**Designation: E2530 – 06**

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

## 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 standard. The values given in parentheses are mathematical conversions to inch-pound units that are provided for information only and are not considered standard.

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 *Definitions of Terms Specific to This Standard:*

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
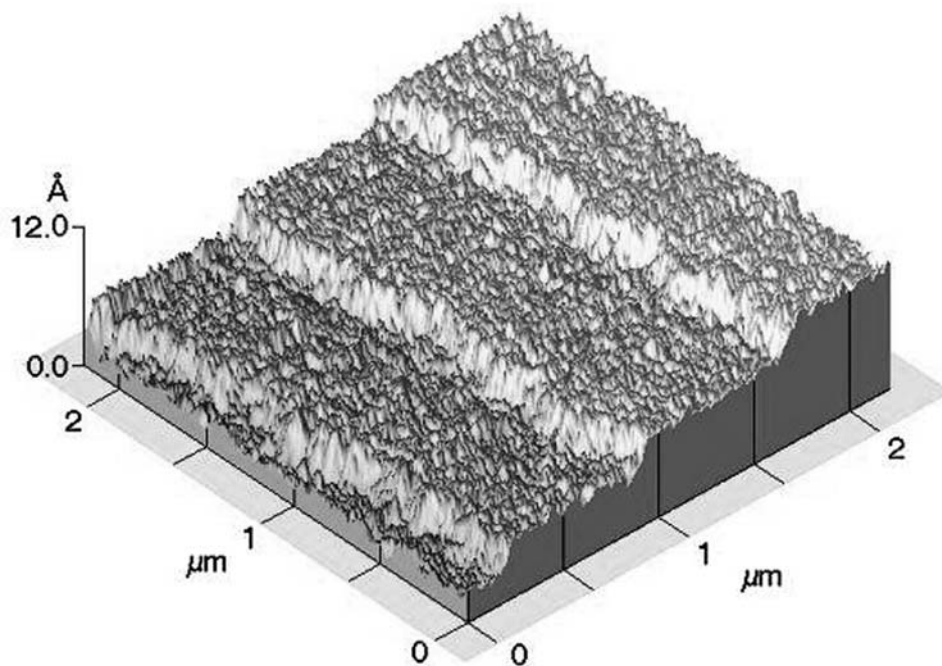
**FIG. 1 Image of a Silicon Monatomic Stepped Surface**

be 314 pm based on X-ray value of lattice parameter of bulk silicon (**1**).[3] However, recent analysis (**2-7**), also taking into account independent measurements by other sources, has 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:

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

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:

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

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[3] The boldface numbers in parentheses refer to a list of references at the end of this standard.
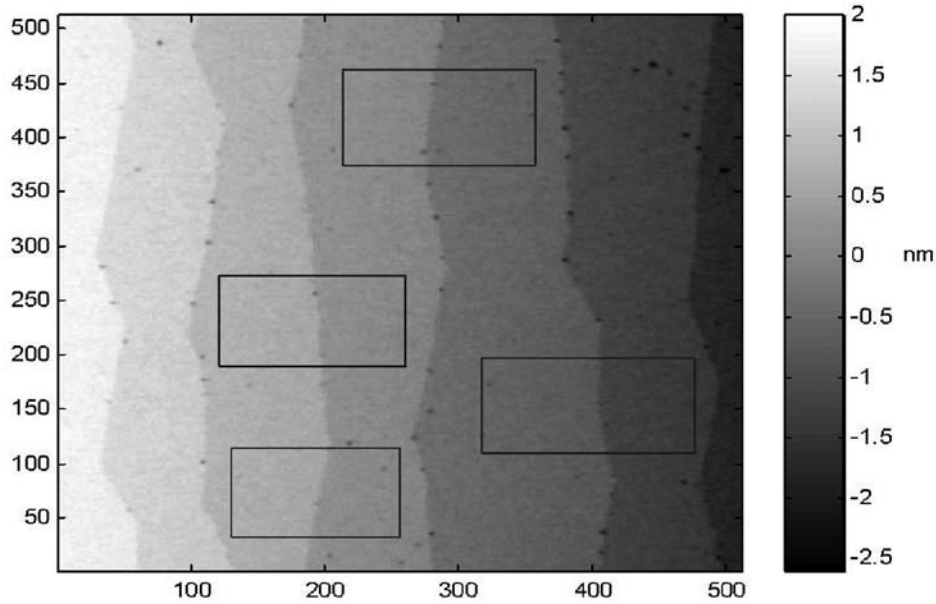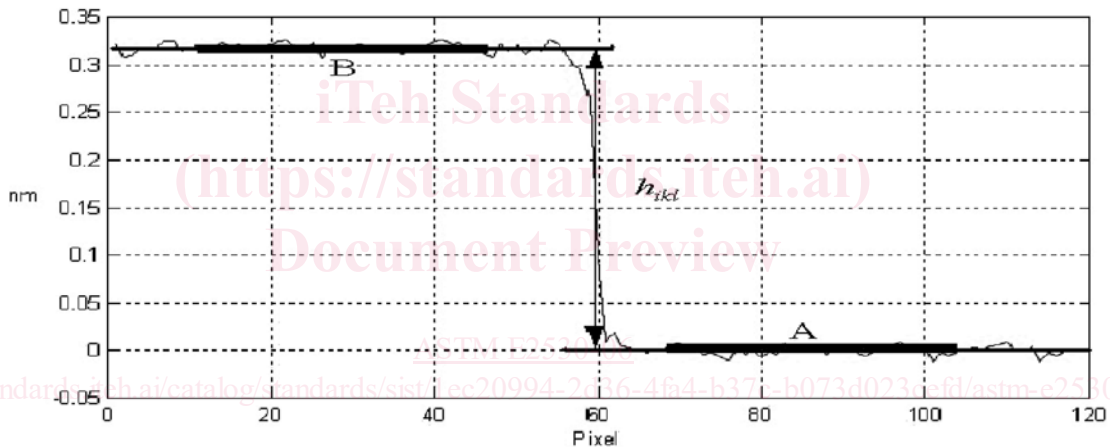
**FIG. 2 Illustration of Selected Areas**



**FIG. 3 Illustration of the Step-Height Algorithm Used**

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:

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%