Submission ID #: 68706
Scriptwriter Name: Sulakshana Karkala
Project Page Link: https://review.jove.com/account/file-uploader?src=20955378

Title: Fabrication and Characterization of High-Q Silicon Nitride Membrane Resonators

Authors and Affiliations:
Atkin D. Hyatt, Oscar A. Flores, Aman R. Agrawal, Charles A. Condos, Dalziel J. Wilson

Wyant College of Optical Sciences, University of Arizona

Corresponding Authors:
Atkin D. Hyatt (atkindavidhyatt@arizona.edu)
Email Addresses for All Authors:
Atkin D. Hyatt (atkindavidhyatt@arizona.edu)
Oscar A. Flores (oscaraf@arizona.edu)
Aman R. Agrawal (amanagrawal@arizona.edu)
Charles A. Condos (cacondos@arizona.edu)
Dalziel J. Wilson (dalziel@arizona.com)

Author Questionnaire

1. Microscopy: Does your protocol require the use of a dissecting or stereomicroscope for performing a complex dissection, microinjection technique, or something similar? **No**

2. Software: Does the part of your protocol being filmed include step-by-step descriptions of software usage? **Yes**

If **Yes**, we will need you to record using screen recording software.

We recommend using the screen capture program OBS. JoVE's tutorial for using OBS Studio is provided at this link: https://review.jove.com/v/5848/screen-capture-instructions-for-authors?status=a7854k

3. Filming location: Will the filming need to take place in multiple locations? **No**

Current Protocol Length
Number of Steps: 26
Number of Shots: 57

Introduction

Videographer: Obtain headshots for all authors available at the filming location.

1.1. **Atkin Hyatt:** Our research explores and exploits the unique optical and mechanical properties of free-standing silicon nitride thin-film resonators.

1.1.1. INTERVIEW: Named Talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B.roll:2.5*

What new scientific questions have your results paved the way for?

1.2. **Atkin Hyatt:** Our fabrication procedure allows us to rapidly design and deploy novel devices, leading to accelerated progress in nanomechanics.

1.2.1. INTERVIEW: Named Talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B.roll:3.5*

What research questions will your laboratory focus on in the future?

1.3. **Atkin Hyatt:** Looking ahead, our group aims to study the quantum limits of angular displacement, develop high-sensitivity on-chip accelerometers, and probe new physics at the table-top scale.

1.3.1. INTERVIEW: Named Talent says the statement above in an interview-style shot, looking slightly off-camera.

Videographer: Obtain headshots for all authors available at the filming location.

Testimonial Questions (OPTIONAL):

Videographer:

- ***Please ensure that all testimonial shots are captured in a wide-angle format, while also maintaining sufficient headspace, given that the final videos will be rendered in a 1:1 aspect ratio.***
- ***Also, kindly note that testimonial statements will be presented live by the authors, offering their spontaneous perspectives.***

How do you think publishing with JoVE will enhance the visibility and impact of your research?

1.4. **Atkin Hyatt, Graduate Research Assistant**: The fabrication of the niche devices used in our research involves many delicate and nuanced steps, some of which are often left out of traditional publications and process flow diagrams. While this may demonstrate a high-level view of the procedure, nanofabrication remains a significant obstacle to entering the field. By publishing our procedure as a video guide, we hope to pull back the curtain in a way not done before.

Can you share a specific success story or benefit you've experienced—or expect to experience—after using or publishing with JoVE? (This could include increased collaborations, citations, funding opportunities, streamlined lab procedures, reduced training time, cost savings in the lab, or improved lab productivity.)

1.5. **Atkin Hyatt**, **Graduate Research Assistant**: Publishing our procedure in detail as a video through JoVE offers a unique opportunity to convey complex processes visually, significantly increasing the speed of training and expanding access to an otherwise esoteric recipe (authors will present their testimonial statements live)

Protocol

2. Spin Coating and Vapor-Phase Priming of Silicon Nitride Wafers for Photolithography Preparation

Demonstrator: Atkin Hyatt

2.1. To begin, obtain a glass Petri dish containing a clean, double sided silicon nitride wafer **[1].** Place a few drops of HMDS *(H-M-D-S)* along the edge of a glass Petri dish using a disposable capillary pipette **[2-TXT]**.

2.1.1. WIDE: Talent holding a glass Petri dish with a double sided Si_3N_4 wafer.

2.1.2. Talent using a capillary pipette to place drops of HMDS along the edge of a Petri dish. **TXT: HMDS: Hexamethyldisilazane**

2.2. Under a fume hood, cover the Petri dish **[1].** Heat it on a hot plate at 90 degrees Celsius for 1 minute to evaporate the HMDS and allow it to adhere to the silicon nitride **[2]**. After priming, place the wafer inside a spin coater **[3].** NOTE: The VO is edited for the additional shot

2.2.1. Shot of the Petri dish being covered inside a fume hood.

2.2.2. Talent placing the dish on a hot plate, covering it, and adjusting the temperature to 90 degrees Celsius.

Added shot: Talent places wafer inside spin coater (Slated as 2.3.3)

2.3. Now, use a needle and syringe to extract no more than 4 milliliters of S1813 *(S-One-Eight-One-Three)* photoresist **[1]**. Gently flick the syringe **[2]** and dispense a small amount of fluid to remove any air bubbles **[3]**.

2.3.1. Talent drawing S1813 photoresist into a syringe fitted with a needle.

2.3.2. Talent flicking the syringe.

2.3.3. Talent pushing out a small droplet to clear bubbles.

2.4. Safely remove the syringe needle and attach a 2-micrometre filter **[1]**. Flick the syringe again **[2]** and dispense 3 to 5 drops to remove air bubbles in the filter **[3-TXT]**.

2.4.1. Talent removing the needle and attaching a 2 µm filter to the syringe.

2.4.2. Talent flicking the syringe with the filter.

2.4.3. Shot of the syringe dispensing several drops. **TXT: Ensure at least 3 mL resist remains in the syringe**

2.5. Deposit the resist drop by drop onto the centre of the wafer to form a large pool **[1]**. As the pool expands, continue depositing resist near its edge to keep it centred **[2]**. Use the needle tip to pop any surface bubbles that appear before proceeding **[3]**.

2.5.1. Shot of the photoresist being dropped onto the center of the wafer. NOTE: 2.5.1 and 2.5.2 are filmed in a single take

2.5.2. Talent adjusting drop placement toward the pool's edge as it grows.

2.5.3. Shot of a needle tip being used to burst visible bubbles on the resist surface.

2.6. Now lock the spin coater lid and set it to spin to create a uniform 1.5 micrometre thick layer across the wafer **[1-TXT]**. Remove the wafer and place it on a hotplate to soft bake **[2].** After 1 minute, remove the wafer from the hot plate **[3]**. NOTE: The VO is edited for the additional shot

2.6.1. Talent placing the wafer inside the spin coater and locking the lid. **TXT: 3000 rpm, 30 s, Acceleration: 3000 rpm/s**

Added shot: 2.6.1a. Talent removing wafer from spin coater and placing it on hot plate

2.6.2. Talent lifting the baked wafer off the hot plate using tweezers.

3. Photolithography for Patterning Resonator Structures on Silicon Nitride Wafers Using a Maskless Aligner

Demonstrator: Atkin Hyatt

3.1. To define the device patterns, load the coated wafer into a maskless alignment photolithography machine **[1].** Center it to minimize global rotation and offset errors **[2]**.

3.1.1. Talent placing the wafer into the maskless alignment photolithography system. NOTE: 3.1.1. and 3.1.2 are filmed in a single take

3.1.2. Talent adjusting it to center.

3.2. Load the populated wafer file onto the computer and convert it to a compatible format for the MLA software **[1]**. Begin patterning the topside layer with resonator designs **[2]**.

3.2.1. SCREEN: Show user opening the wafer file on the computer and converting it to MLA-compatible format. NOTE: 3.2.1-3.3.2 SCs are filmed by the videographer

3.2.2. SCREEN: Show user initiating the patterning of the resonator designs on the topside layer.

3.3. After alignment, select the option to include global rotation error **[1]**. Load beam exposure parameters by setting the beam intensity to 140 millijoules per square centimetre at a wavelength of 375 nanometers **[2-TXT]**.

3.3.1. SCREEN: Show user selecting global rotation error inclusion from the software menu. NOTE: 3.3.1 and 3.3.2 are filmed in a single take

3.3.2. SCREEN: Show user entering beam exposure parameters: 140 mJ/cm^2 at 375 nm.

TXT: Begin exposure when all parameters have been entered

3.4. Now fill a large dish with approximately 75 milliliters of MF-319 *(M-F-Three-One-Nine)* developer and another with deionized water **[1]**.

3.4.1. Talent pouring MF-319 developer into one dish and deionized water into another.

3.5. Once the photolithography exposure is complete, unload the wafer from the photolithography machine **[1].** Then transfer it into the MF-319 developer for 20 seconds **[2]**. Gently swirl the dish in a circular motion to agitate and remove the exposed resist for an additional 40 seconds **[3-TXT]**.

3.5.1. Shot of the wafer being removed from the photolithography machine.

3.5.2. Talent transferring the exposed wafer into the MF-319 developer dish and letting it sit undisturbed. NOTE: 3.5.2 to 3.6.1 are filmed in a single take

3.5.3. Talent swirling the dish gently to agitate the developer. **TXT: If resist remains, swirl for 30 s more**

3.6. Then transfer the wafer to the deionized water dish to dilute off residual developer **[1]**. Rinse both sides of the wafer using a spray gun **[2]**.

3.6.1. Talent moving the wafer into the deionized water bath.

3.6.2. Talent rinsing both sides of the wafer with a spray gun.

3.7. Use a compressed nitrogen gas gun to remove remaining water from the wafer surface, applying low pressure and proper orientation to prevent damage **[1]**.

3.7.1. Talent drying the wafer using a compressed nitrogen gun at a low angle and low pressure.

4. Reactive Ion Etching and Chip-Scale Wet Etching for Fabrication of Free-Standing Silicon Nitride Resonators

Demonstrator: Atkin Hyatt

4.1. Load the developed wafer into a reactive ion etcher to transfer the resist patterns onto the silicon nitride film **[1]**. Run the etching process for 5 seconds per cycle with the given settings **[2].** Repeat the process until all wafer regions not protected by the resist layer turn silver, indicating the substrate is exposed **[3]**. Finally, remove the wafer from the reactive ion etcher for backside patterning **[4]**.

4.1.1. Talent placing the wafer into a reactive ion etcher chamber.

4.1.2. Talent setting the etch parameters.
AND
TEXT ON PLAIN BACKGROUND:
Etch Settings to remove 30 nm of Si_3N_4 [40]
Gas composition: 30 sccm of sulfur hexafluoride (SF_6) and 10 sccm of Argon (Ar)

High frequency bias: 50W
Power of inductively coupled plasma: 1000 W
Chamber pressure: 10 mTorr
Video Editor: Please play both shots side by side

4.1.3. Shot of the wafer surface turning silver under the etcher.

4.1.4. Talent opening the etcher and retrieving the processed wafer.

4.2. To release the patterned resonators from their silicon substrate and produce free-standing films, devices are processed at the chip scale **[1].** Carefully peel the diced chips from their tape **[2]**. Dip the chips into a sonicated acetone bath for at least 30 seconds to remove resist and surface contaminants **[3-TXT]**.

4.2.1. Footage of chips being peeled from tape. NOTE: The shot has been modified NOTE: 4.2.1-4.2.3 are filmed in a single take

4.2.2. Talent peeling off diced chips from tape using tweezers.

4.2.3. Talent lowering chips into a sonicated acetone bath. **TXT: Rinse in IPA, then dry with compressed N_2**

4.3. Optionally, to thin the silicon nitride film or remove stuck-on contaminants, dip the chip into a 10 percent hydrofluoric acid buffer solution to remove 1.55 nanometers of silicont nitride per minute **[1]**.

4.3.1. Talent dipping the chip into a clearly labeled hydrofluoric acid buffer solution using tweezers.

4.4. To prevent contamination, immediately place the cleaned chip into a custom PTFE *(P-T-F-E)* holder under a fume hood **[1-TXT]**.

4.4.1. Talent transferring chip into a custom PTFE holder inside a fume hood. **TXT: PTFE: Polytetrafluoroethylene**

4.5. The chip holder is then gently lowered into a 45% solution potassium hydroxide bath maintained at 86 degrees Celsius **[1-TXT].** Cover the solution to maintain a uniform thermal gradient **[2]**.

4.5.1. Talent placing the PTFE chip holder into the potassium hydroxide bath. NOTE: 4.5.1 and 4.5.2 are filmed in a single take

4.5.2. Talent covering the container. **TXT: Set up camera next to etch vessel for long-distance monitoring**

4.6. After the silicon around the resonator is fully etched and the film is released, stop the reaction by removing the vessel from the hot plate **[1]**. Without exposing the chip, gradually pipette out the potassium hydroxide solution **[2]** and replace it with deionized water, always maintaining the liquid level above the chip to prevent shattering the device **[3]**.

4.6.1. Talent turning off the hot plate and carefully removing the vessel.

4.6.2. Talent pipetting out KOH. NOTE: 4.6.2 and 4.6.3 are filmed in a single take

4.6.3. Talent pipetting in deionized water while maintaining constant liquid coverage.

4.7. To avoid cross-contamination, transfer the chip holder and a clean beaker into a fresh bath of deionized water **[1]**. Then transfer the chip holder into the clean beaker containing deionized water **[2].** After the transfer, remove both beakers **[3].**

4.7.1. Talent placing a clean beaker and beaker with chip into a large water bath.NOTE: 4.7.1 and 4.7.2 are filmed in a single take

4.7.2. Talent lifting the chip holder and placing it into a clean beaker of deionized water.

4.7.3. Talent removing both beakers

4.8. Gradually replace the deionized water with isopropyl alcohol in the same way, ensuring the liquid level always remains above the chip. Repeat with methanol **[1-TXT].** After the last bath, remove the chip and dry it carefully along the device plane using a gentle nitrogen stream **[2]**.

4.8.1. Talent pipetting out deionized water while simultaneously adding isopropyl alcohol into the beaker. **TXT: Perform 2 IPA baths then 2 methanol baths**

4.8.2. Talent removing the chip from the methanol bath and blow-drying it along the chip plane with a nitrogen gun. NOTE: This shot is filmed in 2 takes. Take one shows the removal of the chip, and take 2 shows drying of the chip.

5. Optical Lever-Based Displacement and Energy Ringdown Measurements of Silicon Nitride Resonators

Demonstrator: Atkin Hyatt

5.1. Device characterization is performed with optical lever readout. To set up an optical lever, place a focusing lens after a fiber collimator and a beamsplitter between the focusing lens and the sample **[1].** Focus a collimated laser beam onto the sample with a spot size similar to the largest feature being measured **[2-TXT]**. Align the laser beam close to normal incidence onto the sample by retroreflecting into an optical fiber, which simplifies later alignment **[3]**. NOTE: The VO is edited for the moved shot

5.2.1. Talent positioning a beamsplitter in the optical path between the focusing lens and the sample. NOTE: This shot is moved here as per author's request

5.1.1. Talent adjusting a laser beam onto the sample surface to achieve appropriate focus. **TXT: Optical lever sensitivity is proportional to both spot size and reflected power** NOTE: 5.1.1 and 5.1.2 are filmed in a single take

5.1.2. Talent fine-tuning the beam angle for retroreflection into an optical fiber at normal incidence.

5.2. ~~Now, insert a beamsplitter between the sample and the focusing lens to pick off the reflected light from the resonator [1]~~. Place a balanced photodetector along the reflected beam path, positioned beyond the Rayleigh length of the beam to maximize sensitivity **[1]**.

5.2.1. ~~Talent positioning a beamsplitter in the optical path between the focusing lens and the sample.~~ (NOTE: This shot is moved before 5.1.1)

5.2.2. Talent aligning a balanced photodetector at an appropriate distance along the beam path.

5.3. With the laser incident on the split or quadrant photodetector, the detector output is sent to a digitizer. Compute the real-time power spectral density of the signal using the Fast Fourier Transform method **[1]**.

5.3.1. SCREEN: 68706_screenshot_1.mp4: 00:00-00:20

5.4. To identify specific mode peaks, compare the location of thermal noise peaks in the broadband signal to predicted eigenfrequencies from simulations **[1]**. Take a root mean square average of several power spectral density estimates to confirm the peaks **[2]**.

5.4.1. SCREEN: 68706_screenshot_1.mp4: 00:20-00:30

5.4.2. SCREEN: 68706_screenshot_1.mp4: 00:30-01:00

5.5. Begin energy ringdown by tracking the integral under the power spectral density peak at the resonant frequency to measure stored energy **[1]**. To drive the mode, apply energy either by placing a piezo actuator on the side of the or by sending a second laser beam to the sample at the resonant frequency **[2]**.

5.5.1. SCREEN: 68706_screenshot_2.mp4: 00:05-00:20

5.5.2. Talent operating piezo actuator or aligning a second modulated laser beam onto the sample.

5.6. Once the drive is stopped, track the exponential decay of the oscillation energy to determine the damping rate **[1]**. Then calculate the Modal Q by dividing the resonant frequency by the damping rate **[2]**.

5.6.1. SCREEN: 68706_screenshot_3.mp4: 00:07-00:13

5.6.2. SCREEN: 68706_screenshot_3.mp4: 00:13-00:20

Results

6. Results

6.1. COMSOL *(comm-sol)* simulations of the ribbon geometry revealed a 53-kilohertz flexural mode **[1]** and a 70-kilohertz torsional mode **[2]**. The flexural mode has the largest angular displacement halfway between the centre of the ribbon and its fillets **[3]**.

6.1.1. LAB MEDIA: Figure 4A. *Video editor: Highlight the red-blue deformation shape in the upper plot*

6.1.2. LAB MEDIA: Figure 4A. *Video editor: Highlight the red-blue deformation shape in the lower plot*

6.1.3. LAB MEDIA: Figure 4C.

6.2. A GDSII *(G-D-S-Two)* design was created with 37 chips of 12 square millimeters each, showing a variety of geometries for fabrication **[1]**.

6.2.1. LAB MEDIA: Figure 4B.

6.3. Power spectral density analysis demonstrates the effectiveness of the optical lever in reading out the angular displacement of both modes **[1]**.

6.3.1. LAB MEDIA: Figure 5A. *Video Editor: Sequentially highlight the top and the bottom graph*

Pronunciation Guide

1. **Chromobacterium**
   - **Pronunciation link (Merriam-Webster Medical Dictionary)**: https://www.merriam-webster.com/medical/chromobacterium (Wikipedia, Merriam-Webster, YouTube, Merriam-Webster, Merriam-Webster)
   - **IPA (American English)**: /ˌkroʊmoʊbækˈtɪriəm/
   - **Phonetic Spelling**: kroh-moh-bak-TEER-ee-uhm
2. **Hexamethyldisilazane** (HMDS)
   - **Pronunciation link**: No confirmed link found in Merriam-Webster
   - **IPA (Estimated American English)**: /ˌhɛksəˌmɛθəlˌdɪsɪˈlæzeɪn/
   - **Phonetic Spelling**: heks-uh-meth-uhl-dis-ih-LAY-zayn
3. **Photolithography**
   - **Pronunciation link (Merriam-Webster)**: https://www.merriam-webster.com/dictionary/photolithography (Merriam-Webster, Merriam-Webster, pronouncekiwi.com)

- **IPA (American English)**: /ˌfoʊˌtoʊ-lɪˈθɑgrəfi/
- **Phonetic Spelling**: foh-toh-lih-THOG-ruh-fee

4. **Photoresist**
    - **Pronunciation link**: No confirmed link found in Merriam-Webster
    - **IPA (Estimated American English)**: /ˌfoʊtoʊˈriːzɪst/
    - **Phonetic Spelling**: foh-toh-REE-zist
5. **Reactive Ion Etcher**
    - **Reactive**:
        - **Link**: No confirmed link found
        - **IPA**: /riˈæktɪv/
        - **Spelling**: ree-AK-tiv
    - **Ion**:
        - **Link**: No confirmed link found
        - **IPA**: /ˈaɪən/
        - **Spelling**: EYE-on
    - **Etcher**:
        - **Link**: No confirmed link found
        - **IPA**: /ˈɛtʃər/
        - **Spelling**: EHCH-er
6. **Silicon Nitride**
    - **Silicon**:
        - **Link**: No confirmed link found
        - **IPA**: /ˈsɪlɪkən/
        - **Spelling**: SIL-ih-kuhn
    - **Nitride**:
        - **Link**: No confirmed link found
        - **IPA**: /ˈnaɪtraɪd/
        - **Spelling**: NY-tryd
7. **Polytetrafluoroethylene (PTFE)**
    - **Pronunciation link**: No confirmed link found
    - **IPA (American English)**: /ˌpɑliˌtɛtrəˌflʊəroʊˈiθiːlən/
    - **Phonetic Spelling**: pah-lee-TEH-truh-FLUHR-oh-EE-thee-luhn
8. **COMSOL** (software name, pronounced "comm-sol")
    - **Pronunciation link**: No confirmed link found
    - **IPA (American English)**: /ˈkɑmˌsɒl/
    - **Phonetic Spelling**: KOM-sol