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Title: Aesthetically Enhanced Silica Aerogel Via Incorporation of Laser Etching and Dyes

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Author Questionnaire

1. **Microscopy**: Does your protocol require the use of a dissecting or stereomicroscope for performing a complex dissection, microinjection technique, or similar? **N**

2. **Software:** Does the part of your protocol being filmed demonstrate software usage? **Y**

Videographer: Please film the screen as a backup for all SCREEN shots

3. **Interview statements:** Considering the Covid-19-imposed mask-wearing and social distancing recommendations, which interview statement filming option is the most appropriate for your group? **Please select one**.

☒ Interviewees wear masks until the videographer steps away (≥6 ft/2 m) and begins filming. The interviewee then removes the mask for line delivery only. When the shot is acquired, the interviewee puts the mask back on. Statements can be filmed outside if weather permits.

4. **Filming location:** Will the filming need to take place in multiple locations (greater than walking distance)? **N**

Protocol Length
Number of Shots: **48**

Introduction

1. Introductory Interview Statements

REQUIRED:

1.1. **Mary K. Carroll**: This protocol presents the first visual methodology for etching and cutting plain and dyed aerogels using a laser engraving system **[1]**.

1.1.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B-roll: 3.11.1., 4.8.2.*

REQUIRED:

1.2. **Ann M. Anderson**: The main advantage of this technique is its simplicity, as it allows aerogel monoliths of any shape, size, or color to be etched **[1]**.

1.2.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B-roll: 3.2.1., 5.2.*

OPTIONAL:

1.3. **Allison M. Stanec**: Video presentation allows demonstration of the practical considerations of the protocol, including the placement of the aerogel and the alignment of the laser cutter **[1]**.

1.3.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B-roll: 4.5.1.*

Protocol

2. Aerogel Monolith Fabrication and Laser Engraver Print File Preparation

2.1. To prepare a mold for aerogel monolith fabrication, select a three-part steel mold consisting of a top, middle, and bottom part **[1]** with outer dimensions of 15.24 x 14 centimeters **[2]** and a 10- x 11-centimeter cavity in the center **[3]**.

2.1.1. WIDE: Talent selecting/picking up mold

2.1.2. Shot of mold *Video Editor: please "measure"/emphasize outer dimensions*

2.1.3. Use 2.1.2. *Video Editor: please outline/emphasize center cavity*

2.2. The top part of the mold should have seven 0.08-centimeter vent holes on each side **[1]**.

2.2.1. Shot of side of mold with 7 vent holes

2.3. Use diluted soap and a rough textured sponge to scrub the top, middle, and bottom parts of the mold **[1]** and dry all of the cleaned parts of the mold with a paper towel **[2]**.

2.3.1. Mold being scrubbed **NOTE: Only one part was filmed which was sufficient.**
2.3.2. Mold being dried

2.4. After drying, use individual, disposable, acetone-dipped cleaning wipes to wipe the mold until the cleaning wipe remains clean **[1]**.

2.4.1. Mold being wiped, with beaker of acetone visible in frame

2.5. Next, use 2000-grit sandpaper to lightly sand all of the surfaces until the mold is smooth to the touch and any residue from previous uses has been removed **[1]**, paying extra attention to the inside of the middle mold where the aerogel is formed **[2]**.

2.5.1. Mold being sanded
2.5.2. Inside of mold being sanded

2.6. Clear the vent holes in the top mold part with compressed air **[1]** and manually apply approximately 2.4 milliliters of high-vacuum grease in a thick, even, 1-2-millimeter layer over the entire top-connecting surface of the bottom mold **[2]** and approximately 1

milliliter of grease in a thick, even 1-2-millimeter layer to the outer half of the bottom-connecting surface of the top mold **[3]**.

2.6.1. Air being flowed through vent holes
2.6.2. Grease being applied to bottom of mold
2.6.3. Grease being applied to top of mold

2.7. Apply approximately 0.5 milliliters of high-vacuum grease in an even, less than 0.5-millimeter layer to the inside surfaces of the top and bottom molds **[1]** and use a disposable cleaning wipe to wipe away excess grease until the surface feels smooth and no stickiness from the grease remains **[2]**.

2.7.1. Grease being applied to inside top and bottom mold surface(s) *Videographer: Important step*
2.7.2. Mold being wiped *Videographer: Important step*

2.8. Manually apply 0.5 milliliters of high vacuum grease in an even, less than 0.5-millimeter layer of grease to the inside surface of the middle mold **[2-TXT]**.

2.8.1. Grease being applied to middle mold inside surface **TEXT: Do not wipe away excess grease**

2.9. To prepare the etching pattern file, open a new document in an appropriate drawing application **[1]** and add the desired text or image of any size and line width directly to the document. Then save the file **[2]**.

2.9.1. Talent opening document, with monitor visible in frame

2.9.2. SCREEN: 2.9.2JoVE.mp4. 00:01-00:20.

3. Etching Procedure

3.1. To etch an aerogel monolith, first turn on the laser engraver, vacuum exhaust system, and attached computer **[1-TXT]**.

3.1.1. WIDE: Talent turning on engraver and/or system and/or computer **TEXT: Here 50 W CO_2 laser engraver/cutter shown NOTE: Turning on of the engraver and exhaust system was filmed.**

3.2. Measure the size of the aerogel monolith surface that will be etched **[1-TXT]** and open the etching pattern file **[2]**.

3.2.1. Monolith being measured **TEXT: See text for monolith preparation details**
3.2.2. Talent opening file, with monitor visible in frame

3.3. Set the document's dimension size to correspond to the measured aerogel monolith size **[1]** and open the lid of the laser engraver **[2]**.

3.3.1. SCREEN: 3.3.1JoVE.mp4. 00:01-00:27.
3.3.2. Talent opening lid

3.4. Use a gloved hand to place the aerogel onto the laser engraver platform **[1]** and align the aerogel in the top-left corner with the aerogel touching the top and left rulers **[2]**.

3.4.1. Talent placing aerogel onto platform
3.4.2. Aerogel being positioned touching rulers *Videographer: Important step*

3.5. Flip the V-shaped magnet manual focus gauge attached to the laser upside down **[1]** and press **Focus** on the laser engraver **[2]**.

3.5.1. Talent flipping magnet
3.5.2. Talent pressing focus

3.6. Protect the aerogel monolith with a disposable cleaning wipe **[1]** and use the up arrow on the laser engraver control panel to move the laser engraver platform until the bottom part of the manual focus gauge just touches the aerogel **[2]**.

3.6.1. Wipe being placed *Videographer: Difficult step*
3.6.2. Platform being moved *Videographer: Important/difficult step*

3.7. Remove the wipe **[1]** and return the gauge to its original position **[2]**.

3.7.1. Shot of gauge just touching aerogel, then wipe being removed
3.7.2. Gauge being returned to original position

3.8. Close the laser engraver lid **[1]** and click **File** and **Print** in the drawing program. Select the laser engraver as the print location and open the Preferences window to set the printer properties **[2]**.

3.8.1. Talent closing lid
3.8.2. SCREEN: 3.8.2JoVE.mp4. 00:01-00:11.

3.9. Select the **Raster** mode and set a **DPI** (D-P-eye) of 600, a **Speed** of 100%, and a **Power** of 55%. Confirm that the piece size matches the measured aerogel monolith size and click **Apply** and **Print [1]**.

3.9.1. SCREEN: 3.9.1JoVE.mp4. 00:01-00:24.

3.10. On the front panel of the laser engraver, click **Job** and select the corresponding file name. Click **Go [1]**.

3.10.1. Talent clicking Job, file being selected, then Go being clicked

3.11. When the laser engraver finishes **[1]**, click **Focus** and use the down arrow on the laser engraver front control panel to lower the base **[2]**.

3.11.1. Aerogel being engraved *Videographer: Important step*
3.11.2. Talent clicking Focus and down arrow

3.12. Use a gloved hand to gently transfer the aerogel from the laser engraver platform to its container **[1]** and click **Trash** to purge the job from the engraver **[2]**.

3.12.1. Talent transferring aerogel from platform to container
3.12.2. Talent clicking Trash/purging engraver

4. Cutting Procedure

4.1. To cut an aerogel monolith, turn on the laser engraver, vacuum exhaust system, and attached computer **[1]** and measure the size of the aerogel monolith surface to be cut **[2]**.

4.1.1. WIDE: Talent turning on exhaust system
4.1.2. Surface being measured

4.2. For general cutting, open a new document in the drawing program **[1]** and enter the dimensions for the document size to correlate with the measured aerogel monolith size **[2]**.

4.2.1. Talent opening document, with monitor visible in frame
4.2.2. SCREEN: 4.2.2JoVE.mp4. 00:01-00:09.

4.3. Use the hairline width drawing tool to create the pattern that will be cut, and position the pattern to match the desired cut location on the aerogel **[1]**.

4.3.1. SCREEN: 4.3.1JoVE.mp4. 00:01-00:22.

4.4. Clean a 0.0127-millimeter-thick sheet of stainless steel foil large enough to cover the base of the aerogel monolith with acetone **[1]** and place the foil onto the laser engraver platform **[2]**.

4.4.1. Foil being cleaned
4.4.2. Foil being placed onto platform

4.5. Align the aerogel and stainless steel foil in the top left corner with the aerogel touching the top and left rulers **[1]** and adjust the gauge position as demonstrated **[2]**.

4.5.1. Aerogel and foil being aligned
4.5.2. Gauge being lowered toward wipe-covered aerogel **NOTE: Shot includes multiple steps.**

4.6. Click **File** and **Print** in the drawing program and select the laser engraver as the print location and open the Prefererences window to set the printer properties **[1]**.

4.6.1. SCREEN: 4.6.1JoVE.mp4. 00:01-00:11.

4.7. Select the **Vector** mode and set the **DPI** to 600, the **Speed** to 3%, the **Power** to 90%, and the **Frequency** of 1000 hertz **[1]**. Confirm that the piece size matches the measured aerogel size **[2]**. The depth of the cut will vary with the laser speed **[3]**.

4.7.1. SCREEN: 4.7.1JoVE.mp4. 00:01-00:28.
4.7.2. Talent confirming piece and aerogel sizes
4.7.3. LAB MEDIA: Table 4 *Video Editor: please emphasize Cut Depth column*

4.8. Click **Job** and **Go** to initiate the cutting as demonstrated **[1]**. After collecting the sample, use a foam brush to gently remove any small pieces of ablated aerogel left on the face of the monolith that was in contact with the laser **[2]**.

4.8.1. Talent clicking Job and Go

4.8.2. Sample being collected, then pieces being brushed away **NOTE: Do not include shot of broken aerogel.**

Results

5. Results: Representative Silica Aerogel Laser Etching and Dyeing

5.1. For this aerogel mosaic approach **[1]**, the same pattern was cut into three different dyed aerogel monoliths **[2]** and the pieces were reassembled into a mosaic pattern **[3]**.

5.1.1. LAB MEDIA: Figure 8
5.1.2. LAB MEDIA: Figure 8 *Video Editor: please sequentially emphasize Figures 8a, 8b, and 8c*
5.1.3. LAB MEDIA: Figure 8 *Video Editor: please emphasize Figures 8d and 8e*

5.2. It is possible to etch designs on smaller monolithic pieces as demonstrated to obtain visually interesting arrangements **[1]** under natural **[2]** and ultraviolet light conditions, even on smaller pieces **[3]**.

5.2.1. LAB MEDIA: Figure 9
5.2.2. LAB MEDIA: Figure 9 *Video Editor: please emphasize Figure 9a*
5.2.3. LAB MEDIA: Figure 9 *Video Editor: please emphasize Figure 9b*

5.3. As illustrated **[1]**, native aerogels can be etched with patterns of various density **[2]**, with photographs printed onto the front surface of a planar aerogel **[3]**, onto a curved surface **[4]**, and onto fluorescein-dyed aerogels **[5]**.

5.3.1. LAB MEDIA: Figure 10
5.3.2. LAB MEDIA: Figure 10 *Video Editor: please emphasize Figures 10a-10c*
5.3.3. LAB MEDIA: Figure 10 *Video Editor: please emphasize Figure 10d bottom image*
5.3.4. LAB MEDIA: Figure 10 *Video Editor: please emphasize Figure 10e bottom right image*
5.3.5. LAB MEDIA: Figure 10 *Video Editor: please emphasize Figure 10f*

5.4. In this scanning electron microscope image of an etched silica aerogel **[1]**, the interface between the etched "lines" **[2]** and the almost smooth, un-etched nanoporous aerogel can be observed **[3]**.

5.4.1. LAB MEDIA: Figure 11a
5.4.2. LAB MEDIA: Figure 11a *Video Editor: please emphasize upper right venation pattern*
5.4.3. LAB MEDIA: Figure 11a *Video Editor: please emphasize left/bottom left smooth gel*

5.5. Etching causes the ablation and melting of the surface material into filament-like structures **[1]**, as observed in this image showing the effect of a single laser pulse of the aerogel **[2]**.

5.5.1. LAB MEDIA: Figure 11b

5.5.2. LAB MEDIA: Figure 11b *Video Editor: please emphasize oval pulse in center left of image*

Conclusion

6. Conclusion Interview Statements

6.1. **Allison Stanec**: Careful preparation, including contrast adjustment, of the images to be etched is critical to achieving high-quality results **[1]**.

6.1.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B-roll: 3.9.1.*

6.2. **Ann M. Anderson**: Further refinement of the laser etching method could provide an alternative to micromachining of the silica aerogels **[1]**.

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

6.3. **Mary K. Carroll**: Aesthetic aerogel monoliths could find application in a wide variety of areas, including artwork and sustainable buildings **[1]**.

6.3.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B-roll: 4.8.2.*