Optics Design and Fabrication for a Lung Interferometry-Radiography System

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Louisiana State University and Agricultural and Mechanical College

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OPTICS DESIGN AND FABRICATION FOR A LUNG INTERFEROMETRY-RADIOGRAPHY SYSTEM

A Thesis

Submitted to the Graduate Faculty of the Louisiana State University and Agricultural and Mechanical College in partial fulfillment of the requirements for the degree of Master of Science

in

The Department of Physics & Astronomy

by

Rachael Lyn Blair
B.A., Carthage College, 2021
August 2024
Acknowledgments

There are many people who have helped me in some form throughout my studies. First, I thank my research advisor, Dr. Kip Matthews, for giving the most thoughtful advice and lending his endless expertise. I was honored to have the chance to work further with him outside of classes.

Thank you to my supervisory committee, Dr. Les Butler, Dr. Krystal Kirby and Dr. Phillip Sprunger. Their time and counsel was greatly appreciated. Thank you to Paige Whittington for her help through all administrative steps and handling my trip to Maryland. I thank Dr. Kyungmin Ham as I contacted her many times with questions about the diffraction gratings. Thank you to Dr. Sergi Lendinez, for training me in microfabrication and problem solving with me.

Finally, I thank my classmates Chloe DiTusa, Grant Debevec, Richard Lesieur, Hunter Meyer and Lam Lay for their emotional support and kindness. Thank you to my family, especially my mom, Tracy, and my dad, Brett. It was incredibly hard being so far away from them but they have always supported my dreams and knew this was an opportunity I could not miss. Thank you to Liam O’Connor, for dealing with my lows and highs, and being here with me through this relentless time.

The work was supported by the National Heart Lung and Blood Institute of the National Institutes of Health, through grant 1R41HL158414-01A1. Additional support came from the Dr. Charles M. Smith Jr. Distinguished Professorship in Medical Physics. Research was performed at the CAMD Nanofabrication Facility and NIST Center for Nanoscale Science and Technology.
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**Nomenclature, Symbols, Acronyms**

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<th>Acronym</th>
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<tbody>
<tr>
<td>CAMD</td>
<td>Center for Advanced Microstructures and Devices</td>
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<tr>
<td>CNST</td>
<td>Center for Nanoscale Science and Technology</td>
</tr>
<tr>
<td>COPD</td>
<td>Chronic Obstructive Pulmonary Disease</td>
</tr>
<tr>
<td>COVID-19</td>
<td>Coronavirus Disease 2019</td>
</tr>
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<td>CT</td>
<td>Computed Tomography</td>
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<td>DRIE</td>
<td>Deep Reactive Ion Etching</td>
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<tr>
<td>FESEM</td>
<td>Field Emission Scanning Electron Microscope</td>
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<tr>
<td>HMDS</td>
<td>Hexamethyldisilizane</td>
</tr>
<tr>
<td>ICP</td>
<td>Inductively Coupled Plasma</td>
</tr>
<tr>
<td>ISO</td>
<td>International Standards Organization</td>
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<tr>
<td>MEMS</td>
<td>Micro-electromechanical Systems</td>
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<tr>
<td>NFF</td>
<td>Nanofabrication Facility</td>
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<td>NIST</td>
<td>National Institute of Standards and Technology</td>
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<tr>
<td>RF</td>
<td>Radio-frequency</td>
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<tr>
<td>RIE</td>
<td>Reactive Ion Etching</td>
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<tr>
<td>SAS</td>
<td>Small-angle Scattering</td>
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<tr>
<td>SEM</td>
<td>Scanning Electron Microscope</td>
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<td>UV</td>
<td>Ultraviolet</td>
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Abstract

Purpose: High prevalence of lung diseases leads to many people worldwide needing spirometry and imaging testing. The typical imaging done uses chest radiography and/or low-dose CT. Lung tissue is difficult to visualize, making them substandard for monitoring lung disease progression. An improvement on these systems is x-ray interferometry which produces additional images that provide better visualization of changes in the lung tissue. The set-up of x-ray interferometry builds upon other imaging systems in that it includes diffraction gratings.

Materials: This thesis focused on implementing the fabrication processes to create diffraction gratings for a lung imager prototype that is currently under development. Diffraction gratings can be made through microfabrication using lithography and etch tools. Silicon wafers went through photolithographic processes using photoresist and a developer to form the grating pattern. Dry or plasma etch was used to evaluate different recipes to produce deep, vertical trenches. The processes were initially developed at the Nanofabrication Facility at the LSU Center for Microstructures and Devices. Once the processes were understood, production of the gratings was carried out at the Center for Nanoscale Science and Technology at NIST, where a direct-write lithography and deep silicon etch tool were used with similar recipes established at CAMD.

Results: The developed recipes allowed the fabrication of gratings with the desired properties of feature size width, sharp cornered features, spacing between features (pitch), and high aspect ratio depths. Characterization using imaging and other tools showed that a fast etch rate, vertical walls, and consistent fabrication could be achieved. Etch rates of 2.7 µm/min and 3.4 µm/min for phase and absorption gratings respectively were achieved.
Discussion: We have created phase and absorption diffraction gratings suited for our interferometry design. These processes should be adaptable to other models of lithography and etch tools. Lessons learned along the way include using a high resolution light source to pattern sharp features and a dedicated deep silicon etch tool as this avoids gas contamination leading to significantly improved fabrication quality. The processes developed and demonstrated in this thesis will facilitate the fabrication of similar grating designs for future interferometry development projects.
Chapter 1. Introduction

1.1. Significance

According to the American Cancer Society, in 2022, lung cancer was the leading cause of cancer death [1] and according to the World Health Organization, 3.32 million deaths in 2019 were from chronic obstructive pulmonary disease (COPD), a disease that increases the risk for lung cancer [2]. Both bear substantial economic burdens, lung cancer medical care being $14.2 billion in 2018 [3] and COPD being $32.1 billion in 2010 with a projected $50 billion in 2020 [4].

COPD is a common lung disease and can be characterized by respiratory symptoms like dyspnea, chronic cough, sputum production, wheezing, and chest tightness [5]. Some severe cases may even present fatigue, weight loss, and anorexia. There are many risk factors that make a person more susceptible to the disease; examples include tobacco smoking, environmental exposures, occupational exposures, asthma, genetic abnormalities, and recently acute effect from COVID-19 [6]. With symptoms and risk factors present, a spirometry test would be carried out to determine the severity level of airflow obstruction and to give a diagnosis. Spirometry alone can not identify other potential diseases. Chest radiography and computed tomography (CT) are used for lung imaging for further diagnosis. Poor radiographic contrast of lung tissue with these imaging systems limits adequate visualization of damage to the lungs. Specifically for CT, radiation dose to the patient getting a scan is comparatively high which restrains the frequency of CT scans for following disease progression. An imaging system with high sensitivity, low dose, and low cost is urgently needed for diagnosing and managing lung diseases.
1.2. Specific Aims

The overall project centers around creating a practical and clinically applicable lung x-ray interferometer. This work documents the methods of creating the diffraction gratings for the interferometer. Stated are the specific aims for such work:

1.2.1. Specific Aim 1

Develop the process and skills of microfabrication to fabricate diffraction gratings that can be used to produce large-area gratings. The Nanofabrication Facility (NFF) at the Center for Advanced Microstructures and Devices (CAMD) had the necessary tools to develop and implement the photolithography process of transforming a silicon wafer into a grating. The workflow started with exposing the grating pattern onto the wafer which had a layer of photoresist. The pattern was developed to create openings in the pattern then dry etched to form trenches in the silicon substrate.

1.2.2. Specific Aim 2

Produce multiple copies of the diffraction gratings required for the lung interferometry imaging system. The final gratings were fabricated at the Center for Nanoscale Science and Technology (CNST) at the National Institute of Standards and Technology (NIST) using the techniques developed in Aim 1. The CNST provides several kinds of high-level and efficient equipment for photolithography and etch processes such as the Heidelberg MLA150 Maskless Aligner and SPTS Omega c2L Rapier deep Silicon etcher. Inspection of grating designs and measurements of etch rate, selectivity, and sidewall angle are done with advanced imaging and metrology equipment.
1.3. Background

1.3.1. Radiography and Computed Tomography

X-ray production begins within an evacuated tube which contains a negative potential cathode and positive potential anode [7]. A cloud of electrons are produced from the cathode filament when a current is applied. The electrons are accelerated by the applied voltage towards the high atomic number target on the anode illustrated in Figure 1.1. Once they interact with the target, a larger percentage of the electron interactions forms heat and a smaller percentage interacts with the target’s nuclei resulting in emitted photons or bremsstrahlung x-rays. The energy of these x-rays are a continuous spectrum with the maximal photon energy being equivalent to the peak voltage potential of the x-ray tube. Many times the spectrum is intentionally filtered, by placing some material (e.g. aluminum or copper) to remove low energy x-rays and thus lowering patient dose. With filtration in place, one can estimate the average energy of the polyenergetic spectrum. This is also known as effective energy. The effective energy of a beam can be determined to give a penetration power estimate. The effective energy of an x-ray beam is about one-third the maximum energy value represented by Equation 1.1 [7].

$$E_{effective} = \frac{E_{max}}{3}$$ (1.1)

Conventional radiography and CT uses absorption of such x-rays in tissues to form an image. As x-rays are passing through the patient, they are mainly interacting with air (or lung tissue), soft tissue and bone. Each material produces different levels of attenuation due to their compositions which influence the local x-ray absorption coefficient [7]. The x-ray absorption coefficient is dependent on the density and effective atomic number of
Figure 1.1. Schematic of an x-ray tube connected to a high voltage source. The positive terminal is connected to the anode and the negative terminal is connected to the cathode. The small red circles represent electrons traveling towards a target, gray rectangle. The yellow beams are the produced x-rays exiting through a window. Image courtesy of Bushberg textbook [7].

The material that the x-ray passes through. Bone has high values for density and effective atomic number while soft tissues have a small range of densities and an effective atomic number that is similar to water; lung tissue has lower density compared to soft tissues. This means bone, soft tissue, and lung tissue have high contrast relative to each other, but contrast between soft and lung tissue is limited.

1.3.2. Talbot-Lau X-ray Interferometry

X-ray interferometry imaging is an imaging technique that can address the deficiencies for lung visualization of current diagnostic imaging systems. It can be integrated with projection radiography, tomosynthesis, and CT. Interferometry can form images using absorption, diffraction, and small-angle scattering (SAS) of x-rays. Diffraction happens at the boundary between different materials (i.e. air:tissue) and SAS happens when x-rays pass through an object and scatter at certain angles depending upon what the x-rays interacted with inside an object. The x-ray interactions that cause diffraction and SAS
do not depend on density and atomic number in the same way as absorption. Thus the additional images from diffraction and SAS give information different from the absorption image that improves the overall visualization of the lungs as shown in Figure 1.2. In fact, SAS leads to dark-field images that have relatively high inherent lung contrast, which displays features of the lungs that an absorption image would not show [8, 9, 10]. An example could be a lung nodule that is unclear to see from overlying rib in an absorption image but visible in a dark field image in which the bone signal is greatly reduced.

Figure 1.2. Comparison of a traditional absorption image to a dark-field image of a mouse. In the absorption image, the bones have high contrast and are most clearly seen. In the dark-field image, the notable difference is the lungs which have high contrast. The hairs on the mouse are clearly visible as well. This illustrates the dark-field images potential utility to image the lungs. Image courtesy of Troy Jacobs [11].

The x-ray interferometer pertaining to this project utilizes the Talbot effect where a plane wave of x-rays passes through a periodic diffraction grating introducing a periodic phase modulation which creates an intensity pattern [9, 12]. The x-rays would be altered in phase and amplitude, and the extent of these changes depends upon the interference of x-rays within an object [13].
Another important feature of our x-ray interferometer is its scanning geometry. The detectors used in our system are approximately 23 cm in length which is comparable to the size of a human lung. However, the sensitive area of the detectors, is only about 6.5 cm in length. Our system utilizes a scanning geometry to cover the full extent of the lungs by scanning vertically which moves the gratings active area over the full anatomical area of interest [14].

1.3.3. Microfabrication

Microfabrication encompasses many different applications such as microelectronics, microsensors, micro-electromechanical systems (MEMS), microfluidics, and more [15]. Integrated circuits are an example of the technology that can be made through microfabrication. The material of many microfabricated devices is silicon because of its electrical and mechanical properties, and durability. The diffraction gratings created for x-ray interferometry applications use similar microfabrication processes as creating integrated circuits [16, 17, 18, 19, 20]. These microfabrication processes use Ultraviolet (UV) light to form patterns in photosensitive chemicals, and either heat wet chemicals or generate plasma to react with exposed silicon.
Chapter 2. Materials & Methods

2.1. Microfabrication at CAMD NFF

Microfabrication consists of various methods to create devices in the micrometer range [15]. Similarly, nanofabrication works in the nanometer range. This kind of work is done in a cleanroom which controls the environment such as airflow, temperature, humidity and vibrations. Any fluctuations in the environment from ideal conditions can permanently ruin devices. Every cleanroom has class levels representing the particle count per volume. Minimizing the circulation of unwanted particles will protect devices from being contaminated or destroyed. As another action to prevent the introduction of outside particles from entering the cleanroom, users dress in bunny suits with hoods, hair-nets, sterile gloves, and rubber clogs. Some areas of cleanrooms might not need as much protection from particles, in which case, only a lab coat may be needed. An example of a class level is class 100/ISO class 5 cleanroom. This means 100 particles of 0.5 \( \mu \text{m} \) particle size, per cubic foot. The newer ISO class standards converts this to particles per cubic meter. Both LSU CAMD NFF and NIST CNST are class 100/ISO class 5 cleanrooms. NIST CNST has some areas of higher and lower class levels but the main laboratory space is class 100/ISO class 5.

The device fabricated in the end starts as a plain wafer as shown in the top-left of Figure 2.1. Silicon is a common wafer material because it is inexpensive, very durable, has a high melting point, and comes in many sizes and resistivities making it an ideal material for microfabrication. It is a crystalline structure and is grown into different forms such as single crystalline, polycrystalline and amorphous. The silicon wafers used for the gratings
Figure 2.1. Diagram of the general microfabrication workflow. *Adapted from Zhitian et al. [19].*

were single crystalline material and oriented by the Miller Index of 110 crystal structure. Depending on the etch process, a thin film or hard mask may be desired. For wet etching, the wafer starts as a silicon substrate with a dielectric thin film of silicon nitride deposited on top. Wet etching was tried earlier in the development process but the gratings used for our system were made with dry etching. Therefore, only the process of dry etching is described in this thesis. For dry etching, the wafer can be bare-silicon or use a hard mask; bare-silicon wafers were used for this project.

Optical lithography or photolithography is a method used to create the diffraction grating pattern. The main steps were to expose and develop. Before beginning the expose step, since a thick photoresist was used to enable deep etching, we de-hydrated the surface of the wafer to allow adequate adhesion of the photoresist. Silicon is hydrophilic and
Figure 2.2. Photo of Durham Magneto Optics Ltd’s MicroWriter ML3 Pro setup in CAMD NFF cleanroom. The monitor on the right runs the software needed for operating the tool itself seen on the left. The chamber opens by pressing the circular silver button on the white front panel to the right. This unlocks the door and it hinges up towards the ceiling. The sample is placed in the center of the chamber and aligned to the aligning marks.

absorbs any moisture from ambient humidity so first a dehydration bake is done to remove all moisture from the surface. Next, hexamethyldisilizane (HMDS) was spin coated onto the wafer which adds a methyl (CH$_3$) group to oxygen sites of hydroxide (OH) to make the wafer hydrophobic [21]. After this, a layer of positive photoresist, AZ 12XT-20PL-10, was spin coated on to serve as a photosensitive polymer [15]. There are two kinds of photoresist: negative and positive. Negative means exposed areas are insoluble and won’t go away when exposed with light. Positive is the opposite and was the type of photoresist used for our wafers. After coating the wafer, the photoresist was soft-baked to remove any solvent. The entire wafer with the layer of positive photoresist was placed into a photolithography machine (Durham Magneto Optics Ltd’s MicroWriter ML3 Pro) with an i-line 365 nm UV light source shown in Figure 2.2.
Figure 2.3. A screenshot of the CleWin software used for designing photomask patterns. The black background is the unexposed photoresist and the purple rectangles is exposed silicon. This image displays the G2 pattern with a 10 \( \mu m \) feature size and 20 \( \mu m \) period. In parentheses, the measurements for the G1 pattern are given. The trench and support structure lengths are 100 \( \mu m \) and 10 \( \mu m \), respectively.

The grating pattern was loaded into the Microwriter software to become the “photomask”. Mask layout editing software (CleWin) was used to design the pattern shown in Figure 2.3. The software was where the grating array pattern was first formed and precise feature sizes were specified. Unlike some other x-ray interferometry gratings, a bridge pattern was implemented to give the narrow trenches a stronger structure. The UV light enters through the openings in the photomask to create the pattern on the wafer.

Once the exposure ended, a post exposure bake was done to finalize the exposure process. Next, the wafer needed to be developed so that the exposed photoresist was removed. The optimized time for development varies greatly across feature sizes and doses. Development time tests need to be done to determine the correct amount of time, especially for new patterns. It was important to determine this to avoid under or over
development because the pattern may not form at all or lose adhesion, respectively.

There are various methods for development such as immersion baths, puddles, or spray. Immersion baths were utilized for these wafers. Once developing was done, the wafer now had parts with unexposed photoresist and parts with exposed silicon. Parts with exposed silicon were the pattern that was loaded into the photolithography machine software.

The final step was to etch into the substrate (i.e. the exposed silicon areas). Dry etching is also known as plasma etching or reactive ion etching (RIE). Deep reactive ion etching (DRIE) is a specific type of RIE which is used for etching deep structures. The etch plasma created in the chamber etches the areas of the pattern that have exposed silicon down to a desired depth. The etch plasma also reacts with the unexposed photoresist areas, but it etches at a much slower rate. One needs to balance etching the exposed silicon areas but not degrade the unexposed photoresist areas too much. Selectivity quantifies this where it is the etch rate of silicon to the etch rate of the photoresist. The DRIE recipe can affect the selectivity greatly depending on the input parameters. This is not the only characteristic of DRIE to consider; other parameters can affect the etch rate and ion anisotropy shown in Figure 2.4.

The type of DRIE technique used is called the Bosch Process, named after German engineering company Robert Bosch GmbH. The process uses a deposition and etch plasma to create trenches with high aspect ratios and vertical walls. This was desired for our grating pattern where G1 and G2 require aspect ratios of 20:1 and 10:1, respectively. The plasma etching tool (Oxford ICP-DRIE System 100 ICP180) used to implement this Bosch process is shown in Figure 2.5.

Following Figure 2.6 from left to right, the deposition plasma is made from energized
Figure 2.4. Flowchart of plasma etch parameters and their respective effects which dictate the characterization of the etch rate, selectivity, and anisotropy. Flowchart courtesy of Franssila textbook [15].

$C_4F_8$ gas which acts as a fluoropolymer to coat all surfaces and sidewalls of the trenches. It protects the sidewalls from subsequent etches since many etch cycles will be carried out. Next, the etch plasma is created from energized $SF_6$ gas which consists of ions and free radicals. First, the ions will be accelerated toward the wafer and will bombard the bottoms of the trenches to remove the fluoropolymer coating. The free radicals follow after and etch the bottom of the trenches. Each step lasts seconds so the gases are pulsed throughout the entire process. Testing to determine the satisfactory time for each step may need to be done but generally it will be 1-7 seconds.

Once the desired etch depth was reached, it was now safe to remove the photoresist mask. Depending upon which photoresist used, there is a recommended photoresist remover and removal method. For AZ 12XT-20PL-10, it was recommended to use AZ
Figure 2.5. Photo of the Oxford ICP-DRIE System 100 ICP180 setup in CAMD NFF cleanroom. The upper left hand compartment is the loadlock which is where the sample is placed. This creates a vacuum sealed environment that safely transfers the sample into the chamber. The chamber sits above the company logo and has a small circular port hole where the plasma glow can be seen. Image courtesy of CAMD NFF website [22].

Figure 2.6. Diagram showing the three steps of the Bosch process. Stage 1 on the left shows the passivation gas, C₄F₈, entering the chamber and coating the sample. Stage 2 in the middle shows the etch gas, SF₆, entering the chamber which becomes energized and creates fluorine radicals and ions. The radio-frequency (RF) power accelerates the ions to the surface of the sample to remove the passivation film from the bottoms of trenches. Stage 3 on the right shows the fluorine radicals etching isotropically into the exposed silicon. Diagram courtesy of Oxford Instruments [23].
400T photoresist stripper heated as a bath to 80°C. If there was residual photoresist after using the stripper, an O₂ descum was applied. Before the descum, most of the residual photoresist should be clear otherwise they may get redeposited into the trenches.

The three main tools of measurement used were a contact profilometer (Figure 2.7), an optical microscope (Figure 2.8) and a scanning electron microscope (Figure 2.9). The profilometer (Contact Profilometer Tencor P2) uses a stylus that makes contact with the surface of the wafer and can be moved across the surface of the wafer. It measures depth differences which can represent the depth of an etch when the stylus was placed near the edge of a feature. This can be used to measure how much an area had been etched and from that an etch rate was calculated. There are limitations when the feature size is smaller than the stylus diameter. The stylus was not able to reach our trench bottoms; for this reason height measurements were estimated from a depth pad that was added near the edge of a wafer.

The optical microscope (Nikon OPTIPHOT-88) was used for visual analysis but also offered some quantitative analysis through measurement software. Best practices were to use the microscope after each process step to ensure each step was working properly. This was used to observe if areas of exposed silicon were completely clear of photoresist or fully developed. During resolution tests, it could be observed if the features had sharp corners. It was also used to measure the feature size as that can vary after exposure.

The scanning electron microscope (SEM Hitachi S4500) uses an electron beam that passes through magnetic lenses focusing the beam onto the sample. The sample or wafer was broken by a diamond scribe to allow imaging of the cross section of the wafer. SEM gives superior depth of field for top views compared to optical microscopes but it gives
Figure 2.7. Photo of the Contact Profilometer Tencor P2. The clear window hinges open where the sample can be placed on the stage. The stage can be controlled by the keyboard with changes in the x, y, z, rotation and tilt. The monitor above the stage shows the sample surface and displays the step height data. *Image courtesy of CAMD NFF website [22].*

Figure 2.8. Photo of Nikon OPTIPHOT-88 Optical Microscope.
Figure 2.9. Photo of SEM Hitachi S4500. The sample exchange chamber is hidden behind the large cylindrical column where the electrons enter. The small pad with a joystick near the keyboard moves the stage in the x, y, z, rotation and tilt. The keyboard is where the electron beam can be turned on. Images show up on the right monitor screen. Adjustments to the images such as brightness and contrast are also on the keyboard. The video feed above the monitors shows the sample in the chamber. Image courtesy of CAMD NFF website [22].

even better cross-sectional views because you can see sidewall angle and sidewall surface quality [15]. This can be used to visualize the anisotropy (i.e. the smoothness of the walls) and measure the sidewall angle. The Quartz Personal Computer Imaging (PCI) image analysis program enables measuring of final etch depths which can be used to calculate the etch rate.

2.2. Microfabrication at NIST CNST

The overall process follows similar steps as developed at the CAMD NFF. However, different machines and techniques were used while at the NIST CNST. This section reviews the types of machines utilized and highlights differences in techniques. The process started with de-hydrating the wafer with HMDS. The CNST had a HMDS Prime oven (Yield Engineering Systems) which simultaneously baked the wafer and applied the
Figure 2.10. Photo of Filmetrics F40-UV Reflectometer. The objective on the left is situated above where the sample would be placed. The monitor on the right shows the software that displays the reflectance data. A baseline must first be taken using the sample, a bare-silicon wafer and mirror for background. *Image courtesy of NIST CNST website [24].*

HMDS vapor. This can be done with batches of wafers. Next, the wafers were ready to be spin-coated with positive photoresist at one of the solvent hoods. The CEE Apogee Spinner was utilized which had pre-programmed spin settings and an adjacent hot plate for soft baking. One tool not available at CAMD was a reflectometer. The CNST had the Filmetrics F40-UV Reflectometer shown in Figure 2.10 which measured reflectance of UV and visible light on the silicon wafer covered with photoresist. This gave a measurement of photoresist thickness which can be used to make sure enough resist was spun-on for the etch process later on. Most of the time the spin curves are sufficiently accurate but this method was another way to confirm photoresist thickness.

The lithography tool (Heidelberg MLA150 Maskless Aligner) used at the CNST is shown in Figure 2.11 installed with two diode lasers, 365 and 405 nm. The tool is
Figure 2.11. Image of Heidelberg MLA150 Maskless Aligner. The sliding door can be opened by pressing the small white button to the right of the door. The sample can be placed on the stage and aligned according to its diameter. The vacuum button to the right should be pressed to seal the sample to the stage. The sample will be moved under the light source once the sliding door is shut. *Image courtesy of NIST CNST website [24]*.

compatible with KLayout which is a dedicated mask design application. The designs made in KLayout were what made the photomask, identical to how the ML3 Pro works. No changes to the designs were made compared to the work done at CAMD NFF. After the post exposure bake, the wafers went through development and the CNST offered the spray and puddle techniques with the Suss MicroTec Delta12AQ Automated Resist Developer shown in Figure 2.12. The tool had three different developers available: AZ400K, AZ MIF300 and CD26. The developer used for these wafers was AZ MIF300, same as what was used at the NFF. The wafer was placed on a vacuum stand which secures the wafer during the entire application process. The developer was sprayed onto the surface and puddles were formed on the surface of the wafer.

Several optical microscopes (Nikon L200) were located throughout the cleanroom and
could be used for the same purposes as at CAMD NFF. Shown in Figure 2.13, they had similar software in that feature measurements could be made to check accuracy of feature sizes. It was typically used after the development and photoresist removal steps to observe proper formation of features and take additional measurements to verify consistency in features by the end of the process.

After confirming the correct pattern formation, the process continued to the etching step. The etch tool (SPTS Omega c2L Rapier Deep Silicon Etcher) used is shown in Figure 2.14. This tool provided deep trenches and smooth sidewalls at quick etch rates. It applied the same Bosch process as the Oxford tool except this tool is designated for only deep silicon etching. This means only gases relevant to the Bosch process were available and only silicon wafers can be processed on. There were a few other requirements for use
such as the mask of the wafer, it must be SiO$_2$ or photoresist. The wafer must be flat and the backside of the wafer must be clean. If the wafer had some residual photoresist or developer spots on the backside, IPA or acetone can be used to clean this.

Once etching completed, the photoresist layer was removed. Each type of photoresist has recommendations on the type of remover to use. Usually a heated wet chemical is used and can be used in conjunction with RIE since the Bosch process hardens the photoresist layer. The CNST had several Reactive Ion Etcher Unaxis 790’s shown in Figure 2.15 that have O$_2$ etch recipes to remove residual photoresist. These recipes were very similar to the O$_2$ clean recipes used at CAMD NFF.

Two additional tools of measurement were a contact profilometer (Figure 2.16), and a field emission scanning electron microscope (Figure 2.17). The CNST offered a stylus
Figure 2.14. Photo of SPTS Omega c2L Rapier Deep Silicon Etcher. The loadlock is behind the silver door (left side of image) where the sample can be loaded. Behind this is a robotic arm that transfer the sample to the processing chamber. The chamber is the central black compartment which does have a small port hole but difficult to see from this photo. *Image courtesy of NIST CNST website* [24].

Figure 2.15. Photo of Reactive Ion Etcher Unaxis 790. The silver chamber with handle on the left is where the sample is placed. There is a small port hole adjacent to the handle where the plasma glow can be seen. The monitor on the right has the software to control venting and pumping the chamber, and where recipes are loaded. *Image courtesy of NIST CNST website* [24].
Figure 2.16. Photo of Bruker DekTak XT Stylus Profilometer. Inside the tool, the stage is the porcelain white square seen on the right. This is where the sample is placed and vacuum sealed. The stage can be controlled by the software seen on the monitor with changes in the x, y, z, rotation and tilt. The monitor to the left shows the sample surface and displays the step height data. *Image courtesy of NIST CNST website* [24].

A profilometer (Bruker DekTak XT) and it had all the same functions and settings but in a more user-friendly environment. It still measured depth differences or step heights. It can be used after the photoresist removal step to check if the photoresist had been fully removed from the surface. It can assess the tops and bottoms of trenches, and observe if surfaces were relatively smooth. To measure etch rates and observe sidewall smoothness, the CNST offered the Zeiss Gemini 500 Field Emission Scanning Electron Microscope. One major difference between this tool and what was offered at CAMD NFF, was that this system uses a field emission electron gun rather than producing electrons via thermionic emission. This created very high resolution and high quality images at low and high voltages.

For the wafers with the absorption grating only, they needed to be cut down from a circular wafer to a square region comprising the grating active area. This required the use of a dicing saw (ADT 7132) shown in Figure 2.18. It had four different kinds of blades
Figure 2.17. Photo of Zeiss Gemini 500 Field Emission Scanning Electron Microscope. The sample exchange chamber is to the right of the large cylindrical column and has a rod sticking out. This is where the sample and sample holder are screwed in and transferred into the stage. The electron column is where the company logo is. The monitors to the right have the imaging software where the electron beam can be turned on. The small pad with a joystick near the keyboard moves the stage in the x, y, z, rotation and tilt. Adjustments to the images such as brightness and contrast are on the keyboard. Image courtesy of NIST CNST website [24].
for various materials. The 27HEFE blade used for our silicon wafers was 35 \( \mu m \) wide and creates cuts 45 \( \mu m \) wide. Measurement resolution for checking distances was 0.1 \( \mu m \) and positioning accuracy of the blade was 0.5 \( \mu m \). Parameters inputted included wafer shape, wafer material, wafer diameter, wafer thickness, index of cut, and cut depth.

Figure 2.18. Photo of ADT 7132 Dicing Saw. *Image courtesy of NIST CNST website* [24].

2.3. Testing

To create high quality diffraction gratings and achieve the desired specifications, typical microfabrication tests need to be carried out at each step of the photolithographic and etch processes. Analysis was done using metrology and imaging equipment described earlier.
2.3.1. Photolithography and etching tests at CAMD NFF

Photolithography tests done at CAMD NFF included dose, focus and resolution tests. Dose tests consisted of exposing small areas of photoresist with different exposure doses to determine which value fully imprints the pattern. Focus tests set the depth of focus of the UV light at different depths in the photoresist. This is typically used for thicker photoresists and lower quality resolutions. Resolution tests were done by setting the small areas to different resolutions which means the smallest feature that can be printed according to the depth of focus used [25]. In addition, development tests were done after exposure and they were simply time tests to determine how long the pattern needs to be developed. The optical microscope was used to observe which test area formed the bridge array pattern. Etch tests were done to characterize etch rate, selectivity and sidewall angle for specific dry etch recipes. Equations 2.1 and 2.2 were used to calculate the etch rate and selectivity of the recipe, respectively. $d_{Si}$ represents the amount of silicon etched and $d_{PR}$ represents the amount of photoresist etched. $t$ represents the total amount of time for one loop of the Bosch process.

\[
Etch\ rate = \frac{d_{Si}}{t} \tag{2.1}
\]

\[
Selectivity = \frac{d_{Si}/t}{d_{PR}/t} \tag{2.2}
\]

The Bosch process had not been tried on the current plasma etcher so several initial tests were done to better understand the process then optimization was done to achieve the fastest etch rate, highest selectivity, and nearly right angled trench walls.
2.3.2. Photolithography and etching tests at NIST CNST

An addition to the previous photolithography tests was a photoresist thickness measurement test. This ensured the correct amount of resist had been coated on and that it was an adequate mask for etching. Photolithography tests done at NIST CNST mainly were a dose test for both patterns. The design of the dose test was the same as what was done at CAMD NFF with several test areas spanning the wafer. Six doses were tried for each pattern ranging from 80 to 155 mJ/cm². The optical microscope was used afterwards to see which dose formed the bridge pattern and reached the desired feature size. There were no focus or resolution tests. From previous data and projects, there was a recommended development method which suited this project. Therefore, a development test was unnecessary. Etch tests measured for an etch rate and a more accurate selectivity. Selectivity was more accurate because the post-etch photoresist thickness was measured with the Filmetric tool. An etch rate was calculated from this and compared with the silicon etch rate. Equations 2.1 and 2.2 were used to calculate these values. Sidewall angle measurements were more a visual assessment. The NIST CNST Bosch recipes had been proven to form vertical walls and flat bottom trenches; as a result parameter optimization was unnecessary.
Chapter 3. Results

3.1. Photolithography and etching tests at CAMD NFF

The target dimensions of the gratings are shown in Table 3.1 and illustrated in Figure 2.3. The phase grating (G1) has a period of 7.7 \( \mu \)m and a trench depth of 77 \( \mu \)m meaning it has an aspect ratio of 20:1. The absorption grating (G2) has a period of 20 \( \mu \)m and a trench depth of 100 \( \mu \)m meaning it has an aspect ratio of 10:1.

Table 3.1. Target grating dimensions for both the phase G1 and absorption G2. In parentheses are the possible variations in depth. Etching is not exact every time which leads to a tolerance for the trench depth.

<table>
<thead>
<tr>
<th>Grating</th>
<th>Period (( \mu )m)</th>
<th>Feature Size (( \mu )m)</th>
<th>Trench Depth (( \mu )m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>G1</td>
<td>7.7</td>
<td>3.85</td>
<td>77 (± 5)</td>
</tr>
<tr>
<td>G2</td>
<td>20</td>
<td>10</td>
<td>100 (± 10)</td>
</tr>
</tbody>
</table>

The process for the use of AZ-12XT-20PL-10 photoresist is shown in Table 3.2 with modifications made to the development and photoresist removal steps that were given by the manufacturer. Modifications to the reference processes can cause unexpected results than what the manufacturer provides. Understandably, some modifications needed to be made due to limited abilities within a cleanroom. However, doing this required an understanding that this may change expected results. The reference process for development suggested using a puddle method but this was not offered at the NFF so an immersion bath was done instead. Also, the reference process for photoresist removal stated to use sonication at the end of the photoresist removal step, however, this caused the trenches to collapse shown in Figure 3.1, therefore sonication was not used at this point.

The results of the photolithography tests are summarized in Table 3.3 and correct
Table 3.2. Sequential process for use of AZ-12XT-20PL-10 photoresist.

<table>
<thead>
<tr>
<th>Process step</th>
<th>Parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>Prime</td>
<td>Spin-on HMDS</td>
</tr>
<tr>
<td>Coat</td>
<td>2000 rpm for 60 sec for 10 µm thickness</td>
</tr>
<tr>
<td>Soft Bake</td>
<td>110°C for 3 min</td>
</tr>
<tr>
<td>Expose</td>
<td>dependent on design</td>
</tr>
<tr>
<td>Post Exposure Bake</td>
<td>90°C for 60 sec</td>
</tr>
<tr>
<td>Develop</td>
<td>AZ 300MIF immersion bath</td>
</tr>
<tr>
<td>Post Develop Bake (Hard bake)</td>
<td>100-115 °C for 2-3 min</td>
</tr>
<tr>
<td>Photoresist removal</td>
<td>AZ 400T stripper; 2 x 80°C immersion bath for 20 min then IPA and DIW immersion bath for 5 min</td>
</tr>
<tr>
<td></td>
<td>DO NOT USE SONICATION</td>
</tr>
</tbody>
</table>

Figure 3.1. SEM image of trenches collapsed after sonication. In the center of the image, it shows what remains of two sidewalls. The jagged silicon around this is collapsed trenches.
formation of the patterns are shown in Figures 3.2 and 3.3. Since a thicker photoresist was utilized, higher exposure doses were needed with the G1 pattern at 400 mJ/cm$^2$ and G2 pattern at 300 mJ/cm$^2$. To achieve accurate and sharp features, fine writing resolutions were used with the G1 pattern. Closely related to the writing resolution is the focus correction where broader writing resolutions allow more depth of focus. This allowed for a -10 um focus correction for the G2 pattern which had a larger feature size. The development time varies with exposure dose and depth of focus. The G1 pattern took 90 seconds and the G2 pattern took 210 seconds to develop. The post develop bake was set at a higher temperature and longer time for the G1 pattern since it had a smaller feature size.

Some examples of incorrect pattern formation are shown in Figures 3.4 and 3.5. They show some of the results from the test pads for the G1 pattern. This illustrates the importance of using the right dose, focus, resolution and development time to form the desired pattern. In Figure 3.4, it shows a test pad that was set to 300 mJ/cm$^2$, 0.6 µm writing resolution, and -10 µm focus correction. From this image, we could see rainbow swirls where the features should have formed which is a sign of under-development. In Figure 3.5, it shows a test pad that was set to 420 mJ/cm$^2$, 1 µm writing resolution, and -10 µm focus correction. From this image, we could see the features lose their spacing between each other which is a sign of over-development and a need for a finer writing resolution.

Once the photolithography steps were determined, the wafer began to go through etching. Table 3.4 lists the different etch recipes used with the OXFORD ICP-DRIE System 100 ICP180. A reference Bosch recipe was provided by Oxford and modifications
Table 3.3. Photolithography parameters for both the phase G1 and absorption G2 gratings using AZ-12XT-20PL-10 photoresist. In parentheses were the ranges tested for each parameter.

<table>
<thead>
<tr>
<th>Grating</th>
<th>Exposure Dose (mJ/cm²)</th>
<th>Writing Resolution (µm)</th>
<th>Focus Corrections (µm)</th>
<th>Development Time (sec)</th>
<th>Post Develop Bake</th>
</tr>
</thead>
<tbody>
<tr>
<td>G1</td>
<td>400 (270-420)</td>
<td>0.6 (0.6, 1 &amp; 2)</td>
<td>0 (-10 to +10)</td>
<td>90 (90-210)</td>
<td>3 min at 115°C</td>
</tr>
<tr>
<td>G2</td>
<td>300 (120-550)</td>
<td>1 (1, 2 &amp; 5)</td>
<td>-10 (-10 to +10)</td>
<td>210 (30-570)</td>
<td>2 min at 100°C</td>
</tr>
</tbody>
</table>

Figure 3.2. Microscope image of G1 pattern after development. The rectangular structures are exposed silicon and were etched into trenches. The in-between structures are unexposed photoresist.
Figure 3.3. Microscope image of G2 pattern after development.

Figure 3.4. Microscope image of incorrect formation of G1 pattern after 210 seconds development. The dose was set to 300 mJ/cm², the writing resolution was set to 0.6 µm, and the focus correction was set to -10 µm.
Figure 3.5. Microscope image of incorrect formation of G1 pattern after 210 seconds development. The dose was set to 420 mJ/cm$^2$, the writing resolution was set to 1 µm, and the focus correction was set to -10 µm.

were made thereafter to better suit our depth goal. As shown in the table, there were many parameters to change and it is not limited to this table. The main constant of the Bosch process was the temperature which was set at 15°C for all recipes. The first recipe used a high deposition gas flow, referred to as the high gas flow recipe. The pressure was set at 30 mTorr and the time at each step was 9 seconds. Generally, the RF and inductively coupled plasma (ICP) were set at the same power between all recipes. The etch gas was set at 65 sccm and the deposition gas was set at 100 sccm. There was much more fluoropolymer than etch plasma in this recipe. The second recipe used a lower pressure, referred to as the low pressure recipe. All parameters were set the same except the pressure, gas flow, and time. The gas flows were reduced by about a half and the difference between each steps flow was less. The time was also reduced by a third for
Table 3.4. Listed are the etch parameters for different types of Bosch recipes. (a) high gas flow recipe (b) low pressure recipe (c) continuous recipe with cooldowns

<table>
<thead>
<tr>
<th>(a) Step</th>
<th>Temp. (°C)</th>
<th>Pressure (mTorr)</th>
<th>RF Power (W)</th>
<th>ICP Power (W)</th>
<th>SF₆ (sccm)</th>
<th>C₄F₈ (sccm)</th>
<th>Time (sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Depo.</td>
<td>15</td>
<td>30</td>
<td>12</td>
<td>750</td>
<td>1</td>
<td>100</td>
<td>9</td>
</tr>
<tr>
<td>Etch</td>
<td>15</td>
<td>30</td>
<td>40</td>
<td>750</td>
<td>65</td>
<td>1</td>
<td>9</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>(b) Step</th>
<th>Temp. (°C)</th>
<th>Pressure (mTorr)</th>
<th>RF Power (W)</th>
<th>ICP Power (W)</th>
<th>SF₆ (sccm)</th>
<th>C₄F₈ (sccm)</th>
<th>Time (sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Depo.</td>
<td>15</td>
<td>15</td>
<td>12</td>
<td>750</td>
<td>5</td>
<td>55</td>
<td>3</td>
</tr>
<tr>
<td>Etch</td>
<td>15</td>
<td>15</td>
<td>40</td>
<td>750</td>
<td>40</td>
<td>5</td>
<td>7</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>(c) Step</th>
<th>Temp. (°C)</th>
<th>Pressure (mTorr)</th>
<th>RF Power (W)</th>
<th>ICP Power (W)</th>
<th>SF₆ (sccm)</th>
<th>C₄F₈ (sccm)</th>
<th>Time (sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Depo.</td>
<td>15</td>
<td>20</td>
<td>40</td>
<td>750</td>
<td>5</td>
<td>80</td>
<td>3</td>
</tr>
<tr>
<td>Etch</td>
<td>15</td>
<td>20</td>
<td>40</td>
<td>750</td>
<td>80</td>
<td>5</td>
<td>7</td>
</tr>
</tbody>
</table>

the deposition step and shortened two seconds for the etching step. The third recipe ran continuously, referred to as the continuous recipe with cooldowns. This is compared to the two other recipes which incorporated oxygen cleans every 20 cycles. One cycle meant one deposition step and one etching step. The continuous recipe increased the pressure and gas flow relative to the low pressure recipe. The pressure was set at 20 mTorr, the deposition gas set at 80 sccm and the etch gas set at 80 sccm.

Figure 3.6 is a SEM image taken after the first etch test that used the high gas flow recipe for the G2 pattern. It aimed to reach a depth of about 100 µm and was about 3.4 µm away from that goal. Achieving the exact desired depth is difficult as characterized etch rates can fluctuate. However, differences of ± 10 µm will not affect the intended outcome of the gratings. The trench openings and tops of the structures were aimed to be 10 µm. Due to the curved nature of the SEM stage, the image was taken on an
Figure 3.6. SEM image of the high flow Bosch recipe. Typically the trenches are solid rectangles but due to rough cleaving, there was a cut through the trench walls. The measurements between trench walls and widths were about 10 µm. The etch depth was measured to be about 96.6 µm.

angle, therefore the measurements from the trenches and tops of structures were slightly stretched. The measurements taken in Figure 3.6 estimated the goal of 10 µm.

Figure 3.7 was the result of an etch test using the low pressure recipe for the G2 pattern. It was clear compared to Figure 3.6 that the recipe was not run long enough to achieve a depth of 100 µm. The recipe was stopped after 250 cycles to save time and calculate an etch rate quicker. The depth of 39.4 µm and total time were then used to calculate the etch rate of this recipe which is listed in Table 3.5. The tops of the structures in this recipe estimated the goal of 10 µm feature sizes. The beginning of this test started with the high gas flow recipe but then was switched to the low pressure recipe. This is marked by the 5.4 µm that was etched by the high gas flow recipe showing a clear transition to the low pressure recipe.
Figure 3.7. SEM image of the low pressure Bosch recipe. There is a visible distinction between where this test was switched from the high gas flow recipe to the low pressure recipe. The trench width was measured to be about 10 µm. The etch depth was measured to be about 39.4 µm but contributions from the low pressured recipe was 34 µm.

Figure 3.8. SEM image of the continuous Bosch recipe. The trench width was measured to be about 10 µm. The etch depth was measured to be about 41.3 µm.
Table 3.5. Etch characterizations of the three etch recipes for the G2 pattern. (a) high gas flow recipe (b) low pressure recipe (c) continuous recipe with cooldowns

<table>
<thead>
<tr>
<th>Recipe</th>
<th>Etch Rate ($\mu$m/min)</th>
<th>Selectivity (average)</th>
<th>Anisotropy (Sidewall angle °)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(a)</td>
<td>1.4</td>
<td>N/A</td>
<td>97</td>
</tr>
<tr>
<td>(b)</td>
<td>0.8</td>
<td>5</td>
<td>89</td>
</tr>
<tr>
<td>(b)</td>
<td>1.4</td>
<td>11</td>
<td>88</td>
</tr>
</tbody>
</table>

Figure 3.8 was the result of an etch test using the continuous recipe with cooldowns. Again, compared to Figure 3.6, the recipe was not run to a depth of 100 $\mu$m for the same previous reason. The depth of 41.3 $\mu$m and total time were used to calculate the etch rate for this recipe. The trenches and tops of structures tended to vary from 10 $\mu$m ± 4 $\mu$m. However, as mentioned earlier, measurements taken from these areas were crude and should be taken as estimates for judging feature sizes. Measurements from the optical microscope were more accurate and reliable.

The results of the etch tests are summarized in Table 3.5 and values were extracted from Figures 3.6, 3.7, and 3.8. The selectivity was a rough estimate taken from measurements during and at the end of etching. Accuracy of this value may be affected by fluoropolymer deposits in the depth pad measurement area (e.g. the high gas flow recipe). The sidewall angle measurements were taken using the same PCI software tool as measuring depths and features sizes. The high gas flow recipe resulted in a 1.4 $\mu$m/min etch rate, and sidewall angle of 97°. The low pressure recipe resulted in a slower etch rate of 0.8 $\mu$m/min, much lower selectivity of 5, and almost a sidewall angle of 90°. The continuous recipe resulted in an etch rate of about 1.4 $\mu$m/min, selectivity of 11, and sidewall angle of 88°.
3.2. Photolithography and etching tests at NIST CNST

The target dimensions were the same as Table 3.1 and what is illustrated in Figure 2.3. A different type of photoresist was used at the CNST, SPR 220 3.0. The process for the use of this photoresist in shown in Table 3.6. The only modification to the reference process included using AZ 300MIF developer for 60 seconds times two spray puddles instead of MF24A for 60 seconds with one spray puddle. This was the recommended development method by the CNST staff and the automated developer tool did not provide MF24A. This eliminated the need to perform development tests. An addition to the process was performing an O\textsubscript{2} etch after using a wet chemical to remove the resist layer. This cleaned up any re-deposited resist and was an extra layer of cleaning.

Table 3.6. Sequential process for use of SPR220 3.0 photoresist.

<table>
<thead>
<tr>
<th>Process step</th>
<th>Parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>Prime</td>
<td>YES oven with HMDS vapor</td>
</tr>
<tr>
<td>Coat</td>
<td>1500 rpm for 60 sec for &gt;3 ( \mu )m thickness</td>
</tr>
<tr>
<td>Soft Bake</td>
<td>115°C for 90 sec</td>
</tr>
<tr>
<td>Expose</td>
<td>dependent on design</td>
</tr>
<tr>
<td>Post Exposure Bake</td>
<td>115°C for 90 sec</td>
</tr>
<tr>
<td>Develop</td>
<td>AZ 300MIF 60 sec x 2 puddles</td>
</tr>
<tr>
<td>Photoresist removal</td>
<td>Remover 1165; 2 x 80°C immersion bath for 20 min then DIW immersion bath for 5 min then 5 min RIE O\textsubscript{2} etch</td>
</tr>
</tbody>
</table>

The photolithography test results are summarized in Table 3.7 and correct formation of the patterns are shown in Figures 3.9 and 3.10. The photoresist thickness used here was more than typical resists, but much less than what was attempted at the NFF. After dose tests, it was seen that 140 mJ/cm\textsuperscript{2} exposure dose was adequate for both patterns. The writing resolution was fixed at 1 \( \mu \)m and focus corrections do not need to be made. The
Table 3.7. Photolithography parameters for both the phase G1 and absorption G2 gratings using SPR 220 3.0 photoresist. In parentheses were the ranges tested for each parameter.

<table>
<thead>
<tr>
<th>Grating</th>
<th>Exposure Dose (mJ/cm²)</th>
<th>Writing Resolution (µm)</th>
<th>Focus Corrections (µm)</th>
<th>Development Time (sec)</th>
<th>Post Develop Bake</th>
</tr>
</thead>
<tbody>
<tr>
<td>G1</td>
<td>140 (80-155)</td>
<td>1</td>
<td>N/A</td>
<td>120</td>
<td>N/A</td>
</tr>
<tr>
<td>G2</td>
<td>140 (80-155)</td>
<td>1</td>
<td>N/A</td>
<td>120</td>
<td>N/A</td>
</tr>
</tbody>
</table>

Figure 3.9. Microscope image of G1 pattern after development. The period was measured as 7.79 µm. The distance between features was measured as 3.84 µm. The feature size was measured as 3.76 µm.

60 seconds times two spray puddle method worked for this resist and dose, consequently total development time was 120 seconds. A post develop bake was not needed for this kind of resist.

The etch test results are summarized in Table 3.8 and values were extracted from Figures 3.11, 3.12, 3.13, and 3.14. Since these wafers were <110> orientation, the cutting lines were on an angle to the pattern which can be seen in the FESEM images. Due to this, it made measurements of feature sizes and sidewall angles anomalous as the cut
Figure 3.10. Microscope image of G2 pattern after development. The period was measured as 20.68 $\mu$m. The distance between features was measured as 9.67 $\mu$m. The feature size was measured as 10.75 $\mu$m.

Figure 3.11. FESEM image of G1 pattern after 100 loops of Bosch process. The trench width was measured to be about 3.683 $\mu$m. The etch depth was measured to be about 32.15 $\mu$m.
Figure 3.12. FESEM image of G1 pattern after 260 loops of Bosch process. The trench width was measured to be about 4.039 μm. The etch depth was measured to be about 64.62 μm.

Figure 3.13. FESEM image of G2 pattern after 100 loops of Bosch process. The trench width was measured to be about 10.94 μm. The etch depth was measured to be about 39.36 μm.
Figure 3.14. FESEM image of G2 pattern after 265 loops of Bosch process. The trench width was measured to be about 11.48 µm. The etch depth was measured to be about 84.21 µm.

Table 3.8. Etch characterizations of the etch recipe for the G1 and G2 patterns, and photoresist.

<table>
<thead>
<tr>
<th>Pattern</th>
<th>Etch Rate (µm/min)</th>
<th>Selectivity</th>
<th>Anisotropy (Sidewall angle °)</th>
</tr>
</thead>
<tbody>
<tr>
<td>G1 (100 loops)</td>
<td>3.0</td>
<td>35</td>
<td>≈90</td>
</tr>
<tr>
<td>G1 (260 loops)</td>
<td>2.3</td>
<td>30</td>
<td>≈90</td>
</tr>
<tr>
<td>G2 (100 loops)</td>
<td>3.7</td>
<td>43</td>
<td>≈90</td>
</tr>
<tr>
<td>G2 (265 loops)</td>
<td>3.0</td>
<td>39</td>
<td>≈90</td>
</tr>
</tbody>
</table>

makes them angled. Nevertheless, the depth measurement was reliable which yields etch rate and selectivity calculations. Selectivity measurements were more accurate having access to a reflectometer to measure photoresist thickness after etching. An initial dose test was done for both patterns and these wafers were also used to calculate an etch rate. The deep silicon etch tool was set to 100 loops as a starting point. The etch rate from this test for the G1 pattern was 3.0 µm/min and for the G2 pattern was 3.7 µm/min. The
selectivity for the G1 and G2 patterns was 29 and 34, respectively. The next test was to pattern the wafers to their full scale. The G1 pattern was approximately 60x60 cm², and the G2 pattern was approximately 50x50 cm². Using the etch rate from the previous tests, an estimation about the number of loops needed to reach the desired depths was made. For the G1 pattern, it was estimated 260 loops could reach a depth of 77 µm. For the G2 pattern, it was estimated 265 loops could reach a depth of 100 µm. The etch rate from this test for the G1 pattern was 2.3 µm/min and for the G2 pattern was 3.0 µm/min. The selectivity for the G1 and G2 patterns was 31 and 39, respectively. Both test pad wafers (100 loops), yielded a photoresist etch rate of approximately 0.086 µm/min. The second round of test wafers (260 and 265 loops), yielded a photoresist etch rate of approximately 0.076 µm/min. Figures 3.12 and 3.14 show that the number of loops was underestimated by 13-16 µm for the desired depths. Linear extrapolation was used to make another number of loops estimate that reached the desired depths. The angle measurement tool within the FESEM software was crude for accurate sidewall angle measurements, but through visual analysis across the wafer the tests produced nearly 90° walls.
Chapter 4. Discussion and conclusion

4.1. Discussion

4.1.1. Workflow efficiency

We fabricated phase and absorption diffraction gratings through microfabrication techniques. We went through a series of photolithography and etching tests to determine the process and equipment needed to create the gratings. Two nanofabrication facilities were used, the locally accessible CAMD facility and the national NIST facility. Here we summarize key features and capabilities: We utilized a mask-less direct write lithography tools with, CAMD exposure times in excess of 4 hours. The computer connected to the photolithography tool at CAMD NFF struggled with memory storage for dense patterns causing slow writing. The NIST lithography tool required exposure times less than 10 minutes. We utilized reactive ion etching at CAMD with a plasma etch tool taking several days and three fills of the liquid nitrogen reservoir. Reactive ion etching performed at NIST was with a Bosch deep silicon plasma etch tool taking 45 minutes. The recommendation for CAMD NFF to achieve similar etch times to NIST CNST is to tune the parameters for the etch recipes to achieve faster etch rates. The goal is an etch recipe that will finish within an hour especially since the liquid nitrogen reservoir can maintain the desired temperature for hours but not days. Increasing the power and gas flows by 3× and 5×, respectively would bring the recipes closer to what was used at NIST CNST.

4.1.2. Photolithography validation efforts

We tested various parameter combinations such as doses, foci, resolutions and development times to achieve the desired feature size and periodicity with sharp corners.
Using the AZ-12XT-20PL-10 photoresist, we found from the test pads ranging from 270 to 420 mJ/cm$^2$ that 400 mJ/cm$^2$ accurately formed the G1 pattern. Additionally, between 120 to 550 mJ/cm$^2$ that 300 mJ/cm$^2$ formed the G2 pattern. For each dose set, the writing resolution and focus was varied as well. For example, three test pads were set to 300 mJ/cm$^2$ but varied in focus: -10, 0 and +10 µm. In another row, this was replicated but with a different writing resolution. This was applied to the range of doses stated earlier creating a matrix of test pads. We immersed the wafer after exposure in developer and set different times to inspect which test pad fully developed the exposed photoresist.

Using the SPR 220 3.0 photoresist, we found from the test pads ranging from 80 to 155 mJ/cm$^2$ that 140 mJ/cm$^2$ accurately formed the G1 pattern. Additionally, between 80 to 155 mJ/cm$^2$ that 140 mJ/cm$^2$ formed the G2 pattern. No writing resolution, focus corrections or development time tests were necessary.

4.1.3. Photolithography alternatives: Masks

We obtained reliable inspection results from the Filmetric (only available at NIST), optical microscope and SEM tools. With the NIST tools, we were able to calculate a silicon etch rate and selectivity for each pattern. Although the values were acceptable, further improvements can be made to increase the selectivity by using a thicker photoresist. However, this presents difficulties with the lithography tool as a defocus correction will need to be applied. This will lead to the need of a focus test similar to what was done at CAMD NFF. The SPTS deep Silicon etcher does allow for one type of hard mask: SiO$_2$. Having a hard mask such as SiO$_2$, would greatly increase selectivity [18, 19] and avoid the need to run a focus test. A RIE step would need to be done before the deep silicon
etch in this case. The photoresist mask, SPR 220 3.0, was narrowly thick enough to reach each desired depth with their respective feature sizes. Therefore, goals to etch deeper with these parameters may not be feasible. Absorption gratings with deeper trenches for our feature size would have increased visibility. To achieve deeper results, it is suggested to use a thicker photoresist or hard mask as described above.

4.1.4. Photolithography alternatives: Pattern and mask design

Another way to increase visibility via lithography design is to increase the ratio of trench length to supporting structure length. The purpose of these structures were to keep the trenches from collapsing and giving them more stability. For this project, the ratio was 10:1 where the next fabrication could use 20:1 or 50:1. Increasing this ratio should decrease the number of completely transmissive areas thereby increasing interferometry visibility. Other lithography designs such as the Sunray design, shown in Figure 4.1, could be tried to increase visibility, however fabrication of this design is complex and was beyond the scope of this project.

Figure 4.1. The photoresist mask (on left) and final grating (on right) using the Sunray design. The red arrows point to stability areas for the fragile structures. Image courtesy of Trimborn et al. [20].
4.1.5. Etching using the Bosch Process

The commonalities and differences seen amongst the etch recipes created at CAMD NFF were illustrated in the SEM images. Not included as an image was the full wafer after etching. The areas of exposed silicon appeared rough and dark, especially noticeable in the depth pad area. This is why it was not possible to measure an accurate resist etch rate for the high gas flow recipe. After shortening the deposition time and decreasing the pressure, the areas previously “burnt” looking were much “cleaner”. There was too much fluoropolymer being deposited onto surfaces which caused re-deposits onto the wafer. There were also specks observed on the surface of the wafer in arbitrary locations. The pressure decrease helped to control the directionality of the ions hence the more vertical sidewalls seen in the later recipes. However, there were signs of an undercut forming and this greatly slowed the etch rate. The etch recipe at NIST CNST uses a higher pressure so there is room to improve without sacrificing time. Their etch tool is capable of handling higher power and gas flows, but further testing needs to be done to confirm if CAMD NFF’s tool can do the same.

We were able to use a well-defined etch recipe at NIST CNST that formed vertical walls and deep trenches. Some characterization needed to be done because the results from one etch recipe was dependent on the design of the pattern. The area of exposed silicon, feature sizes, pattern density, and aspect ratio, are a few parameters that depend on the etch rate. The more exposed silicon, the etch rate slows because more free radicals will be “eaten” up. This can lead to a discrepancy in etch rate between dose tests and the full size as dose tests tend to be designed to take up less area. Feature sizes affect the etch
rate in that smaller feature sizes have slower etch rates. A narrower opening will interact with fewer free radicals than a wider opening would. This explains why the G1 pattern etches at a slower rate than the G2 pattern because it has a smaller feature size (e.g. 3.85 \( \mu m \) vs. 10 \( \mu m \)). Pattern density in this project was very dense which also contributed to slow etch rates. This dependency is similar to the amount of exposed silicon because a more dense pattern will have more exposed silicon than a less dense pattern. Aspect ratios are the etch depth to feature size where high ratios would signify deep etches with small feature sizes [19, 26, 27, 28]. The trench to feature size ratio in this project is considered a relatively high aspect ratio which can also attribute to slow etch rates.

The effects of needing well-balanced etch parameters can be seen between the work done at CAMD NFF versus NIST CNST. The silicon etch rate for the G2 pattern averaged amongst the three recipes was 1.2 \( \mu m/min \) at the CAMD NFF compared to 3-3.7 \( \mu m/min \) at NIST CNST. It was learnt at the CNST that each etch process should have its own tool to avoid contamination of other gases. However, the etch tool at the CAMD NFF performs various etch processes in addition to the Bosch process which means there could have been contamination throughout Aim 1. At the least, a thorough chamber clean needs to be run before and after a Bosch process to remove any contamination using the appropriate kind of gas to clean. Such chamber cleaning was attempted for all of the Bosch etch recipes at the CAMD NFF. However, a clean should only be run before and/or after the process, not during which was applied in two of the recipes to help with matching network issues. It was necessary at the time to enable etching beyond 20-30 cycles, but it does change the chamber conditions for each run.
4.2. Conclusion

The immediate next step of grating assembly for the interferometry system is to develop an absorption grating holder and alignment system for tiling several gratings together to create a large FOV. Thus far, an aluminum plate has been cut out with two windows approximately 150x50 mm$^2$. Six gratings, cut out approximately 55x55 mm$^2$ will be laid onto the plate with three gratings in each window. In the end, another aluminum plate will need to be created for six more gratings. Each aluminum plate corresponds to capturing one lung. Additional alignment pieces are created through microfabrication with two parallel trenches that match a trench of identical width on the gratings. The trench on the gratings is etched superior to the grating area and spans across the wafer. Hypodermic needles will be laid into the trenches of the alignment pieces and gratings, with separate needles for the top and bottom row of gratings. This method targets minimizing the translational movement of the gratings. Testing to be done at this step includes optical alignment with a laser and visualizing the fringe pattern produced.

Another crucial next step is to fill the absorption gratings trenches with metal. The metal will be a highly x-ray absorbing material such as Gd$_2$O$_3$. This transforms the x-ray fringe pattern to a lower frequency thereby allowing the detectors to resolve the pattern within its spatial resolution capabilities. This nanoparticle filling method has been done at other universities (i.e. Munich Technical University and Tohoku University) [29, 30, 31]. The Gd$_2$O$_3$ is in the form of a nanoparticle/polymer slurry and this substance is troweled into the grating structures.

Once the interferometer has been fully constructed and the diffraction gratings are
aligned and secured in place, testing for image quality and other performance metrics will be done. This should take the project closer to the goal of conducting a pilot study of COPD patients. The fabrication of diffraction gratings is a tedious and specialized step in interferometry, but they make it possible to form key images such as phase contrast and dark-field necessary for imaging lung diseases. The fabrication process can be learned, replicated, and altered as evident by this research project.
Appendix A. DMO MicroWriter ML3 Pro Instructions

Sample requirements

- No minimum wafer size.
- 195 mm x 195 mm maximum writing area.
- 230 mm x 230 mm x 15 mm maximum wafer size.

Load a wafer

- Press button with red light, located on bottom right of the front of the tool to open the latch.
- Carefully place wafer inside following the white guidelines, shut the door and press the button with the red light again.

Align wafer

- On the right hand side of the software, there is a text box to enter a wafer thickness. Enter your wafer thickness here and press the green check mark button.
- Next, focus the microscope onto the wafer by clicking the checker pattern button. Focus at all desired magnifying levels (e.g. 10x, 20x, 50x). The button below the thickness text box, applies an autofocus which can be done instead of the checker pattern button. The bar below the autofocus button should be almost fully green as this means the microscope is focused.
- Now to center the wafer, click the tab at the top “Wafers”. This will bring up a window to input the wafer diameter. Enter the wafer diameter in mm. The edge exclusion option excludes a certain outer diameter when centering. Once all this is inputted, press the button with the wafer and centered target. Again, the bar below this button will fill fully green once completed.

Prepare pattern

- Upload CleWin file of pattern by clicking “File” and “Upload file”. The job list will show the pattern on the screen.
- Parameters to change at this step are quality, dose correction, focus correction, resolution, wafer position, or job list builder for dense patterns. It is up to the user to choose what is best for the desired pattern.
Exposure

- Input a resist sensitivity ("Dose") and global focus. Once inputted, click the "Tools" tab and select "Start rendering".

- Once the pattern has been rendered and a time estimate has been given, click the green traffic light to begin the exposure. If the process needs to be stopped, click the red traffic light.

- To take the wafer out, press the button with the red light and use tweezers to remove. Quickly transition to development or leave wafer in machine until ready. The photoresist is light sensitive so it could ruin the formation of the pattern.
Appendix B. Oxford ICP-DRIE System 100 ICP180 Instructions

Sample requirements and preparing for use

- Wafers up to 6” in diameter.
- Si, SiO\textsubscript{2}, SiN\textsubscript{x}, PMMA, etc. wafers are acceptable.
- The sample chamber needs to be cooled to a specific temperature, typically around 15°C. Twist open the liquid nitrogen tank knob which is outside the cleanroom, on the backside of the space. The fill knob should be opened, not the vent knob. Once opened, this can take 20 minutes to several hours. To speed up the process, before every use, disconnect the liquid nitrogen line and shoot N\textsubscript{2} gas into the fill line from inside the cleanroom.
- Check the physical loadlock by looking into the circular window.
- Check that all the yellow handles to the right of the machine are on open (there should be four).
- Check that the machine is on and if it is not, press the green square button located on the front facing panel of the machine.
- Sign into the PTIQ software with the credentials given by the cleanroom manager.

Load a wafer

- First check if the loadlock needs to be vented, as it may have been pumped down by the previous user. To vent, at the bottom of the screen click “Manual” then “Transport” then “Vent”.
- Once loadlock is vented, a message will pop up confirming this and now the sample can be placed in the loadlock. The loadlock is located to the left with a small circular window and black handle. Holding onto the black handle, lift up and to the left to swing open the door. Carefully and quickly, place wafer in by making contact with the two prongs and facing the wafer flat towards the process chamber. Close door shut.
- To vacuum seal the loadlock, at the same location as the “Vent”, click “Pump”.

Condition the chamber

- Before starting a recipe, clean the process chamber with O\textsubscript{2} plasma.
• There is an option on the “Automatic” tab where you select a recipe to include an O\textsubscript{2} plasma clean recipe.

• Select the box for “Condition chamber” and select an O\textsubscript{2} plasma clean recipe.

• This recipe can be run whenever, even as the main recipe with a dummy wafer.

• If you notice the plasma doesn’t strike (glow) or the bias voltage is roughly zero, something is not right. One solution is to change the recipe to first strike at a higher pressure then add another step after to set the pressure lower.

**Clean the chamber**

• After finishing a recipe, clean the process chamber with O\textsubscript{2} plasma.

• Same as conditioning the chamber, select the box for “Clean chamber” and select an O\textsubscript{2} plasma clean recipe.

**Run a recipe**

• There are many recipes already created within the PTIQ software. They may be found under the “Recipes” tab. These recipes appear in the drop-down menu on the “Automatic” tab.

• On the “Automatic” tab, it will ask to enter a Bosch ID. Enter an identifier name perhaps something related to the kind of pattern on the wafer.

• After this, click on the recipe drop down menu and select the desired recipe.

• As described above, this is also where selections for conditioning and post-process cleaning can be made.

• At this point, the recipe should be ready and the “Run” button should change color. Click this button when ready to run recipe.

• Click “Manual” then “ICP” and this will display all pertinent information to the gases, pressures, powers, temperature, recipe details, etc. It is recommended to navigate to this page during the recipe to monitor if there is enough gas, if the plasma strikes and generates a bias, if the temperature has reached the goal temperature, and if there are any errors.

• At this point, everything should run automatically and finish. If there are any errors or problems, call for the cleanroom manager.
Unload a wafer

- This works the same as loading a wafer except in reverse.

- If the recipe successfully runs, it will automatically complete this step. In the event the recipe fails, to manually unload, first make sure the “Process Gauge” is about $4 \times 10^{-5}$ mTorr and click “Unload”. Once unloading into the loadlock, click “Vent”.

- If done using for the day, make sure to “Pump” to keep the loadlock sealed and prevent outside foreign contaminants from entering.

Replacing liquid nitrogen tanks

- Twist fill knob close and with a wrench loosen only the top most fitting to the right to disconnect the line.

- Wheel the tank outside to the liquid nitrogen filling station. Bring glasses, gloves and wrenches. For those who have sensitive hearing, ear plugs are recommended.

- Once at filling station, feel knob on filling station to make sure it is not vibrating as that means the line is closed. Take hooked connector and attach to fill nozzle on tank by tightening the top most fitting to left. Leave two notches open when tightening.

- Twist open the vent knob first (4 times), fill knob second (4 times), and filling station knob third. The pressure in the tank will begin to go down and escape out the vent knob which is now open. The filling can take awhile (about 30 minutes) especially during warm months. The filling station knob will need to be adjusted according to the pressure. Maintain the pressure around 16-18 and stop the filling at 12-13 volume.

- Once reached, quickly twist filling station knob close first (it might vibrate a little), fill knob second, and vent knob third. Do this quickly so the pressure does not decrease too much.

- Detach the hooked connector by turning handles slightly to let pressure escape first then fully disconnect. There may be ice on the hooked connector-fill nozzle joint which can be brushed off with gloves. Return the hooked connector with ice and all onto stand.

- Wheel tank back inside to its original location. Re-attach the plasma etcher chamber line and tighten only the top most fitting to the left to connect line. Two notches can be left open so that the next person can disconnect it.
References


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Vita

Rachael Blair was born in Milwaukee, Wisconsin in 1999. Rachael grew up in Muskego, Wisconsin where she attended high school and graduated in 2017. She went to Carthage College and earned a Bachelor of Arts in Physics in 2021. After learning about medical physics in her undergraduate studies, she wanted to pursue that career path. She enrolled in LSU’s Medical Physics Master of Science program. Subsequent to her anticipated graduation in 2024, she will begin residency at the University of Minnesota.