

Single Crystal Critical Dimension Reference Materials (SCCDRM): Process Optimization for the Next Generation of Standards

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ABSTRACT

Critical dimension atomic force microscopes (CD-AFMs) are rapidly gaining acceptance in semiconductor manufacturing metrology. These instruments offer non-destructive three dimensional imaging of structures and can provide a valuable complement to critical dimension scanning electron microscope (CD-SEM) and optical metrology. Accurate CD-AFM metrology, however, is critically dependent upon calibration of the tip width. In response to this need, NIST has developed prototype single crystal critical dimension reference materials (SCCDRMs).

In 2004, a new generation of SCCDRMs was released to the Member Companies of SEMATECH – a result of the fruitful partnership between several organizations. These specimens, which are fabricated using a lattice-plane-selective etch on (110) silicon, exhibit near vertical sidewalls and high uniformity and can be used to calibrate CD-AFM tip width to a standard uncertainty of about ± 1 nm.

Following the 2004 release, NIST began work on the “next generation” of SCCDRM standards. A major goal of this thrust was to improve upon the SCCDRM characteristics that impact user-friendliness: the linewidth uniformity and cleanliness. Toward this end, an experiment was designed to further optimize the process conditions. The first round of this experiment was recently completed, and the results show great promise for further improvement of the SCCDRM manufacturing process.

Among other observations, we found that the minimum linewidth and linewidth uniformity were primarily sensitive to different factors - and can thus be independently tuned to meet our future goals – which include linewidths as small as 20 nm and a standard uncertainty due to non-uniformity at the ± 0.5 nm level. Our future work will include a new refining experiment to further optimize the important factors that we have identified, and extension of the methodology to a monolithic 200 mm implementation.

Keywords: CD-AFM, metrology, CD, linewidth, SCCDRM, standards, calibration, traceability, uncertainty

1. INTRODUCTION

Critical dimension atomic force microscopes (CD-AFMs) are rapidly gaining acceptance in semiconductor manufacturing metrology. These instruments offer non-destructive three dimensional imaging of structures and can provide a valuable complement to CD-SEM and optical metrology. Accurate CD-AFM metrology, however, is critically dependent upon calibration of the tip width.¹⁻³ In response to this need, NIST has developed prototype single crystal critical dimension reference materials (SCCDRMs).²⁻⁵

In 2004, a new generation of SCCDRMs was released to the Member Companies of SEMATECH – a result of the fruitful partnership between NIST, SEMATECH, and VLSI Standards.[†] These specimens, which are fabricated using a lattice plane selective etch on (110) silicon, exhibit near vertical sidewalls and high uniformity and can be used to calibrate CD-AFM tip width to a standard uncertainty of about ± 1 nm.

[†]Certain commercial equipment is identified in this paper to adequately describe the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology nor does it imply that the equipment identified is necessarily the best available for the purpose.

Following the 2004 release, NIST began work on the “next generation” of SCCDRM standards. A major goal of this thrust was to improve upon the SCCDRM characteristics that impact user-friendliness: the linewidth uniformity and cleanliness. Toward this end, an experiment was designed to further optimize the process conditions. The last phase of this experiment was recently completed, and our results showed great promise for further improvement of the SCCDRM manufacturing process.

Among other observations, we found that the minimum linewidth and linewidth uniformity were primarily sensitive to different factors - and can thus be independently tuned to meet our future goals. Additionally, we observed feature widths as small as 20 nm and a standard uncertainty due to non-uniformity as low as ± 0.45 nm. The results also point the way to natural extensions of the experiment and process.

We are currently working on a refining experiment to further optimize the important processing factors that we have identified. Our strategic goal is to develop a NIST Standard Reference Material (SRM) based on our SCCDRM technology. We are considering both a chip-based (as in the 2004 release) and a monolithic 200 mm wafer implementation for this standard. The measurement focus is expected to be on isolated lines that are most suitable for CD-AFM calibration, but we are also exploring application of this technology to grating standards for scatterometry.⁶

1.1 CD-AFM Tip Width Calibration

There are many important metrology applications of CD-AFM in semiconductor manufacturing. However, the most common is linewidth metrology – for measurement of gate CD or as a reference tool for in-line critical dimension scanning electron microscopes (CD-SEMs). For this application, it is especially important to minimize the uncertainty of the CD-AFM width measurements.

The scale calibration uncertainties that limit the accuracy of CD-AFM pitch measurements also apply to linewidth metrology, but these are not typically the most significant sources of uncertainty in deep submicrometer linewidth measurements. Instead, the most challenging source of uncertainty in width measurements is the calibration of the tip width and shape.

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 the uncertainty in the value of the tip width to subtract as the zeroth order tip width uncertainty.^{1,2}

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 (*i.e.*, shape in the orthogonal axis) are thought of as being higher-order tip effects. The idealized geometry does not incorporate any of these considerations. While higher order effects can be important in the measurement of less uniform structures such as photoresist lines, these effects are often negligible for measurement of relatively uniform structures – particularly those with near-vertical sidewalls.

CD-AFM users have typically developed in-house reference standards for tip width calibration – often based on SEM or transmission electron microscope (TEM) cross sections. But the resultant uncertainty of such standards can be significant. Tip characterizer samples - which have a sharp ridge that can be used to calibrate tip width - are commercially available. However, scanning such samples can result in tip damage, and the standard uncertainty of tip calibrations based on this method is limited to about ± 5 nm.

The NIST response to this pressing need for more accurate CD-AFM tip calibration was the development of single crystal critical dimension reference materials (SCCDRM).²⁻⁵ These specimens, which are fabricated using a lattice-plane-selective etch on (110) silicon, exhibit near vertical sidewalls and high uniformity. As a result of this project, CD-AFM tip width can now be calibrated with 1 nm standard uncertainty.

This dramatic impact of the SCCDRM project on CD-AFM metrology will continue to be expanded by the next generation thrust through more uniform and user-friendly samples, and through collaborative cross-comparisons we are performing with a commercial vendor of similar standards.⁷

2. SCCDRM PROCESS OPTIMIZATION EXPERIMENT

The SCCDRM project has an extensive history and background that have been previously described.^{2-5, 8-11} This history includes several developmental releases prior to the 2004 release to the SEMATECH Member Companies. While there have been some differences in the material and process throughout this evolution, the use of selective etching on (110) Si has always been at the core. And, although we are currently considering other options, the use of a buried oxide as an etch stop and for electrical isolation has also been consistent.

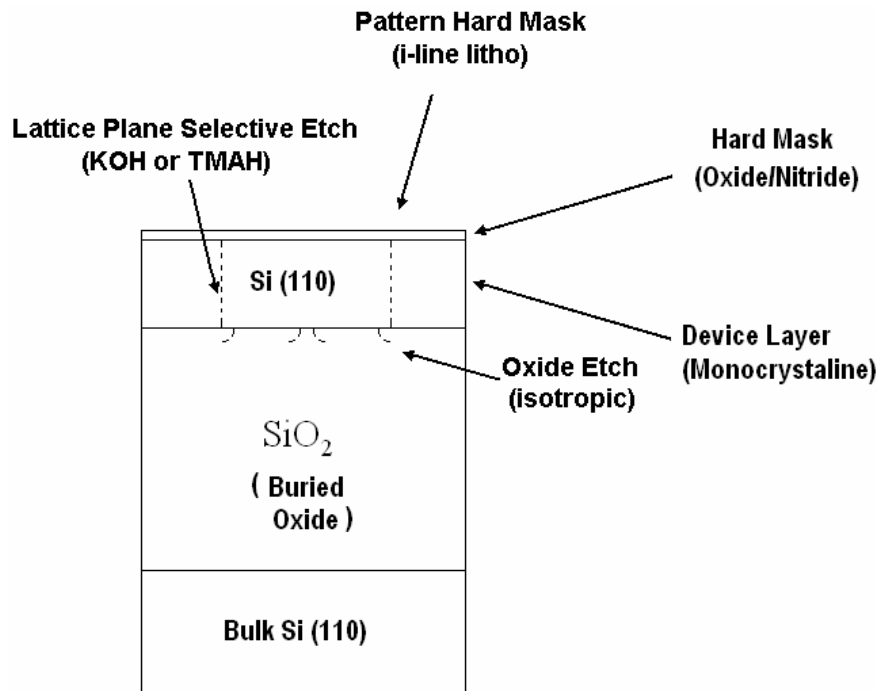


Figure 1. Schematic of SOI starting material and SCCDRM process. For the 2004 release and current experiment, the device layer is approximately 160 nm thick and the buried oxide is about 390 nm. The typical hard mask thickness is about 10 nm. Although earlier experiments used a silicon nitride hard mask, oxide was used for the 2004 release and current work. The etchant for the 2004 release was TMAH, but KOH was used for the present work.

Access to the most advanced optical lithography was another challenge for the project. Consequently, i-line (365 nm) lithography was used for the 2004 release – coupled with significant over-etching to reduce the linewidths to the desired level. As part of our expanding partnership with SEMATECH, we expect to use 193 nm litho for some future work. However, for the current optimization experiment, we used the same starting material and litho as the 2004 release.

The typical hard mask thickness is about 10 nm. In some of our earlier experiments, a silicon nitride hard mask was used because nitride has greater resistance to the selective etch step. However, the post etch removal of a nitride mask is significantly more difficult than the removal of an oxide. In some prior experiments, particles or residue from the hard mask remained on the final features. Therefore, we used an oxide hard mask in both the 2004 release and the current optimization experiment.

In figure 1, we illustrate some of the major characteristics of the current SCCDRM material and process. The substrate and device layer – in which the features are patterned – are both (110) crystalline silicon and are separated by a buried oxide. For the 2004 release, the device layer was approximately 160 nm thick and the buried oxide was about 390 nm.

The silicon-on-insulator (SOI) starting material was most commonly obtained by using an implantation process – such as separation by implantation of oxygen (SIMOX), but some experiments were also performed using bonded-and-etched-back-silicon-on-insulator (BESOI). Due to the expense and difficulty of procuring 200 mm or larger (110) Si wafers, much of the developmental work was performed using 150 mm SIMOX wafers as the starting material.

Following the lithography and pattern transfer to the hard mask, the wafers are diced into individual chips for the remainder of the process. A lattice-plane-selective etch then defines the features in the device layer. During the prior generations of this project, both tetra-methyl-ammonium hydroxide (TMAH) and potassium hydroxide (KOH) were evaluated as etchants for this step.

For the 2004 release of SCCDRMs, TMAH was used as the selective etchant. This was motivated largely by the use of an oxide hard mask which has a greater resistance to TMAH than to KOH. For the current process optimization experiment, however, we realized this was unnecessary due to the low total etch times involved. We therefore migrated to a KOH etch – which has a larger relative lattice-plane selectivity than TMAH.⁹

2.1 Design of Process Optimization Experiment

Although the final feature characteristics of the SCCDRM will have some dependence on both the properties of the starting material and performance of the lithography, we focused on the etch-related factors for our current process optimization experiment. This is relatively practical since these factors are under our direct control and can thus be readily adjusted.

Table I. Details of the SCCDRM process optimization experiment using six two-level factors in a 2⁶⁻² fractional design. This method allows estimation of all main effects and some two-factor interactions. Since the starting material and litho were not as readily adjusted, our current work focused on etch-related factors and cleaning steps.

	Pre-Thin Hard Mask	Etchant Concentration and Etch Time	Ultrasonic Agitation During Etch	Hard Mask Removal	Ultrasonic Agitation During IPA Cleaning	Duration of IPA Cleaning	Ultrasonic Agitation During IPA Cleaning	Duration of IPA Cleaning	Post-SEM Cleaning Procedure	
	1	2	3		4	5	4	5	6	
Experimental Treatments (A-P) and Chips (1-2)	A1/A2	No	25% KOH for 25 s	No	18 s BOE	No	30 s	No	30 s	Acetone
	B1/B2	10 s BOE	25% KOH for 25 s	No	8 s BOE	No	100 s	No	100 s	Acetone
	C1/C2	No	25% KOH for 25 s	No	18 s BOE	Yes	100 s	Yes	100 s	Ozone
	D1/D2	10 s BOE	25% KOH for 25 s	No	8 s BOE	Yes	30 s	Yes	30 s	Ozone
	E1/E2	No	12.5% KOH for 35 s	No	18 s BOE	No	100 s	No	100 s	Ozone
	F1/F2	10 s BOE	12.5% KOH for 35 s	No	8 s BOE	No	30 s	No	30 s	Ozone
	G1/G2	No	12.5% KOH for 35 s	No	18 s BOE	Yes	30 s	Yes	30 s	Acetone
	H1/H2	10 s BOE	12.5% KOH for 35 s	No	8 s BOE	Yes	100 s	Yes	100 s	Acetone
	I1/I2	No	25% KOH for 25 s	Yes	18 s BOE	No	30 s	No	30 s	Ozone
	J1/J2	10 s BOE	25% KOH for 25 s	Yes	8 s BOE	No	100 s	No	100 s	Ozone
	K1/K2	No	25% KOH for 25 s	Yes	18 s BOE	Yes	100 s	Yes	100 s	Acetone
	L1/L2	10 s BOE	25% KOH for 25 s	Yes	8 s BOE	Yes	30 s	Yes	30 s	Acetone
	M1/M2	No	12.5% KOH for 35 s	Yes	18 s BOE	No	100 s	No	100 s	Acetone
	N1/N2	10 s BOE	12.5% KOH for 35 s	Yes	8 s BOE	No	30 s	No	30 s	Acetone
	O1/O2	No	12.5% KOH for 35 s	Yes	18 s BOE	Yes	30 s	Yes	30 s	Ozone
	P1/P2	10 s BOE	12.5% KOH for 35 s	Yes	8 s BOE	Yes	100 s	Yes	100 s	Ozone

Obtaining new starting material or having more lithography performed would be more costly and time consuming, whereas the etching is performed in our laboratory facilities at NIST. More importantly, our overall technical objectives were to reduce feature size and non-uniformity, and we believe that these characteristics have a greater sensitivity to the etch conditions than to the details of the initial lithography.

Barring major defects in crystallinity of the device layer or on the mask pattern, we would expect the selective etch to smooth out most of the width non-uniformity of the features due to imperfections in the mask or the lithography step. But the extent to which this is accomplished will depend on how close the etch conditions are to the idealized model.

In addition to the primary etch conditions, we also incorporated cleaning factors into our experiment. Though not seemingly a central issue, the cleaning process is very important – for the removal of both particulates and hydro-carbon deposition from scanning electron microscope (SEM) inspection. Both types of contamination were of concern during the 2004 release.

Our experiment for optimization of the SCCDRM fabrication process employed a model with six two-level factors in a 2^{6-2} or quarter-replicate design. This method, shown in table I, allows for estimation of all main effects and some two-factor interactions from only 16 runs instead of the 64 that would be required in a full factorial design.¹² The six factors were:

1. Pre-thinning of the oxide hard mask before silicon patterning,
2. Concentration of KOH and etch duration used for patterning,
3. Use of ultrasonic agitation during patterning,
4. Use of ultrasonic agitation during cleaning of chips with iso-propyl alcohol,
5. Duration of cleaning with iso-propyl alcohol, and
6. Use of acetone or acetone and gaseous ozone to remove carbon deposits following SEM inspection.

The process flow generally follows the order of the factors above and from left to right across the columns in table I. However, factors 2 and 3 pertain to the same process step – the lattice-selective silicon etch – and thus occur simultaneously. This is also true of factors 4 and 5 – which describe the cleaning step after patterning.

Note also that factor 1 has a complementary process step – the hard mask strip – which is shown in table I, but which is not an independent factor. This is because the total etch time using the buffered oxide etch (BOE) is held constant at 18 seconds – which was determined to be sufficient to remove all of the oxide. Therefore, the variable described by factor 1 indicates whether the oxide mask is stripped in one step following the lattice-selective silicon etch, or in two steps including a pre-thinning.

Optical inspection, when necessary to verify chip condition, was performed on the chips during the process steps involving the first five factors. At that point, CD-AFM measurements were performed to determine the linewidth and non-uniformity of the target features. A Veeco SXM320[†], which is installed in the Advanced Measurement Laboratory (AML) at NIST, was used for these measurements. Then an SEM inspection was performed and the final cleaning step (factor 6) was completed. The SEM measurements were performed by an external supplier.

The SCCDRM chips have a large number of features on them, and the measurement of all of them by either AFM or SEM would be prohibitive. For this experiment we focused on the variable linewidth grid (VLG) targets. These are also referred to as high resolution transmission electron microscopy (HRTEM) targets, because they were used for the HRTEM calibration step in the 2004 release – and were also the features calibrated for distribution.

For the first phase of AFM measurements, which was performed after the process steps corresponding to the first five factors, we selected two types of target on each chip. One was the narrowest design-width target that had all six features intact, and was, in general, different for every chip. This choice was necessary to assess the factor effects on minimum linewidth.

The second target measured on each chip was one with the largest design-width – which was always the same. This choice would potentially allow us to detect any influence related to defects in the mask, and it also provides independent confirmation of the factor effects on non-uniformity determined from the measurements of the smallest design-width targets.

Following the first phase of AFM measurements, the SEM measurements were performed only on the surviving-smallest-design width targets. An AFM re-check of selected targets was then performed to assess the level of hydrocarbon contamination deposited by the SEM. A typical level of contamination is a few nanometers thick.

The last cleaning step, described by factor 6, was used to remove any contamination deposited during the SEM inspection. Although alternative cleaning processes may be explored in the future, we decided to compare an acetone

clean with an acetone and ozone clean for this factor. Following the final clean, those features that had been measured by SEM were measured again using the AFM.

2.2 Analysis of Results

We analyzed the CD-AFM data to determine the sensitivity of two feature characteristics to the experimental factors. These characteristics were the minimum linewidth and linewidth non-uniformity. To quantify the non-uniformity, we used a metric that was developed as part of our uncertainty analysis for the 2004 release.

Essentially, this metric involves the use of a triangular weighting function over the central 0.5 μm of the AFM data set – or 20 line scans – to calculate an average and standard uncertainty of the linewidth in that region. This choice was motivated by our treatment of TEM/AFM relative positioning uncertainty for the 2004 release, and the details have been previously published.^{4,5}

In figures 2 and 3 the factor effects plots¹² for minimum linewidth and non-uniformity are shown. For clarity, only the effects of the first five factors are included in these plots. These results are calculated from the AFM measurements taken on the smallest surviving target on each chip. However, the results taken from the same target on each chip (i.e., largest design width) lead to a substantively similar conclusion. This means that – as expected – there is no significant dependence on the specific target selected.

Effects Plots for Minimum CD

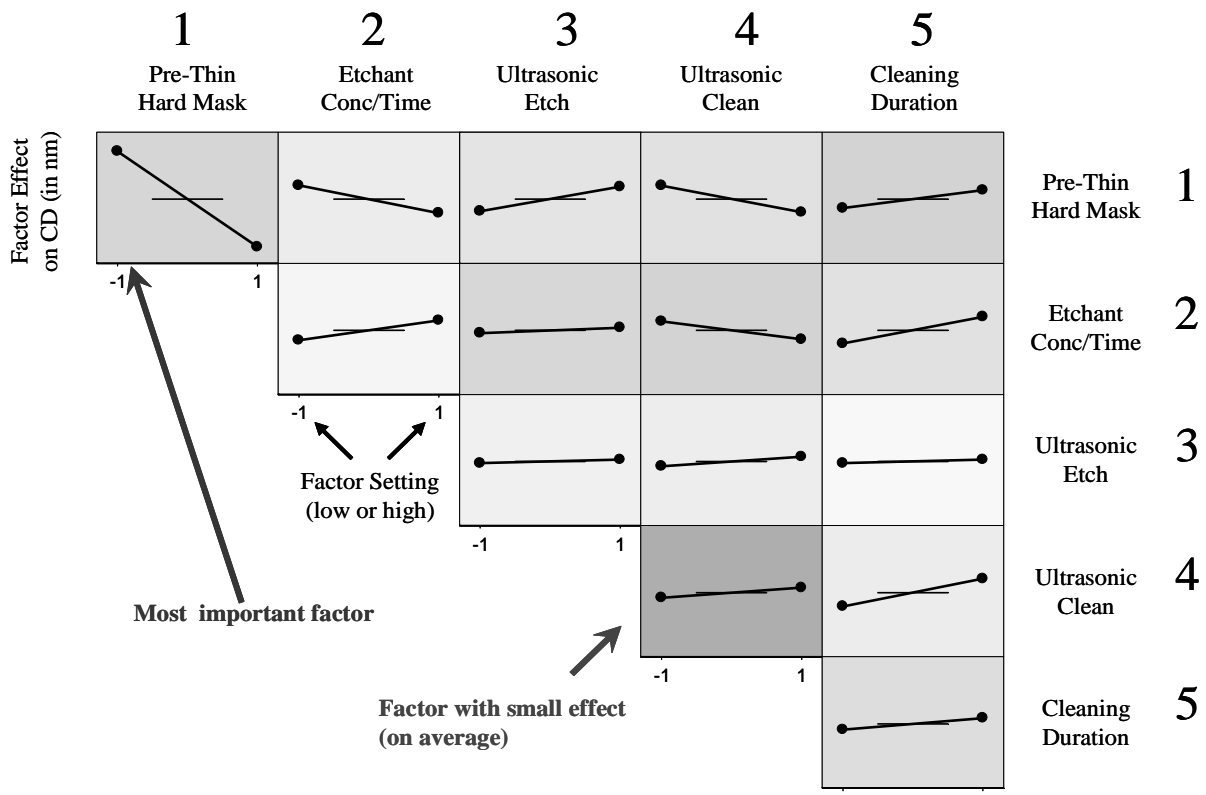


Figure 2. Factor effects plot for minimum linewidth. The most important single factor effect is pre-thinning of the oxide hard mask prior to the silicon etch. (Notes: (1) Common background color indicates confounded effects; (2) factor six is not included.)

Effects Plots for CD Non-Uniformity

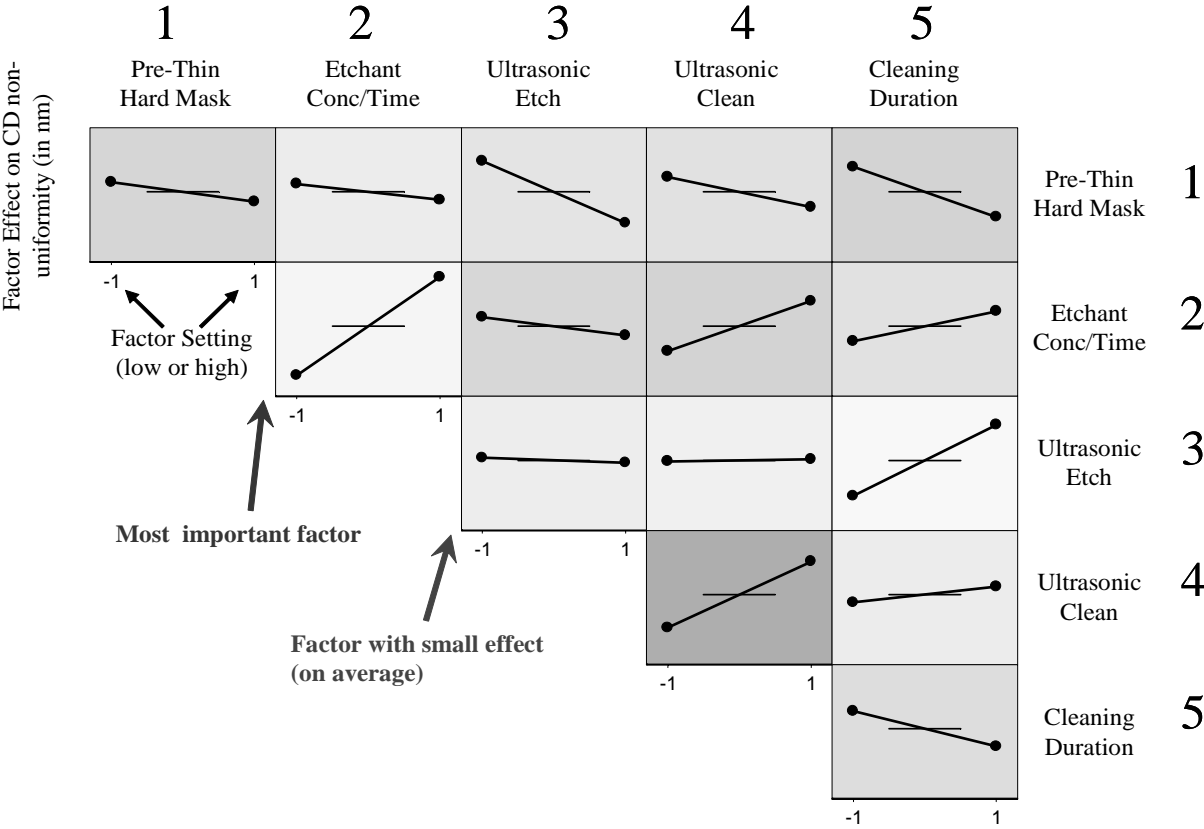


Figure 3. Factor effects plot for linewidth non-uniformity. The most important single factor effect is the etchant concentration and time. (Notes: (1) Common background color indicates confounded effects; (2) factor six is not included.)

As the factor effects plots reveal, the minimum linewidth that survived etching is sensitive primarily to pre-thinning the hard mask and has little dependence on the remaining factors.¹² This is physically reasonable since the narrowest features will be quickly undercut during oxide etch which strips the hard mask. Consequently, removing as much hard mask as possible up front can be expected to increase the survival of very narrow lines.

The non-uniformity has sensitivity to several factors and two-factor interactions, but very little sensitivity to pre-thinning of the hard mask. However, the single largest sensitivity is to the etchant concentration and etch time factor. This is also physically reasonable since we believe that local variations in the etch environment (e.g. concentration gradients) are a significant contributor to the observed non-uniformity of the final features.

A lower etchant concentration applied for a longer period of time is more likely to result in average etch conditions that are more uniform and may thus be expected to produce structures with lower non-uniformity. This conclusion is consistent with our experimental observations.

In figures 4 and 5, plots taken from the AFM measurement of the least and most uniform process configurations are shown. These figures illustrate the observed range of non-uniformity and the results of the best process conditions. As can be seen in figure 5, we were able to achieve linewidths as low as 20 nm. The standard uncertainty due to non-uniformity in the central 0.5 μm section of that feature, as defined by the metric discussed above, is ± 0.45 nm. With

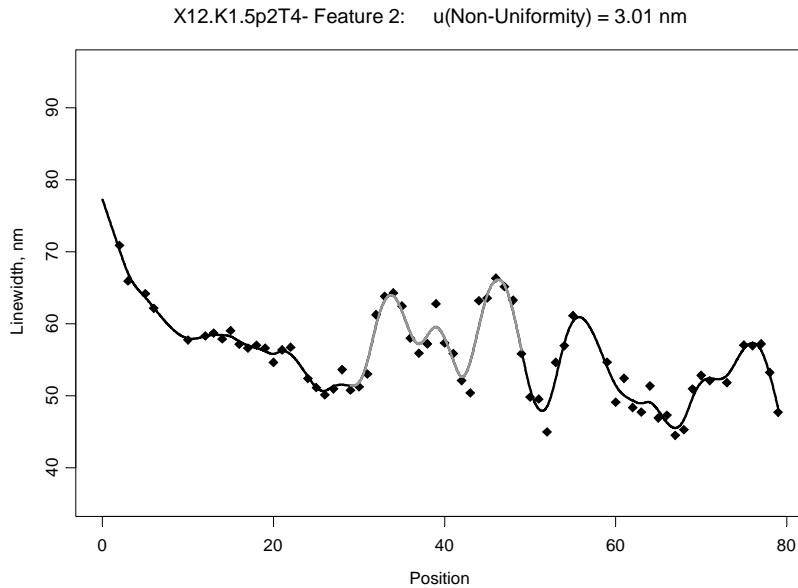


Figure 4. Example of CD-AFM data on feature from worst process condition. The plot shows the linewidth calculated from individual linescans taken along a $2 \mu\text{m}$ length of the feature. The orange curve indicates the portion of data used to calculate the non-uniformity metric.

The non-uniformity exhibits a statistically significant dependence on two factors and one two-factor interaction. As discussed above, the importance of the factor for etchant concentration and etch time is not surprising, and the effect of ultra-sonic cleaning is also not unreasonable – especially if one speculates that some of the apparent non-uniformity is due to residue on the features. However, the significant dependence of the non-uniformity on the two-factor interaction between ultra-sonic etch and cleaning duration is not fully understood. But we can clearly conclude from these results that further investigation of all significant effects identified in our experiment is warranted.

In our ongoing extension and refinement experiments, we are exploring other levels of the etch factor and alternative cleaning processes which were considered too aggressive for the current experiment. The so-called ‘piranha’ clean,

repeated AFM measurements to reduce statistical uncertainty, this is low enough to support our goal of developing standards that have an expanded uncertainty of about $\pm 1 \text{ nm}$ ($k = 2$).

In figures 6 and 7 the normal probability plots¹² are shown for both minimum linewidth and non-uniformity. These figures clearly demonstrate that statistically significant conclusions can be drawn from the factor effects plots.

Although the minimum CD has some statistically significant dependence on two two-factor interactions, both of these involve

pre-thinning of the hard mask – which is the only factor that has a significant main effect – a dependence we believe is well understood.

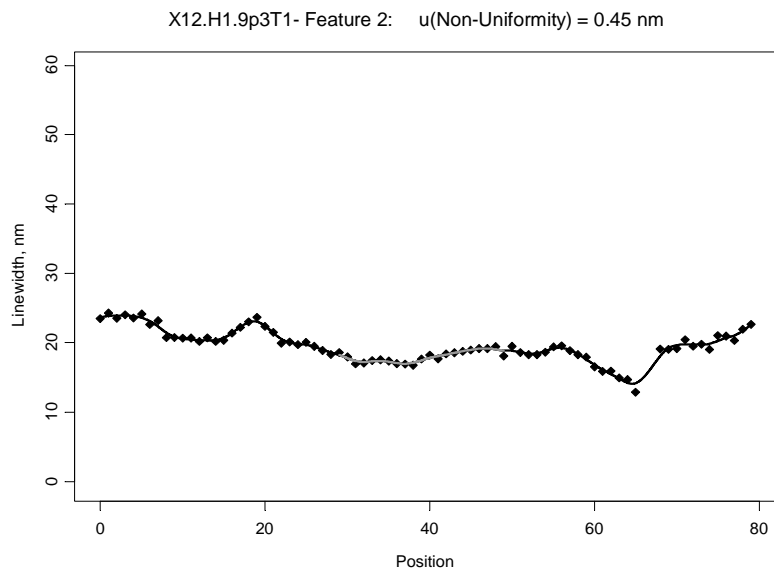


Figure 5. Example of CD-AFM data on feature from best process condition. The plot shows the linewidth calculated from individual linescans taken along a $2 \mu\text{m}$ length of the feature. The orange curve indicates the portion of data used to calculate the non-uniformity metric.

CD

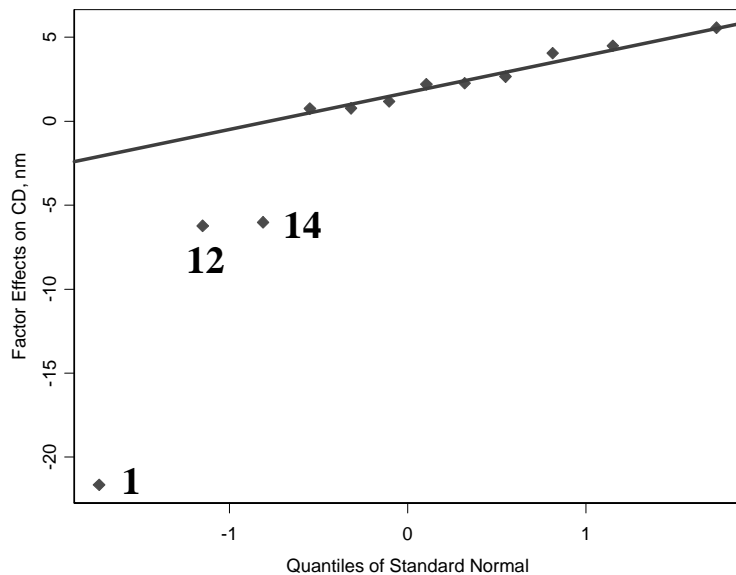


Figure 6. Normal probability plot for factor effects on minimum linewidth showing the dramatic dependence on pre-thinning of the hard mask (factor 1) and statistical significance of the observation.

uncertainties of about ± 1.5 nm ($k = 2$) in the 2004 release – is the bulwark of this response.

Following the 2004 release, NIST began work on the “next generation” of SCCDRM standards, and we have now completed a designed experiment to improve our fabrication process and reduce the non-uniformity of the features. We found that the minimum linewidth and linewidth uniformity were primarily sensitive to different factors - and can thus be independently tuned to meet our future goals.

Presently, we are working on refining experiments to further optimize the important factors that we have identified. Our strategic goal is develop a NIST Standard Reference Material (SRM) based on our SCCDRM technology. We are considering both a chip-based (as in the 2004 release) and a monolithic 200 mm wafer implementation for this standard. The measurement focus is expected to be on isolated lines that are most

which uses a mixture of sulphuric acid (H_2SO_4) and hydrogen peroxide (H_2O_2), is one candidate. This cleaning process is commonly used in the micro-electro-mechanical systems (MEMS) industry to remove resist and other organic residue.¹³

Although these experiments were all performed processing individual chips, we expect most of our conclusions to carry over to the processing of intact 200 mm wafers. Currently, we are exploring both SOI and bulk (110) 200 mm implementations, and will report the results of preliminary experiments elsewhere.¹⁴

3. SUMMARY AND PLANS

The NIST response to the pressing need for greater accuracy in CD-AFM metrology has been a robust and multifaceted program of standards development and industrial collaboration. The SCCDRM project – which produced artifacts for tip width calibration with typical expanded

Non-Uniformity

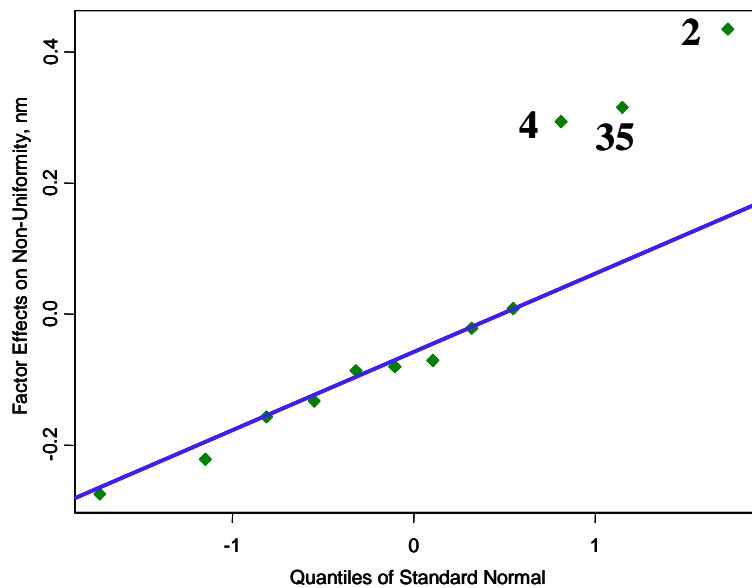


Figure 7. Normal probability plot for factor effects on linewidth non-uniformity showing the statistical significance of three effects with the largest being the factor for etch time and etchant concentration (factor 2).

suitable for CD-AFM calibration, but we are also exploring application of this technology to grating standards for scatterometry.

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