Proceedings of the Fifth International Conference on Precision, Meso, Micro, and Nano Engineering, Edited by B. Anil, K. Sunilkumar, and V. Radhakrishnan (Allied Publishers Pvt. Ltd., Chennai, India, 2007) pp 13-18.

# Nano- and Atomic-Scale Length Metrology

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## Abstract

We review several ongoing projects at NIST and SEMATECH in linewidth and step height metrology. Linewidth measurements using critical dimension atomic force microscopes have quoted uncertainties down to  $\pm 0.63$  nm, with coverage factor (k) = 1. Step height measurements of the 0.3 nm monatomic step heights on Si(111) have been performed with an uncertainty of approximately  $\pm 4$  pm (k=1), and a procedure for using these surfaces as step height calibration standards has been developed as ASTM Standard 2530-06.

*Keywords*: atomic force, documentary standard, linewidth, step height, transmission electron microscopy

### 1. Introduction

We review several ongoing projects at NIST and SEMATECH in nano-scale length metrology of linewidth and step-height. These properties are of growing importance to the function and specification of semiconductor devices as the dimensions of semiconductor devices shrink to the 50 nm level and below. The ability to manufacture ever smaller linewidths leads to semiconductor elements with increasing speed and storage Critical dimensions (linewidths) of density. semiconductor features serve as the driving specification in the International Technology Semiconductors (ITRS) [1]. Roadmap for Accurate metrology is required to determine whether manufacturing specifications for critical dimensions are being achieved. Calibrated step heights provide a source of calibration for the zscales of atomic force microscopes, which are used to measure surface roughness and feature heights with sensitivities down to atomic scales.

#### 2. Linewidth

Cresswell et al. [2] have developed a physical standard of calibrated linewidths, called the Single Crystal Critical Dimension Reference Material (SCCDRM). The standards consist of Si chips with Si(110) single crystal surfaces with patterns of six calibrated lines (Fig. 1) having widths ranging from about 70 nm to about 225 nm.



#### Fig. 1 Layout of a single pattern of the SCCDRM [2] showing six lines having widths decreasing from top to bottom. Arrows incorporated into the design indicate the calibrated measurement positions for AFM scans.

These lines are fabricated using directional etching techniques, which produce vertical sidewalls and uniform widths. The lines have been measured by critical dimension atomic force microscopes (CD-AFM) at NIST and SEMATECH. An important source of uncertainty here is the width of the AFM probe itself, which causes an offset in the measured results. These probe tip offsets are determined using high-resolution transmission electron microscopy (HR-TEM), when the same linewidths are measured by both techniques and the results are compared. Figure 2 shows the offsets measured between the CD-

AFM results and the HR-TEM results for measurements on twelve lines.

Also shown is the resulting average tip-offset correction and its uncertainty of  $\pm$  0.58 nm, with coverage factor (*k*) = 2 [3], an important source of uncertainty for the CD-AFM linewidth measurements. The combined uncertainty of the calibrated widths also includes non-uniformity of the lines, AFM reproducibility, and statistical variations of the measurements, along with

uncertainty in the offset correction. The combined uncertainty ranges down to  $\pm$  0.63 nm (k =1) depending on the linewidth being calibrated. Recently we have verified the small uncertainties of these results by two additional independent comparisons using the same general approach of comparing CD-AFM with TEM measurements of the same linewidths [4, 5]. A result of Orji et al. [5] for one of these comparisons on an 18 nm linewidth is shown in Fig. 3.



Fig. 2 Offsets between the linewidths measured with CD-AFM and HR-TEM [2]. Measurements were taken on six lines each on chips A7 and D1. For each, "1" is the narrowest line and "6" the widest. The weighted mean offset is the point shown at the right and it functions as an additive correction factor to subsequent CD-AFM results. The uncertainties shown are k = 2.



Fig. 3 Comparison [5] of 18 nm linewidths measured with HR-TEM, HAADF-TEM, CD-AFM 1 located at SEMATECH and CD-AFM 2 located at NIST. The uncertainties shown are all standard uncertainties, k = 1.

A slightly different TEM imaging method, called high angle annular dark field scanning TEM (HAADF-TEM), was used here and is shown along with HR-TEM results and results obtained with CD-AFM 1 at SEMATECH and with CD-AFM 2 at NIST. The agreement between all the measurements is very good. Differences between any two results are smaller than their combined uncertainties.

We are currently researching a second technique for linewidth measurement, which is intended to be independently traceable to the SI unit of length. The image stitching approach [6] relies on the high-resolution surface imaging capability of an atomic force microscope fitted with a nanotube probe. Because the orientation of the nanotube is likely not perpendicular to the surface, an AFM scan of a line feature with such a probe yields a relatively sharp accurate profile on one side and a distorted profile on the other. The image stitching procedure is shown in Fig. 4. Each side of a semiconductor line is imaged by turns with minimal distortion, and the two images are stitched together by matching the topographies measured on the top of the line. The result is a single image of a line having minimal distortion of the steep sidewalls on both sides. The current uncertainty of this approach, about  $\pm 18$  nm (k=1), is limited by the width of readily available nanotubes on AFM cantilevers.

Compliance of the nanotube will also likely become a significant source of uncertainty for the image stitching approach as the mounted nanotube widths become smaller. In response, we are developing models for the deflections of AFM probes resulting from interaction with the measured surface [7]. The models include the mechanical properties of the probe and a Lennard-Jones model for the force of interaction as a function of distance between the tip and surface. The deflection of a nanotube probe upon contact is governed by a snap-in / snap-out behavior [8]. We have done a preliminary model calculation of the snap-in deflection for a probe model similar to the probe used in the image stitching work discussed above and obtained a deflection of about 1.1 nm. This contributes to one component of the 18 nm uncertainty stated above.

## 3. Step Height

Traceable, commercially available step height standards may be obtained ranging down to heights of about 7 nm. These step heights are useful for the calibration of the z-range of surface profiling instruments such as stylus instruments, optical profilers, and atomic force microscopes. For calibration at smaller heights, one can envision the need for calibrated step height standards of approximately 3 nm, 1 nm, and 0.3 nm. The monatomic steps on single crystal Si(111) (Fig. 5) have been studied [9] for their usefulness as step height standards at the 0.3 nm level of amplitude. We have performed an independent calibration of the 0.3 nm monatomic step height and have been working on a practical procedure for using it to calibrate atomic force microscopes working at the atomic z-scale level.



Fig. 4 Illustration of the AFM image stitching concept. Between the first image and the second image, the sample is rotated 180°.





The measurements of the Si(111) step height were performed using a calibrated atomic force microscope (C-AFM) [10]. With this instrument, displacements in the z-direction are measured using a capacitance gauge. In turn, the capacitance gauge is calibrated in situ with a displacement interferometer, generally on each step height measurements dav that are Our result for the monatomic step performed. height, averaged over six sets of measurements taken over five months on two different samples, was 304 pm ± 8 pm (k=2) [11, 12]. This value may be compared with a value of approximately 314 pm calculated from x-ray diffraction of bulk silicon to determine the lattice constant. The latter value has an extremely low fractional uncertainty

of  $6X10^{-8}$  (*k*=2) but does not represent a direct measurement of the step itself, which resides on the surface. Using a Type B uncertainty analysis [3], we combined these two results along with values obtained by electron and x-ray diffraction of the stepped surface (Fig. 6) to provide a recommended calibration value of the Si(111) step height of 312 pm ± 12 pm (*k*=2) [11, 12], which is then useful for calibrating atomic force microscopes working at their highest levels of magnification.

We then developed a procedure for calibration of atomic force microscopes using the Si step. The procedure included a surface sampling procedure and a profile-based step height algorithm. Α laboratory comparison of measurements of five laboratories, including our own, was then completed to test the usefulness and variability of the procedure. The results suggest that calibration of the z-scale of an atomic force microscope can be performed with an uncertainty of about 7 % (k = 2) using a reasonable sampling procedure. A documentary standard based on this approach was developed and approved by ASTM Subcommittee E42.14 on STM/AFM and was recently published [13]. The profile-based algorithm is illustrated in Fig. 7. The step height is calculated from fitted straight lines of equal length and with equal offsets from the center of the step. This algorithm provides a stable result, which is insensitive to slope errors and to curvature errors in the surface profiles.



Fig.6 Comparison of four values for the monatomic step height on a Si(111) single crystal surface. The recommended value [11, 12], shown at the right, is the result of a Type B analysis [3] of four measured values, which are, from left to right, the bulk lattice value determined by x-ray diffraction, low energy electron diffraction (LEED), the C-AFM work discussed here, and grazing incidence x-ray diffraction. The uncertainties shown are k = 2.



Fig.7 Typical profile of a Si(111) monatomic step illustrating aspects of the step-height algorithm used to calculate the step height (H). The data used to fit the straight lines are shown schematically by the bold double arrows.

The Si(111) steps may be manufactured using ultrahigh vacuum techniques or an experimental wet chemistry approach [13, 14] and have also been available commercially\*[15]. Gaps still remain in the chain of standards for calibrated step heights. However, Powell et al. [16] have developed structured, stepped samples on SiC with 1 nm step heights. If this effort is successful for producing useful, economical step height standards at the 1 nm level, then the only gap in a series of available step height standards ranging in height from several  $\mu$ m to 300 pm with at least two calibrated step values per decade of height would be at the 3 nm step-height level.

#### 4. Continuing Work

The SCCDRMs were distributed to member companies of SEMATECH. Work has begun to develop a similar linewidth standard as a NIST Standard Reference Material, and a similar linewidth standard is now available commercially [17]. Over the past several years, line edge roughness (LER) has become an important quality issue for semiconductors and is now a quantity specified in the ITRS [1]. Orji et al. have been exploring the capability for measuring LER with CD-AFM [18]. For studies of this nature, Park et al. [19] have developed procedures for fabrication of carbon nanotube probes with controlled orientations and shapes and are researching the behavior of these new probes when mounted in CD-AFM. Work on a calibration procedure for AFMs using Si (111) monatomic steps is considered to be complete with the publication of ASTM 2530-06 [13]. The procedure likely can be extended to using 1 nm steps as well for step height calibration standards.

#### 5. Acknowledgements

The authors are grateful to J. Marshall and J. Kramar for their comments concerning this manuscript and to J. Waters for assistance in its preparation. The work was supported in part by the Office of Microelectronic Programs at NIST

\* Certain commercial equipment, instruments, or materials are identified in this paper to foster understanding. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

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