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Title: Improved Polydimethylsiloxane (PDMS) Double Casting via Silicone Oil Treatment for Densely Packed Microstructure Replication

Authors and Affiliations:
Yoonjae Lee, Taehoon Kim, Jae Hun Seol

School of Mechanical and Robotics Engineering, Gwangju Institute of Science and Technology

Corresponding Authors:
Jae Hun Seol (jhseol@gist.ac.kr)
Email Addresses for All Authors:
Yoonjae Lee (yoonjae10@gm.gist.ac.kr)
Taehoon Kim (enormerit@gm.gist.ac.kr)
Jae Hun Seol (jhseol@gist.ac.kr)

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? **No**

3. **Filming location:** Will the filming need to take place in multiple locations? **Yes, 50 m apart in the same building**

Current Protocol Length
Number of Steps: 14
Number of Shots: 31

Introduction

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

1.1. **Yoonjae Lee:** To address demolding challenges in dense and high-aspect-ratio microstructures, we modified the PDMS double casting process by adding a silicone oil treatment step applied with a tensioned thread.

1.1.1. INTERVIEW: Named Talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B.roll:2.6*

What advantage does your protocol offer compared to other techniques?

1.2. **Yoonjae Lee:** Our protocol offers a simple, cost-effective solution that avoids the need for expensive equipment or specialized materials, enabling reliable replication of dense microstructures without damage.

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

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

1.3. **Taehoon Kim:** Our laboratory will focus on automating the oil application process and developing custom silicone oils to identify optimal conditions for improved reproducibility and precision.

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.

Protocol

2. Fabrication of PDMS mold from a Silicon Mold

Demonstrator: Yoonjae Lee

2.1. To begin, mix a solution of hexane and OTS *(O-T-S)* in a 40 to 1 volume ratio using a clean glass beaker and a stirring rod **[1-TXT]**. Cover the beaker with aluminum foil to minimize evaporation **[2]**.

2.1.1. WIDE: Talent mixing hexane and OTS in a clean glass beaker using a stirring rod. **TXT: OTS: Octadecyltrichlorosilane**

2.1.2. Talent covering the glass beaker with aluminum foil.

2.2. Sonicate the sealed glass beaker into a room-temperature ultrasonic bath filled with water **[1-TXT]**. Then immerse the pre-etched silicon mold with a hole array into the prepared hexane-OTS solution **[2]**.

2.2.1. Talent placing the covered glass beaker into an ultrasonic bath. **TXT: Sonication: 40 kHz, 135 W, 5 min**

2.2.2. Talent placing the silicon mold vertically into the solution using tweezers.

2.3. Now use clean tweezers to gently lift the entire silicon mold vertically out of the solution **[1]**. Immediately transfer it to a fresh beaker containing neat hexane **[2].** Then rinse by gently agitating it back and forth 5 to 10 times **[3]**.

2.3.1. Talent lifting the silicon mold from the beaker using tweezers.

2.3.2. Talent transferring the mold to a fresh beaker with neat hexane.

2.3.3. Talent rinsing the mold in neat hexane by moving it gently back and forth.

2.4. Gently dry the mold using a nitrogen blower held approximately 3 centimeters away for 6 to 10 seconds until no visible moisture remains **[1]**.

2.4.1. Talent drying the mold using a nitrogen blower from a short distance.

2.5. Next, mix the PDMS *(P-D-M-S)* base and curing agent in a 10 to 1 weight ratio in a clean plastic Petri dish for 15 minutes **[1-TXT]**. Place the mixture into a chamber and degas at 160 torr until no visible air bubbles remain **[2]**.

2.5.1. Talent mixing the PDMS base and curing agent in a Petri dish. **TXT: PDMS: Polydimethylsiloxane**

2.5.2. Talent placing the Petri dish into a vacuum chamber for degassing.

2.6. Lightly coat a clean plastic Petri dish with silicone oil of 100 centistokes viscosity using a lint-free wiper pre-soaked in the oil **[1]**. Then place the OTS-coated silicon mold face-up in the centre of the coated Petri dish **[2]**.

2.6.1. Talent wiping the surface of the Petri dish using an oil-soaked lint-free wiper.

2.6.2. Talent positioning the coated silicon mold in the Petri dish.

2.7. Slowly pour the degassed PDMS mixture over the mold until it is fully covered **[1]**. Degas the filled mold again in the vacuum bubble remover until no visible bubbles remain **[2]**.

2.7.1. Talent pouring the polydimethylsiloxane mixture over the mold.

2.7.2. Talent placing the Petri dish back into the vacuum chamber.

2.8. Now, place the Petri dish on a preheated hot plate set to 90 degrees Celsius to cure the PDMS **[1]**. Once cured, gently peel the PDMS mold from the silicon mold, ensuring the microstructure remains intact **[2]**.

2.8.1. Talent placing the Petri dish on the hot plate.

2.8.2. Talent carefully peeling the cured polydimethylsiloxane mold from the silicon mold.

3. Fabrication of PDMS Product from PDMS Mold

3.1. Place the fabricated PDMS mold on a hot plate set to 150 degrees Celsius **[1]**. After 3 days of thermal aging, immerse the mold in silicone oil of 100 centistokes viscosity **[2]**.

3.1.1. Talent placing the polydimethylsiloxane mold onto a hot plate.

3.1.2. Talent submerging the mold in silicone oil.

3.2. Degas the oil-immersed mold under vacuum at 160 torr for 10 to 15 minutes **[1]**. Then, remove the mold using tweezers **[2]** and gently wipe the surface 3 to 5 times with a clean lint-free wiper to leave a uniform thin film of oil **[3]**.

3.2.1. Talent placing the mold in a vacuum chamber for degassing.

3.2.2. Talent removing the mold with tweezers.

3.2.3. Talent wiping the mold with a lint-free wiper.

3.3. Next, prepare a PDMS mixture at a 10 to 1 base to curing agent weight ratio, using the same mixing and degassing method as before **[1]**. Gently pour the degassed mixture onto the treated mold, allowing it to spread and reach at least halfway up the micropillars **[2]**.

3.3.1. Talent preparing and degassing the polydimethylsiloxane mixture.

3.3.2. Talent pouring the mixture over the mold, showing it rising along the micropillars.

3.4. Secure a 0.5-micrometre nylon thread under gentle tension **[1]**. Swipe the thread once or twice across the PDMS surface in one direction to reduce excess oil between micropillars **[2]**. Then degas the mold with the thin PDMS layer in a vacuum chamber until no air bubbles remain **[3]**.

3.4.1. Talent securing the nylon thread under tension.

3.4.2. Talent swiping the thread across the mold surface.

3.4.3. Talent placing the mold into a vacuum chamber.

3.5. To fabricate a hole-array pattern PDMS product, lightly coat a clean plastic Petri dish with silicone oil **[1]**. Place the PDMS mold into the coated Petri dish **[2]** and pour the degassed PDMS mixture over it until the desired height is reached **[3]**.

3.5.1. Talent coating the Petri dish with silicone oil.

3.5.2. Talent placing the mold in the dish.

3.5.3. Talent pouring the polydimethylsiloxane mixture.

3.6. Degas the mold assembly once more to eliminate any trapped air bubbles **[1]**. Then place the Petri dish on a preheated hot plate set to 110 degrees Celsius and cure until fully solidified **[2]**. After curing, gently insert clean tweezers between the mold and the replica and carefully demold the final product from the mold **[3]**.

3.6.1. Talent placing the Petri dish into the vacuum chamber.

3.6.2. Talent placing the Petri dish on a hot plate.

Added shot: Talent removing the mold with tweezers.

Results

4. Results

4.1. The silicon mold featured a dense array of circular holes measuring 143 micrometres in diameter with 150 micrometre spacing **[1]**, and a depth of 284 micrometres, yielding a 2 to 1 aspect ratio **[2]**.

4.1.1. LAB MEDIA: Figure 3A.

4.1.2. LAB MEDIA: Figure 3B.

4.2. The PDMS mold replicated from the silicon template produced micropillars measuring 142 micrometres in diameter **[1]** and 283 micrometres in height closely matching the original dimensions **[2]**.

4.2.1. LAB MEDIA: Figure 4A.

4.2.2. LAB MEDIA: Figure 4B.

4.3. The PDMS replica thermal aged 1 day and subsequently treated with 100 centistokes silicone oil, showed severe tearing and disintegration of wall structures during demolding **[1]**. After 2 days of thermal aging followed by 100 centistokes silicone oil treatment, distinct pillar outlines became visible, but the walls remained heavily damaged due to insufficient release **[2]**.

4.3.1. LAB MEDIA: Figure 5A.

4.3.2. LAB MEDIA: Figure 5B.

4.4. With 3 days of thermal aging and no oil layer, cross-linking between cured and uncured PDMS resulted in recombined structures **[1]**. Using low-viscosity MR-100 *(M-R-One-Hundred)* oil after 3 days of thermal aging led to poor coverage and tearing, as the oil drained off vertical sidewalls **[2]**. High-viscosity oil formed a thick coating, preventing mold filling and causing the final product to appear wavy and malformed **[3]**.

4.4.1. LAB MEDIA: Figure 5C.

4.4.2. LAB MEDIA: Figure 5D.

4.4.3. LAB MEDIA: Figure 5E.

4.5. Under optimized conditions of 3 days thermal aging and 100 centistokes silicone oil coating, the final replica preserved hole-patterned microstructures with 148 micrometres diameter **[1]** and 280 micrometres height, showing minor dimensional variation but intact structure **[2]**.

4.5.1. LAB MEDIA: Figure 6A.

4.5.2. LAB MEDIA: Figure 6B.

Pronunciation Guide:

1. Polydimethylsiloxane

Pronunciation link:
https://www.howtopronounce.com/polydimethylsiloxane YouTube+11How to Pronounce+11How to Pronounce+11

IPA: /ˌpɑː.liˌdaɪˌmɛθ.əlˈsɪl.ək.sɑn/
Phonetic Spelling: pah-lee-dye-meth-uhl-sil-uhk-sahn

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2. Octadecyltrichlorosilane (OTS)

Pronunciation link:
https://www.pronouncekiwi.com/Octadecyltrichlorosilane precision.fda.gov+10pronouncekiwi.com+10pronouncekiwi.com+10

IPA: /ˌɒk.təˌdɛs.ɪlˌtraɪ.klɔːr.oʊˈsɪl.eɪn/
Phonetic Spelling: ok-tuh-DES-il-try-KLOR-oh-sil-ayn

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3. Hexane

Pronunciation link:
https://www.merriam-webster.com/dictionary/hexane YouGlish+2How To Say Guide+2Forvo.com+2

IPA: /ˈhɛk.seɪn/
Phonetic Spelling: HEK-sayn

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4. Sonicate / Sonication

Pronunciation link:
https://www.merriam-webster.com/dictionary/sonicate NIST WebBook+2How to Pronounce+2Wikipedia+2

IPA: /ˈsɒn.ɪ.keɪt/
Phonetic Spelling: SON-ih-kate

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5. Degas / Degassing

Pronunciation link:
https://www.merriam-webster.com/dictionary/degassing merriam-webster.com

IPA: /diːˈgæs/
Phonetic Spelling: dee-GAS

6. Centistokes

Pronunciation link:
https://www.merriam-webster.com/dictionary/centistoke merriam-webster.com

IPA: /ˈsɛn.tiˌstoʊks/
Phonetic Spelling: SEN-tee-stohks