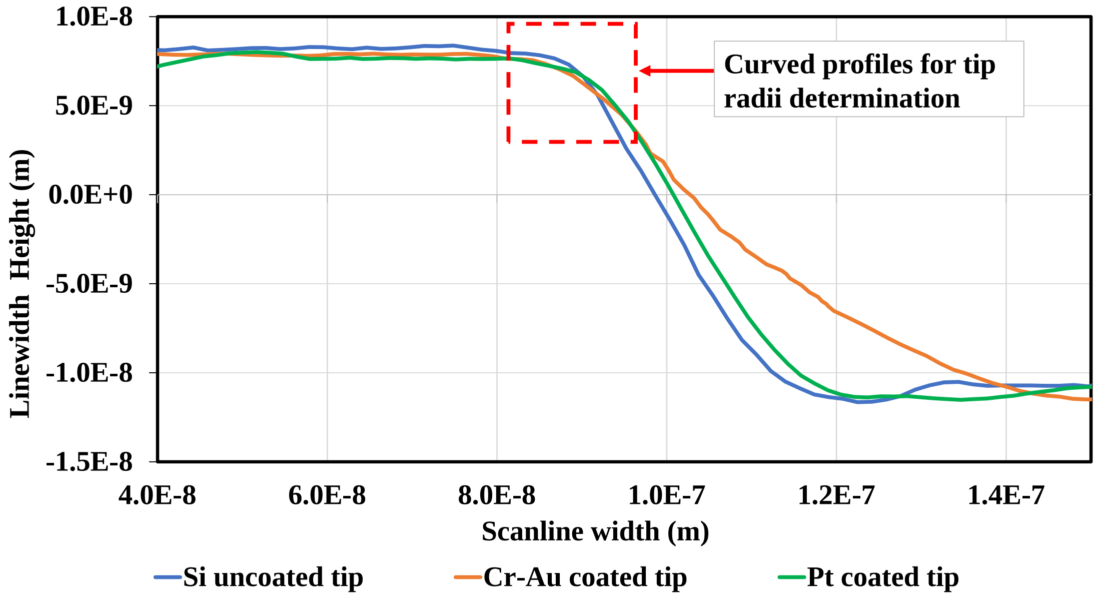

Figure 4 Comparison of the AFM scanline profiles of all tips scanning across the corner edge of the calibration grating structure of 20 nm producing the curves profiles for tip radius determination

Curve fitting with nonlinear regression

The arc curves of all tips obtained from Figure 4 are used to determine the curvature radii. The data points of tip position in x-y coordinate for each arc curve are imported into MATLAB. Then, we used a nonlinear regression function in MATLAB to fit the arc curve to the measured data points as the procedure explained follows. The nonlinear regression function of a circular curve on the x – y plane: y = $(x_1{}^2 + x_2{}^2 +$

$b_1x_1 + b_2x_2 + b_3$) was conducted for fitting the circular curve (a red curve in Figure 6) to the measured data on the cartesian x_1 and x_2 axes (blue stars in Figure 5, typical data of the Si uncoated tip) and finding the center of the arc. The b_1, b_2, and b_3 are vector parameters. Then, the mean distance (X_m & Y_m) from the center to those data points on that arc was estimated by the function itself. Finally, the tip radius can be calculated by equation R = sqrt $(({X_m}^2 + {Y_m}^2) - B_3)$, where B_3 is an error term (see Supporting Information S3).

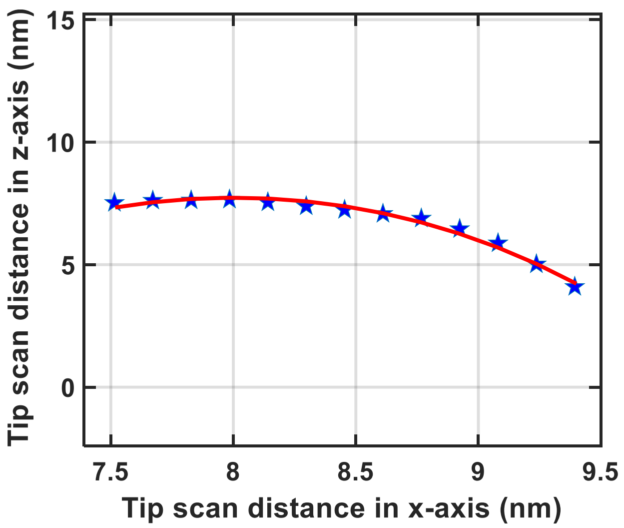

Figure 5 A typical curve fitting using the measured data points of the Pt-coated tip with the nonlinear regression function

Result and Discussion

Fitting all the tip scan-profile curves obtained allows us to get the estimated values of tip radii as follows. The radius of the Si uncoated tip is ~10.54 nm, which is smaller than the radius of Pt coated tip (~25.78 nm) followed by the Cr-Au coated tip (~29.61nm) and consistent with the standard nominal radii specified by the manufacturer (MikroMasch). This is pronounced that nonlinear regression method gives an accurate estimate because the values of Root Mean Squared Error (RMSE) from the fitting results are very small, that is, 2.67e-18 for the Si uncoated tip,3.43e-18 for the Pt coated tip, and 4.82e-18 for the Cr-Au coated tip, while R-squared values of all fitting results are infinity since the R-squared is not valid for Nonlinear Regression. However, there might have few errors on the Cr-Au coated tip scanline that moved up a bit more in an approach regime (see Figure 4) while sliding toward the sidewall of the grate height. Possibly, some particles stick to the tip surface due to contamination on the grating substrate, resulting in relatively high RMSE. This can be a further work for future researchers who want to calibrate the tip radius before the AFM experiment. They need to perform a thorough cleaning process for the AFM tip.

Conclusion

Three different AFM tips have been gone through the actual measurement of the radius using the in-situ probe characterizer, HS-20MG Height Calibration Grating Standard, under sub-nano scanning load. The curvature radius of each tip was estimated by fitting the measured data points of the tip position on the curved scanline at the edge corner of the grating structure with a nonlinear regression function. The results have shown that the tip radius obtained from the in-situ measurements and the good curve fitting with nonlinear regression function is in the range of the nominal values (< 30 nm of Pt-coated tip, < 35 nm of Cr-Au coated tip) given by the manufacturer, except for the radius of the Si uncoated tip which is larger than the nominal range about 2.54 nm. The curved profiles of both coated and uncoated show similar curved profiles, but different curvature radii and can be fitted for the tip radius with the nonlinear regression function. From curve fitting results, it indicates that our estimation model is reasonably accurate. However, more accuracy can be done if we have more data points of the AFM tip position at the curved scan profile (Figure 4) for the nonlinear regression curve fitting.

Supporting Information

Supporting Information S1: Height images of the scanned specimens of calibration standard grating within 10 μm x 10 μm

Supporting Information S2: Height images of the scanned corners of the calibration gate at smaller areas

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Author Information

Corresponding Authors

Perawat Boonpuek

School of Manufacturing Engineering, Institute of Engineering, Suranaree University of Technology, Nakhon Ratchasima 30000, Thailand

Email: perawat@sut.ac.th

Phone: +66-89-078-9857

Authors

Jonathan R. Felts

Advanced Nanomanufacturing Laboratory, Department of Mechanical Engineering, Texas A&M University, College Station, Texas 77840, USA

Author Contributions

Devised and Designed experiments[1, 2], performed experiments[1], developed experimental programs[1,2], interpreted results [1, 2], composed the written research work[1, 2].

Conflicts of interest

There are no conflicts to declare.

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