Photoresist cross-sectioning with negligible damage using a dual-beam FIB-SEM: A high throughput method for profile imaging

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Imaging of photoresist cross sections in a focused ion beam (FIB)–scanning electron microscope (SEM) is demonstrated with negligible damage. An *in-situ* chromium sputtering technique is used to deposit metal on the site of interest, replacing the conventional and more damaging metal deposition by high energy ion decomposition of metal-organic precursors. Here, a high current ion beam is rastered over a small chromium target suspended over the wafer surface resulting in a less damaging metal deposition step. The subsequent resist critical dimensions measured via FIB-SEM are calibrated against profile measurements taken by critical dimension atomic force microscopy, implemented here as a reference measurement system (RMS) without the influence of beam exposure. The use of a nondamaging RMS allows an accurate measurement of resist damage during imaging. As a practical demonstration of this sputtering method, a 50 nm 1:1 line/space array in extreme ultraviolet photoresist is analyzed through focus and exposure.

I. INTRODUCTION

In-Fab focused ion beam–scanning electron microscopy (FIB-SEM) platforms can greatly improve throughput time for taking cross sectional images and are currently used for a variety of process development applications[1,2] as well as in-line process control.[3] A full wafer automated FIB-SEM recipe begins with feature alignment by pattern recognition. At each site, metal (usually W or Pt) is deposited to prevent sample charging and to facilitate an even milling step. Deposition is done by ion-assisted decomposition of an organometallic precursor [such as $W(CO)_6$]. This is followed by a milling step where the ion beam sputters a wedge into the wafer, exposing a cross section at the site of interest. The cross-section face can be decorated with XeF_2 to provide added material contrast. Finally, the site is imaged by an electron beam positioned at a fixed angle above the wafer horizon. The above sequence can take 10 – 20 min to complete per wafer site compared to roughly 1 h for a conventional laboratory SEM on a wafer piece.

Frequently, a 30 keV ion beam is used to protect and mill the site to be cross sectioned. However, this limits the applicability of the FIB-SEM technique to robust materials that do not degrade under the influence of an electron or ion beam. Materials such as photoresists are notoriously susceptible to beam damage. Figure 1 shows a comparison of 50 nm wide photoresist lines (193 nm lithography) acquired from a conventional laboratory-based cross section of a cleaved wafer fragment and an automated FIB-SEM cross section taken on a full wafer using a 30 keV ion beam to coat the site with tungsten. The FIB-SEM image exhibits severe damage from the metal deposition step. The resulting resist lines are a fraction of the height of the lines measured in a conventional SEM. Using lower ion beam energies of 5 and 3 keV at the expense of deposition rate can reduce but not eliminate damage to resist lines.

PREPRINT: See published version at
JS Clarke, MB Schmidt, NG Orji "Photoresist cross-sectioning with negligible damage using a dual-beam FIB-SEM: A high throughput method for profile imaging"
***J. Vac. Sci. & Technol. B* 25, 2526 (2007); https://doi.org/10.1116/1.2804516**

In this article, we demonstrate a negligible-damage method of measuring cross sections using a FIB-SEM with- out sacrificing automation and throughput. Critical dimension atomic force microscopy (CD-AFM) is used to calibrate the resist profiles in the absence of electron or ion beam exposure. The metal deposition step is then accomplished by *in situ* sputtering of chromium from a target placed just above the wafer surface using the 30 keV ion beam. FIB- SEM results are compared to the CD-AFM reference and practical application of extreme ultraviolet (EUV) resist screening is demonstrated. The time and resources required to obtain the information from the cross sections can be widely reduced allowing for a greater number of more comprehensive information turns during lithography development.

II. EXPERIMENT

The goal of this work is to ultimately compare a FIB- SEM cross section to a conventional cross section and validate that no measurable difference is observed in the resist profile between the two techniques. This first requires a reference metrology capable of measuring the small dimensions of advanced lithography, but which preferably does not ex- pose the sample to electrons or ions. This is underscored by the resist damage shown in Fig. 1. Second, the photoresist sample will still need a nondamaging metal protection layer prior to imaging in order to prevent charging and allow an even FIB mill. This will require an *in-situ* metal deposition method that does not impede analysis time or influence the image quality of a FIB-SEM cross section.

To characterize the profile shape, we used the CD-AFM,[4] which is an ideal reference instrument for our purposes be- cause of its nondestructive nature and the sidewall profiling ability. The specific CD-AFM used in this study is implemented as a reference measurement system (RMS), where it is calibrated with samples that are closely traceable to the SI definition of length and used to monitor the performance of faster and less accurate tools. Details of the calibration and the uncertainty budgets of the CD-AFM are well documented.[5] Previously, the instrument has been used for measurement procedure development, standards development,[6] and instrument accuracy evaluations. Hence, in addition to independently verifying profile information, the RMS implementation ensures that the width and height values are traceable at the nanometer level.

Here, the measurements were made on a series of line features with a nominal width of 50 nm using the Veeco X3D 340 CD-AFM. The scan size for the images ranged from 3 to 6 μm along the fast scan axis, and 2 μm in the slow scan axis. The scan rate for all of the images was 0.477 Hz. Each final image is the summation of 30-line scans, which has been chosen to optimize signal to noise and minimize scanning time. The resist linewidth measurements for the CD-AFM were taken at 20% from the top and bottom of the lines in order to avoid the small footing or top rounding of these resist features.

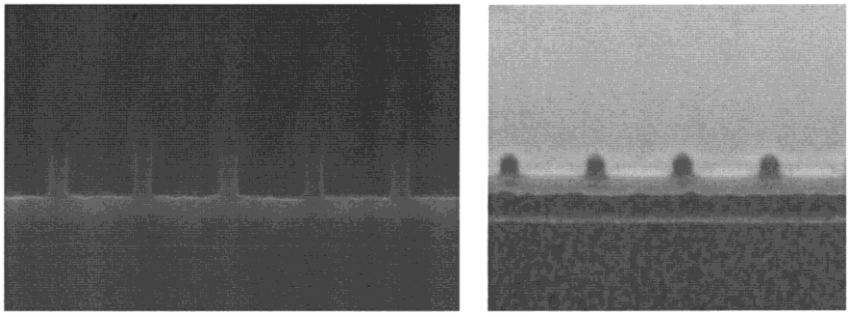
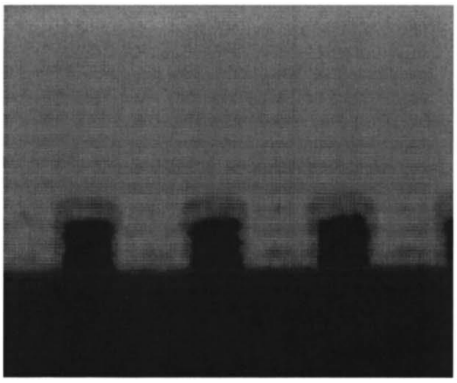


FIG. 1. Two images of the same 193 nm photoresist are shown. The resist is patterned to have a CD of 50 nm, a height of 150 nm, and a pitch of 250 nm. A conventional SEM image from a cleaved wafer fragment is shown on the left. A standard FIB- SEM image is shown on the right, where the resist lines show catastrophic damage (top loss).

III. FIB-SEM: A PATH TO REDUCED DAMAGE

It is common practice to first sputter coat a thin layer of conductive metal (such as Au) onto a wafer fragment when performing a manual cross section on a laboratory-based SEM. This is especially important for charge reduction when looking at samples such as oxides or photoresists. We there- fore hypothesize that sputter deposition of a conductive material from a target onto a patterned photoresist would reduce or eliminate damage during the ion deposition protection step of the FIB-SEM process.

To validate this technique, a laboratory-based argon plasma sputter tool was used to deposit nickel from a target onto a surface of patterned 193 nm resist. Nickel was chosen over gold for this experiment in order to align with Fab contamination requirements. The deposited nickel was non- conformal with a depth of 50 nm on the top of the resist lines and $<$20 nm on the resist sidewall. No attempt was made to improve metal conformality. The sample was then coated in an FEI Strata DB-STEM-237 small stage FIB-SEM tool with 30 keV ion-deposited W to a depth of 1 μm and milled and imaged using the process outlined previously. An image of the nickel coated sample is shown in Fig. 2 along with a table comparing resist height and width measured from CD- AFM (before metal deposition) and FIB-SEM. The two measurement techniques have matching dimensions at the 85% confidence interval. This indicates that sputter deposition as a sample protection scheme in FIB-SEM tools can allow for resist cross sections and imaging.



	Height (nm)		Width (nm)	
	Avg	Std Dev	Avg	Std Dev
CD-AFM	153.9	0.3	107.5	0.4
Sputtered Ni	151.5	2.5	105.9	2.6

FIG. 2. Cross section of a 193 nm pho- toresist is shown on the left. A nonconformal layer of nickel is sputtered to a maximum depth of 50 nm on top of the resist lines. The sample was then imaged in a FIB-SEM using the conventional metal deposition method prior to milling. The CDs measured from the resulting image are compared with CD-AFM measurements from the sample prior to Ni coating. Results conform within the 85% confidence interval.

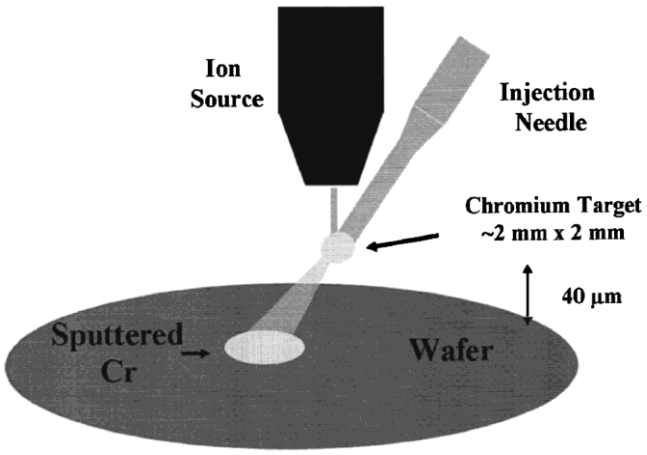


FIG. 3. Diagram of the *in-situ* Cr sputter method is shown. A Cr target is positioned on a retractable needle, which can be positioned in front of the FIB-SEM ion source. The wafer is then positioned very close to the sputter target. This narrow mechanical distance helps us to localize the spread of deposited material. The ion beam sputters chromium from the target and onto the wafer.

From an integrated perspective, it is not practical to add a sputter deposition chamber onto a full wafer FIB-SEM tool. Likewise, a localized plasma sputter capability in the FIB- SEM vacuum chamber could lead to unwanted metal deposition on the electron optics, thereby affecting image quality. The approach taken here is shown in Fig. 3, with all modifications being made to a FEI Defect Analyzer 300HP platform. A 2 – 4 mm^2 chromium target is placed onto a retract- able metallic (nonmagnetic) gas needle valve within the vacuum chamber. This needle is similar to the needles that inject the organometallic precursor used for conventional metal deposition or XeF_2 used for sample decoration. Chromium is chosen here because of its fabrication compatibility relative to Au, due to its small grain size after deposition, and because of its relatively low cost. The Cr target is positioned within tens of microns to the wafer surface. Spacing was optimized through experiment to allow safe clearance of the needles, to maximize metal deposition rate, and to minimize the spread of sputtered material onto the wafer. A high current Ga^{++} ion beam ($>$20 nA) is used to sputter the Cr from the target to the wafer. The deposition rate is $2-5$ nm/min of sputtering time, which is several orders of magnitude slower than using direct ion beam deposition. The resulting sputtered region is roughly 600 μm in diameter under the experimental conditions used here. As the ion beam impinges the Cr at a shallow angle, there is appreciable reflection of high energy ions onto the wafer surface directly below the target.
This results in a $=$40 μm diameter region of damage at one edge of the sputtered area that is similar in nature to the resist erosion shown in Fig. 1. Subsequent milling and imaging of the wafer site takes place at a location in the middle of the sputtered area, but well away ($>$200 μm) from the observed damage area.

The full processing sequence is accomplished in several steps. First, the site is coated with sputtered Cr to a depth of 5 – 25 nm. Special consideration is made to optimize the sputtering direction parallel to line/space pattern in order to promote even coverage. This coating step can take up to 5 min and is sufficient to fully encapsulate a resist line/space array. With the resist capped by the Cr, tungsten is then ion deposited to a depth of $>$400 nm. The purpose of the second metal layer deposition is to allow a more even mill. The deposited W should not penetrate the sputtered Cr even at depths of 5 – 10 nm. Once the deposition is complete, a 50 pA ion beam is used to mill the cut face into the wafer that is large relative to the feature of interest. A 2 keV, 10 pA electron beam is used to image the resist profile at an angle of 38° above the wafer horizon. The e-beam focus and stigmation are then automatically adjusted on the cut face in a location away from the feature of interest. Finally, a fast $=$0.5 s) nonintegrated scan of the resist line is taken over a 1–2 μm field of view. This minimizes e-beam induced resist shrinkage to the same level as measured in a competitive CD-SEM.[7] We conjecture here that the $=$0.5 μm thick layer of protective metal provides structural integrity to the cross-section face, further minimizing resist shrinkage. The entire process (sputter to image) can be accomplished in as little as 10 min, and as stated above, is fully automated, thus removing operator dependence and time expenditure.

IV. RESULTS AND DISCUSSION

Images taken from a CD-AFM and the FIB-SEM are shown in Fig. 4 from the same site on a 193 nm resist wafer. For the purposes of illustrating the Cr sputter process recipe, a 20-minute-long sputter deposition step is used. The Cr is observed as a conformal layer up to 50 nm thick on top of the resist surface. Practically, sputter deposition times as short as 2 min have been successfully used for photoresist protection.

In Fig. 5, conventional FIB-SEM processing recipes and the sputter deposited Cr recipe described above are statistically compared to the CD-AFM data. Three different ion beam accelerating voltages are used for ion deposition in the conventional recipes. The 30 keV recipes show severe resist height loss when compared to the CD-AFM results. Other gentle ion depositions using a specially tuned 5 or 3 keV ion beam exhibits less resist damage, but CD change is still significant. By comparison, the site processed using sputter deposited Cr during the FIB-SEM protection step is in statistical agreement (85% confidence interval) with the CD-AFM for mean and standard deviation of resist CD and resist height. A fast, automated cross section of photoresist has thus been demonstrated with negligible damage. As a practical demonstration of this approach, an EUV photoresist was patterned with a 50 nm 1:1 line/space array in a focus-exposure matrix using the Cr sputter approach. This pattern represents the leading edge of current litho- graphic capabilities and poses a strenuous test of the Cr sputter approach. A total of 20 sites were imaged and the processing time was optimized to 10 min per site. As this process was fully automated, no man-hours were consumed to collect this data beyond starting the recipe. A through- focus slice of this matrix is shown in Fig. 6. Bossung behavior is observed in this series of images, which can be used to characterize the process window of this photoresist. Several resist wafers can be loaded and imaged in one full sequence. In addition, these resist cross sections can be used to optimize the measurement settings of a top-down CD-SEM in order to improve measurement accuracy and can be used to validate the optical aetherometry models required for CD process control.

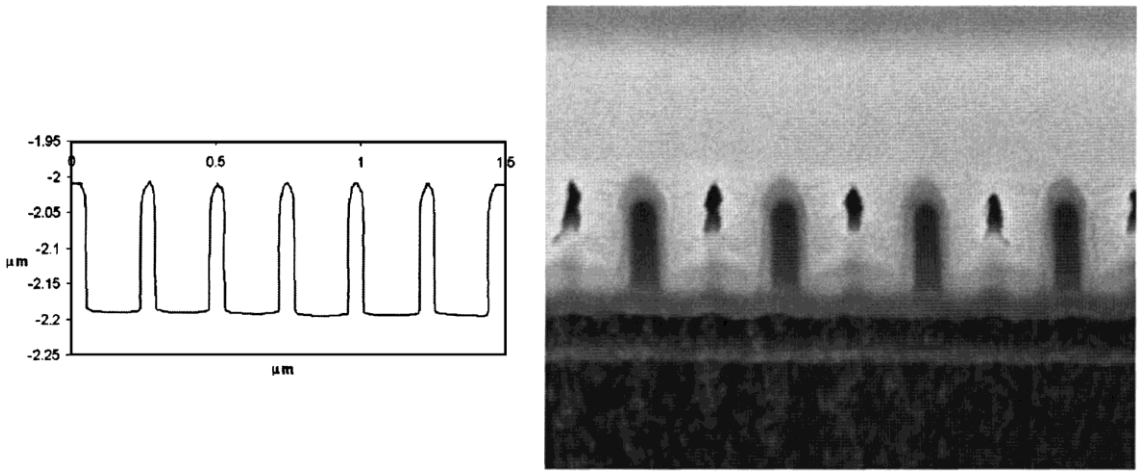


FIG. 4. Image on the left is the reconstruction of a CD-AFM scan of a 193 nm photoresist line space array. The corresponding FIB-SEM image is shown on the right. Prior to imaging, the sample was coated *in situ* with sputtered chromium. The Cr is observed as a light gray conformal layer. Tungsten is then deposited by ion-assisted deposition on top of the protective Cr coating. The voids in between the resist lines are due to poor gap fill of the ion-deposited W and are inconsequential to the resist measurement.

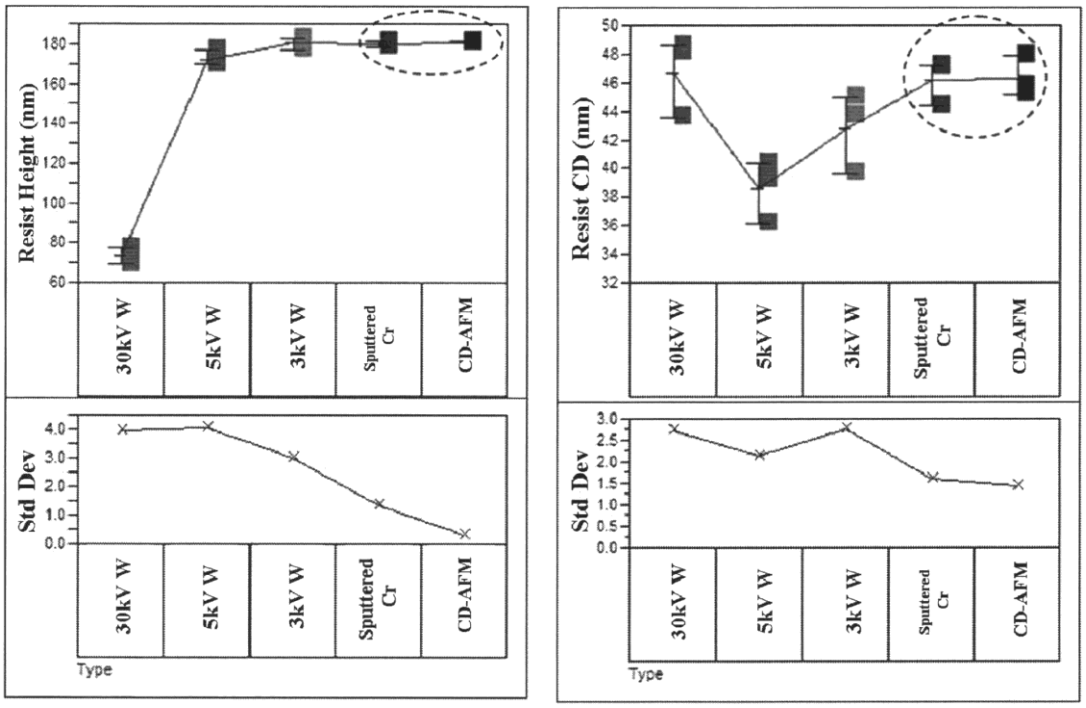


FIG. 5. Dimensions of different sample coating methods are compared to CD-AFM. Each method is the weighted average of three different resist lines. Note that the error bars shown correspond to the statistical standard uncertainties for each weighted mean. The conventional FIB-SEM method of using a 30 keV ion beam shows severe damage to resist height. Gentler variations of the ion-deposited W recipe exhibit less damage, but results are statistically different from CD-AFM. Only the sample first coated with sputtered Cr shows resist height and CDs that are in statistical agreement to the CD-AFM (85% confidence interval).

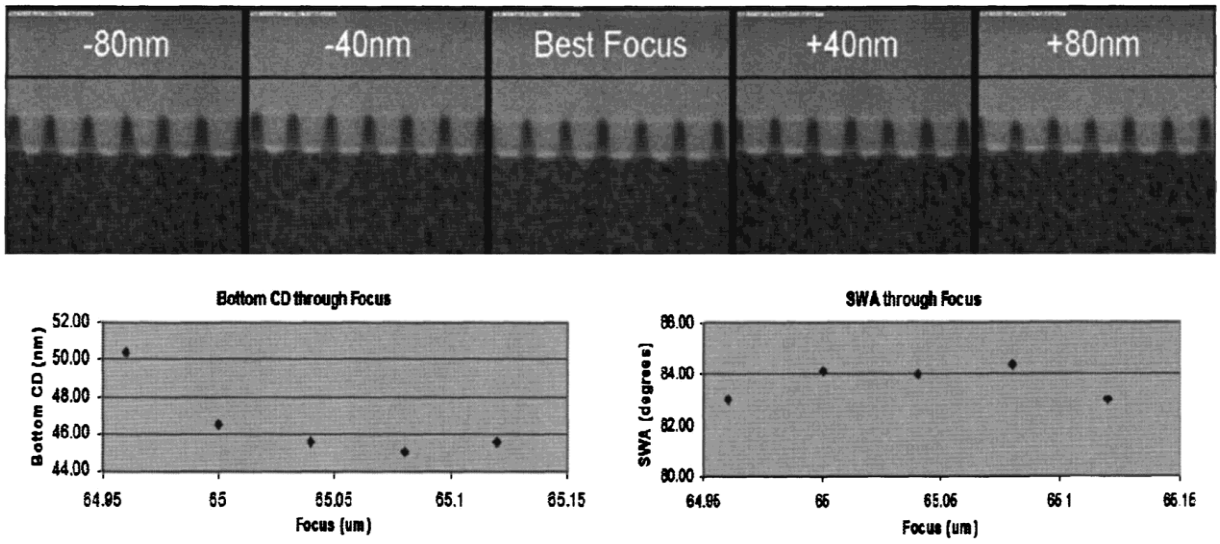


FIG. 6. Processing cross sections of a 100 nm thick EUV photoresist are shown through focus (top). These images took approximately 10 min of processing time per site and represent a subset of a full focus-exposure matrix. Images were measured using signal processing software. Resist bottom CD (bottom left) and sidewall angle (bottom right) are plotted as a function of focus and exhibit good Bossing behavior.

V. CONCLUSIONS

In-Fab FIB-SEM tools can greatly improve the time to information in research and development; however, conventional processing of photoresists results in an unacceptable level of damage to profile shape and dimension. Here, *in situ* chromium sputter capability was demonstrated in a FIB- SEM system in order to gently coat the wafer surface prior to FIB milling. A chromium target, optimized for geometry with respect to the wafer, minimizes resist damage while maintaining an even metal coating. Profiles obtained from CD-AFM act as a calibration to these cross-section images, allowing verification of the damage during site preparation and imaging. Furthermore, the use of CD-AFM as a reference allows bias-free optimization of imaging conditions for electron and ion beam metrologies. Finally, damage for a 193 nm photoresist pattern was determined to be negligible using this technique, and extensions have been made to 100 nm pitch EUV patterns. Processing time was roughly 10 min per site, and improved time to information in lithography development is achieved.

ACKNOWLEDGMENTS

One of the authors (N.G.O.) and the NIST contribution to this work were supported by the NIST Office of Microelectronic Programs (OMP). The authors acknowledge Pat Paludal of Intel Corporation, Stacey Stone of FEI Company, Ben Bunday of SEMATECH, and Ronald Dixson of NIST for helpful discussions and review of this manuscript. Certain commercial equipment are identified in this article 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 are necessarily the best available for the purpose.

REFERENCES

1. A. Volinsky, L. Rice, W. Qin, and N. D. Theodore, FIB failure analysis of memory arrays, *Microelectron. Eng.***75**, 3 (2004). https://doi.org/10.1016/j.mee.2004.03.088

2. V. G. M. Sivel, et al. Application of the dual-beam FIB/SEM to metals research, *J. Microsc.* 214, 237 (2004). https://doi.org/10.1111/j.0022-2720.2004.01329.x

3. G. Franco, Z. Dsouza, D. Mello, and M. Weschler, Proceedings of the IEEE International Symposium on Semiconductor Manufacturing pp. 394–397. (2005)

4. Y. Martin and H. K. Wickramasinghe, Method for imaging sidewalls by atomic force microscopy, *Appl. Phys. Lett*. **64**, 2498 (1994). https://doi.org/10.1063/1.111578

5. N. G. Orji, et al. Progress on implementation of a CD-AFM-based reference measurement system, *Proc. SPIE* 6152, 61520O, (2006). https://doi.org/10.1117/12.653287

6. R. G. Dixson, et al. Traceable calibration of critical-dimension atomic force microscope linewidth measurements with nanometer uncertainty *J. Vac. Sci. Technol. B* **23**, 3028 (2005). https://doi.org/10.1116/1.2130347

7. B. Bunday, et al. ISMI Tech Transfer Document ID No. 04114595C-ENG, 2006; Nonconfidential, available on SEMATECH website, http://www.sematech.org