Refining the Process of Achromatic Holographic Lithography

by

Daniel B. Olster

Submitted to the Department of Electrical Engineering and
Computer Science
in partial fulfillment of the requirements for the degree of

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Achromatic holographic lithography (AHL) provides a means of producing highly coherent large area grating structures. Such structures are of interest in a number of applications, in particular atomic interferometry experiments. In this thesis, a method is discussed for fabricating 100 nm period gratings in photoresist using an ArF excimer. A detailed discussion of the alignment procedure of an AHL apparatus is provided to serve as a guide for future grating fabrication. Recommendations are made for improvements of the apparatus.

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Chapter 1

Introduction

Fine period gratings are of interest in a number of applications, including quantum-effect electronics[1], optoelectronics[2], diffractive optics[3], and atomic interferometry[4][5]. For many of the applications mentioned, especially atomic interferometry, the gratings must be free-standing, as they are used as transmission gratings.

The fabrication of such gratings with periods in the deep-submicron regime poses a number of technical obstacles. Some of the issues that must be addressed are how to produce the membranes upon which the gratings will reside, how to image the grating structure on the membrane, and how to process the exposed membranes to produce free-standing gratings. In addition, it is desirable to be able to control the line-to-space ratio and to produce low distortion gratings over large areas.

Researchers elsewhere have proposed the use of electron beam techniques in the fabrication of gratings[5]; however, these methods usually produce gratings with low coherence, and thus, they have small usable areas. Within the Submicron Structures Laboratory (SSL), holographic techniques are used to lithographically expose the gratings. These techniques have been demonstrated to produce far superior results.

Due to the unavailability of short-wavelength lasers with suficiently long coherence lengths conventional holographic techniques cannot be utilized to create 100 nm-period gratings. Therefore, other means of imaging the gratings must be sought. This work focuses on the technique of achromatic holographic lithography (AHL) that is used to produce 100nm gratings in PMMA. A theoretical discussion of AHL is provided, followed by a detailed description of an AHL apparatus. Procedures for aligning the AHL apparatus are discussed. Recommendations for improving the system are provided in addition to a brief discussion of follow-up work.

Chapter 2

Theory

2.1 Conventional Holographic Lithography

Coherent holographic lithography involves the interference of two expanded beams on a photosensitive material. If two plane-waves of the same wavelength are made to interfere, a standing wave will result. The period of this standing wave is given by:

$$\Lambda = \frac{\lambda}{2sin\theta} \tag{2.1}$$

where θ is the incident angle of the two beams relative toe the normal and λ is the wavelength of the light (see Figure 2-1).

From the above relation, it is clear that if we hope to produce a grating with a 100 nm period, the wavelength of the light source must be less than or equal to 200 nm. Thus, a source in the deep ultra-violet (UV) portion of the electro-magnetic spectrum is required.

The lack of coherent sources with suitable power below 257 nm eliminates conventional holography as a viable option. An ArF excimer laser produces high-power radiation at 193 nm; however, it suffers from poor spatial and temporal coherence. In order to overcome the poor spatial coherence of high-power sources in the range that is required, achromatic techniques are employed.

2.2 Achromatic Holography

Achromatic Holographic Lithography (AHL) is a technique that can be used to expose gratings of period Λ using radiation of any wavelength less than 2Λ. In

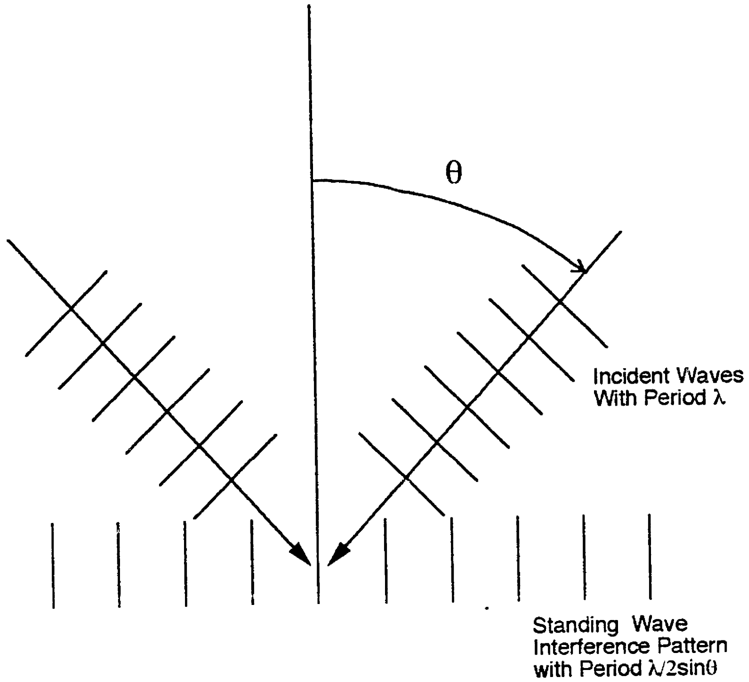


Figure 2-1: Interference of two plane-waves and resulting standing wave pattern

addition, this process is, to first order, insensitive to the low degree of spatial and temporal coherence that deep UV lasers, such as the ArF excimer laser, display.

The process involves the use of two gratings of period 2Λ. Light incident on the first grating at normal incidence will be split into symmetric positive and negative diffracted orders. These diffracted orders will then impinge on the second grating which will produce additional diffracted beams. Two of these beams recombine on the photoresist-coated substrate located behind the second grating (see Figure 2-2).

If we consider the first order diffracted beams from the first grating, we find that it is diffracted at an angle β given by the grating equation:

$$\sin\beta + \sin\theta = m\frac{\lambda}{p} \tag{2.2}$$

where m is the diffraction order, θ is the angle of incidence, and p is the period of the

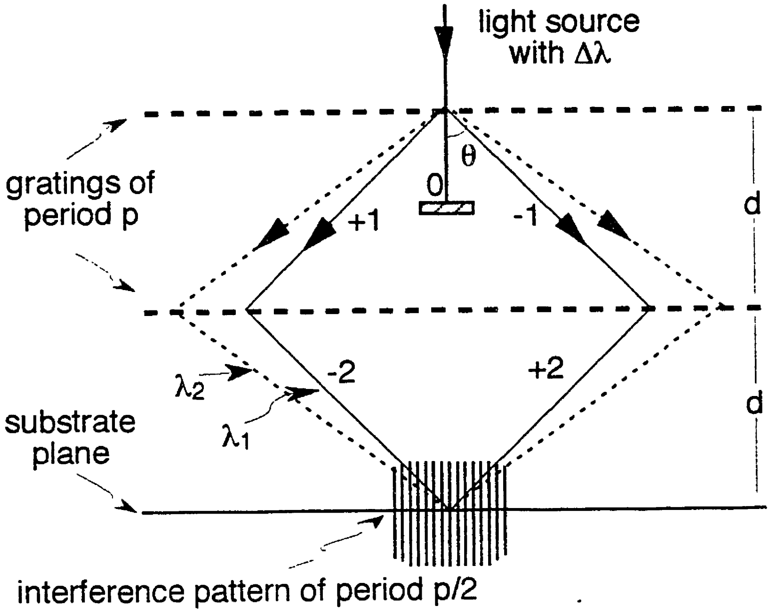


Figure 2-2: The first fixed grating acts as a beam-splitter, while the second serves to recombine the second-order beams which interfere at the substrate.

fixed gratings. For normal incidence, θ goes to zero. Thus, we find that:

$$\beta = \arcsin \frac{\lambda}{p} \tag{2.3}$$

After traversing the gap spacing d, the first-order diffracted beams then strike the second grating at an angle β. The second order beams will be diffracted at an angle γ given by:

$$\sin \gamma + \sin \beta = 2\frac{\lambda}{p} \tag{2.4}$$

From which it follows that,

$$\gamma = \arcsin \frac{\lambda}{p} \tag{2.5}$$

Thus, the +1-2 and -1+2 (where the digits refer to the diffraction orders from the first and second gratings, respectively) diffracted beams impinge on the substrate at an angle γ. If the substrate is located a distance d from the second grating, a standing wave will be exposed on the substrate, with period Λ given by,

$$\Lambda = \frac{\lambda}{2\sin\gamma} = \frac{\lambda}{2\frac{\lambda}{p}} = \frac{p}{2} \tag{2.6}$$

Therefore, the +1-2 and -1+2 beams interfere on the substrate to form a standing wave of period $p/2$. It is clear now, why this process is achromatic. The angle, γ is a function of the wavelength light used. The gap spacing, phase plate thickness, and wavelength determine the required size of the phase plate surfaces.

The above derivation assumes identical spacing between the two fixed gratings and between the second grating and the substrate. In addition, it assumes that the source beam impinges at precisely normal incidence. For strong interference, we require that the path length difference of the two interfering beams is significantly smaller than the coherence length of the laser.

The optical path-length difference that results from an angle of incidence θ radians from normal is given by:

$$\Delta L = (\Delta(\sqrt{n^2 - (\frac{\lambda}{p} + \theta)^2} - \sqrt{n^2 - (\frac{\lambda}{p} - \theta)^2}) + \Delta'(\sqrt{n^2 - (\frac{\lambda}{p} + \theta)^2} - \sqrt{n^2 - (\frac{\lambda}{p} - \theta)^2})) \tag{2.7}$$

where Δ and Δ' are the gap variations shown in Figure 2-3 and n is the index of refraction of the phase plates.

From the grating equation, it can be shown that for $\lambda = 193$ nm and $p = 200$ nm, $\theta_{max} = 2^o$. This is the largest incident angle that will produce first-order diffracted beams. Under the assumption that the gaps vary by at most 1 μm, the most severe angle of incidence results in a path length difference of only 0.3 μm. The coherence length of the laser was measured and found to be 70 μm.[6] Clearly, the angle of incidence will not preclude interference as a result of exceeding the coherence length of the system.

However, a non-normal angle of incidence contributes to the already large spatial incoherence of the ArF beam. The depth of focus of the system, calculated as a

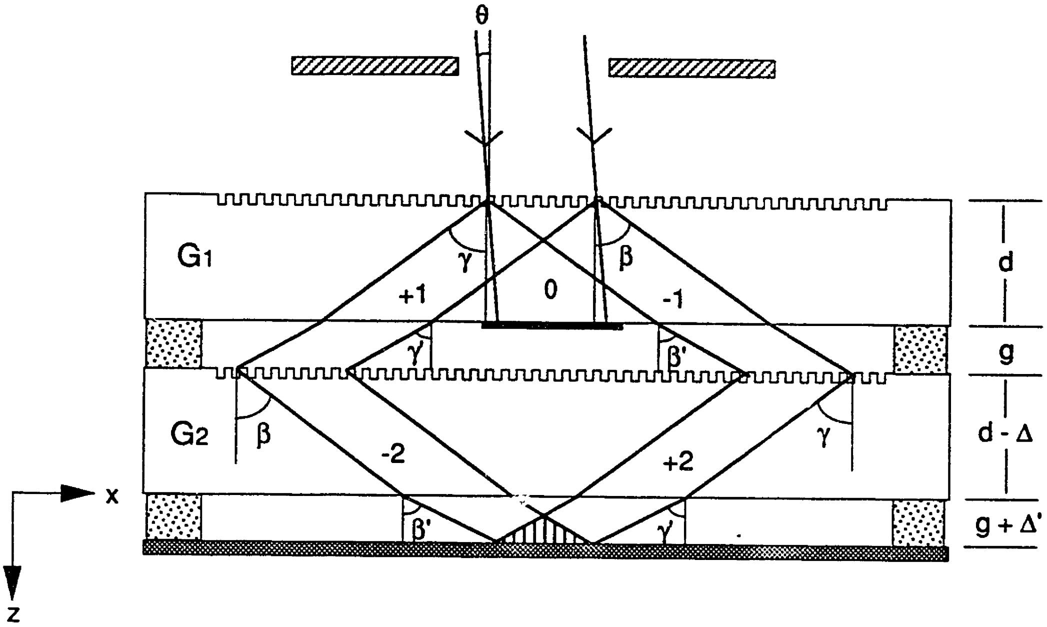


Figure 2-3: Ray picture used to determine optical path lengths through the system

function of the beam angular divergence, $\Delta\theta$, is found to be:

$$\frac{\sqrt{p^2 - \lambda_o^2}}{2\Delta\theta} \tag{2.8}$$

This expression assumes normal angle of incidence and equality of gaps ($\Delta = \Delta' = 0$).[6] For $\lambda_o = 193$ nm and $\Delta\theta = 6$ mrad, the depth of focus is found to be about 4 μm. Thus, the cumulative deviation from ideal conditions that is tolerable is 4 μm. If the variations exceed this value, the substrate will not be in the image plane, and no gratings will be observed.

Chapter 3

Procedures

An apparatus to perform achromatic holographic lithography was designed and constructed by A. Yen.[6] The apparatus consists of two 12.7 mm thick fused silica discs into which 76.2 mm diameter 200 nm-period phase gratings were etched, and a pinchuck fabricated in silicon. One of the silica plates is rigidly mounted, while the other is on a translational platform, adjustable over several micrometers. The pinchuck is also mounted on an adjustable platform. A thin aluminum strip lies between the two silica discs to block the zero-order light. The whole assembly, shown in Figure 3-1, is mounted on a thick aluminum block which rests on an optical table. A top-view of the optical table, as of the writing of this paper is shown in Figure 3-2

Prior to his leaving MIT, A. Yen was able to get high-contrast gratings fairly consistently. Sometime between his departure and my arrival, the system was perturbed. I believe the laser was accidentally bumped and consequently misaligned with the rest of the AHL system. As a result, much effort was spent implementing and refining the alignment procedures described by A. Yen.[6]

3.1 Laser Optics

The light source is an ArF excimer laser capable of producing well over 1 watt of radiation at 193 nm. The beam is Gaussian and has a rectangular profile measuring $\sim$2.5 cm horizontally by $\sim$0.5 cm vertically. The beam divergence is about 6 mrad in the horizontal direction and 2 mrad in the vertical direction.

Upon my arrival, it was determined that the front laser optic required cleaning. It was fogged up as a result laser-induced deposition.[6] It was removed and cleaned with a slurry of tin oxide and deionized water. It was replaced; however, it was not

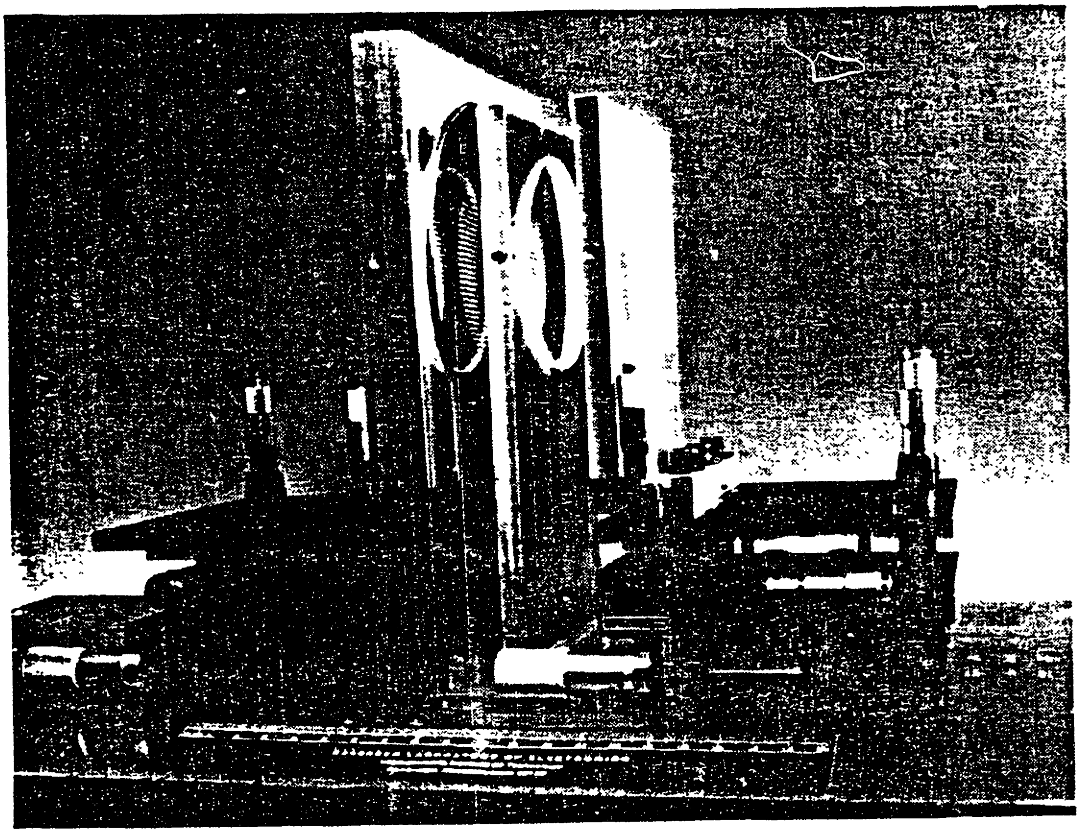


Figure 3-1: Achromatic Holographic Apparatus for Generating 100 nm-Period Gratings

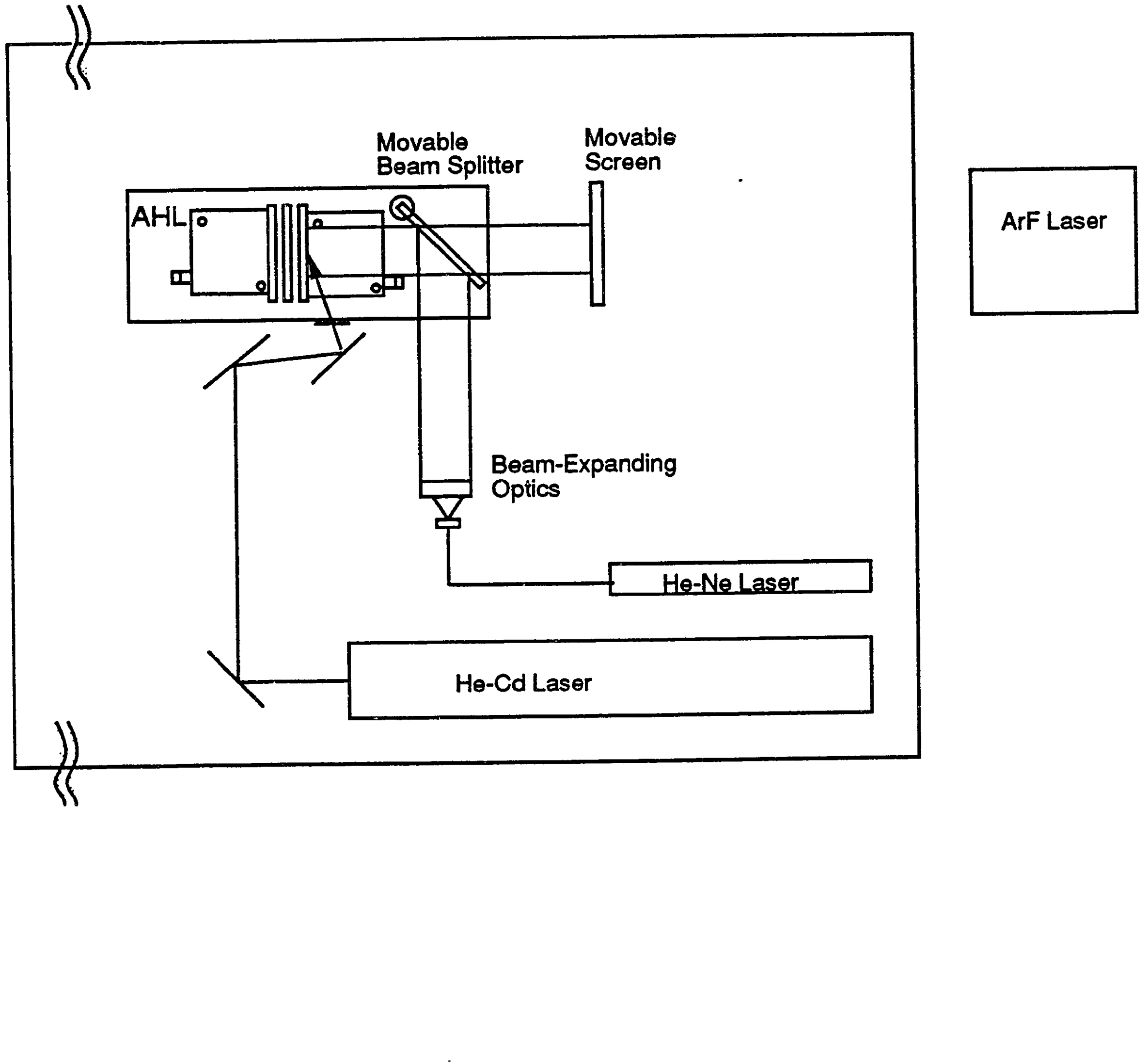


Figure 3-2: Layout of optical table. He-Ne laser and He-Cd laser are used for alignment of AHL apparatus.

initially aligned. This proved to be a source of added angular divergence and loss of power. Once discovered, the misalignment was corrected by following the procedure described in the laser documentation.

As a result of removing and replacing the mirror, the laser had to be passivated following the procedure in the laser manual and in A. Yen's thesis. This is a rather lengthy process which took several days to complete. To summarize the process, the laser cavity is filled with ArF and the laser is run until the power drops to 50%. This procedure is repeated until the half-power point occurs after 5x10^4 shots. At this point, the laser is said to be passivated.

3.2 Establishing Equal Gaps

The gaps between the two silica plates and between the fixed plate and substrate are controlled by spacers. In this case, the spacers of choice are strips of scotch tape. Strips of 100 um-thick tape provide large contact area for lateral stability. Additionally, thickness variation between strips of tape from a given roll are minimal. Gap variations can be controlled to within ~1 um over the ~76 mm area.[6]

3.3 Alignment of Fixed Gratings

The grating lines of the silica plates must be made parallel to assure that the diffracted beams meet and interfere on the substrate. A He-Cd laser (λ = 325 nm) is used for this alignment. For λ = 325 nm and p = 200 nm, the grating equation predicts the existence of a single back diffracted beam. For an angle of incidence of $\sim$ 54.3°, this back diffracted beam returns to the source.

The He-Cd beam is passed through a pinhole in a piece of sheet-metal coated with flourescent paint (the beam is in the UV). It is made to impinge on front silica disc at close to ~54.3°, away from the zero order block. The back-diffracted beams are observed adjacent to the pinhole as shown in Figure 3-3

When the plates are out of alignment, the back-diffracted beams from the two

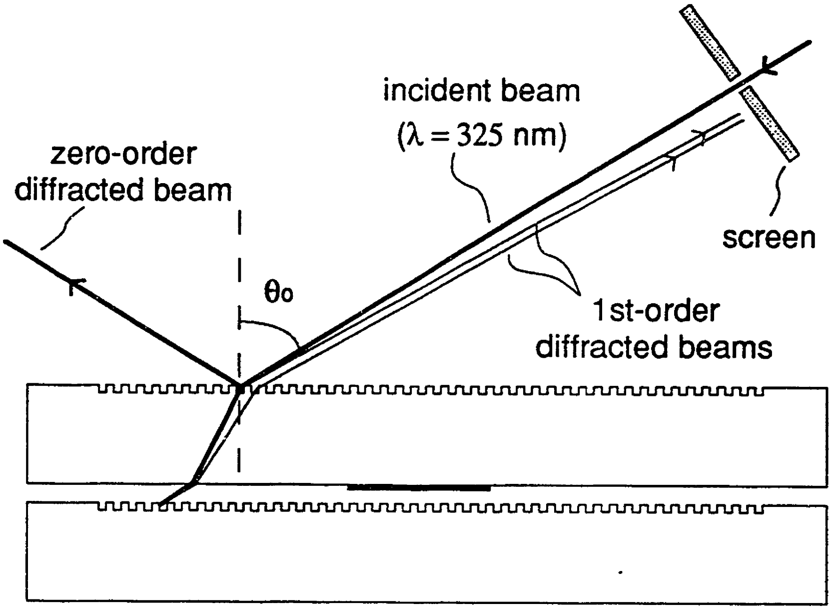


Figure 3-3: Establishing parallelism of phase grating lines using a HeCd laser

plates will be vertically displaced. The micrometers are used to make the beams overlap. Very fine adjustments can be performed with the gratings in contact with the scotch tape; however, for gross alignments, they should be separated to avoid damage to the spacers. When the gratings are nearly parallel, fringes will be observable in the overlapping beams. Careful adjustments can yield one fringe over the ~1 mm spot size. Proper and improper alignments are shown in Figure 3-4 Thus, the gratings can be made parallel to well under 1 mrad.

3.4 Parallelism of Gaps

All gaps in the AHL setup must be made as parallel as possible to avoid exceeding the depth of focus budget. Parallelism adjustments are performed interferometrically using a He-Ne laser. The beam from the He-Ne laser is expanded 75 times using beam-expanding optics. The expander consists of a 3.2 mm aperture with a focal length of -4 mm. The collimating lens has an aperture of 76.2 mm and a focal length

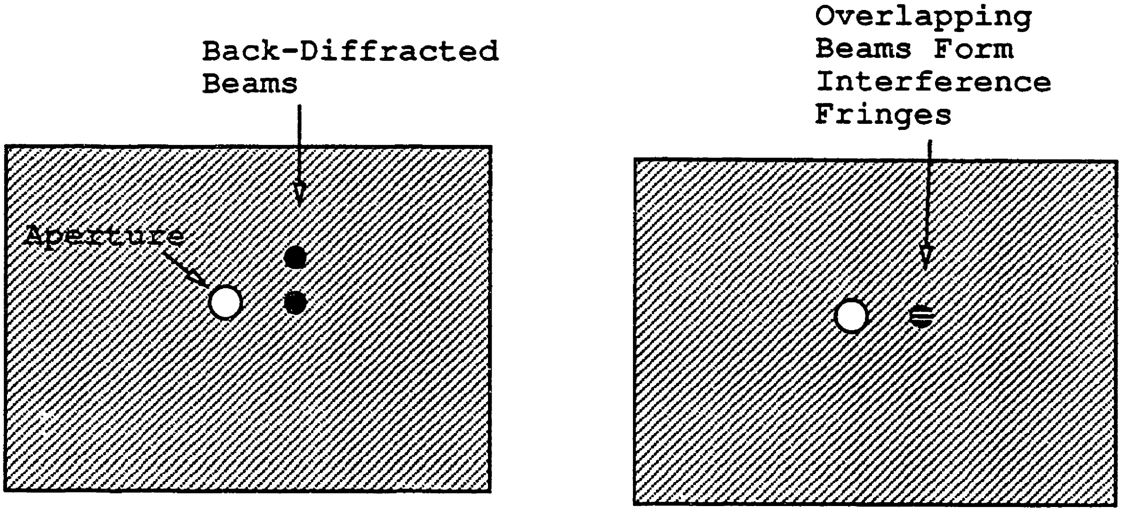


Figure 3-4: a) Improper rotational alignment of gratings results in displacement of back-diffracted beams; b) If properly aligned, fringes will be visible in overlapping beams.

of 300 mm. Both are mounted on a sliding track.

To assure the collimator is correctly positioned, the two silica discs should be separated and a lab clean-wipe placed in the gap. The beam splitter is positioned as shown in Figure 3-5. The interference of the reflected beams from the two surfaces of the first disc is observed on a white screen. The surfaces of the disc are known to be flat to within 0.1 um. The beam collimator should be moved along the track on which it is mounted until only one fringe is observed on the screen. The collimator should now be screwed securely in place. The clean-wipe can now be removed. Since dust may have been introduced in this process, the system should be blown off with N_2.

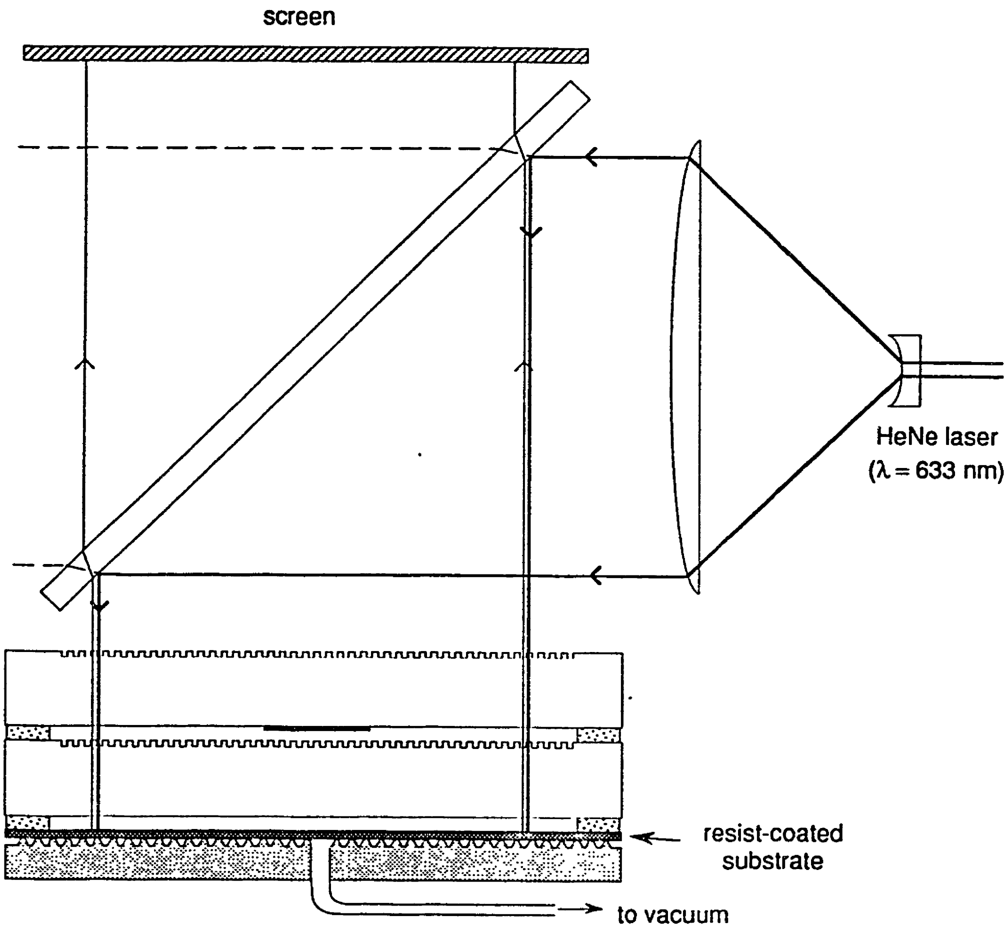


Figure 3-5: Top-view of AHL setup during He-Ne alignment procedure.

3.4.1 Plate-Plate Gap

To establish parallelism between the two silica plates, they are now brought into contact. The pinchuck should now be moved away from the fixed silica disc and a clean-wipe inserted in the gap. The micrometers of the front grating should be adjusted until 1-2 fringes are observed on the screen. Slight movements of the micrometers will not disturb the parallelism alignment performed with the He-Cd. To assure that this is the case, the optical table is setup to allow both alignment procedures to proceed concurrently.

3.4.2 Plate-Substrate Gap

To establish parallelism between the silica plates and the chucked wafer, the pinchuck is brought into contact with the spacers. Fringes should be observed on the screen. The micrometers of the pinchuck mount should be manipulated until a minimum number of fringes is observed. If bullseye patterns are seen in the interferogram, the wafer should be removed, and the pinchuck checked for dust particles. Blowing with a nitrogen jet should remove the dust. With high quality wafers, as few as 2-3 fringes will be observed across the surface of the wafer.

3.5 Intensity Balancing

Ideally, the ArF laser should strike the first grating at normal incidence to achieve intensity balance between the two interfering arms. However, due to a defect in the silica gratings, the positive and negative diffracted orders carry differing amounts of power. This is presumed to be due to an asymmetric blazing of the gratings. To compensate for this, the incident beam should be made to impinge at an angle slightly off-normal. This angle should be small enough to avoid severely degrading the depth of focus. Different regions of the quartz plates are known to display varying degrees of blazing.

3.6 Substrate Preparation

Silicon wafers with an anti-reflection coating of poly methyl methacrylate (PMMA) crosslinked by *bis*-azide, 4,4'-diazidodiphenyl sulfone (DDS) 4:3 were used as the substrate. The ARC is crosslinked by deep UV (260 nm) irradiation of 200mJ/cm^2 and subsequent baking at 160°C for 30 minutes. The photoresist is PMMA. It is baked for 1 hour at 180°C. The 1400Åof ARC and 1200Åof PMMA were spin-coated.[6]

Chapter 4

Results

Initially, it was not possible to produce exposures that displayed any periodic structures. Several of these problems can be attributed to the fact that we work so close to the limits of the depth of focus of the system.

4.1 Substrate Uniformity

Initial problems resulted from using wafers with inadequate thickness control. Even on the pin chuck, thickness variations of several microns were noticed. This places a serious strain on the 4 um depth of focus budget. When high-quality wafers (MEMC brand) were used the number of fringes seen during the He-Ne substrate alignment was drastically reduced. Figure 4.1 shows a sketch of good and bad quality wafers after adhering to the pinchuck.

4.2 Blazing Effects

Alignment of the incident angle to compensate for the blazing of the silica gratings proved to be very difficult. A region of the quartz gratings known to have minimal blazing, about 1/3 down from the top, was used. According to A. Yen, the reflected spot, after passing the beam through a 3 mm aperture, should image adjacent to the pinhole.

Several exposures were done in which the development rate of the first-order spots were compared. Adjustment to the angle of incidence were made until the spots seemed to be equally exposed. However, this misalignment proved to be substantially more than A. Yen found it to be. It was determined that it was best to follow A.

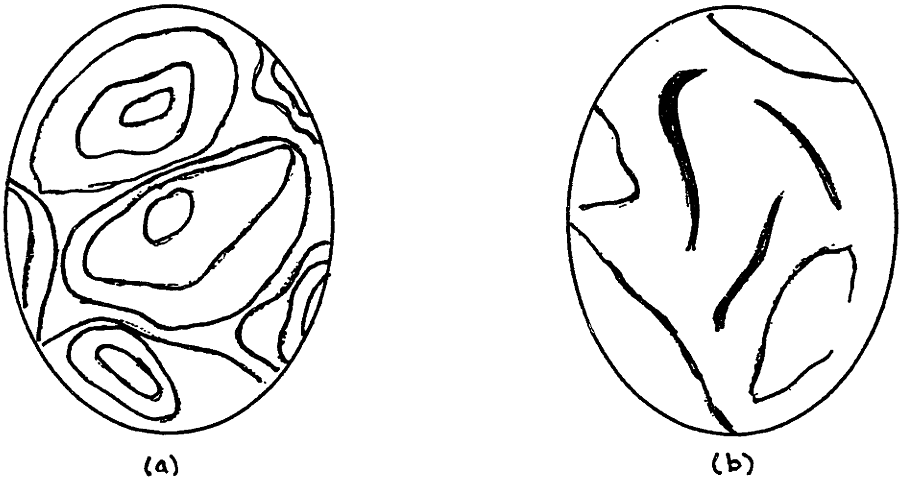


Figure 4-1: Interferogram of a) an inadequately flat wafer and b) a highly flat wafer shown pictorially after adhering to pinchuck

Yen's results by placing the reflected spot to the right of the aperture until a better method of balancing the intensities can be found.

4.3 Dose and Development

Figuring out proper exposure doses and development times also proved to be quite difficult. It is speculated that the available power meter (an antique bolometer) is out of regulation, and not accurate. A dose of 0.15 watts in the apertured beam corresponded to a development time of less than 10 seconds in 60:40 MIBK:IPA at 21°. Immersion development was preferred over spray developing for the ability to control the temperature of the developer.

4.4 Analysis of an Exposed Wafer

A spot-map of an exposed wafer is shown in Figure 4.4. The two outer spots are the zero order spots of the second silica grating. The next two inner spots are the +1-1 and -1+1 beams. The center region is the overlap of the +1-2 and -1+2 beams. This center region is where we expect to find gratings.

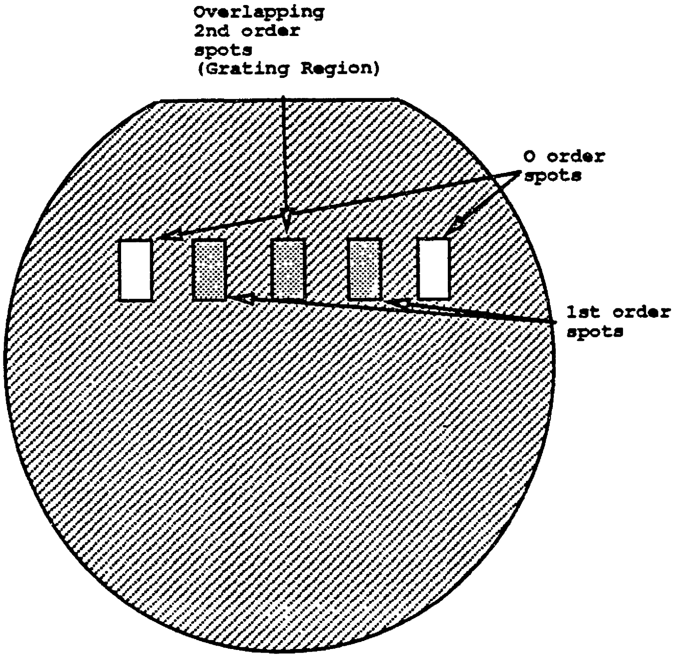


Figure 4-2: Imaging of diffracted beams on the substrate

If all goes well, there should be gratings in the center region. A scanning electron microscope (SEM) is used to view the sample. Consistent results were not obtained. An SEM photograph of the best exposure obtained, shown in Figure 4-3 displays signs of poor contrast (peaks have developed along with valleys). In addition, it appears the ARC has not worked adequately. Also, the quality of the grating lines was not consistent across this sample.

It is presumed that the gaps are not adequately controlled, and a variation exists between the plate-plate gap and plate-substrate gap. This would suggest that the depth of focus of the system is reduced, and the substrate plane may be out of the

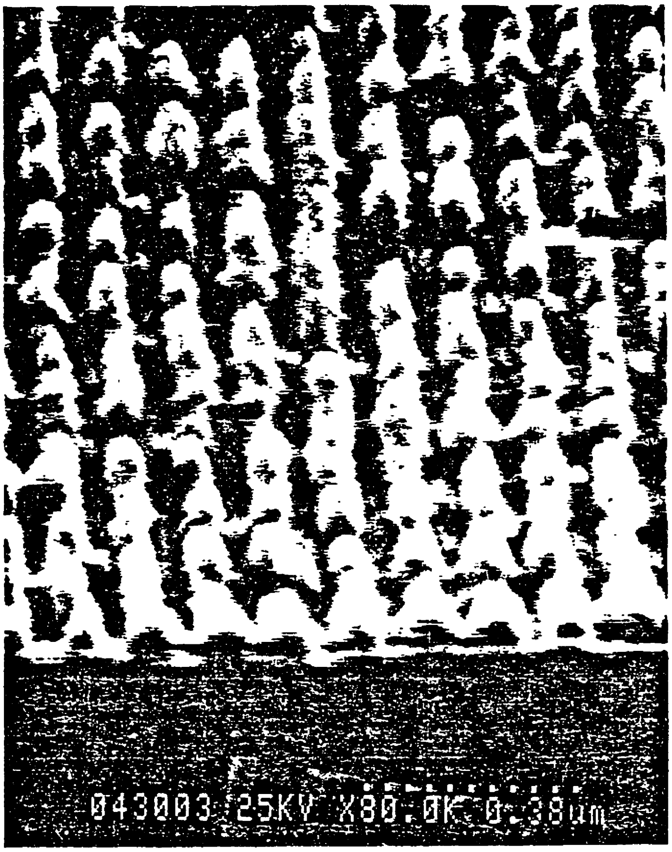


Figure 4-3: Best grating pattern obtained in PMMA on ARC

image plane. If time allowed, the tape spacers would be replaced to attempt to correct for this possible source of problems.

Chapter 5

Discussion

5.1 Improving the System

Several aspects of the AHL apparatus should be modified to make the process more robust. First, a laser should be acquired with higher spatial coherence. As it stands, we work on the fringe of the depth of focus. A laser with a higher degree of spatial coherence will be more forgiving of misalignments in the apparatus.

Other possibilities of increasing the degree of spatial coherence can be explored. Increasing the distance between the aperture in front of the laser and the aperture in front of the AHL would limit the angular range of the beams striking the AHL. Also, decreasing the size of the apertures would help to increase the spatial coherence. While this defeats the ultimate objective of producing large-area gratings, it might provide and indication of why the system is not operating correctly.

An easier method of balancing the two interfering beams should be devised. This may involve fabricating new 200 nm silica phase gratings of higher quality. One option that was explored was to remove the pinchuck and measure the power in the beams directly with a power meter. The first and second order beams were too weak to measure with the available power meter; however, a better meter might be able to do an adequate job.

The one parameter of the system that was not readily checkable via simple interferometric means was the gap spacing. A precision spacer with specified and consistent thickness should be sought. Scotch tape has a large contact area to provide lateral stability; however, the thickness variation from piece to piece is questionable. Also, the effects of being compressed within the AHL system for large periods of time are unknown. Undoubtedly, there is some relaxation and compression. There is nothing to ensure that that degree of relaxation is the same for the plate-plate gap and the plate-substrate gap. Optical fiber has been proposed as a precision spacer. While it will not provide the type of lateral stability that scotch tape does, such stability is likely secondary to assuring equal gaps.

Another possibility, though a bit drastic, would be to mount the entire system such that the phase plates are horizontal to the table surface. In this orientation, gravity would do the work of assuring uniform gaps. If a good spacer were used (such as optical fiber), this might be a viable option. The procedure for alignment of the grating line orientations, which is currently done using the He-Cd laser will have to be modified

5.2 Follow-Up Work

Once high-contrast gratings are produced in PMMA, etching techniques will be employed to produce free-standing gratings in silicon nitride (Si_3N_4). The process will make use of silicon wafers coated with a thin layer of Si_3N_4 (on the order of 1000Å-2000Å). These wafers will be back-patterned with small windows (~1 mm by ~0.1 mm) in photoresist. Reactive ion etching will remove the silicon nitride on the back of the wafer, leaving the silicon exposed.

AHL will then be used to expose 100 nm period gratings on the front side in PMMA. Without developing, a perpendicular support structure is exposed on the PMMA using a flood deep-UV exposure tool. The support structure consists of a 5 μm period grating with 1 μm spaces and 4 μm lines. The 1 μm spaces are exposed and subsequently developed away. The entire exposure is then developed.

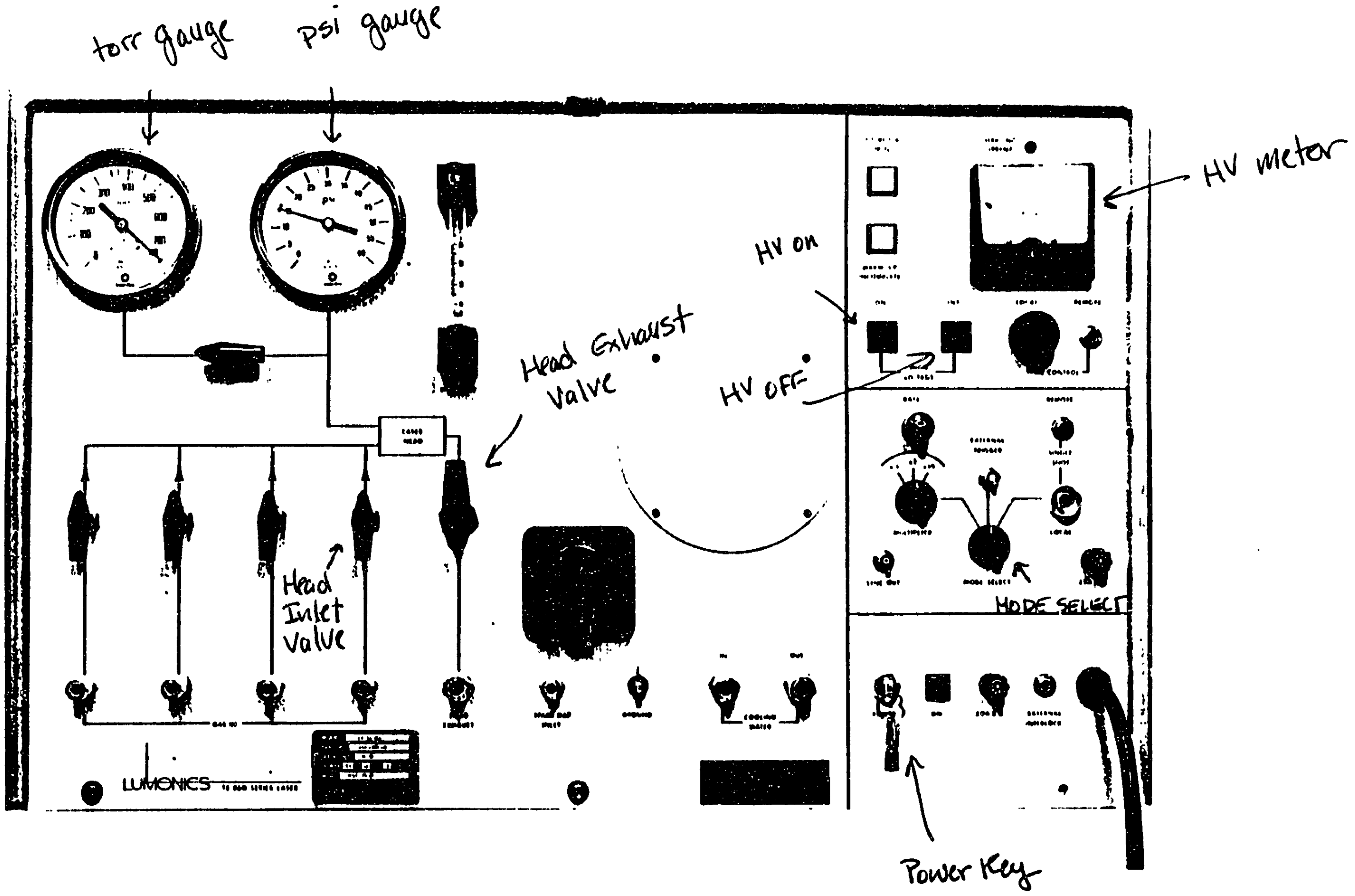
A suitable metal reactive ion etch mask will then be shadow-evaporated on the grating structures. The angle and thickness of the shadow evaporation can be used to achieve a desired line-to-space ratio. Reacitve ion etching will remove the ARC and nitride in the space regions of the grating. The final step is to remove the silicon under the nitride. This can be accomplished with a KOH etch. The final result will be free-standing silicon nitride gratings with 100 nm periods and customized line-widths.

Appendix A

ArF Laser Operation Run Sheet

1. Turn power key on.
2. Turn circulating fan on.
3. Open cooling water inlet and return valves by door.
4. Open head-exhaust valve slightly. Continue to open valve until vacuum pump makes knocking sound.
5. When psi gauge drops below 15psi, switch to torr gauge. Laser should pump down to <10torr. (Tap gauge to make sure needle isn't sticking.)
6. Close head-exhaust valve.
7. Verify that He tank and purge valve are closed.
8. Open ArF (premixed) tank 1/4 turn.
9. Slowly open gas inlet valve to avoid pulling a vacuum on the gas tank. Regulator pressure should remain >10psi. Switch to psi gauge. When 15psi is reached, inlet valve can be opened fully.
10. Fill cavity to 31psi and close inlet value.
11. Place power meter in front of beam path.
12. Set mode select to single-shot.
13. When warm-up is complete, HV off button will light. Press HV on and turn HV control to 30kV.

14. Set rep-rate to 30Hz.
15. Turn mode select to repeat (all the way CCW). Laser should no be firing. Let it run for 5-10 minutes to stabilize.
16. Vary HV and rep-rate to achieve desired power output.
17. HV off will stop laser from firing.
18. At the end of the day, ArF must be pumped out of chamber. Close ArF tank, and repeat pump-down procedure (steps 4-7)
19. Open the He tank 1/4 turn and open purge valve. Fill chamber as before to 20psi.
20. Turn power off.
21. Close water inlet and return valves.
22. Turn circulating fan off.



REAR CONTROL PANEL

FIG. 5-1

Appendix B

AHL Run Sheet

1. Start laser as described in Appendix A
2. Turn on power supply for beam-stearing stepper motor
3. Remove the plexi-glass cover from the AHL. Remove the front window from the plexi-glass cover
4. Slide pinchuck back away from silica plate with micrometer. Slide back manually and insert aluminum spacing block to give easier access to pinchuck.
5. Visually inspect pinchuck and silica plate for dust particles. Blow any dust off with N_2 jet.
6. Pick up sample with vacuum wand. Place against pinncuck and turn on vacuum to pinchuck. If wand is still stuck to wafer, press release button
7. Remove aluminum spacing block. Bring substrate into contact with tape spacers with micrometer.
8. Swing beam splitter into position for He-Ne alignment. Remove aperture in front of AHL if it is there. Place white screen in front of AHL. Turn on He-Ne.
9. First align the two quartz plates. Only a very slight adjustment of the micrometers should be required.
10. Align the substrate by minimizing the number of fringes seen. There may be dust particles behind the wafer. If bullseye patterns are seen, wafer should be removed and the dust should be blown off.

11. Place aperture in position. A post-it paper can be used to mark the correct position to place the aperture in so that the zero order beam is adequately blocked.

12. Cover the AHL with the plexi-glass case. Be sure that the front window is removed

13. Swing the beam splitter out of the way.

14. Leave the screen in front of the AHL, and begin firing the laser. Measure the power of the apertured beam. It should be around 1.4 watts for exposure. Allow the laser to run for several minutes to stabalize. Then, place the He-Ne screen over the top beam stearing optic to block the beam.

15. Move the power meter out of the path of the beam, and bang the table in case it is resting in an unstable position. Wait a few minutes before beginning the exposure to allow the vibrations to settle.

16. Move the beam stearing mirror to the top or bottom line (whichever it is closest to.

17. Remove the screen in front of the laser and begin sweeping the beam.

18. Do three passes of the beam (up-down-up or down-up-down).

19. Turn of the high-voltage to the laser and turn of the circulating fan.

20. Follow loading procedure to remove sample.

21. Immersion develop in a 60:40 mixture of MIBK:IPA at 21°C for 10 seconds

22. Grating should now be present in center exposed region.

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