# Pattern Fidelity in Nanoimprinted Films using CD-SAXS<sup>1</sup>

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# ABSTRACT

The primary measure of process quality in nanoimprint lithography (NIL) is the fidelity of pattern transfer, comparing the dimensions of the imprinted pattern to those of the mold. As a potential next generation lithography, NIL is capable of true nanofabrication, producing patterns of sub-10 nm dimensions. Routine production of nanoscale patterns will require new metrologies capable of non-destructive dimensional measurements of both the mold and the pattern with sub-nm precision. In this article, a rapid, non-destructive technique termed Critical Dimension Small Angle X-ray Scattering (CD-SAXS) is used to measure the cross sectional shape of both a pattern master, or mold, and the resulting imprinted films. CD-SAXS data are used to extract periodicity as well as pattern height, width, and sidewall angles. Films of varying materials are molded by thermal embossed NIL at temperatures both near and far from the bulk glass transition (T<sub>G</sub>). The polymer systems include a photoresist, representing a mixture of a polymer and small molecular components, and two pure homopolymers. Molding at low temperatures (T-T<sub>G</sub> < 40 °C) produces small aspect ratio patterns that maintain periodicity to within a single nanometer, but feature large sidewall angles. While the pattern height does not reach that of the mold until very large imprinting temperatures (T-T<sub>G</sub>  $\approx$  70 °C), the pattern width of the mold is accurately transferred for T-T<sub>G</sub> > 30 °C. In addition to obtaining basic dimensions, CD-SAXS data are used to assess the origin of loss in pattern fidelity.

Keywords: Nanoimprint Lithography, CD Metrology, X-ray scattering, sub-100 nm lithography

#### 1. INTRODUCTION

Nanoimprint lithography (NIL) is a low-cost, effective nanofabrication tool for patterning arbitrary structures with critical dimensions (CD) well below 50 nm. To date, novel patterns with feature sizes smaller than 5 nm have been demonstrated<sup>1,2</sup>. Representing a potential solution for true nanofabrication, NIL is now listed in the International Technology Roadmap for Semiconductors as a potential next generation lithography for semiconductor fabrication<sup>3</sup>. In addition to semiconductors, the potential of NIL to pattern polymers, and perhaps other materials,<sup>4</sup> of arbitrary chemistry promises to provide a low-cost fabrication alternative to a host of developing technologies including nanoelectronic machines (NEMS), photonic waveguides, high density storage devices, and microfluidic devices.<sup>5-8</sup> Fabricating devices with NIL will require precise control over dimensions of the imprinted pattern, even on the level of nanometers. The fidelity of pattern transfer is therefore a critical indication of the suitability of a specific set of NIL parameters, requiring precise dimensional characterization of both the imprinted pattern and the mold. However, current metrologies face significant challenges in characterizing pattern fidelity. In particular, the need to examine the original mold in a non-destructive manner restricts the application of traditional imaging techniques such as scanning or transmission electron microscopy. Visible light based metrologies also face increasing challenges as feature sizes shrink to an order of magnitude below the probing wavelength.

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We have developed a non-destructive metrology capable of sub-nm precision in dimensional measurements. Critical Dimension Small Angle X-ray Scattering (CD-SAXS) measures the diffraction from a repeating pattern by transmission X-ray scattering (see figure 1). Using X-rays with an appropriate energy (> 13 keV), the flux through silicon wafers, glass, and quartz substrates is sufficient to measure most common molds as well as the imprinted patterns. The sub-Angstrom, greatly enhances the ability to extract information on patterned media through diffraction, even in dense patterns of < 10 nm in width. This technique has been previously applied to the study of photoresist materials,<sup>9,10</sup> however the capability to measure both the mold and the imprinted pattern with identical instrumental parameters makes CD-SAXS an ideal candidate for NIL metrology. Here, we apply CD-SAXS to a critical issue in NIL, namely the fidelity of pattern transfer.

Process parameters that dictate the fidelity of pattern transfer in thermally embossed NIL include imprinting temperature, pressure, polymer molecular weight, and surface chemistry of the mold.<sup>11-14</sup> A critical issue in NIL is the reduction of length scales from the macroscopic to length scales that approach molecular dimensions. Within these levels of confinement, the rheological properties of high molecular weight polymers are expected to be controlled by the polymer/mold surfaces after flow begins into the mold. The initial stages leading to flow into the mold, where the mold initially deforms the film surface, may be dictated by the surface properties of the unimprinted film. This region may be particularly important when imprinting at small  $T-T_G$  and large pressures. Since the resulting patterns in this region are likely to be of small aspect ratio, high precision metrologies of shape and pattern replication are required. As mold filling begins, prior studies using molds with large cavities (> 1  $\mu$ m) have shown that the cavity walls fill first, through a capillary wetting mechanism, followed by a filling of the cavity interior.<sup>11</sup> As the cavity size decreases, however, the filling mechanism is completely dictated by the surfaces and becomes similar to "plug flow" found in macroscopic systems. For cavities with widths of < 500 nm, we expect a simple flow that fills the entire width when the polymer readily flows (i.e. T >> T<sub>G</sub>). Under these conditions, and relatively vertical sidewalls, the height of the imprinted pattern will depend on the processing variables until complete mold fill is achieved. In contrast, the average values of sidewall angle and line width may be relatively independent of the same variables. Finally, warpage of a pattern, even under optimal conditions, may occur due to residual stresses in the patterns, resulting in restoring forces, and uneven mold removal, resulting in systematic pattern tilt.<sup>13,15</sup>

Using these observations of the NIL process, it is possible to understand the efficacy of processing parameters simply from a knowledge of the resulting pattern shape. Large aspect ratio patterns that mimic the mold dimensions are expected from optimal processing parameters. In this case, the pattern height may be the best indicator of the distance from optimum conditions. Optimal conditions may still result in pattern warpage through restoring forces, while mold removal can serve to cause a systematic tilt across a wide area. In this study, we use CD-SAXS to compare the final shape of a series of polymers imprinted under varying temperatures to the analogous dimensions of a silicon mold.

# 2. SCATTERING MODEL

The diffraction of a collimated X-ray beam passing through a nanoimprinted pattern can be modeled by the relationship  $I(\mathbf{Q}) = \Omega F(\mathbf{Q}) F^*(\mathbf{Q})$ , where  $\Omega$  is a  $\mathbf{Q}$  independent constant.  $F(\mathbf{Q})$  is the Fourier transform of a function describing the relative positions of mass in the pattern, where  $\mathbf{Q}$  is the resulting inverse space vector, and \* denotes the conjugate value. Approximations used to obtain this form of  $I(\mathbf{Q})$  are considered valid in the limits of the CD-SAXS geometry, namely a transmission geometry and a low probability of multiple scattering. A detailed discussion of the CD-SAXS models are provided elsewhere<sup>10</sup>, and the discussion here is limited to the modeling of pattern fidelity. The loss of phase information in the conjugate product makes an analytical extraction of  $F(\mathbf{Q})$  from  $I(\mathbf{Q})$  impossible. The primary method of determining dimensions is therefore to build a real space model of the pattern cross section and fit the resulting Fourier transform to the CD-SAXS data.

Using this premise, a real space model cross section of an isolated nanoimprinted line in the real space x-z plane is given in figure 1b. After Fourier transformation, the predicted scattered intensity is given in the  $Q_x$ - $Q_z$  plane. The model line shape used here is a trapezoid characterized by a width, W, height, H, and two sidewall angles,  $\beta_1$  and  $\beta_2$ . The width and height are manifested as periodic functions along  $Q_x$  and  $Q_z$  respectively, with minima occurring at  $W = 2\pi n/Q_x$ , where n is an integer, and  $H = 2\pi n/Q_z$ . The two sidewalls produce characteristic intensity ridges along  $Q_z = \beta_1 Q_x$  and  $Q_z = -\beta_2 Q_x$ . Extending the model of an isolated line to a series of periodically spaced lines results in a series of diffraction peaks at  $Q_x = 2\pi/L$ , where L is the repeat period of the pattern. While the spacing of the peaks results from the lattice, the relative intensities follow the form of the isolated line predicted in figure 2. The model used here is therefore a periodic lattice of trapezoidal lines, with parameters of periodicity, average line width and height, and two sidewall angles. Finally, an excess decay of  $I(q_x)$  beyond that predicted by the trapezoidal model is expected from the distribution in periodicity. This distribution can arise from random variations in the average line position, where the line width is constant, or from variations in the line width that also affect the periodicity. In both cases, this factor is approximated by an effective Debye-Waller factor similar to that derived for fluctuations in crystal lattices. The resulting quantity is the root mean square average of the deviation in periodicity across the beam spot size,  $\langle \delta L^2 \rangle^{1/2}$ , where the brackets indicate an ensemble average. As described here, each of these parameters is independently specified by the form of the intensity in the  $Q_x - Q_z$  plane.



**Figure 1.** Schematic of CD-SAXS instrumental geometry (left) showing the X-ray beam (solid thick lines) in transmission through the patterned sample. The intensity, I, is measured on a 2-D detector as a function of scattering angle, 2 $\theta$ , and converted to I(q<sub>x</sub>), where q<sub>x</sub> is defined in the text. A series of measurements are performed at varying angles of incidence to the sample, where the sample rotation angle,  $\omega$ , is shown. After conversion from the qx- $\omega$  plane, the intensities from a trapezoidal cross section would appear as predicted in the model calculation on the right, plotted as I as a function of the Fourier components, Q<sub>x</sub> and Q<sub>z</sub>.

#### **3. EXPERIMENTAL**

Polymers used in this study included atactic Poly(methyl methacrylate) (PMMA), atactic Poly(styrene) (PS), purchased from Microchem Inc.<sup>16</sup>, and S1813 photoresist from Shipley Co. The PMMA has a number average relative molecular mass ( $M_{r,n}$ ) of 950,000 and polydispersity (PDI) of 2.2, and the PS has  $M_{r,n} = 50,000$  with a PDI = 2.1. All Films were spun cast from solution on 200 mm diameter silicon wafers and annealed afterward to remove excess solvent. The resulting film thicknesses were measured by profilometry and found to be  $\approx 310$  nm for both the PMMA and PS, and  $\approx 260$  nm for the S1813. The nanoimprint mold consists of a set of parallel lines and spacings with a line:space ratio of approximately 3:1. The line-space pattern covers a square with sides of 5 mm. CD-SAXS measurements were performed near the center of the imprinted area. The mold was fabricated in silicon oxide via 193 nm optical photolithography using a JSR 1237R resist on a 200 mm SEMI-standard Si wafer. Nanoimprint lithography was performed on an Obducat NIL 4 (Series 1) machine. Imprinting was conducted in the thermal embossing mode for 10 min at a pressure of 6 MPa. Subsequently the mold was cooled to below T<sub>G</sub> before the sample was removed from the mold. For each sample, scanning electron microscopy (SEM) images were taken of identically prepared samples after cleaving to provide cross sectional views of the patterns. The SEM images reveal a residual, unpatterned layer that is > 100 nm thick in all samples.

CD-SAXS experiments were performed at the 5-ID-D beamline of the DND-CAT at the Advanced Photon Source, part of Argonne National Laboratory. An X-ray energy of E = 17 keV was selected using a double monochromator, corresponding to a wavelength of  $\lambda = (0.0729 \pm 0.0001)$  nm. At this wavelength, silicon wafers used in microelectronic fabrication, with total thickness  $\approx 0.5$  mm, have a transmission of  $\approx 0.45$ . The sample is oriented with the pattern on the detector side to reduce any possibility of beam damage. We further note that prior studies on various patterns in photoresists have not demonstrated measurable changes in pattern shape during exposure to the X-ray beam. Diffraction of the pattern is recorded on a 2-Dimenionsal CCD detector with 2048 x 2048 pixels. The detector was set at a distance of  $(700 \pm 1)$  cm from the sample. Intensities were recorded as a function of the scattering vector,  $q (= 4\pi/\lambda \sin(\theta)$ , where 2 $\theta$  is the angle of diffraction as depicted in figure 1). Mapping of the Q<sub>x</sub>-Q<sub>z</sub> plane described above requires a series of measurements at varying angles of sample rotation,  $\omega$ . The relationship  $I(Q_x,Q_z) = I(q, \omega) R(\omega)$ , where R is a rotation matrix projecting the vector q into the Q<sub>x</sub>-Q<sub>z</sub> plane, is then used to transform the data into the sample coordinate system.



**Figure 2.** CD-SAXS data from a silicon master (top left) shown as a function of  $\omega$  and  $q_x$  is compared to analogous data from a 2-D model of a trapezoidal cross section (bottom left). Also shown is a comparison of intensity contours from experiment (open circles) and the model (solid line) at constant  $q_x = 0.225 \text{ nm}^{-1}$  (top right) and constant  $\omega = -0.4^{\circ}$  (bottom right).

## 4. RESULTS AND DISCUSSION

CD-SAXS measurements of a silicon oxide master are shown in figure 2. The cross section of the pattern is obtained by fitting the diffracted intensity of the pattern to the form of a model cross section over a wide range (-45 to +45)° of incident angles. Both experimental and resulting model data are plotted as a contour map of intensity in the  $\omega$ -q<sub>x</sub> plane.

Prominent in this form of the data are the Bragg diffraction peaks along the  $q_x$  axis. As the sample is rotated, the Bragg peak positions smoothly vary with the projected repeat distance. The maximum period defines the condition of a beam at normal incidence to the plane of the pattern. The intensity ridges created by the sidewalls can be observed at  $\omega = \beta_1$ and  $\omega = \beta_2$ , providing a direct measure of the sidewall angles. Visual examination of the data in figure 2 therefore supports the validity of the trapezoidal cross section as a model of the mold. The fitted dimensions of the mold, and subsequent imprints, are given in Table 1. We note that the conservation of length scales between periodicity, line width, and space width results in two identical solutions, representing values for W and L - W. Distinguishing between the two solutions using CD-SAXS alone requires additional analysis described elsewhere.<sup>10</sup> Here, the mold is known to have a 3:1 ratio of line and space width, and the line width is assigned the larger value. A similar redundancy exists in the vertical orientation of the trapezoid, however the nature of the NIL process usually limits molds to structures with positive sidewall angles. Finally, while CD-SAXS provides a value of average periodicity with sub-nm precision, we note that the distribution of periodicity is measured to be  $\langle \delta L^2 \rangle^{1/2} = 3$  nm. It is important to realize that this distribution must be included in any direct comparison between average techniques, such as CD-SAXS, and microscopies that examine individual or small numbers of features. In addition to variations in line position, where the lines are otherwise perfect, the Debye-Waller factor will reflect the presence of defects. However, a quantitative connection between types of defects and this factor will require further investigation. Regardless, the Debye-Waller factor is an indication of the density of defects in long range periodicity and provides a basis for comparison in subsequent imprints.



**Figure 3.** Cross section SEM images of a line space pattern imprinted into a PMMA film (left). The white bar represents a scale of 100 nm. In addition, CD-SAXS data from an identically prepared sample is shown as a function of rotation angle,  $\omega$ , and  $q_x$  (middle) and compared to the resulting model prediction using the trapezoidal cross section (right).

In figure 4, SEM images of a pattern imprinted into PMMA is shown in both "top down" and cross sectional views. Here, the patterns have been imprinted at a small T-T<sub>G</sub> (= 15 °C). Given the high molecular weight of the polymer, it is not surprising that the mold does not fill completely and patterns with a small aspect ratio result. Prior studies of thermally embossed NIL have typically used values of T-T<sub>G</sub>  $\approx$  100 °C as a result, even for polymers of lower molecular weight.<sup>14</sup> Nevertheless, the diffraction peaks from an identically prepared sample show that the long range order of the mold has been successfully transferred, maintaining the same periodicity to within a single nanometer with only a small increase in the Debye-Waller factor ( $\approx$  1 nm). The lack of visually sharp ridges in the 2-D map results from a combination of a low pattern height, broadening the intensity ridges from the sidewalls, and possibly a large distribution in sidewall angle among the lines (see figure 4). As may be expected, the trend toward low aspect ratio patterns continues with the imprinting of a PS sample at T-T<sub>G</sub>  $\approx$  5 °C. While SEM images indicate a pattern exists on the PS sample, with a height on the order of 10 nm, CD-SAXS did not detect any diffraction peaks. The lack of diffraction may be due in part to a vanishingly small pattern size, but may also reflect a high density of defects in the patterns. From this data, we suggest that a minimum positive value of T-T<sub>G</sub> on the order of 10 °C may be required to create a pattern with long range order. However, even largely non-optimal conditions of imprinting serve to reproduce the basic dimensions laterally, resulting in only a 6 nm decrease in average line width for the PMMA sample. A capability to

produce patterns with varying aspect ratio, simply by changing temperature, may be useful to produce, for instance, surfaces with controlled morphology. Surface morphology control could play a role in applications such as in nanostructured surfaces for lubrication, bottom-up assembly, and morphologically induced mixing in microfluidic channels. The strong diffraction from the PMMA sample suggests that small aspect ratio patterns can be characterized sufficiently with CD-SAXS provided a sufficient degree of long range order exists.



**Figure 4.** CD-SAXS data from nanoimprinted PMMA films shown as a function  $\omega$  and  $q_x$ . Shown are data from a film imprinted at T-T<sub>G</sub> = 70 °C (top left) and T-T<sub>G</sub> = 80 °C (bottom left). Data are compared to results of fitting to a trapezoidal model for each film (middle) and cross sectional SEM's of a sample prepared under nominally identical conditions (right). The white bar represents a length scale of 100 nm in the SEM images.

The disparity in dimensions between the mold and the low temperature PMMA and PS samples are likely due to difficulties in flowing a polymer at temperatures approaching the  $T_G$ . To achieve highly conformal imprints, it is known that large values of T-T<sub>G</sub> ( $\approx$  40 °C or larger) are required for most commercial NIL imprinters. Data from both PS and PMMA films imprinted at higher temperature demonstrate significant increases in pattern height and follow the dimensions of the mold very closely. The heights of the patterns are all within 20 nm of the original mold, but the high aspect ratio also makes the patterns subject to systematic leaning. For the case of a PMMA pattern imprinted at T-T<sub>G</sub> = 70 °C, the pattern remains relatively vertical with two positive sidewall angles, and replicates the molds dimensions to within a 1 and 6 nm in width and height respectively. These results suggest that the temperature, time, and pressure are nearly optimal for this polymer. For the PS and a subsequent PMMA imprint, the dimensions also approximate those of the mold, however tilting of the lines is significant enough to make one of the sidewall angles negative. Since the CD-SAXS data represent an average over hundreds of lines, this tilting occurs systematically over a large (100 µm X 100 µm) area and is therefore likely to be caused during mold removal.



**Figure 5.** Cross section SEM images of two S1813 films imprinted at  $T-T_G = (20 \text{ to } 30)$  °C and  $T-T_G = (60 \text{ to } 70)$  °C. Images show a small increase in pattern height with increasing temperature. The white bar represents 100 nm in the SEM images.

The generality of NIL as a nanofabrication tool is further illustrated in figure 7, where a photoresist (Shipley S1813) film is imprinted at T = 130 °C. While the photosensitive nature of a photoresist is not required for thermally embossed NIL, the chemical composition of photoresists are naturally tuned to possess high etch resistance. NIL of photoresists could therefore provide the dimensions required of next generation lithography while maintaining the established etch properties of photoresists. As with the simpler homopolymers, the resist system shows conformality at  $T-T_G > 20$  °C, maintaining periodicity and line width. In addition, this particular sample shows conformality in sidewall angle. The SEM images of figure 5 further demonstrate a capability to control pattern aspect ratio simply through temperature.

The limitations of CD-SAXS are general to techniques that provide average measures, namely an inability to see small concentrations of defects, especially when the defects are not spatially correlated. Further development of the technique will attempt to provide quantitative measures of the distributions in each of these parameters, in a manner similar to the Debye-Waller factor in the periodicity, as well as addressing more complex cross sectional shapes. Given the transmission geometry of the technique, and its capability to do measurements on the time scale of seconds,<sup>15</sup> it is also conceivable that in-situ measurements are possible, directly following the flow of material into the mold.

Table 1. Results of fitting CD-SAXS data to a trapezoidal cross sectional model. Shown are the
samples, their imprinting temperature relative to their bulk glass transition temperature, and the resultin
periodicity (L), line width (W), line height (H), sidewall angles ( $\beta_1$ and $\beta_2$ ), and deviation in periodicity
The values are provided with standard uncertainties. The value of W for the silicon mold represents the
width of the space and not the width of the actual line, to provide a clearer comparison with the imprinte
patterns.

Sample	$T_{imprint}$ - $T_{G}$	L (nm)	W (nm)	H (nm)	β <sub>1</sub> (°)	β <sub>2</sub> (°)	$<\delta L^{2}>^{1/2}$
	(°C)						( <b>nm</b> )
Silicon	NA	$358.4 \pm 0.5$	$111.5 \pm 1.0$	$167 \pm 3$	$5.6 \pm 0.1$	$5.6 \pm 0.1$	$3.2 \pm 0.5$
Mold							
PMMA	$\approx 10$	$358.4\pm0.5$	$105.5 \pm 1.0$	$50 \pm 10$	$10 \pm 2$	$3\pm 2$	$4.4 \pm 1.0$
PMMA	pprox 70	$358.4\pm0.5$	$111.5 \pm 1.0$	$161 \pm 5$	$2.2\pm0.5$	$5.8 \pm 0.5$	$3.0 \pm 0.5$
PMMA	pprox 80	$358.4\pm0.5$	$96.5 \pm 1$	$149 \pm 10$	$-4.0 \pm 1$	$11.1 \pm 1$	$3.5 \pm 0.5$
PS	$\approx 5$	NA	NA	NA	NA	NA	NA
PS	$\approx 45$	$358.4\pm0.5$	$99.5 \pm 1.0$	$151 \pm 10$	$12 \pm 1$	$-6.0 \pm 1$	$3.5 \pm 0.5$
S1813	$\approx$ (20 to 30)	$358.4\pm0.5$	$105.5 \pm 1$	$140 \pm 1$	$5.4 \pm 0.5$	$5.6\pm0.5$	$3.5 \pm 0.5$
S1813	$\approx$ (50 to 60)	NA	NA	NA	NA	NA	NA

## **5. CONCLUSIONS**

A technique based on transmission X-ray scattering, termed Critical Dimension Small Angle X-ray Scattering (CD-SAXS) was used to characterize the fidelity of pattern transfer in thermally embossed NIL patterns. Line/space patterns with 1:3 spacing were imprinted in films of PMMA, PS, and S1813 photoresist and their cross section characterized non-destructively. Dimensions were compared to analogous measurements of the silicon oxide master and cross sectional SEM of nominally identically prepared. Patterns were modeled with a trapezoidal cross section, providing sub-nm precision in periodicity, nanometer level precision in pattern width and height, and sub-degree precision in sidewall angle. The technique also reveals slight tilts in the resulting structures, likely due to deformation during mold release, as well as sensitivity to more complex cross sections. Finally, the transmission geometry of CD-SAXS suggests the possibility of non-destructive, in situ metrology of the molding process.

# ACKNOWLEDGEMENTS

Funding for this research was provided by the NIST Office of Microelectronic Programs. This work was funded in part by the NIST Office of Microelectronic Programs and the National Institute of Health. The Advanced Photon Source is supported by the U.S. Department of Energy under contract no. W-31-109-Eng-38. The mold was provided by A. Mahorowala of the IBM T. J. Watson Research Center, Yorktown Heights, NY.

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- 16. Commercial equipment and materials are identified in this paper only to adequately specify experimental procedure. In no case does this imply endorsement or recommendation by the National Institute of Standards and Technology.