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Fabrication of 200 nanometer period centimeter area hard x-ray absorption gratings by multilayer deposition

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Abstract

We describe the design and fabrication trials of x-ray absorption gratings of 200 nm period and up to 100:1 depth-to-period ratios for full-field hard x-ray imaging applications. Hard x-ray phase-contrast imaging relies on gratings of ultra-small periods and sufficient depth to achieve high sensitivity. Current grating designs utilize lithographic processes to produce periodic vertical structures, where grating periods below 2.0 μm are difficult due to the extreme aspect ratios of the structures. In our design, multiple bilayers of x-ray transparent and opaque materials are deposited on a staircase substrate, and mostly on the floor surfaces of the steps only. When illuminated by an x-ray beam horizontally, the multilayer stack on each step functions as a micro-grating whose grating period is the thickness of a bilayer. The array of micro-gratings over the length of the staircase works as a single grating over a large area when continuity conditions are met. Since the layers can be nanometers thick and many microns wide, this design allows sub-micron grating periods and sufficient grating depth to modulate hard x-rays. We present the details of the fabrication process and diffraction profiles and contact radiography images showing successful intensity modulation of a 25 keV x-ray beam.

1. Introduction

Grating-based x-ray phase-contrast imaging has advanced rapidly in the past few years with the potential for better image contrast and lower radiation dose over conventional absorption radiography and computed tomography (CT) [1–19]. In a typical x-ray grating interferometer [9, 20], at least one grating that operates in the transmission mode is used to diffract an incident x-ray beam into two or more beams. The beams propagate along different paths through a sample to arrive at a detector plane, where they interfere with each other coherently to produce a fringe pattern. Variations of the refractive index in the sample will lead to a relative difference in the path length or phase delay between the interfering beams, which is detectable as a shift of the fringe pattern. Additionally, random scattering of the beams in the sample reduces their mutual coherence, resulting in decreased amplitudes of the interference fringes. By these effects a grating interferometer is able to detect both x-ray refraction and diffraction in the sample.

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The sensitivity of the interferometer is dependent on the degree of separation between the diffracted beams, which in turn is dependent on the line density of the grating. High line densities (small grating periods) are desirable for achieving high sensitivity. However, since hard x-rays have good penetrating power, the gratings should also be deep enough to substantially modulate the beam, particularly for absorption gratings. New UV and x-ray lithography techniques have been developed to make hard x-ray absorption gratings of 2.0 μm periods[21–23], although for smaller periods, it becomes difficult to maintain the thickness of the gratings due to the extreme aspect ratios of the vertical structures.

Alternatively, a stack of vacuum-deposited thin layers can function as a grating in the horizontal transmission mode [24–26]. The layers can be several nanometers thick and millimeters wide, and therefore as a transmission grating the depth-to-period ratio can be very high. Kim and co-authors successfully produced Talbot fringes of 0.39 μm period using a multilayer stack in a broadband synchrotron beam centered at 10 keV[26]. However, the limited height of a multilayer stack ($< 50 \mu\text{m}$)[27] precludes full-field imaging of large samples. In order to apply the multilayer approach to the fabrication of full-field imaging gratings, we describe a grating design called multilayer grating array (MLGA) which we believe can surmount the above limitations and realize sub-micron period hard x-ray gratings over large areas[28]. We describe the fabrication of absorption gratings of 200 nm period, 30 to 100 depth-to-period ratios (60 to 200 aspect ratios), and 20 mm $\times$ 20 mm area. We present contact radiography images of the transmitted x-ray beam through these gratings at 17.5 keV and 25 keV.

The MLGA design is shown in figure 1. It consists of a staircase substrate, on which a number of alternating layers of x-ray transparent (silicon) and x-ray opaque (tungsten) materials are deposited. Each step of the staircase holds a multilayer stack. When illuminated horizontally by an x-ray beam, each step works as a micro-grating. The entire staircase is an array of micro-gratings that covers a large area. Continuity between adjacent micro-gratings can be achieved if the bilayer thickness P is $H/2N$, where H is the height of each step, and N is the number of bilayers in the stack. A key point to note is that the bilayer thickness is the period of the grating.

Since the angle of the x-ray beam relative to the overall grating plane is oblique, such gratings are designed to work in oblique-incidence interferometers as illustrated in figure 2. We showed previously that they are equivalent to normal-incidence grating interferometers[29, 30]. In the following sections we describe the fabrication steps, including UV lithography of the silicon substrate, magnetron sputter coating of the multilayers, and cross-section and contact radiography examinations of the gratings.

2. Material and Methods

2.1 Fabrication of the Staircase Substrate

Referring to figure 3, the staircase substrate is made from an off-cut silicon wafer by anisotropic wet-etching[31–34]. The design took into consideration several requirements. First is that the width of the steps should be sufficient to modulate the x-ray beam. In the example of an absorption grating of W/Si multilayers for 17.5 keV x-rays, the step width should be greater than the penetration depth in tungsten, which is 5.7 μm . Secondly, the blaze angle of the steps (figure 3) should not be too large to avoid excessive deposition on the side wall of the steps, and should not be too small since the effective vertical size of the grating is $L \sin(\theta)$, where L is the length of the staircase and θ is the blaze angle. Too small a blaze angle will result in a small effective grating area. After several multilayer coating trials, we settled on a 28° blaze angle for the substrates.

The fabrication work flow is shown in figure 4. The wafers were deposited with a low stress nitride layer of 30 nm, which was used as the masking layer in the two subsequent KOH etches. Alignment features were patterned into the perimeter of the wafers using contact lithography, dry etched into the nitride layer using tetrafluoromethane and oxygen gas, then KOH etched in a 30% KOH solution at 90°C for 20 minutes to expose the <111> crystal planes. The angle of deviation between the crystal plane and the nominal <111> side of the alignment mask window was measured with a microscope (figure 4). This angle was the error of mask alignment to the crystal plane. It was then corrected for in the second photo lithography step, where the staircase pattern was etched into the wafer. The alignment step proved necessary, because alignment using the machined flat on the wafer was inaccurate by as much as 1.5°.

In the second lithography step, periodic bars were patterned onto the wafers, etched into the nitride layer, and then KOH etched at 80°C (figure 4). It proved to be difficult to precisely time the under-etch of the plateaus under the bars due to residual alignment errors (~ 0.1°) and fluctuations in the KOH solution temperature and activity. For example, the 34.6 µm step substrate had a KOH etch time range of 27–38 minutes, while the 10 µm step substrate etch time was about 6–8 minutes. In order to monitor the progression of the under-etch to realize plateau widths of less than 1.0 µm, test etch features of smaller dimensions were added to the mask. The main grating area nitride bars of the 34.6 µm step substrate were 5 µm with test feature bars of 3 and 4 µm (figure 5); the 10 µm step substrate bars were 1.5 µm with 1.0 and 1.3 µm test feature bar widths. These features were large enough that they could be macroscopically viewed during the KOH etch; a change in the reflectivity denoted when the nitride bars had been completely under-etched, conveying the progression of the under-etch in the main grating region.

Finally, the smoothness of the <111> surface was affected by the purity of the KOH solution. Light microscopy images revealed the surface roughness indicative of KOH impurities due to lack of contamination control in a multi-user facility (figure 6). A specialized KOH bath was procured, and wafers were etched in fresh KOH solution after etching a clean Si wafer to lower the fast initial etch rate. This precaution of using a specialized bath resolved surface roughness issues.

2.2 Magnetron Sputtering Deposition of the Multilayers

In an absorption grating, the multilayer stack consists of alternating layers of x-ray transparent and x-ray absorbing material. We chose the material combination of silicon and tungsten in our trials. Referring to figure 1, the ideal multilayer structure should have uniform layer thickness across the height of the stack and over the grating area. Additionally, good directionality of deposition is desired so that coating on the side walls of the steps is minimized. We used both the 10 µm and 34.6 µm step substrates for multilayer deposition. Our target grating period was 200 nm. Accordingly, the target multilayer parameters for the 10 µm step substrate were 46 alternating W/Si layers of 102.0 nm individual layer thickness to fill the step height of 4.69 µm (figure 3); the target parameters for the 34.6 µm step substrate were 162 alternating W/Si layers of 100.2 nm individual layer thickness to fill the step height of 16.24 µm.

Multilayer deposition by DC magnetron sputtering was performed at the Advance Photon Source of Argonne National Laboratory. The deposition system consists of four large vacuum chambers in series, each 16 inches in diameter and 66 inches long. Three CTI model CT-8 cryo pumps and an Alcatel ADP 81 dry pump provide a base pressure of about 10⁻⁸ Torr for the system. Four 3-inch-diameter magnetron sputter guns are deployed facing up in four wells 15" apart on the bottom of the deposition chamber. Samples were placed facing down on a sample holder and loaded into a carrier, which can be moved from chamber to

chamber by a computer-controlled transport system. The distance from the top of the sputter target to the sample holder is about 12 cm. During the deposition, the substrates were moving linearly at constant speeds. The coating uniformity along the direction perpendicular to the moving direction was improved through a contoured mask over the sputter gun (figure 7). The depositions were carried out at ambient temperatures and at an Ar pressure of 2.3 mTorr. The sputter guns were operated at a constant power of 215 W. They were programmed to turn on 4 sec before the substrate was moved over and to turn off after a desired thickness was deposited. Multilayer coatings were achieved by alternatively passing the substrates back and forth over sputter targets of W and Si. The thickness of each layer was controlled by the translation speeds and the number of loops over the gun. The coating parameters for each layer were calculated according to its thickness requirement, test results of test runs, and the growth rate corrections due to target erosions. Test samples of W and Si thin films were grown and measured using a surface profiler before multilayer growth.

It is known that W/Si multilayers have a tendency to peel off from a Si substrate[25]. Care has been taken to break a long coating process into many shorter ones while maintaining vacuum in the chamber, so that the sample was able to cool without accumulating too much thermal stress.

To improve the directionality of coating, the substrates were mounted on a tilted stage with a side-shielding chimney, such that the deposition vapor was collimated, and the areas presented by the side walls towards the sputter target were reduced (figure 7). We tested two tilt angles in the two substrates, which were 9° in the 10 μm step substrate and 15° in the 34.6 μm step substrate. A trade-off for these measures was reduced deposition rate. Indeed, the total run time, including cooling breaks, for the 10 μm step substrate was 16 hours, and for the 34.6 μm step substrate was 48 hours.

After the multilayers were deposited, a protective epoxy layer of SPR 220–7.0 several microns in thickness was spun onto the surface. A small strip of the grating was then diced for cross-section examination using scanning electron microscopy (SEM). The procedure for the sample preparation follows the ones developed for x-ray multilayer zone plates[35]. The front (multilayer coated) side of the small strip was bonded to a cover wafer using epoxy, then diced along the length of the staircase to expose a cross-section surface. The surface was polished with chemical-mechanical polishing and refined with ion beam ablation to reveal the multilayer structure. Under SEM the multilayer structures were inspected with respect to side-wall deposition and other extra accumulation of materials beyond the ideal configuration. The layer thicknesses were measured at the bottom, middle, and top of the multilayer stack at different locations over the length of the grating. The measured values were then compared with the target values.

2.3 Contact Radiography of the Multilayer Gratings

To observe the transmitted x-ray beam through the gratings, we performed high resolution contact radiography at the 2-BM beam line of Advanced Photon Source, Argonne National Laboratory (Chicago, Illinois, US). The contact radiography was based on the method developed by Spiller and co-authors for x-ray microscopy[26, 36] with one modification. We coated <100> silicon wafers with the electron beam resist ZEP520A (Zeon Corporation) to be used as x-ray film plates. The thickness of the resist layer was 250 nm. The gratings were packaged with the plates with their front side facing the resist. The packages were positioned in the x-ray beam at 28° incidence angle. The x-ray beam transmitted through the gratings before encountering the resist. The exposure times were several minutes. The exposed plates were developed in 40°C xylene for one minute and rinsed with isopropyl alcohol. Our modification of the method is that instead of SEM, we used atomic force microscopy (AFM) to image the height profile of the resist layer. The ZEP520A is a positive

tone resist, so higher x-ray intensity resulted in more resist being removed in the developing process and lower remaining height. The height profile is therefore an imprint of the transmitted x-ray intensity.

For radiography of the 10 μm step sized grating we used a beam energy of 17.5 keV. For the 34.6 μm step sized grating we used 25 keV beam energy.

2.4 Slit Diffraction Study

A slit diffraction study was performed on the 34.6 μm step sized grating at 25.17 keV on the beam line. In order to see how a parallel beam is diffracted by the grating, a horizontal tungsten slit of 60 μm width was placed in the beam in front of the grating, and the diffraction pattern was recorded by an x-ray camera at 600 mm distance from the grating. The pixel size of the camera was 1.5 μm . Based on the profile of the diffraction pattern, the percentage of energy that was diffracted into the ± 1 and higher orders were calculated. The result was compared with an ideal absorption grating of a square wave absorption profile.

3. Results

3.1 Cross-Section Evaluation of the Multilayer Gratings

Figure 8a shows the cross-section of the grating which was fabricated on the 10 μm step substrate. The tilt angle of the substrate during multilayer coating was 9° . We see the desired stack of alternating layers in the middle of the multilayer structure, but with substantial deposition on the side wall of the substrate step. There is also a tapered section of the multilayers towards the inner edge of the step, and conformal rounding of the multilayers at the ledge of the step. Additionally, as illustrated in figure 8a, the flat portions of the top layers are slightly tilted relative to the bottom layer by 1.1° . Because of the gradual tilting of the layers, the extrapolated planes of the layers will converge at a distance of approximately 0.24 mm from the stack.

The thicknesses of the individual tungsten and silicon layers as well W/Si bilayers were measured at various locations throughout the cross-section. The bilayer thickness is effectively the period of the grating and therefore of the most interest. The measured values are plotted in figure 8b. Generally, the bilayers were thinner than the target thickness of 204 nm, with an average value of 179.6 nm which was 12% below target. This resulted in a visible mismatch between the top of one micro-grating and the bottom of the adjacent micro-grating in figure 8a.

In comparison, the grating which was fabricated on the 34.6 μm step substrate had less material deposition on the side walls of the steps (figure 9a). For this grating the substrate was tilted 15° during sputter coating, which was larger than the 9° tilt of the previous grating. In the resulting multilayer structure, the tapered and the rounded sections at the ends still exist. However, the flat portions of all layers are parallel to each other as can be seen in figure 9a. The measured bilayer thicknesses are graphed in figure 9b for various locations along the staircase and within each micro-grating stack. The average bilayer thickness throughout the grating was 191.9 nm, which was 4% below the target value of 200.4 nm. The maximum bilayer thickness was found at the bottom of the stack in the middle of the stair case; the minimum at the top of the stack at the top of the staircase. The measured bilayer thicknesses ranged between 177.5 nm and 208.2 nm.

3.2 Contact Radiography of the Multilayer Gratings

The AFM image of the resist layer which was exposed with the 10 μm step grating is shown in figure 10a, and the height profile of the resist layer across the multilayer stack is graphed

in figure 10b. The periodic oscillation of the x-ray intensity is visible in the bottom half of each multilayer stack, but diminishes towards the top of the stack. The fringe period is approximately 200 nm, although the resolution of the image was primarily determined by the grainy appearance of the surface of the resist. The gradual loss of the fringe contrast towards the top of the stack is consistent with the gradual tilt of the layers relative to the floor surface of the substrate, which was seen in the cross-section SEM images and described in the previous section. This resulted in a gradual misalignment of the layer planes with the incident beam. Additionally, the gap seen between adjacent multi-layer stacks is consistent with the fact that the measured thicknesses of the layers were below the target value by 12% on average.

The AFM image and height profile of the resist layer which was exposed with the 34.6 μm step grating are shown in figure 11. Here the periodic fringes can be seen throughout the height of each multilayer stack. The AFM image was taken at the mid section of the staircase. Cross-sectional measurements showed that at this location, the height of the multilayer stack matches the height of the substrate step (figure 9b). Therefore, there was no apparent gap at the boundary between two adjacent stacks in the image such as the one seen in the previous grating. However, there was an oscillation of the height profile at the boundary that likely came from edge diffraction at the top surface of each stack.

3.3 Slit Diffraction Study

The slit diffraction pattern from the 34.6 μm step sized grating at 25.17 keV is shown in figure 12. The central 0th diffraction order and a number of side lobes can be seen. Based on the intensity profile of the diffraction pattern, the percentage of energy that was diffracted into the ± 1 and higher diffraction orders was 69%. For comparison, a perfect absorption grating of square wave transmission function should diffract 50% of the beam energy into the side lobes, while a perfect π phase shift grating of a square wave phase shift function and no absorption diffracts all of the beam energy into the side lobes. The fact that the measured percentage of diffracted energy is above the ideal absorption grating indicates that the tungsten layers in the multilayer stacks were not perfect absorbers, but rather acted as a combination of absorbers and phase shifters. The asymmetry of the diffraction pattern is likely the result of the imperfections in the structure of the multilayer stack, including the tapered and rounded ends of the stack.

4. Conclusion and Future Development

Multilayer deposition is an attractive option for the fabrication of x-ray gratings due to the possibility of sub-micron grating periods and virtually unlimited depth-to-period ratios. However, a significant obstacle for full-field imaging has been the limited height of a multilayer stack that can be deposited in practical terms. Here we show that by magnetron sputter deposition on a staircase substrate, we can fabricate an array of thousands of micro-gratings to cover a large area in a single deposition run. Each micro-grating is microns tall and has a grating period of approximately 200 nm and depth-to-period ratio of 100:1, which is substantially smaller than the 2.0 μm period that has been achieved with lithographic techniques. Even with the imperfections in the layer structure, contact radiography showed that intensity modulation was successfully achieved at 200 nm period for 25 keV x-rays. In the slit diffraction pattern 69% of the beam energy was diffracted into the ± 1 and higher diffraction orders. We found that an important parameter in determining the multilayer structure is the tilt angle of the substrate in the deposition chamber. For silicon $\langle 111 \rangle$ substrates of 28° degree blaze angle, a 15° tilt worked better than 9° in terms of better uniformity of layer thickness across the width of each step of the substrate and less side wall deposition.

The results also point to several areas that require further development. The uniformity and accuracy of the thickness of individual layers are important for the array of micro-gratings to work as a single continuous grating. For this purpose we need to accumulate sufficient deposition data on a given MLGA substrate, and perhaps employ in situ measurements of the layer thicknesses[37]. The tapered and rounded sections of the multilayer structure should be minimized. This may require further lithographic steps that specifically remove these sections, or a more directional deposition technique. Reducing the plateaus at the edge of the substrate steps may also help reduce the rounded ends of the multilayers. This may require steps to dry etch the plateaus using a third round of lithography[38].

Additionally, because of the oblique incidence angle of the x-ray beam, the effective thickness of the substrate that the beam has to penetrate is over twice the actual thickness. To reduce attenuation in the substrate, it may be desirable to thin out the backside of the substrate wafer, especially at lower x-ray energies. Lastly, the edge diffraction effects seen at the top surface of the multilayer stacks should be minimized (figure 11). This may be accomplished by choosing a suitable material for the top cover layer (figure 1) that matches the index of refraction of the silicon substrate, for example, silicon dioxide.

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Figure 1.

Idealized multilayer grating consisting of an array of micro-gratings. The multilayers are deposited on a staircase substrate in a single deposition run. The stack on each step fills the height of the step precisely. When an x-ray beam transmits horizontally through the stacks, they function as micro-gratings with period equal to the bilayer thickness. A protective polymer coating is applied over the multilayers. The bottom and top of the staircase are marked.

Figure 2.

Oblique-incidence x-ray interferometer suitable for multilayer gratings. In this three-grating interferometer, the three multilayer gratings are arranged in parallel, tilted planes and equidistant from each other. The source grating G_0 and analyzer grating G_2 are absorption gratings (e.g. W/Si multilayer grating), and the middle G_1 is a phase grating (e.g. Ni/Si multilayer grating). The diffraction beams that coherently interfere with each other are represented by the red lines. Such oblique-incidence interferometers have their equivalent normal incidence forms as explained in the text. The imaged object can be placed either in front of or behind the G_1 grating as in normal incidence setups.

Figure 3.

SEM image of a staircase substrate. The Si staircase of step size $34.6\ \mu\text{m}$ and blaze angle 28° was fabricated by KOH etching a $\langle 111 \rangle$ wafer cut 28° towards $\langle 001 \rangle$; the plateau refers to the residual flat area at the tip of each step. The exposed $\langle 111 \rangle$ planes are the floor surface and side wall of the steps. The multilayers will be deposited on the wider floor surface.

Figure 4.

Alignment and staircase substrate fabrication. (a) A rectangular alignment feature (darker gray) was aligned to the wafer flat in the first lithography step, then KOH etched to a depth of 50–70 μm to expose the $\langle 111 \rangle$ planes (paler gray hexagon). (b) The grating bars of the mask were aligned to the etched $\langle 111 \rangle$ plane on the wafer and patterned onto the wafer in the second contact photo lithography step. (c) The bars were dry etched into the nitride masking layer, then (d) KOH etched to yield the staircase grating. Finally, (e) the nitride strips were removed from the plateaus using Transetch-N (H_3PO_4) etchant, leaving the bare Si staircase substrate ready for multilayer deposition.

Figure 5.

View of test features for under-etch control. This image of a wafer diced into four samples demonstrates the change in reflectivity in the test grating features outlined by the rectangle (from left, features #1 and #3 have $3\text{ }\mu\text{m}$ nitride bars, #2 and #4 have $4\text{ }\mu\text{m}$ nitride bars). When the nitride bars in a test feature were completely under-etched, the feature would appear more reflective. Monitoring of the under-etch of the test features indicated the under-etch progression in main grating areas.

Figure 6.

Comparison of rough and smooth substrate surfaces. (a) A dark-field light microscopy image of a 34.6 μm step size staircase substrate shows rough $\langle 111 \rangle$ surfaces due to impurities in the KOH bath. (b) A substrate etched in fresh KOH solution shows smooth $\langle 111 \rangle$ surfaces.

Figure 7. Schematics of the magnetron sputter multilayer deposition setup. The side-shielding chimney and the tilting of the substrate together reduce coating on the side walls of the steps of the substrate. The substrate and the attached chimney move over the contoured mask at a constant speed to help improve uniformity of coating on the substrate.

Figure 8.

Cross-section evaluation of the 10 μm step size multilayer grating which was sputter coated at 9° substrate tilt angle. (a) SEM image of polished cross-section shows substantial deposition on the side wall of the substrate step, tapering of the multilayers towards the inner edge of the step, and conformal rounding of the multilayers around the plateau. The yellow lines are parallel to the floor surface of the step and serve as visual guides to illustrate how the layers gradually tilt upwards toward the top of the multilayer stack. The height of the stack is less than the step height of the substrate. The peeling appearance of the few layers at the top is a polishing artifact. (b) The graph presents the measured W/Si bilayer thicknesses, i.e. the period of the grating, at the bottom, middle, and top of each step stack across the entire 20 mm staircase grating (see figure 1 for naming convention). The average thickness of the bilayers was 179.6 nm, 12% below the target of 204 nm.

Figure 9.

Cross-section evaluation of the 34.6 μm step size multilayer grating which was sputter coated at 15° substrate tilt angle. (a) SEM image of polished cross-section shows less side wall deposition than the 10 μm step grating (figure 8a). The yellow lines are parallel to the floor surface of the step. Using them as reference it can be seen that the flat portions of the layers remain parallel throughout the height of the multilayer stack. The irregular appearance of the top few layers is a polishing artifact. (b) The graph presents the measured W/Si bilayer thicknesses at the bottom, middle, and top of each step stack across the length of the 20 mm staircase grating (see figure 1 for naming convention). The average bilayer thickness was 191.9 nm which was 4% below the target thickness of 200.4 nm, although the thicknesses in the mid-section of the staircase reached the target value.

Figure 10.

Contact radiography of the 10 μm step multilayer grating. (a) The AFM scan of e-beam resist layer exposed with the 10 μm step grating in a 17.5 keV beam shows visible fringes in the bottom half of each multilayer stack with gradual loss of fringe contrast and gaps between stacks. (b) The height profile of the resist demonstrates the periodic oscillations of the x-ray intensity with a fringe period of approximately 200 nm. The gradual tilt of the multilayers and incomplete filling of the steps (figure 8a) caused the loss in fringe contrast and gap at the top of each stack, respectively.

Figure 11.

Contact radiography of the 34.6 μm step multilayer grating. (a) The AFM image of e-beam resist layer exposed with the 34.6 μm step grating in a 25 keV beam shows visible fringes throughout the height of the multilayer stack. (b) The height profile of the resist demonstrates uniform modulation of the x-ray intensity at the period of 200 nm, with an additional oscillation at the boundary of the step that likely came from edge diffraction at the top of the multilayer stack.

Figure 12.

Slit diffraction pattern by the $34.6\ \mu\text{m}$ step multilayer grating at $25.17\ \text{keV}$. A $60\ \mu\text{m}$ slit was placed in the parallel beam in front of the grating, and an image of the diffracted beam was taken at $600\ \text{mm}$ distance down stream from the grating. The image is shown in the inset, and the diffraction profile obtained from the image is plotted. The percentage of energy that was diffracted into the side lobes was 69% .