Study on the Effects of X-ray Energy and Beam Size on CD-SAXS Measurement Precision

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Abstract

As the semiconductor industry advances toward sub-7-nm nodes, Critical Dimension Small-Angle X-ray Scattering (CD-SAXS) has become an important technique for the quantitative measurement of nanoscale variations. This study investigates the influence of X-ray beam size and photon energy on the precision of critical dimension measurements. Critical dimensions measured with different spot sizes exhibit deviations from expected values, and both excessively large and small beam sizes are detrimental to confidence interval optimization. With increasing incident energy, X-ray transmittance increases while the scattering cross-section decreases, leading to a gradual reduction in the signal-to-noise ratio of diffraction peaks, thereby diminishing CD-SAXS measurement precision. Optimal precision was achieved at an energy of 12 keV using a smaller beam size and an effective trapezoidal model: average pitch of $100.4 \pm 0.2 nm$, $linewidthof 49.8 \pm 0.2 nm$, $heightof 130.0 \pm 0.2 nm$, $andsidewallangleoflessthan 1.1 \pm \$0.1°$. These results provide important guidance for CD-SAXS laboratory construction and X-ray equipment development, offering strong support for research in related fields.

Full Text

Preamble

Study on the Impact of X-ray Energy and Beam Size on CD-SAXS Measurement Precision

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With the development of the semiconductor industry below the 7 nm node, Critical Dimension Small Angle X-ray Scattering (CD-SAXS) has emerged as a powerful tool for quantitatively measuring nanoscale deviations. In this work, the effects of X-ray beam size and photon energy on the accuracy of critical dimension measurements were systematically investigated. Measurements using different beam spot sizes showed varying deviations from expected values, with both excessively large and small beam sizes failing to improve confidence intervals. As incident energy increases, the X-ray transmission rate increases while the scattering cross-section decreases, resulting in a gradual reduction of the signal-to-noise ratio of diffraction peaks and consequently diminishing CD-SAXS measurement accuracy. Optimal accuracy was achieved at 12 keV with a smaller beam size. Using an effective trapezoid model, the results yielded an average pitch of 100.4 ± 0.2 nm, width of 49.8 ± 0.2 nm, height of 130.0 ± 0.2 nm, and a sidewall angle of 1.1 ± 0.1°. These findings provide crucial guidance for future CD-SAXS laboratory development and X-ray instrument construction, offering robust support for research in related fields.

Keywords: critical dimension small angle X-ray scattering, nonlinear fitting, beam size, X-ray energy, chip

Introduction

In the rapidly evolving fields of microelectronics and nanotechnology, the semiconductor industry is undergoing significant transformation. Manufacturers continuously strive to enhance chip performance, add functionalities, and integrate more components onto chips [1–7]. As integrated circuits advance, planar architectures can no longer meet development needs, prompting the adoption of advanced processes such as 3D transistor designs and intricate patterning techniques. The fin-based field-effect transistor (FinFET) remained the mainstream device option until 2021, when gate-all-around (GAA) designs emerged and dominated at smaller dimensions due to their superior electrostatic control. Meanwhile, techniques such as 3D stacking and 3D very large-scale integration (3DVLSI) have introduced numerous additional steps to integrated circuit production processes [8, 9]. In these processes, the grating serves as a crucial microstructure playing a vital role in chip manufacturing. With technological advancements, the critical dimensions of chips have gradually decreased, requiring more accurate and high-resolution measurement techniques. The rapid reduction in grating size poses considerable challenges for precise measurement.

Among methods used for measuring grating dimensions, Scanning Electron Microscopy (SEM) and Atomic Force Microscopy (AFM) can detect local critical dimensions in gratings [10]. Optical Critical Dimension (OCD) is another widely applied technology, especially for studying non-planar FinFET devices on silicon

insulators [11, 12].

Small angle X-ray scattering has been extensively employed across numerous scientific disciplines, including physics, chemistry, materials science, and life sciences, as a highly effective technique for examining structural characteristics at the nanoscale [13–18]. It enables non-destructive analysis of nanoparticle dimensions, morphology, and distribution [19]; investigation of biological macromolecule structures [20, 21]; measurement of nanoparticle size, shape, and size distribution; and analysis of thin-film microstructures for solar cells [22, 23]. Notably, the CD-SAXS technique, developed by scientists at NIST, stands out as a promising measurement platform for revealing the three-dimensional configuration of regularly spaced arrays of nanostructured surface elements [24, 25]. CD-SAXS holds potential as a future alternative to OCD measurement methods. This advanced X-ray scattering technique can explore critical dimensions, morphology, structure, and organization of materials at the nanoscale. Being non-destructive, CD-SAXS enables measurement of X-ray scattering angle and intensity distribution in materials, with its key advantage lying in revealing detailed microstructural information at the nanoscale. By precisely measuring critical dimensions such as line width and spacing, CD-SAXS contributes to process control and optimization, offering precision at the nanometer scale while remaining non-destructive.

In the field of advanced 3D microelectronics architecture, CD-SAXS has demonstrated outstanding performance in providing high-precision critical dimension measurements [26]. It has been effectively utilized to investigate widely adopted FinFET grating structures and has validated its measurement capabilities on potential future grating architectures [27]. Since roughness critically impacts transistor performance, CD-SAXS' s ability to measure roughness information has exhibited robust characterization potential under varied conditions, including both synchrotron radiation sources and laboratory environments [28]. Previous research indicates that grating critical dimension precision is affected by X-ray energy, highlighting the need for further studies on how X-ray energies influence measurement accuracy. The technology has marked significant advancements in addressing grating roughness issues, providing important insights for optimizing transistor performance [29, 30]. Notably, CD-SAXS experiments conducted under synchrotron radiation sources not only provide highly accurate data for grating roughness but also lay the foundation for designing and optimizing future grating structures. Furthermore, the successful application of CD-SAXS in laboratory environments has promoted the development and dissemination of CD-SAXS technology.

Currently, CD-SAXS primarily utilizes synchrotron radiation as its X-ray source. Although synchrotron radiation can meet requirements for high brightness and sensitivity, CD-SAXS applications under laboratory X-ray sources still face certain limitations. To address challenges associated with the high cost and constrained experimental conditions of synchrotron sources and to expand the technology' s application domains, establishing laboratory X-ray sources is crucial.

The selection of target materials for laboratory X-ray sources is a critical step in determining X-ray energy spectra to meet specific grating measurement requirements. Additionally, the influence of beam size on measurement precision has not been systematically investigated in previous studies. In this work, we examined the effects of varying beam size and X-ray photon energies on CD-SAXS experimental accuracy.

II. Experimental and Calculation Methods

CD-SAXS measurements were conducted at the small-angle X-ray scattering beamline of the Shanghai Synchrotron Radiation Facility (SSRF). Measurements were performed with 12 keV photons at the center position of the sample, utilizing four different beam sizes. Additionally, a further series of measurements were performed at the same sample position with the same beam size, employing three different photon energies.

A. Critical Dimension Small Angle X-ray Scattering

CD-SAXS measurements were conducted on a trapezoid sample, detailed in Figure S1, which illustrates a trapezoidal profile grating model characterized by several geometrical parameters: pitch L, height h, width W, the sidewall angle (SWA) , and the roughness of the sidewall.

This study utilized transmission small-angle X-ray scattering through a silicon substrate with a thickness of 0.7 mm. The measurement geometry, as elaborated in Figure S2, specifies the q vectors for both the beam and the detector. The configuration aligns the Z-direction with the incoming beam, while the X- and Y-directions are set perpendicular to both the primary beam and each other. The sample was placed on a rotating platform with the line direction of the grating aligned parallel with the rotation axis. Its rotation range was from $-50°$ to $50°$ with a step of $1°$, and the data collection time at each angle was 10 seconds. To facilitate visualization of diffraction data from all tilt conditions simultaneously, a reciprocal space map (RSM) was constructed by extracting the diffracted intensities from each individual tilt condition into a one-dimensional scattering profile.

The scattered intensity I(q) from the system was recorded as a function of the scattering vector q, whose modulus value can be obtained from Equation (1), where 2 is the angle of diffraction (see Figure S2) and λ is the X-ray wavelength.

$$|q| = \frac{4\pi \sin\theta}{\lambda} \tag{1}$$

The diffraction peak separation is given by Equation (2), where L is the period of the grating sample, thus facilitating straightforward acquisition of pitch information. Critical dimensions describing the nanostructure are determined by the

relative intensity and profile of the peaks at different rotation angles ω relative to the incidence.

$$q_x = \frac{2\pi}{L} \tag{2}$$

The three-dimensional configuration of the lines is depicted through a sequence of form factors that have been convoluted with the structure factors from an ideal grating. The general form of the scattered intensity in CD-SAXS is calculated from Equations (3)-(5), where (r) is the shape function [30, 31], L is the pitch, and $*$ denotes the convolution operation. The Debye-Waller factor σDWF describes the interfacial roughness. In application, the Fourier transform of each trapezoid in the model is computed, and results are aggregated to determine the amplitude Af(q), which is then used in the calculation of If(q). The structure factor of the grating reflects the periodic information of the grating nanostructure. Since the Voigt profile serves as a theoretically natural description for the shape of diffraction peaks, the Voigt function' s profile can be adjusted by tuning its parameters to match the shape of each diffraction peak in the CD-SAXS experimental results [32]. Using the structure factor, each Voigt profile is constrained to periodic positions along the qx direction defined by the pitch L. In practice, the structure factor can be constructed by convoluting the Voigt profile with a one-dimensional lattice of Dirac delta functions along the qx direction.

$$A_f(q) = \int \sum \rho(r) * \sum \delta(x - nL) \exp(-iqr) dr \tag{3}$$

$$I_f(q) = |A_f(q)|^2 \tag{4}$$

$$I(q) = I_f(q) \exp\left(-q^2 \sigma_{DWF}^2\right) \tag{5}$$

The analysis of CD-SAXS involves an inverse and iterative approach. This method compares the calculated scattering from a hypothesized form function with the actual scattering data. The trial shape is adjusted iteratively until the calculated scattering aligns with the observed scattering data.

For the trapezoidal grating sample in this trial, the influence of its various parameters on the form factor is illustrated in Figure S3. From these graphs, it can be observed that different critical dimensions of the grating have varying effects on the shape factor. Comparing the influences of each parameter aids in adjusting their ranges to fit experimental data more accurately.

B. Curve Fit

Curve fitting is a prevalent technique in the sciences and engineering to approximate the functional relationship represented by discrete data points [33-

36]. Through mathematical methods, these discrete data points are represented using continuous curves or more densely packed discrete equations, enabling mathematical calculations, theoretical analysis of experimental results, and even estimation for unmeasurable locations. This method plays a crucial role in solving many problems that rely solely on sampling or experimentation to acquire discrete data.

The fitting of experimental data is framed as an optimization problem, with the fitting function' s undetermined parameters including the critical dimensions in CD-SAXS. The optimization objective aims to minimize a certain error metric between the observed experimental data and the function values of the fitting function. A typical optimization goal is to minimize the sum of squares of the errors between the values of the fitting function and the observed data. Alternatively, in cases where the significance or distribution of observed data varies, a weighted sum of squares of errors can be employed as the optimization objective [37, 38].

The least squares approach is the fundamental method for data fitting. For observed data (xi, yi), (i = 1, 2, ···, n), the objective function of the optimization problem is to minimize the sum of squared errors between the observed experimental data yi and the calculated values f(xi, p) of the fitting function f(xi), as illustrated in Equation (6), where (p1, ···, pm) represent the undetermined parameters within the fitting function.

$$\min J(p_1, ..., p_m) = \sum_{i=1}^{n} [y_i - f(x_i)]^2 \tag{6}$$

Theoretical computation of CD-SAXS involves parameters related to several critical dimensions, along with considerations for describing the width of diffraction peaks. Given the high-dimensional parameter space formed by the parameters to be fitted, navigating for optimal solutions within vast datasets poses significant challenges. To address this, an advanced curve fitting approach was employed, combining a stepwise fitting strategy with iterative optimization.

Initially, traditional gradient descent methods were tested but proved inadequate for locating the global optimum within the complex parameter space. Instead, a stepwise fitting approach was adopted, beginning with data visualization and focusing on analyzing the impact of each parameter on the function' s graph. First, partial parameter fitting is performed on specific parameters without considering the influence of others, thereby reducing the dimensionality of the parameter space. Subsequently, an iterative optimization strategy is applied, where multiple starting points are randomly selected within the parameter space, and the loss function (mean squared error) is evaluated for each iteration. The minimum value of the loss function is identified as the optimal solution. To ensure reproducibility, the random seed for the starting points is fixed, and the fitting process is repeated multiple times to confirm consistency. This combined approach has been shown to significantly improve the accuracy

and reliability of the fitting results, thereby enabling efficient navigation of the high-dimensional parameter space in CD-SAXS measurements.

C. Measurement Error Analysis

To ensure measurement reliability, all experimental conditions other than the variables of interest (beam size and X-ray energy) were carefully controlled. The sample environment, detector settings, and other instrumental parameters were kept constant throughout the experiments. Data collection began only after the system had stabilized following changes in beam size or X-ray energy. This rigorous control of experimental conditions minimized potential interference and ensured the validity of the results.

The raw experimental data were pre-processed to ensure accuracy and reliability. Outliers, such as those caused by detector defects (e.g., bad pixels), were identified and removed. Given the large number of data points involved in CD-SAXS fitting, the removal of outliers did not significantly impact the overall dataset. Additionally, the data were normalized to facilitate background scattering subtraction, which reduced the influence of background noise on the fitting process. This normalization step is critical for improving the precision of subsequent analysis.

In small-angle X-ray scattering experiments, Equation (7) provides a fundamental tool for describing the relationship between scattering angle , scattering wavelength λ, and scattering vector q. Simultaneously, Equation (8) offers a method for analyzing the uncertainty of the scattering vector. In this equation, Δ represents the scattering angle error caused by the pixel size, with each pixel size being fixed for the detector used in CD-SAXS measurements. As the energy of the X-rays increases, their scattering wavelength λ becomes shorter, resulting in a decrease in the corresponding scattering angles for each scattering vector q. In such cases, the impact of the scattering angle error Δ induced by pixel size in ($+\Delta$) will increase, resulting in increased uncertainty of the scattering vector. This suggests that increased X-ray energy corresponds with elevated signal error detection. This analysis provides insight into potential sources of errors when conducting small-angle X-ray scattering experiments at different energies, emphasizing the importance of taking appropriate measures to maximize experimental data accuracy and reliability.

$$q = \frac{4\pi \sin\theta}{\lambda} \tag{7}$$

$$\Delta q = \frac{4\pi \sin(\theta + \Delta\theta)}{\lambda} - \frac{4\pi \sin\theta}{\lambda} \tag{8}$$

The detector enables precise measurement of scattered signal intensity. Processing the collected data allows for determination of the peak center position. Typically, the position of the maximum intensity value in the collected data

corresponds to the center of the peak. However, the finite pixel size of the detector introduces inherent error in determining the peak center position within a pixel-sized area. The error in the peak center position caused by pixel size can be described using the uncertainty of the scattered signal, as expressed in Equation (9).

$$\frac{\Delta q}{q} = \sqrt{\left(\frac{\Delta\lambda}{\lambda}\right)^2 + (\cot\theta \cdot \Delta\theta)^2 + \left(\frac{\Delta d}{d}\right)^2 + \left(\frac{\Delta L}{L}\right)^2 + \left(\frac{\Delta\alpha}{\alpha}\right)^2 + \left(\frac{\Delta PSF}{PSF}\right)^2} \tag{9}$$

Where is the scattering angle, α is the beam divergence, d is the beam size, PSF is the point spread function of the small-angle detector, L is the distance from the sample to the detector, and $\Delta\lambda/\lambda$ is the energy resolution of the incident X-ray.

In CD-SAXS measurement experiments, the distance between the sample and the detector is maintained constant. Notably, a decrease in the beam size d, as depicted in Equation (9), results in a reduction in the Δq/q value, implying enhanced accuracy for the measurement results for each scattering vector q. Therefore, a smaller beam size is associated with more precise measurement results. However, the influence of pixel size on the results must be considered. When the beam size is small enough, the detector cannot provide the necessary resolution to accurately measure the scattering vector q due to the constraint of pixel size. If overly small, the beam size can narrow the full-width at half-maximum (FWHM) of the diffraction peak, thus challenging the detector' s ability to accurately characterize the peak' s shape and adversely affecting result accuracy. Careful beam size selection is essential to ensure accurate measurement results and overcome detector resolution limitations.

As the scattering angle increases, the uncertainty of the scattered signal decreases. Therefore, as the magnitude of the scattering vector q increases, the signal uncertainty decreases. Utilizing the center position of the first-order diffraction peak to determine grating periodic information introduces inaccuracy due to uncertainty effects, which become more pronounced with increasing scattering vector q. This phenomenon is evidenced by the concordance between theoretical and experimental peak positions for lower-order diffraction peaks, while higher-order peaks show discrepancies. Considering the characteristics of the structure factor in CD-SAXS, the peak positions of higher-order diffraction peaks can be used to determine the grating period. In this process, the uncertainty in the scattering signal caused by a single pixel can be divided into multiple portions, effectively reducing overall uncertainty and minimizing errors in fitting the grating period.

Meanwhile, gaps between pixels in some detectors may lead to undetected peak values in diffraction patterns, rendering sole reliance on maximum intensity for determining diffraction peak centers inaccurate. The shape and positional infor-

mation of each diffraction peak can be accurately determined through diffraction peak fitting methods. To mitigate this deviation, a multi-peak fitting approach is employed to fit the diffraction peaks in experimental results, simultaneously obtaining position information for each diffraction peak. The final fitting results align the peak centers of the structure factor with the diffraction peaks in experimental results, thereby enhancing measurement accuracy and reliability.

In the process of fitting diffraction peaks, the full-width at half-maximum (FWHM) determines the peak shape and subsequently helps ascertain the peak center position. Insufficient data points can lead to imprecise measurements of the FWHM, affecting both peak fitting and the final autocorrelation coefficient. Although narrower diffraction peaks facilitate peak position localization, they offer fewer data points, which limits detail in peak information. This scarcity of data complicates distinguishing background noise in the overall fitting process. Therefore, selecting an appropriate beam size is crucial for achieving accurate fitting results.

III. Results and Discussion

A. Effect of Different Beam Sizes

Table 1 shows the comparative analysis of CD-SAXS results alongside other measurement techniques. Scanning Electron Microscopy (SEM) can provide information about the grating pitch without delving into depth contrast. Atomic Force Microscopy (AFM) can theoretically measure grating depth, but due to the relatively coarse nature of AFM probes at critical scales, crucial information within surface topographic images might be obscured, leading to potential inaccuracies. Moreover, limitations may arise during grating depth measurements, restricting probe access to groove bottoms due to neighboring gratings. Consequently, AFM struggles to provide precise depth-oriented details, offering only periodic information within the scanned grating sample area.

Optical Critical Dimension (OCD) yields depth information but requires referencing results from AFM and SEM before the data fitting process. Accurate period determination in OCD measurements is critical for obtaining precise structural size information. Figure S4 shows grating measurement results from SEM, AFM, and OCD.

This study investigates the effect of various beam sizes on data acquisition and result precision during the experimental process. Four distinct beam sizes were identified on the grating sample at the small-angle X-ray scattering beamline, with the smallest spot designated as P1 and the largest as P4. These beam sizes were arranged in ascending order. Figure 1 [Figure 1: see original paper] displays the shapes and dimensions of beams P1 to P4.

It is acknowledged that there is a degree of human error inherent in sample placement. Consequently, each angle measurement result is affected by an error in the initial angle. To compensate for this error, the symmetry of Figure 2

[Figure 2: see original paper] can be utilized. Reconstructed images of CD-SAXS measurements from different angles and beam sizes produce an intensity map as a function of qx and qz, shown in Figure 2.

In CD-SAXS measurement, a silver behenate sample is used for calibrating the distance from the sample to the detector, which is set at approximately 2.95 meters. When maintaining a fixed sample-to-detector distance, a smaller beam may result in some data loss near the full-width at half-maximum (FWHM) of the diffraction peak, as shown in Figure 3 [Figure 3: see original paper]. A detailed analysis of results from the P1 group shows that each peak exists relatively independently in the one-dimensional curve. The measured data points are mainly distributed around the peak values of the diffraction peaks and between neighboring diffraction peaks.

Although this experiment rapidly determines diffraction peak positions, a significant drawback is the shortage of data points near the half-maximum of the diffraction peaks, evident in the P1 curve illustrated in Figure 3. In the process of fitting diffraction peaks, FWHM was employed to determine peak shapes and subsequently the positions of peak centers. The scarcity of data points results in large absolute values of the derivatives at the diffraction peaks' half-maximum positions, making it difficult to accurately determine the FWHM of the peak shapes. Insufficient data points lead to inaccurate measurements of the width at half-maximum, which in turn affects diffraction peak fitting and thus the final autocorrelation coefficient.

Figure 4 [Figure 4: see original paper] reveals considerable dispersion in the P1 data points, particularly in the qz direction, where the number of data points is comparatively lower than in other groups. This scarcity makes it challenging to discern the true trend, leading to decreased fitting accuracy and rendering the fitted values less reliable. Notably, the data at qx = 0.252 nm^{-1} are particularly susceptible to noise, which hinders comprehensive assessment of the overall experimental data trend. This increased difficulty in separating background signals complicates distinguishing between useful signals and background noise. Consequently, the fitting algorithm may overfit to noise, leading to significant deviations in fitted values from expected experimental outcomes.

The results of CD-SAXS measurements using different beam sizes are summarized in Table 2 . Observations show that measurements with the smallest beam size exhibit the smallest pitch confidence interval. Although results in this group show minor differences in grating width, height, and sidewall angle compared to expected values (i.e., the known critical dimensions of the reference material used in our experiments), the confidence interval for the sidewall angle was notably large. Even after many experiments, the sidewall angle result still showed high uncertainty in the P1 group. This significant sidewall angle uncertainty derives from the small beam size, which produces experimental data that does not accurately describe diffraction peak shapes, making it challenging to provide precise information about shape factors.

A moderate increase in beam size for group P2 notably narrows the confidence interval without evident deviations in fitting results. Conversely, further beam size increases in P3 and P4 elevated uncertainty in critical dimensions. Specifically, fitting results in group P4 deviate noticeably from those of other groups and from expected values, substantially reducing measurement precision. This deviation can be attributed to the relatively large beam size, resulting in diffraction peaks with comparatively smooth and broadened profiles due to the smear effect of scattering. This peak profile broadening leads to increased uncertainty in confirming exact peak center locations. In CD-SAXS data fitting, using diffraction peak values to discern shape factor trends is imperative for extracting crucial dimensional information about the grating sample. Increased uncertainty in peak center identification renders the fitting algorithm more vulnerable to nearby values, diminishing emphasis on peak centers and adversely affecting shape factor determination and fitting precision. These findings highlight the significant impact of beam size on uncertainty and precision in CD-SAXS measurements, demonstrating that appropriate beam size selection is crucial for obtaining accurate and reliable measurement results.

B. Effect of Different X-ray Energies

To thoroughly analyze the impact of X-ray photon energy on CD-SAXS measurement accuracy, this study performed CD-SAXS assessments on gratings using various X-ray photon energies while maintaining a constant beam size. In CD-SAXS experiments, scientists at NIST recommend conducting tests at energy levels above 17 keV. Higher-energy X-rays facilitate straightforward transmission through the grating sample substrate. However, this simplicity comes at the cost of reduced interaction between X-rays and the grating.

Figure S5 shows the transmission rate of X-rays through a 700 µm silicon substrate at different energy levels. When X-ray photon energy is high, absorption of the grating sample decreases and transmittance increases, resulting in some background scattering passing through the grating and being received by the detector. To perform CD-SAXS measurements using laboratory X-ray sources, the challenge of low intensity must be addressed. Achieving high-quality scattering signals in a short timeframe necessitates improved photon utilization efficiency. Therefore, careful selection of the metal target in the X-ray tube is crucial in designing laboratory X-ray sources. In contrast, synchrotron radiation sources produce X-rays with exceptionally high brightness, resulting in significantly elevated photon flux per unit area. This characteristic allows researchers to acquire substantial information from the detector within a given time period. The utilization of synchrotron radiation sources for CD-SAXS experiments at various X-ray photon energy levels can serve as a reference for choosing metal targets when constructing laboratory X-ray sources.

Reconstructed images from CD-SAXS measurements produce an intensity map as a function of qx and qz, shown in Figure 5 Figure 5: see original paper. The 16 keV test results reveal numerous diffraction peaks, and the peak centers can

provide information about the structure factor of the trapezoidal grating sample. As shown in Figure 5(b), detailed examination of the data is achieved through one-dimensional profile cuts over qx (qz = 0) and over qz (across all peak orders). Lack of clear spacing between diffraction peaks significantly increased mutual interference, and excessively broad peaks reduced resolution, complicating peak position identification. High resolution is crucial in CD-SAXS measurements to accurately determine periodic information. Observation at 16 keV revealed notable broadening of diffraction peaks that caused adjacent peaks to obscure each other, with significant intensity variations. The second-order diffraction peak was heavily influenced by the first-order peak, posing challenges for accurate observation. Furthermore, peak widening considerably decreased the signal-to-noise ratio, creating uncertainty in the relative intensity correlation between signals and noise. Consequently, this decreased the accuracy of fitting results, markedly increasing uncertainty.

The CD-SAXS results obtained at 10 keV and 12 keV are shown in Figure 6 [Figure 6: see original paper]. These results have been normalized to facilitate analysis of the energy effect with various scattering cross-sections on experimental results. Although X-ray transmissivities at 10 keV and 12 keV were lower than at 16 keV, the diffraction peaks were clearly distinct and did not overlap. The clear shapes of diffraction peaks provide advantages in data fitting. Additionally, the diffraction peaks were distinctly separated from background scattering, with significant differences in peak heights and background intensity that make them easy to distinguish. Higher X-ray photon energy leads to increased background scattering, as shown in Figure 6. This decreased contrast can impair the accuracy of peak measurement and analysis. As X-ray energy increases, the absorption capacity of the grating sample decreases while penetration capacity increases, implying that more X-ray photons can pass through the substrate. This is accompanied by corresponding enhancement in background signals that pass through the sample and are received by the detector. Concurrently, the scattering cross-section decreases, resulting in weakening of each scattering event' s intensity, yet an overall enhancement of both diffraction peak signal and background signal occurs, but with decreased signal-to-noise ratio. Figure S5 enables calculation of scattering probability at each energy level, demonstrating that as energy increases, scattering probability also increases. The enhancement of background signal reduces the signal-to-noise ratio of the diffraction pattern, resulting in blurred diffraction peak boundaries and increased width. Analysis of the grating structure becomes more complex due to indistinct positions and peaks of different diffraction orders.

In experimental results at 10 keV and 12 keV, we can readily observe that background scattering remains stable between diffraction peaks, unaffected by the presence of diffraction peaks. This reduces uncertainties introduced by noise during the fitting process. The CD values obtained at three different energies are listed in Table 3 .

TABLE 3. Comparison of CD-SAXS measurements with different

energies.

Parameter	10 keV	12 keV	16 keV
Pitch (nm)	100.8 ± 0.5	100.6 ± 0.3	101.1 ± 1.2
Width (nm)	51.0 ± 0.9	50.1 ± 0.4	50.4 ± 2.4
Height (nm)	131.9 ± 1.7	131.2 ± 1.3	132.4 ± 3.1
Sidewall angle (°)	1.3 ± 0.7	1.2 ± 0.3	1.4 ± 0.4

These results confirm the feasibility of CD-SAXS testing below 17 keV. As X-ray energy increases, grating parameters achieve an optimal solution at 12 keV. The mean values of height, sidewall angle, and pitch at 16 keV exhibit notable discrepancy from expected values, while the mean width value at 10 keV shows considerable discrepancy. Notably, measurement results at 12 keV exhibit the smallest confidence interval. Compared to the other two sets of results, the data at 12 keV are more accurate and relatively stable.

The dependences of CD on X-ray beam size and photon energy are shown in Figure 7 [Figure 7: see original paper]. Clearly, changes in these conditions affect average measurement values, allowing easy observation of confidence intervals for each dataset. By comparing with the grating standard sample dimensions, it is possible to select experimental conditions with high precision and small confidence intervals, providing a basis for determining experimental conditions for future CD-SAXS measurements.

IV. Conclusion

In this work, the critical dimensions of a grating with a half-pitch of 50 nm were investigated using small-angle X-ray scattering with various spot sizes and incidence energies. We proposed an effective fitting method for reconstructing grating structural information and obtained critical dimensions. Optimized CD values were achieved with an X-ray energy of 12 keV and a spot size of 200 µm, with precisions of 0.2 nm for pitch, width, and height, as well as 0.1° for the sidewall angle (SWA). This research demonstrates the significance of beam size and confirms the viability of CD-SAXS experiments within specific energy ranges, laying the foundation for future developments and providing valuable references for laboratory setups. Our study makes a significant contribution to CD-SAXS experimentation and paves the way for further technological advancements.

Despite its advantages, CD-SAXS technology faces several challenges that limit its current applicability. One major limitation is the requirement for a high signal-to-noise ratio (SNR) to accurately infer key dimensions from the data. Achieving high SNR often requires prolonged irradiation times, which can be impractical for routine laboratory use. Additionally, the low flux of laboratory X-ray sources further restricts the efficiency of CD-SAXS measurements. To address these challenges, the Mo X-ray source emerges as a favorable choice

due to its balanced performance characteristics. However, the Liquid-Metal-Jet X-ray source—particularly alloy-rich indium variants—presents a promising alternative, as it enhances flux density by an order of magnitude while achieving smaller spot sizes (100 µm) compared to solid-target sources (500 µm with multilayer-coated Montel mirrors). These advancements directly align with our CD-SAXS measurement accuracy results, where reduced spot size and enhanced flux synergistically improve precision. Future efforts should prioritize optimizing such high-brightness sources alongside algorithmic innovations to streamline data acquisition and analysis, ultimately accelerating the adoption of CD-SAXS in both research and industrial metrology.

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Supporting Information

The Supporting Information shows the structure of the grating sample for CD-SAXS measurement, provides a schematic of the CD-SAXS measurement, and illustrates the effect of different critical dimensions on the shape of the form factor. In addition, the Supporting Information includes graphs of measurement results from other comparative methods. Finally, it shows the scattering cross-section versus transmission curves at different X-ray energies.

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Note: Figure translations are in progress. See original paper for figures.