Supplement: Fabrication protocol

The present series of protocols details how to fabricate both silica microsphere and microtoroid resonant cavities. While silica microsphere resonant cavities are well-established, microtoroid resonant cavities were only recently invented.[1] As many of the fundamental methods used to fabricate the microsphere are also used in the more complex microtoroid fabrication procedure, by including both in a single protocol it will enable researchers to more easily trouble-shoot their experiments.

Protocol Text:

1.) Microsphere Fabrication

1.1) Select a small amount (approximately 5 inches) of optical fiber, strip ~1.5" cladding from one end and clean with either methanol or ethanol (Figure 1a, b).

1.2) If available, cleave the end with an optical fiber cleaver. If not available, cut with wire cutters or scissors such that ~0.5" is left. The advantage of using an optical fiber cleaver is that it produces a very smooth, uniform cut as in Figure 1b. Excessive roughness or defects from a cut may cause uneven reflow, lowering the quality factor of the resulting spheres.

1.3) Expose the cleaned fiber end to 3W of CO_2 laser power focused to a ~500μm diameter spot size for ~1 second (Figure 1c, d, e). This produces spheres ~200μm in diameter; however, the size can be tuned by increasing or decreasing the diameter of the optical fiber. Slightly adjusting the laser intensity may also be necessary to reflow larger or smaller spheres.

2.) Microtoroid Fabrication

2.1.) Design and make a photomask with dark, solid circles, in the spacing and diameter of your choice. It is important to note that the toroids produced will be 25-30% smaller than the circles on the mask. For example, a solid circle with a diameter of 100 microns will produce a toroid with a diameter of approximately 75microns. Also, it is recommended to leave at least 1-2mm of space between each circle and at least 5mm of space between arrays of circles and around the edges of the mask. Since the sample wafers must be carefully handled with tweezers, it is important to leave space for the tweezers to grip without damaging the toroids. The extra space also provides room for a tapered optical fiber to couple light into the finished devices, and allows samples to be cut into smaller arrays more easily. For this procedure, we used a mask with rows of 160μm diameter circles ~1mm apart, with ~5mm of space between each row of circles. The finished toroids are approximately 110μm in diameter.

2.2) Begin with silicon wafers with a 2μm thick layer of thermally grown silica. Cleave the wafers to fit the desired microdisk pattern on the photolithography mask, leaving room for photoresist edge bead. Note that at the beginning of fabrication, it is usually most convenient to etch several arrays of circles on larger pieces of silicon wafers (~several cm x several cm). Larger wafers allow photolithography and BOE etching of more samples at a time, and are more easily handled with tweezers. Later, before the XeF_2 etching step, it is recommended to cleave the larger wafers into smaller arrays to allow faster, more uniform XeF_2 etching.

2.3.) In a fumehood, thoroughly clean the wafers by rinsing with acetone, methanol, isopropanol, and deionized water. Blow the samples dry using a nitrogen or filtered compressed air gun, and place them on a hot plate set to 120°C for at least 2 minutes to dry.

2.4.) After letting the wafers cool, place them in a flammable/solvent fumehood and expose to HMDS for 2 minutes using the vapor deposition method. A simple vapor deposition method: put a few drops of HMDS in a small 10ml beaker, and then cover the wafers and small beaker with a larger glass container to hold the vapor.

2.5.) Place a sample on a spinner with an appropriately sized mount. Using a dropper bottle or syringe and filter, apply photoresist to the sample. Spin coat S1813 photoresist onto each sample for 5 seconds at 500rpm, followed by 45 seconds at 3000rpm. Edge bead removal is not needed if the wafer is sufficiently large so that the edge bead does not interfere with the patterning.

2.6.) Soft bake the photoresist on a hot plate at 95°C for 2 minutes.

2.7.) Using a UV mask aligner and the desired photomask, expose the photoresist-covered samples to a total of $80mJ/cm^2$ of UV radiation.

2.8.) Immerse the samples in MF-321 developer to remove the photoresist which was exposed to UV light. While developing, closely watch as the photoresist is removed from the wafer and dissolved. It is important to stir/swish the container constantly during this process to ensure the photoresist is removed uniformly. For the given parameters, the photoresist takes approximately 30 seconds to develop.

2.9.) When most of the unwanted photoresist has dissolved in the developer, rinse the samples thoroughly under running water, gently blow dry the samples using a nitrogen or air gun, and inspect the samples with a microscope to ensure all undesired photoresist has been removed. If needed, the samples can be immersed again in developer; however, one should be careful not to overdevelop the samples as the desired photoresist patterns could also be damaged. (If the desired patterns are damaged or defective, the photoresist can be removed with acetone and steps 2.1-2.9 can be repeated again).

2.10.) After developing, thoroughly rinse the samples in running water, gently blow dry the samples, and hard bake them on a hot plate at 110°C for 2 minutes. This step heats the photoresist above its glass transition temperature, reflowing the photoresist and partially repairing roughness which occurred during the developing process.

2.11.) Using Teflon containers and the necessary protective equipment, immerse the samples in improved buffered oxide etchant (BOE). BOE contains HF, which etches the silica not covered by photoresist to form circular silica pads on the silicon wafer (Figure 2a-c). Improved buffered HF produces a smoother etch, minimizing roughness in the resulting silica circles. While it is possible to mix buffered HF starting with 49% HF, this can lead to highly variable results as typically only small quantities are made.

2.12.) After approximately 15-20 minutes (depending on the patterns, sample sizes and number of samples), remove the samples from the BOE using Teflon tweezers. Carefully rinse the samples in running water. The silica has been removed when the samples become hydrophobic.

2.13.) After etching, rinsing, and drying the samples, inspect them using an optical microscope. Check to make sure the desired patterns have been etched completely and all the unwanted silica has been removed. If needed, return the samples to the BOE for further etching. One should be careful not to overetch the samples, or the circular patterns underneath the photoresist may be damaged.

2.14.) Once BOE etching is complete, thoroughly rinse the samples in deionized water and blow dry. If the samples are on large pieces of silicon wafer, it is also recommended to cut them (using a dicing saw or diamond scribe) into smaller pieces with individual rows of silica circles. Individual rows of circles are etched more quickly and uniformly in the XeF_2 etching step (2.16). Silicon dust produced by the cutting is removed during cleaning in the next step.

2.15.) Remove the photoresist by rinsing with acetone, methanol, isopropanol, and deionized water, and dry the samples using a nitrogen gun and heating on a 120°C hot plate for at least 2 minutes.

2.16.) Using a XeF_2 etcher, undercut the silicon beneath the circular silica pads to form silica microdisks (Figure 2d-f). The amount etched should be approximately 1/3 of the silica circle's size, so that the resulting microdisk's pillar is approximately 1/3-1/2 of the total disk diameter, as determined by inspection with an optical microscope. The number of XeF_2 pulses and the duration of each pulse depends on the amount of silicon in the chamber and the type of XeF_2 etcher used.

2.17.) After XeF_2 etching, expose the samples to a focused CO_2 laser beam at approximately 12W intensity for ~3 seconds or until a smooth toroid is formed (Figure 2g-i). Depending on the exact size of the disk and the amount of XeF_2 undercut, a slightly higher or lower intensity and exposure time may be needed to form a microtoroid. It is important that the center of the laser beam and the center of the microdisk are aligned, so that the silica microdisk will form a smooth, circular microtoroid.

Additional Comments:

As with any optical structure, maintaining cleanliness at every step of the fabrication process is of critical importance. As there are numerous textbooks written on the topic of lithography and fabrication, the suggestions below are not intended to be comprehensive, but highlight a few of the more common issues researchers have faced.[2,3]

Because the uniformity of the microtoroid's periphery is determined by the uniformity of the initial disk, it is very important to pattern very circular disks. Common problems specific to the microtoroid are: 1) pixilation of photo-masks, 2) poor photolithography (under or over exposure, under or over developing, and rough or uneven etching), and 3) poor adhesion of the photoresist to the silica; here we address each issue individually.

It is very important to acquire high resolution photo-masks. While low resolution photomasks or ink-jet photomasks are readily available, these will result in "pixilated" or jagged circles with will not reflow correctly, resulting in non-circular toroids. The present protocols give UV exposure times for very specific photoresist film thicknesses at specific UV intensities. If different film thicknesses are used or if the photoresist is expired, then a different exposure time will be necessary. It is also advisable to calibrate one's photoaligner to ensure the correct UV exposure is given. Similarly, the time required in developer may vary as it is specific to the photoresist's film thickness and assumes that the photoresist is fully exposed. Finally, if the silica is not exposed to HMDS immediately before the photoresist is applied, the photoresist will not adhere well to the wafer. As a result, when the sample is etched using BOE, it will experience a severe and non-uniform undercut.

There is one other issue which also frequently arises with the toroid fabrication process and is related to the XeF_2 undercutting step. Because of the high degree of selectivity of XeF_2 for silicon over silica, the XeF_2 will not directly etch the native oxide which is inherently present on the silicon wafer. Therefore, it is important to make sure to minimize the potential growth of such an oxide and to further eliminate any further oxide growth by thoroughly purging the XeF_2 etch chamber with Nitrogen. If this is not done, the XeF_2 etch will be extremely rough or pocketed.

Additionally, in order to form a circular structure, it is very important to use an isotropic silicon etchant. While XeF_2 is the most commonly used etchant in the microtoroid fabrication process, there are others, such as HNA which is a mixture of hydrofluoric acid, nitric acid and acetic acid.[3] However, because it contains HF, it is not as selective for silicon as XeF_2 is, and the etching of the silica must be taken into account.

The CO_2 laser reflow process used must be done very precisely to successfully fabricate microspheres and microtoroids. One standard and simple reflow setup is shown in Figure 4 with a list of parts in Table 4. There are many possible ways to build such a setup, and the layout and parts used can vary. However, the design must satisfy two important criteria. First, the distance between the sample and CO_2 laser's focusing lens must be equal to the lens's focal length, so that the sample is located in the focus of the laser beam. Second, the uniformity of the CO_2 laser across the spot and the placement of the device in the center of the spot are extremely important. This requires that all of the free space optics are in alignment, and of course, free space optics can drift with temperature and humidity fluctuations. Example devices which were fabricated with incorrectly aligned optics are in Figure 5. To help avoid these alignment problems, cameras and stages can be used to allow easier, more accurate positioning of a sample under the beam. While using an optical table or vibration isolation is not required, having the reflow components integrated and secured on a breadboard can improve alignment.

If a CO_2 laser is not available, alternative reflow methods can be used. For the microsphere, a hydrogen torch could be used as an alternative method. If this approach is used, it is very important to follow all requisite safety protocols when building the reflow set-up, such as incorporating a flashback arrestor on the hydrogen tank and using a hydrogen torch, to eliminate the potential risk of an explosion. Typically, when this approach is used, a similar imaging system to that described for the CO_2 laser set-up is used for monitoring the reflow process. However, a hydrogen torch will not work for the microtoroid, as the melting temperature of silicon is less than that of silica. The CO_2 laser overcomes this problem, because silica strongly absorbs the laser light while silicon does not. Therefore, we have found that reflow with a properly aligned CO_2 laser beam allows us to obtain the most consistent reflow needed for high quality factor microsphere and microtoroid resonators.

The pair of methods presented here enable the fabrication of ultra-high-Q silica resonant cavities. As a result of their long photon lifetimes, these devices have numerous important applications, particularly within the biological sciences.

Tables of specific reagents and equipment:

Table 1: Microsphere Fabrication Materials

Name of the part	Company	Catalogue number	Comments
Fiber scribe	Newport	F-RFS	Optional
Optical fiber	Newport	F-SMF-28	Any type of optical fiber can be used.
Fiber coating stripper	Newport	F-STR-175	Wire strippers can also be used
Ethanol	Any vendor	Solvent-level purity	Methanol or Isopropanol are substitutes

Table 2: Microtoroid Fabrication Materials

Name of the reagent	**Company**	**Catalogue number**	**Comments**
Silicon wafers with 2μm thermally grown silica	WRS Materials	n/a	We use intrinsic[8], <100>, 4” diameter
HMDS (Hexamethyldisilazane)	Aldrich	440191	
Photoresist	Shipley	S1813	
Developer	Shipley	MF-321	
Buffered HF - Improved	Transene	n/a	The improved buffered HF gives a smoother, better quality etch than plain BOE or HF
Acetone, Methanol, Isopropanol	Any vendor	99.8% purity	

Table 3: Microtoroid Fabrication Equipment

Equipment Name	**Manufacturer**	**Catalogue number**	**Comments**
Spinner	Solitec	5110-ND	Any spinner can be used.
Aligner	Suss Microtec	MJB 3	Any aligner can be used.
XeF_2 etcher	Advanced Communication Devices, Inc.	#ADCETCH2007	

Table 4: CO_2 Laser Reflow Set-up

Name of the part	**Company**	**Catalogue number**	**Comments**

CO_2 Laser	Synrad	Series 48	
3-Axis stage	OptoSigma	120-0770	Available from other vendors as well.
Si Reflector (1” diameter)	II-VI	308325	Available from other vendors as well.
Kinematic gimbal mount (for Si reflector)	Thor Labs	KX1G	Available from other vendors as well.
Beam combiner (1” diameter)	Meller Optics	L19100008-B0	Available from other vendors as well.
4” Focal length Lens (1” diameter)	Meller Optics or II-VI		Available from other vendors as well
Assorted posts, lens mounts	Thor Labs, Newport, Edmund Optics or Optosigma		
Zoom 6000 machine vision system	Navitar	n/a	Requires generic USB camera and computer for real-time imaging. This is purchased as a kit.
Focuser for Zoom 6000 system	Edmund Optics	54-792	Available from other vendors as well.
X-Z Axis Positioners for Zoom 6000	Parker Daedal	CR4457, CR4452, 4499	CR4457 is X-axis, CR4452 is Z-axis, 4499 is mounting bracket.

References:

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