Traceable calibration of critical-dimension atomic force microscope linewidth measurements with nanometer uncertainty

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The use of critical dimension atomic force microscopes (CD AFMs) in semiconductor manufacturing, both for process control and as a reference metrology tool, is increasing. If the tip width is calibrated consistently between measurements, a CD AFM can function as an excellent width comparator. Relative widths can be measured with uncertainties of 1 nm or less. However, to perform accurate measurements, the absolute tip width must be accurately calibrated. Until recently, conventional methods for accomplishing this had standard uncertainties on the order of 5 nm. Recently developed CD reference materials now make it possible to calibrate absolute tip width with uncertainties at the 1 nm level. The highlights of our method are: (1) the use of single-crystal silicon and preferential etching to pattern well-defined and highly uniform features; (2) the use of high resolution transmission electron microscopy (HRTEM) to access the Si lattice spacing directly as a source of traceable width information, and (3) the use of CD AFM to transfer width information from the HRTEM samples. These standards are known as single crystal critical dimension reference materials (SCCDRM), and prototype SCCDRMs have recently been delivered to SEMATECH Member Companies for evaluation. [DOI: 10.1116/1.2130347]

I. INTRODUCTION

The National Institute of Standards and Technology (NIST) and SEMATECH have been working together to improve the accuracy of critical dimension atomic force microscope (CD AFM) dimensional metrology in semiconductor manufacturing. A major component of this collaboration has been the development of a CD AFM reference measurement system (RMS) at SEMATECH using a current generation CD AFM. The implementation of the CD AFM RMS is described in detail elsewhere.[1–4] In this article we focus on CD AFM linewidth metrology and the reduction in uncertainty that is now possible as a result of the NIST/SEMATECH single crystal critical dimension reference material (SCCDRM) project.

In developing our uncertainty budgets for CD AFM pitch, height, and linewidth measurements, we used the approach recommended by the International Organization for Standardization (ISO).[5,6] Generally, this consists of developing an estimated contribution for every known source of uncertainty in a given measurement. Those contributions that are evaluated by statistical methods are known as type A evaluations; other terms, known as type B evaluations, are estimated using some combination of measured data, physical models, or assumptions about the probability distribution.[6] All the terms are then added in quadrature to obtain a combined standard uncertainty for the measurement. (Note that for a type A evaluation, the standard uncertainty will correspond to an observed standard deviation.) The standard uncertainty is usually multiplied by a coverage factor k to obtain an expanded uncertainty. The most common coverage factor used is $k=2$, which would correspond to approximately 95% confidence for a normal (Gaussian) distribution. The standard uncertainties for CD AFM pitch and height measurements are approximately 0.2% and 0.4%, respectively. Prior to the SCCDRM project, CD AFM linewidth measurements were limited to a standard uncertainty of about 5 nm. However, this limit can now be significantly reduced.

A new generation of the SCCDRM samples was released to SEMATECH member companies during late 2004.[7] CD AFM was used to measure the linewidths of selected features on the distributed specimens. To reduce the uncertainty in tip width calibration, a separate transfer experiment was performed in which samples were measured by CD AFM and then sent for high resolution transmission electron microscopy (HRTEM). In this manner, CD AFM could be used to transfer the HRTEM width information to the distributed samples. Consequently, we are now able to reduce the limit on the standard uncertainty ($k=1$) of CD AFM width measurements to 1 nm.

II. FABRICATION OF SCCDRM SPECIMENS AND HRTEM METROLOGY

The background and history of the SCCDRM project has been described in more detail elsewhere.[8,9] Features on the SCCDRM samples are preferentially etched into a {110} silicon-on-insulator substrate, so the sidewalls are near vertical. The starting material is produced using separation by implantation of oxygen and has a low resistivity device layer and a 20 nm thick layer of silicon dioxide to serve as a hard mask. To ensure that the pattern is aligned so that its principal axes are orientated to ⟨112⟩ directions of the silicon lattice, a special-purpose angular fiducial pattern is imaged to the hard mask and transferred to the silicon with a deep,

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lattice-plane-selective etch. Lattice orientation is subsequently determined from visual inspection of the features of the pattern. The wafer is coated in photoresist, which is patterned. The design is then transferred to the hard mask using i-line lithography followed by a buffered oxide etch (BOE) dip. Finally, it is transferred from the hard mask to the SIMOX device layer by a 25 s dip in a 25% solution of tetramethylammonium hydroxide at 83 °C with ultrasonic agitation. The remaining hard mask is removed in BOE.

Due to the crystalline nature of the SCCDRM features, it is possible to use the silicon lattice constant as a source of width information when the structures are measured using HRTEM. Since HRTEM is destructive, however, we used CD AFM as a comparator to measure the relative widths of all the features prior to HRTEM measurement. The SCCDRM chips were prepared for HRTEM using a process that has been optimized to ensure that the surfaces of the reference feature are not damaged. Each step of this three-step process serves to define the region and protect the sidewalls of the features from the subsequent, higher energy, thinning steps. The first step is deposition, by sputtering or evaporation, of a gold-palladium film to protect the surfaces of the reference feature during the process steps which follow. After coating, the sample is placed in a focused ion beam/scanning electron microscope (FIB/SEM) tool to mark the region of interest for cross sectioning with an electron-beam assisted platinum film that is approximately 0.5 μm by 8 μm. The final deposition step is that of a large, 8 μm by 20 μm, protective platinum box. This box covers the central region of the test structure.

At this point, the test chip is removed from the FIB/SEM and tripod polished to a thickness of 30 μm, both in the direction of the cross section and from the back side of the wafer. This 30 μm thick sliver is then silver mounted on a half grid, returned to the FIB/SEM, and thinned along the axis perpendicular to the desired cross section. At the beginning of this process, a 30 kV gallium beam is used for rapid thinning; for final thinning, a 10 kV beam is used to prevent damage to the reference features. This thinning targets the center of the 0.5 μm region defined by the electron-assisted platinum mark and continues until the reference features become electron transparent.

The SCCDRM layout was designed so that six features of different widths could be included in a single TEM cross section. Consequently, seven images were made of each sample: a low magnification (10 k$\times$) image showing all six of the reference features and six high magnification (400 or 600 k$\times$) images of the individual reference features. The images were captured on 80 mm by 100 mm negatives which were scanned at 900 dots/p in.; following this procedure, each of the individual images of the six features is captured with enough detail to resolve the silicon lattice. A high magnification (400 k$\times$) image negative of the narrowest reference feature measured is shown in Fig. 1; shown in the inset is a portion of the edge region showing the silicon lattice and the transition to the surrounding native oxide and metal coating.

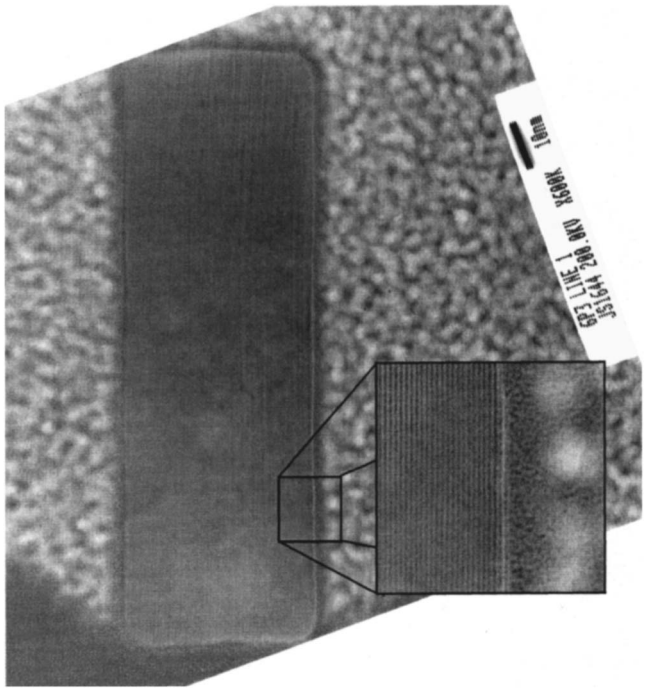

FIG. 1. Negative of the high-magnification (400 k$\times$) HRTEM image of the narrowest feature used. At this magnification, the silicon lattice fringes are visible as can be seen in the enlarged portion of the sidewall shown in the inset.

To extract the linewidth from each HRTEM image, the fringes were counted manually by four operators on most features, and more details of this procedure have been published elsewhere.[10] The agreement between counters was at the subnanometer level. The standard deviation of the results was typically 0.2 nm to 0.9 nm on the features that were ultimately used for the calibration analysis.

III. CD-AFM LINEWIDTH METROLOGY

The operation of CD AFM is more sophisticated than conventional, top-down AFM. Some unique aspects of CD AFM operation are that feedback occurs along both lateral and vertical axes and flared tips are used. This allows imaging of structures with near-vertical sidewalls such as commonly occur in semiconductor processing. Like most AFMs, the displacement metrology in the CD AFM is not intrinsically traceable, and so scale calibration is one important source of uncertainty. Previous descriptions of the RMS project have included uncertainty budgets for CD AFM pitch, step height, and linewidth measurements.[1–4] For linewidth measurements in the 100 nm range, however, the most significant source of uncertainty pertains to correction for the size of the tip.

Although the interaction of an AFM tip with the imaged surface is complex, for many purposes a highly simplified and two-dimensional model can be useful. In this basic model, the effect of the tip is represented as a simple additive offset which must be subtracted from the apparent width to obtain an accurate measurement. We refer to this offset as the zeroth order tip correction, and there is an uncertainty component that represents the uncertainty in this offset. Since the

offset is determined from the apparent width of a reference structure, the zeroth order uncertainty component is closely linked to the uncertainty in the width of the reference structure. Prior to the SCCDRM project, the standard uncertainty in this reference width was estimated to be 5 nm.[2,3]

It is important to note that the standard uncertainty in the zeroth order correction represents an uncertainty in the absolute value of the linewidth. For some applications, the absolute uncertainty is of less interest than the relative uncertainty between two given width measurements. This uncertainty can be significantly smaller than 5 nm. For some circumstances, this relative uncertainty between two width measurements will be on the order of 1 nm or less.

The finer details of the tip-sample interaction, pertaining to things like flare radius, feature sidewall angle, feature corner radius, and the three-dimensional nature of both the tip and sample are thought of as being higher-order tip effects. The idealized treatment of the tip width as an additive offset does not incorporate any of these considerations. We have given more discussion of higher order tip effects elsewhere.[1] Because these effects have a strong dependence on the specific geometry of each tip and feature, it is difficult to make general statements about the resulting uncertainties, and it is necessary to make a specific assessment for every measurement. Previously, these effects were often of secondary concern since these components were typically smaller than the uncertainty in the zeroth order correction. However, with the current reduction in the zeroth order uncertainty, characterization and correction for these effects will become more important in CD AFM width measurements.

IV. SCCDRM METHODOLOGY AND CALIBRATION UNCERTAINTY ANALYSIS

For the SCCDRM project, the features had a geometry that was close to an ideal rectangular cross section. Consequently, the higher order tip effects were generally negligible in the measurements. Therefore, when using the CD AFM as a width comparator on the SCCDRM structures, the relative uncertainty between measurements is largely dependent upon the stability of the tip width and the consistency of whatever tip width calibration method is used. Note that the method does not need to be accurate in an absolute sense. That is, a bias is acceptable, but it must be consistent from one measurement to the next.

We achieved consistency by using one of the SCCDRM chips as a monitor sample to anchor the tip width calibration. The monitor sample was measured before and after every measurement of a target sample. In this manner, we could detect any change in the tip width and achieve tip calibration consistency among our measurements. Once the relative widths of the SCCDRM features were measured, those samples chosen for HRTEM were cross sectioned. Once these transfer samples were measured by HRTEM, the linewidths of the remaining samples were then known relative to the TEM values. The silicon lattice constant is extremely well known relative to the SI meter, but establishing traceability of linewidth results based upon HRTEM also requires a careful methodology in order to rule out possible sources of error. While there are potential effects that could lead to biased results and increased uncertainty, these were addressed by our methodology and results.

During the SCCDRM project, we used four HRTEM transfer samples—each having six features of different widths. The HRTEM fringes were counted, and the images were analyzed in detail. Based upon the visibility of the fringes and the uniformity of the interfaces, we concluded that most potential biases pertaining to sample preparation (e.g., surface effects, membrane distortion, FIB-induced damage) were not an issue. However, we did conclude that there was a probable issue involving contamination and its removal during the HRTEM sample preparation.

The results of our analysis suggested that two of the HRTEM samples had been slightly contaminated, probably with hydrocarbon deposited during the prior SEM inspection, at the time of the AFM measurements. This caused a bias in the AFM/HRTEM comparison for those two samples. However, the results from the other two samples were in agreement, and we concluded that these were not biased by contamination. This conclusion is also consistent with a difference in preparation between the samples: The two chips biased by contamination received a less aggressive cleaning process following the SEM inspection.

Another important internal check on our results was consistency of the scale calibration. The lateral scale of the CD AFM was independently calibrated during the development of the RMS.[3,4] During the SCCDRM measurements, the scale calibration was closely monitored to ensure its stability. To check the absolute value of the scale calibration, the observed slope of the AFM/HRTEM regression curve, $0.996 \pm 0.005 (k=1)$, was used. Since this is consistent with unity (i.e., in agreement with the prior scale calibration), we were able to treat the HRTEM/AFM comparison data as a direct measure of the bias in the existing tip width calibration and calculate the offset between the HRTEM and the AFM results. Therefore, we did not need to rely on the HRTEM results to establish the scale calibration, but only to establish the value of the effective tip width during the measurements.

The observed offsets between the HRTEM and AFM results for each of the features used are shown in Fig. 2. The expanded uncertainties shown include the reproducibility of both the HRTEM and AFM results, as well as the influence of relative positioning uncertainty and feature nonuniformity. Using a weighted average to combine the results from all 12 features allows a determination of the offset with an expanded uncertainty of less than 1 nm.

The measured offset of 1.03 nm was well within the 5 nm uncertainty of the prior tip calibration. Since both calibrations are traceable and intended to represent the same measurand, this observed agreement is significant. Furthermore, this comparison also supports our conclusion that two of the HRTEM chips suffered from contamination. The two chips thought to be contaminated showed larger offsets between the HRTEM result and the prior AFM tip characterizer result.

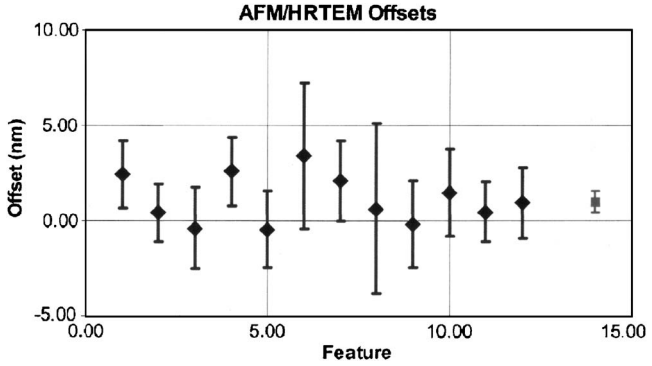

FIG. 2. Comparison of the individually estimated AFM offsets and the weighted-mean offset. The uncertainties shown in the plot are the expanded uncertainties ($k=2$). The uncertainties include the reproducibility of both the HRTEM and AFM results and the influence of relative positioning uncertainty. The weighted average and expanded uncertainty ($k=2$) are 1.03 nm±0.58 nm.

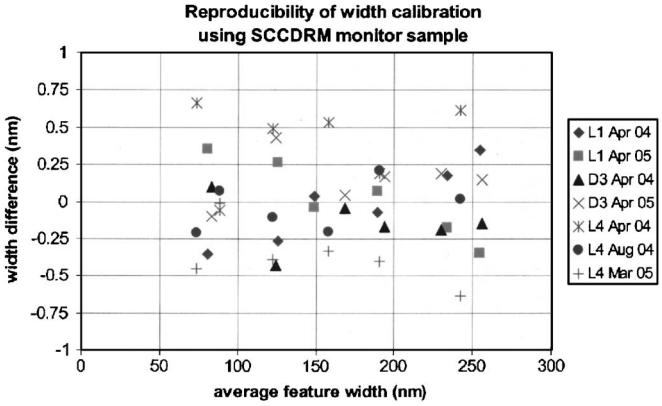

FIG. 3. Reproducibility of linewidth measurements on three SCCDRM chips: K143 L4, K147 L1, and K143 D3. All widths are measured relative to the SCCDRM monitor sample, indicating that a realization of tip width relative to the SI meter can be accomplished with a standard uncertainty of 1 nm or less.

For some of the features, the differences were as large as 12 nm—enough to be in unambiguous statistical disagreement.

Potential biases in the HTREM results pertaining to sample preparation were discussed above. With the exception of contamination, we believe that such effects are probably less than 0.1 nm. As to potential biases in the HRTEM metrology itself, all known effects would only impact the fringe contrast and visibility which would bias the determination of the silicon/oxide interface. However, in our analysis of the HRTEM images we only measured the location of the oxide/Au–Pd interface. The uncertainty associated with this determination results largely from interface uniformity and is included in the statistical uncertainty of the HRTEM widths determined by different counters. Consequently, all significant sources of uncertainty have been included in our analysis.

A. Reproducibility of SCCDRM tip width calibration

The reproducibility of measurements on three SCCDRM chips (numbers K143 L4, K147 L1, and K143 D3) is illustrated in Fig. 3. All widths were measured relative to the SCCDRM monitor sample (chip K162 K). The history includes results obtained over 1 year using two different heads on the CD AFM. The measurements taken in April of 2004 were part of the main body of measurements for the SCCDRM project. Followup measurements on the K147 L1 and K143 D3 chips were taken in April of 2005. The K143 L4 chip was installed in the CD AFM as a system monitor during the year. Although only three measurement sets on the L4 chip are shown in the figure, many more results were available, and some have been discussed elsewhere.[1]

The observed variability indicates that the typical reproducibility is approximately 0.5 nm (1σ), and this conclusion also is supported by the complete monitor history on the L4 chip. This means that it is possible to realize a consistent calibration of tip width at the level of 0.5 nm. The results of the SCCDRM project also permit this calibration to be traceable with a standard uncertainty ($k=1$) of 1 nm or less.

V. SUMMARY AND FUTURE WORK

NIST has completed the SCCDRM project, and chips were distributed to SEMATECH member companies for evaluation. On the distributed chips, the feature widths ranged from approximately 50 to 250 nm, and the expanded uncertainties ($k=2$) were typically 2 nm, although some were as low as 1.5 nm. As a result of this work, it is now possible to traceably calibrate CD AFM tip width with a standard uncertainty ($k=1$) of 1 nm or less. In future work, we hope to refine the fabrication process to improve feature uniformity, reduce the presence of contamination, and include data acquired over multiple measurement runs to reduce the statistical uncertainty.

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