Standard Operating Procedure

TU/e

SOP details

Title	Silanization of Si wafer master mould and Ormomould
Description	This SOP describes how to perform silane treatment to the Si wafer TopoChip and Ormomould to increase hydrophobicity
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SOP number	1.3
Version number	2

	Name	Date	Signature
Prepared	Phani Krishna Sudarsanam	26-05-2020	
Revised	Mehmet Tas	11-02-2021	
Reviewed			
Authorized	Jan de Boer	18-4-2023	

Version changes

Version	Name	Date	Changes made
1	Phani Krishna Sudarsanam	26-05-2020	Made in TU/e
2	Mehmet Tas	11-02-2021	Revised, more details and figures added.
3	Jan de Boer	23—3-2021	Reviewed and commented with track changes
4	Mehmet Tas	11-05-2021	Comments are addressed, document is ready to finalise
5	Burcu Gumuscu	20-5-2021	Revised, finalised.

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1 Purpose

This SOP describes how to treat the Silica (Si) mould or Ormomould with chemical agents that make the surface of the moulds hydrophobic to allow easy release of the imprinting polymer film without damaging the mould or decreasing the yield.

2 Principle

Silanization is the covering of a surface with organofunctional alkoxysilane molecules. If a substrate surface contains hydroxyl groups, it can be silanized. Hydroxyl groups attack the alkoxy groups on the silane which results in forming a stable covalent Si-o-Si bond. In TopoChip production, this treatment is used for producing a passivation layer on Si master mould surfaces to aid release of Polydimethylsiloxane (PDMS) and prevent the PDMS from adhering to the master. Additionally, Ormomoulds are also silane treated before imprinting polymer surfaces via hot embossing to assist release of the polymer films without damaging the Ormomould and improve its reusability with high surface imprinting yield.

3 Before You Start

The Si wafer master mould or Ormomould should be cleaned with pressurized nitrogen to remove any dust particles before proceeding with the oxygen plasma treatment (SOP 1.2) to make the surface clean to increase the formation of the silane monolayer. This SOP can be used for treatment of both Si mould and Ormomoulds. Before performing this process, users must obtain the general microfabrication lab introduction (Feynman lab) from the lab technicians and silanization training by a superuser.

4 Required materials

4.1 Workplace

This SOP can be performed in the microfabrication lab (Gemini- Noord, Feynman lab). Follow the safety protocols instructed by the lab managers and perform the experiment accordingly in allocated locations in the lab. Silanization must be performed in a fumehood.

4.2 Equipment and disposables

- Fumehood
- Vacuum desiccator (Bookable on Labagenda, located in silanization fumehood)
- Nitrogen spray gun
- Plastic pipette (2ml) (LP Italiana, supplier VWR)
- Disposable glass vial (5 ml) (VWR generic screw top vials)

4.3 Chemicals

- Trichloro(1H,1H,2H,2H-perfluorooctyl) silane (Sigma, Cat. No: 448931) – This silanizing agent is in the safety cupboard – corrosives shelf)

5 Procedure

5.1 Working procedure

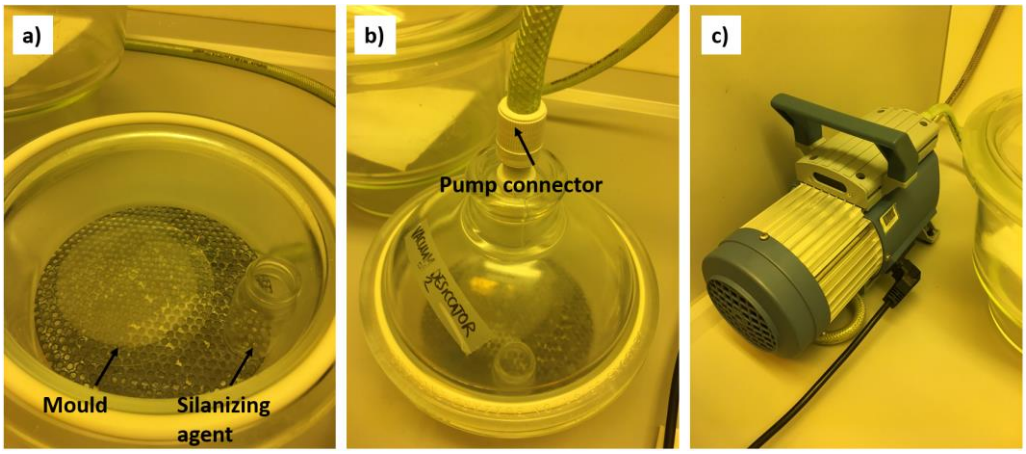
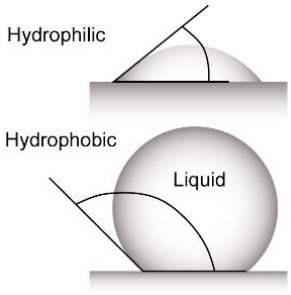


Figure 1. Silanization setup in the fumehood. (a) Dessicator where the mould and silanizing agent are placed in. The silanizing agent is put into a glass container without a cap, (b) pump connection, (c) pump.

This process should be carried out inside the fumehood. Silanizing agent bottle should be opened inside the fumehood. Below are the steps to perform the silanization treatment.

1. The cleaned Si/Ormo mould is placed in a glass or plastic petri dish with topographies facing upwards in the center of the vacuum desiccator. (see Figure 1a)
2. Using a plastic pipette, a single drop of silane is carefully pipetted into a disposable glass vial (5 ml) which is kept inside the desiccator next to the mould. The bottle should not be opened until the setup is ready and should immediately be closed and secured to prevent spillage and unnecessary evaporation since it is a dangerous chemical. It should be placed in the safety cupboard in the dedicated shelf right away.
3. Place the lid back onto the desiccator with the rubber seal in place to retain the vacuum overnight.
4. Connect the tube from the motor of the vacuum pump to the desiccator and place the screw valve in open position (see Figure 1b) .
5. Switch on the vacuum pump and pump the air out of the desiccator for 5 minutes (see Figure 1c).
6. Switch off the pump and close the valve (screw down) and disconnect the tube from the desiccator.
7. Discard the gloves and get a new pair if you have other work to do in the cleanroom.
8. Label the overnight pass which you can find in the lab with your details and leave the fume hood closed overnight. Make sure you book the dedicated dessicator on Lab agenda for the duration of the silanization process.
9. Once the treatment is complete, vent the desiccator slowly and open the desiccator's lid to get the mould out. If the valve is released fast, the wafer may flip over due to vacuum inside of the desiccator.
10. The glass vial can be deposited in the waste bin dedicated for the fume hood. Clean the desiccator with iso-propanol using tissue wipes.

11. Pipette 250 µl of demi water using a micropipette on the mould and observe the shape of the water droplet. Preferably, apply the water on the corners and not in the middle of the wafer or where the microstructures are present.
12. If the surface is silanized and the surface becomes hydrophobic, there is a change in the contact angle of the water droplet on the surface and it pushes away the droplet off the surface. This process can be observed by eye, a hydrophobic surface is characterized by a water droplet having a contact angle of >= 90^{o} with the surface. If quantitative analysis are required, a contact angle measurement setup can be used.



5.2 Safety.

Work in the microfabrication lab in Gemini-Noord according the safety regulations. Follow the instructions given in the lab introduction by the lab managers

Read through the MSDS form of the silanizing agent. It is present in the shared SOP folders. You can also access it here on Sigma Aldrich's website.

6 Waste

When working in the microfabrication lab, handle waste according to guidelines which are labelled at the waste disposal based on its categories as given below in the table 1.

7 References

SOPnr	Title
1.2	Plasma oxygen treatment

Technische Universiteit **Eindhoven** University of Technology

Categorisation of hazardous waste substances

HELP PROTECT SAFETY, HEALTH AND THE ENVIRONMENT

					N/A
				N/A	N/A
Category I (diluted) inorganic acids with heavy metals pH ≤ 7	*Category II* (diluted) inorganic caustic solutions pH ≥ 7	*Category III* low halogen content organic substances	*Category IV* high halogen content organic substances	*Category V* special waste substances	*Category VI* waste substances containing special risks
laboratory chemicals (original packaging for chemicals still containing residues), contact Waste management & logistics, phone no 4343					Radioactive substances
Diluted inorganic acids (nitric acid, hydrochloric acid, etc.)	Diluted inorganic hydroxides	Liquid organic substances (alcohol, acetone, toluene, etc.)	Liquid halogenated organic substances (i.e. substances containing fluorine, chlorine, bromine or iodine)	Preparations and specimens	Biological waste
Heavy metal cations in solution (e.g. zinc, copper, nickel, lead)	Heavy metal ions in solution (e.g. zinc, copper, nickel, lead)	Solid and pasty low-halogen organic substances (including plastics)	Solid and pasty high-halogen organic substances (including plastics)	Chemically contaminated packaging/equipment	gas cylinders and pressurised containers
Cations and anions of heavy metals	Anions of metalloids	Mineral lubrication and system oils (such as sump oil)	Pesticides (containing halogen-based compounds)	Small, hazardous waste	
Solutions containing fluoride	Solutions containing cyanide	Oil emulsions (such as drill, grinder, roller and cutter oils containing water)	Waste oils contaminated with substances containing halogens	All other chemical waste that cannot be placed in categories I to IV or VI . *See the TU/e Science Park Waste Substances Manual*	For the disposal of the waste substances listed above, contact the **SBD, BVF or Waste management & logistics**
Waste liquid from plating and pickling baths (acid), fixing salt	Photograph developer and activator	Pesticides (halogen-free)			
For the disposal of the waste substances listed above, contact the **Waste management & logistics**					

General tips

- Read the 'Waste Disposal' instructions and the TU/e Science Park Waste Substances Manual (AMSO website)
- Make sure waste is categorised correctly
- Correctly label waste
- Watch out for accidental reactions (if you have any doubts, see the list of hazardous combinations in the TU/e Science Park Waste Substances Manual)
- Be aware of the fire risks associated with Category III and IV substances
- Work neatly and tidily, use personal protective equipment
- Use the correct packaging for collecting waste
- Always close (empty) packaging
- If possible, only open packaging in a fume cupboard
- Fill packaging to a maximum of 90% (up to the indicator strip)
- For the temporary storage of packaging, follow the 'Storage of hazardous substances in accordance with PGS 15' procedure (Appendix 1 of the TU/e Science Park Waste Substances Manual)
- Call the TU/e Waste Centre before collection
- If you have any doubts about specific types of waste or questions concerning (the above) procedures, etc., you should always contact your faculty's local health, safety and environment co-ordinator

Important telephone numbers:

Waste Management & logistics	(040 247) 4343
(AMSO)	(040 247) 3500 (if there is no answer call 3355)
Radiation Protection Service (SBD)	(040 247) 3355 (if there is no answer call 3500)
Biological agents safety officer (BVF)	(040 247) 2239
Emergency telephone number	**(040 247) 2222**

PDO/AMVS/2014

Table 1: Categories of hazardous liquid waste