The Abdus Salam
International Centre for Theoretical Physics

United Nations
Educational, Scientific
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International Atomic
Energy Agency

SMR.1670 - 12

INTRODUCTION TO MICROFLUIDICS

8 - 26 August 2005

Microfabrication for Microfluidics

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University of Twente, Enschede, The Netherlands

Microfabrication for microfluidics

Summer School in Microfluidics
ICTP, Trieste, Italy

Han Gardeniers
University of Twente

The basic questions

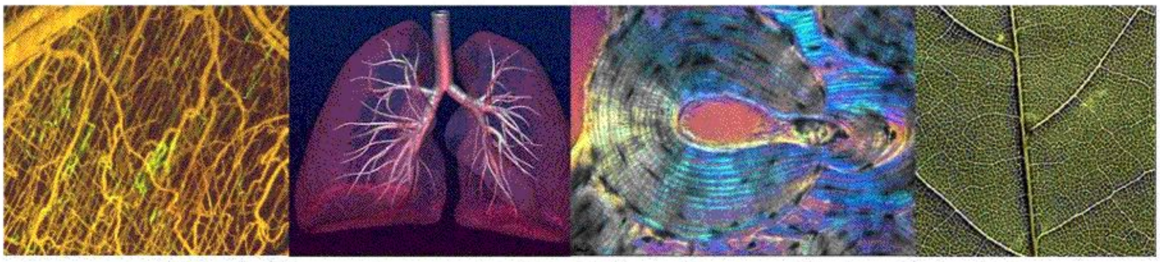
What are the materials of interest to us microfluidicists?

How do we bring them into shape?

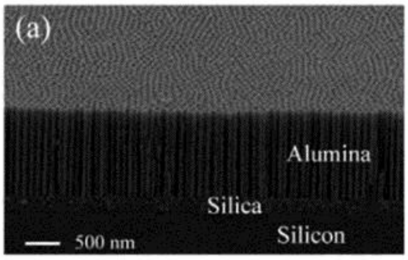
How do we put them into place?

When used in our microfluidic application, how reliable are the materials ?

The micro/nanofluidic world

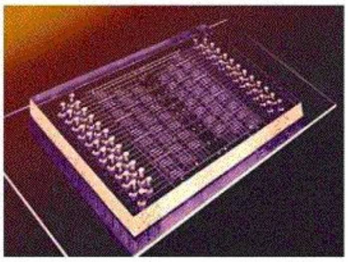


Biological flow systems (animal blood / lung / bone capillaries, plant veins)



Porous solids

Glass / PEEK / stainless-steel tubes / capillaries



Machined microchannels

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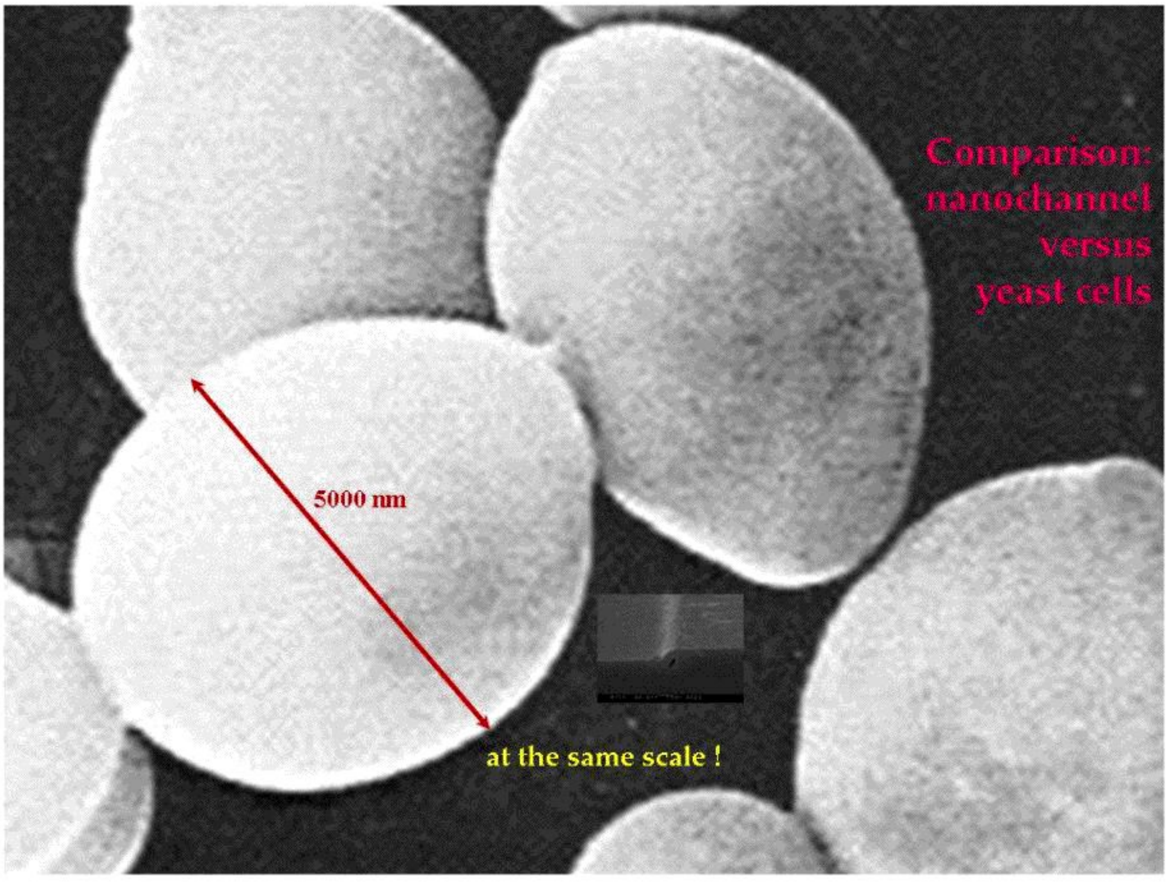
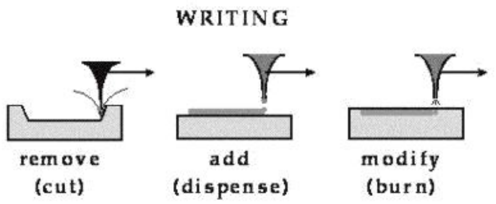
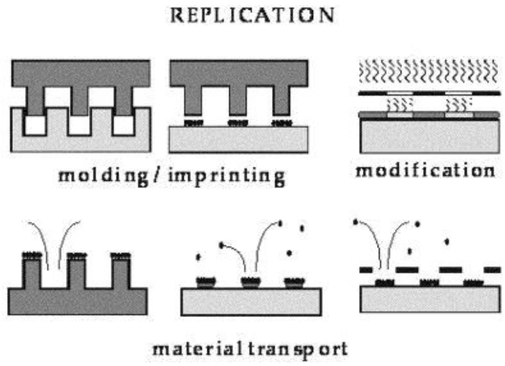
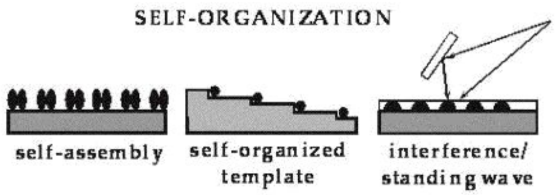


How to make a micro(nano)structure?



dig - glue -fry





photons - atoms - ions - molecules
particles - gels - melts

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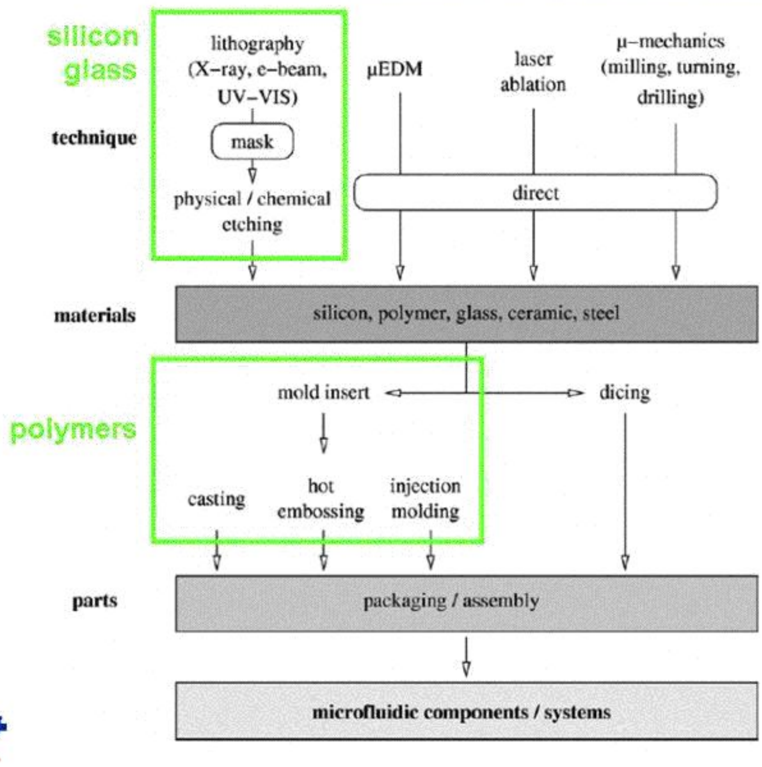
Microfabrication overview



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The philosophy of microfabrication

Batch fabrication principles in IC technology

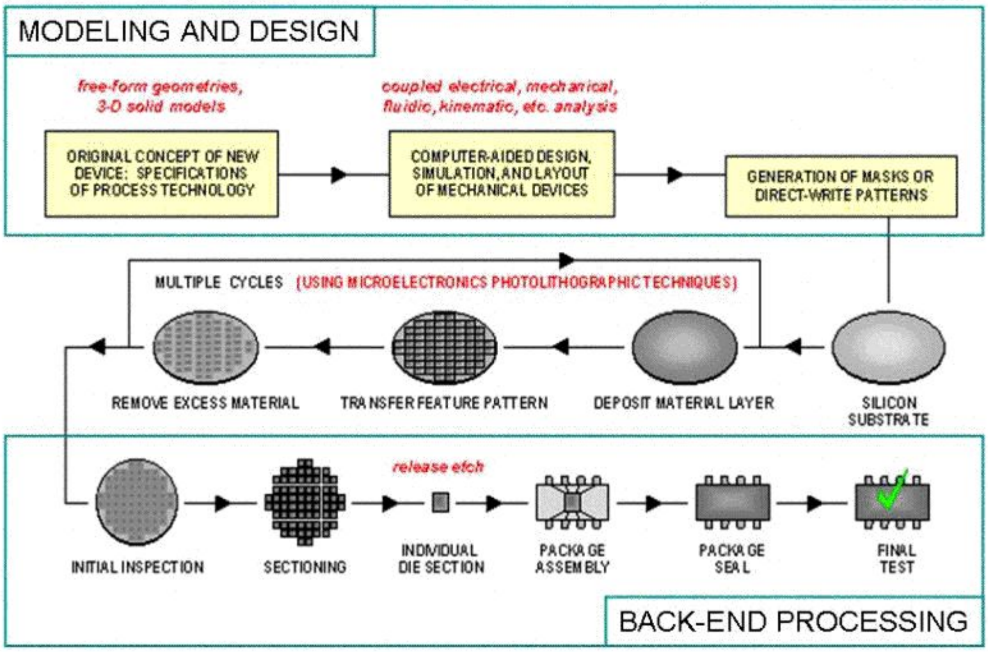
1. Computer aided design

2. Transfer of pattern to substrate by means of optical imaging (photolithography)

3. Batch fabrication: simultaneous treatment, therewith minimizing variation in process quality; costs of a process step are distributed over thousands of components

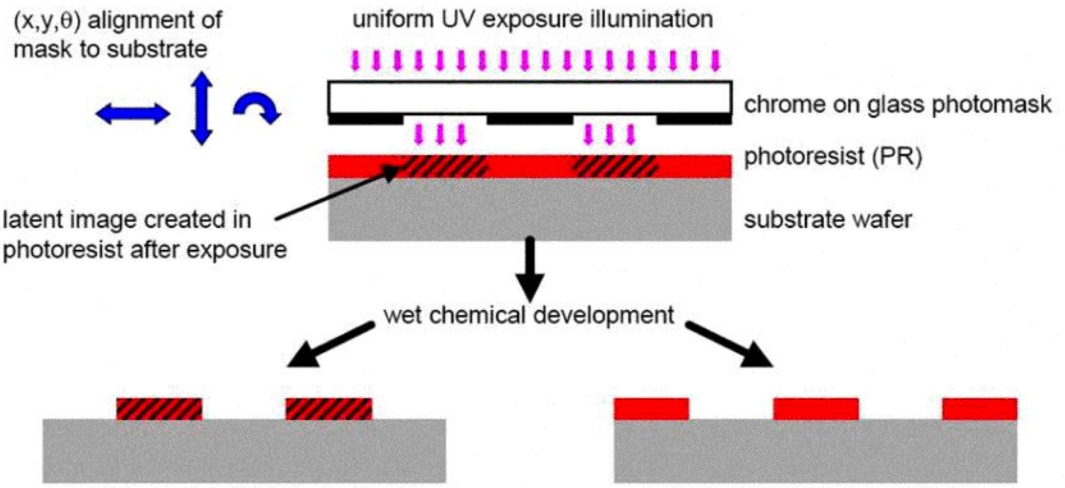
4. Linking a large number of identical components with high packing density to get to an intelligent system of high quality

Batch microfabrication process



copied from: MEMS Tutorial by M.A. Michalicek

Overview of Align/Expose/Develop Steps

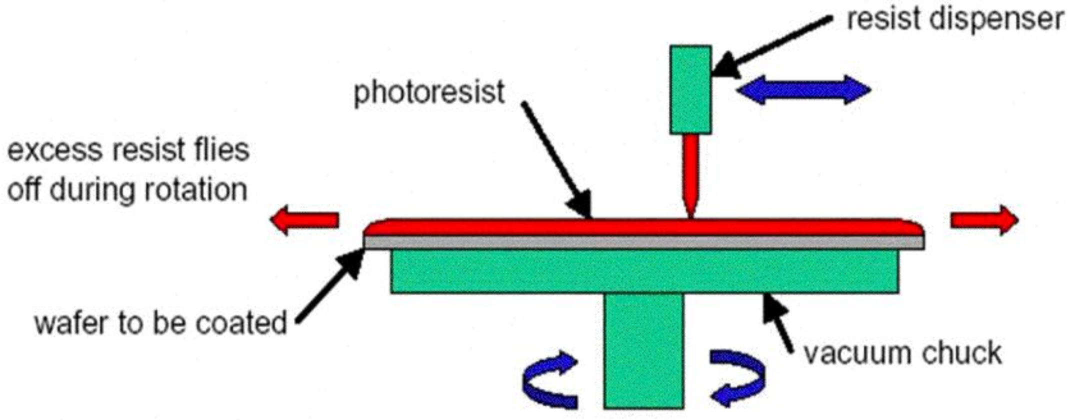


NEGATIVE PHOTORESIST
Photoresist is photopolymerized where exposed and rendered insoluble to the developer solution.

POSITIVE PHOTORESIST
Exposure decomposes a development inhibitor and developer solution only dissolves photoresist in the exposed areas.

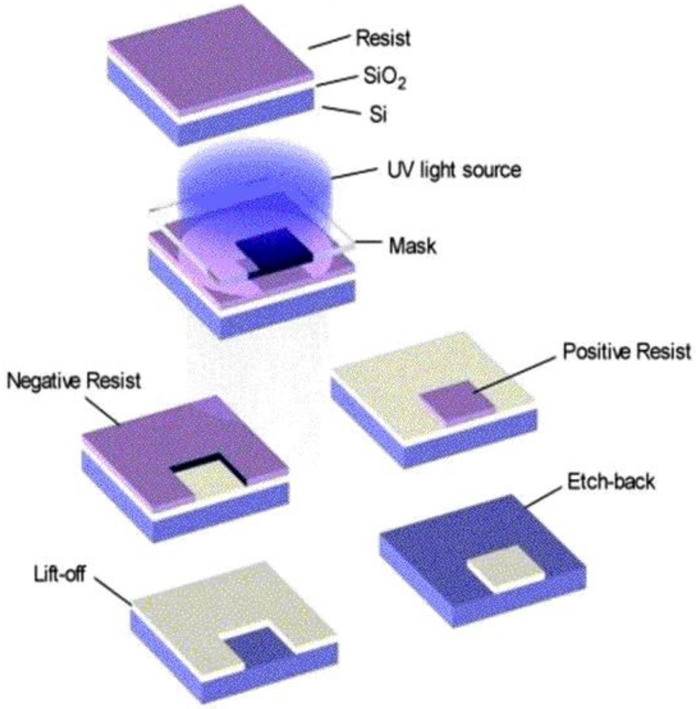
R. B. Darling / EE-527

Photoresist Spin Coating



http://www.engr.washington.edu/~cam/PROCESSES/NEWtutorial.html

Photolithography: pattern transfer



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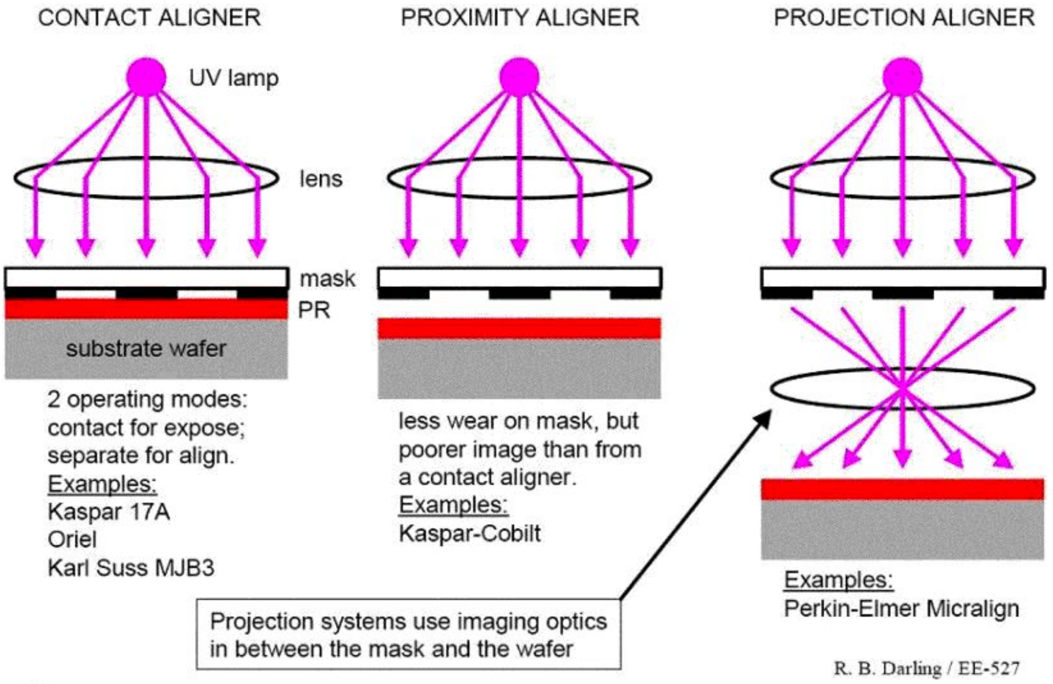
Alignment and Exposure Hardware



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Mask aligners

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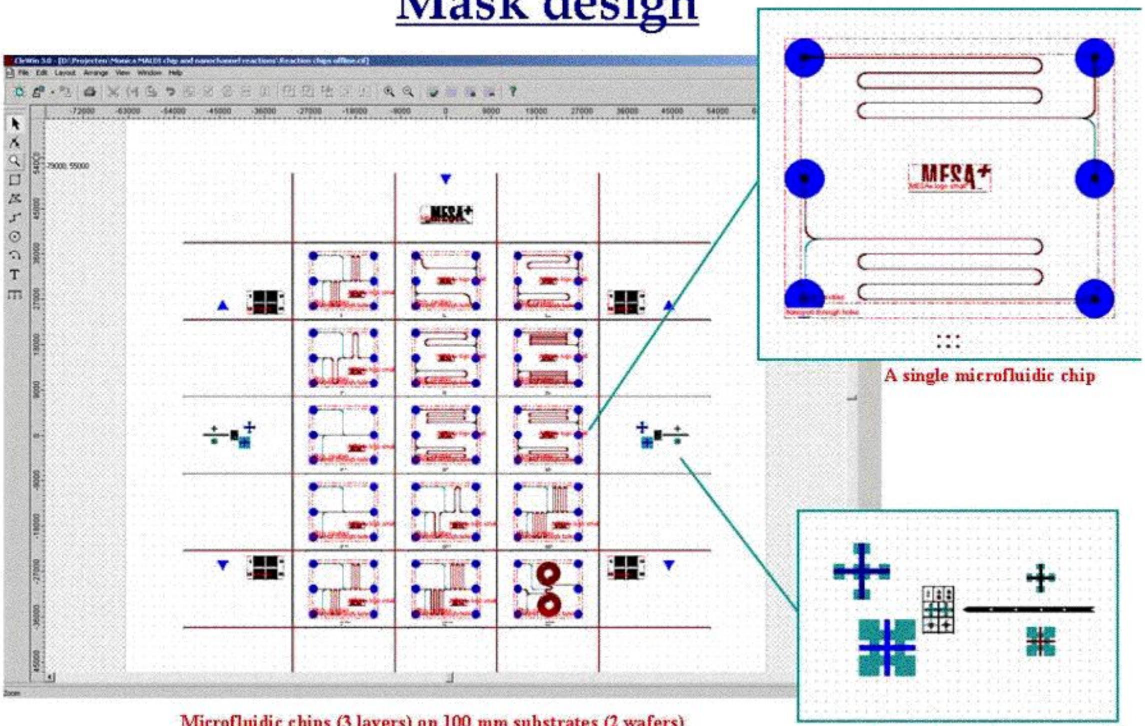
Mask design



Microfluidic chips (3 layers) on 100 mm substrates (2 wafers)

☞link to CLEWin

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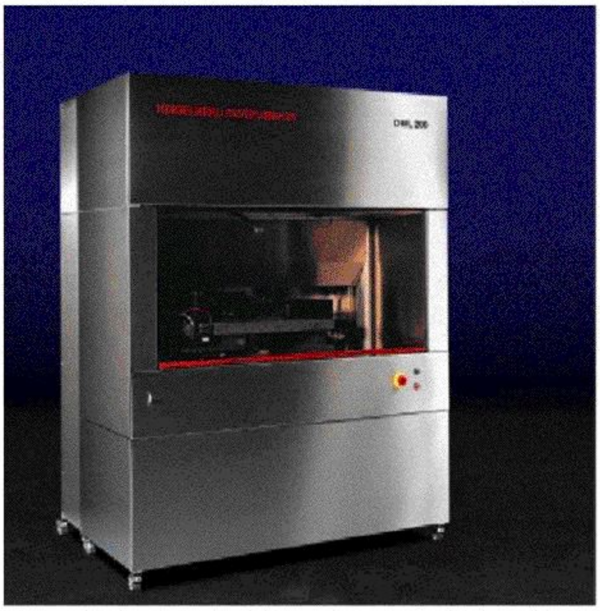
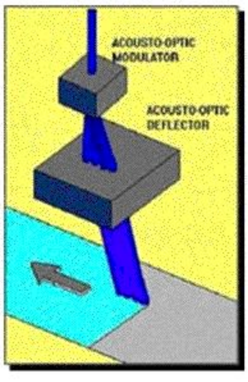
Mask fabrication e.g. by laser beam writing





High Accuracy Photomask and Direct Write Lithography Systems

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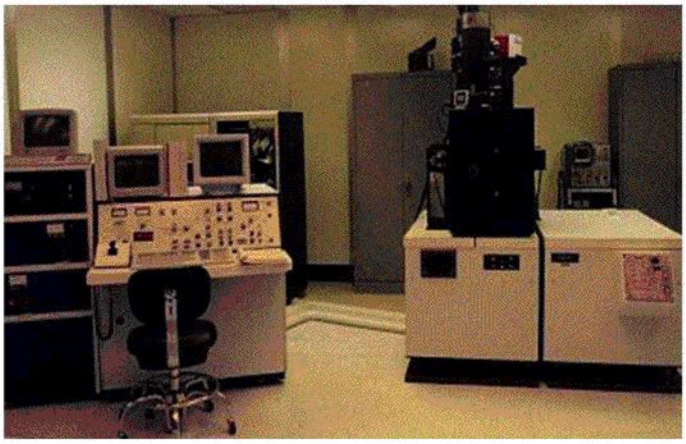
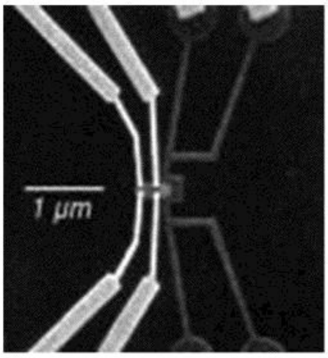
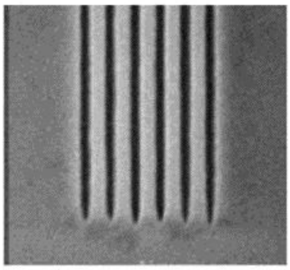
Nanostructures: E-beam lithography







33nm Trenches

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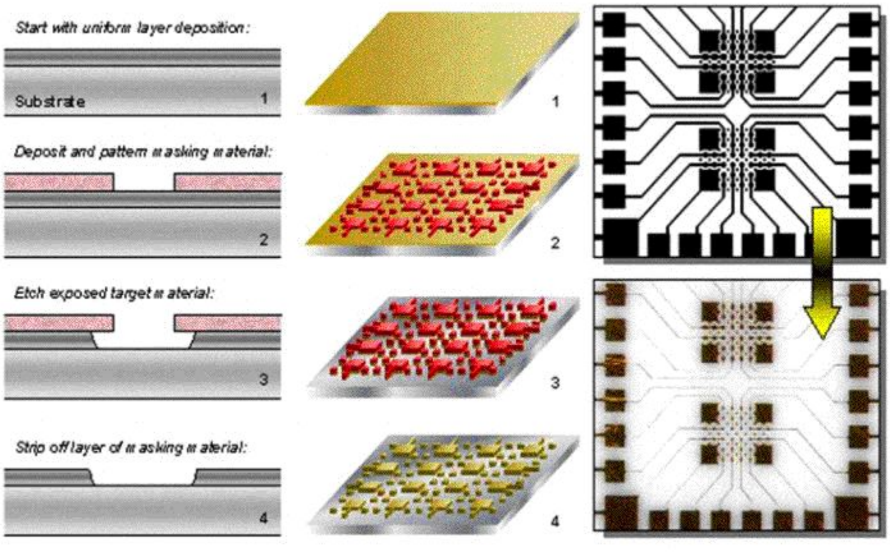
Etching features in thin films



copied from: MEMS Tutorial by M.A. Michalicek

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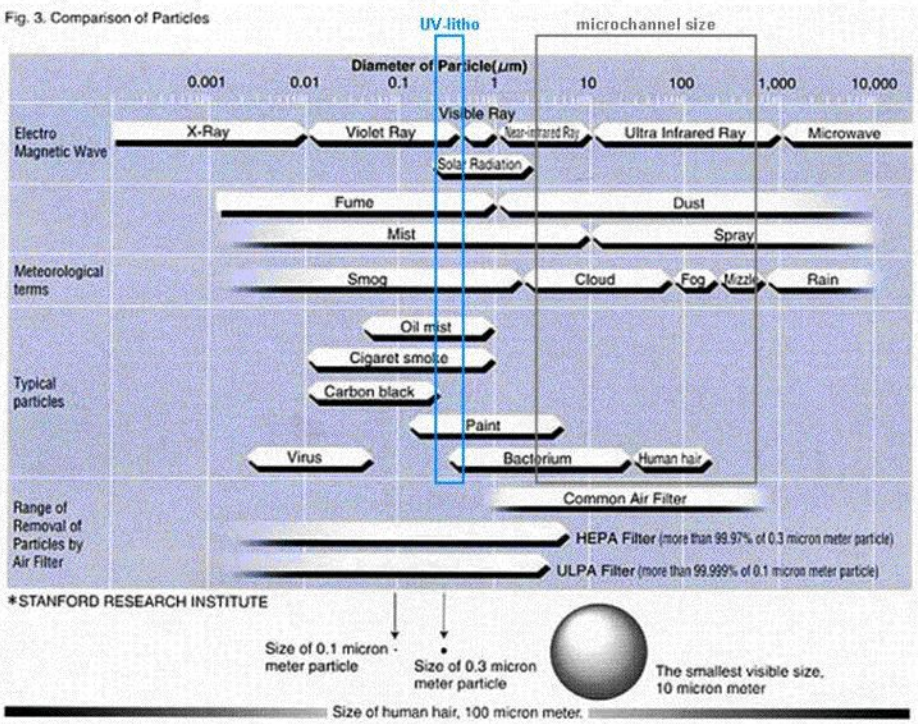
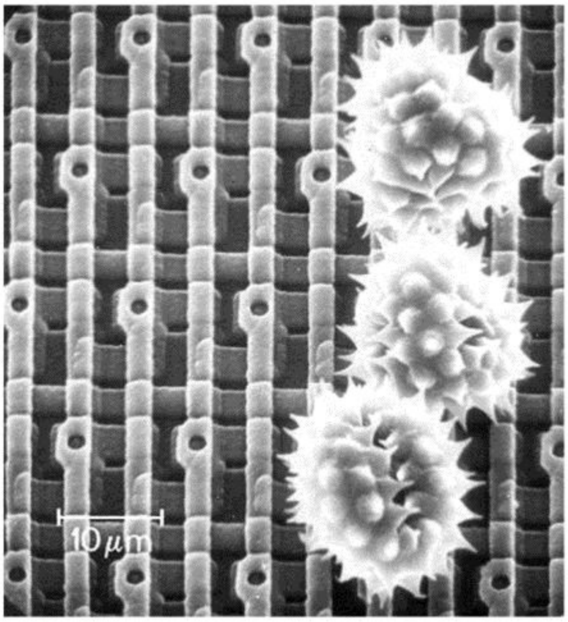
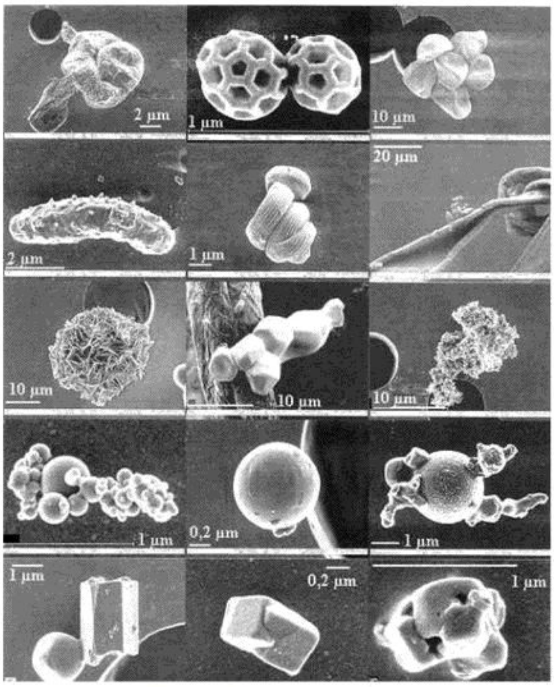
Intermezzo: where to do your microfabrication?

Microfabrication expert desperately looking for a dust-free location





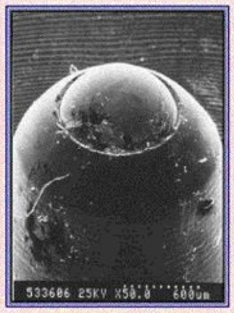
Pollen on microelectronic circuit



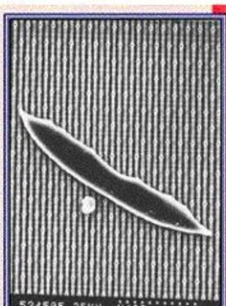
The air we breath

Humans as a source of particles

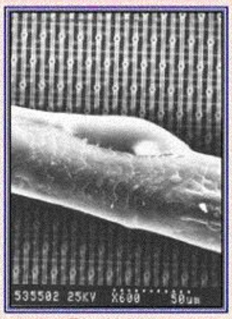
Dirt on a necklace



Tip of a ball point writer

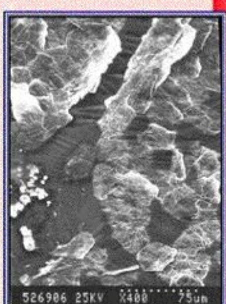


A piece of nail polish on a 256 k DRAM chip



Hair with hair spray

Inside of finger ring



Flakes of skin

Dirty people

Table 3. Dust Generation from Human Body and Quantity*

(1) Particles (over 0.3 μm)

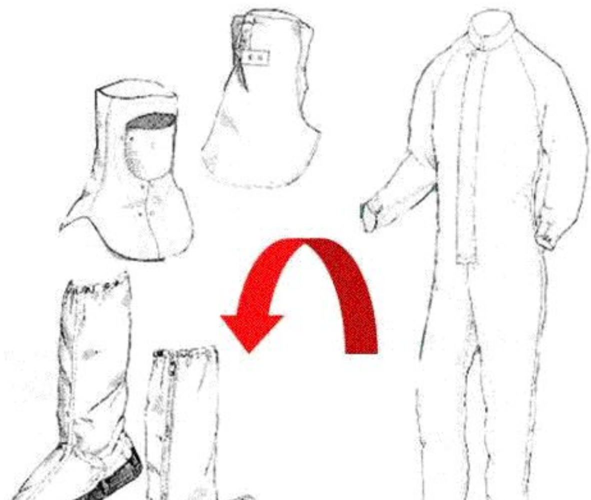
Kind of Movement	Nos. of Particles/ per minute ($\geq$0.3μm)
Sitting or standing (on movement)	100,000
Sitting (slightly moving head, arm and hands)	500,000
Sitting (slightly moving body and foot)	1,000,000
Standing up from a sitting position	2,500,000
Walking about 1 meter/per second	5,000,000
Walking about 1.5 meters/per second	7,500,000
Walking quickly	10,000,000
Climbing stairs	10,000,000
Gymnastic exercise	15,000,000 – 30,000,000

(2) Bacteria

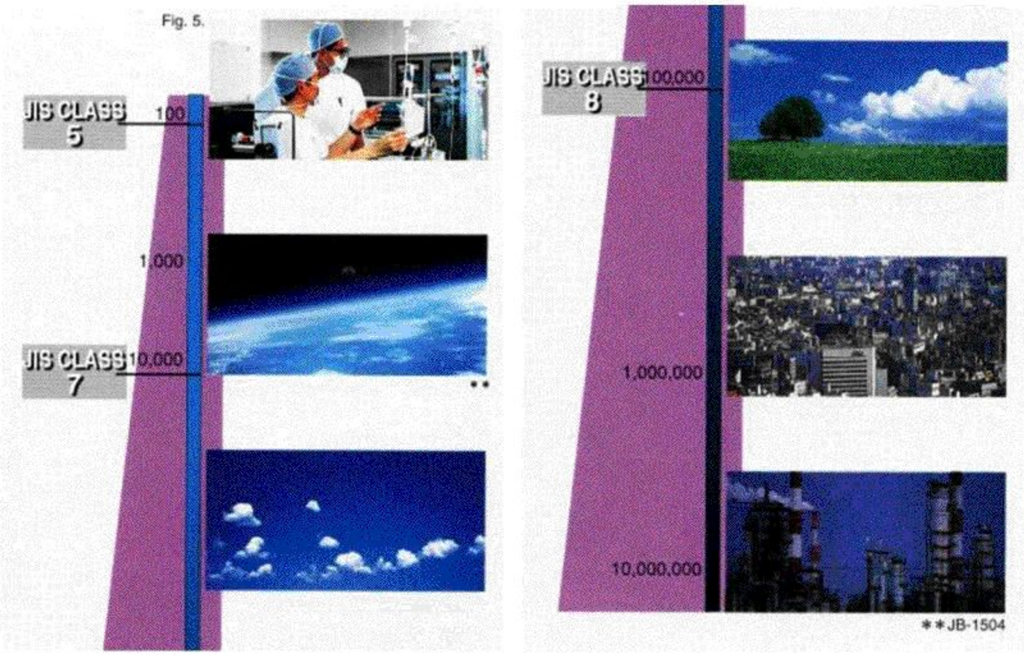
Kind of Movement	Nos. of Bacteria/ per minute
In operation	
Under strict bacterial control	5,000
On average	10,000
Without bacterial control	50,000
In Laboratory:	
Heavy movement	15,000
Medium movement	8,000
Slight movement	4,000

*P.R. AUSTIN : DESIGN & Operation of Clean Room

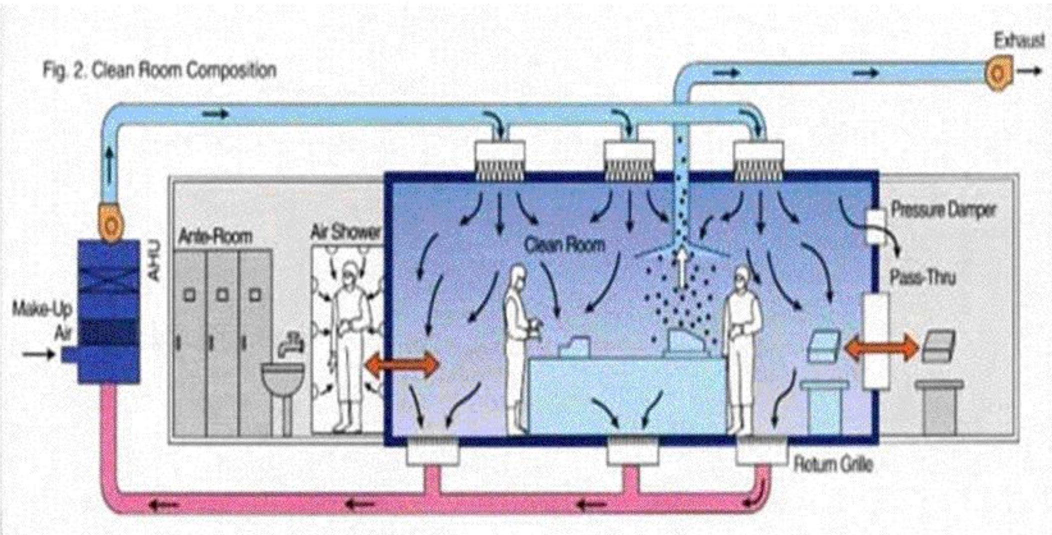
The people that visit a clean room...



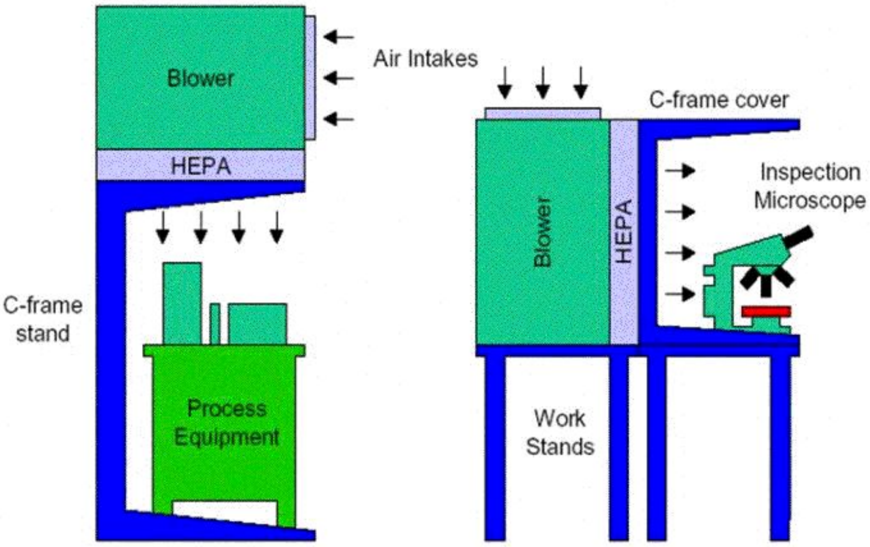
...wear fancy stuff



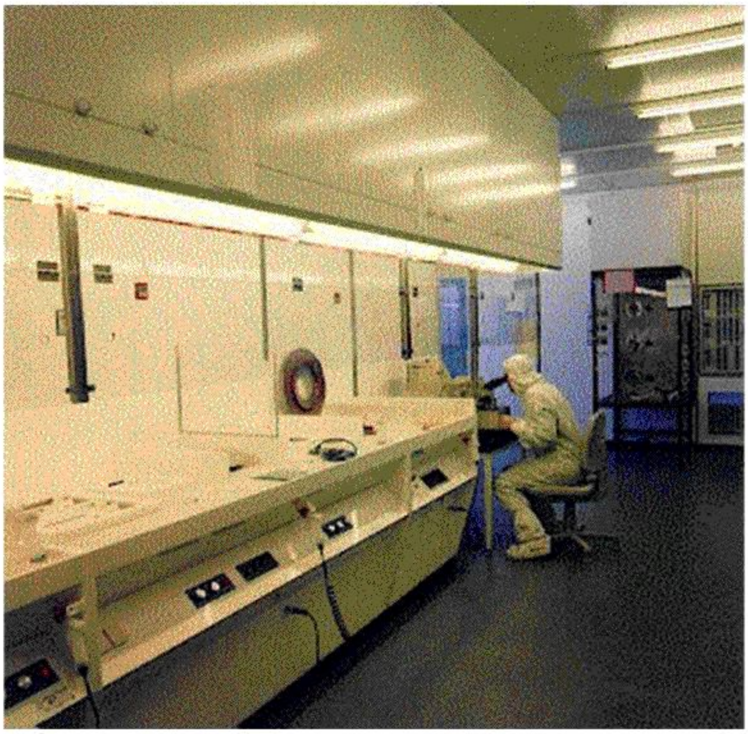
Principle of a clean room



Local clean air generation



http://www.engr.washington.edu/~cam/PROCESSES/NEWtutorial.html



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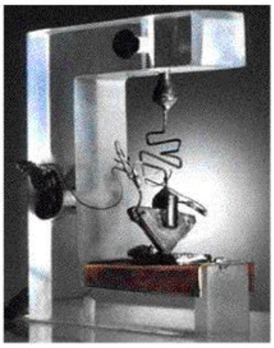
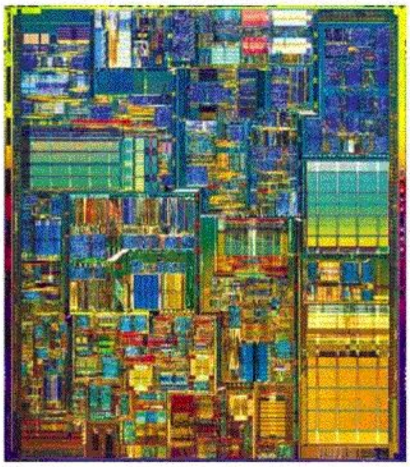
The material silicon

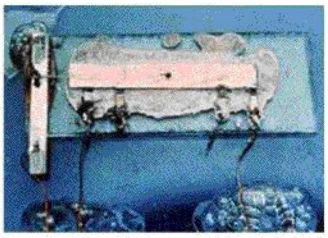
Silicon as an electronic material

A replica of the point-contact transistor created by John Bardeen and Walter Brattain, under the supervision of William Shockley in 1947 (P.S. this was not Si but Ge)

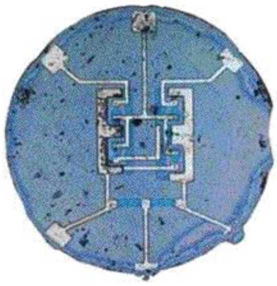


Intel's Pentium 4 contains tens of millions of transistors



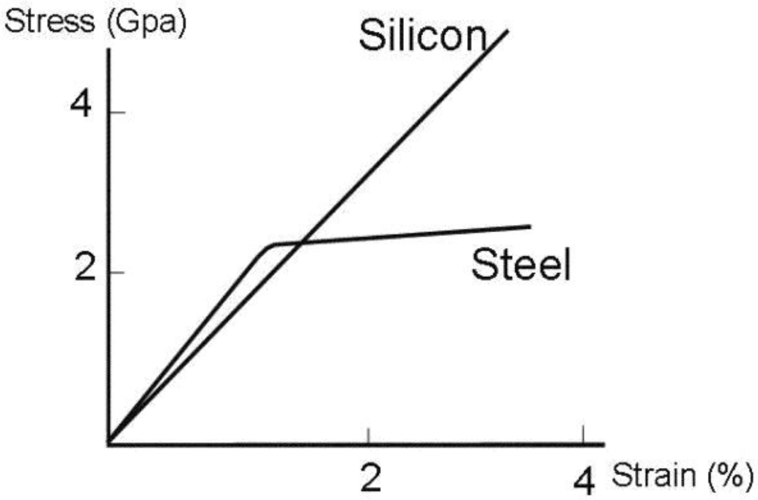


Jack Kilby of Texas Instruments made this, the first integrated circuit, in 1958



This device, developed by Robert Noyce in the late 1950s, was the first commercially available integrated circuit

Silicon as a mechanical material



stress-strain curve for silicon compared to a typical metal

Silicon as a Mechanical Material

KURT E. PETERSEN, MEMBER, IEEE

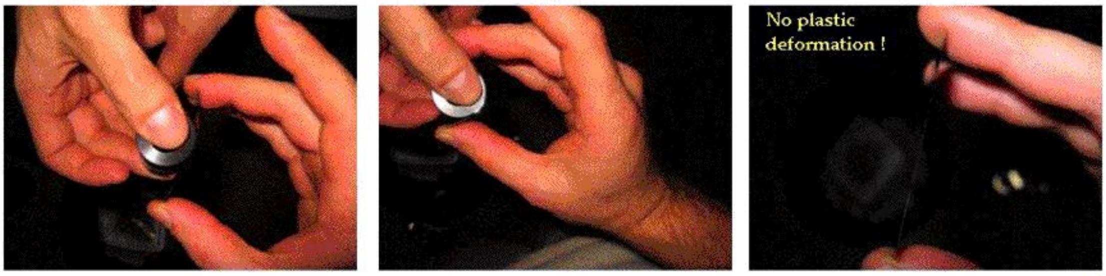
Abstract—Single-crystal silicon is being increasingly employed in a variety of new commercial products not because of its well-established electronic properties, but rather because of its excellent mechanical properties. In addition, recent trends in the engineering literature indicate a growing interest in the use of silicon as a mechanical material with the ultimate goal of developing a broad range of inexpensive, batch-fabricated, high-performance sensors and transducers which are easily interfaced with the rapidly proliferating microprocessor. This review describes the advantages of employing silicon as a mechanical material, the relevant mechanical characteristics of silicon, and the processing techniques which are specific to micromechanical structures. Finally, the potentials of this new technology are illustrated by numerous detailed examples from the literature. It is clear that silicon will continue to be aggressively exploited in a wide variety of mechanical applications complementary to its traditional role as an electronic material. Furthermore, these multidisciplinary uses of silicon will significantly alter the way we think about all types of miniature mechanical devices and components.

I. INTRODUCTION

miniaturized mechanical devices and components must be integrated or interfaced with electronics such as the examples given above.

The continuing development of silicon micromechanical applications is only one aspect of the current technical drive toward miniaturization which is being pursued over a wide front in many diverse engineering disciplines. Certainly silicon microelectronics continues to be the most obvious success in the ongoing pursuit of miniaturization. Four factors have played crucial roles in this phenomenal success story: 1) the active material, silicon, is abundant, inexpensive, and can now be produced and processed controllably to unparalleled standards of purity and perfection; 2) silicon processing itself is based on very thin deposited films which are highly amenable to miniaturization; 3) definition and reproduction of the device shapes and patterns are performed using photographic techniques which have also, historically, been capable of high

K.E.Petersen, Proc. IEEE 70, 420-457 (1982)



Wrapping a 100um-thick wafer around a post with 1" diameter, from: http://www-bsac.eecs.berkeley.edu/~mattlast/research/getbent/

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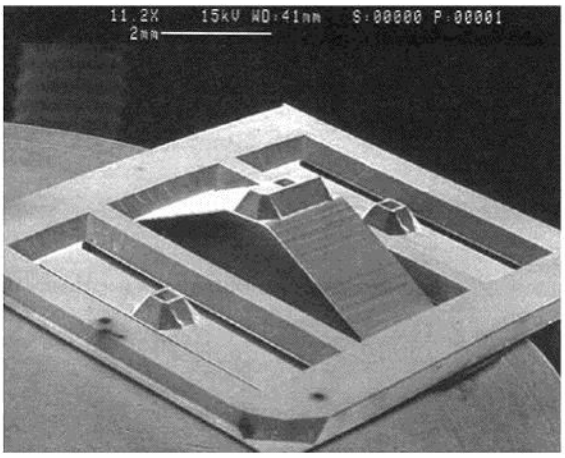
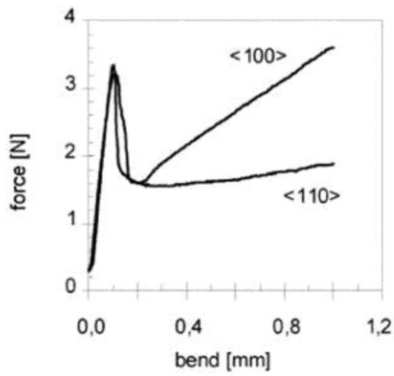
Silicon plastic deformation at high T





Typical force–bend diagram for differently orientated Si-beams at 900 °C and 0.1 mm/min bending rate

J. Frühauf et al. J. Micromech. Microeng. 9, 305–312 (1999)

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Silicon is brittle

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Properties of silicon vs. other materials

	Yield strength (GPa)	Knoop Hardness (Kg/mm^2)	Young's Modulus (GPa)	Density ($1000 Kg/m^3$)	Thermal expansion ($10^{-6}/°K$)
Diamond	53	7000	10.35	3.5	1.0
SiC	21	2480	7.0	3.2	3.3
Si_3N_4	14	3486	3.85	3.1	0.8
Si	7	850	1.9	2.3	2.33
Stainless steel	2.1	660	2.0	7.9	17.3
Al	0.17	130	0.7	2.7	25

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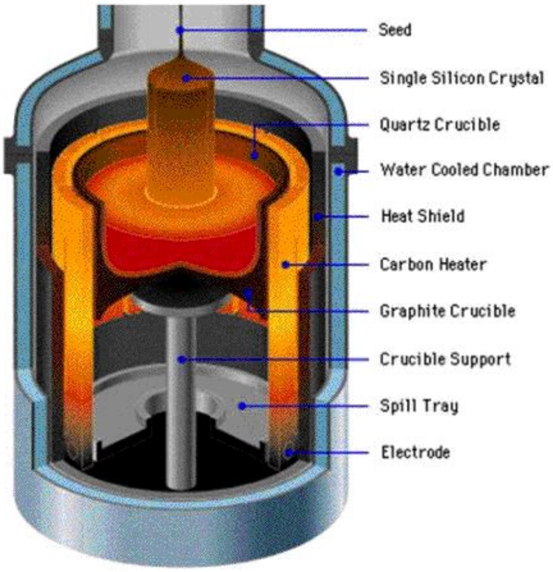
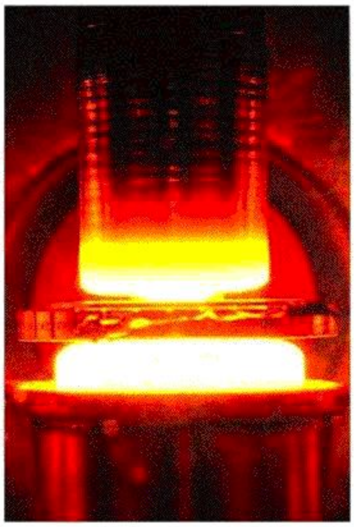
How silicon crystals are made

Czochralski



Float zone



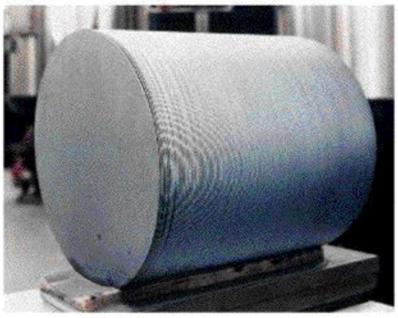
Poly and mono crystals

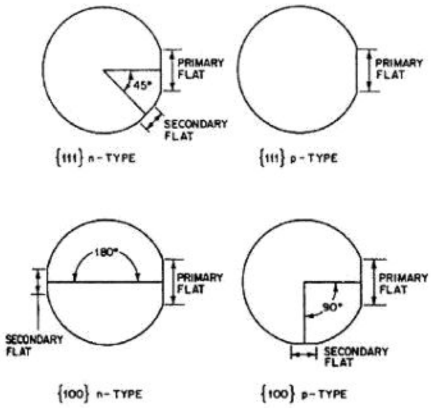
Silicon "wafers"

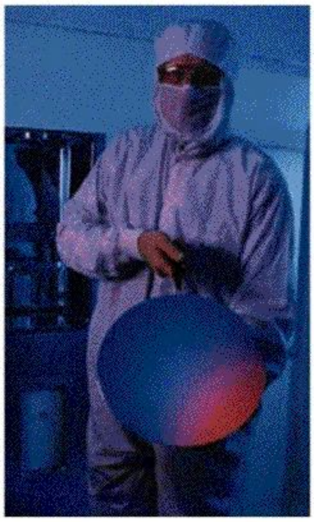
from 10 microns

thickness

to 3 mm







size up to 12"(30 cm)

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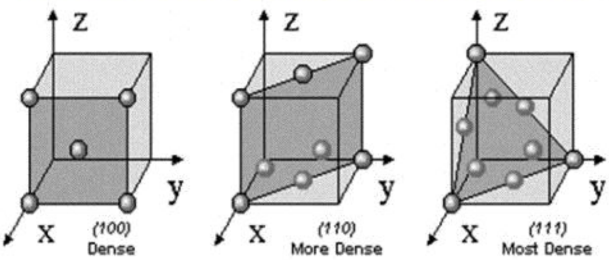
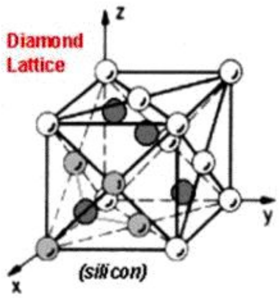
Silicon crystal planes

Miller indices identify crystal planes from the unit cell:





☞MDL-link

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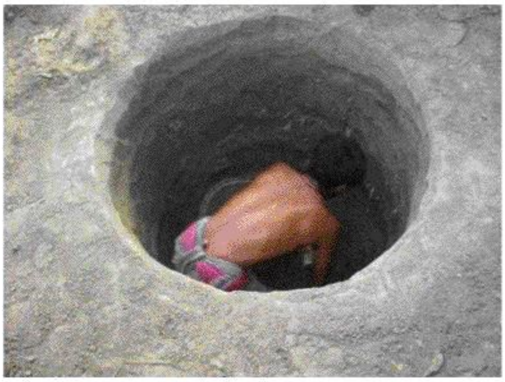
Bulk micromachining of silicon

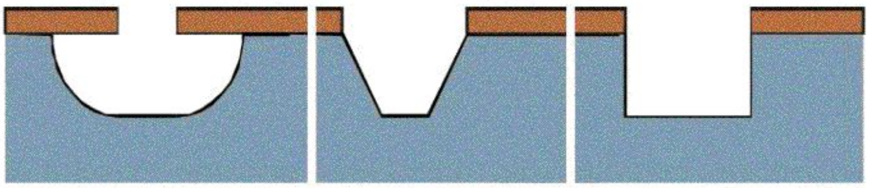
Let's start digging into the material....



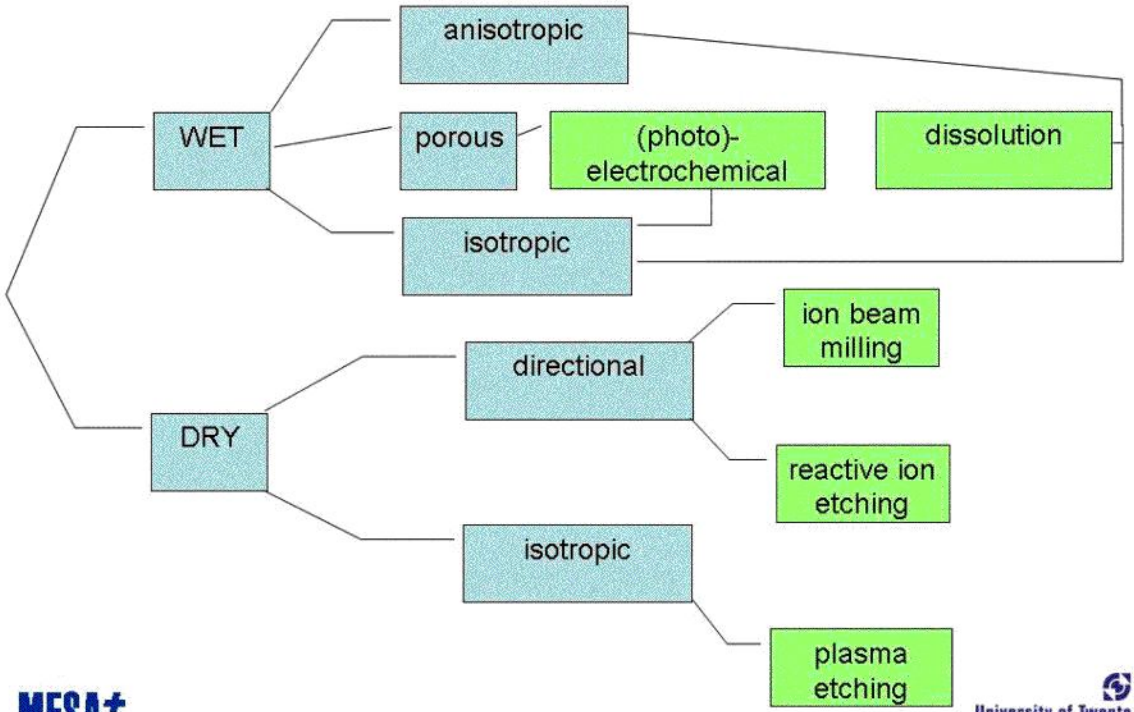
Bulk micromachining of silicon

- Thin film deposition (masking material)
- Patterning by photolithography and selective layer etching
- Selective silicon etching:

isotropic anisotropic directional



Categories of bulk micromachining



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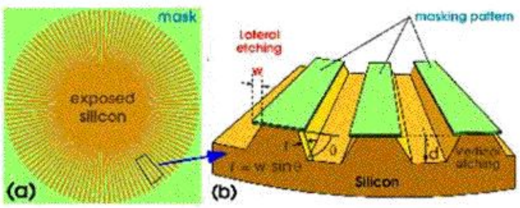
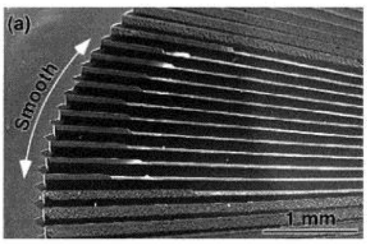
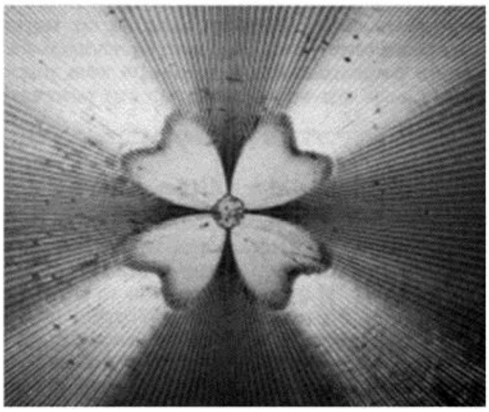
Silicon anisotropic etching

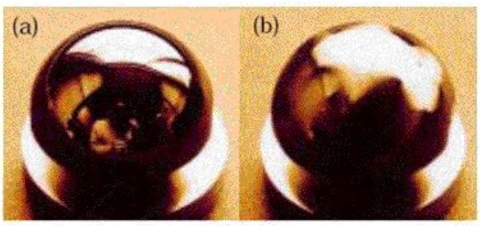


wagon wheel pattern



R.A. Wind e.a. J. Phys. Chem. B 106, 1557-1569 (2002)





Silicon test piece before and after etching
K. Sato, Nagoya University

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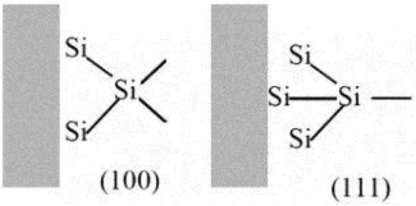
Silicon anisotropic etching

Typical conditions are: 25 wt-% KOH in water at 75 °C

{100} etches about 1μm/min

Selectivity {100} vs. {111} plane ~ 100



Overall chemical reaction:

$$Si + 4OH^{-} + 4H_2O \longrightarrow Si(OH)_6^{2-} + 2H_2 \text{ (bubbles)}$$

(can form oligomers and ultimately silica)

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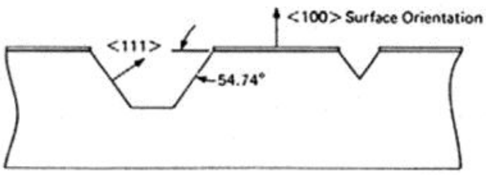
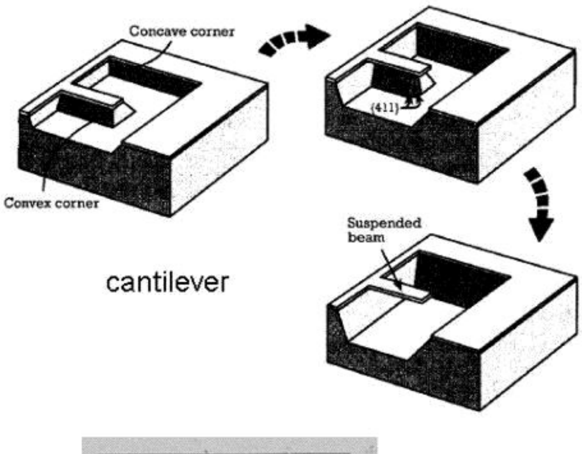
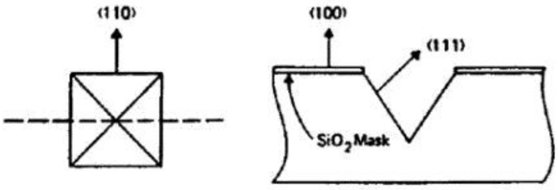
Etching of {100}

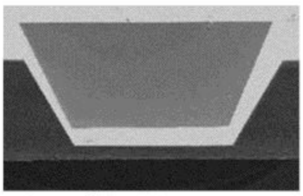




cantilever



pyramidal shape



membrane

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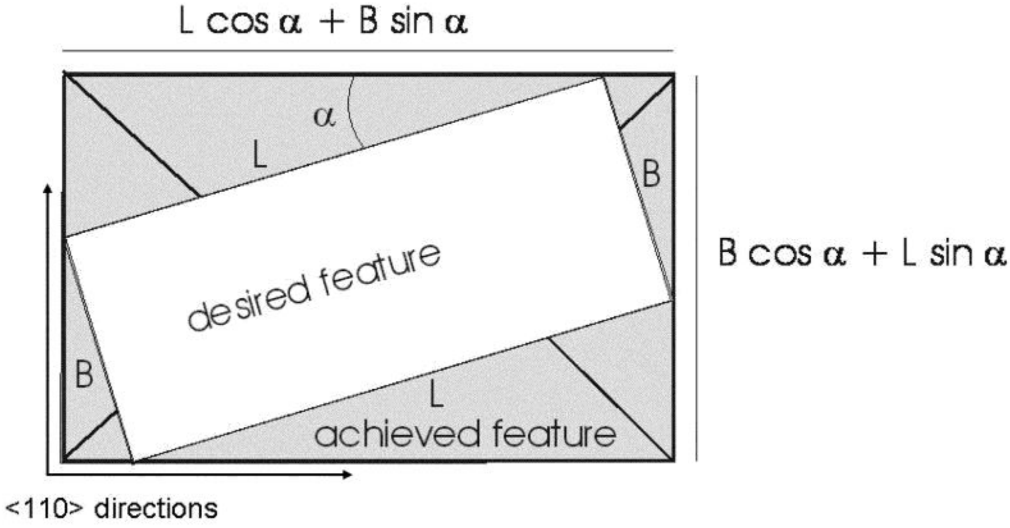
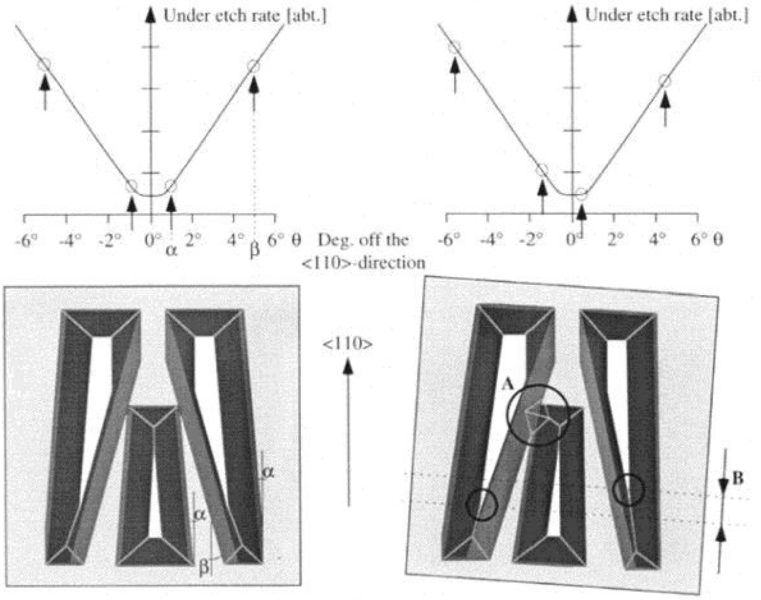
Result of misalignment to crystal directions



Precise alignment to crystal directions

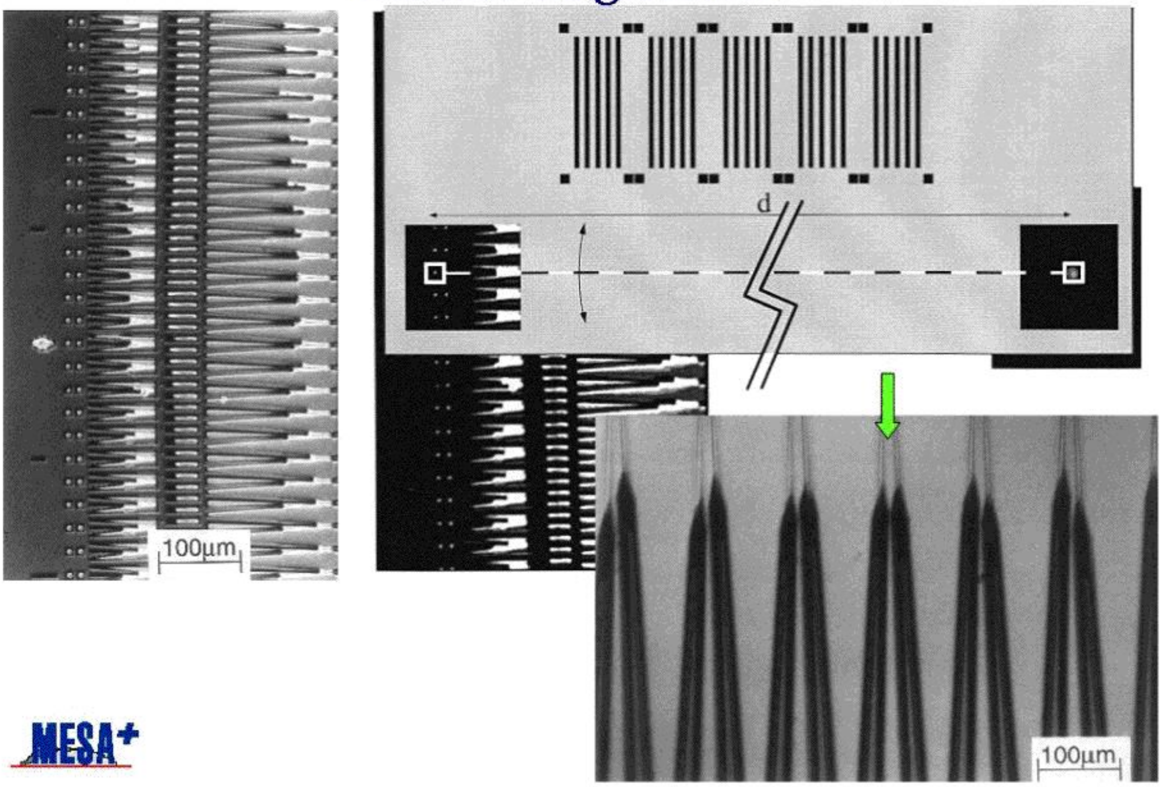


A symmetrical etching figure arises if the mask is perfectly aligned with the [110]-direction (left). Misalignment (right) can be detected by: (A) tapered ridges; (B) tapered grooves.

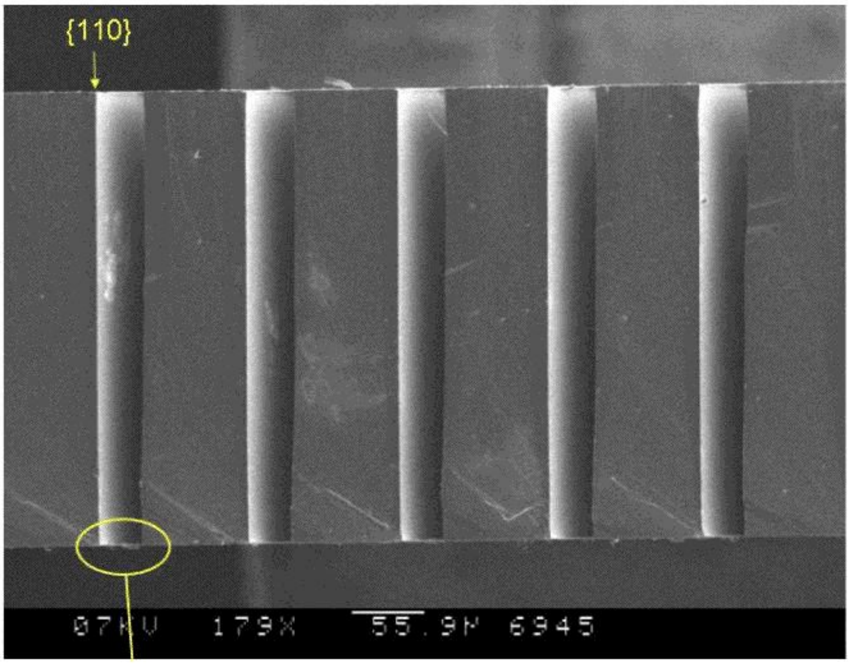
Etching time ca. 20 minutes. Best precision: 0.05°

From: M. Vangbo e.a. J. Micromech. Microeng. 6, 279-284 (1996)

Precise alignment



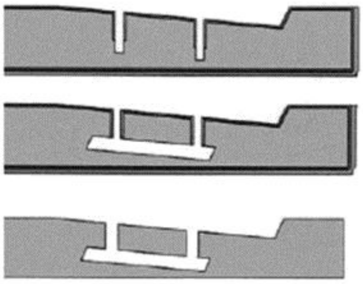
Etching of {110} wafers

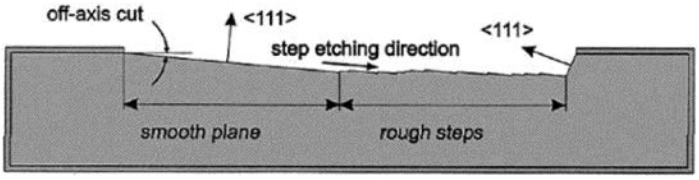


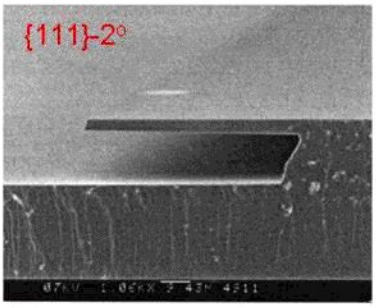
PdAg membrane

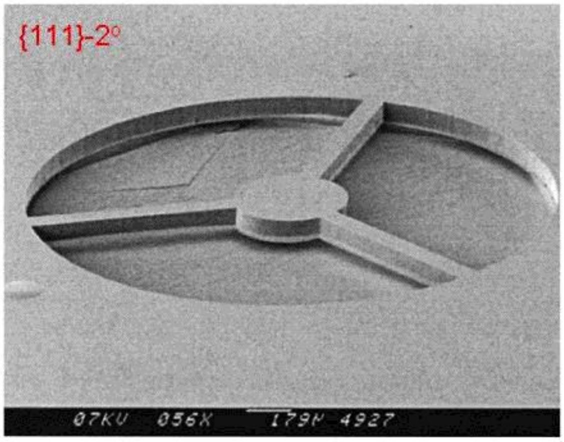
Hien e.a. J. MEMS 12, 622-629 (2003)

Etching of {111} wafers



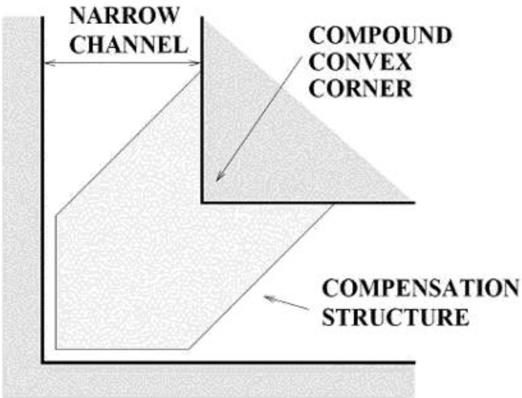


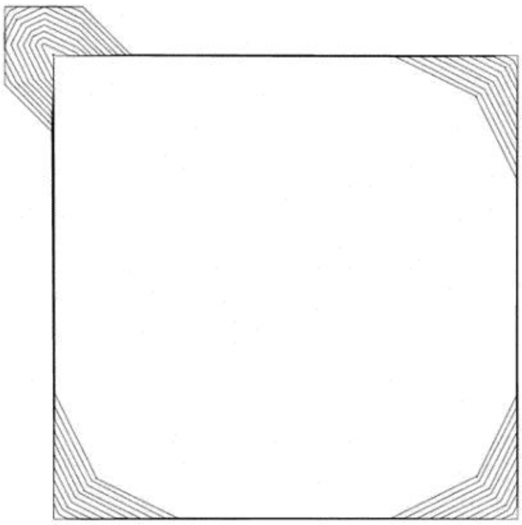




Oosterbroek e.a. J. MEMS 9, 390-398 (2000)

Convex corner compensation

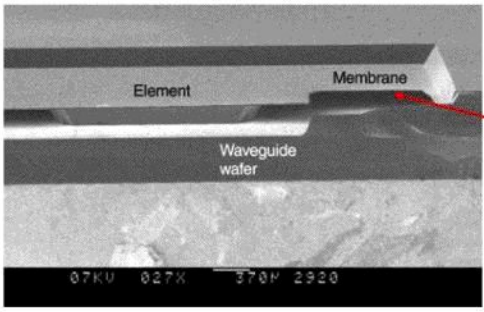


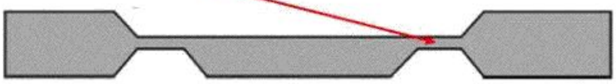


Simulation of etched profile

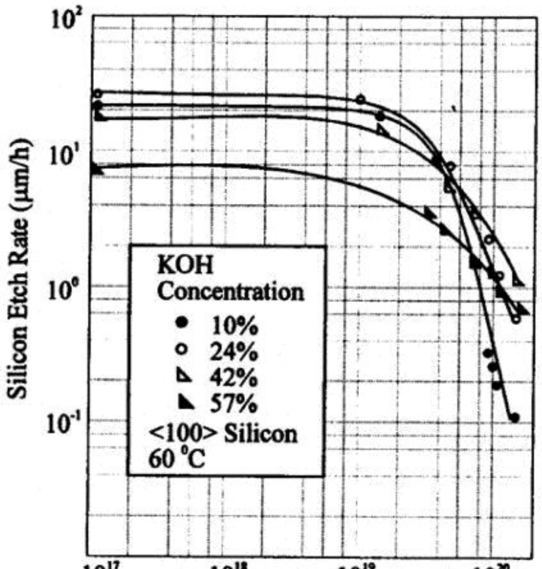
Etch stops for KOH etching

- Time:
  - simple but inaccurate
- Thin layer:
  - simple but feature depth = wafer thickness
- B-doping:
  - reliable but stress in doped layer
- Electrochemical:
  - no stress but complex (especially for batch)





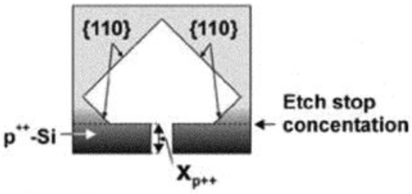
Boron etch stop

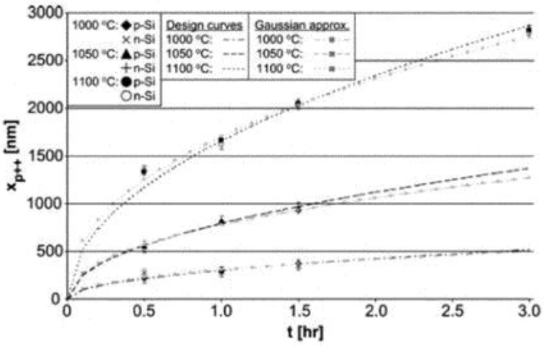


Boron layer by:
-diffusion
-implantation
-epitaxial growth

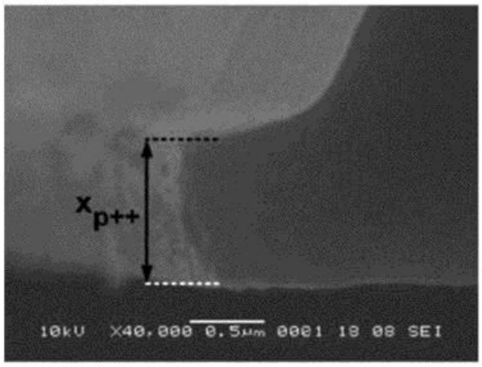
Seidel e.a. J. Electrochem. Soc. 137, 3626-3632 (1990)

Boron etch stop



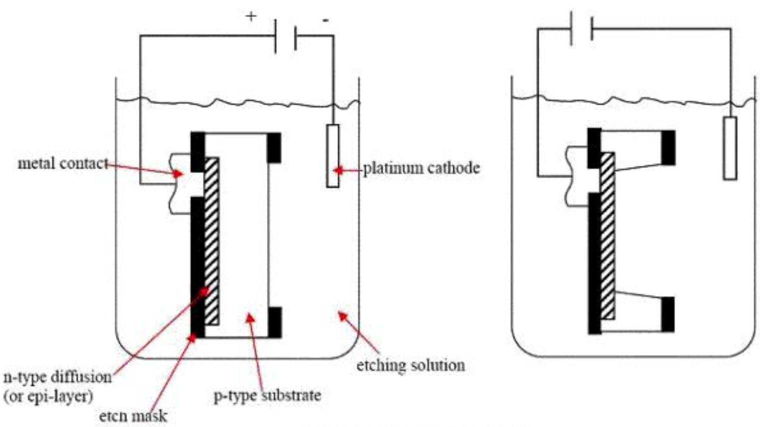


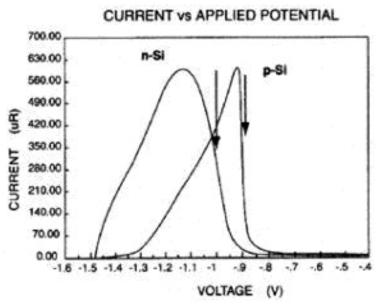
membrane thickness vs. diffusion conditions



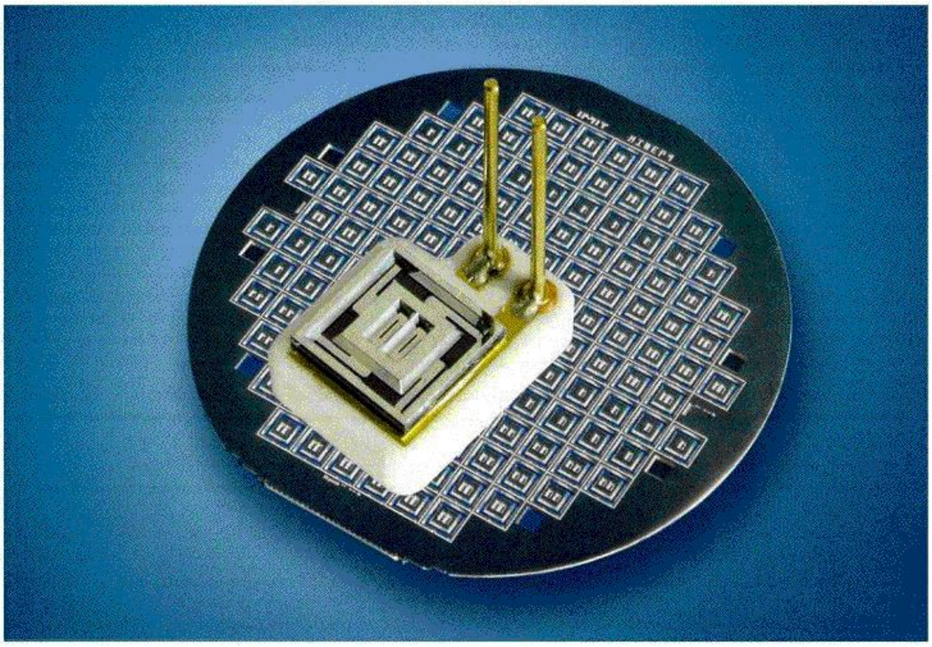
Tiggelaar e.a. Sens. Act. A 119, 196-205 (2005)

Electrochemical etch stop





Example: silicon microvalves

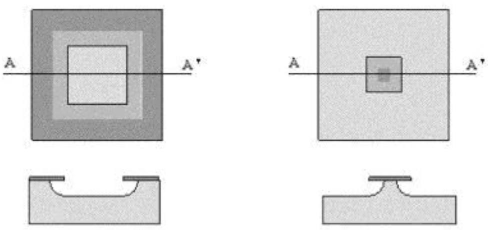


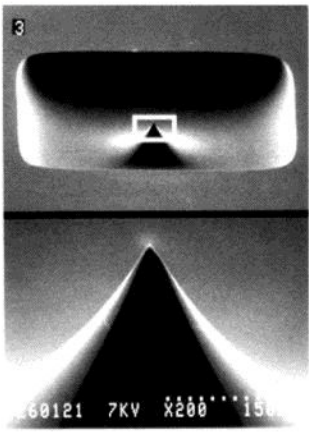
Source: HSG IMIT, Villingen-Schwenningen, D

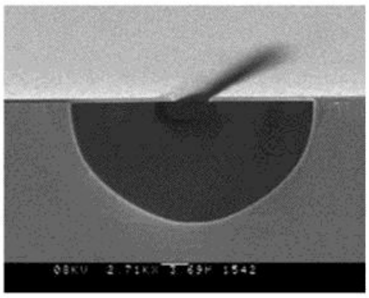
Isotropic etching (wet)

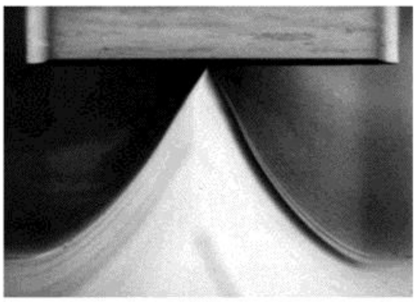
Mixtures of HNO_3 - HF in water or acetic acid (HNA)

Etch rate ~ 1µm/min (but can be much higher)

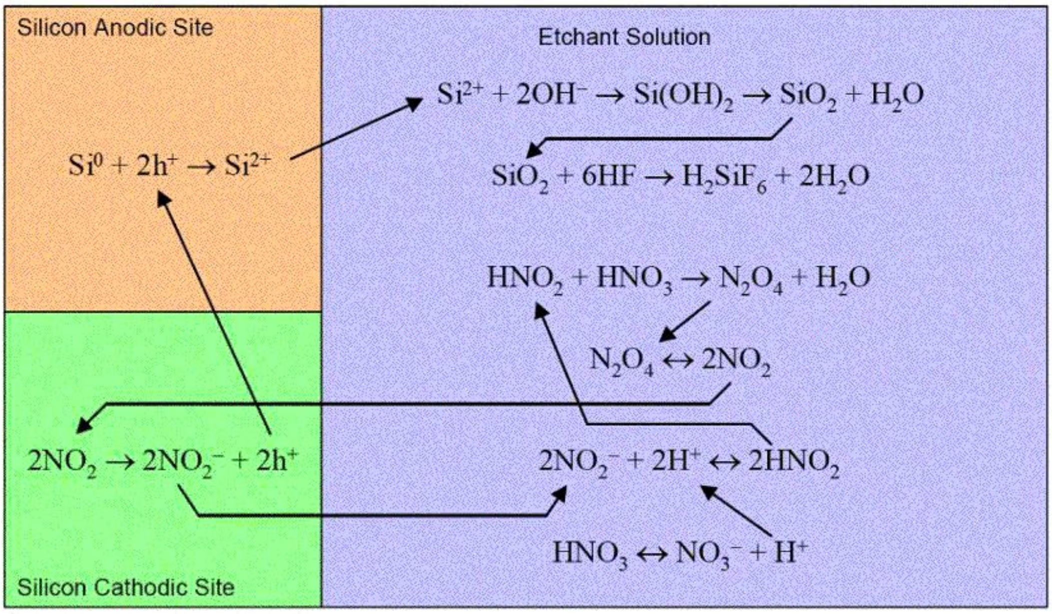






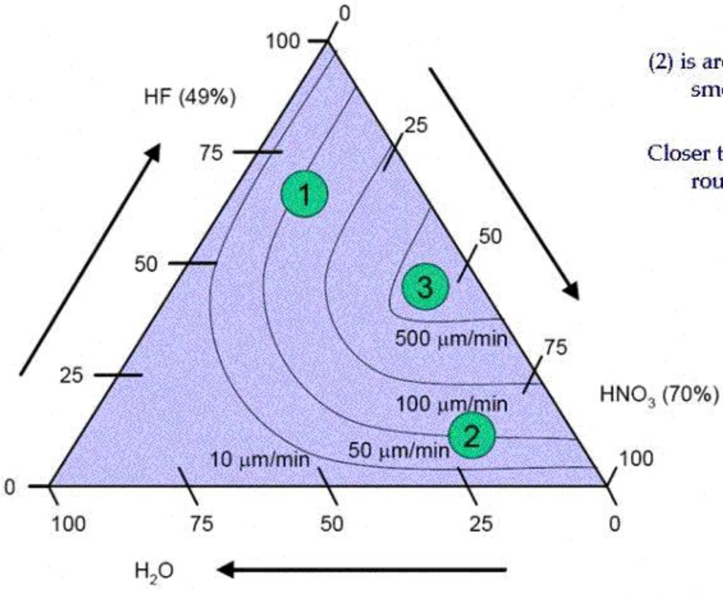


Isotropic etching using HNA



R. B. Darling / EE-527

Isotropic etching - etching rates

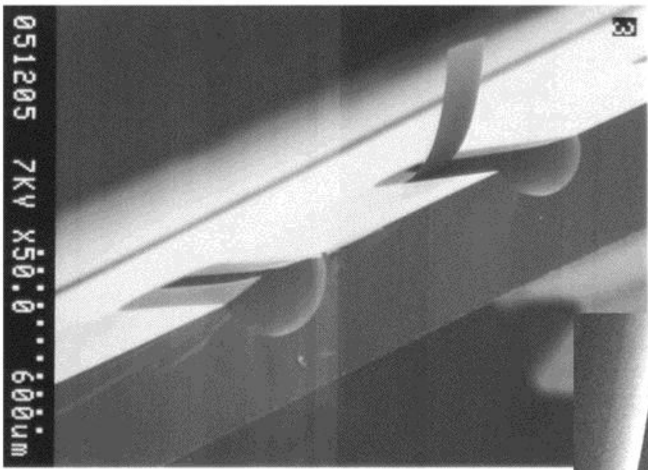


(2) is area for polishing, gives smooth surfaces

Closer to (1) gives higher roughness

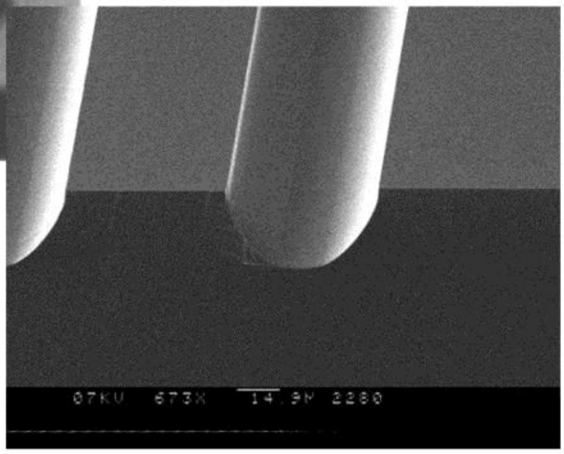
R. B. Darling / EE-527

Smooth microchannels



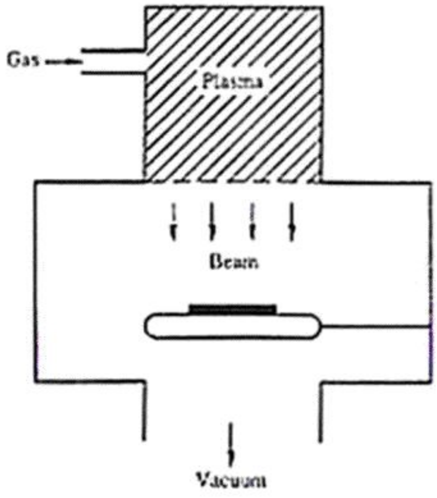
with silicon nitride mask layer

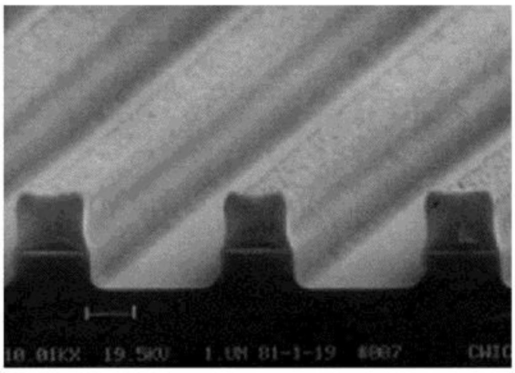
without mask



Ion beam etching (dry)

Material removal by physical impact => etching is directional
Ionized inert gas (e.g. Argon)
Etch rate ~ 5 nm/min

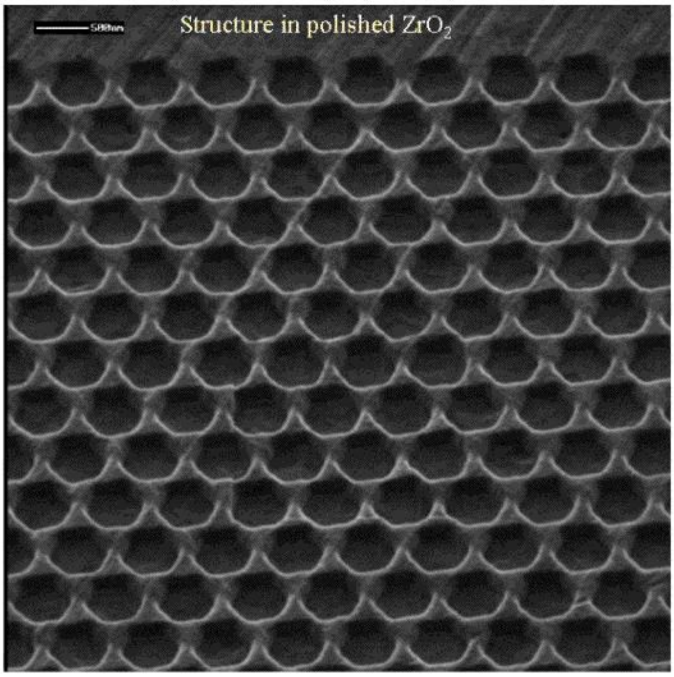


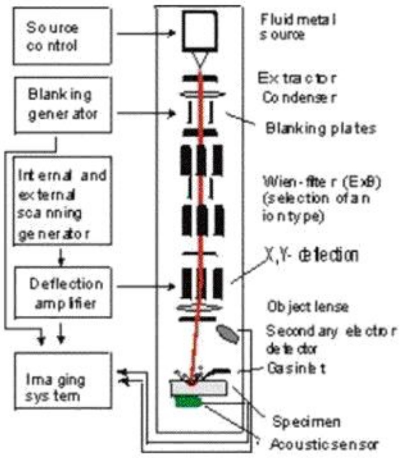


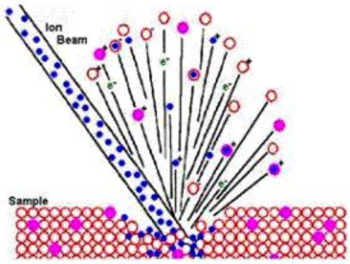
(Photoresist still on pattern)
Paul May, Bristol University 1997

University of Twente
The Netherlands

Focused ion beam etching







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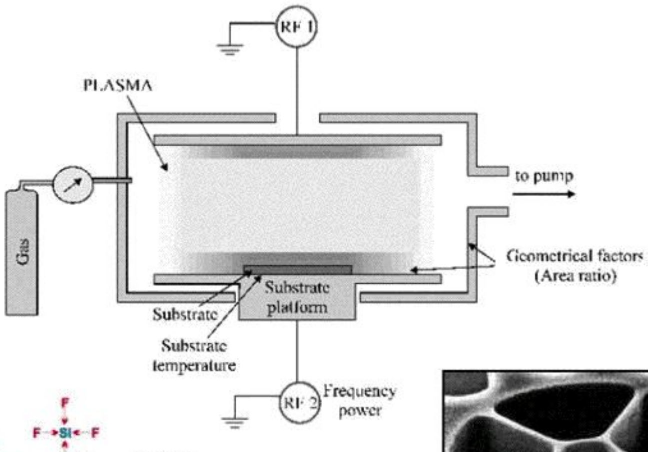
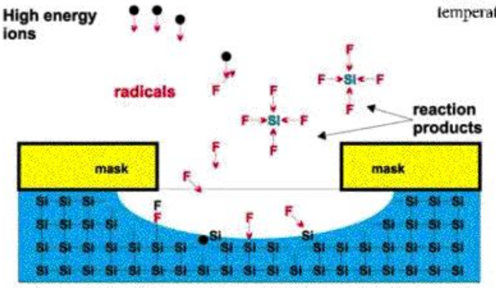
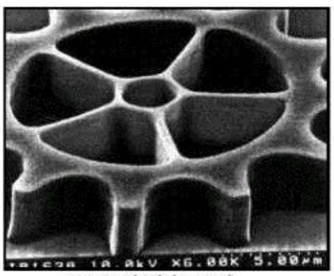
Reactive Ion Etching (RIE)







www.oxfordplasma.de

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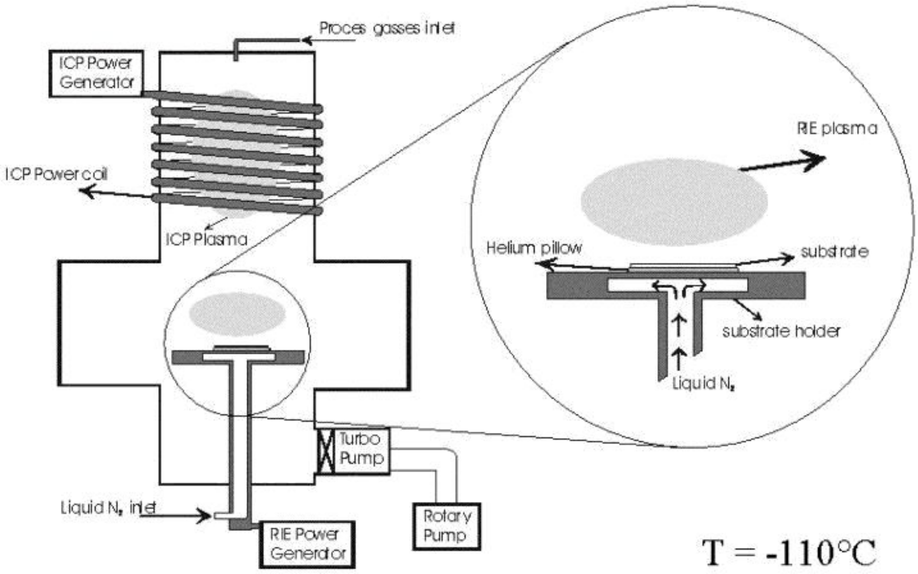
Deep Reactive Ion Etching (DRIE)



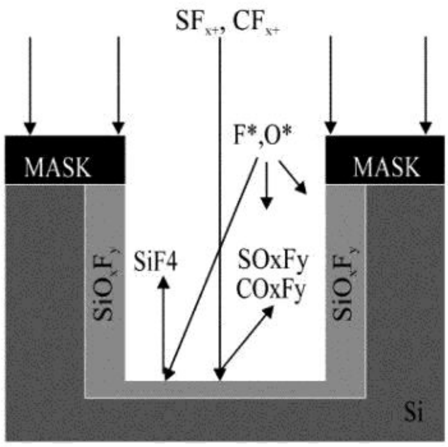
T = -110°C

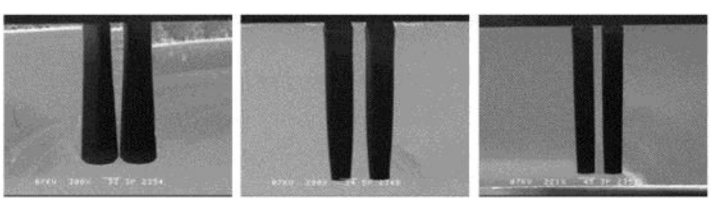
Deep Reactive Ion Etching (DRIE)

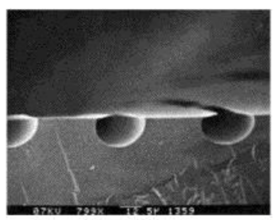
SF_6 gas etches the silicon
O_2 gas passivates the sidewalls

Isotropic or directional, depending on the settings!

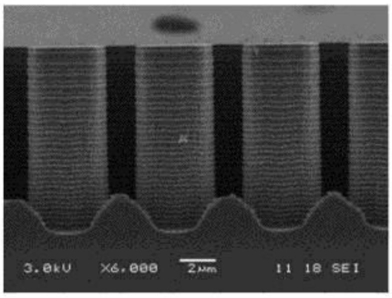
Etch rate > 5 μm/min

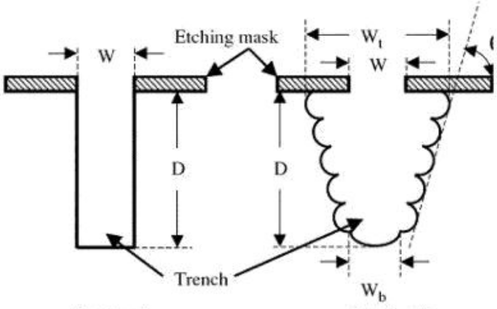


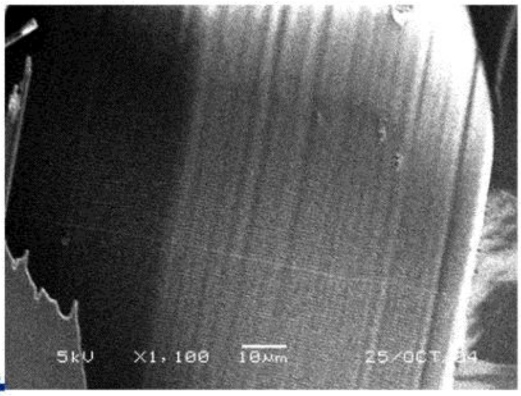


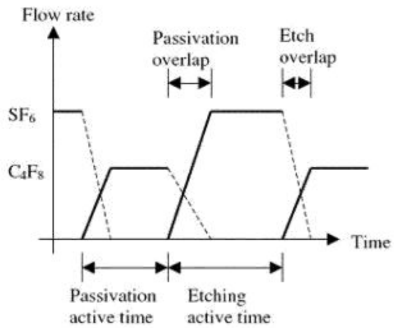


Deep RIE via "Bosch process"









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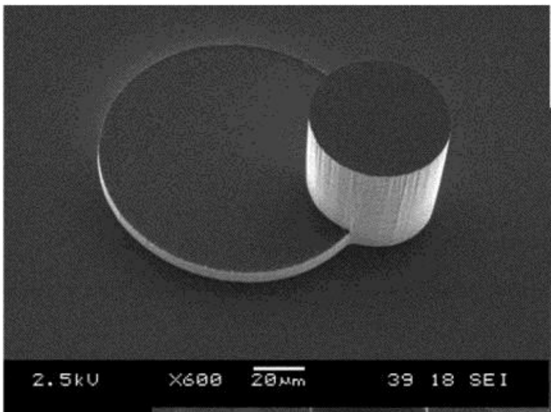
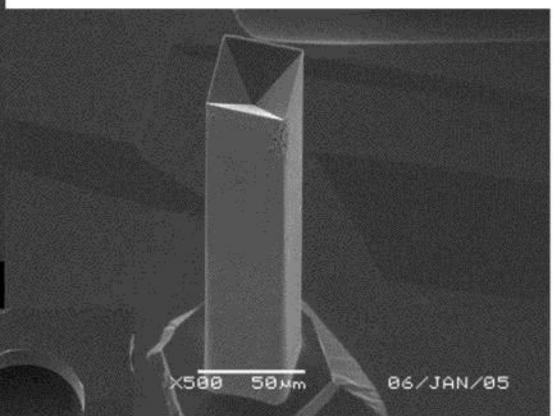
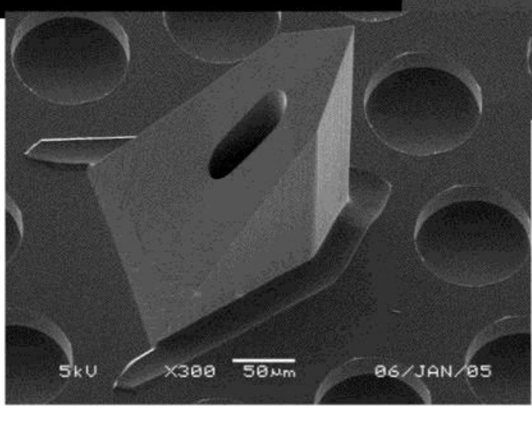
Some examples







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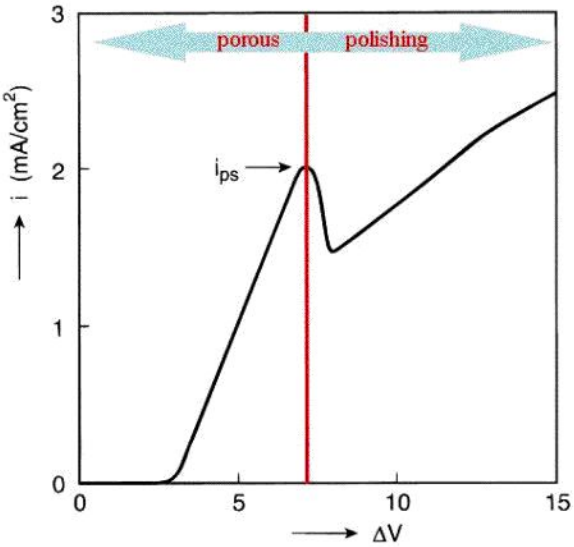
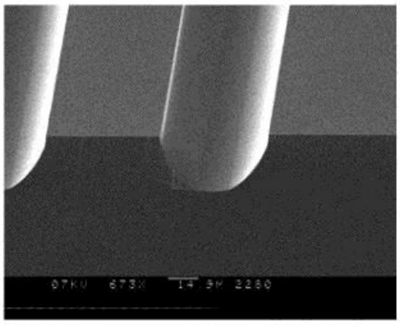
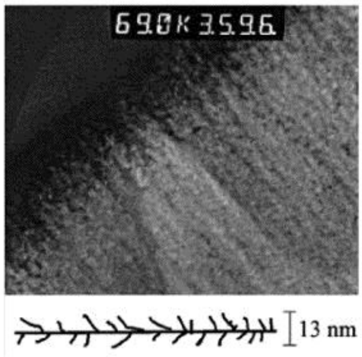
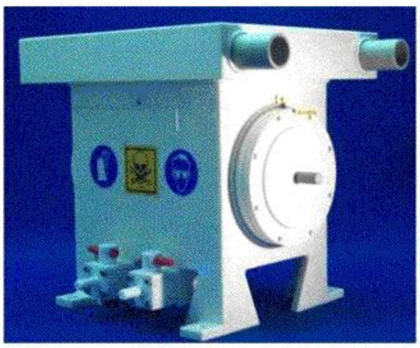
Anodic etching of Si

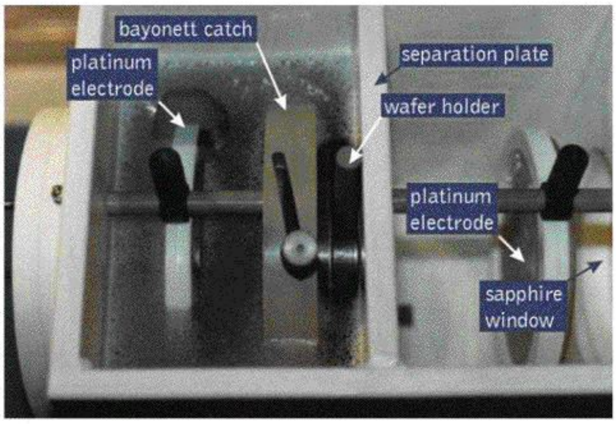






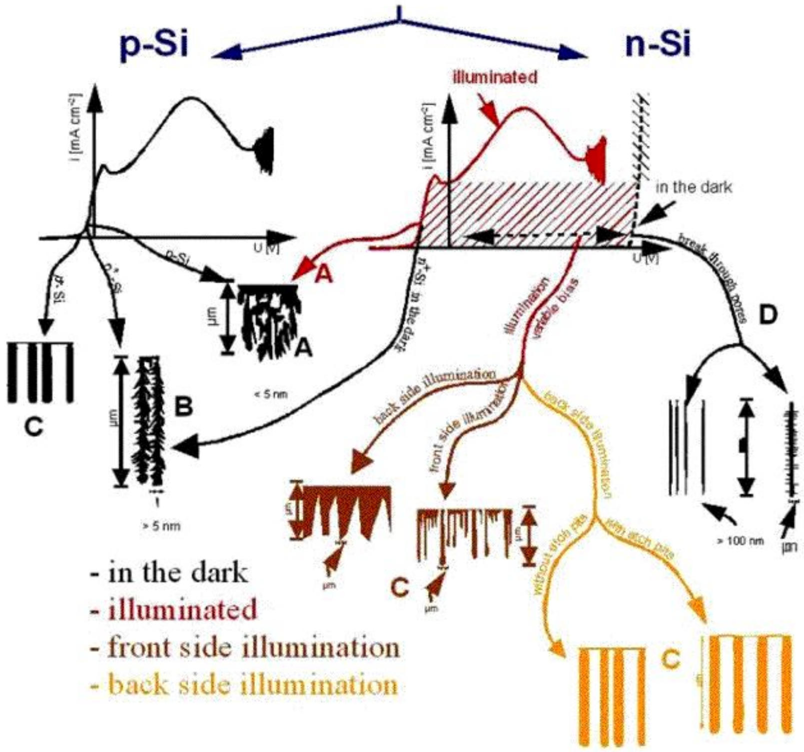
Equipment





from: http://www.ammt.com/

Pores in Silicon in aqueous Electrolytes



from: http://www.tf.uni-kiel.de/matwis/amat/por_model/all_theo/all1.html

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Regular pore formation

Influence of the space charge region (SCR)

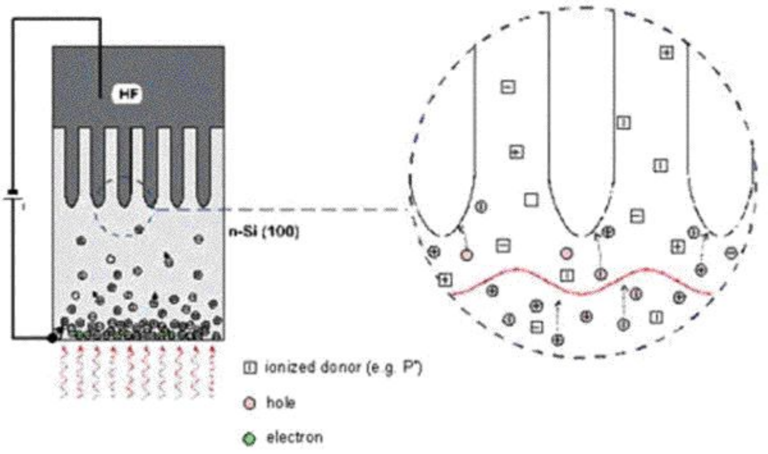
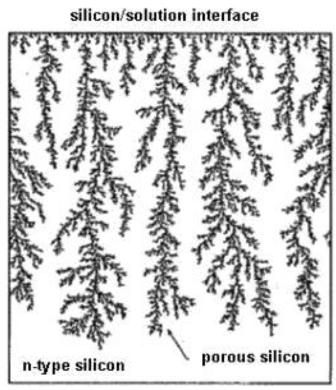


photo-electrochemically controlled process



random process

R.L. Smith e.a. J.Appl.Phys. 71, R1-R22 (1992)

http://www.mpi-halle.mpg.de/~porous_m/Si_pore_growth.html

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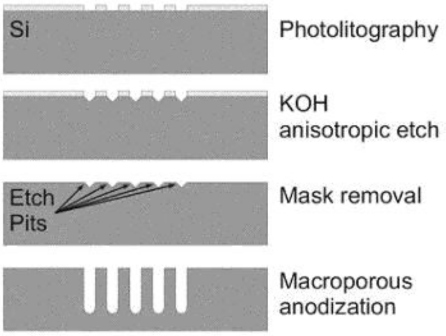
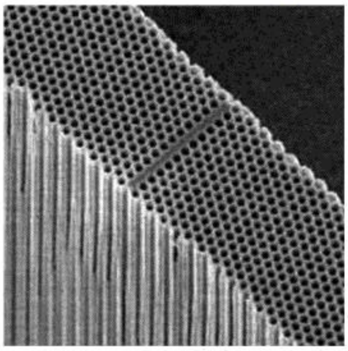
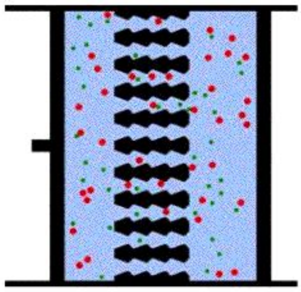
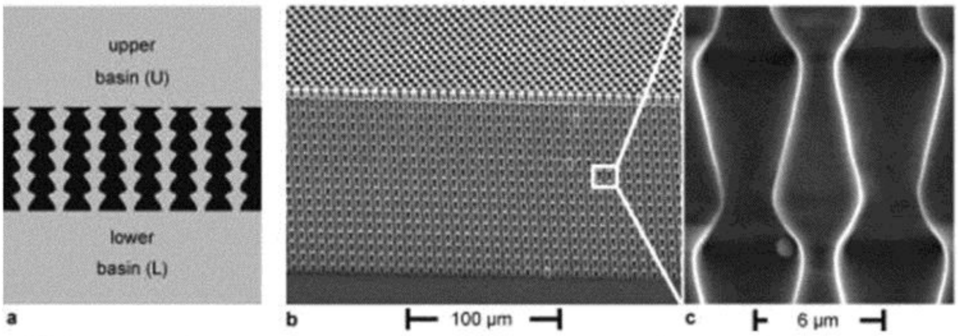
Procedure and examples



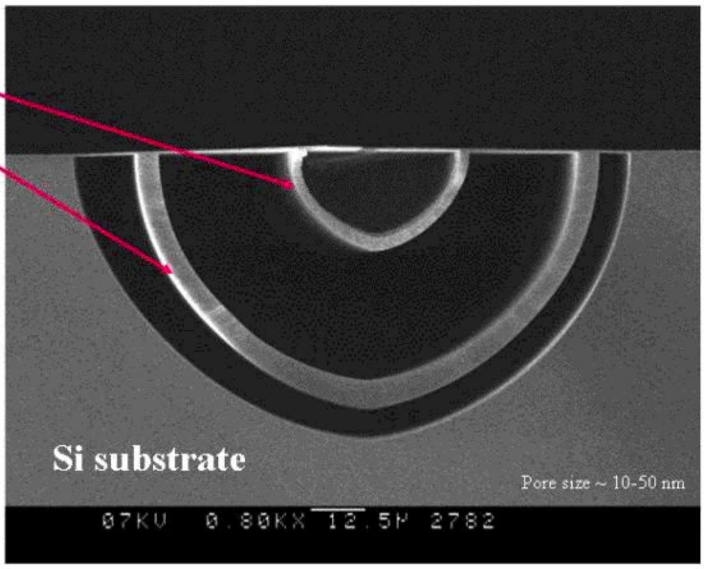






Asymmetric pores in a silicon membrane acting as massively parallel brownian ratchets"
S. Matthias & F. Müller, Nature 424, 53-57 (2003)

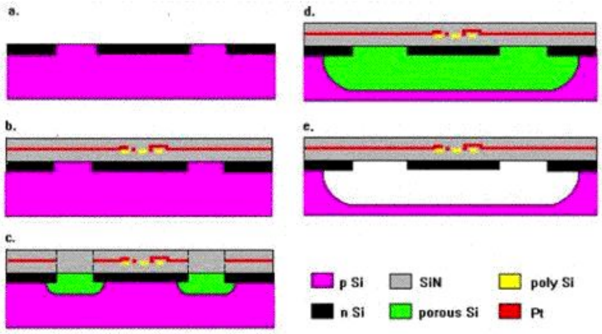
Porous silicon membranes in microchannel

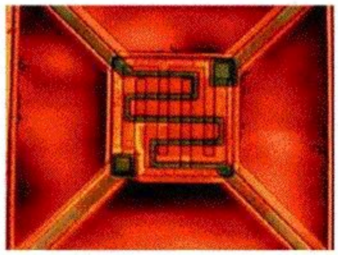


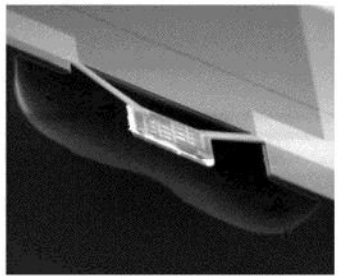
Switching of conditions during etching:

- porous Si formation (low V)
- isotropic etching (high V)

Porous Si micro hotplates for chemical sensing





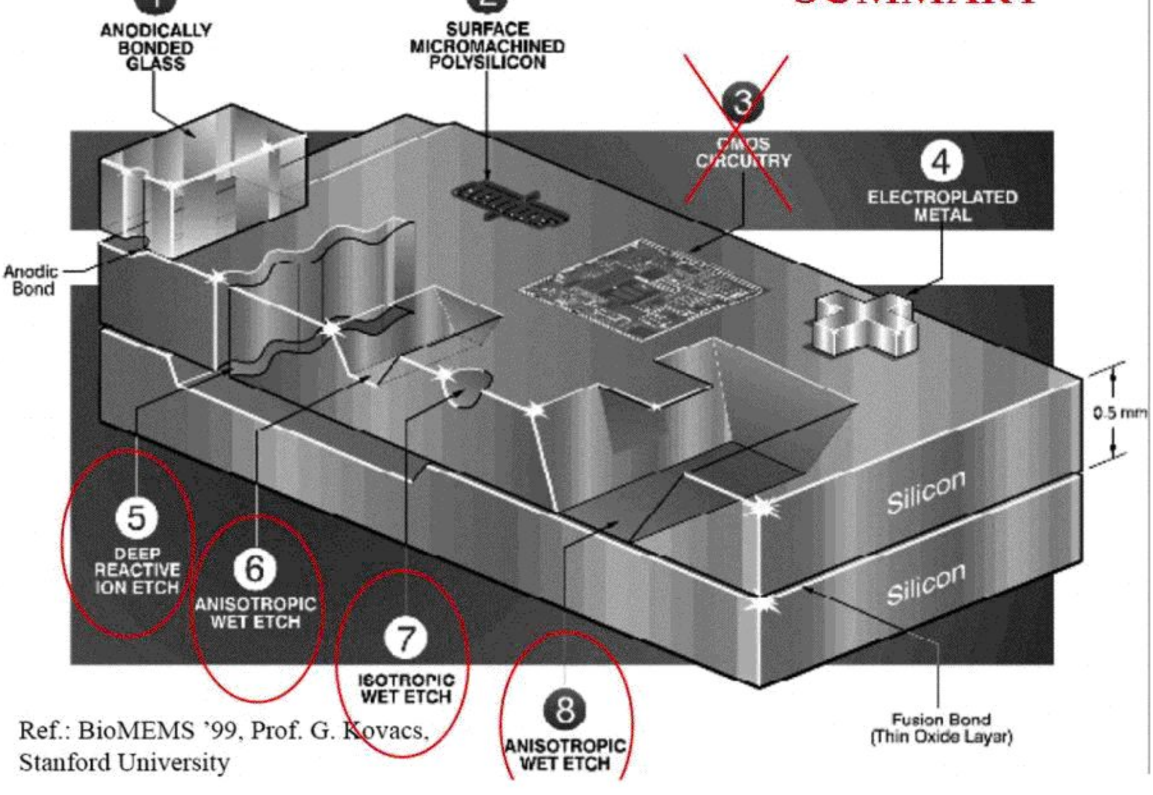


100x100 μm membrane
poly Si heater, Pt temp. sensor
30 mW for 300 ºC
2.2 μm po. Si, size 19-55 nm, 75 % po.
eff. surface area 270 times geom. area

MESA+

University of Twente
The Netherlands

SUMMARY



Ref.: BioMEMS '99, Prof. G. Kovacs, Stanford University