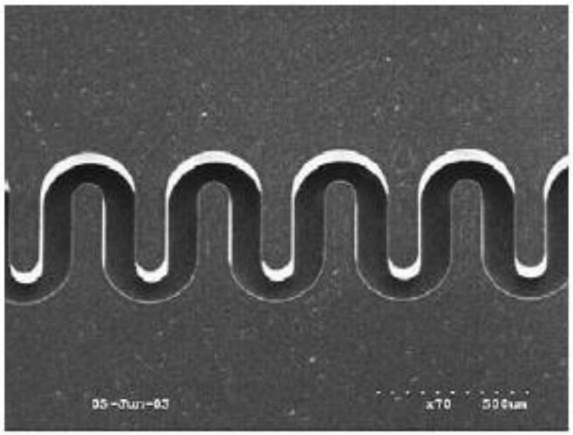
Microfabrication for fluidics

sami.franssila@aalto.fi

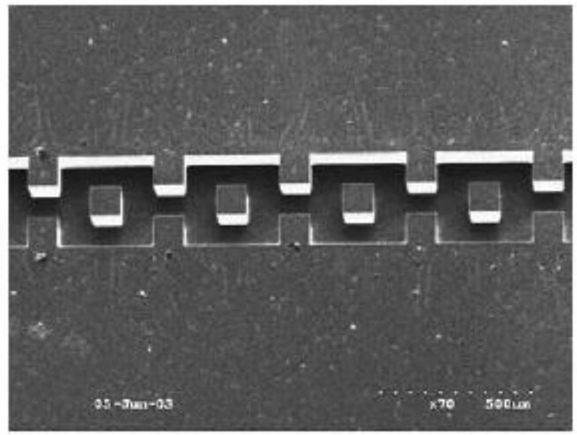
Contents

- Optical lithography
- Etching
- Polymer replication
- Bonding
- Thin films
- Structure-materials-processes pros & cons
- Materials property comparison

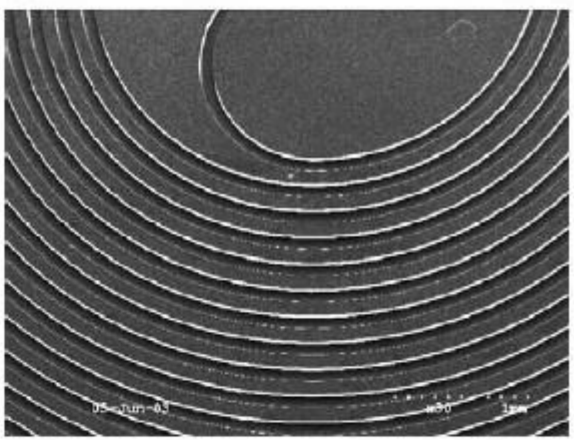
Channels, top view (layout view)



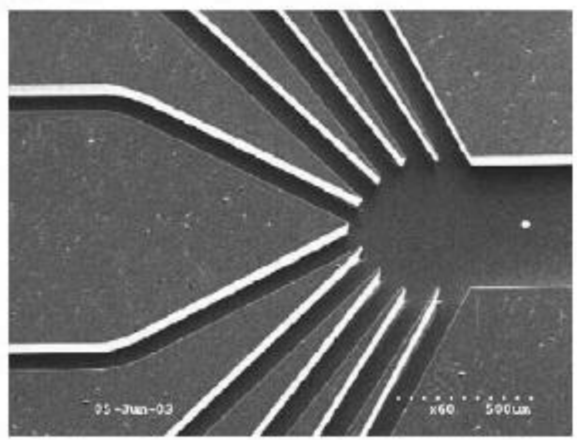
(a)



(b)



(c)



(d)

Typical scales:

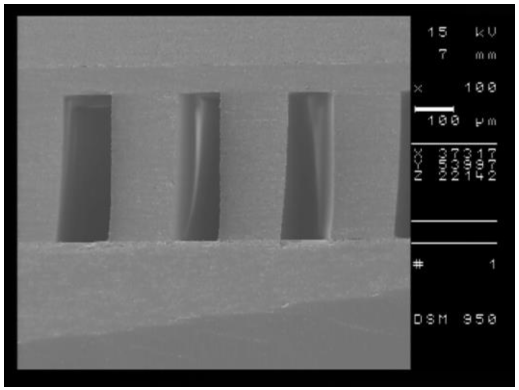
Individual channels 10-100 µm wide

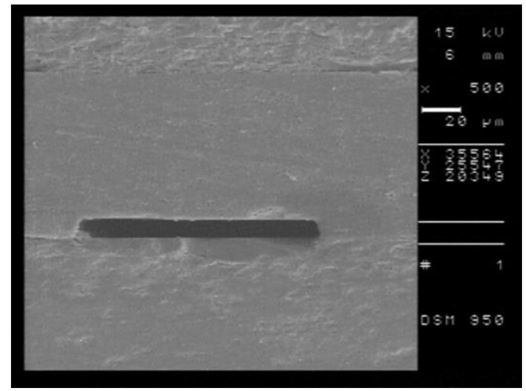
Large reservoirs: millimeter sizes (volume 0.1 nl if 100 µm high)

H.-S. Noh et al. / Sensors and Actuators B 102 (2004) 78–85

Channels, cross section view

SU-8 epoxy polymer





Typical channel heights:

5-500 µm

Glass

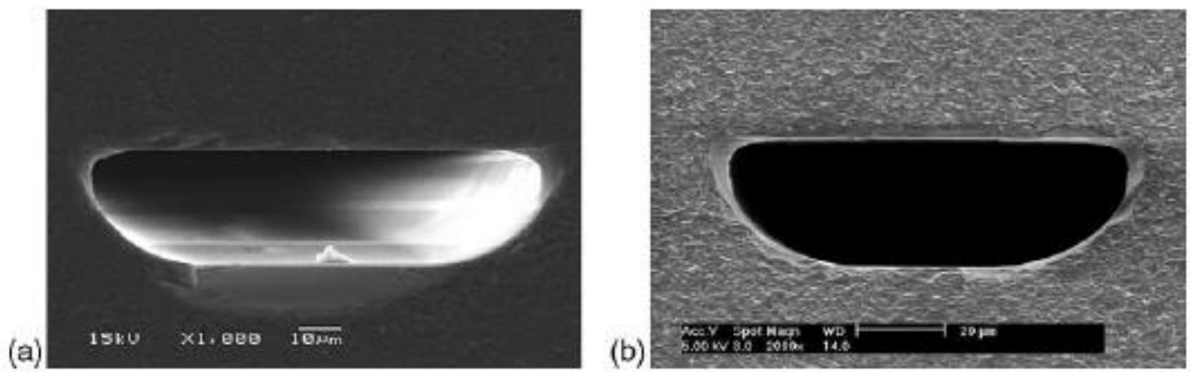
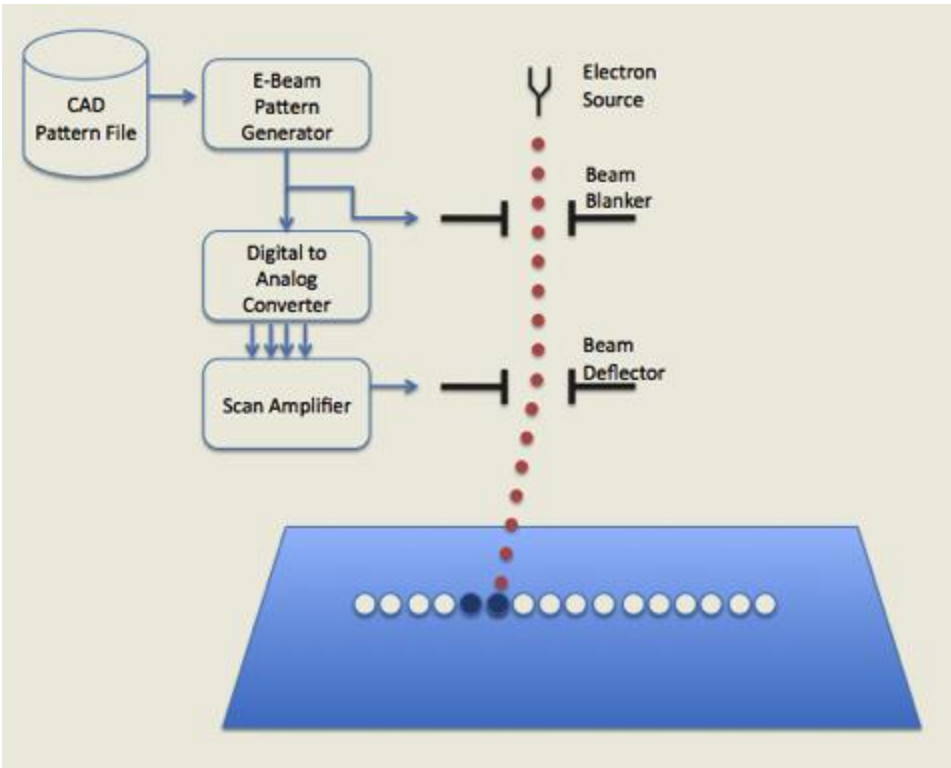


Fig. 6. SEM images of the cross-section of a microchannel in (a) quartz and (b) glass.

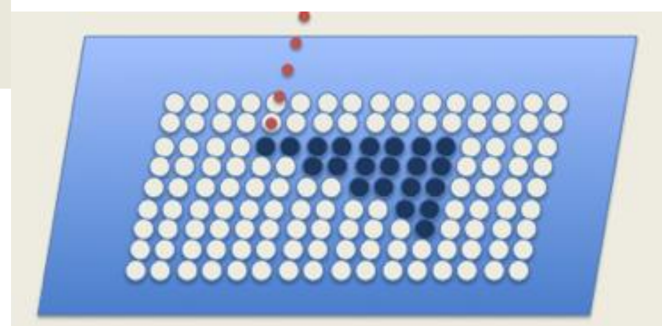
M.-S. Kim et al. / Sensors and Actuators B 107 (2005) 818–824

Original pattern generation



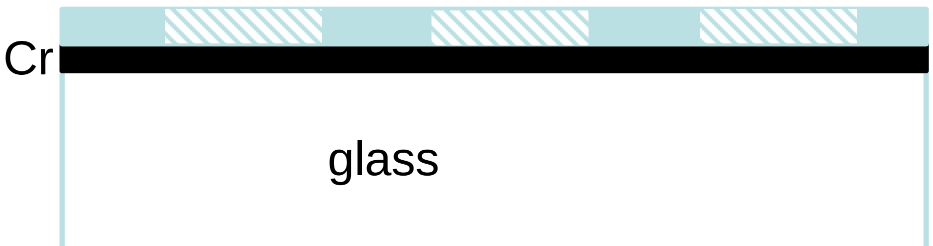
Laser/electron beam exposes polymer pixel-by-pixel, creating desired shapes.

Pixel-by-pixel the pattern emerges, slowly. Writing time hours, even days.



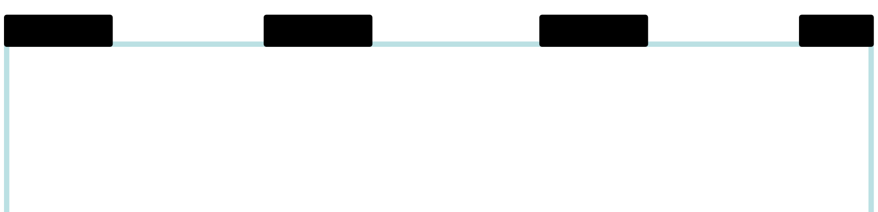
https://ebeam.wnf.uw.edu/ebeamweb/doc/exp/exp/exposure_basics_1.html

Creating a photomask



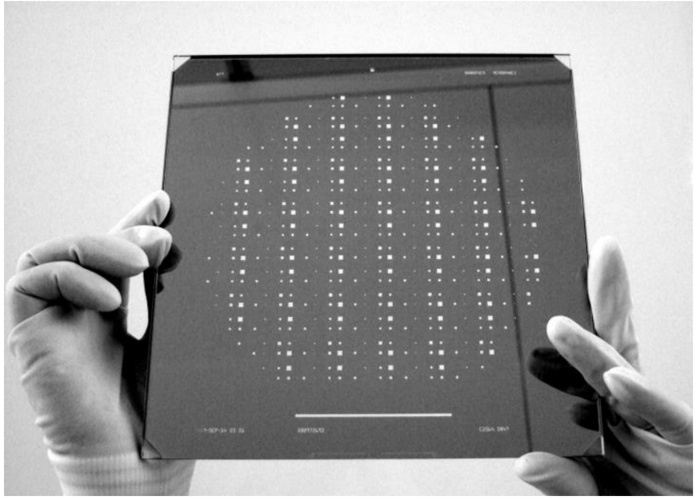
Laser/e-beam has caused local polymerization, which leads to solubility difference.

Polymer acts as a protective coating during Cr acid etching.

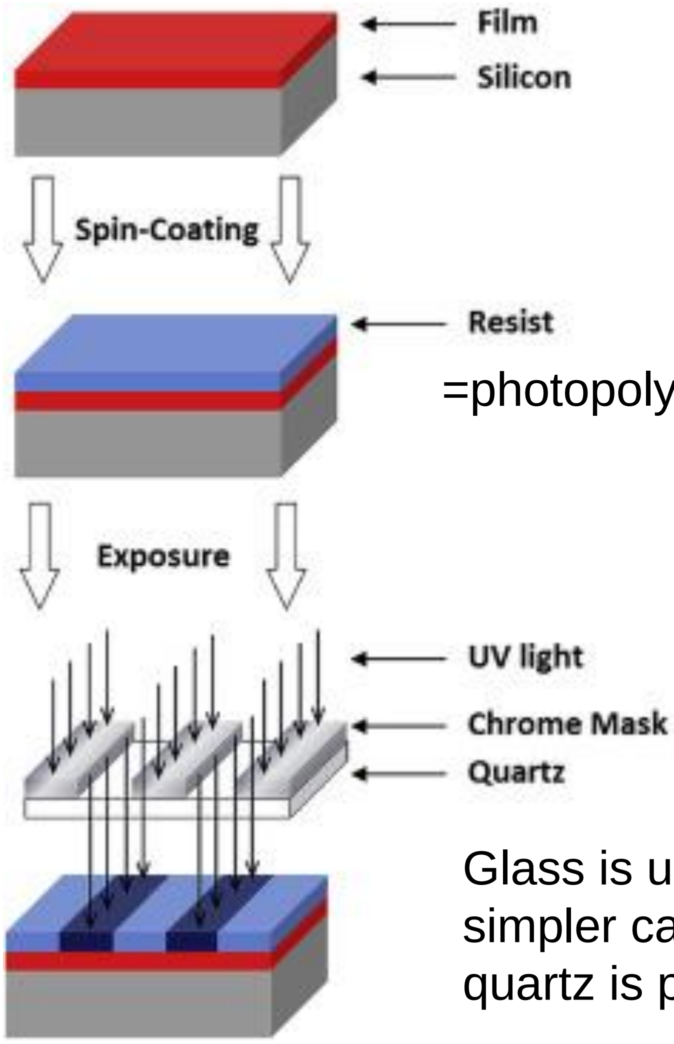


Polymer is removed, and we have Cr-pattern on glass plate. We call this a photomask.

Patterning with a mask



Photomask = glass plate with metal patterns (=opaque areas) and open areas (=transparent at UV-VIS wavelengths)



=photopolymer

Glass is used in simpler cases; quartz is premium.

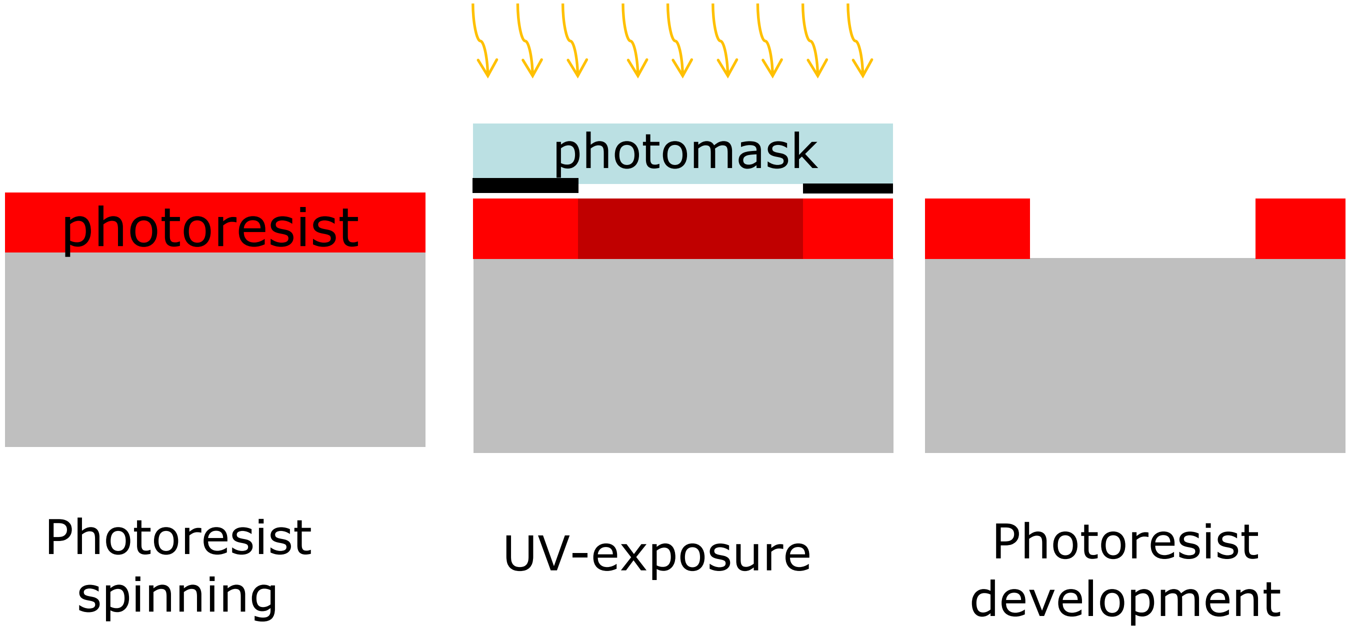
Lawson & Robinson: Frontiers of Nanoscience Volume 11, 2016, Pages 1-90

Lithographic patterns

Spin coat photoresist (photoactive polymer)

Expose thru mask with UV light (solubility changes)

Develop: exposed parts solubility differs from non-exposed ➔ pattern emerges



Mask cost vs. feature size

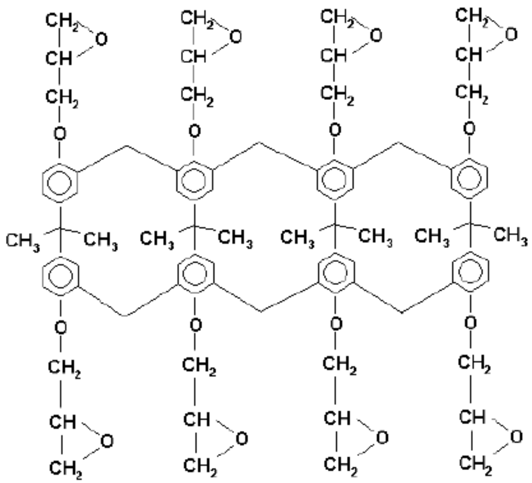
Office laser printer enables ~ 100 µm wide lines	0.1 €
Industrial laser printer ~ 10 µm wide lines	10 €
Dedicated microfabrication laser ~ 1 µm wide lines	1000 €
Electron beam system ~ 0.1 µm wide lines	10 000 €

Most microfluidic structures are in 10-100 µm range

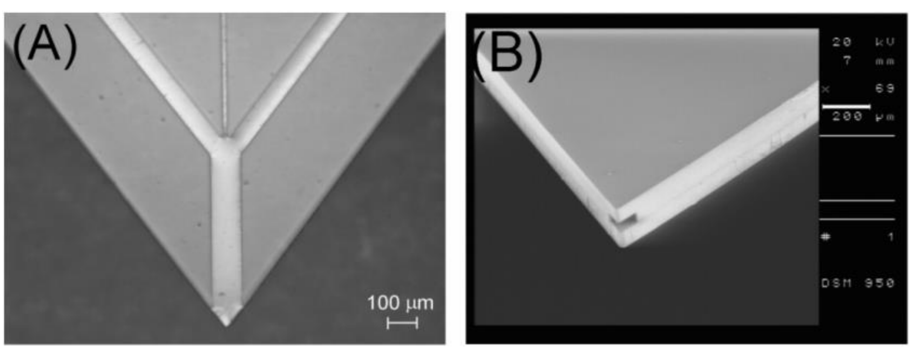
Fabrication example 1:
SU-8 Fabrication by UV-lithography

The SU-8 resist contains 3 principal components:

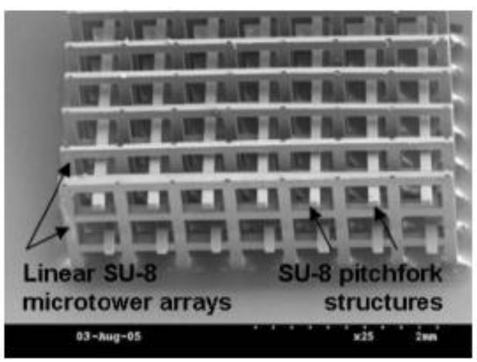
- Resin (defines mechanical & thermal properties)
- Photoinitiator (defines optical properties)
- Solvent (defines viscosity)



• Wide range of possible thicknesses by spin coating 100 nm – 1 mm



SU-8 electrospray ionization tip

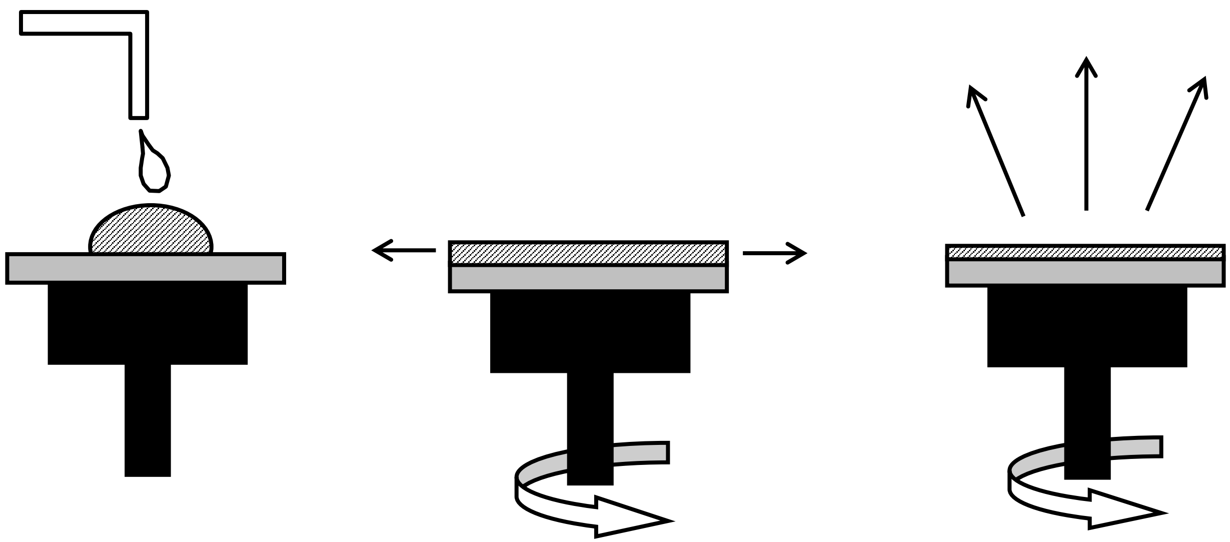


SU-8 neuron growth scaffold

Fabrication of SU-8 channels

SU-8 spinning

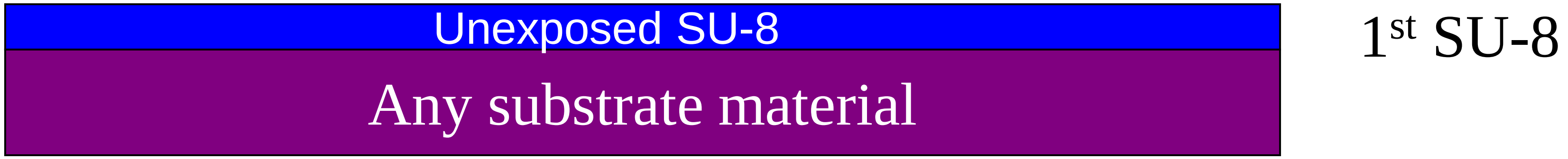
Viscosity and spinning speed determine thickness,
for example, 20 µm thick SU-8 layer for fluidic chip.



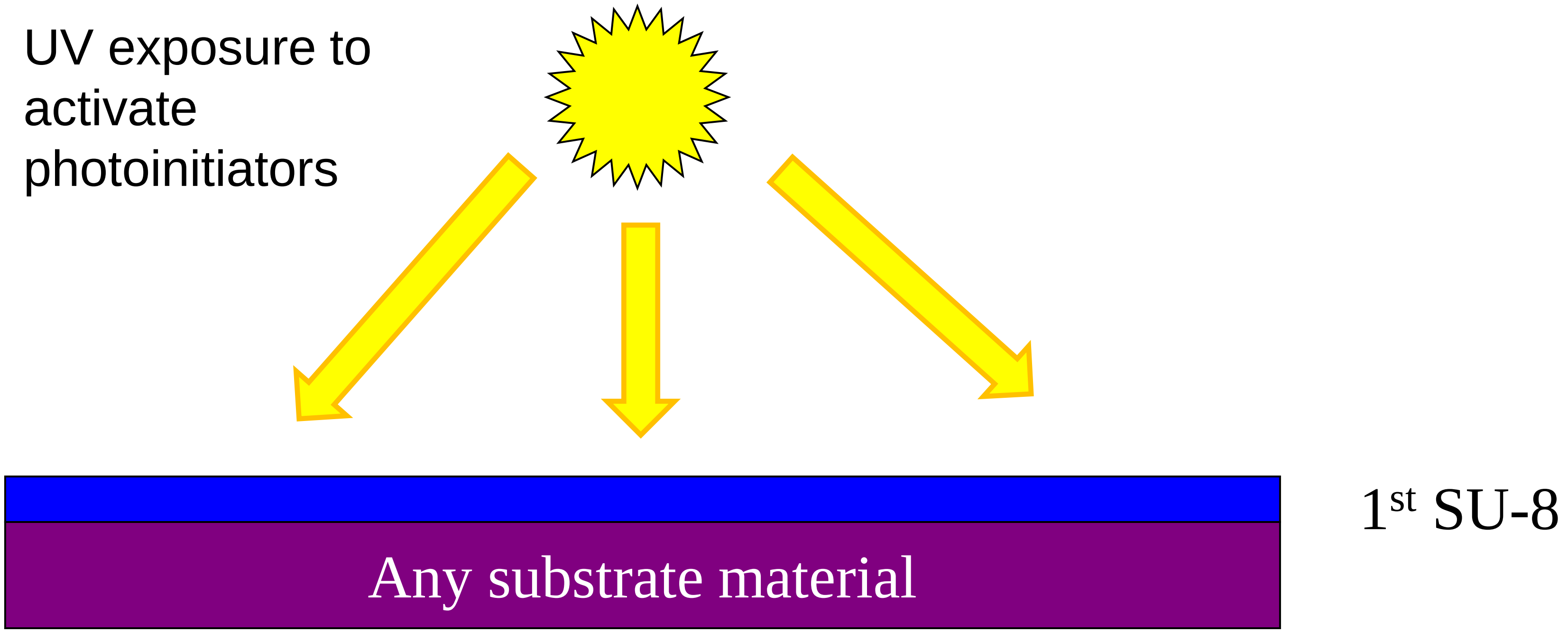
Franssila: Introduction to Microfabrication

Soft bake to remove solvent

Soft bake at 95°C to remove solvent, time depends on the thickness



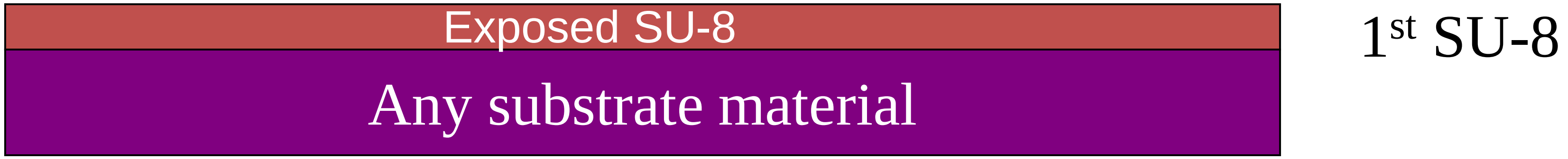
UV exposure to polymerize



Another bake to finalize polymerization

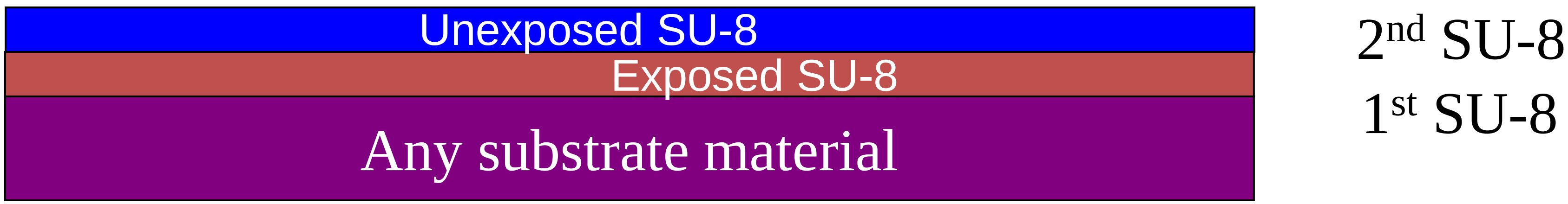
Post exposure baking at 95°C to cross link SU-8

Activated photo initiators and thermal energy allow for cross linking



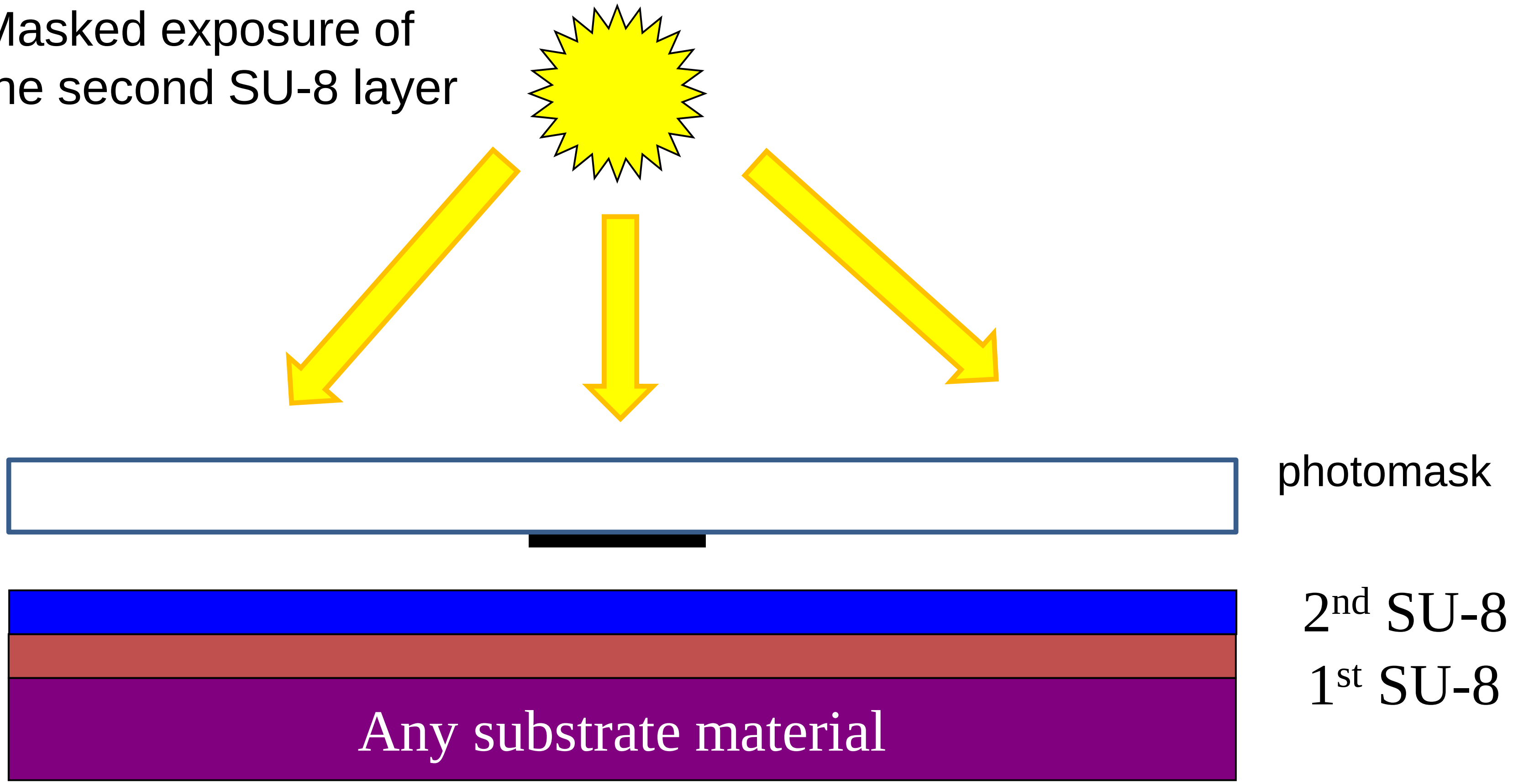
Second layer for channel formation

Spin coating second SU-8 layer
Soft bake at 95°C to remove solvent



UV-exposure thru a mask

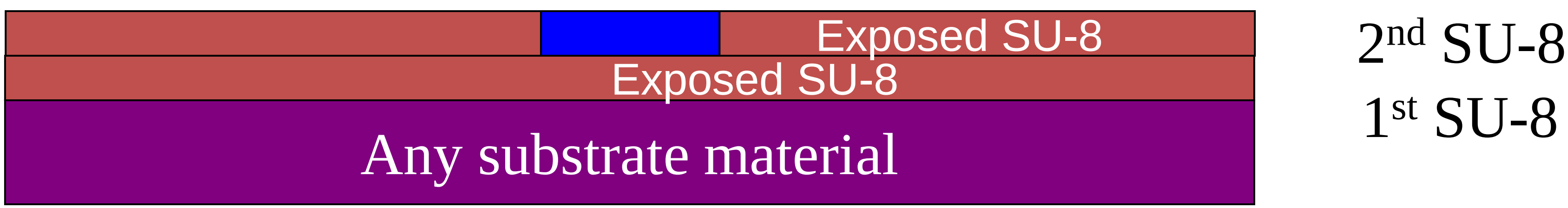
Masked exposure of the second SU-8 layer



Another bake…

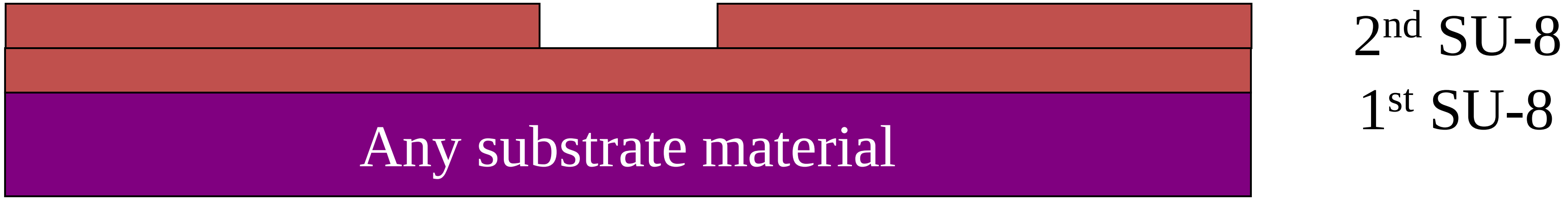
Post exposure baking at 95°C to cross link exposed SU-8

The area protected by mask does not crosslink due to inactive photo initiators



Development leads to channels

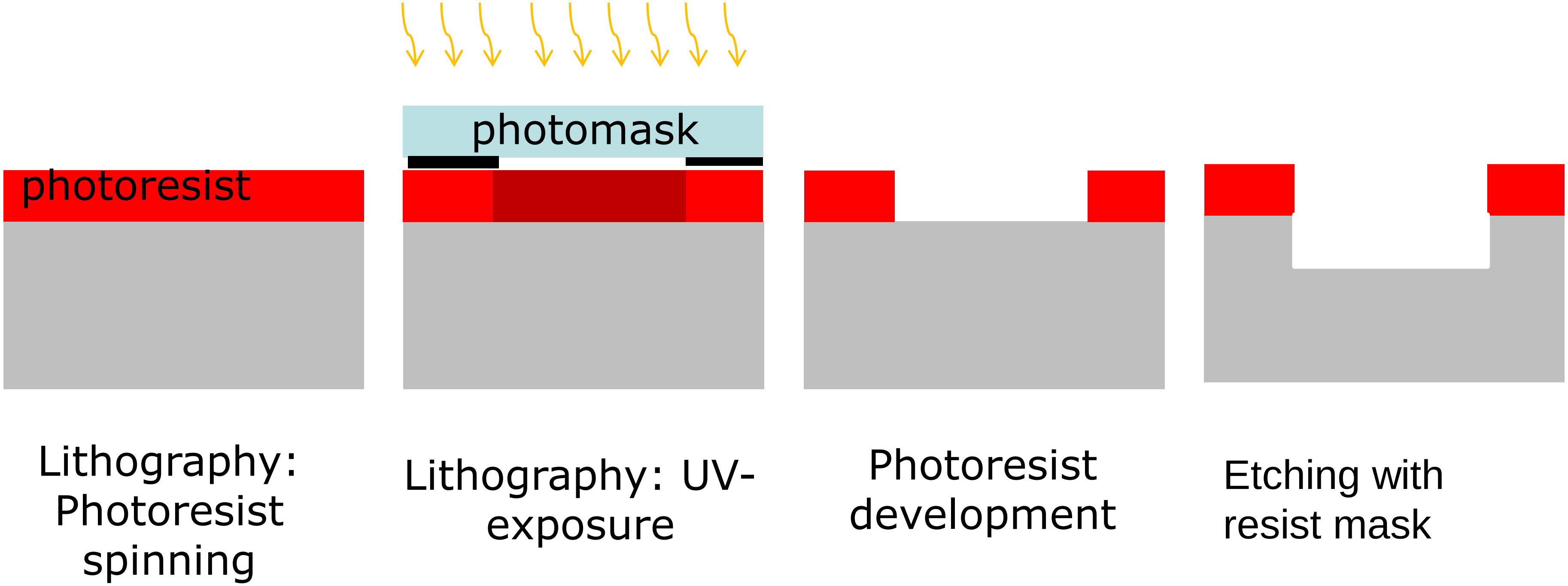
Developing the wafer in an organic solvent (PGMEA) to remove non-cross linked SU-8



Lithography + etching

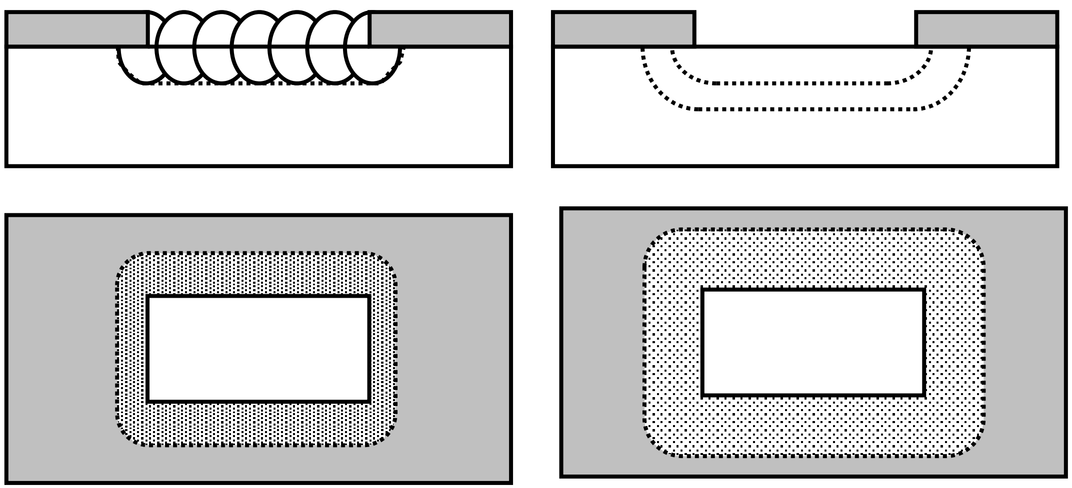
Use photoresist as a mask to etch a channel

Benefit: we can etch many materials, and we are not limited to polymers.

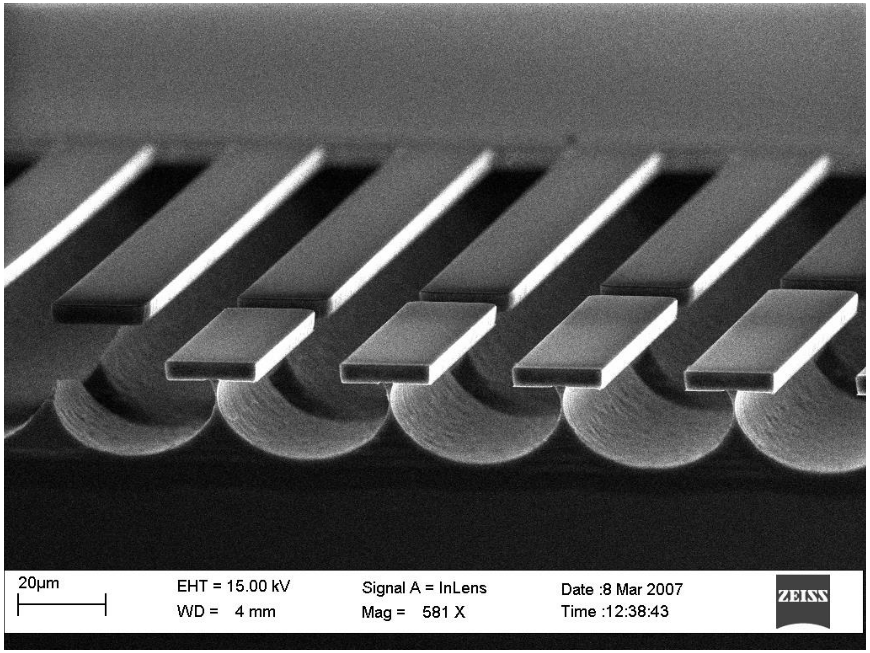


Isotropic etching

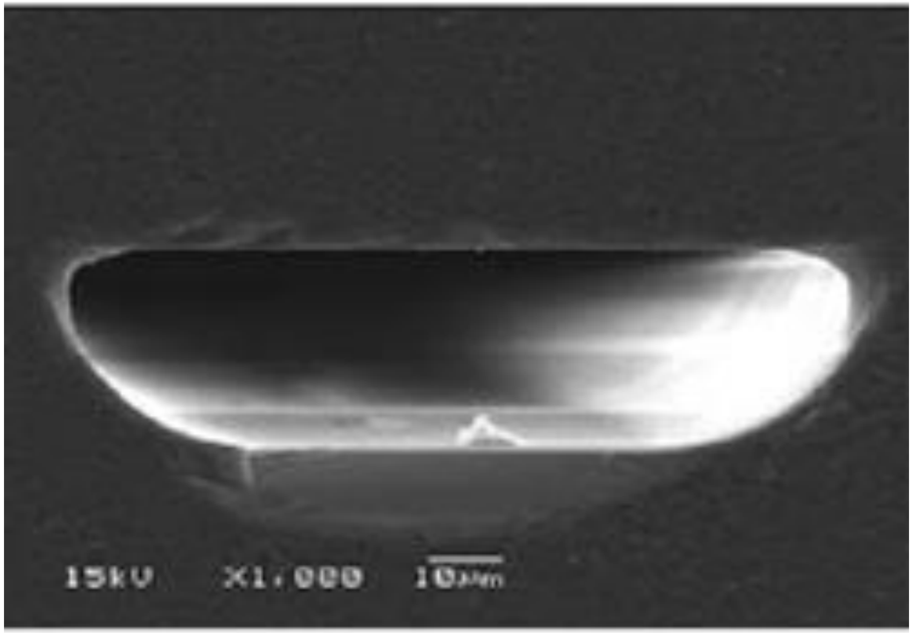
- Proceeds as a spherical wave
- Undercuts structures (proceeds under mask)
- Most wet etching processes are isotropic
- HF etching of SiO_2 and glass, H_3PO_4 etching of Al



Isotropic etch profiles

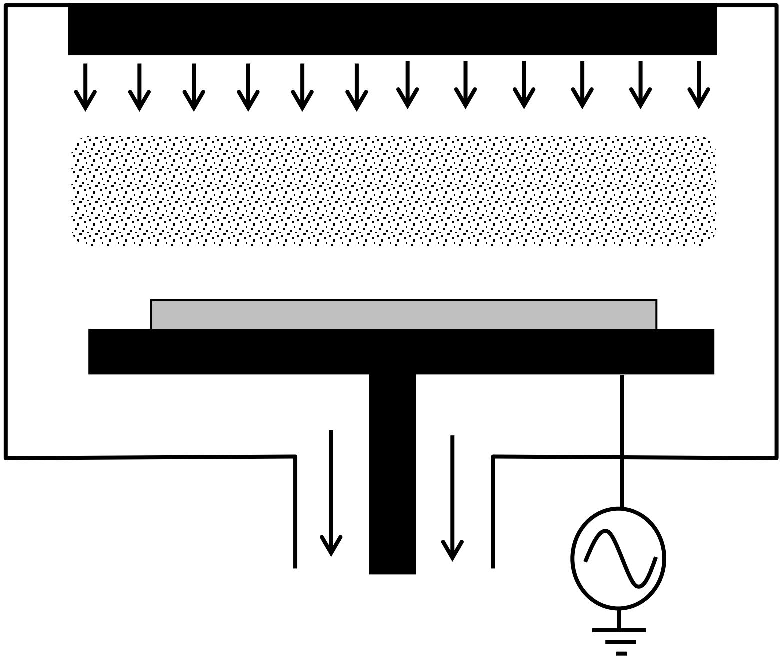


Silicon etched in isotropic SF_6 high pressure plasma



Glass wafer etched in HF (with roof bonded)

Plasma etching/RIE



vacuum 1 mTorr-3 Torr

13.56 MHz RF

SF6 feed gas to etch silicon:

e- + SF_6 ➔ e- + SF_5* + F*
Si + 4F* ➔ SiF_4 (g)

Reactive neutrals very reactive

➔High etch rate

Ions provide directionality

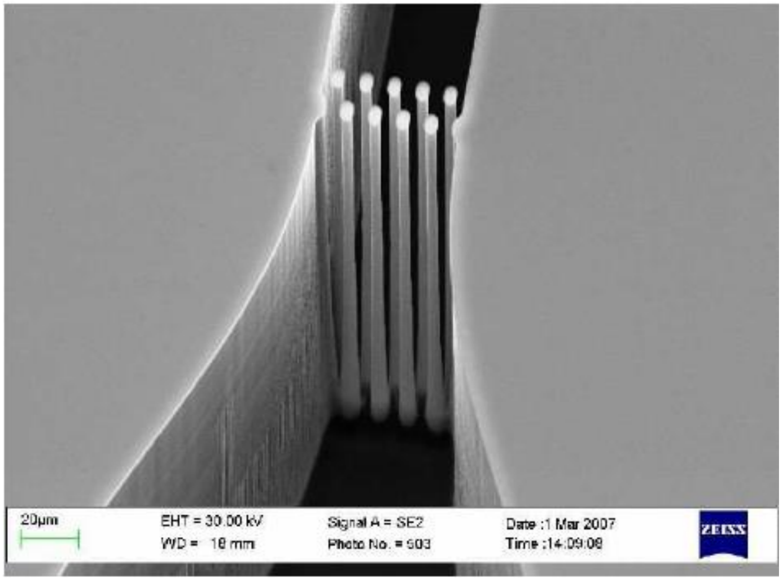
➔ vertical walls

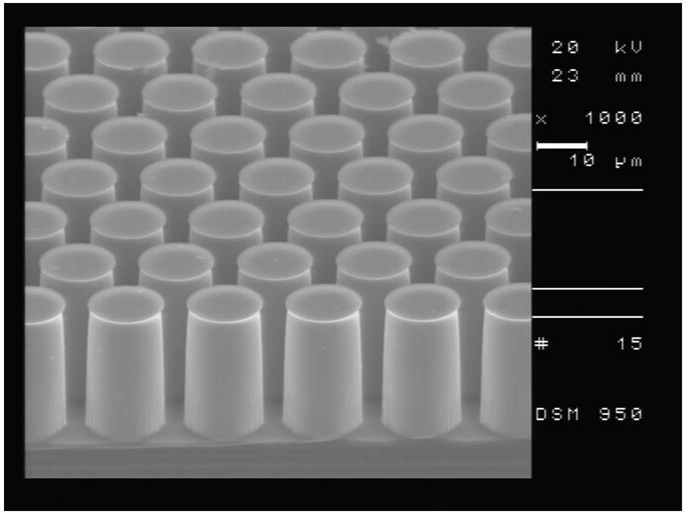
Note 1: RIE = Reactive Ion Etching = plasma etching
Note 2: ions have minor role; excited neutrals major

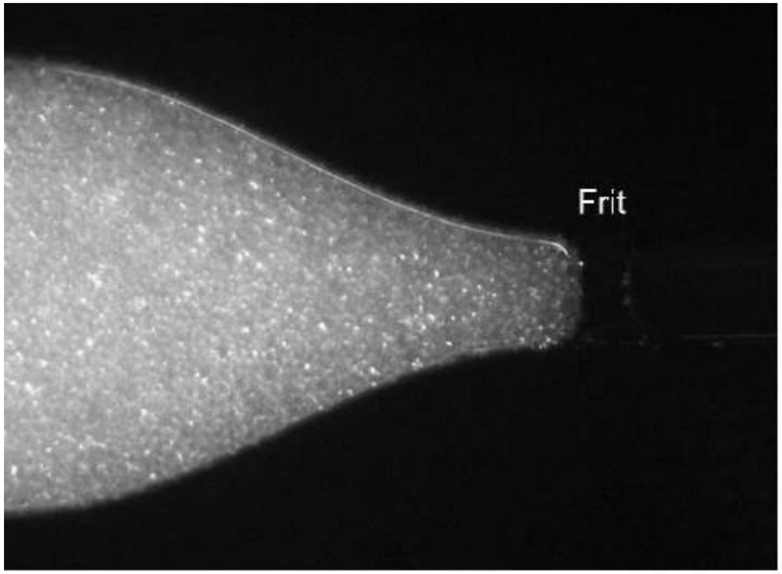
Plasma etching of silicon

Possibility to make vertical walls

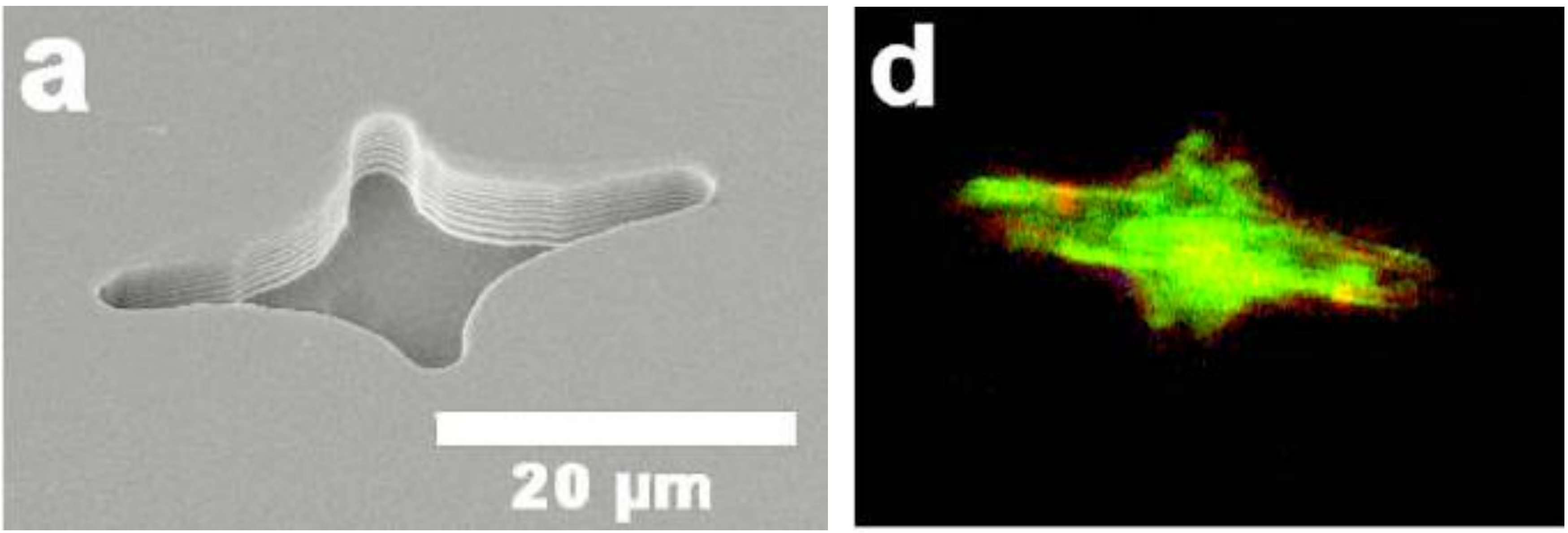
➔ narrow gaps





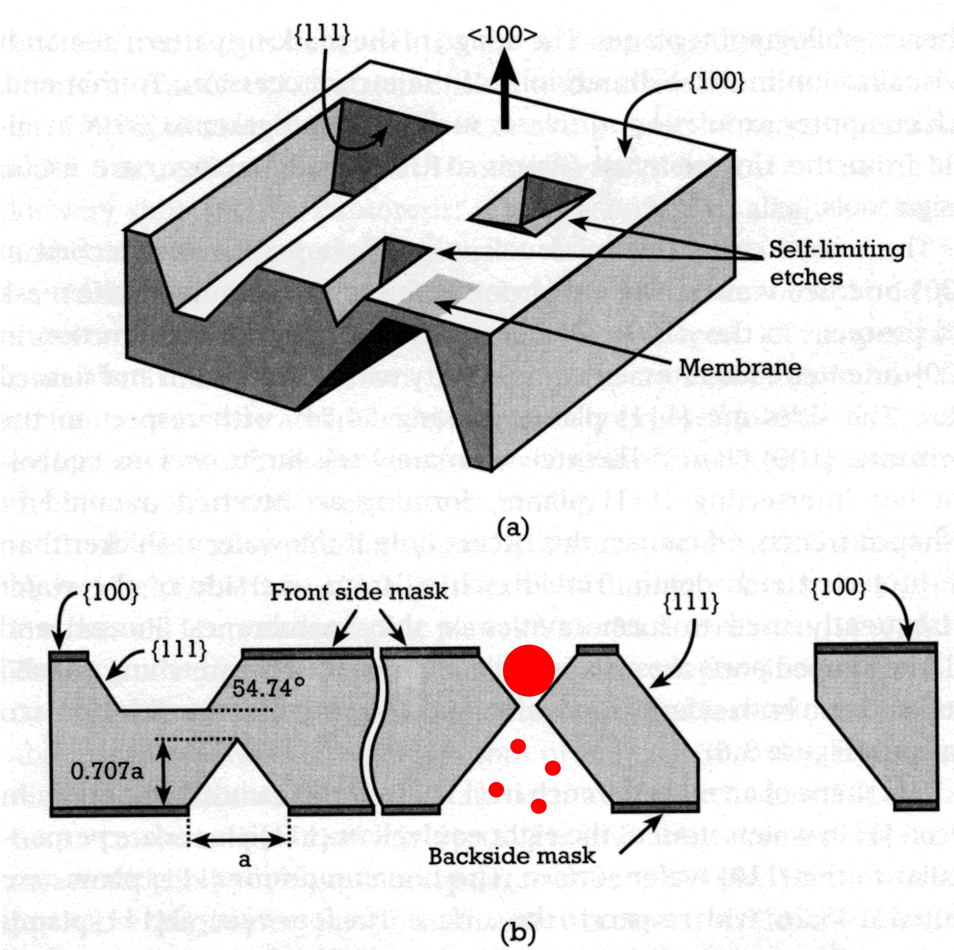


Cell growth forced into arbitrary shapes formed by plasma etching



Dusseiller *et al.*: Microfabricated three-dimensional environments for single cell studies

Alkaline etching of silicon



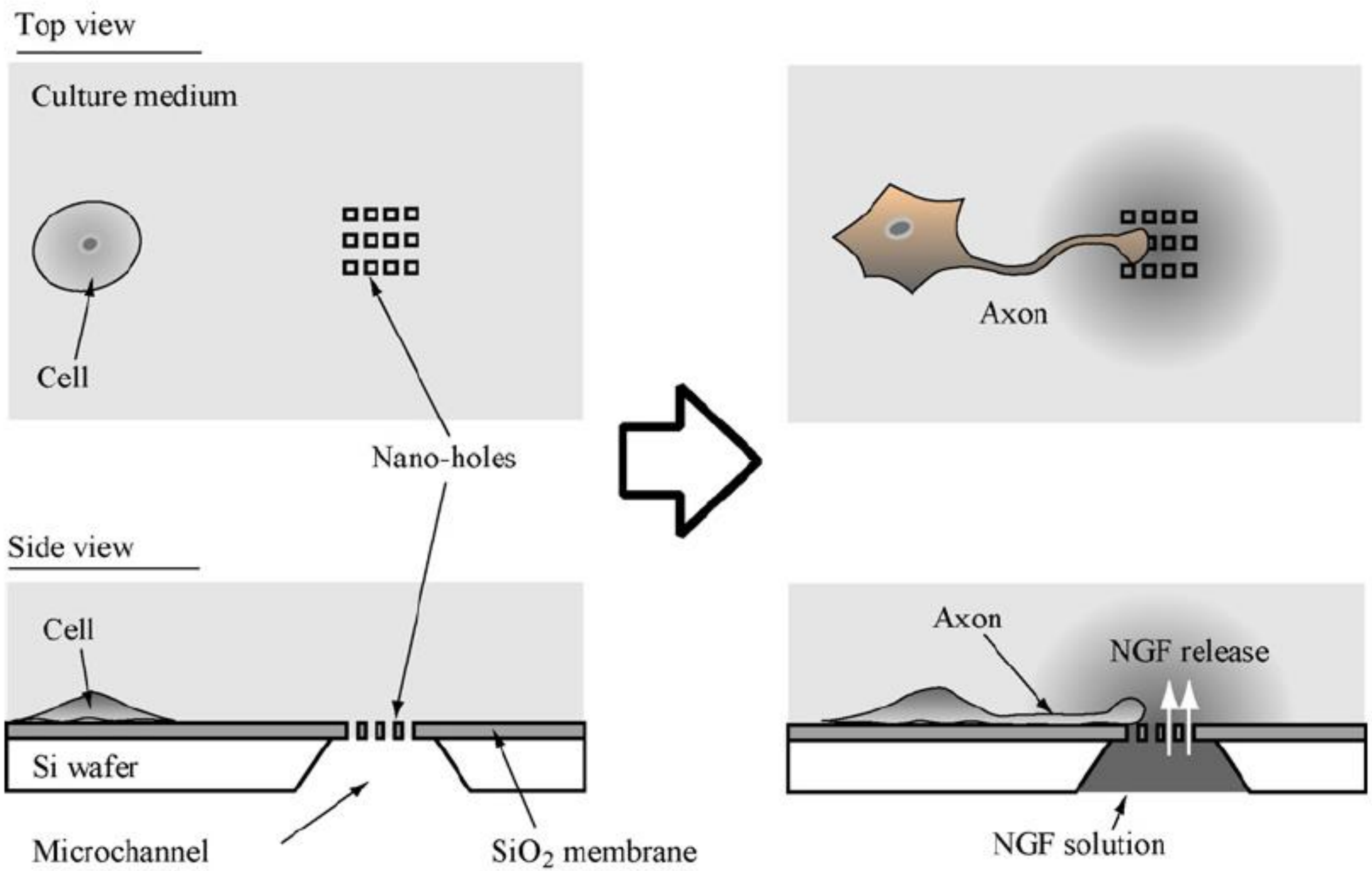
(100) and (110) crystal planes etch fast: 1 µm/min typical

(111) plane etches slow, 10 nm/min typical

Uses SiO_2 as etch mask

(photoresist and oxide etching is used to make patterns in oxide, and this is immersed in KOH)

Cell growth stimulator: wet and plasma etching



Y. Nakashima, T. Yasuda / Sensors and Actuators A 139 (2007) 252–258

Three ways to etch silicon

Isotropic wet etching:

- easy and fast
- not suitable for small structures

Plasma etching:

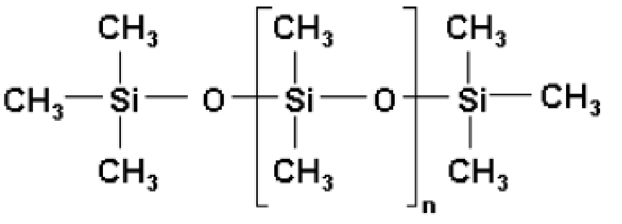
- when vertical walls are needed
- when small structures are needed

Alkaline wet etching:

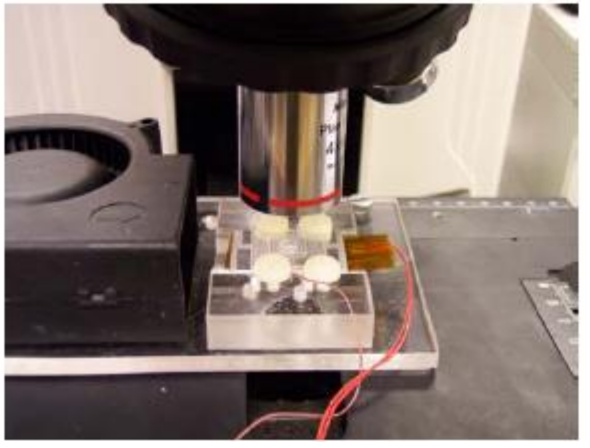
- crystal plane defined shapes
- only applicable to silicon

Fabrication example 2: PDMS by replication molding

PDMS elastomer is the most widely used polymer for microfluidics



- Very easy to fabricate by casting
- Non-toxic
- Excellent optical properties
- Thermally stable but huge CTE
- Ease of sealing and bonding
- Permeable to O_2 and H_2O (not always positive)



PDMS PCR chip

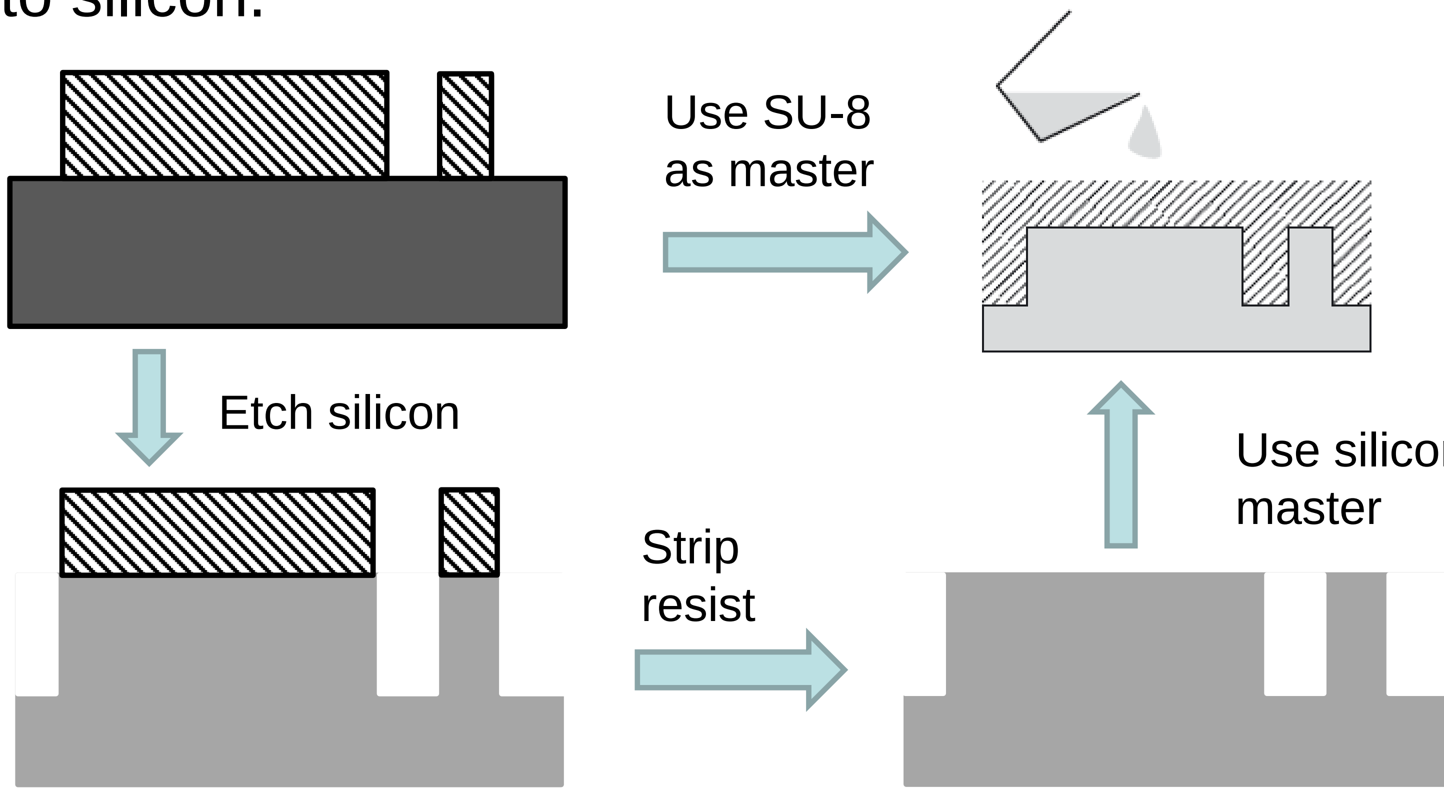
Problems:

- Hydrophobicity
- Poor solvent compatibility
- Elasticity (sometimes)

PDMS replica moulding

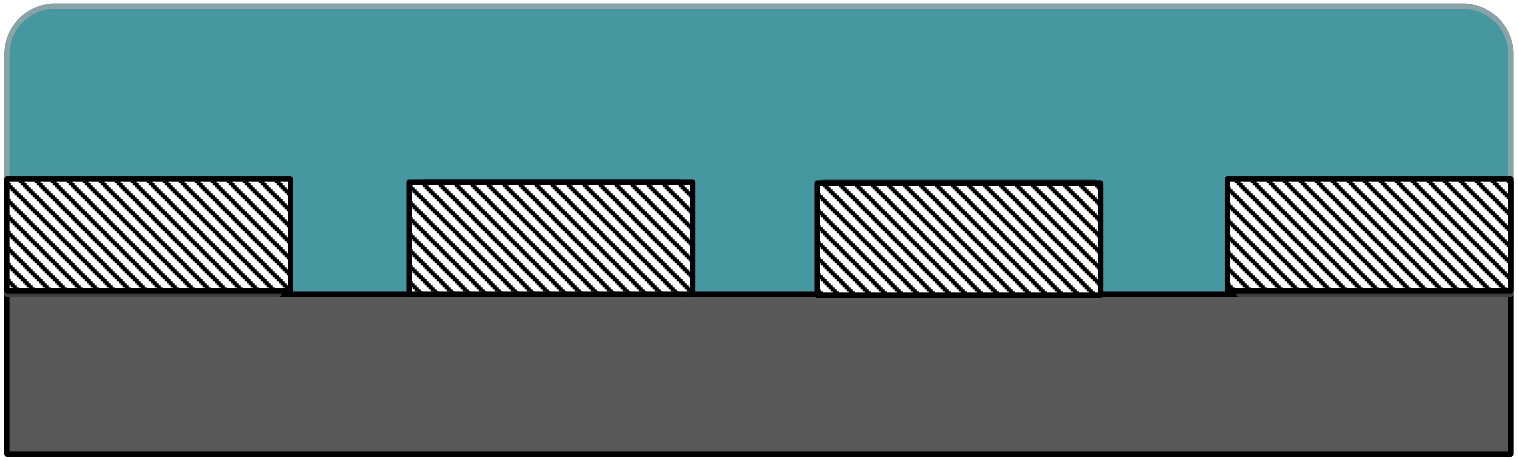
Lithography as usual, resist patterns.
Use resist directly, or etch into silicon.

Then casting liquid PDMS prepolymer on your pattern



PDMS replica moulding (2)

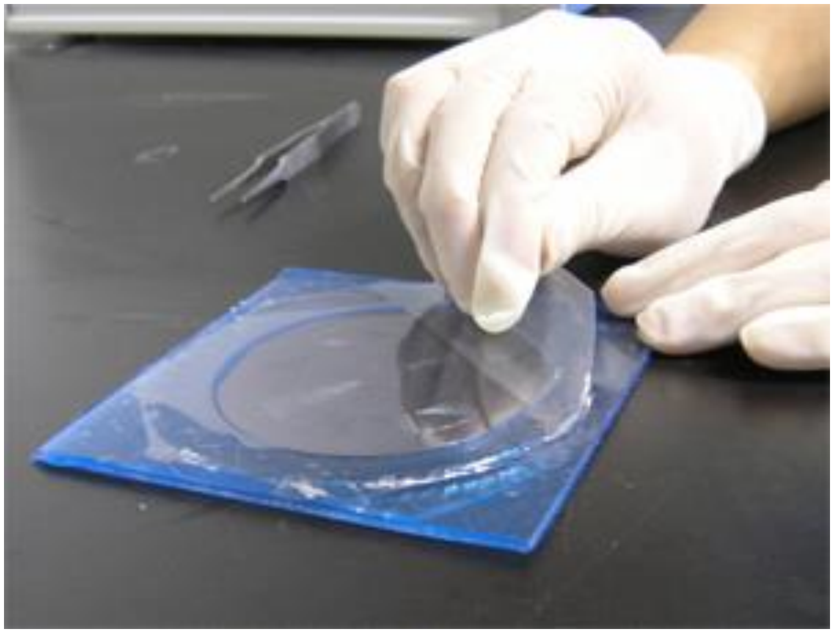
- PDMS crosslinking by thermal activation.



•Possible curing times e.g.

- 1 day in room temperature
- 3 hours in 50°C
- 1 hour in 90 degrees

PDMS properties



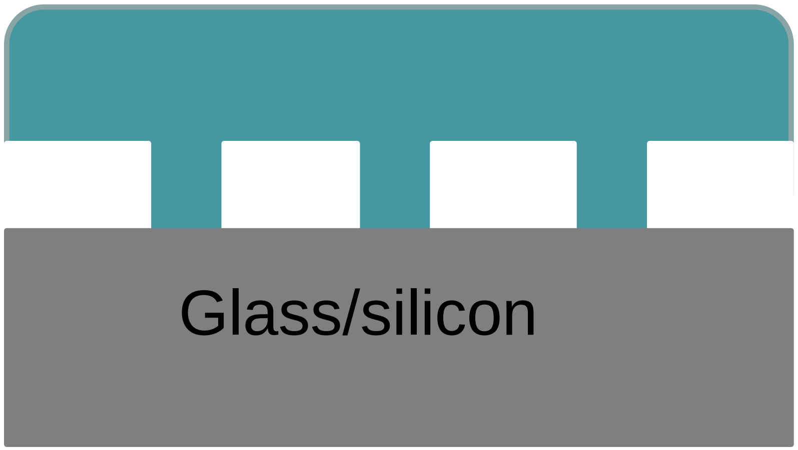
Peeling from master mould

Elastic
➔ peeling easily from master wafer

Transparent
➔ optical detection

OK with fluids of biological interest (water-based); but adsorption may be a problem

PDMS properties (2)



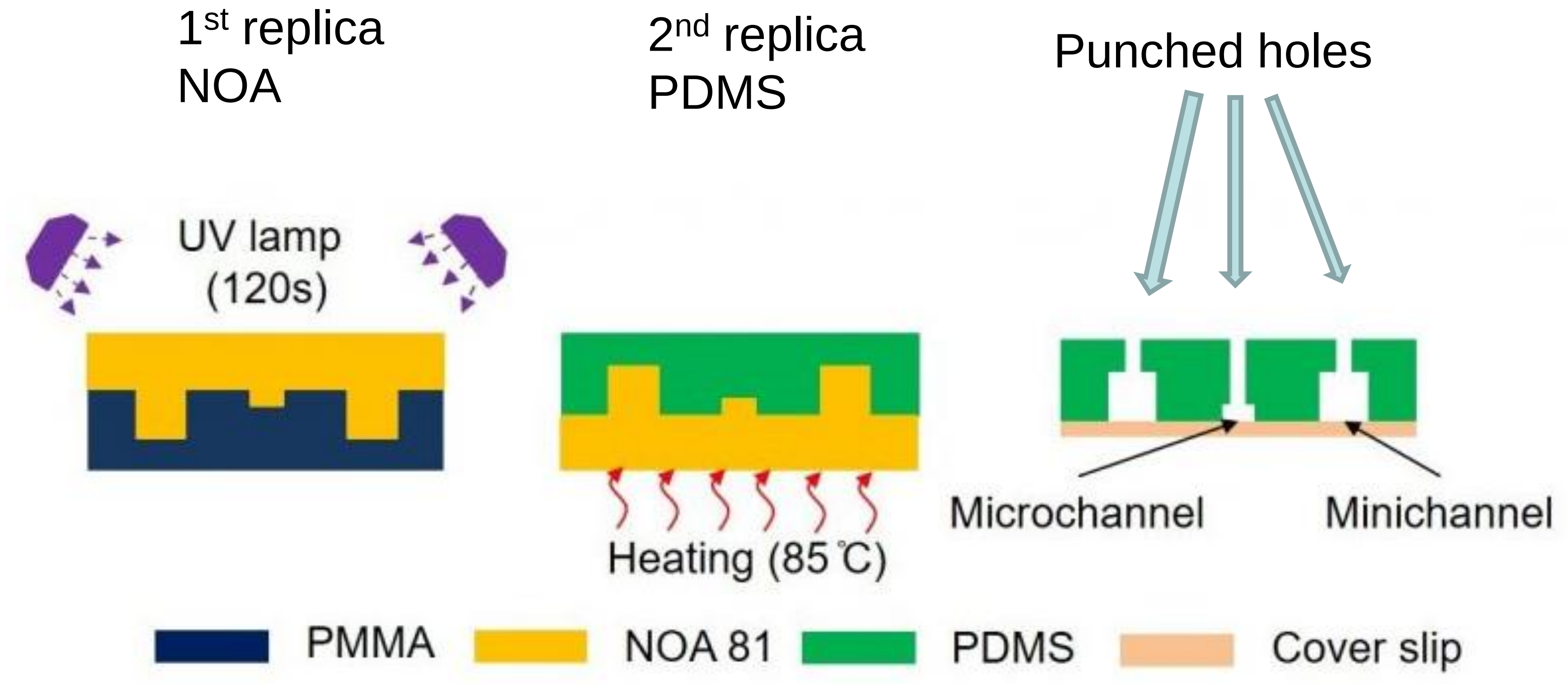
Bonding to a silicon wafer

Self-adhesive
➔ bonding to a clean wafer

Soft
➔ makes intimate contact even with a rough surface

Water vapor and oxygen permeable
➔ cells can breathe under PDMS (but water will escape if system is heated)

Double replication

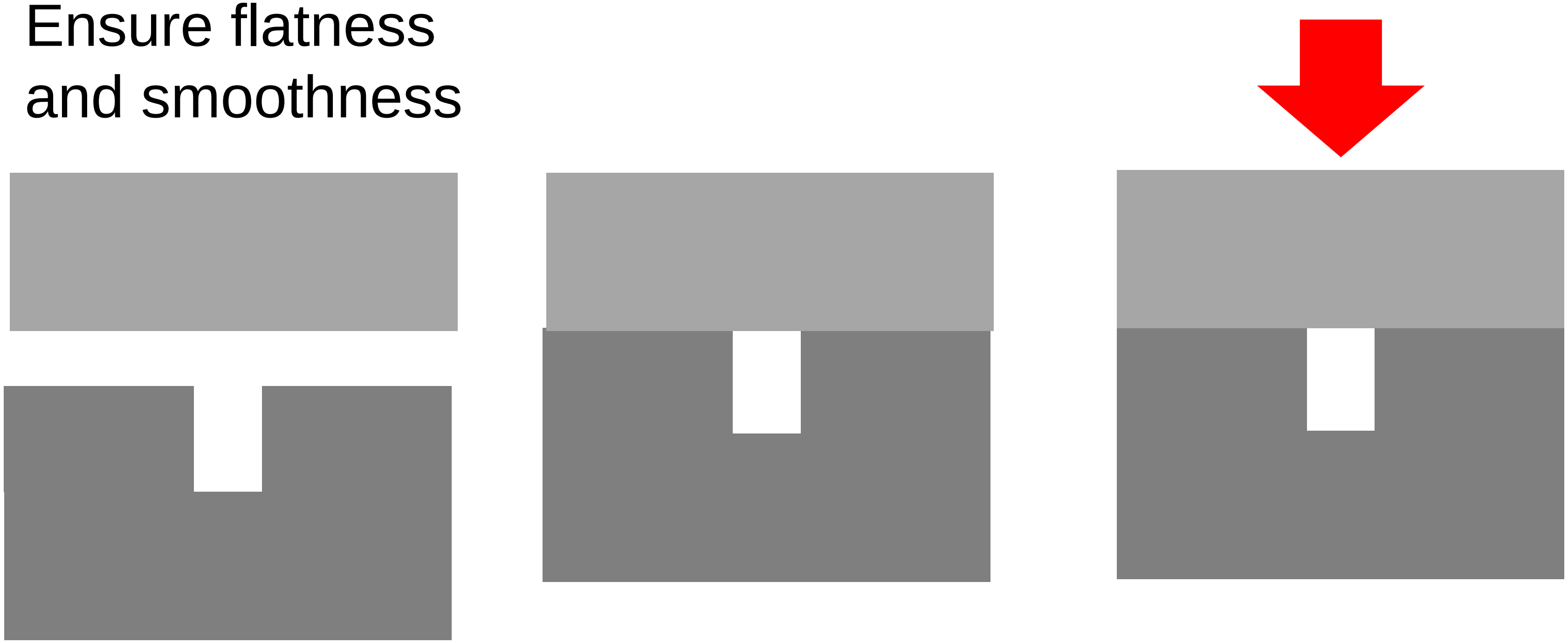


NOA= Photoactive polymer

Zhibin Yan, Xiaoyang Huang, Chun Yang. Rapid prototyping of single-layer microfluidic PDMS devices with abrupt depth variations under non-clean-room conditions by using laser ablation and UV-curable polymer. Microfluid Nanofluid (2017) 21:108

Channels by bonding

Ensure flatness and smoothness

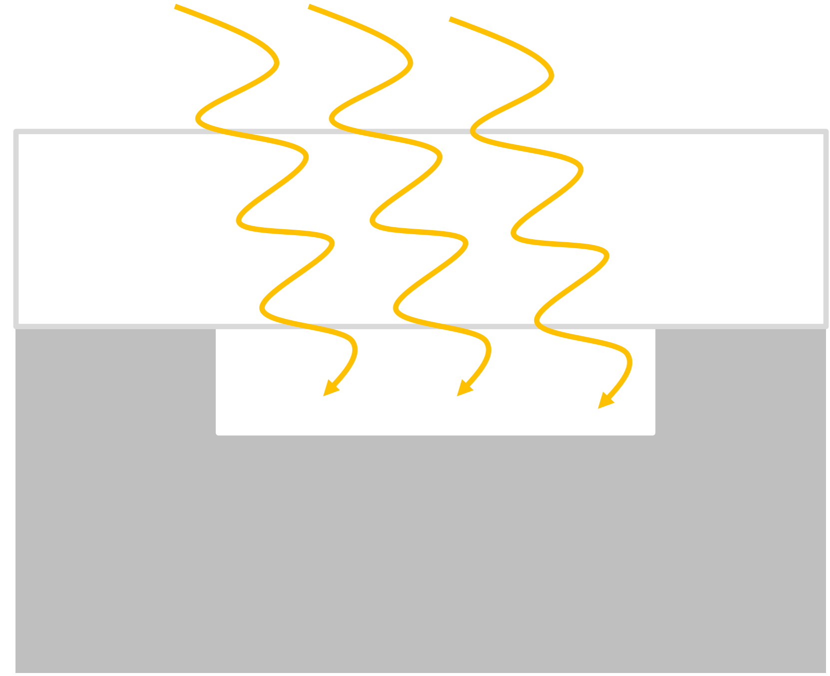


Clean the surfaces
1) Particle removal
2) Surface chemistry

Join the wafers (at room temperature)

Apply force (pressure, heat, voltage) to strengthen the bond

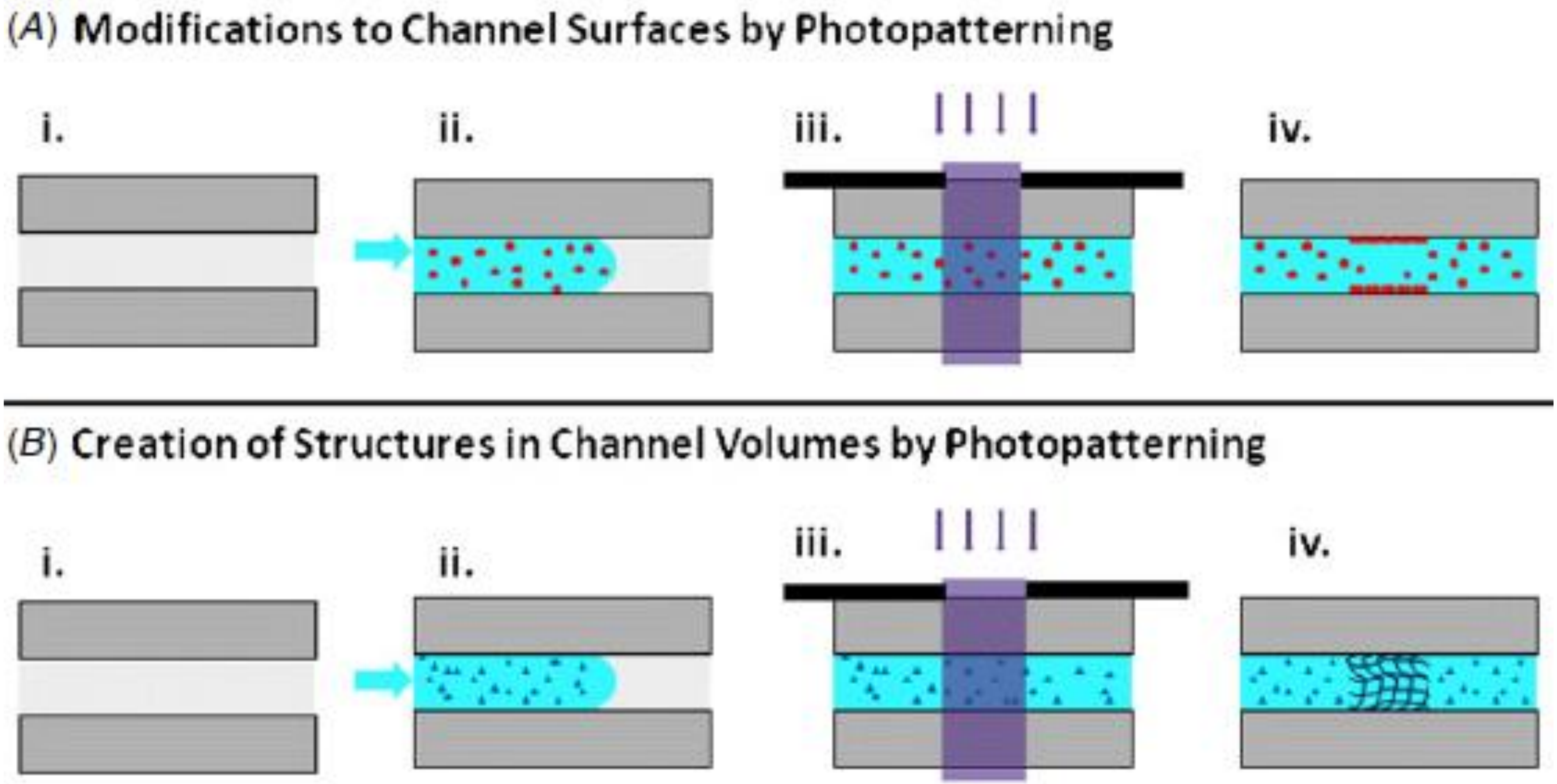
Etched channel + bonded roof



-more permanent materials (e.g. silicon instead of polymer)
-fewer materials

-transparent roof
-dielectric material (depends if an electrical device)

Patterning inside microstructures



A M Tentori and A E Herr 2011

Direct/thermal bonding

Two identical materials bonded:

-silicon-to-silicon ca. 1000^{o}C
-glass-to-glass ca. 600^{o}C
-polymer-to-polymer ca. 100-200^{o}C

Polymer thermal bonding

Raise temperature above T_g

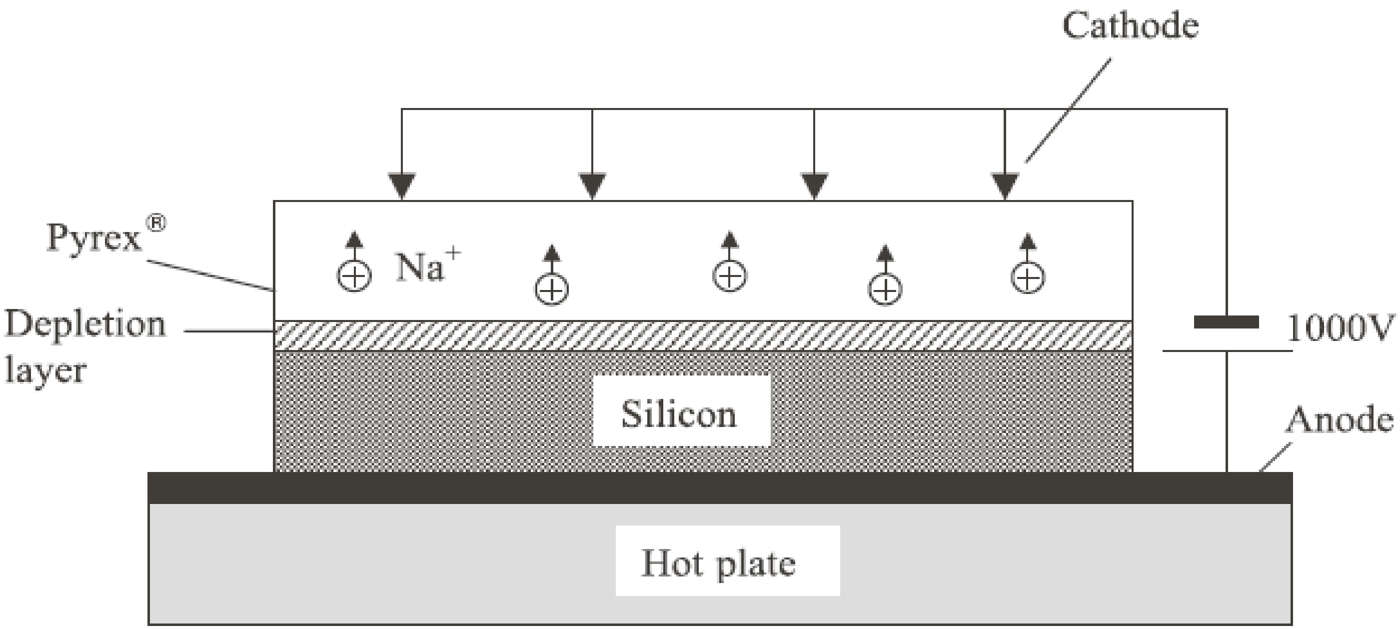
➔ softening

➔ intimate contact

➔ cool down below T_g

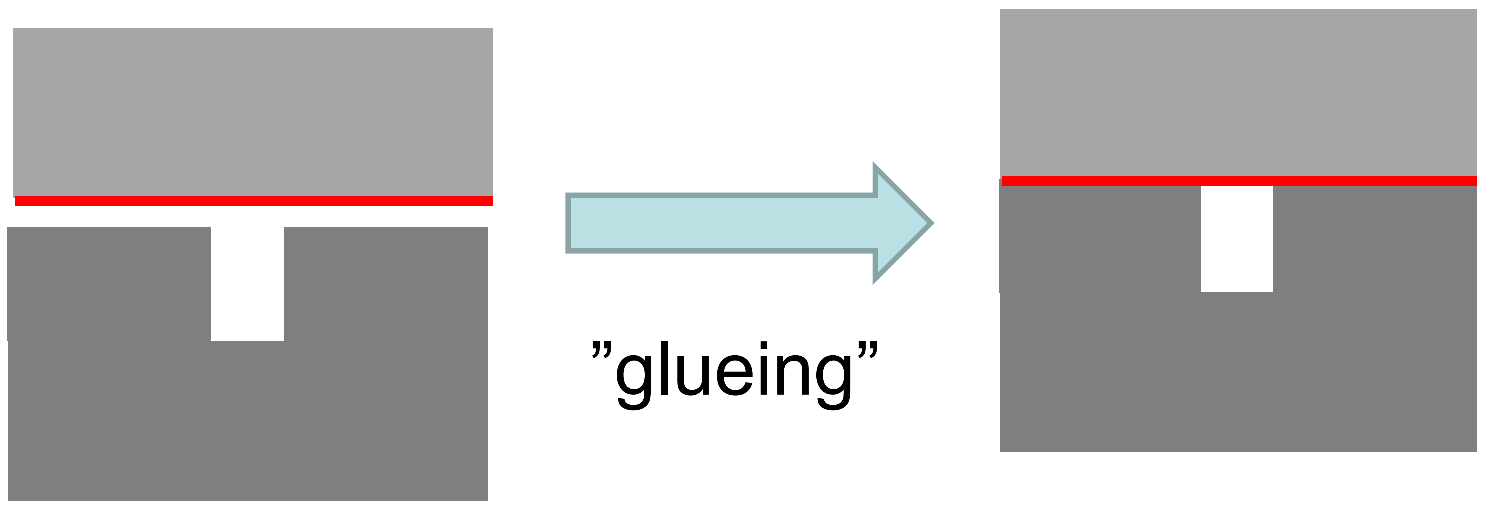
➔ bond interface indistinguishable from bulk materials (because same bonds !)

Si-glass anodic bonding



Double Sided Heating:	**e.g. 350°C**
High Voltage:	**e.g. 500V**
Bonding Atmosphere:	**e.g. 1 mbar nitrogen**

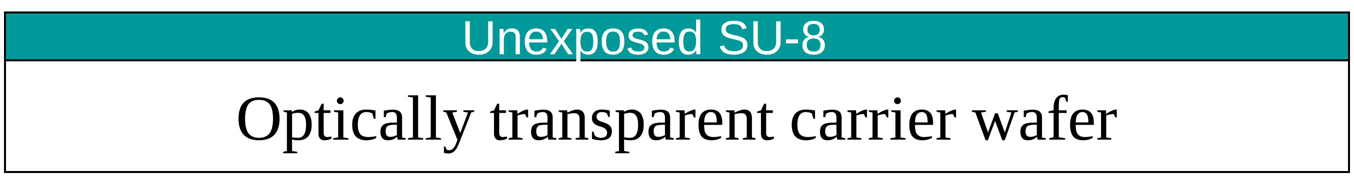
Adhesive bonding



-any two materials can be joined by polymer interlayer
-glue remains in the structure, and may react with chemicals
-temperature is ca. 100-200^{o}C only
-no pressure needed

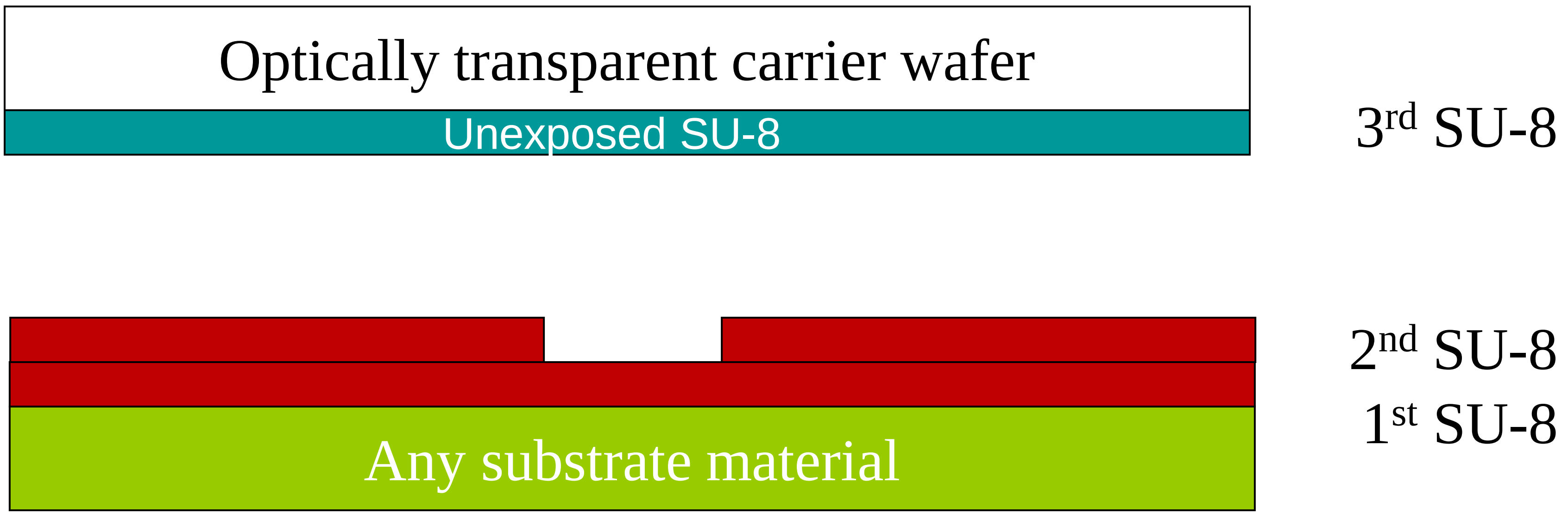
Bonding of SU-8 channels

Start of the bonding process.
Spin coating a SU-8 layer on top of a temporary transparent substrate
Soft bake at 95°C to remove solvent



3rd SU-8

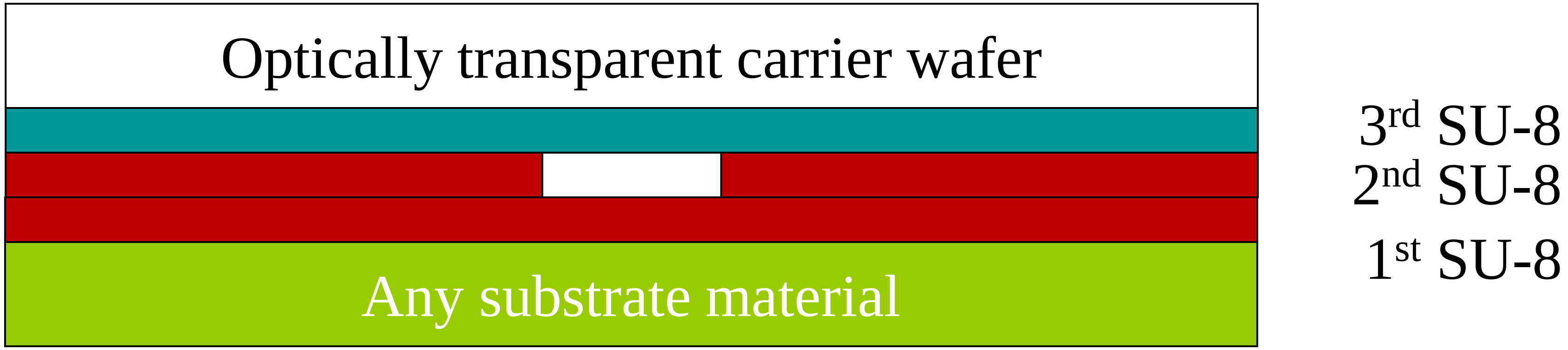
Carrier wafer with adhesive layer



Preliminary bonding @ RT

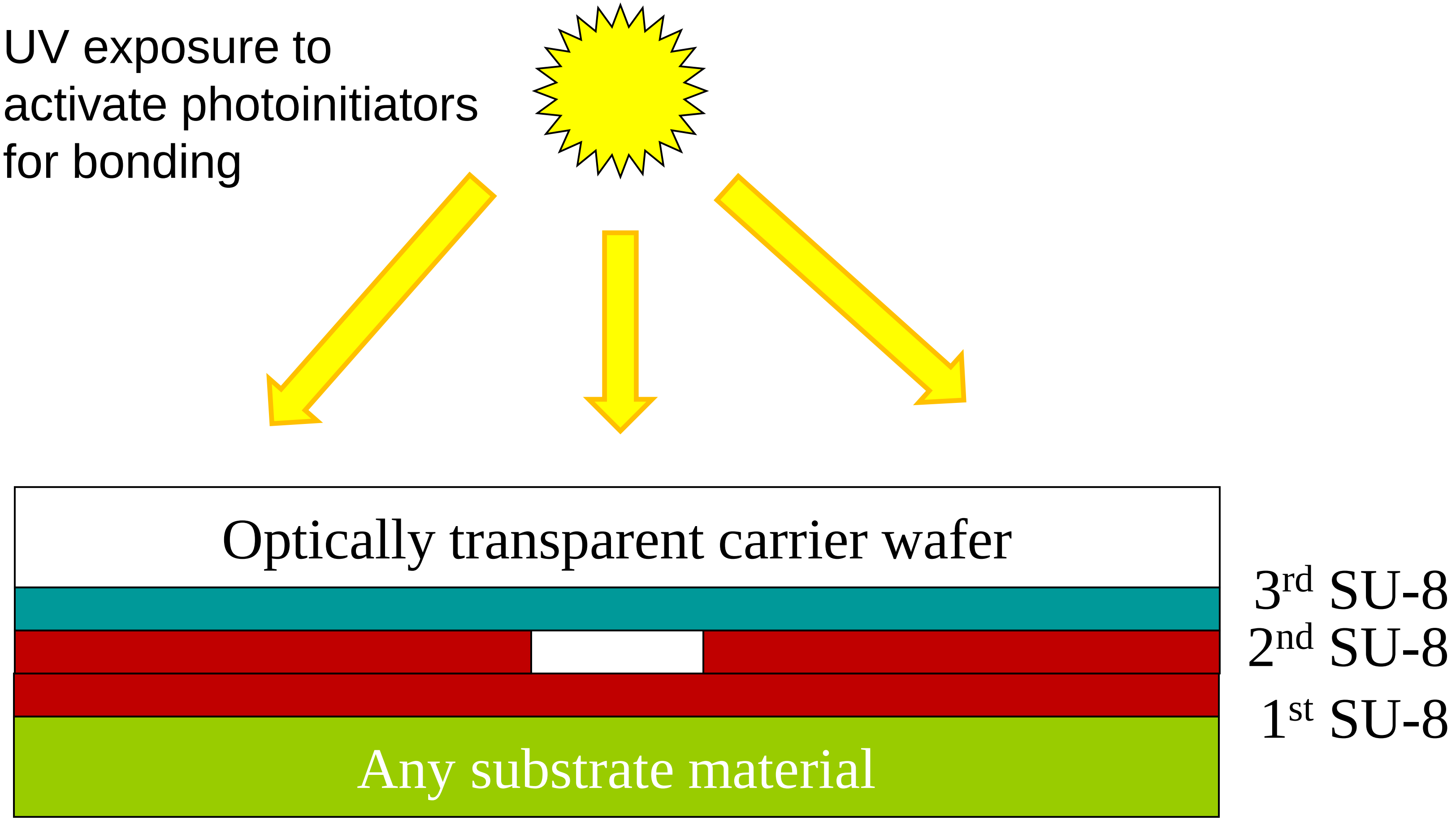
Bonding a cover lid which has an unexposed SU-8 layer

This is adhesive bonding (with thermal extra energy)



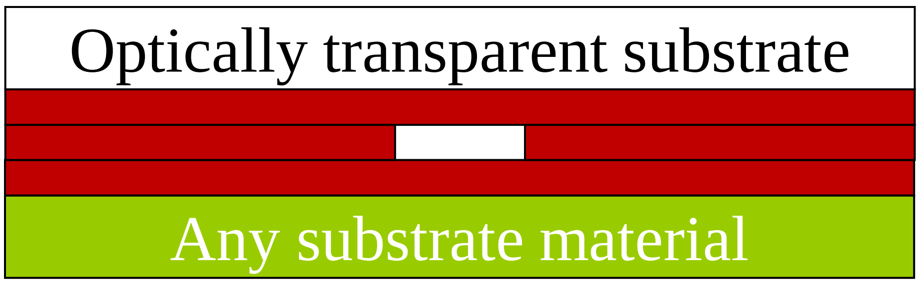
UV-exposure thru carrier wafer

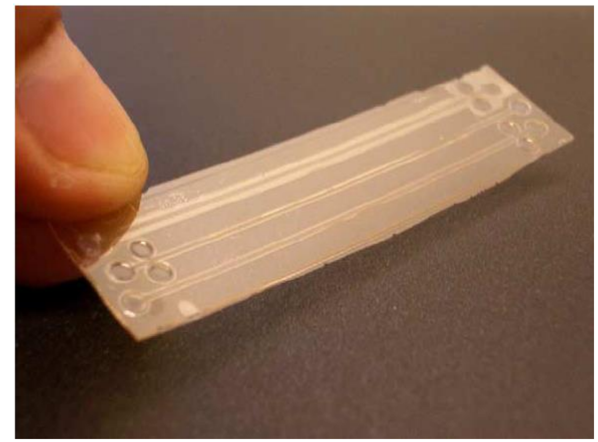
UV exposure to activate photoinitiators for bonding



Final curing of exposed SU-8

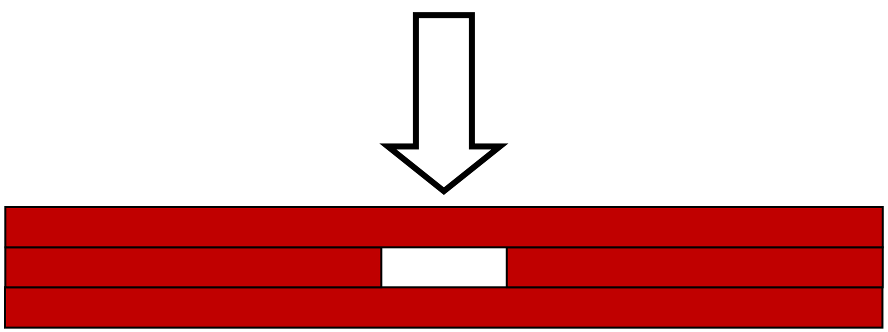
Post exposure bake to complete the bonding

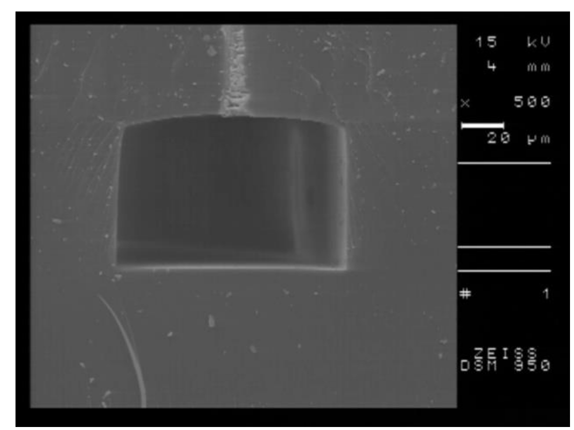




Freestanding SU-8 microchip

Removing the substrates (optional)

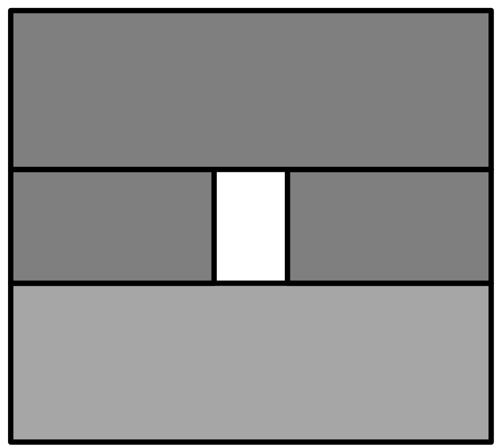




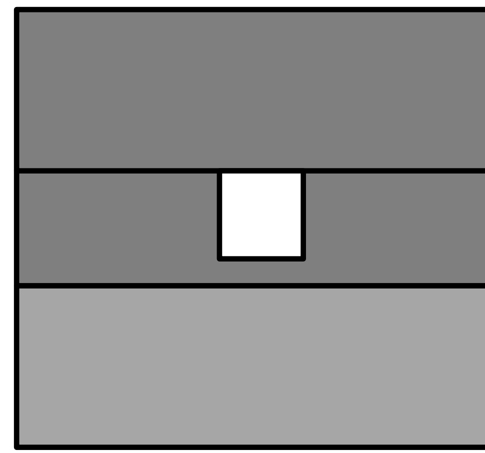
Number of materials

Substrate, wall and cap
➔ 3different materials

Walls and cap identical
➔ 2 materials



Floor, wall and cap same material
➔ only 1 material



Wetting, charging, adsorption… easier to control if we have fewer materials present.

Channel considerations

Materials

- silicon (semi)conducting, opaque
- glass (insulator), transparent
- polymer (insulator), transparent or opaque

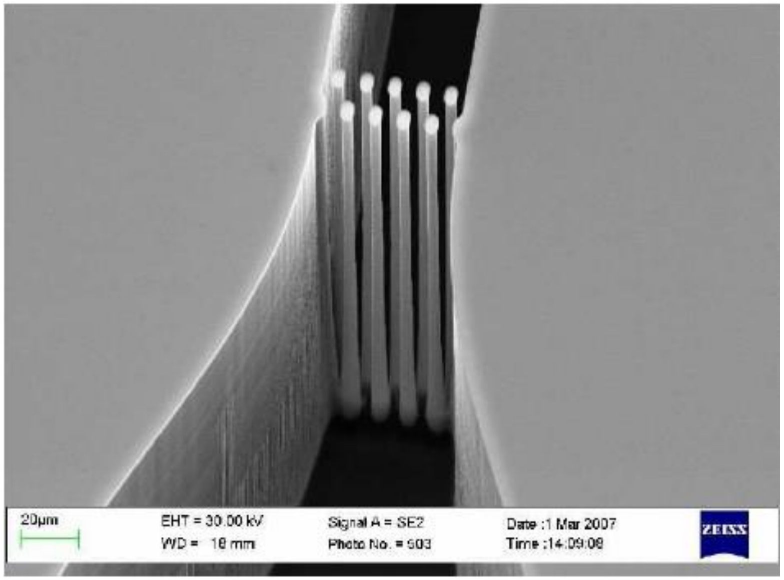
Channels

- vertical/round/slanted
- smooth/polished/textured/porous
- surface charging/electric double layer
- surface-volume ratio

Fluidics

- wetting/hydrophilic/hydrophobic
- self-filling/capillary forces
- flow dynamics/Reynolds number
- size effects/diffusion
- thermal effects/Fourier number

Lithographically defined sieve

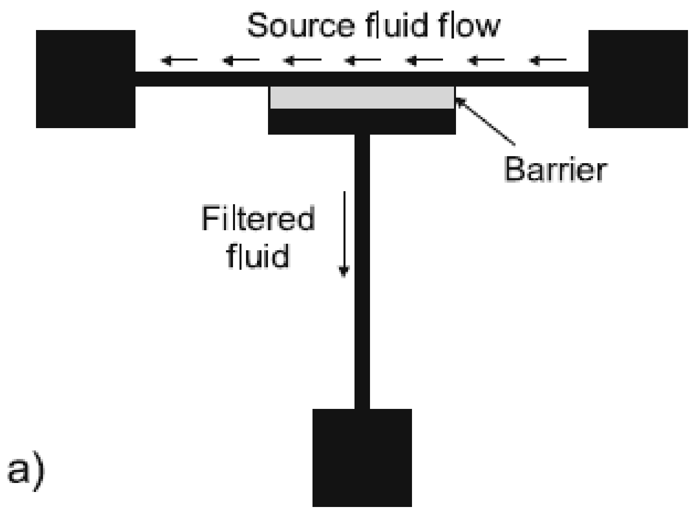


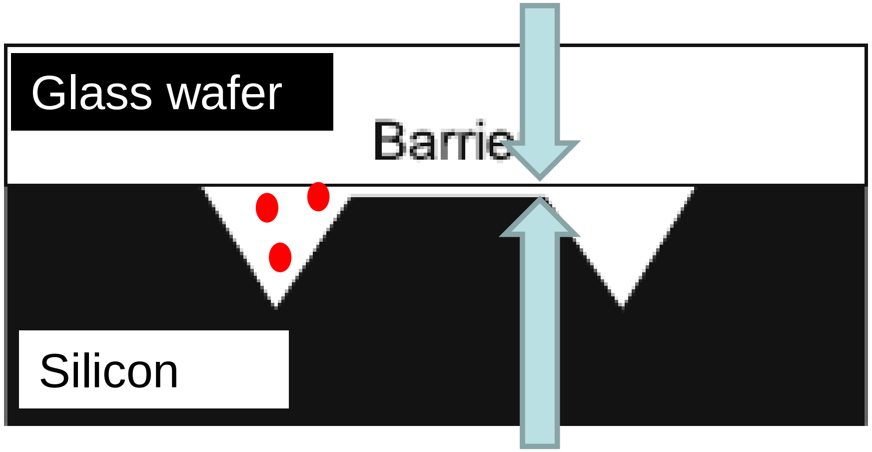
Sieve fabricated by lithography and plasma etching.

Sieve pass size determined by lithography capability.

1 µm pass size very expensive.

Bonded sieve





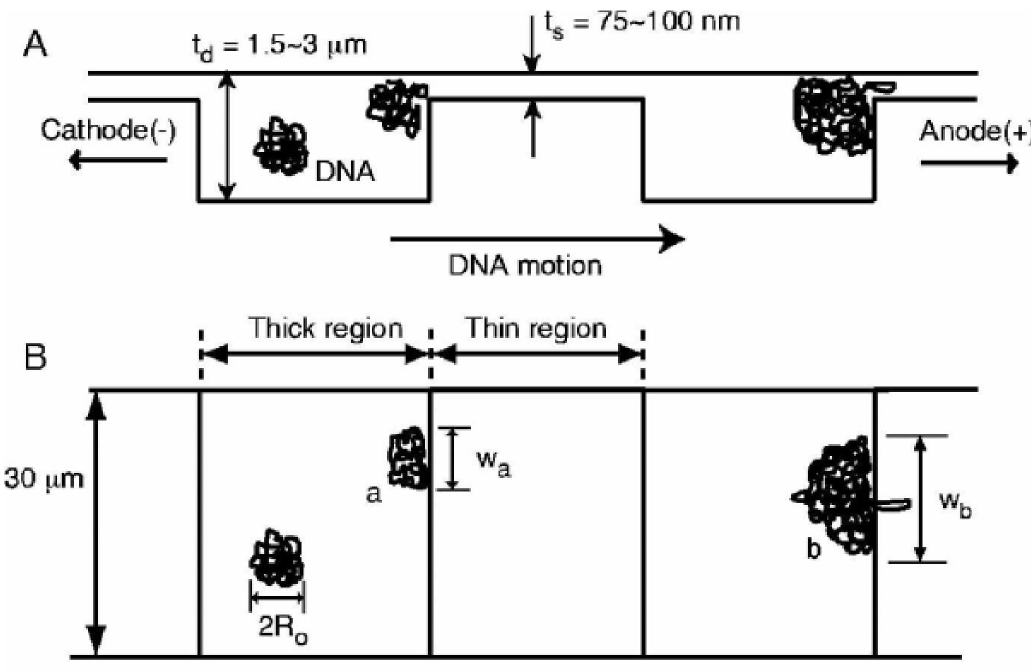
1 µm pass size for a bonded sieve is piece of cake.
100 nm is easy, too.

Sieve barrier height is determined by etching, not lithography

J.P. Brody, T.D. Osborn, F.K. Foster, P. Yager, Sens. Actuators A 54 (1996) 704–708.

Nanofluidics: molecular size ≈ channel size

Gap size 75-100 nm easy by etching + bonding.



Side view

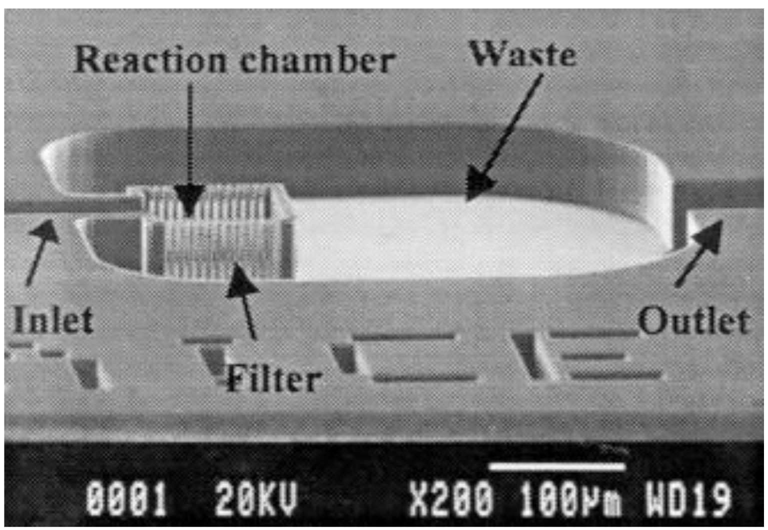
Top view

J. Han, H.G. Craighead, Science 288 (2000) 1026.

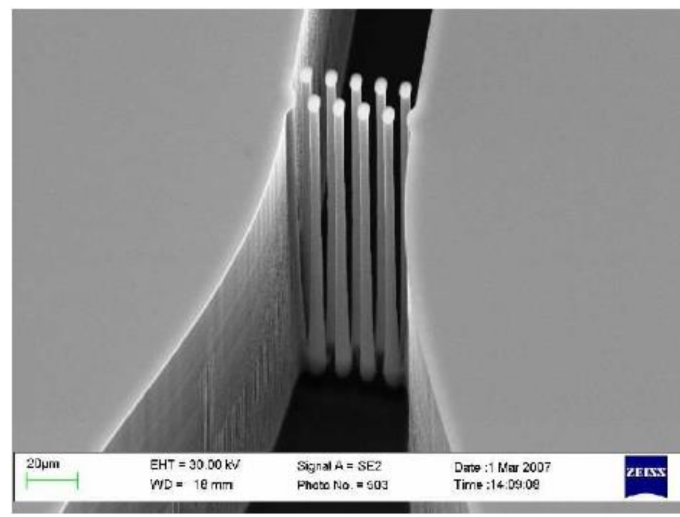
Bonding area and tightness

Bonding large areas is easier than bonding small areas.

Both must be bonded for leak-tightness.

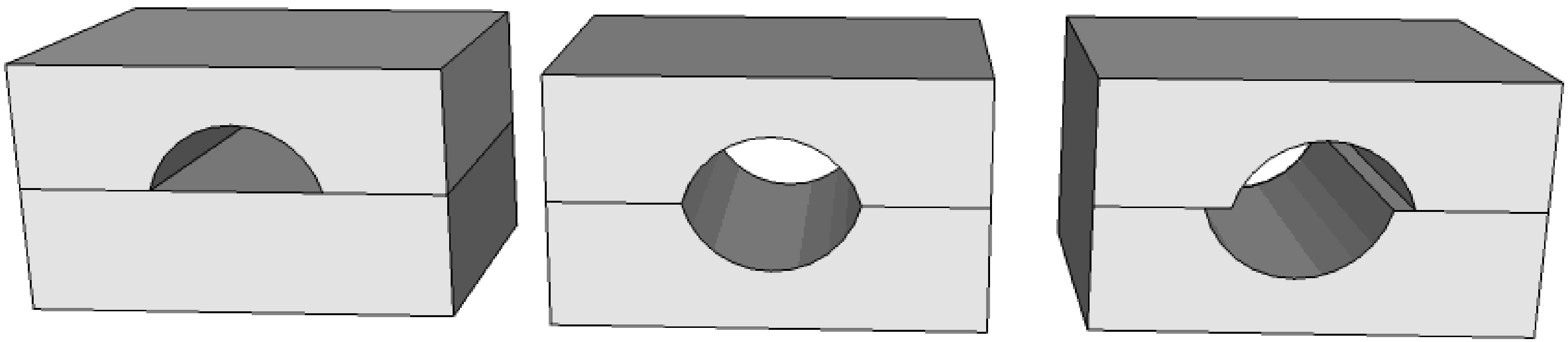


Andersson et al: Electrophoresis 2001, p. 249



Kai Kolari, VTT

Bond alignment



None

Simple equipment enough

Perfect

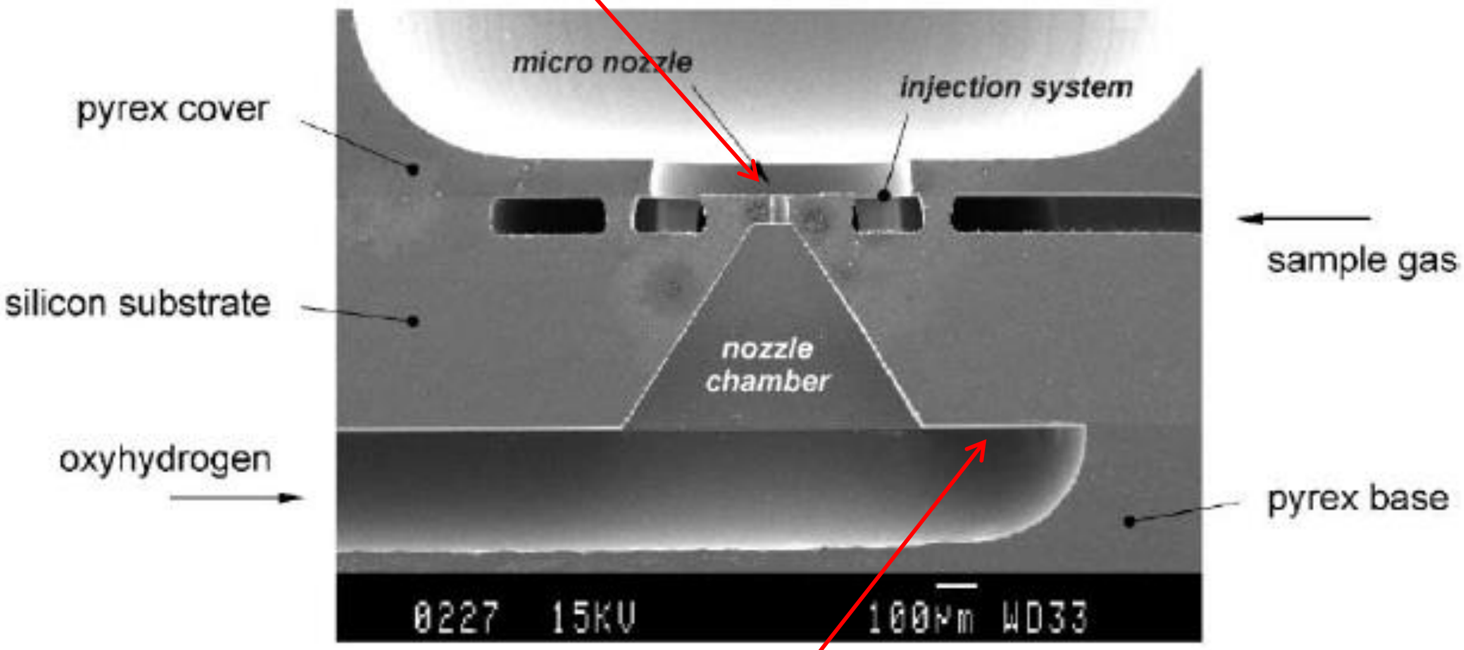
Requires advanced tools

Misaligned

Some devices more sensitive to misalignment than others.

Bond alignment (2)

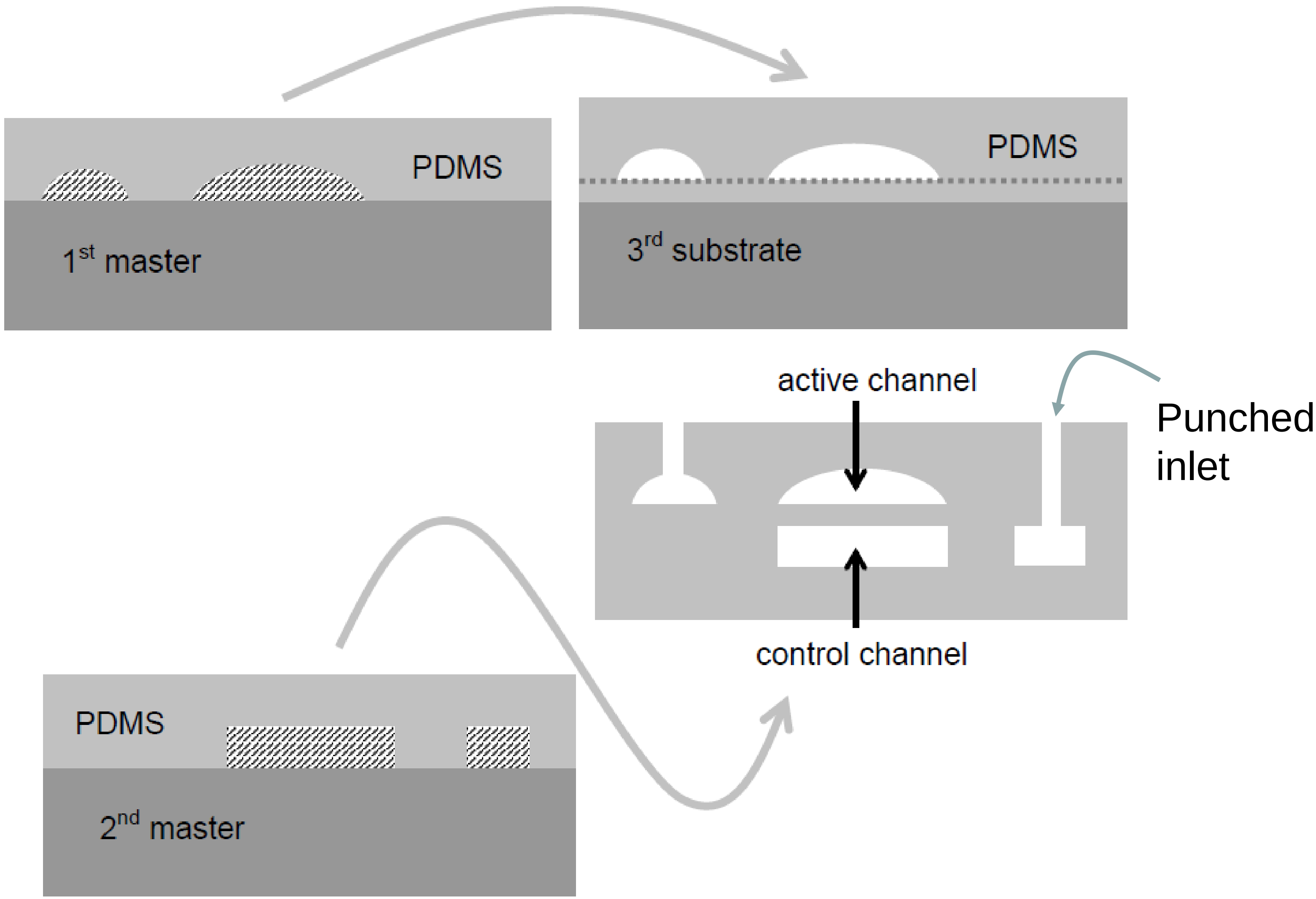
Critical alignment, nozzle in middle of injection system



Non-critical alignment, large volume gas reservoir

Zimmermann et al: Sensors and Actuators B 83 (2002) 285–289

Multiple PDMS layers bonded



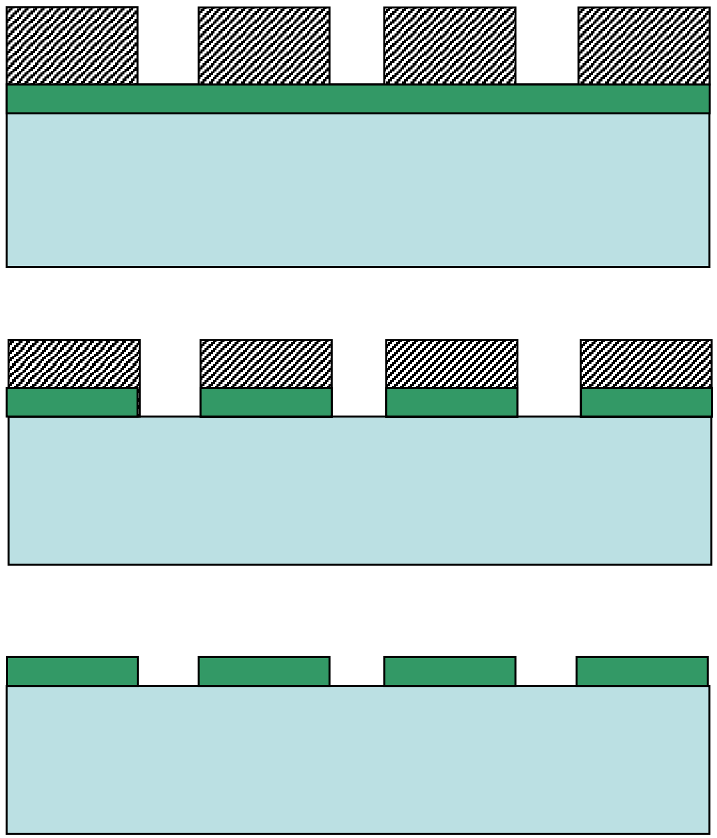
Thin films

Thin films are layers 1-1000 nm thick

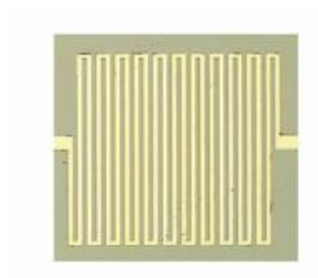
They serve several functions:

-heater electrodes (W, Pt, TiN, Al, ...)
-temperature sensors (Pt)
-catalysts (Pt, Pd, ...)
-mirrors (many metals; $\lambda/4$ dielectric stacks))
-electrodes for electrical sensing (Pt, Pd, Au, ...)
-electrical isolation (SiO_2, Si_3N_4)
-optical coatings (filters, windows, ...)
-antisticking coatings (Teflon)
-protective layers (SiO_2, Si_3N_4, Cr, Al, etch masks)

Thin film heater processing



1. Metal film evaporation
2. Photoresist spinning & baking
3. Lithography with resistor mask

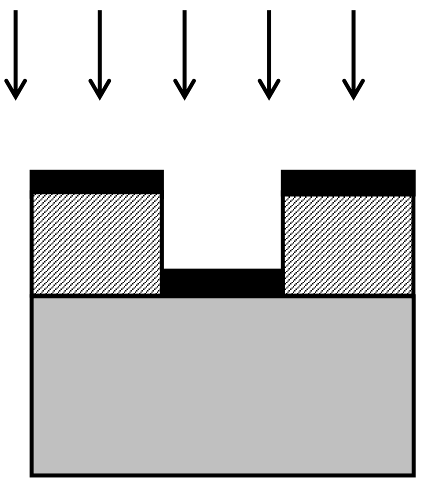


4. Resist image development
5. Metal etching (in acid)
6. Photoresist stripping

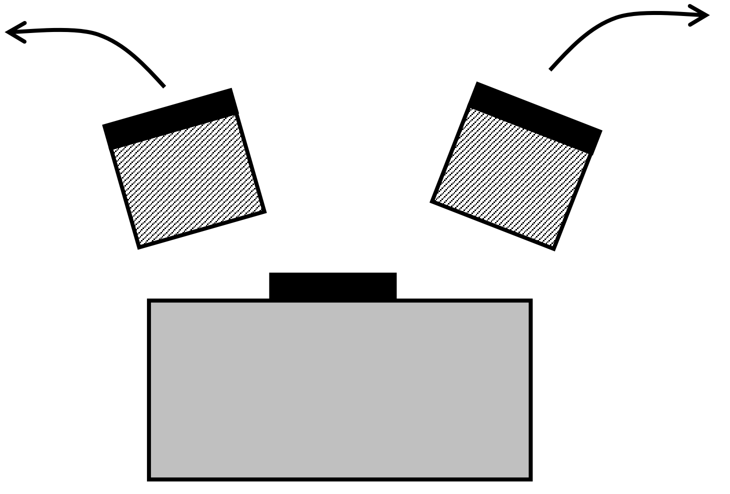
Can be done on any wafer !

Glass wafers, polymer, ...

Alternative metal patterning: Lift-off



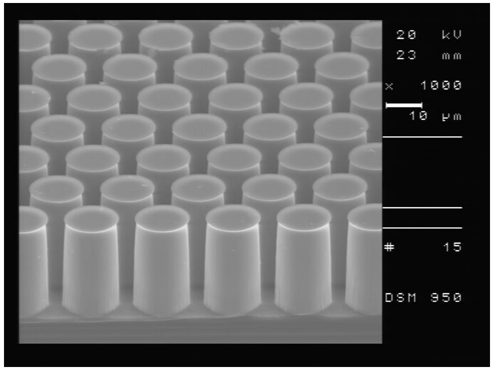
Litho + deposition



Resist lift-off

Silicon: pros and cons

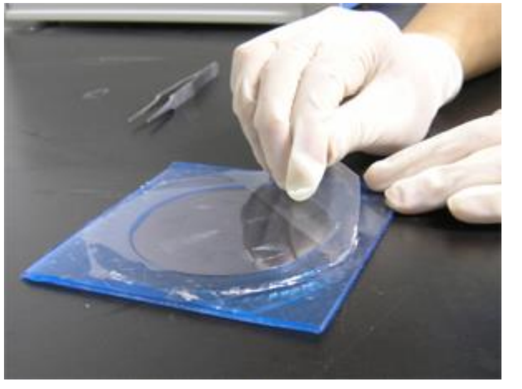
- micromachining technologies exist
- surface modification technologies exist
- smooth and flat surfaces
- bonding to glass (400^{o}C) and to silicon (1000^{o}C)
- adhesive bonding to polymers
- integration of electrical, optical and thermal functions
- not transparent
- semiconducting
- 10-20 €/wafer (100 mm diameter)



PDMS: pros and cons

Easy processing by replica molding

Easily bondable, self adhesive



Optically transparent, electrical insulator

Biocompatible

Hydrophobic

Oxygen and water vapor permeable

Absorbs solvents and salts

Max temperature <100^{o}C (H_2O permeable…)

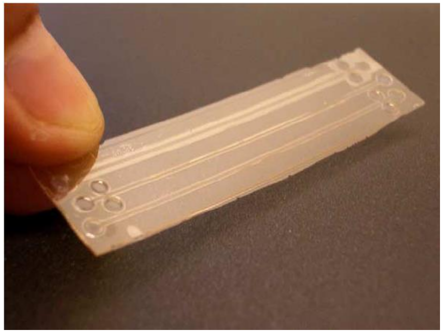
Huge thermal expansion 300 ppm/^{o}C

100-500 €/kg (1-5 €/100 mm diameter, 5 mm thick piece)

SU-8: pros & cons

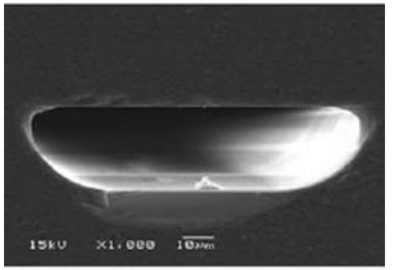
- Easy to process; feature size and thickness range very large
- Thermally and chemically stable (for a polymer)
- Easy to bond to itself
- Not fully transparent & autofluorescence
- High stresses often encountered

➔ non-planarity



Glass: pros & cons

- Surface chemistry well known from lab glassware
- Transparent in VIS-IR
- If you need UV-transparency, use silica/quartz: pure SiO_2



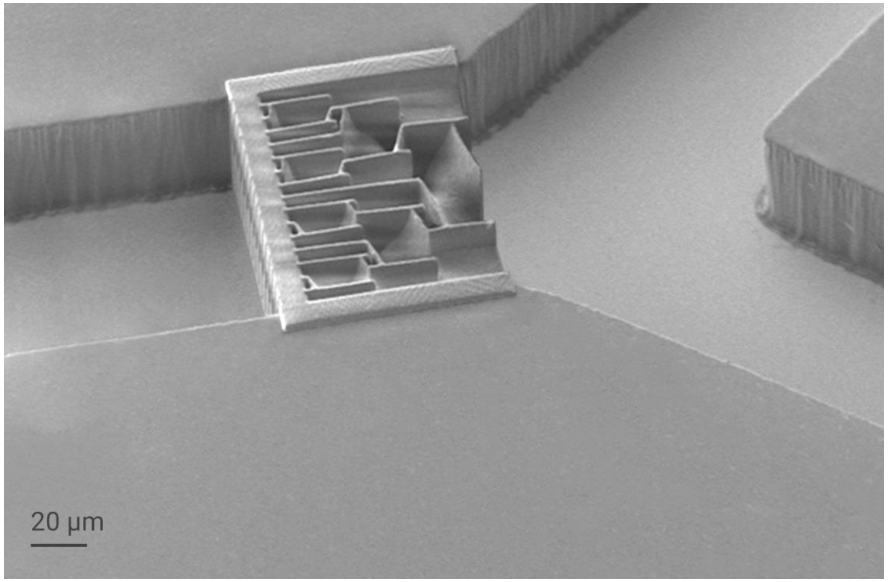
- Difficult to etch, only isotropic shapes
- Anodic bonding to silicon easy & strong

More exotic fabrication

Because feature sizes extremely small

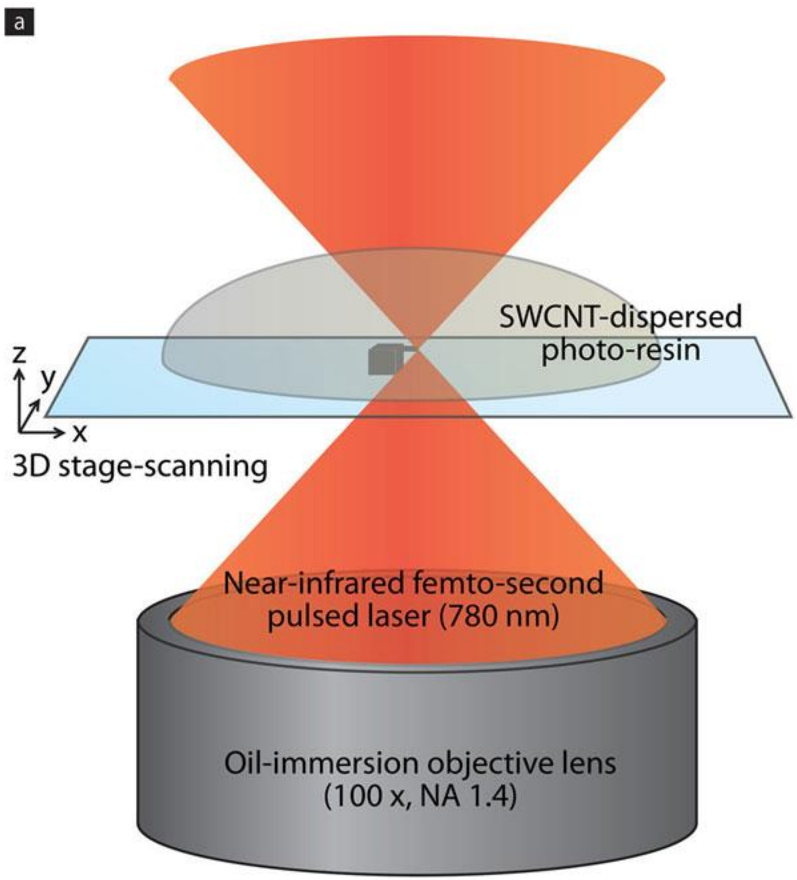
Because 3D structures

Maybe both !



Nanoscribe GmbH

2-photon photopolymerization (2PPP)



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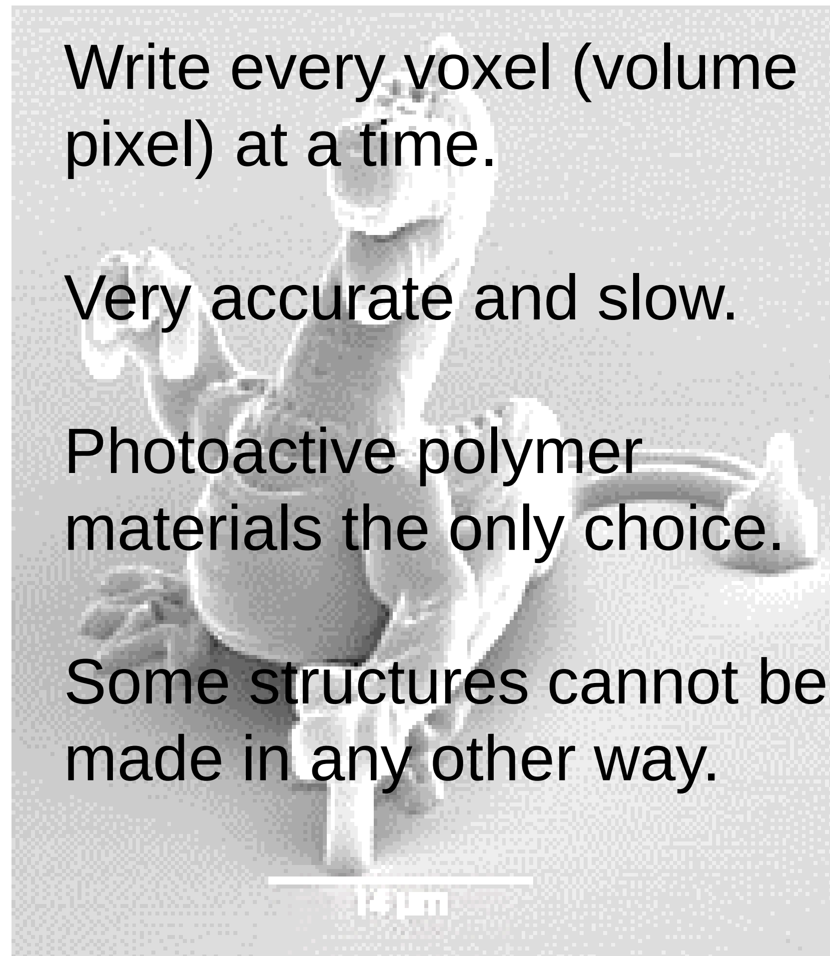


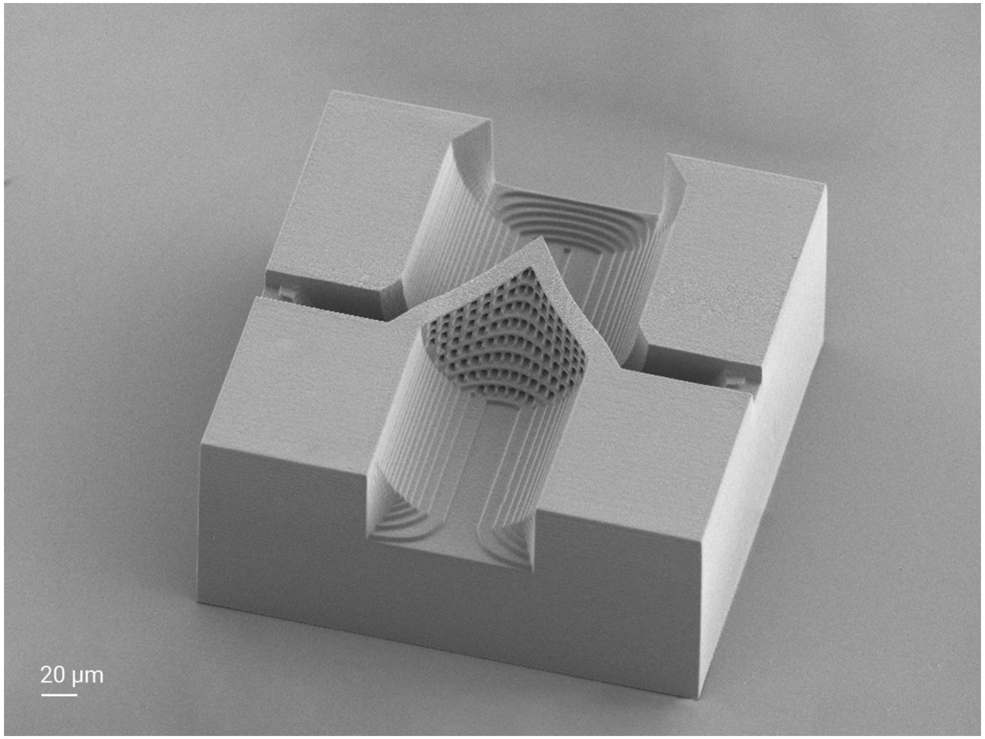
Write every voxel (volume pixel) at a time.

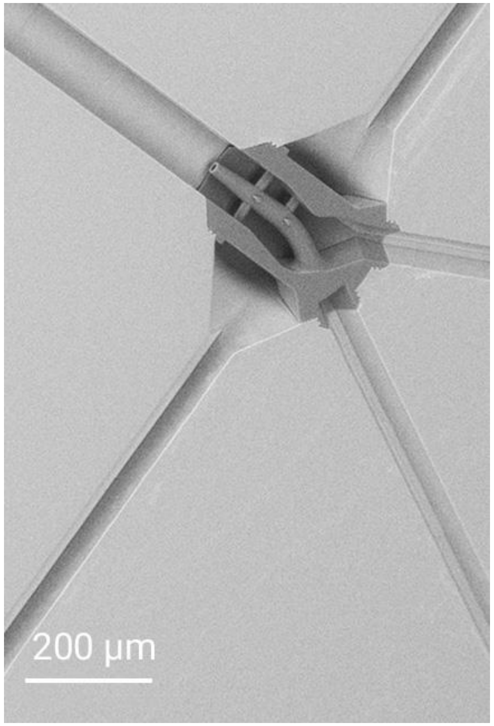
Very accurate and slow.

Photoactive polymer materials the only choice.

Some structures cannot be made in any other way.

Microfluidic filters by 2PPP





Sizes down to 100-200 nm.
Shape freedom.

Slow, and only suitable for experimentation.

Nanoscribe GmbH