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PRECISE ROTATIONAL ALIGNMENT OF
X-RAY TRANSMISSION DIFFRACTION GRATINGS

Sherry L. Hill

March 28, 1988

Lawrence
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Work performed under the auspices of the U.S. Department of Energy by the Lawrence Livermore Laboratory under Contract W-7405-Eng-48.

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PRECISE ROTATIONAL ALIGNMENT OF
X-RAY TRANSMISSION DIFFRACTION GRATINGS

Sherry L. Hill

Lawrence Livermore National Laboratory
University of California
Livermore, CA 94550

ABSTRACT

Gold transmission diffraction gratings used for x-ray spectroscopy must sometimes be rotationally aligned to the axis of a diagnostic instrument to within sub-milliradian accuracy. We have fabricated transmission diffraction gratings with high line-densities (grating periods of 200 and 300 nm) using UV holographic and x-ray lithography. Since the submicron features of the gratings are not optically visible, precision alignment is time consuming and difficult to verify in situ. We have developed a technique to write an optically visible alignment pattern onto these gratings using a scanning electron microscope. (SEM). At high magnification (15000 X) several submicron lines of the grating are observable in the SEM, making it possible to write an alignment pattern parallel to the grating lines in an electron-beam-sensitive coating that overlays the grating. We create an alignment pattern by

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following a 1-cm-long grating line using the SEM's joystick-controlled translation stage. By following the same grating line we are assured the traveled direction of the SEM electron beam is parallel to the grating to better than 10 μ radian. The electron-beam-exposed line-width can be large (5 to 15 μ m wide) depending on the SEM magnification, and is therefore optically visible. The exposed pattern is eventually made a permanent feature of the grating by ion beam etching or gold electroplating. The pattern can be used to accurately align the grating to the axis of a diagnostic instrument. More importantly, the alignment of the grating can be quickly verified in situ.

INTRODUCTION

With the development of advanced submicron fabrication processes we can now produce gold transmission diffraction gratings with submicron periods (200 nm and 300 nm periods) for use in x-ray spectroscopy. We have produced two types of gratings: one type is fabricated on a thin support membrane, for x-ray lithography applications; the other is freestanding with a support grid, for spectroscopy of x-rays from laser produced plasmas.¹ Under certain experimental conditions, we must rotationally align the grating to the axis of a diagnostic instrument with great accuracy. Of primary concern is the absolute determination of the direction of the grating lines. We are not able to use visible diffraction techniques or optically image the grating lines because they are below the resolution limit of visible light. Previously, a polarization technique² had been used to determine the grating orientation; however, the results are limited to milliradian accuracy and

must be performed on a laboratory bench. This makes final in situ alignment verification difficult if not impossible. We report here on a new alignment procedure that determines grating direction to a few microradian accuracy and allows us to verify rotational alignment using optical imaging techniques.

PROCEDURE

In LEAD program's laboratory for Submicron Structure Technology, we have fabricated thick gold transmission diffraction gratings with submicron periods. The fabrication of submicron period gratings^{3,4} utilized several state-of-the-art processes including x-ray lithography, for fabrication of freestanding thick gold gratings (Appendix A), and holography lithography, for fabrication of x-ray lithography masks (Appendix B). After grating fabrication, we incorporated our alignment technique, which required overcoating the gold grating with a high-resolution electron-beam-sensitive material (Appendix C), polymethylmethacrylate (PMMA). We used the electron beam of a scanning electron microscope (SEM) to "write" a pattern in the PMMA overcoating which made it possible to record the position of the electron beam relative to the grating. The PMMA coating must be thin (≤ 250 nm) in order for the SEM to image the underlying gold grating. We were not able to image through layers of PMMA greater than 500 nm because of a decrease in resolution resulting from the lateral spreading of the electron beam due to scattering.⁵

Our application required modifications to the SEM; a joystick-controlled-motorized stage was added to a Bausch and Lomb Nanolab LE2100 SEM, Fig. 1. The stepper motors were configured to allow minimum motion of approximately 32 nm per step in the X and Y directions. The stage translation speed was determined by the stepper motor controller, programmed to rotate at various frequencies (steps per second). The direction of travel was controlled by the joystick allowing the operator to move the stage along the direction of a single grating line. By using minimal motion, we were able to follow a single grating line without deviating more than one-half a line width (50 nm) over the length of the line (≥ 1 cm). This resulted in an alignment pattern (a line) in the PMMA overcoat written parallel to the gold grating to within ± 5 μ rad. Several grating lines were displayed on the CRT screen at any time, and an electronic cross-hair on the screen aided the operator in identifying and following one particular grating line (Fig. 1, top). The width of the electron-beam-exposed alignment line recorded in the PMMA was directly proportional to the SEM magnification used. The greater the magnification, the smaller the area scanned by the electron beam. By operating at approximately 15000 X, we were able to produce an alignment line 5 μ m to 15 μ m wide. The width of the exposed line was also dependent upon the operating voltage (kV) and length of exposure time (translation stage speed). We typically operated at 15 kV and translation stage speeds ranged from 0.05 to 0.25 mm/min. Even though the width of the SEM written line varied, the center of the line remained accurately aligned to the grating. After electron-beam exposure, the alignment pattern was developed for one minute with a mixture of 50%

methyl-isobutyl-ketone (MIBK) and 50% isopropyl-alcohol. After development, the alignment pattern (Fig. 2A) was made a permanent feature of the grating by ion-milling (Appendix D) or gold-electroplating, using the PMMA pattern as a mask. Afterwards, the PMMA was removed by soaking in chlorobenzene, followed by exposure to UV ozone for final cleaning. Under certain conditions, the electron beam overexposed a line in the PMMA causing it to act as a negative resist and therefore, insoluble in the developer. The primary cause of over exposure was a decrease in translation stage speed which allowed the electron beam to dwell in one area too long. However, the areas on either side of the overexposed line were exposed to backscattered electrons only and having received less exposure, were able to develop normally (Fig. 2B). This resulted in two parallel lines (Fig. 3), which served equally as well as the single line for subsequent processing steps and eventual alignment.

Additional processing of the freestanding gratings required alignment and fabrication of a support grid relative to the SEM written line. This was accomplished by spinning 2.0 μm of photoresist onto the grating and exposing a grid pattern (0.01-mm-period grating with 0.003-mm-wide gold bars orthogonal to a 0.25-mm period grating) with the use of a mask aligner. The SEM written line was clearly visible in the mask aligner. Once the mask was aligned parallel to the SEM written line, the photoresist was exposed and developed. The grid pattern was then gold-electroplated creating a permanent support grid with gold bars running parallel and perpendicular to the submicron period grating (Fig. 4). Any one of the gold bars of the support grid can now be used

for alignment purposes. The final processing step included etching away the silicon wafer leaving a freestanding grating supported by gold grid bars and a silicon support ring (Appendix A).

CONCLUSIONS

Using a modified scanning electron microscope we are able to write an optically visible alignment pattern parallel to a submicron period grating to within ± 5 μ radians. By implementing this technique, we are able to rotationally align the submicron period grating to the axis of a diagnostic instrument with 10 to 100 times the accuracy previously achieved. This permanent alignment pattern is currently being used for initial grating alignment as well as in situ alignment verification.

ACKNOWLEDGEMENTS

I would like to thank N. M. Ceglie and A. M. Hawryluk for their encouragement, support and patience in this work, and D. Carlo, G. L. Howe and H. E. Petersen for their expert technical assistance.

Appendix A

OVERVIEW OF THICK GOLD GRATING FABRICATION PROCEDURES

1. Evaporate 10 nm of Cr and 10 nm of Au onto clean two-inch silicon wafers using a vacuum deposition system at a pressure of 2×10^{-7} Torr.
2. UV ozone samples five minutes each just prior to spinning a filtered 4% PMMA solution. (Filter pore size = $0.2 \mu\text{m}$). A spin speed of ~3000 rpm for 60 sec results in a PMMA thickness of ~350 - 400 nm. Bake at 180 C for 30 min in flowing nitrogen. (Thicker PMMA layers can be spun for thicker gold gratings).
3. After choosing an appropriate mask, the grating pattern is exposed into the PMMA using x-ray lithography.³

X-ray lithography parameters: Chamber base pressure = 1.0×10^{-6} Torr

Source to sample distance = 8 cm

Gun voltage = 5.0 keV

Gun current = 60 ma

Filament current = 30 amps

Anode power = 300 W

Mask contact voltage = 30 V

Mask contact capacitance = 4 nf

Exposure time = 11 hrs.

The samples are developed using 50% MIKB (methyl-isobutyl-ketone) and 50% isopropyl alcohol until the monitor areas are fully developed.

4. Sel-Rex BDT 510 gold electroplating solution is used to deposit gold between the resist lines. A room temperature bath at a pH of ~8 is used. The anode to cathode spacing is ~2.5 cm, and a bias voltage of 0.8 V. A 1.0 ma current produces a plating rate of ~6.0 nm per min. Deposit ~ 250 - 350 nm gold between the resist lines.
(Thicker gold can be plated if a thicker layer of PMMA was used. As a rule, use a PMMA layer ~ 100 nm more than the final gold thickness).
5. Remove the PMMA with a 10 min soak in chlorobenzene followed by a five minute exposure to UV zone.
For a case in which the x-ray lithography mask did not already have an alignment line steps #6-10 will create a permanent alignment pattern on the gold grating.
6. Spin ~250 nm PMMA onto the samples for the SEM alignment line. A 4% PMMA solution is spun at 4.5 K rpm for 60 sec and baked at 180°C for 30 min in flowing nitrogen. (See footnote #1)
7. An alignment pattern ~1 cm long is written in the PMMA using an SEM.
SEM parameters: kV = 15
Mag. = 15000 X
Working Dist. = 7.2 mm
Stage controller: x divisor = 50,000
y divisor = 10,000
8. The alignment pattern exposed in PMMA is developed using 50% MIBK and 50% isopropyl alcohol for ~1 min.

9. The gold electroplate process is used to deposit gold into the developed alignment pattern. Ten to fifteen minutes of electroplating at 0.8 V is sufficient time to deposit a permanent alignment pattern.
10. The PMMA is removed by soaking the sample in chlorobenzene for 10 min followed by a 5 min exposure to UV ozone.
11. Spin $\sim 2.0 \mu\text{m}$ photoresist for fabrication of the support structure. AZ Products 4210 photoresist is spun at $\sim 4 \text{ K rpm}$ for 30 sec and baked at 90°C for 30 min.
12. After choosing the appropriate mask pattern, a mask aligner is used to align the SEM written alignment pattern to the parallel lines on the mask. After a 35 sec exposure, the photoresist is developed in AZ 400 K developer 1:4 with water for 1 min.
13. Samples are examined in the SEM for alignment verification.
14. The support pattern is gold electroplated to produce a permanent support grid $\sim 1.5 - 2.0 \mu\text{m}$ thick which is precisely aligned to the underlying submicron period grating. Remove the photoresist with acetone.
15. Black wax is painted on all areas of the sample that are not to be etched, including; the entire front surface as well as a circular ring surrounding the grating on the back side which will act as a support ring.
16. The remaining silicon is etched in a solution of 97% HF and 3% HNO_3 acids to obtain a gold transmission grating on a silicon support ring.
17. After etching in acid for $\sim 20 \text{ min}$, the samples are rinsed in DI water followed by isopropyl alcohol.

Footnote #1: In subsequent experiments, we found a PMMA thickness of 150 nm easier to image through and equally functional. A 2% solution of PMMA in chlorobenzene was spun at 2.4 K rpm for 60 sec and baked at 180°C for 30 min in flowing nitrogen.

Appendix B

OVERVIEW OF X-RAY LITHOGRAPHY MASK FABRICATION

1. Spin $\sim 1.25 \mu\text{m}$ polyimide onto clean two-inch silicon wafers.
Pyralin 2545 polyimide produced by DuPont is spun at 3.5 K rpm for 120 sec. Bake at 150°C for 15 min with nitrogen and vacuum, increase to 250°C for 1 hour, increase to 300°C for 2 hours without nitrogen and vacuum.
2. UV ozone samples for 3 min just prior to evaporating 10 nm Cr and 120 nm Au.
3. UV ozone 3 min just prior to spinning ~ 70 nm photoresist (AZ Products 4007 photoresist). Spin at 4 K rpm for 60 sec and bake at 90°C for 30 min.
4. A 300 nm period grating pattern is exposed in the photoresist using holographic lithography.³ Develop with AZ 400 K developer 1:4 with DI water for 1 min.
5. Create a monitor area by removing a small section of unexposed photoresist from the edge of the wafer with a cotton-tipped swab soaked in acetone.
6. Transfer the pattern into the underlying gold by ion milling (Appendix D). The photoresist serves as a mask while energetic Ar ions sputter etch the gold until the gold in the monitor area is removed.
7. Remove the photoresist with acetone and spin PMMA for the SEM alignment technique. Use the procedure outlined in Appendix A, procedures #6-8.

8. Transfer the SEM alignment pattern into the gold by ion milling.
Remove the PMMA by soaking in chlorobenzene for five minutes and exposing to UV ozone for three minutes.
9. Spin ~250 nm Pyralin 2545 polyimide 1:1 with thinner. Spin at 4 K rpm for 60 sec and bake for three hours gradually increasing from room temperature to 250°C with nitrogen and vacuum.
10. UV ozone for three minute just prior to spinning ~1.0 μm AZ 4110 photoresist. Spin at 4 K rpm for 30 sec and bake at 90°C for 30 min.
11. Expose and develop a 50 μm grid pattern.
12. Transfer the grid pattern into the polyimide ~50 nm deep by etching in an oxygen plasma asher at 100 W for ~5 min.
13. Remove the resist with acetone and UV ozone for three minutes.
14. Bond a 1-3/4 inch diameter PVC tube to the face of the sample with five min. epoxy.
15. Black wax all silicon areas not to be etched along the edge of the wafer and overlapping 1/4 inch onto the PVC tube to prevent acid from leaking into the tube.
16. The remaining silicon wafer is etched in a solution of 97% HF and 3% HNO_3 acids for ~20 min until the silicon is etched leaving a gold grating supported by a polyimide membrane.
17. Carefully rinse in DI water and blow dry with nitrogen.
18. A vespel (see footnote #1) ring, 2-1/2 cm OD, 2 cm ID is bonded to the polyimide membrane with 24 hour curing epoxy (BI-Pak, Trabond, BA-2115).

19. The vespel ring is removed from the PVC tube by cutting through the excess membrane outside the ring with a knife.
20. Place a drop of 24 hour epoxy on the inside edge of the vespel ring and evaporate ~50 nm Al onto the membrane to establish electrical continuity from the membrane to the ring.

Footnote #1: Vespel is the trade name of a plastic produce by DuPont.

Appendix C

SPINNING OF PMMA

The alignment technique required overcoating the gold grating with a high-resolution electron-beam-sensitive material, polymethylmethacrylate (PMMA). Prior to spinning the PMMA, the samples were cleaned by submerging in the freon PCA, acetone and methyl alcohol for five minutes each with ultrasonic agitation. The final cleaning step was a three minute exposure to UV ozone. The electron-beam-sensitive material PMMA was a 4% solution in chlorobenzene produced by KTI Chemicals Incorporated. A 0.2 μm filter was used for dispensing the PMMA to prevent particle contamination. To achieve a PMMA thickness of ~ 250 nm the samples were spun at ~ 4.5 K rpm for 60 sec. The samples were baked at 180°C for 30 min in flowing nitrogen.

Appendix D

ION MILLING

Ion milling is one method used to transfer the SEM alignment pattern into the gold grating. The PMMA served as a mask allowing the exposed gold to be sputter etched by energetic argon ions.

Ion mill parameters were as follows:

Source gas	Argon
Chamber pressure	2×10^{-4} Torr
Filament current	5.25 amps
Beam current	75 ma
Beam voltage	500 volts
Acceleration current	15 ma
Acceleration voltage	250 volts
Emission current	125 ma
Filament current	2.88 amps
Time etched	3 min
Cycle shutter	10 sec on/30 sec off

FIGURE CAPTIONS

1. Modified Bausch and Lomb Nanolab LE2100 scanning electron microscope (SEM) with a joystick-controlled-motorized stage (top). CRT screen with electronic cross-hair display.
- 2a. (Top) After nominal exposure, the electron-beam-exposed area developed normally.
- 2b. (Bottom) Lines on either side of the overexposed alignment pattern received less exposure and were able to develop normally.
3. Two parallel lines produced by SEM overexposure condition. Lines have been gold electroplated.
4. Gold plated support grid is aligned to SEM written line for use in rotational alignment of the submicron period grating.

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