Calibration of 1 nm SiC step height standards

T.V. Vorburger, A. Hilton, R.G. Dixson, N.G. Orji,
National Institute of Standards and Technology, Gaithersburg, MD, 20899 USA

J.A. Powell,
Sest, Inc., Middleburg Hts., OH 44130 USA

A.J. Trunek,
Ohio Aerospace Institute, Cleveland, OH 44142 USA

P.G. Neudeck, P.B. Abel,
NASA Glenn Research Center, Cleveland, OH 44135 USA

ABSTRACT

We aim to develop and calibrate a set of step height standards to meet the range of steps useful for nanotechnology. Of particular interest to this community is the calibration of atomic force microscopes operating at their highest levels of magnification. In previous work we fabricated and calibrated step height standards consisting of the lattice steps on the (111) surface of single crystal Si and provided a recommended value of 312 pm ± 12 pm. In the current work we report traceable measurements of 1 nm step height specimens fabricated on the (0001) 4H-SiC surface. In this, we are seeking to fill in the range between the newly available 300 pm steps and 8 nm steps, which are the smallest available commercially. The step height measurements were performed using a calibrated atomic force microscope (C-AFM) calibrated with respect to the wavelength of light along all three axes of motion. Analysis of the measurements yields an average step height value of 0.981 nm with a combined standard uncertainty of ± 0.019 nm (k = 1), reasonably consistent with the expected value of 1.00851 nm derived from the parameters of the SiC crystal lattice.

Keywords: step height, atomic force microscope, nanometrology, silicon carbide, calibration, traceability, ASTM, metrology, atom-based standards

1. MOTIVATION AND BACKGROUND

Step height standards are used for calibration of the z-scale of a wide range of surface measurement instruments, including stylus instruments, optical profilers, and atomic force microscopes (AFMs). These instruments in turn are used in a wide range of measurement applications, such as:
- the roughness of mechanical, optical, and semiconductor surfaces,
- the heights of semiconductor features,
- the thicknesses of coatings, and
- pole tip recession for magnetic data storage.

The step height standards may be two sided, as shown by the upper diagram in Fig. 1,[1,2] or may be one sided, as shown by the lower diagram in Fig. 1[3]. The steps may also range widely in height. For example, one vendor offers step height standards with heights as small as 8 nm or as high as 24 μm. Taller step heights are also available commercially or they may be fabricated by wringing on a platen two adjacent gauge blocks with slightly different thicknesses. The step height is then the difference in thickness between the two adjacent gauge blocks.

Commercial step heights are often fabricated by film deposition or surface etching. For years the smallest commercially available step height was approximately 8 nm. However, calibrated step heights are required down to the atomic (sub-nanometer) levels in order to provide calibration and traceability for AFMs operating at their highest levels of magnification for measuring semiconductor and optical surfaces.

$$d = C - (A + B) / 2$$
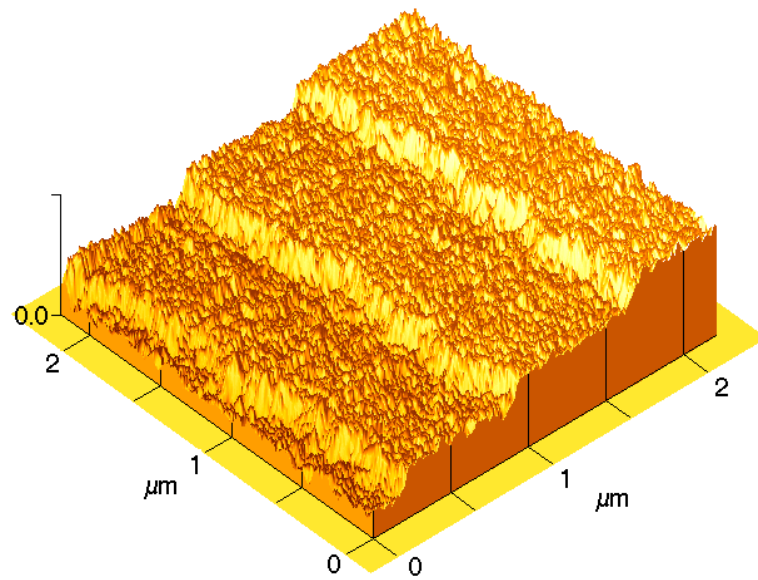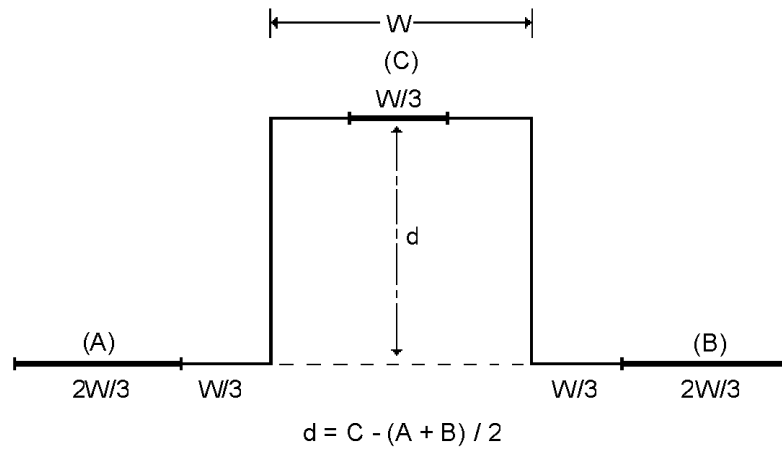
measurement procedure described in ASTM 2530-06 makes it feasible to calibrate an AFM at the atomic level using the Si(111) steps and derive a value for the z-gain of the instrument with reasonable uncertainty, approximately 6 % (k = 2). The physical standards themselves are available commercially or may be fabricated[3,4,6,10].

However, there is now a gap in the availability of step height standards between 300 pm and 8 nm. To fill this gap, at least two height standards need to be developed, one between about 2 nm and 3 nm and one at about 1 nm. Calibrated height measurements between these levels can routinely be attained if the measurement tools have moderately linear sensitivity to surface height.

In this paper, we discuss the fabrication and measurement of a prototype 1 nm step height standard patterned from lattice steps in SiC, and we provide a traceable result for the measured step height.

2. SiC STEP HEIGHT FABRICATION

Various SiC polytype crystals grow via step-flow growth from steps produced by screw-dislocations. A given polytype is produced by a particular bilayer stacking sequence along the crystal c-axis (parallel to the screw dislocations). The SiC step height standard is fabricated from an "on-axis", Si-face, commercial 4H-SiC (0001) wafer (four-bilayer repeat sequence). Miscut angles for the Si-face are specified to be less than 0.5°. Detailed descriptions of the fabrication process, developed at the National Aeronautics and Space Administration (NASA) Glenn Research Center, are given elsewhere [11-13].

The essential fabrication steps are as follows: First, deep etch a polished Si face of the wafer to produce an array of rectangular mesas (about 100 μm on an edge) that are aligned with an edge parallel to a <1120> crystal direction. At this mesa size, most mesas will be free of screw dislocations using currently available commercial wafers. Second, carry out epitaxial deposition in a manner that causes existing steps to grow to the edge of the mesa by "step flow" growth on the SiC wafer. Those mesas that are free of screw dislocations will become step-free. Third, etch these samples in a hydrogen etch at about 1450 °C for about 5 min. The etching of the step-free mesas will produce a "step-flow etch" from the edges of the mesa to produce a four-sided, pyramidal step structure on each mesa with the following characteristics (See Fig. 2). Two opposite faces of the pyramid will have 0.5 nm steps (two-bilayer steps) and the other two faces will have 1 nm steps (four-bilayer steps) with a single step on one side of the crest consisting of only two bilayers. The horizontal distance between steps will be about 1 μm. The 1 nm steps will be parallel to the <1120> direction and the 0.5 nm steps will be perpendicular to the 1 nm steps. The end of each 1 nm step splits into two 0.5 nm steps.
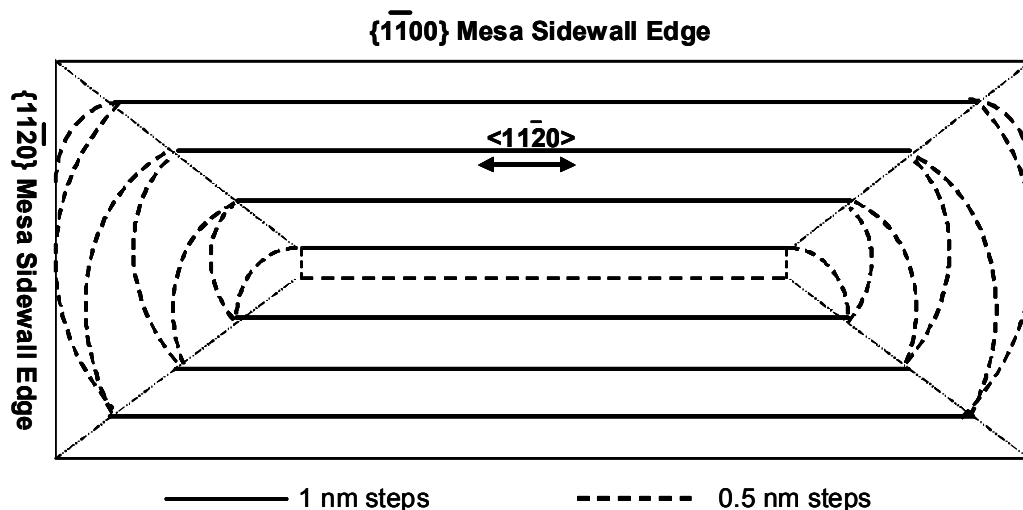

Figure 2. Schematic diagram of the fabricated SiC pyramidal structure showing the crystal directions and the splitting of the 1 nm steps around the edges.

3. MEASUREMENT SYSTEM

The calibrated atomic force microscope[14,15] (C-AFM) was used to calibrate areas on the 1 nm step height standards. The C-AFM is a custom AFM constructed by the National Institute of Standards and Technology (NIST) for dimensional metrology applications. It is intended primarily to calibrate physical standards for other AFMs. The design, performance, and uncertainties of the system have been discussed elsewhere, so we give only a short summary here. The C-AFM has metrology traceability via the 633 nm wavelength of the I_2-stabilized He-Ne laser—a recommended radiation for the realization of the meter with visible light—for all three axes. This is accomplished using heterodyne laser interferometers. The C-AFM employs a scanning-sample design. A piezoelectrically driven two-axis flexure stage, with a 100 μm range, is used to translate the sample in the x and y directions. It has negligible straightness and angular motion deviations. Heterodyne laser interferometers monitor the x-y displacement, and a digital signal processor in the controller is used to allow closed loop control of the lateral sample position. This eliminates the scale calibration and linearity problems of the scanners used in most commercial instruments. The vertical (z) position of the sample is adjusted with a piezoelectrically-actuated, flexure-guided transducer with an integrated capacitance sensor. This z-stage provides one axis of rectilinear motion with negligible straightness error and an angular motion error of approximately 0.31 μrad/μm of travel, and the capacitance sensor provides measurement of the z-stage extension with high repeatability and high resolution. To achieve traceability, the capacitance sensor is calibrated *in situ* using a third interferometer each day that the system is used to make a z-scale measurement.

For the present work, the system was operated in contact mode with a commercial AFM head having a sensitive optical-lever force transducer. Low thermal expansion materials and kinematic mounts are used to minimize drift in the sensitive components of the system, and the instrument is operated in a temperature-controlled laboratory with a stability of less than 0.1 °C.

4. MEASUREMENTS

NASA Glenn provided NIST with two SiC chips to be measured and calibrated. These were labeled 0408 and 0409. Each sample is a 3 mm × 4 mm SiC chip, 1 mm thick, etched with a number of rectangular mesas, which are approximately 200 μm × 50 μm in size. Each mesa, if well-formed, should consist of a chisel-shaped pyramid structure which contains a staircase of steps on each of its four facets. Figure 3 shows an area of a chip containing about nine of these mesas, with the letter labels "R S T" across the top and the number labels "9 8 7" on the right. Two of the nine mesas are clearly defective and the pyramidal structure is poorly developed, but the other seven mesas reveal a well developed pyramidal structure, such as mesa S8. The areas on the top and bottom faces of the pyramid S8 and others there contain the 1 nm steps, typical of those measured for this paper. These steps split around the side areas (see Fig. 4), and the short faces left and right therefore contain 0.5 nm steps, which were not measured for this paper. A further close-up (Fig. 5) of a small area straddling the top of the chisel-pyramid clearly shows the 1 nm steps, now oriented left and right.

A clean pyramid was selected for measurement on each SiC chip. These areas are denoted as N7 on chip 0408 and as R2 on chip 0409. On each pyramid, twenty distributed small areas containing steps were selected for individual step height measurements. Figure 6 contains a schematic diagram of the step height sampling plan. A topographic image of a single profile on one of these areas is shown in Fig. 7. The image contains approximately 2000 data points and is approximately 2 μm in length. These profiles were analyzed for step height and the results averaged over all profiles in the topography images on the pyramid as further described below.

5. ANALYSIS AND RESULTS

Twenty topographic images were measured on each sample. For both samples, the image at position 3a was not used because the step transition in these images was poorly defined. For the remaining images, a step height was calculated for each profile from a rectangular area that was manually selected to have relatively clean profiles with few anomalies or outliers. In addition, a few profiles in the selected regions yielded calculated step heights less than 0 and were not included in the final tally. Depending on the image, the number of selected profiles ranged from 21 to about 67. As shown in Fig. 7, the height of a one sided step is calculated as the vertical distance between two straight lines fitted to equal-length profile sections equally spaced from the middle of the step transition. The lateral position of the step transition is defined to be the position of the steepest slope in the profile after a 20-point moving average has been temporarily applied to the profile. For future measurements an automated criterion could be developed to select profiles that do not contain outlier data points, but this function was not developed for the present measurements.
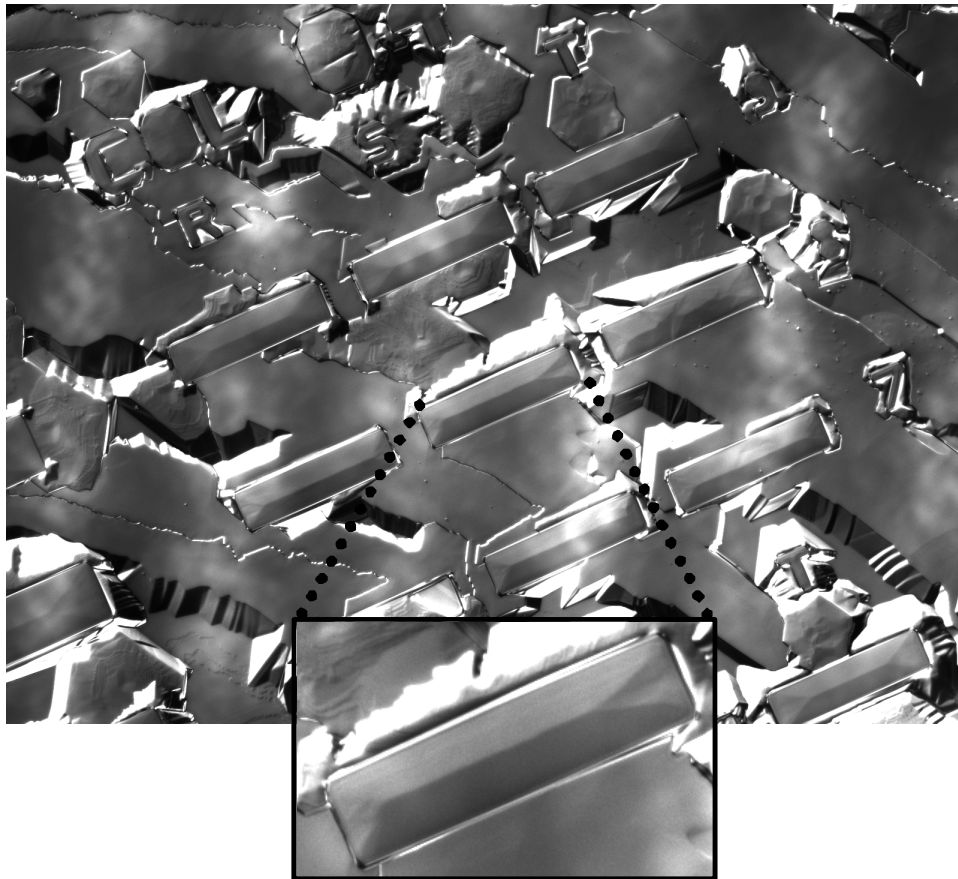

Figure 3. Photo-micrograph of a small number of fabricated mesa structures on the (0001) 4H-SiC surface. The etched labels R S and T are shown along the top and the labels 9, 8, and 7 along the side. The mesas are approximately 200 μm × 50 μ in size. Area S8, also shown magnified, is a good example of a clearly fabricated pyramidal structure that could be used as a step height standard.

The mean step height for each of the 19 regions was then calculated by averaging the step heights obtained from the individual profiles. These mean values are shown in Table 1. The sample mean and its standard deviation were then calculated from the values for the 19 regions for each sample, and a grand average for both samples was taken as the mean of the two sample values. The value of 0.981 nm represents the measured height of the SiC step height as determined by the C-AFM. The external standard deviation of 0.0099 nm shown in Table 1 is the standard deviation of

the two sample means, 0.9875 nm and 0.9736 nm. This quantity is slightly larger than the internal standard deviation of 0.0080 nm, so we take the external standard deviation as the Type A statistical uncertainty of these results. The combined uncertainty estimated from all sources is calculated in the next section.
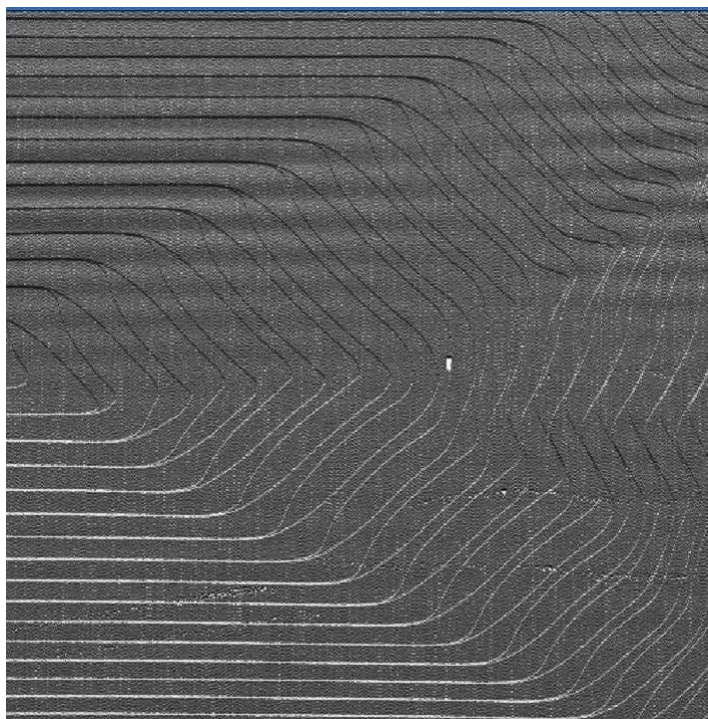

Figure 4. AFM image of one edge of a SiC pyramid structure showing the splitting of the 1 nm steps. The field of view is about 25 μm × 25 μm.
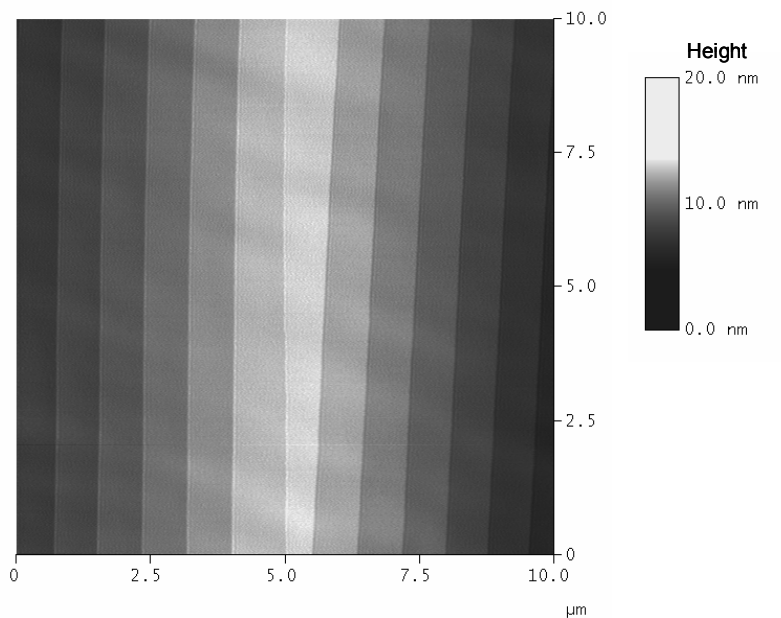

Figure 5. AFM topographic image showing the peak of a pyramidal structure on 4H-SiC and individual 1 nm steps on staircases leading left and right.
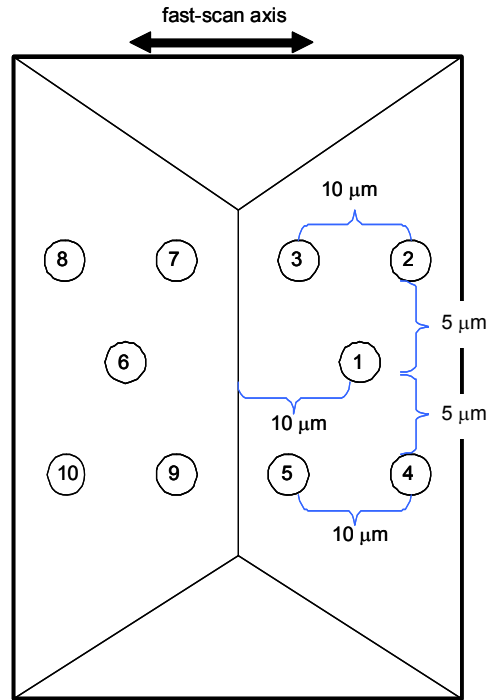
Figure 6. Schematic diagram of a SiC target area. Two images were taken at each of the 10 circled areas, for a total of 20 images per sample. Each scan size was about 2 μm × 2 μm. Each image size was about 2000 pixels × 256 profiles.
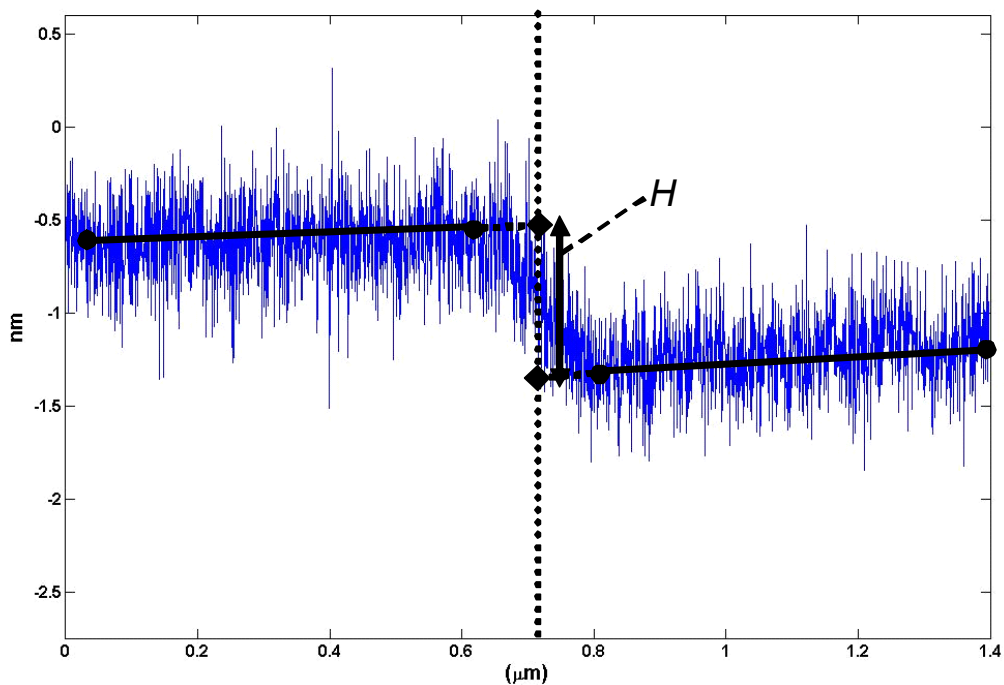

Figure 7. Example of a single profile of measured data of a SiC step height measured with the C-AFM. Also shown is a schematic diagram of the algorithm used to calculate the step height. The solid lines show the straight lines fitted between the filled circles, and the dashed extensions show the extrapolations to the middle of the step transition.

6. UNCERTAINTY BUDGET

A number of sources of uncertainty must be taken into account to establish the overall uncertainty for the measurements of the SiC step height. A general uncertainty budget for the C-AFM for measurements in the *z*-direction has been published elsewhere[15]. Table 2 shows the uncertainty budget with all estimated components for calibration of the SiC 1 nm step height using the C-AFM. In accordance with current standards for describing uncertainties of measured results, the components are classified as Type A or Type B[16,17]. Terms evaluated exclusively by statistical methods are classified as Type A components. Other terms, evaluated using some combination of measured data, physical models, or assumptions about the probability distribution of potential errors, are classified as Type B components. Each component of uncertainty is briefly described in the paragraphs below.

6.1 Repeatability and step height uniformity

Numerous sources of noise in the C-AFM measurement system produce noise in the measured profiles (as shown in Fig. 7) and variations in the measured surface profiling results. Variations also arise from non-uniformities in the measured sample. Both sources of variation are accounted for here by taking successive topographic images over different step heights, calculating the average step height for each image, and then calculating the standard deviation of the mean of the results as described in Section 5. For these data, the external standard deviation is used.

6.2 Capacitance gauge scale calibration, reproducibility

This is one of several components of uncertainty arising in the calibration of the capacitance gauge by interferometric displacement measurement over a height range of approximately 1 μm. This procedure is performed each day that the capacitance gauge is used for step height measurements. The calibration procedure yields a value for the sensitivity of the capacitance gauge that is approximately 3.588 nm/mV, but which varies from day to day. Because the sources of this variability are not well understood, we assume that this overall variability could produce variability in the measured step height and we, therefore, include the day-to-day reproducibility of capacitance gauge sensitivity in the uncertainty budget. We treat the component as a Type B uncertainty component, using the scale calibration factors of 3.590 nm/mV and 3.586 nm/mV, recorded on the two days that step-height data were taken on mesas R2 and N7.

6.3 Capacitance gauge scale calibration, Abbe offset

Potential Abbe offset between the axis of the capacitance gauge and the interferometer axis, coupled with angular motion error of the piezo-electric displacement transducer in the z-direction produces a potential error in the measured sensitivity factor of the capacitance gauge. The angular motion of the transducer has a linear error of approximately 0.31 μrad/μm, and the Abbe offset is measured to be approximately 3 mm, thus producing a fractional uncertainty in the height measurement shown in the tables.

6.4 Capacitance gauge scale calibration, cosine error

A cosine error results if the direction of the laser axis is not parallel to the direction of motion of the capacitance gauge. This potential error appears to be limited by the squareness of the connector that fastens the capacitance gauge to the sample platform. An upper limit of 1° is estimated for this error, which leads to the quoted uncertainty shown in Table 2.

6.5 Capacitance gauge scale calibration, voltage measurement

Possible nonlinearity in the voltage measurement system for the capacitance gauge contributes a very small uncertainty to the measurement.

Table 1. Results of Step Height Calculations from C-AFM Profiling Data on Two Stepped SiC Surfaces.

NASA SiC Sample

	Sample 409 Area R2 22-Jan-09		Sample 408 Area N7 23-Mar-09	
	Height	Stdev.	Height	Stdev.
Position	nm	nm	nm	nm
1a	1.0352	0.0894	1.0570	0.0773
1b	1.0185	0.1093	0.9197	0.0738
2a	0.9730	0.1084	0.9574	0.1247
2b	1.0476	0.1350	0.8898	0.0549
3a*				
3b	0.9510	0.0893	0.9639	0.0677
4a	1.0457	0.1094	0.9087	0.0591
4b	0.9994	0.1288	0.9863	0.0516
5a	0.9986	0.1048	0.9216	0.0640
5b	1.1049	0.1113	0.9804	0.1223
6a	1.0129	0.1203	1.0144	0.0728
6b	0.9973	0.1134	0.9596	0.0924
7a	0.8943	0.0726	1.0210	0.1076
7b	0.9828	0.0515	0.9905	0.0756
8a	0.9650	0.1080	1.0117	0.0744
8b	0.8949	0.0728	1.0241	0.0942
9a	0.9715	0.0686	0.9705	0.0629
9b	0.9579	0.0937	1.0160	0.0795
10a	0.9869	0.1277	0.9101	0.0780
10b	0.9260	0.0511	0.9952	0.0729
Sample Average	0.9875	0.2269	0.9736	0.0188
External Stdev.	0.0523		0.0466	
Stdev. of the mean	0.0120		0.0107	

Average Step Height =   0.9806   nm

Internal Stdev.   0.0080   nm
External Stdev.   0.0099   nm

* Position 3a on both samples was not used because of the poorly resolved step transitions.

6.6 Step height measurement, Abbe offset

When a step height measurement is made, any Abbe offset between the capacitance gauge axis and the AFM probe tip leads to an error in the height measurement, when coupled with the angular motion error of the z-stage. This component is similar in form and is estimated to be slightly smaller than the other Abbe uncertainty term described above.

6.7 Step height measurement, cosine error

An error in the measured step height could result if the normal direction of the sample surface is not parallel to the direction of motion of the capacitance gauge. This term is estimated from the apparent slope of the sample surface as the sample is translated over a wide scan range of 85 μm.

6.8 Capacitance gauge nonlinearity

The measured sensitivity of the capacitance gauge varies systematically as the calibration scan length is varied from about 1 μm to about 0.4 μm. This nonlinearity causes uncertainty in the measured step height unless the amplitude of the measured step is precisely the same as the calibrated length of travel of the capacitance gauge. An estimate of the size of this effect over all measurement scales is made, based on measurements of the change of the measured sensitivity with height.

6.9 Calculation uncertainty

The quoted grand average of 0.981 nm is calculated from selected profiles in the 19 topographic images. To test the sensitivity of the result to calculation procedure, we re-analyzed the data in two ways. First, we averaged the step height values obtained from all profiles in all images except for image 3b on 23-Mar-09, which had the most poorly defined step transition. We also changed the lengths of the fitted straight lines. This approach yielded a mean value of 0.986 nm. Then, we used only 36 images but selected larger areas in each image. This approach yielded a mean value of 0.997 nm. We conclude that the result for the average step height is not sensitive to specific choices of data regions. However, the largest difference (0.997 nm – 0.981 nm = 0.16 nm) is taken as an estimate of the uncertainty due to the choice of calculation procedure and is included in the uncertainty budget.

The combined standard uncertainty (u_c) is found by taking the quadratic sum of all the components. The formula and calculated value for the combined standard uncertainty is given at the bottom of Table 2. Therefore, the overall result for the SiC step height is 0.981 nm with a standard uncertainty ($k = 1$) of ± 0.019 nm. This value is in reasonable agreement with the expected value of (1.00851 ± 0.00002) nm (k=1) derived from bulk X-ray diffraction[18].

Table 2. Uncertainty Budget ($k = 1$) for Measurement of the SiC Step Height with the C-AFM

Component	Standard Uncertainty
Type A	
Repeatability and step height uniformity – external standard deviation of two results	0.0099 nm
Type B	
Capacitance gauge scale calibration, reproducibility	0.00056 H*
Capacitance gauge scale calibration, Abbe offset	0.00093 H
Capacitance gauge scale calibration, cosine error	0.00009 H
Capacitance gauge scale calibration, voltage measurement	0.00002 H
Step height measurement, Abbe offset	0.00082 H
Step height measurement, cosine error	0.00007 H
Capacitance gauge nonlinearity	0.00266 H
Calculation uncertainty	0.016 nm
Combined standard uncertainty	$[(0.0188 \text{ nm})^2 + (0.0030H)^2]^{1/2}$ = 0.019 nm

*The symbol H indicates that the uncertainty is proportional to the measured value of step height H.

7. SUMMARY AND FUTURE WORK

We have developed a procedure for calibration of a SiC single lattice step height using the C-AFM. The procedure requires a modest number of measurements to sample the surface of the standard and produces a value with a small uncertainty. Because a number of different steps were sampled, the result represents the calibrated value for a step fabricated with the procedure discussed in Sec. 2. SiC steps can now be used as calibration standards for AFMs operating at nearly their highest magnification ranges. The signal-to-noise ratio of the measurements is high enough that only a modest number of measurements should be required to perform a calibration.

The straightforward algorithm used here for calculating step height is similar to the algorithm already described in standard ASTM 2530-06[3], and the use of SiC as a potential source of calibration is mentioned briefly there. Versions of the algorithm have been used by NIST for years to calibrate single-sided step heights[19] and a FORTRAN implementation was published in 1983[20]. As has been discussed by Fu[21], this algorithm is certainly more robust against profiling artifacts such as curvature and slope variations than more commonly used algorithms such as simple area subtraction or the histogram method. It should be straightforward to install the algorithm into commercial AFM controllers.

Since SiC is inert to wet chemicals at room temperature, the standards have a very stable surface and once fabricated, should last for years in a reasonably clean laboratory environment. A wet acid clean, including those known to remove any native oxide that might form in air, could be used to attack foreign contaminants that might accumulate on the surface over time. The fabrication technology was developed by NASA Glenn Research Center. An effort is underway to transfer the fabrication technology to metrology suppliers. Once that is accomplished, SiC standards would be available to industrial users of AFMs for calibration of their AFMs.

The SiC step height at 1 nm fills an important gap in the range of available step height standards. The linearity of profiling instruments, on the whole, is good enough that a calibration standard does not need to be closely matched in height to a sample required to be measured. Commercial step height standards are often available as a series of steps differing in height by factors of two or three. The SiC lattice steps together with the Si (111) step height standards available commercially can fulfill the need for step heights to calibrate the atomic scale vertical ranges of AFMs. There remains only one gap—between 1 nm and 8 nm—in the range of standards available from macroscopic regimes down to 0.3 nm. If a step height of 2 nm to 3 nm were commercially available, that would fill the gap. But what fabrication process should be used for this? It is not yet apparent whether it would be more practical to fabricate such a step by atom-based techniques, such as those used for Si(111) and SiC, or by traditional etching or deposition.

One approach to fabricating different step heights would be to use different morphologies of SiC. By orienting a rectangular mesa with the long edge rotated 90° from those in the pattern shown in Fig. 4, the pyramid forms with predominantly 0.5nm (straight) steps rather than 1 nm steps. One can then create both 1nm and 0.5nm calibration step heights on one chip. Alternatively, by starting with the 6H SiC polytype, calibration specimens with 0.75 nm and 1.5 nm steps on each chip could be produced. One can then envisage a fairly "rational" step height sequence starting from the Si(111) 0.3 nm commercially available baseline step. To eliminate the small remaining gap, a step of 3 nm to 4 nm could be fabricated with etching or deposition.

8. ACKNOWLEDGMENTS

REFERENCES

[1] ASME B46.1 – 2002, *Surface Texture (Surface Roughness, Waviness, and Lay)*, (Amer. Soc. Mech. Engrs., New York, 2003).

[2] ISO 5436-1: 2000, *Geometrical Product Specifications (GPS) — Surface texture: Profile method; Measurement standards — Part 1: Material measures*, (International Organization for Standardization, Geneva, 2000).

[3] ASTM 2530-06, *Standard Practice for Calibrating the Z-Magnification of an Atomic Force Microscope at Subnanometer Displacement Levels Using Si (111) Monatomic Steps,* (ASTM International, West Conshohocken PA, 2006).

[4] M.Suzuki, Y. Kudoh, Y. Homma, and R. Kaneko, Monatomic step observation on Si(111) surface by force microscopy, Appl. Phys. Lett. **58**, 2225-2227 (1991).

[5] M. Suzuki, S. Aoyama, T. Futatsuki, A.J. Kelly, T. Osada, A. Nakano, Y. Sakakibara, Y. Suzuki, H. Takami, T. Takenobu, and M. Yasutake, Standardized procedure for calibrating height scales in atomic force microscopy on the order of 1 nm, J. Vac. Sci. Technol. **A14**, 1228-1232 (1996).

[6] V. Tsai, X.-S. Wang, E. D. Williams, J. Schneir, and R. Dixson, Conformal Oxides on Si Surfaces, Appl. Phys. Lett. **71**, 1495-1497 (1997).

[7] V.W. Tsai, T. Vorburger, R. Dixson, J. Fu, R. Köning, R. Silver, and E.D. Williams, The Study of Silicon Stepped Surfaces as Atomic Force Microscope Calibration Standards with a Calibrated AFM at NIST, in D. G. Seiler, A. C. Diebold, W. M. Bullis, T. J. Shaffner, R. McDonald, and E. J. Walters, eds. *Characterization and Metrology for ULSI Technology: 1998 International Conference*, CP449 (Amer. Inst. Phys., Woodbury, NY, 1998) pp 839-842.

[8] R. Dixson, N. Orji, V. Tsai, E. Williams, R. Kacker, T. Vorburger, H. Edwards, D. Cook, P. West, and R. Nyffenegger, Silicon Single Atom Steps as AFM Height Standards, Proc. SPIE **4344**, 157-168 (2001).

[9] N. Orji, R. Dixson, J. Fu, and T. Vorburger, Traceable Pico-meter Level Step Height Metrology, Wear **257**, 1264-1269 (2004).

[10] J. Fu, H. Zhou, J.A. Kramar, R.M. Silver, S. Gonda, Dependency of Morphology on Miscut Angle for Si(111) Etched in NH_4F, Appl. Phys. Lett. **82**, 3014-3016 (2003).

[11] J.A. Powell, P.G. Neudeck, A.J. Trunek, and P.B. Abel, Step Structures Produced by Hydrogen Etching of Initially Step-Free (0001) 4H-SiC Mesas, Materials Science Forum **483-485**, 753-756 (2005).

[12] J.A. Powell, P.G. Neudeck, A.J. Trunek, G.M. Beheim, L.G. Matus, R.W. Hoffman, and L.J. Keys, Growth of step-free surfaces on device-size (0001) SiC mesas, Appl. Phys. Lett. **77**, 1449-1452 (2000).

[13] P.B. Abel, J.A. Powell, and P.G. Neudeck, US Patent 6,869,480 - Method for the production of nanometer scale step height reference specimens (Mar. 22, 2005).

[14] R. Dixson, R. Köning, V.W. Tsai, J. Fu, and T.V. Vorburger, Dimensional Metrology with the NIST calibrated atomic force microscope, Proc. SPIE **3677**, 20-34 (1999).

[15] R. Dixson, R. Köning, J. Fu, T. Vorburger, and B. Renegar, Accurate Dimensional Metrology with Atomic Force Microscopy, Proc. SPIE **3998**, 362-368 (2000).

[16] JCGM 100:2008, *Evaluation of measurement data—Guide to the expression of uncertainty in measurement* (Joint Committee for Guides in Metrology, September, 2008)
http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf.

[17] B. N. Taylor and C. E. Kuyatt, *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*, NIST Tech. Note 1297, (Nat'l. Inst. Stds. & Technol., Gaithersburg, MD, 1994).

[18] H.J. Chung and M. Skowronski, High-resolution X-ray diffraction and optical absorption study of heavily nitrogen-doped 4H–SiC crystals, J. Cryst. Growth **259**, 52-60 (2003).

[19] E.C. Teague, *Evaluation, Revision, and Application of the NBS Stylus/Computer System for the Measurement of Surface Roughness*, NBS Technical Note 902 (U.S. Dept. Commerce, Wash., DC, 1976).

[20] T.V. Vorburger, *FASTMENU: A Set of FORTRAN Programs for Analyzing Surface Texture*, NBSIR 83 2703 (Dept. of Commerce, Washington, DC, 1983) p. 15.

[21] J. Fu, V.W. Tsai, R. Köning, R. Dixson, and T.V. Vorburger, Algorithms for calculating single atom step heights, Nanotechnology **10**, 428-433 (1999).