Designation: E 2530 – 06

Standard Practice for Calibrating the *Z*-Magnification of an Atomic Force Microscope at Subnanometer Displacement Levels Using Si *(111)* Monatomic Steps[1]

This standard is issued under the fixed designation E 2530; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers a measurement procedure to calibrate the *z*-scale of an atomic force microscope using Si *(111)* monatomic step height specimens.

1.2 *Applications*—This procedure is applicable either in ambient or vacuum condition when the atomic force microscope (AFM) is operated at its highest levels of *z*-magnification, that is, in the nanometer and sub-nanometer ranges of *z*-displacement. These ranges of measurement are required when the AFM is used to measure the surfaces of semiconductors, optical surfaces, and other high technology components.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ISO Standards:*[2]

ISO Draft International Standard 25178-2 Geometrical Products Specification (GPS)---Surface Texture: Areal----Part 2: Terms, Definitions and Surface Texture Parameters.

ISO Committee Draft 25178-6 Geometrical Products Specification (GPS)---Surface Texture: areal----Part 6: Classification of methods for measuring surface texture.

ISO/TS 21748: 2004 Guidance for the Use of Repeatability, Reproducibility and Trueness Estimates in Measurement Uncertainty Estimation

GUM: 1993 Guide to the Expression of Uncertainty in Measurement

3. Terminology

3.1

3.1.1 *Atomic force microscope (AFM)*, *n*—surface topography measurement method whereby the surface height is sensed from the mechanical force of attraction or repulsion between a probe tip and a surface.

3.1.2 *Coordinate axes*, *n*—coordinate system in which surface topographic measurements are performed.

3.1.2.1 *Discussion*—It is usual to use a rectangular coordinate system in which the axes form a right Cartesian set, the *x*-axis being the direction of tracing colinear with the mean line, the *y*-axis also nominally lying on the real surface, and the *z*-axis being in an outward direction (from the material to the surrounding medium).

3.1.3 *Si(111)*, *n*—single crystal surface of silicon, oriented near the *(111)* crystal plane and perhaps with either a grown oxide overlayer or native oxide layer, which, when prepared appropriately, contains a large number of separated monatomic steps and atomically smooth terraces between them.

3.1.4 *x-y displacement stage*, *n*—mechanical means used to move a probe with respect to a surface (or vice versa) along the two coordinate axes in the plane of the surface.

3.1.5 *z-magnification (alternatively, z-sensitivity or z-scale)*, *n*—term that describes the sensitivity of the output of a surface profiling instrument to displacements in the *z*-direction.

4. Significance and Use

4.1 Careful use of this practice can yield calibrated *z*-magnifications traceable to the SI unit of length with uncertainties (k = 2) of approximately 7 % over height ranges of approximately 1 nm.

5. Calibration Specimen

5.1 The physical step height specimen is the vicinal Si *(111)* surface (see Fig. 1), defined in 3.1.3, containing numerous monatomic steps, having on average a calibrated step height value. The calibrated value has been traditionally assigned to be 314 pm based on X-ray value of lattice parameter of bulk silicon (**1**). However, recent analysis (**2-7**), also taking into account independent measurements by other sources, has

[1] This test method is under the jurisdiction of ASTM Committee E42 on Surface Analysis and is the direct responsibility of Subcommittee E42.14 on STM/AFM.

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[2] Available from International Organization for Standardization (ISO), 1 rue de Varembé, Case postale 56, CH-1211, Geneva 20, Switzerland, http://www.iso.ch.

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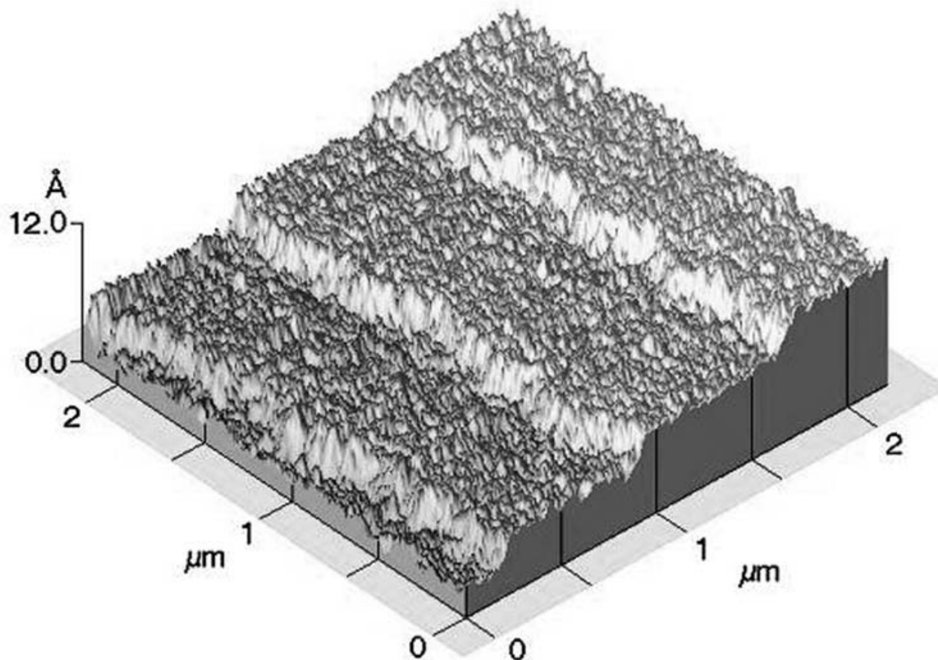

FIG. 1 Image of a Silicon Monatomic Stepped Surface

produced a current recommended value of 312 + 12 pm (k = 2). The difference is small in comparison with other sources of uncertainty in the procedures for measuring sub-nanometer level surface topography. Future measurements using advancing technology will likely produce further changes in the recommended value and reduction in its uncertainty.

6. Apparatus

6.1 The procedure is suitable for instruments, such as the atomic force microscope (AFM), which measures surface topography at high resolution in all three coordinate axes. The vertical (*z*) resolution must be close to or less than 0.1 nm, sufficient to resolve the 0.3-nm step heights. The lateral (*x*, *y*) resolution must be sufficient to resolve clearly steps separated by approximately 0.3 µm or less depending on the closeness of orientation of the surface normal to the Si *(111)* crystal direction.

7. Procedure

7.1 Perform setup and preliminary calibration of the AFM according to the manufacturer's instructions.

7.2 Mount the Si *(111)* specimen on the sample holder and align the Si *(111)* surface so that the step edges are as parallel as possible to the *y*-axis of the *x*-*y* displacement stage.

7.3 Measure a clean area of the specimen containing a fairly evenly separated series of steps and terraces. Avoid areas in which the steps pile up near a surface defect.

7.4 Measure an area containing about five or six terraces and monatomic steps.

7.5 Choose and block outline about four rectangular sub-areas of the measured step, each containing a single step approximately in the center (see Fig. 2). The topographic data *z* (*x*,*y*) in each sub-area should consist of a set of profiles *z*(*x*) each containing a single-sided step.

7.6 For each profile, calculate the step height using a single-sided step algorithm using two fitted straight lines, as schematically shown in Fig. 3. The measured step height, H_{meas}, is given by:

$$H_{meas} = (a_1 x_T + b_1) — (a_2 x_T + b_2) \quad (1)$$

Where:

x_T = the x-coordinate of the center of the step transition,
a_1 and b_1 = the parameters of the upper straight line, and
a_2 and b_2 = the parameters of the lower straight line.

7.7 If the surface profile has significant curvature, the two straight lines must be of equal length along the *x*-direction and equally spaced in *x* from the center of the step transition.

7.8 In each sub-area, average the step height results obtained for each profile over the set of profiles to obtain an average step height for the entire sub-area.

7.9 Average the results for the four areas to yield a step height value, H_{meas}, and calculate the standard deviation of the four results.

7.10 Recalibrate the *z*-scale of the AFM by correcting the gain or piezoelectric scanner sensitivity value of the instrument by the ratio:

$$R = 312\ pm/H_{meas} \quad (2)$$

7.10.1 Different instruments will have different recipes for accomplishing this. It may require several iterations to achieve a satisfactory result.

8. Report

8.1 The report shall contain the following information as appropriate:

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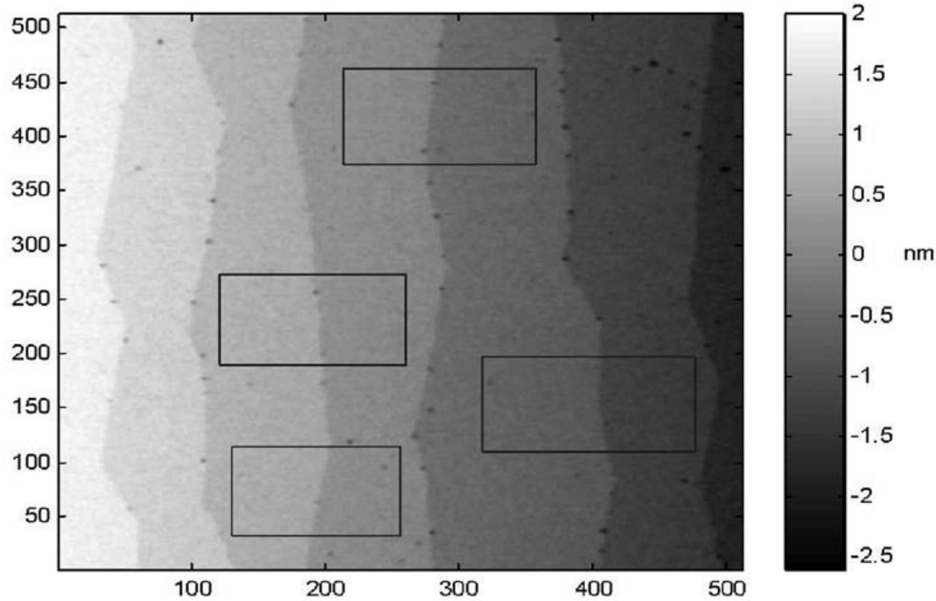

FIG. 2 Illustration of Selected Areas

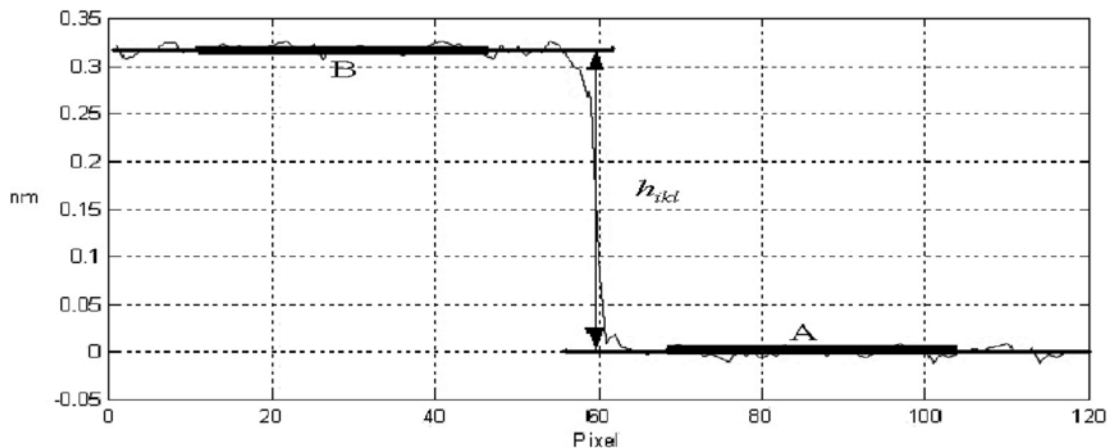

FIG. 3 Illustration of the Step-Height Algorithm Used

8.1.1 Date of test;

8.1.2 Operator identification;

8.1.3 Approximate temperature of the environment;

8.1.4 Description of specimen including vendor and specimen serial number;

8.1.5 Type, brand, model and serial number of instrument used;

8.1.6 Magnification settings of the instrument along all three coordinate directions;

8.1.7 Total number of measured sub-areas and their approximate locations and sizes;

8.1.8 An approximate number of data points in the measured profiles, and an approximate number of profiles used for a typical sub-area;

8.1.9 Average value of H_{meas} using the previously determined magnification factor before calibration;

8.1.10 Amount of correction, ratio of $312/H_{meas}$;

8.1.11 Typical graph of one of the measured profiles; and

8.1.12 Uncertainty budget showing all sources of uncertainty, both Type A and Type B, and the combined uncertainty calculated according to GUM: 1993.

8.1.13 As an example of an uncertainty budget, the one shown below is an extension of uncertainty budgets for NIST step measurement using master calibration steps.

8.1.13.1 *Type A*—(statistical) (k=1)

(1) Instrument noise and uniformity of sample to be measured (to be assessed by the user)—2%

(2) Instrument noise and Si *(111)* sample uniformity (to be assessed by user)—2%

8.1.13.2 *Type B*—(k=1)

(1) Z-scale nonlinearity (must be assessed by the user)—2%

(2) Si *(111)* step height estimation—1.9%

8.1.13.3 The above uncertainty budget is for an instrument calibrated by with Si *(111)* steps and subsequently used to measure an unknown sample. If the purpose of calibration is to

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determine the scale of the instrument only, the first item of type A in above example can be ignored.

8.1.13.4 The quadratic sum of the above values leads to an expanded uncertainty (k=2) of approximately 7 % given in Section 4.

9. Uncertainty

9.1 The combined standard uncertainty of the recalibrated scale includes the (Type A) relative standard deviation ($1\delta/H_{meas}$) obtained in 7.9 and a relative (Type B) standard uncertainty of 1.9 % for the recommended value of 312 pm, as well as other components associated with the particular instrument that must be estimated by the user. All components should be added in quadrature to yield a relative combined standard uncertainty.

9.2 A freshly made Si *(111)* surface should yield a smooth surface which leads to a good signal-to-noise ratio (that is, step height-to-rms roughness). However as time lapses and the surface of Si*(111)* degrades due to moisture and oxygen present in the ambient, the signal-to-noise ratio will reach unacceptable level at which time a new sample will be required

10. Potential Alternative

10.1 The procedure described in Section 7 is not limited to a Si *(111)* monatomic step specimen only. Similar procedures could be developed with lattice-step specimens fabricated from different materials and with different heights such as possibly 1-nm silicon carbide steps (**8, 9**). The linearity of the instrument can also be checked by using two different monatomic step height specimens.

11. Keywords

11.1 atomic force microscope; atomic steps; AFM; calibration; measurement; silicon; z-magnification;

ANNEX

(Mandatory Information)

A1. PROCEDURE FOR FABRICATION OF SI(111) MONATOMIC STEP HEIGHT SPECIMENS

INTRODUCTION

The subcommittee is aware of at least one commercial source for a Si atomic step specimen; however we describe two fabrication methods here for those who want to make their own.

A1.1 The Si *(111)* monatomic step specimen can be produced either by thermal heating in an ultra-high vacuum (UHV) chamber or by wet-chemical etching (see Fig. A1.1 and Fig. A1.2). However, for this step height calibration purpose, the heating method is preferred because of the better surface quality obtained.

A1.2 The average terrace width, *w*, is determined by the wafer miscut angle, θ, from the *(111)* plane and described by $w = 0.314\ \text{nm}/\tan\theta$.

A1.3 Thermal Technique—The heating can be produced by resistive heating through the sample itself or electron bombardment from the backside of the sample.

A1.3.1 Ultrasonically clean the sample.

A1.3.2 Outgas the sample at 600°C for more than 10 h.

A1.3.3 To maintain a stringent vacuum ($<10^{-7}$ Pa) and to avoid contamination, thermal cycle the sample with increasing temperature until the sample can be held at 1200°C for 1 min

A1.3.4 Cool down the sample rapidly to 900°C and follow by a slow cooling (1°C/min) to 700°C.

A1.3.5 Turn off the heating power and remove the sample until it reaches room temperature.

A1.4 Wet-Chemical Etching—The largest terrace that can be produced by this recipe is around 200 nm (miscut angle ~0.08°). It is limited by the etching mechanism. Proper tools and safety equipment are required to handle chemicals, particularly in handling of hydrofluoric acid (HF).

A1.4.1 4:1 mixture (by volume) of 98 % H_2SO_4: 30 % H_2O_2 at 40°C for 30 min to remove any organic residues.

A1.4.2 Ultrasonic rinse in ultra-pure water for 10 min twice.

A1.4.3 2 % HF for 1 min to remove the native oxide on the surface.

A1.4.4 5:1:1 mixture of H_2O: 35 % HCl: 30 % H_2O_2 at 80°C for 10 min to remove any metallic contaminants and to re-oxidize surface.

A1.4.5 Ultrasonic rinse in ultra-pure water for 10 min twice.

A1.4.6 Etching samples in argon-bubbled 40 % NH_4F, to reduce the dissolved oxygen in NH_4F.

A1.4.7 5- to 10-s ultra-pure water rinse.

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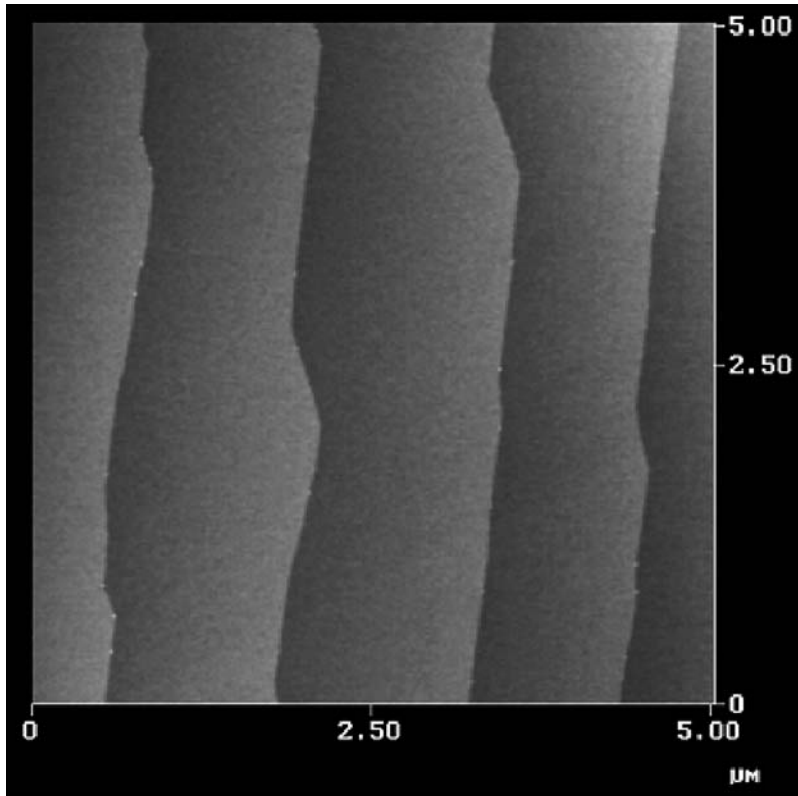

FIG. A1.1 Surface Produced by Thermal Heating (0.015° Miscut)

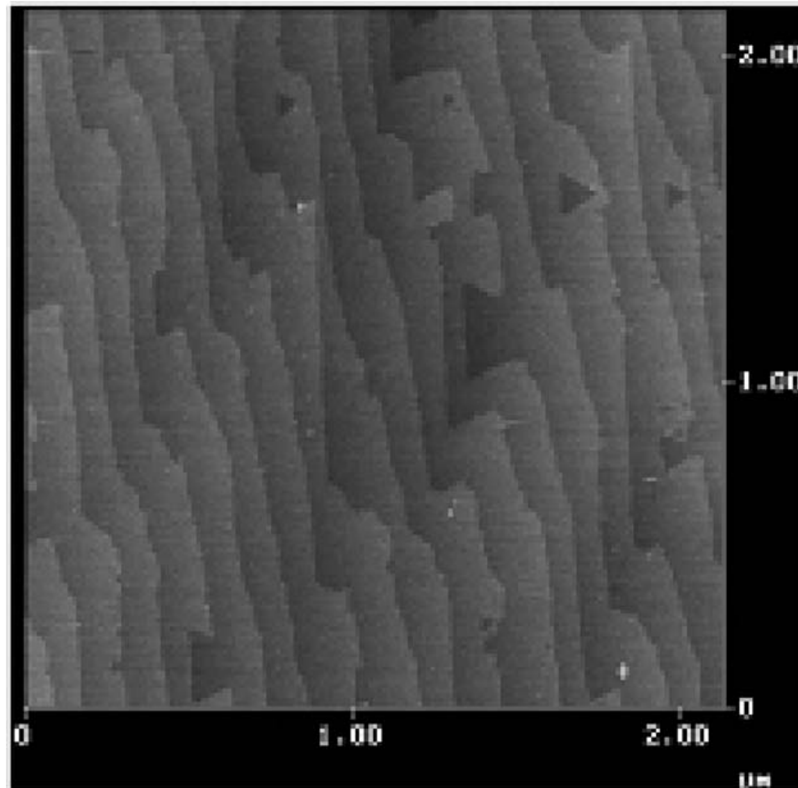

FIG. A1.2 Surface Produced by Wet-Chemical Etching (0.1° Miscut)

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REFERENCES

(1) CODATA International Recommended Values of Fundamental Physical Constants, http://physics.nist.gov/cuu/Constants/index.html.

(2) Tsai, V. W., Vorburger, T., Dixson, R., Fu, J., Köning, R., et al, "The Study of Silicon Stepped Surfaces as Atomic Force Microscope Calibration Standards with a Calibrated AFM at NIST," in Characterization and Metrology for ULSI Technology: 1998 International Conference, D.G. Seiler, A.C. Diebold, W.M. Bullis, T.J. Shaffner, R. McDonald, and E.J. Walters, Eds., AIP Press, 1998, pp. 839-842.

(3) Dixson, R. et al, "Silicon Single Atom Steps as AFM Height Standards," in Proc. SPIE 4344, Santa Clara, CA, March 2001.

(4) Suzuki, M. et al, "Standardized Procedure for Calibrating Height Scales in Atomic Force Microscopy on the Order of 1 nm," *J. Vac. Sci. & Technol.A*, Vol 14, No. 3, 1996 pp.1228-1232.

(5) Kacker, R., Datla, R., and Parr, A., "Combined Result and Associated Uncertainty from Inter-laboratory Evaluations Based on the ISO Guide," Metrologia,Vol 39 No. 3, 2002, pp. 279-293.

(6) Fu, J., Tsai, V. W., Köning, R., Dixson, R., and Vorburger, T. V., "Algorithms for Calculating Single Atom Step Heights," Nanotechnology, Vol 10, 1999, pp. 428-433.

(7) Ultra Clean Society Standard, Ultra Clean Society, Cosmos Hongo Bldg., 4-1-4 Hongo, Bunkyo, Tokyo 113, Japan.

(8) Abel, P. B., Powell, J. A., Neudeck, P. G., Method for the production of nanometer scale step height reference specimens, US Patent 6869480.

(9) Powell, J. A., Neudeck, P. G., Trunek, A. J., and Abel, P. B., "Step Structures Produced by Hydrogen Etching of Initially Step-Free (0001) 4H-SiC Mesas," in Materials Science Forum, vol. 483-485, Silicon Carbide and Related Materials 2004, R. Nipoti, A. Poggi, and A. Scorzoni, Eds. Switzerland: Trans Tech Publications, 2005, pp. 753-756.

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