Appendix A

EXPERIMENTAL PROTOCOLS

A.1 Polystyrene Nanofilm Preparation

The quality of the solid nanofilms which were then liquefied at the start of the molten nanofilm experiments detailed above is of critical importance. In the case of the flat plate experiments described in Ch. 3, Ch. 4, and Ch. 5, the presence of defects can lead to nonlinear growth or dewetting, which obscures the desired instability and corrupts the measured wavelengths and growth rates. Defects will also destroy the optical properties of the micro-optical devices described in Ch. 6 and Ch. 7 because they are scattering centers which induce optical loss. The preparation of high quality nanofilms starts with pure solvents to make sure that additional contaminants are not introduced. Specifically, the polystyrene (PS) solvent was toluene and it dissolves both standard washbottles and plastic filters. This causes contamination in the final films, so any materials used during nanofilm preparation were either glass or metal. To reduce all sources of contamination precise filtering was performed during preparation and while dispensing the solution for spin coating. The nanofilm preparation procedure has been divided into two sections: one devoted to the initial creation of the solution and the other devoted to the spin coating process. Note that all of these steps were accomplished in labs which were not a cleanroom, although this would have been helpful. Instead, polyester filters were placed in the air registers of the room and changed annually. To further reduce the level of airborne contaminants a HEPA filter (Alen BreatheSmart) was run continuously on its highest setting near the fume hoods.

A.1.1 Dissolving and Filtering Polystyrene in Toluene

1. Begin preparations by donning a lab coat, nitrile gloves, safety glasses, a hair net, and a surgical mask. The use of a hair net and surgical mask are to reduce the amount of contamination that is introduced from external sources into the solution.

2. Rinse a glass bottle and a glass beaker with toluene and dry with nitrogen. **DO NOT USE TOLUENE FROM A WASHBOTTLE.** Toluene dissolves the bottle and leads to contaminants in the resulting nanofilms. Instead, pour

the toluene from the large 4 L glass bottle into a smaller beaker and use it directly from the beaker. Refill this secondary beaker as needed throughout the process. The cleansed beaker will be the initial mixing vessel and the bottle will house the final solution. The toluene in the washbottle is only for cleaning the spin coater.

3. Rinse a small metal scoopula with toluene and dry with nitrogen. Tear off a small section (approximately 2” x 2”) of weighing paper and put it onto the balance. Tare the balance.

4. Weigh out the appropriate amount of polymer onto the weighing paper for your desired solution concentration. Several different weight percent solutions and typically make at least 10 mL of solution at a time. In 10 mL of toluene, the amount of PS that is needed for the corresponding weight percent is listed: 1% = 87.9 mg; 2% = 177.6 mg; 4% = 362.5 mg; 8% = 756.5 mg.

5. Fill the initial mixing beaker with 10 mL of toluene and carefully pour the polymer on the weighing paper into this solution. Cover and leave solution to dissolve while performing the next three steps.

6. Clean the glass syringe with stainless steel Leur lock tip and glass plunger (Cadence Science) by rinsing in pure toluene from a glass beaker. Separate the two pieces and clean them individually. Then dry them using nitrogen gas.

7. Disassemble and clean each part of the stainless steel filter holder (Whatman) with toluene individually. Dry each component with nitrogen and then re-assemble in the same order (large donut gasket beneath the steel mesh, flat spacer above steel mesh) with the Anodisc filter (0.02 micron pore size) inserted directly above the steel mesh and below the flat spacer. Attach the filter holder assembly to the end of the 5 mL glass syringe. The holder will screw into the metal end of the syringe. Both of these seals should be at least finger tight and should not leak during normal filtering operations.

8. Clean the metal scoopula with toluene, dry it with nitrogen and put it away. If the polymer solution is not completely mixed at this point, it can be swirled by hand to promote mixing. In cases of high PS weight percentage, the solution can also be sonicated for one to two minutes. The solution can also be heated

on a hotplate to promote mixing, but care must be taken so that toluene does not evaporate during this process and so generally sonication is preferred.

9. Once the solution has mixed completely, remove the plunger from the 5 mL glass syringe and pour about half the 10 mL solution into the syringe. Replace the plunger and apply just enough pressure so that the solution passes through the filter at a rate of approximately 1 drop per second. Do not exert too much pressure to prevent the Anodisc filter from cracking which will cause solution to leak out of the connection between the filter holder and the syringe. Refill the syringe and repeat this process until the entire solution has been filtered. After filtering approximately two full syringes, the filter will tend to be saturated with material and the pressure required to filter the solution will increase markedly. This is more of a problem at high weight percentages (e.g. at 8% this typically occurs after one full syringe) and can be remedied by cleaning the filter holder and inserting a new Anodisc filter, as in Step 7.

10. Label the solution with the date, the preparation parameters such as the type of polymer and weight percentage, and any other relevant information.

11. Clean up the rest of the materials by disassembling the filter holder, disposing of the Anodisc filter in the glass disposal container, and rinsing each piece of the filter holder with toluene. Dry the pieces and then reassemble the holder. Wrap the filter holder in aluminum foil to prevent contamination from dust and store in the cabinet. Rinse the plunger and glass syringe separately with toluene and then dry with toluene. Store in the box and return it to the cabinet. Dispose of any remaining toluene that is not in the large 4L bottle by cleaning the initial mixing beaker and then disposing of any other toluene in the waste container.

A.1.2 Spin Coating Nanofilms

1. Begin preparations by donning a lab coat, nitrile gloves, safety glasses, a hair net, and a surgical mask. Clean the glass syringe and the filter holder as in Steps 6 and 7 in Sec. A.1.1.

2. Rinse a glass bottle and a glass beaker with toluene and dry with nitrogen. **DO NOT USE TOLUENE FROM A WASHBOTTLE.** Toluene dissolves the bottle and leads to contaminants in the resulting nanofilms. Instead, pour

the toluene from the large 4 L glass bottle into a smaller beaker and use it directly from the beaker. Refill this secondary beaker as needed throughout the process. The cleansed beaker will be the initial mixing vessel and the bottle will house the final solution. The toluene in the washbottle is only for cleaning the spin coater.

3. Grab a clean 2” silicon wafer from the pack by grasping the flat edge and place it onto the spin coater. Try to stay as close to the edge as possible while maintaining mechanical stability because anywhere the tweezers touch becomes contaminated with defects in the resulting nanofilm. Silicon wafers are generally cleanest as they come from the manufacturer and that more defects are introduced by attempting to clean them (with solvents, piranha, HF, etc.).

4. Turn on the spin coater (Cee-100, Brewer Science) and select the program by pressing ‘Run’ and then the program number. There is an initial centering step that should be completed before the solution is applied to the wafer. This step can be completed by pressing ‘Start’ once. If the wafer is not centered, it can be adjusted and tested again by pressing ‘0’. All of the programs for the spin coater should be listed on the blackboard next to the fume hoods. They can all be modified and the interested user should consult the manual for a full listing of the possible options. If a program is changed, then the list on the blackboard should be updated.

5. Once the wafer is centered, draw solution slowly into the 5 mL glass syringe without the filter holder attached. Take care to have a constant pull speed to avoid introducing extraneous bubbles into the solution. Dispense three to five drops of solution back into the container to fill the dead space in the filter holder. Dispense the solution slowly onto the wafer opposite the flat edge to avoid spreading defects. Dispense close to the wafer so that a meniscus forms and the solution can be dispensed continuously without the formation of drops. To improve reproducibility, dispense solution until the point when the entire wafer is covered by solution. An alternative technique to this is to always draw the same amount of solution into the syringe (1 mL, for example) and then dispense the entire syringe. When utilizing this approach, care must be taken to fill the dead space of the filter holder before measuring the correct amount of solution in the syringe.

6. Close the spin coater and start the spin coating process which is typically a 30 second spin at 3000 RPM with an acceleration of 1000 RPM/s. Once the chuck has stopped rotating, visually inspect the quality of the film, looking for dust and comets in the film. After inspection, immediately place the wafer into a 2" wafer carrier. Label the carrier with the date, your initials, the type of solution, and any other information relevant to this wafer. The sample is now ready for film height measurement with the ellipsometer or deformation in the experimental setup.

A.2 Film Thickness Measurements through Ellipsometry

The thickness of transparent thin films can be measured through ellipsometry and this technique was used extensively above to measure the thickness of our PS nanofilms. However, there are several important limitations of the instrument, a Rudolph Auto EL III Ellipsometer. First, it cannot measure the thickness of thicker films (greater than about a micron). Second, the film thickness measurements are periodic in the film height and if the initial guess is not sufficiently close to the true value, it can report the wrong thickness. This effect can be mitigated by measuring at another wavelength (this ellipsometer can measure at 405 nm, 546.1 nm, and 632.8 nm), since the periodicity is wavelength dependent. If the same thickness is measured at two different wavelengths then it is probably the correct value for your film. The third limitation of this instrument is that it has difficulty with multiple transparent layers. When the refractive index difference between two layers is small, there is relatively little reflection from that interface which means that the observed signal is small. Even with these limitations, the ellipsometer was quite effective at measuring the thickness of PS nanofilms on silicon substrates. The steps in a typical thickness measurement are detailed below.

1. Begin preparations by donning a lab coat, nitrile gloves, safety glasses, a hair net, and a surgical mask.

2. Turn on the ellipsometer using the key on the lower left part of the ellipsometer, flip the switch on the upper left part of the ellipsometer which controls the illumination source, and flip the rocker switch on the upper right part of the front panel which controls the alignment light. Load the sample on to the stage. Adjust the position of the sample so that the ellipsometer spot hits a section that is flat and free from defects.

3. Adjust the sample height. Start by pulling the magnifier at the bottom of the periscope outwards. If the sample is at the correct height the circle should have crisp edges and be in focus. If it is not, then turn the large wheel underneath the stage until the bright spot is focused.

4. Adjust the sample tilt. Push the magnifier at the bottom of the periscope towards the ellipsometer as far as it will go. The bright spot should be centered on the reticle, completely within the black circle. If it is not, then adjust the three smaller screws beneath the stage until it is. After adjustment, all screws should be snug so that tilt is not affected by small vibrations.

5. After hitting enter to confirm that the sample was inserted and aligned, press the RUN button and select the appropriate program from the white board opposite the ellipsometer. Each of these programs can be modified and for a full list of details and options consult the ellipsometer manual. There are two main types of programs that we use. Programs 00 through 02 calculate both the film thickness and the refractive index while programs 03 through 05 only calculate the film thickness and assume the refractive index is fixed. When possible use 03 through 05 because they are slightly more accurate and repeatable, but the refractive index of the film material must be known from a different measurement to use these. When running a program it will typically prompt the user to provide a guess for the film thickness and the film refractive index. Provide a reasonable guess for the film thickness, otherwise an incorrect value could be reported due to the periodicity of the technique. Typically, the approximate thickness can be estimated by eye using the film color. There are several tables around the lab which show color as a function of film thickness for PS films on silicon wafers. They can also be derived using the equations of thin film interference.

6. Pay attention to the ellipsometer display as the printer does not work effectively, for the ellipsometer in the lab. The film thickness (and refractive index if the chosen program calculates it) value will flash briefly on this screen and should be written down. Generally, these measurements are quite repeatable so if the value was not written down, then the program can be run again. The ellipsometer will also provide the raw values which can be used if something more complex than a thickness measurement is desired.

A.3 SU-8 UV Photolithography on Sapphire

This set of instructions will detail how to make the photoresist spacers which determine the size of the air gap above the molten nanofilm. When making the spacers, great care was taken to make sure that they were all the same height and free from defects which would disturb the parallelism. This is in contrast to most photolithographic applications where the surface of the photoresist is unimportant since it is typically used as a binary mask. For the flat plate experiments described in Ch. 3, Ch. 4, and Ch. 5 only a set of spacers was deposited onto the sapphire. For the micro-optical device fabrication described in Ch. 6 and Ch. 7, another set of deposition steps was required which defined the pattern on the superstrate that influenced the film growth. This second pattern was of a different height than the spacers, which is why two distinct photolithography exposures were necessary. These fabrication steps were performed in a class 1000 cleanroom to reduce contamination.

1. Start with a clean sapphire window. This could be a new window directly from the manufacturer (Meller Optics) or one that has been cleaned with piranha (see Sec. A.4). Typically, if a window has a damaged or unwanted pattern it can be removed with piranha and then rinsed with acetone followed by a rinse with isopropyl alcohol (IPA). This should be visually inspected to ensure that it is free from dust and other contamination. Small amounts of dust can usually be removed with a quick blast from the nitrogen guns in the hood.

2. Prepare for future baking steps by setting two hotplates in the cleanroom to 65 °C and 95 °C.

3. Attach the sapphire window to a larger glass slide using Shipley S1813 photoresist to reduce the presence of the edge bead. The reduction of edge bead makes the spacers more uniform. To do this, place a drop of S1813 underneath a window and bake at 95 °C for 10 minutes for 3/8" diameter windows (15 minutes or longer for the 1” diameter windows). Remove from the hot plate and let cool to approximately room temperature. At this point, there is no danger of overbaking the resist as the longer it bakes, the better the adhesion of sapphire to the glass. Note that the use of specifically S1813 is not required. This resist was used because it would not dissolve in SU-8 developer or the IPA rinse which immediately followed. As such, multiple spinning and exposure steps could be achieved sequentially without reapplying the adhesion photoresist.

4. OPTIONAL: Level the spin coater to improve the uniformity of the spacer heights. If this has been done recently and the spin coater has not been disturbed, then this should only need to be done infrequently (perhaps annually for the spin coater in Watson 153 and much more frequently for the spin coater in the cleanroom). Take a silicon wafer and put it onto the desired chuck. Dispense IPA using a plastic syringe held vertically in the middle of the wafer and watch which direction it spreads fastest. Then, adjust the feet of the spin coater to raise that direction. Alternatively, that direction can be shimmed with a piece of Technicloth. Spin the wafer briefly to remove the IPA. Repeat this procedure of dispensing IPA and then leveling until the IPA spreads evenly in all directions. At this point, the spin coater is level and spinning should produce films uniform to within a few percent of the total film thickness over the whole wafer.

5. Load the sapphire window on the glass slide into the spin coater. Dispense the SU-8 solution steadily and evenly to cover the entire window. With the higher viscosity solutions, areas which were not covered initially tend not to be covered after spinning, so it is important to have complete coverage initially. Spin the window at 3000 RPM for 60 seconds with a 3000 RPM/s acceleration. This can be varied and will depend on both the SU-8 solution viscosity and the desired film thickness, but it is a good place to start. The standard SU-8 2010 solution can be diluted using cyclopentanone according to the table in the MicroChem datasheet [32] to produce different viscosities. NOTE: when using 2” square glass slides as substrates, do not use the PTFE holder as the vacuum is not strong enough to keep the glass slide from breaking at 3000 RPM. Instead, place it directly on the chuck with no holder in place.

6. Pre-bake the window for 1 minute at 65 °C; then transfer it immediately to the 95 °C hotplate for 2 minutes. Remove from the hotplate and let the sample cool to approximately room temperature. These baking times have been fairly resilient for films of any thickness up to 10 microns, but for more precise times recommended by MicroChem for different thickness regimes consult the datasheet [32].

7. Load the sample into the mask aligner and align the middle of the sample beneath the desired pattern. Expose the sample for 60 seconds for thin films (<2 microns) and 90 seconds for thick films (>2 microns). Alignment of the pattern can be achieved by using the large enclosing ring which can be seen

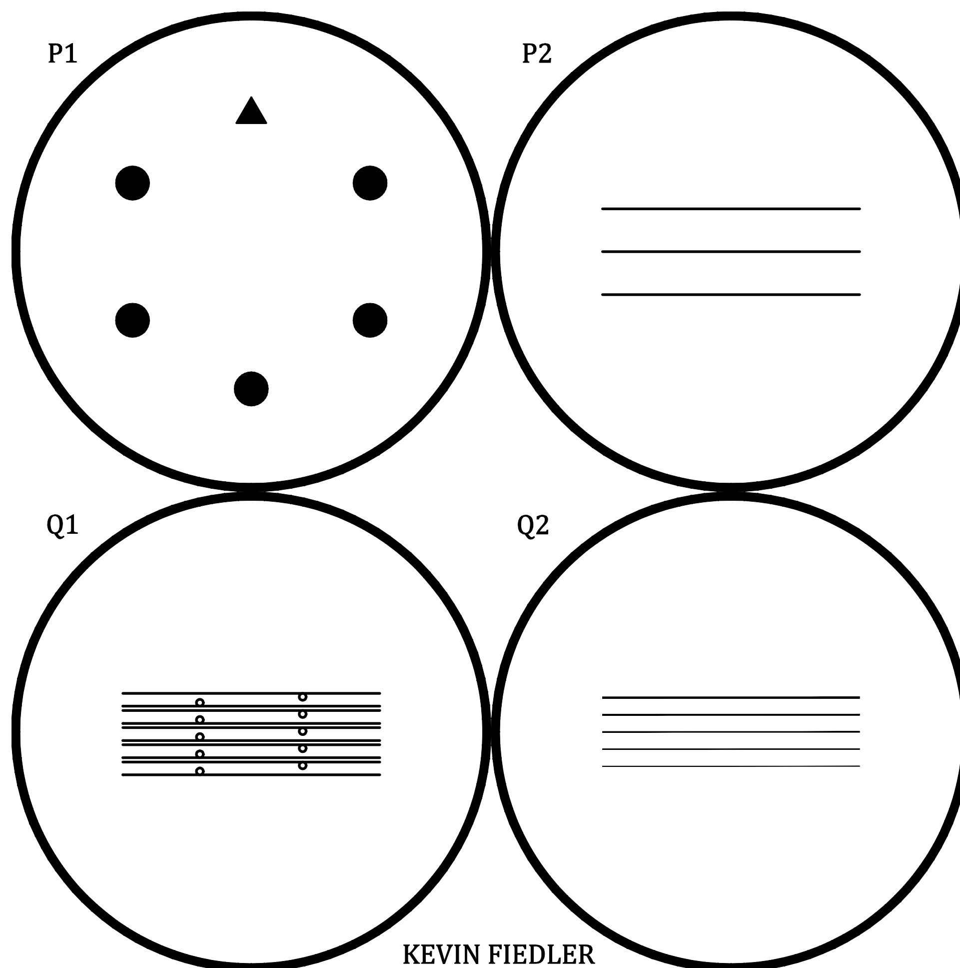
around each pattern in Fig. A.1. After coarse alignment of the sample under the pattern, precise alignment can be achieved by translating the sample in the vertical direction until it is barely visible through the top of the outer ring. Then, the top of the window should be aligned with the top of the pattern. Move the sample so that it is just hidden under the pattern and shift your focus to the bottom of the pattern. Count the number of turns on the translation micrometer that it takes for the sample to just become visible at the bottom of the pattern. Go backwards half the number of turns it took to reach the bottom and the sample should be aligned with the center of the pattern as long as the micrometers do not have too much slop when changing directions.

8. After exposure, remove the sample from the mask aligner and post-bake the window for 2 minute at 65 °C; then transfer it immediately to the 95 °C hotplate for 4 minutes. Remove from the hotplate and allow the sample to cool to approximately room temperature.

9. Develop the sample by complete immersion and agitation for 30 seconds in SU-8 developer. Immediately dip the sample in IPA for approximately 15 seconds and then rinse with fresh IPA. The sample can then be dried with nitrogen. If the features are not completely developed, put it back into developer for approximately 15 seconds and repeat the process. Thicker films will typically require longer development times, but it is better to err on the side of less development because the sample can always be developed more. The features are not fully developed if there is a milky residue on the sample when you initially put it in the IPA. The residue will also be visible under the microscope as a set of small dots. Once development is complete the features should be crisp and well-defined. With thin, high-aspect ratio features, the development and drying process can destroy the desired pattern. If this becomes a problem, do not agitate the sample during the SU-8 developer or IPA immersion steps. Additionally, do not use the nitrogen gun to dry the sample; let it air dry instead.

10. OPTIONAL: Perform another set of spinning and photolithography exposure steps to define a pattern. When making windows with both spacers and a pattern, Step 5 can be repeated with a different viscosity solution to create a pattern with a smaller height. This is more efficient than proceeding to Step 12 and hard baking both the spacers and pattern separately because the sapphire window only needs to be attached to the glass substrate once and only needs

Figure A.1: SU-8 photomask example



Example of an SU-8 photomask used to make the features on the sapphire windows. The dark regions are areas which would be transparent in the chrome on glass pattern. All these patterns are designed for 1" diameter sapphire windows and are not drawn to scale. Pattern P1 is the hexagonally arranged spacers. The triangle allows for easier orientation of subsequent pattern steps. The remaining patterns are examples of waveguides.

to be hard baked once. The spacers should be patterned first because they are large and will not be affected by the subsequent spinning step. If very fine patterns were made in the first step, then the high viscosity solution for the spacers could remove the features when it is applied.

11. Once the spacers and the pattern are developed completely, remove the window from the glass substrate using acetone. When dissolving the S1813 with acetone, try not to submerge the SU-8 pattern in acetone and remove the window as quickly as possible once it comes off the glass substrate. A moderate amount of pressure is required to remove the window from the glass substrate. If the window sits with the pattern submerged, the acetone will destroy the SU-8 pattern. This sensitivity of SU-8 to acetone will persist until the window has been hard baked. One technique to avoid this issue is to put down a thin layer of acetone so that it barely covers the glass substrate. Then it will not come over the top of the sapphire window into contact with the spacers or pattern. This thin layer can then diffuse under the window to remove the photoresist and allows for a little more flexibility in the time required to remove the window.

12. Hard bake the windows at 200 °C for 2 hours to cure them. This step does not necessarily have to be done in a cleanroom. If it is not done in a cleanroom, then cover the hotplate but do not form a seal. This will allow the SU-8 to degas and cure without being contaminated with dust from the environment.

13. Measure the height of the spacers and the pattern, if it exists, with a profilometer (Ambios XP2). Do this after the window has been hard baked, otherwise the force from the profilometer tip can scratch the SU-8 and leave behind a pattern which will influence the growth of the features in the molten films.

A.4 Cleaning with Piranha Solution

To clean off the SU-8 patterns from sapphire windows or remove PS from a quartz window we typically use piranha solution. This is a mixture of sulfuric acid and hydrogen peroxide, typically in an approximately 3:1 ratio. It will dissolve most organic material, including elemental carbon. Additionally, it will dissolve metals, so it is important that you use PTFE (Teflon) tweezers instead of metal ones. The mixing process is highly exothermic, so it is very important that this procedure is performed in a fume hood on a hot plate and that the mixing container is not held

by hand while pouring. It is also crucial to have another person in the lab for safety purposes.

1. Begin preparations by donning a lab coat, nitrile gloves, safety glasses, a hair net, and a surgical mask. Additionally, an acid apron must be donned when working with acids.
2. Measure approximately 75 mL of sulfuric acid (H_2SO_4) into a beaker. Note that the viscosity of sulfuric acid is significantly different than the viscosity of water and it will not pour in the same way.
3. Pour the sulfuric acid into the glass container where the samples will be cleaned. This container should be located on top of a hot plate during the mixing process because it is quite exothermic.
4. Measure approximately 25 mL of 30% concentrated hydrogen peroxide (H_2O_2) into a different beaker than the one which contained the sulfuric acid.
5. Add the hydrogen peroxide to the sulfuric acid in the mixing container and allow the solution to cool. It will typically steam slightly.
6. Rinse the hydrogen peroxide beaker with deionized water and pour the mixture into the sulfuric acid beaker. Rinse the sulfuric acid beaker with more water and dispose of the resulting solution into the hazardous waste bottle.
7. Insert the samples to be cleaned into the piranha solution and let them sit for approximately 20 minutes. It will be clear that the solution is working if the organic matter turns brown/black and bubbles slowly. Then, the sample is clean once the brown/black material has disappeared. The solution will lose its effectiveness over time because the hydrogen peroxide is light sensitive, so use it soon after it has been prepared. Additionally, the solution can saturate if there is a lot of organic material to be removed. In this case, not all the of the brown/black material will disappear and the samples should be cleaned again.
8. Remove the clean samples from the piranha solution and transfer them to a beaker filled with deionized water using the PTFE tweezers. After all the samples have been removed put them in the rinse bath and put it under a steady stream of deionized water. While the water is overflowing the container,

remove the samples through the stream of deionized water. This process will prevent any residual organic matter which accumulates on the top of the rinse bath from redepositing on the sample as it is removed from the rinse beaker.

9. After removal from the rinse bath, dry the sample with either Technicloth or nitrogen from the fume hoods. The samples are now clean and ready for use.

10. To dispose of the piranha solution after cleaning, pour it into the waste bottle. Then, rinse the mixing container with deionized water and pour this into the waste container as well. Flush all the beakers and mixing containers that have been used with excess water and dry them using paper towels. Leave them in the fume hood to finish drying.

A.5 Wafer Cleaving for Waveguide Isolation

As detailed in Ch. 7, polymeric waveguides were fabricated on substrates which were composed of a 5 micron wet thermal oxide layer grown on a <100> silicon wafer. To produce optical quality end facets which would allow light to be coupled in and out of the waveguide, the waveguide substrate was cracked. Due to the nanoscale heights of the waveguide, they cracked cleanly and simultaneously with the silicon wafer. The cleaving of silicon is a common process in semiconductor processing where a wafer is typically scratched by hand on the unpolished side with a diamond scribe, then flipped over and cracked from the polished side. This technique posed two difficulties for the specific application of cracking waveguides. First, the waveguides were fabricated on the polished side of the wafer and putting the wafer with the polished side down could scratch or otherwise damage the waveguides. Second, the scratches defining the cleave axis must be aligned with the location of the waveguide and this is difficult to do without being able to see the position of the waveguide as the scratches were made. As such, a few modifications to the traditional process were made and are detailed below.

1. Begin preparations by donning a lab coat, nitrile gloves, safety glasses, a hair net, and a surgical mask. Lay out a fresh piece of Technicloth on which the wafer will be cracked. Place the room temperature wafer on the Technicloth with the fabricated waveguides facing up.

2. Scratch the top side of the wafer along the crystal axes near the wafer flat. Align one scratch near the left end of the waveguide, as close to the end as possible while avoiding defects. Align another scratch near the right end of

Figure A.2: Wafer cleaving diagram

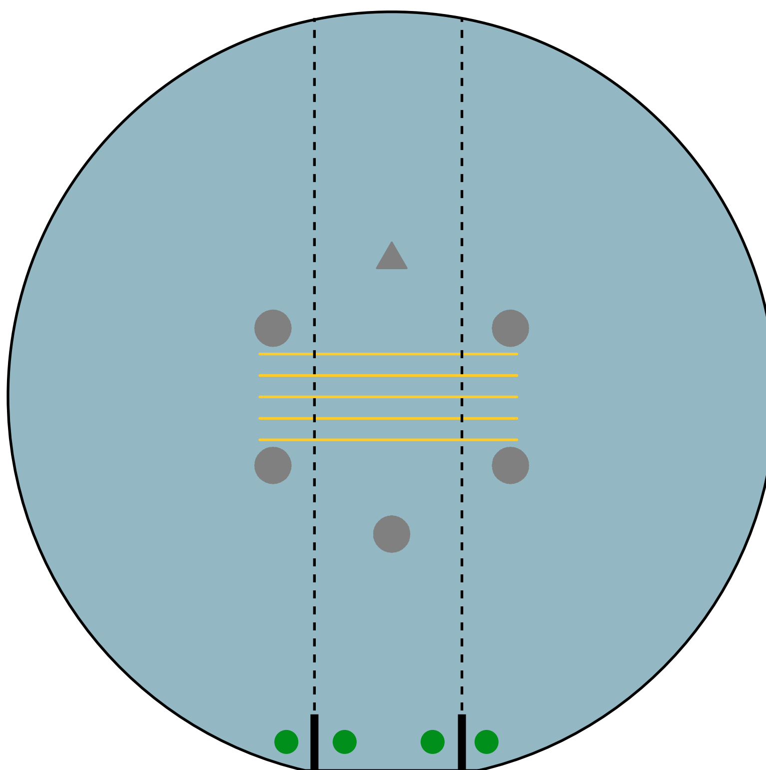


Diagram of the approximate geometry used for cleaving a wafer. The scratches in the wafer are denoted by the thick solid black lines perpendicular to the wafer flat. The dotted lines represent the propagation direction of the cleave which is perpendicular to the waveguides (denoted by the yellow lines). The hexagonally arranged gray objects are the spacers and the green dots are locations where pressure should be applied to propagate the crack through the wafer.

the waveguide, as close to the other end as possible. The scratches only need to be a couple millimeters long and should not produce large amounts of silicon dust. Ideally, the scratches would be aligned to the crystal axes of the wafer (perpendicular to the flat for <100> wafers) and would only require one motion. The approximate location of good scratch locations are shown by the solid black lines near the bottom of Fig. A.2.

3. Insert a straightened paper clip or staple underneath the scratch so that it is aligned parallel to the desired cleave direction. Press on either side of the scratch until the wafer cracks. Press relatively close to the position of the scratch and can use the end of tweezers or toothpicks to push down on the wafer. The approximate location to apply pressure is shown by the green dots in Fig. A.2. Too much pressure will cause the wafer to shatter and if the wafer feels difficult to crack, make the scratches deeper. Typically only two

cleaves are needed to create input and output facets on the waveguide. The undeformed regions near the top and bottom of the wafer make convenient handles to maneuver and mount the sample.

A.6 Abbe Refractometer Refractive Index Measurements

The dispersion of an optical material is a critical parameter when designing structures because the refractive index will determine the required dimensions and other properties. To this end, an Abbe refractometer was used to measure the refractive index of PS at several discrete wavelengths. An Abbe refractometer uses total internal reflection to measure the refractive index of a sample which is sandwiched between two prisms. However, this particular Abbe refractometer (Vee Gee Instruments C10) was originally intended solely for measurement at a wavelength of 589 nm. This instrument was designed using two double Amici prisms to compensate for the dispersion of a broadband illumination source. To allow measurement at discrete wavelengths, this compensating prism was removed and three single wavelength illumination sources were chosen. The measurement wavelengths were 405 nm, 532 nm, and 633 nm. Using three distinct wavelengths allowed the refractive index of materials to be fit as a function of wavelength through the Cauchy equation

$$n(\lambda_{\text{opt}}) = B + \frac{C}{\lambda_{\text{opt}}^2} + \frac{D}{\lambda_{\text{opt}}^4}. \tag{A.1}$$

In this equation, $n(\lambda_{\text{opt}})$ is the refractive index, λ_{opt} is the optical wavelength and B, C, and D are fitting constants. For the PS used above, these coefficients were $B_{\text{PS}} = 1.574$, $C_{\text{PS}} = -0.002248\ \mu\text{m}^2$, and $D_{\text{PS}} = 0.001813\ \mu\text{m}^4$.

In addition to the modifications which were performed on the measurement instrument, the choice of low molecular weight PS also posed challenges. Typically, solid samples must be large and optically polished to provide a smooth interface which can be placed on the refractometer prism. Attempts to fabricate a macroscopic polished sample of PS were unsuccessful because the PS stuck to the molds, such as aluminum foil or glass. It was also too brittle to withstand the mechanical stress of polishing. As a result, molten PS was poured directly on to the prism and allowed to cool within the setup, as will be detailed in the steps below.

For each of the measurement wavelengths the refractometer was calibrated using solutions with known refractive indices. The calibration samples were common liquid samples of acetone, ethylene glycol, toluene, and water. The measurement steps enumerated below were performed for each calibration liquid at each measurement

wavelength. Then the measured refractive index values were compared to literature values and the deviation of the measured refractive index value from the literature value was computed. These deviations were averaged over all the calibration samples independently at each wavelength to provide offsets to use when measuring unknown samples. A full listing of the calibration liquids, their measured refractive indices, and the literature values can be found in Table A.1.

1. Begin preparations by donning a lab coat, nitrile gloves, safety glasses, a hair net, and a surgical mask. Measurements are easier when performed with the room lights turned off, but the lights can be left on during sample preparation in Step 2.

2. Place the sample on the lower refractometer prism and close the refractometer to bring the upper prism into contact with the sample. For liquid samples the solution will be dispensed directly on the prism and should cover the entire prism face. In the case of solid samples, an index matching fluid is typically required to provide good optical contact between the prism and the sample. For the prisms used in this refractometer, 1-bromonapthalene was the index matching fluid. Certain solid samples, such as the calibration sample, will be too large for the upper prism to close, but this should not cause measurement difficulties as long as some illumination passes through the sample. As mentioned above, the PS sample was melted on an external hotplate at approximately 100 °C. The molten PS was removed from the hotplate and immediately poured onto the lower prism and the upper prism was quickly brought into contact with the PS, exerting mild pressure on the still molten sample. Since this sample was initially molten, an index matching fluid was not required to achieve optical contact.

3. Turn off the room lights if this has not already been done. Select the desired illumination wavelength and turn the source on. The source should already be aligned to illuminate the aperture of the upper prism which will transmit through the sample. If it is not, then the steering mirrors can be adjusted. Also note that the aperture should be covered with a Kimwipe which functions as a diffuser. The presence of the diffuser more evenly illuminates the sample and reduces speckle in the transmitted light [71]. This allows for a more precise measurement of the refractive index.

Table A.1: Abbe refractometer calibration data and PS refractive index

	405 nm	532 nm	632.8 nm
Calibration Samples - Measured			
Acetone	1.3325	1.3619	1.3718
Ethylene Glycol	1.4044	1.4335	1.4423
Toluene	1.4865	1.5014	1.5056
Water	1.3038	1.3363	1.3464
Calibration Samples - Literature	405 nm	532 nm	632.8 nm
Acetone [72]	N/A	1.3615	1.3578
Ethylene Glycol [73]	1.4412	1.4335	1.4308
Toluene [73]	1.5211	1.5006	1.4940
Water [74]	1.3427	1.3350	1.3317
Mean Offset	0.0368	-0.0007	-0.0130
Measured PS Values	1.5886	1.5894	1.5930
Final PS Refractive Indices	1.6254	1.5887	1.5800

4. Looking through the eyepiece, adjust the knob on the side of the refractometer so that the middle of the "X" is aligned with the line of demarcation between the light and dark regions. The light region should be the same color as the illuminating beam; if it is not, then there is light contamination from an external source (or a different source is still on). The line should be very distinct in proportion to the wavelength spread of the source.

5. Read the refractive index of the sample off the scale at the bottom of the field of view when looking through the eyepiece. With the room lights turned off, the scale is very dim, so it is usually necessary to turn on the small lamp to the left of the refractometer which illuminates the measurement scale. After measuring this value, compensate the measurement using the calibration values in Table A.1. Typically it is easiest to load a sample, measure all three wavelengths and then load another sample for measurements as opposed to keeping the illumination source constant and cycling through samples.

A.7 Optical Coupling to Polymeric Waveguides

To couple light into the optical waveguides whose fabrication and characterization were detailed in Ch 7, a coupling setup located at the Jet Propulsion Laboratory was used. The basic setup is diagrammed in Fig. 7.4 and this section will detail how to effectively image the output of the waveguide modes.

1. Mount the waveguide sample which has already been cleaved on to a holder. This was typically done using SEM tabs (Ted Pella #16084-1, 12 mm outer diameter) on a long aluminum cantilever. The SEM tabs can be reused between samples although the back of the sample must be free of thermal paste for it to adhere. The back of the sample should be cleaned with IPA and a Kimwipe. Then the sample should be attached to a piece of Scotch tape several times to remove any remaining residue. Cleaning the back of the sample will prolong the life of the SEM tabs.

2. Align the optical fiber to the focal point of the microscope objective so that the smallest spot size is imaged by the Basler cameras. If the sample is currently configured to use a PBS and two cameras, then this step can also ensure the spot is centered simultaneously in both cameras and that they are in the same focal plane. Mark the location of the front of the microscope objective and the fiber tip on the TV/screen which is used to visualize the setup. This gives a good estimate for the focal distance and focal plane of the microscope objective in the present configuration.

3. OPTIONAL: Adjust the polarization controller to change the relative amount of light in the TE and TM polarizations. For certain measurements, it can be desirable for the amount of light to be equal in both the TE and TM polarizations while for other measurements it can be better for more power to be in one of the two polarizations. This is the most convenient point to adjust the polarization because the sample has not yet been inserted. Move the PBS mounted on the optical rail away from the cameras and directly beneath the reflection photodiode (Thorlabs PDA55) and swing the transmission photodiode into place. Cover the setup with the cardboard box cover to reduce environmental effects and adjust the gain of the photodiodes so that they are not saturated. Turn on the multimeters connected to the photodiodes and adjust the polarization controller to the desired polarization settings. When

the polarization has been adjusted, move the PBS back into position between the cameras.

4. Move the optical fiber from the focal plane of the microscope objective and leave plenty of room for the sample to be inserted. Attach the holder to the three axis translational stage. At this point, do a coarse alignment of the optical fiber to one of the waveguides on the chip, both laterally and vertically. This can mostly be done by eye, but will save time during the precision alignment later. Bring the waveguide sample into the focal plane of the microscope objective and align the waveguide to the mark which was made before. Depending on the lighting and camera the waveguide might not be visible. If the waveguide isn't visible this is not concerning as the position will be adjusted later when an image is formed.

5. Bring the optical fiber into coupling position at the input facet of the waveguide sample. This should be done carefully so as to not crash the optical fiber into the sample. Align the sample under 5x optical magnification. For this step, it is necessary to be able to see the interference fringes in the waveguide. If the waveguide is not visible, change the lighting until it is. This is best done with illumination through a dedicated microscope, but can be done with a lamp or the fluorescent room lights. When aligning the fiber, start by moving it to the center of the waveguide. Put a screen after the microscope objective but well before the PBS to get a rough idea of the light passing through the setup in the current configuration. Bing the fiber to the plane of the waveguide from above. There will be many closely spaced fringes on the screen which get further apart as the correct plane is approached. There will also probably be pieces of dust on the waveguide surface that are visible through the observation microscope in the correct plane for alignment. If light is coupled into the waveguide, then it should light up and this will be easiest to see with the room lights turned off. There should also be a faint horizontal line and a bright spot on the screen after the microscope objective. Once coupling to the waveguide has occurred, remove the screen.

6. Adjust the lateral position of the microscope objective so that the bright spot on the screen is aligned to the active part of the camera. Do not adjust the focal distance until the spot is on the camera. Otherwise, finding the correct focal plane will be very difficult. The position or opening of the iris might need to be adjusted, but after this has been done once it should be pretty close for

subsequent testing. If nothing is visible on the camera, then it typically helps to turn up the camera gain significantly so that scattered light is visible. Once the spot is on the camera, adjust the gain and the separation of the microscope objective and the waveguide sample until the image is in focus. Turn down the camera gain so that the image is not saturated and then adjust the separation distance until a clear image is formed.

7. Move the optical fiber to find the desired mode. Typically different modes can be accessed by small lateral translations of the fiber. The vertical position of the fiber can also be adjusted until the maximum coupling to the mode. At this point, images of the modes can be recorded or the transmitted power through the waveguide can be measured using the photodiodes.