

# Small-angle Neutron Scattering Measurements for the Characterization of Lithographically Prepared Structures

Wen-li Wu<sup>1</sup>, Eric K. Lin<sup>1</sup>, Qinghuang Lin<sup>2</sup>, and Marie Angelopolous<sup>2</sup>

<sup>1</sup>*Polymers Division, National Institute of Standards and Technology,  
Gaithersburg, MD 20899-8541.*

<sup>2</sup>*IBM Thomas J. Watson Research Center, P. O. Box 218,  
Yorktown Heights, NY 10598.*

**Abstract.** The continuing decrease in feature sizes in the semiconductor and other nanofabrication industries has placed increasingly stringent demands on current microscopy-based techniques to precisely measure both the critical dimensions and the quality (i.e. line-edge roughness) of these structures. Small-angle neutron scattering (SANS) experiments provide a quick, non-destructive, and quantitative measurement of the three-dimensional shape and quality of lithographically prepared structures as fabricated on a silicon substrate. We demonstrate the application of SANS for the characterization of nanoscale structures using periodic 150 nm parallel lines prepared using standard 248 nm photolithographic processes.

## INTRODUCTION

The continued growth of the semiconductor industry has in part been fueled by advancements in photolithographic processes and materials to enable the economical production of smaller device features. As feature sizes continue to decrease and approach 100 nm, the allowable resolution limits of the lithographic process become more stringent and difficult to measure. The precise measurement of the size and quality of lithographically prepared features as they decrease in size is a challenging metrology issue and is critical for evaluating and improving new lithographic processes and materials. Currently, microscopy-based techniques such as scanning electron microscopy (SEM) and atomic force microscopy (AFM) are the primary tools used to characterize nanoscale structures [1-4]. These experimental methods often require specialized modifications to enable the measurement of either the critical dimension or feature resolution parameters such as the line-edge roughness (LER). In general, both SEM and AFM characterization methods are time-consuming, may require special sample preparation, and are only able to sample a small area of a given wafer. More importantly, these techniques

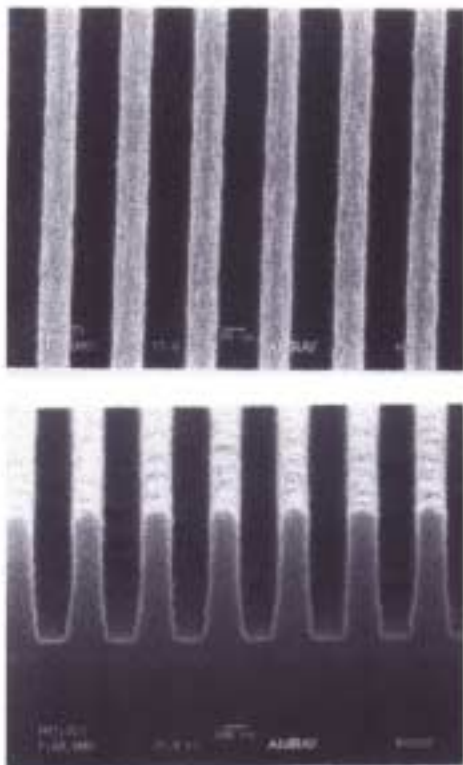
become even more challenging as feature sizes continue to decrease.

In this work, we demonstrate the powerful use of small-angle neutron scattering (SANS) to quickly, non-destructively, and quantitatively characterize both the size and profile (including LER) of lithographically prepared nanoscale structures as prepared on a silicon wafer substrate. Until recently, SANS instruments were unable to measure lithographic feature sizes (greater than 300 nm) and neutron beam fluxes were too weak to measure scattering from thin film structures. Today, higher intensity neutron sources, new focusing optics, and smaller lithographic features allow for the potential of routine SANS measurements. Other important advantages for the use of SANS to measure lithographic structures include a) measurement of structures on silicon because single crystal silicon wafers are generally transparent to neutrons b) a measurement metric statistically averaged over an area of several square centimeters and c) less stringent instrument requirements for the SANS line as lithographic structures decrease in size. As an example, periodic, equally spaced parallel line patterns with a nominal size of 150 nm were prepared on a

silicon single crystal wafer using standard 248 nm optical lithography and placed directly in the neutron beam. Quantitative measurements of the size and average profile of these lines are extracted from the scattering data.

## EXPERIMENT

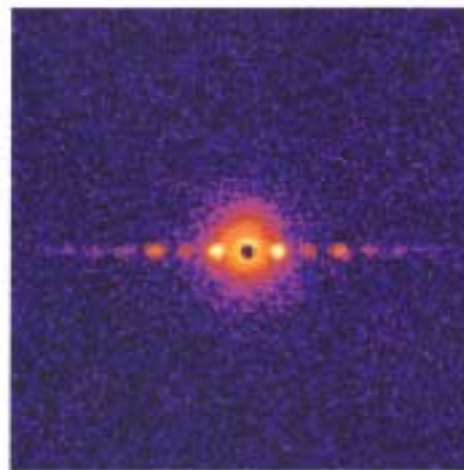
The samples used in this study consist of periodic and equally spaced parallel line structures prepared using conventional 248 nm optical lithography with chemically amplified photoresist chemistry [5]. The structures were nominally 150 nm in size and were patterned and developed from a 620 nm thick photoresist film. The patterned area was 8 mm by 8 mm in size. The patterned silicon wafer is placed directly into the neutron beam. SEM micrographs of these structures are shown in Fig. 1.



**FIGURE 1.** Top-down and side view scanning electron micrographs of the lithographically prepared lines used in the SANS measurement. The lines are nominally 150 nm wide and 0.62  $\mu\text{m}$  in height.

The SANS measurements were performed on the NG7 30 m SANS line under ambient atmospheric conditions at the National Institute of Standards and Technology Center for Neutron Research. The

neutron beam wavelength,  $\lambda$ , was 8.44  $\text{\AA}$  with a wavelength spread,  $\Delta\lambda/\lambda$ , of 11 %. The sample to detector distance was 15.3 m. Newly developed neutron focusing optics consisting of 28 biconcave  $\text{MgF}_2$  lenses were used to access small enough angles to resolve feature sizes up to 300 nm, a previously inaccessible length scale for SANS [6,7]. The accessible  $q$  range (where  $q = (4\pi/\lambda)\sin(\theta/2)$  and  $\theta$  is the scattering angle) for this configuration was 0.0011  $\text{\AA}$  to 0.015  $\text{\AA}$ . Two-dimensional scattering data were collected for up to 6 hr for good count statistics, but usable SANS data were obtained after 10 min. Scattering from the unexposed photoresist film and background radiation were subtracted from the scattering data of the parallel line structures using standard reduction methods. The sample was placed with the substrate perpendicular to the incident neutron beam. In this configuration, the SANS data provide quantitative information about the line repeat distance and the quality of the line structure.



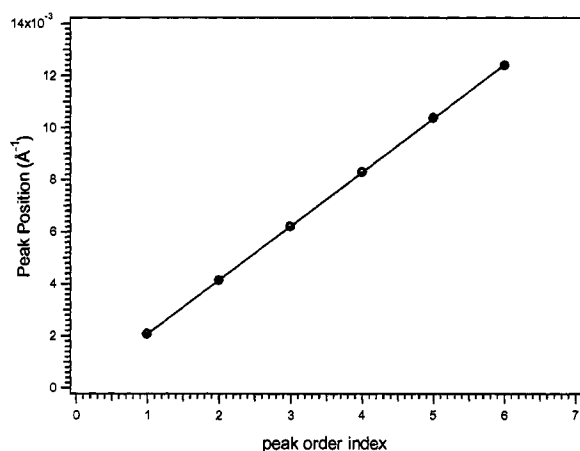
**FIGURE 2.** Two-dimensional SANS pattern from the sample shown in Fig. 1. Six orders of diffraction are observed due to the high resolution of the lithographically prepared pattern.

## RESULTS AND DISCUSSION

The two-dimensional scattering data from the line structures shown in Fig. 1 are shown in Fig. 2. The line structures are aligned with the vertical axis of the detector. Six orders of diffraction peaks are immediately observed in the horizontal axis of the detector. The appearance of six orders of diffraction peaks is indicative of the highly periodic pattern of the fabricated lines. A halo of scattered intensity is also observed at the center of the detector and arises from the photoresist polymer film. After subtracting the

scattered intensity of the polymer film from the data in Fig. 2, the halo disappears. Additionally, an analysis of the widths of the diffraction peaks from this sample indicates that the wavelength spread is sufficient to account for the peak widths.

From an analysis of the peak positions, the critical dimension of the line pattern can be determined. Figure 3 shows the peak position as a function of the diffraction order index. The width of the line structures can be determined from a straightforward linear fit to the data. From Fig. 3, the slope of the fitted line is  $(2.073 \times 10^{-3} \pm 6.0 \times 10^{-6}) \text{ \AA}^{-1}$  corresponding to a feature repeat distance,  $D$ , of  $(3031 \pm 9) \text{ \AA}$ .

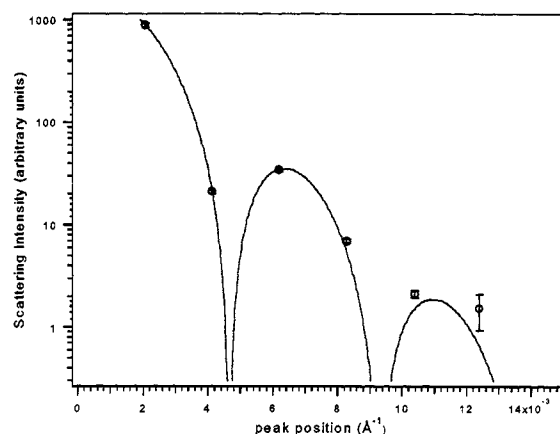


**FIGURE 3.** Peak position plotted as a function of the order of the diffraction peak. The solid line is the best fit line to the data. From this fit, the size of the lines is determined to be  $(3031 \pm 9) \text{ \AA}$  [8].

A more detailed analysis provides a quantitative determination of the average profile of the line structures including a measure of the line-edge roughness. We model the periodic line pattern as a convolution of a periodic delta function with the average cross-section of a line. The cross-section of a line is modeled as a square wave function  $\Pi(H,L)$  (where  $H$  is the height of the line and  $L$  is half the line width) convoluted with a Gaussian function with a half width,  $\xi$ . In this description, the full width at half the maximum height is approximately,  $2(L^2 + \xi^2)^{1/2}$ . Thus, the magnitude of  $\xi$  provides a measure of the line-edge roughness of an average line. More specifically, the SANS data can be modeled by the Fourier transform of the real space description of the line pattern (the form factor,  $F(q)$ ) given by Equation 1.

$$F^2(q) = 4H^2\xi^2 \left( \frac{\sin Lq}{q} \right)^2 \exp\left(-\frac{q^2\xi^2}{2}\right) \quad (1)$$

In Figure 4, the scattering intensity of a given diffraction peak is plotted as a function of the position of the peak. The solid line is the best fit to the experimental data using equation 1. The parameters  $H$ ,  $L$ , and  $\xi$  were found to be  $1.536 \times 10^{-4} \pm 6.87 \times 10^{-6}$ ,  $(675 \pm 30) \text{ \AA}$ , and  $(213 \pm 11) \text{ \AA}$ , respectively.  $H$  is dimensionless in this case, but could be placed on an absolute intensity scale in future experiments. The height of the line could be quantitatively determined if the absolute intensity was known.



**FIGURE 4.** Scattering Intensity of each diffraction peak as a function of the peak position. The solid represents the best fit to the data using the form factor equation (1).

Additionally, the second and fourth order diffraction peaks are visible and less intense than the first and third diffraction peaks. If the lines in the periodic pattern were precisely half the overall repeat distance, the second and fourth order diffraction peaks would be extinguished by minima in the form factor. The appearance of the even order diffraction peaks indicates that the line feature size is slightly less than one half the repeat distance.

The average line structural size and cross-section were determined in a configuration where the sample was placed perpendicular to the incident neutron beam. More three-dimensional information about the average line structure can be obtained by tilting the line pattern with respect to the incident beam. Varying projections of the line profile onto the detector plane provide an elegant method to deduce more specific structural information. These concepts will be tested in the future.

## SUMMARY

Using a model pattern of periodic and equally spaced lines 150 nm wide, we have demonstrated the first use of SANS to quickly, non-destructively, and quantitatively measure both the average dimension and cross-section of lithographically prepared structures. The SANS measurements were facilitated by a convergence of length scales from increases in the observable angular range with newly developed neutron focusing optics, increased neutron beam fluxes, and decreases in feature sizes that can be fabricated lithographically. Unlike microscopy-based metrology methods such as SEM and AFM, SANS measurements can be performed directly on samples as prepared on silicon single crystal substrates and become easier to perform as feature sizes decrease. Although the measurements in this paper focused on a diffraction grating pattern to facilitate the analysis procedure, the formalism to extend the SANS theoretical framework to arbitrary shapes is well-established and will be developed in the future. With these advances, SANS may be used to identify resolution limits in new nanofabrication processes and materials and to serve as an important metrology tool to understand the physical processes that control the resolution of these methods.

## ACKNOWLEDGEMENTS

The authors gratefully acknowledge technical assistance during the SANS measurements from Charles J. Glinka, Paul S. Butler, and Sung-min Choi.

## REFERENCES

1. Martin, Y., and Wickramasinghe, K., *Appl. Phys. Letters* **64**, 2498 (1994).
2. Nelson, C., and Plamateer, S., and Lyszczarz, T., *Proc. SPIE* **3332**, 19 (1998).
3. Reynolds, G. W., and Taylor, J. W., *J. Vac. Sci. Technol. B.* **17** 334 (1999).
4. Reynolds, G. W., and Taylor, J. W., *J. Vac. Sci. Technol. B.* **17** 2723 (1999).
5. Willson, C. G. in *Introduction to Microlithography, 2<sup>nd</sup> Ed.*, edited by L. F. Thompson, et. al., American Chemical Society, Washington, DC, 1994, p. 139.
6. Choi, S. M., Barker, J. G., Glinka, C. J., Cheng, Y. T., and Gammel, P. L., *J. Appl. Cryst.*, in press.
7. Eskilden, M. R., Gammel, P. L., Isaacs, E. D., Detlefs, C., Mortensen, K., and Bishop, D. J., *Nature* **391** 563 (1998).
8. All data in the text and in the figures are presented with the standard error associated with the measurement.