Lecture 10 Part II:

Microfabrication for Microfluidics and Microfluidics Devices

Silicon Etching

Polymer-based Micromachining

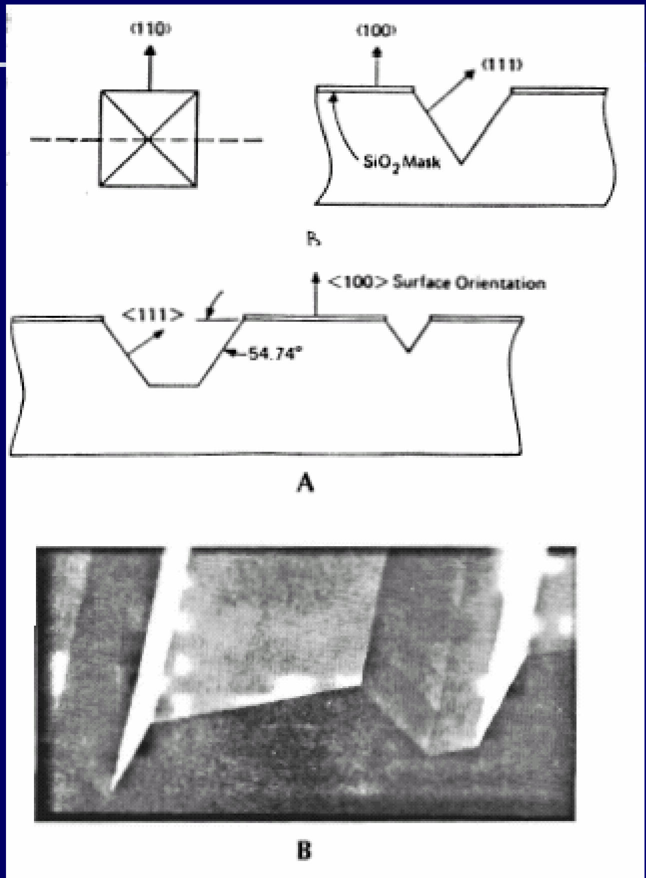
Assembly and Packaging

Biocompartibility

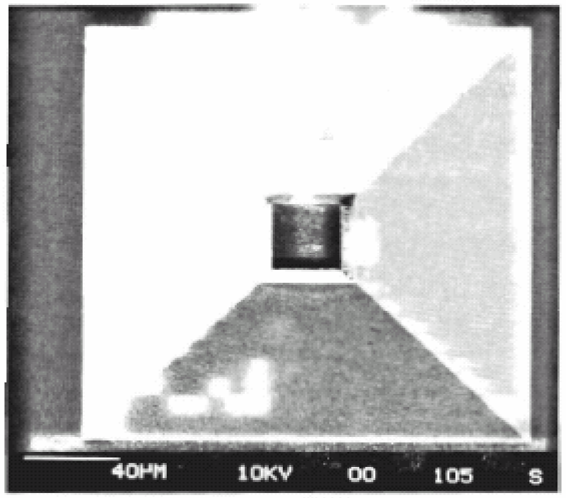
Techniques involved:

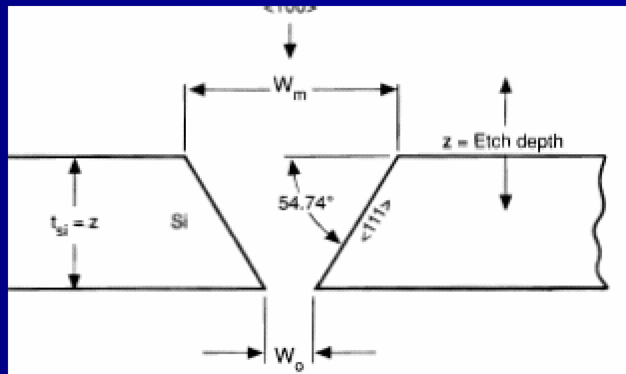
- Wet etching of channels in Si and glass (isotropic, anysotropic)
- Dry etching
- Resist lithography
- PDMS soft lithography
- Hot embossing
- Other machining techniques in plastics, glass etc.
- Bonding

Wet etching of (100) Silicon



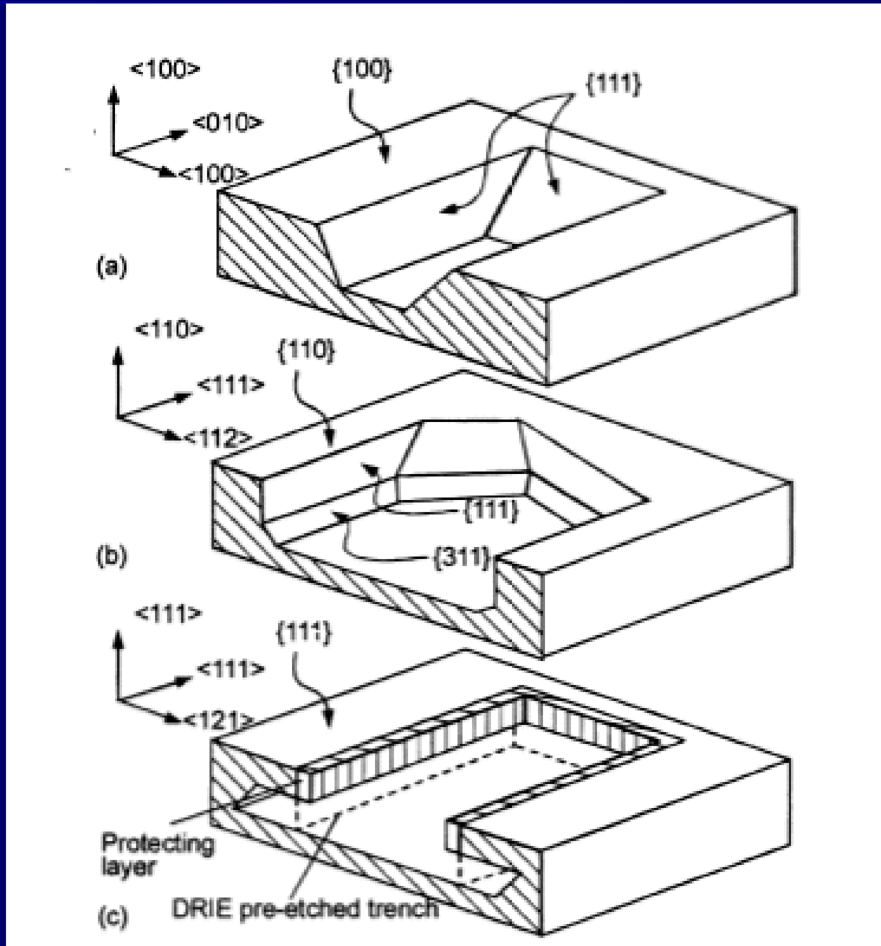
Orifice (A via through Si wafer)





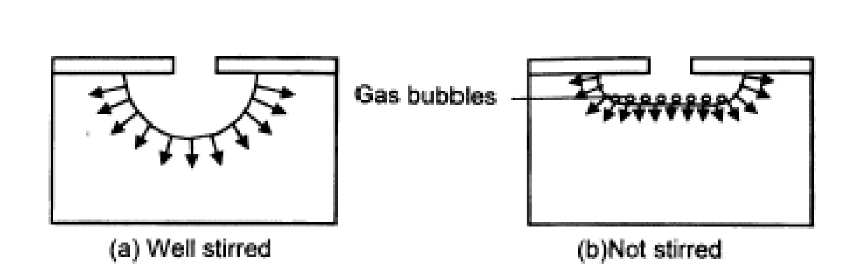
Anisotropic Etching of (100) Silicon

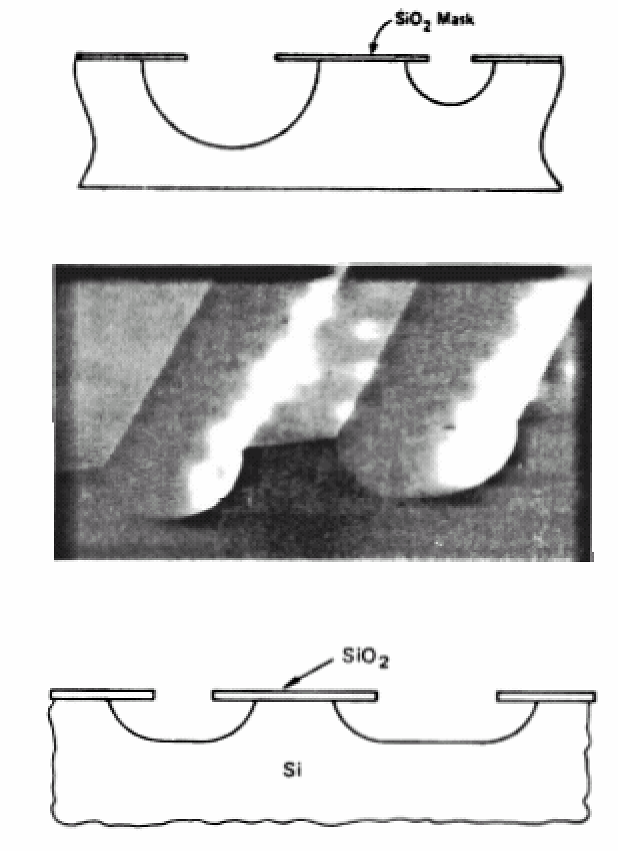
Wet etching of other orientation of Silicon



Isotropic etching of Silicon

Etchant: 66% HNO3 and 34% HF
Etching rate: 5um/min

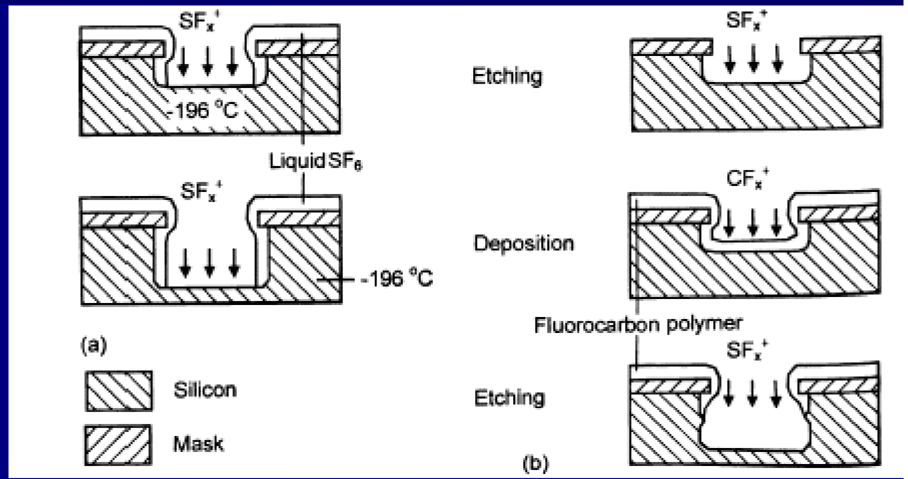




On importance of stirring….

Chemical dry etching

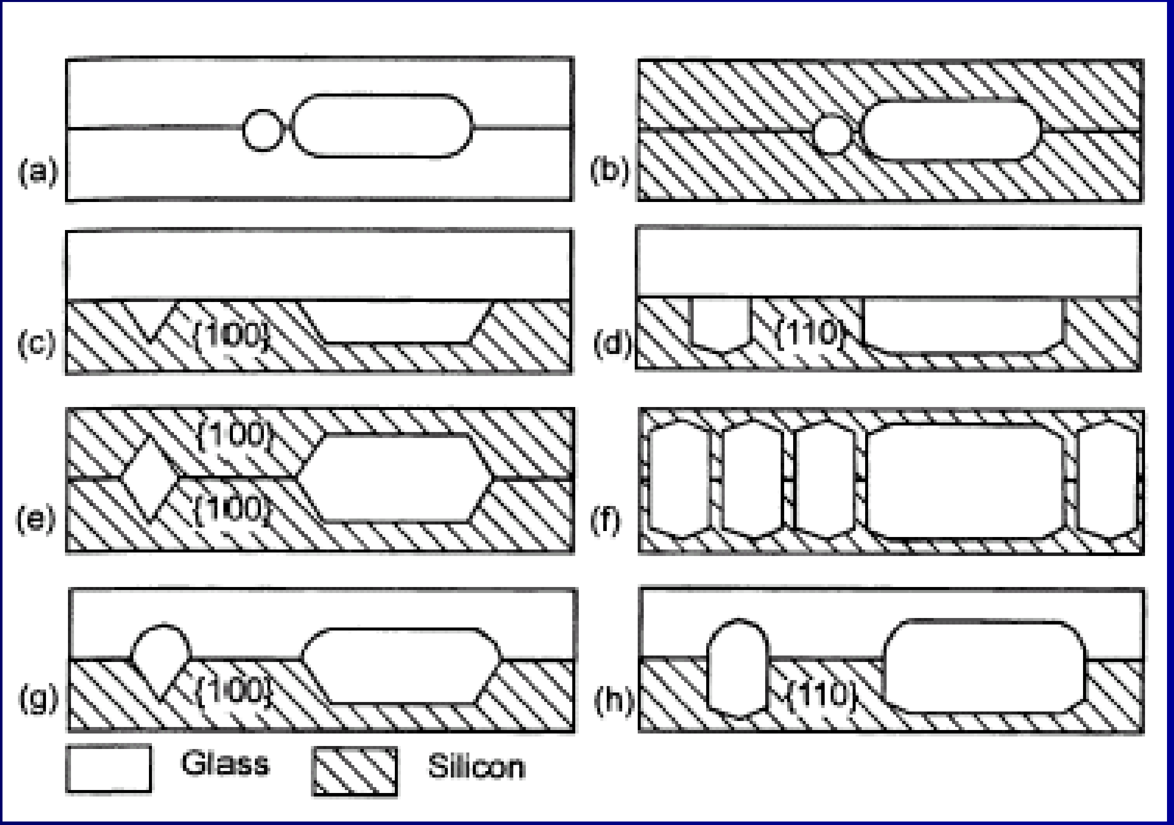
- Deep trenches with high aspect ratios can be made in Si, glass or plastic
- Gases used:
  - Fluorine chemistry (CHF_3, SF_6, CF_4)
  - Chlorine chemistry (HCl, Cl_2)
  - Oxygen



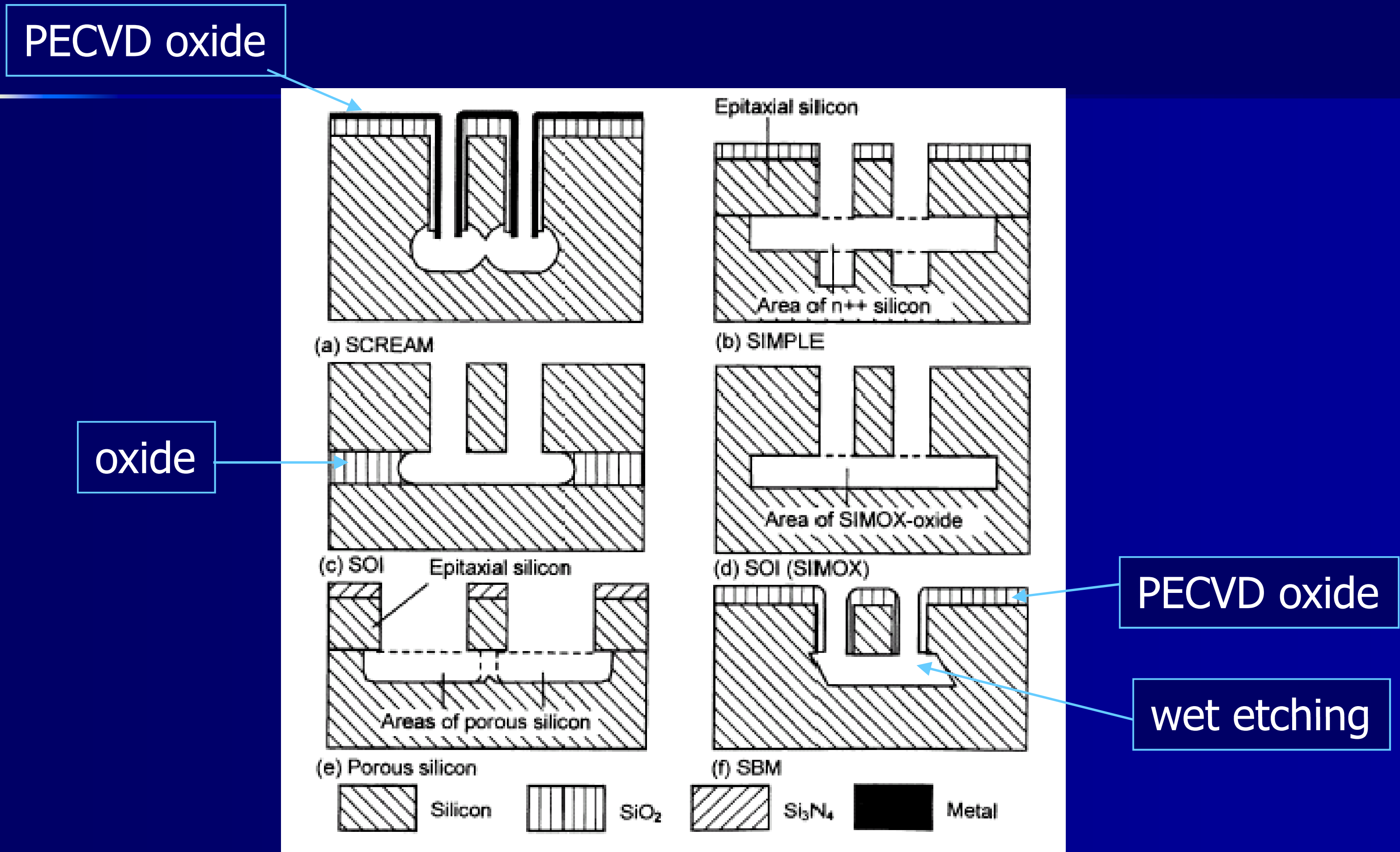
Recipes of Dry Etchant Gases for Thin Films of Functional Materials (After [3])

Material	*Etchant gases*	*Selective To*
Si	BCl_3 / Cl_2, BCl_3 / CF_4, BCl_3 / CHF_3, Cl_2 / CF_4, Cl_2 / He, Cl_2 / CHF_3, HBr, HBr /Cl_2 / He / O_2, HBr /NF_3 / He / O_2, HBr / SiF_4 / NF_3, HCl, CF_4	SiO_2
SiO_2	CF_4/ H_2, C_2F_6, C_3F_8, CHF_3, CHF_3 / O_2, CHF_3 / CF_4, (CF_4/ O_2)	Si (Al)
Si_3N_4	CF_4/ H_2, (CF_4/ CHF_3/ He, CHF_3, C_2F_4)	Si (SiO_2)
Al	BCl_3, BCl_3 / Cl_2, BCl_3 / Cl_2 /He, BCl_3 / Cl_2 /CHF_3 / O_2, HBr, HBr / Cl_2, HJ, $SiCl_4$, SiCl / Cl_2, Cl_2 / He	SiO_2
Organics	O_2, O_2/CF_4, O_2/SF_6	

Bulk micromachined channels



Silicon surface micromachining



e 3.13 Single crystalline silicon surface micromachining: (a) SCREAM; (b) SIMPLE; (c) SOI

Polymer based micromachining

- Thick resist lithography
- Polymeric based micromachining
- Soft lithography
- Microstereo lithography
- Micromolding

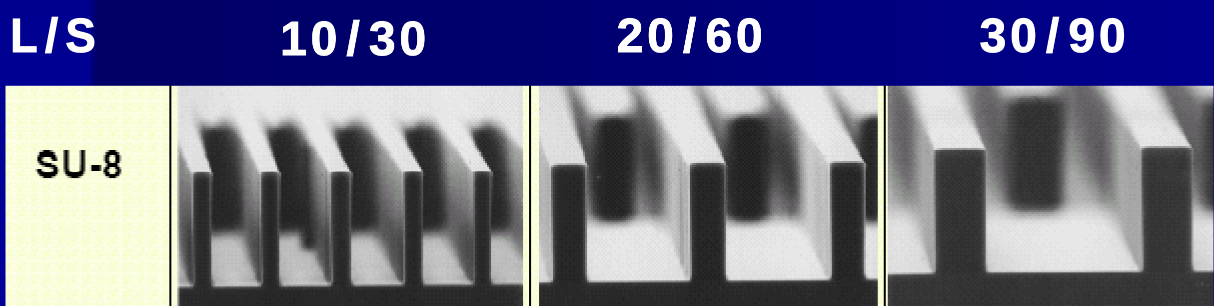
SU-8 resist

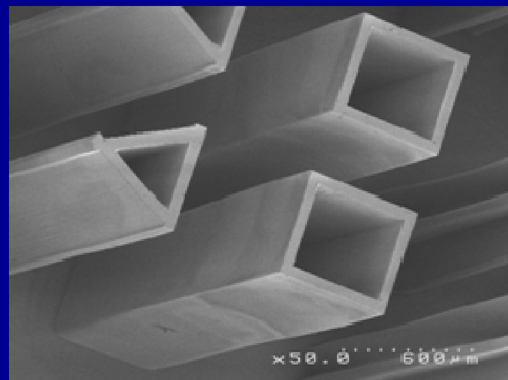
- Negative photoresist for NUV exposure

Film Thickness of Different SU-8 Types at a Spin Speed of 1,000 rpm (After [76, 77])

Type	*Kinematic Viscosity (m^2/s)*	*Thickness (μm)*
SU-8 2	4.3×10^{-5}	5
SU-8 5	29.3×10^{-5}	15
SU-8 10	105×10^{-5}	30
SU-8 25	252.5×10^{-5}	40
SU-8 50	$1{,}225\times10^{-5}$	100
SU-8 100	$5{,}150\times10^{-5}$	250

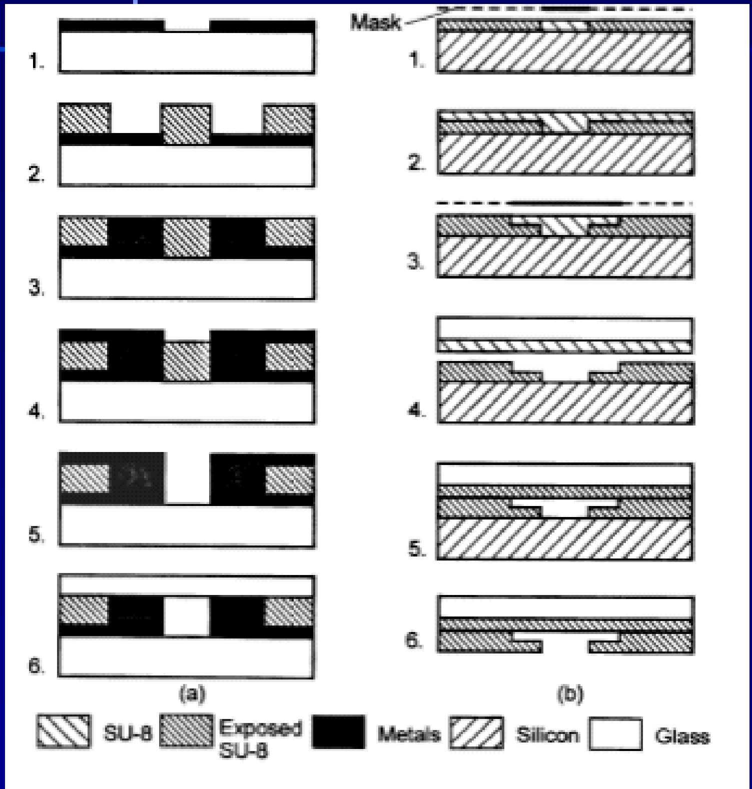
Really thick layers in one spin!



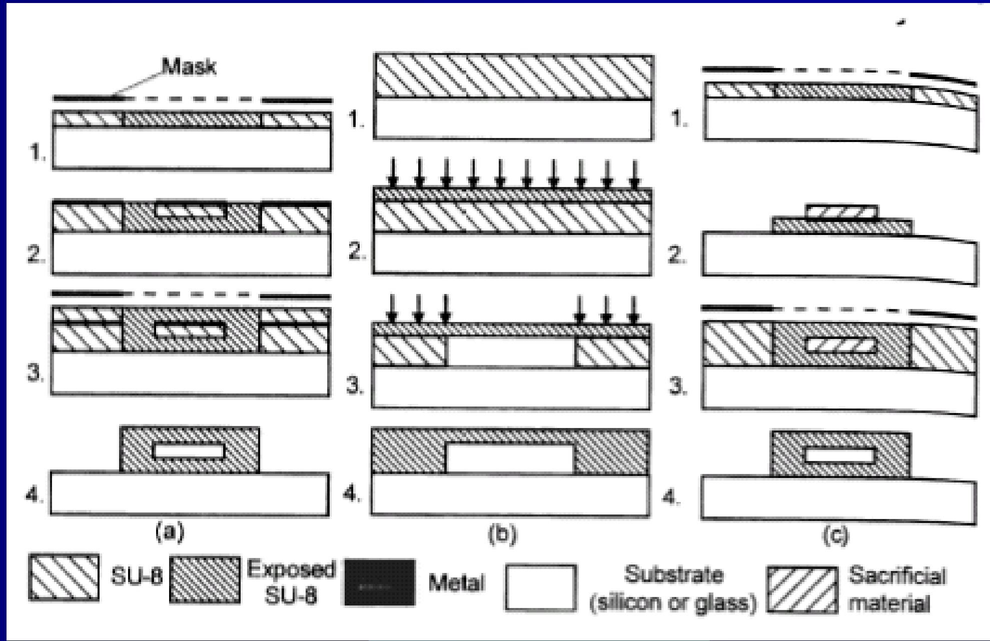


Example of SU-8 structures

Fabrication of open channels



Fabrication of covered channels

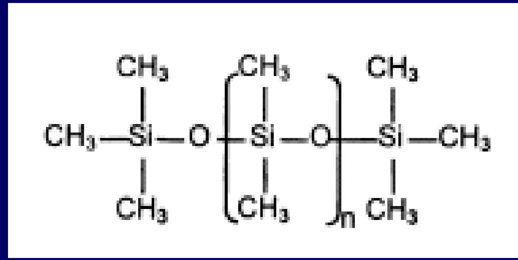


embedded mask

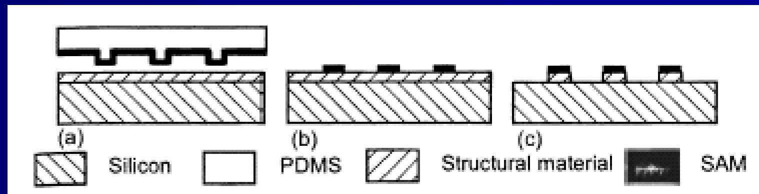
selective proton writing

sacrif.layer

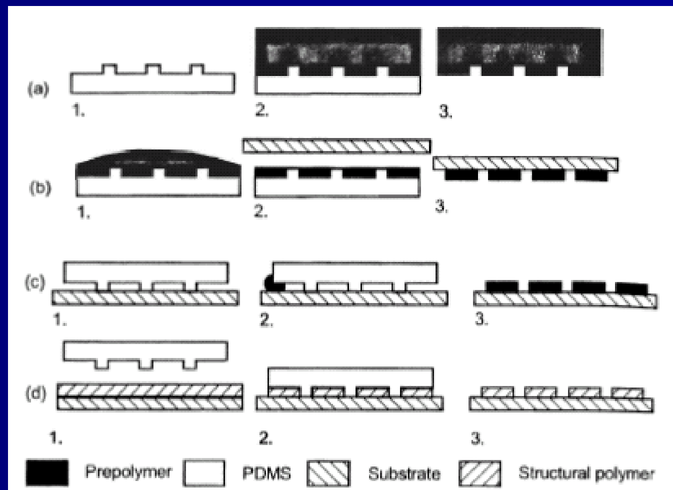
Soft lithography



- Uses elastomeric stamp, usually PDMS (Polydimethylsiloxane) to transfer the pattern.
  - Microcontact printing

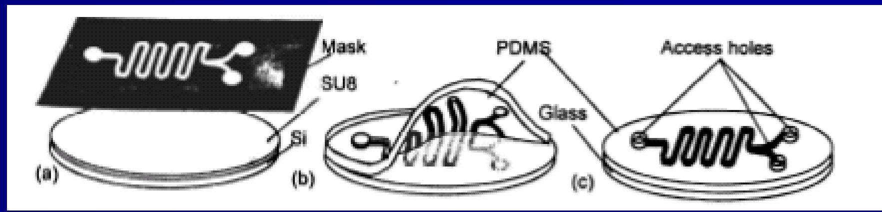


  - Micromolding



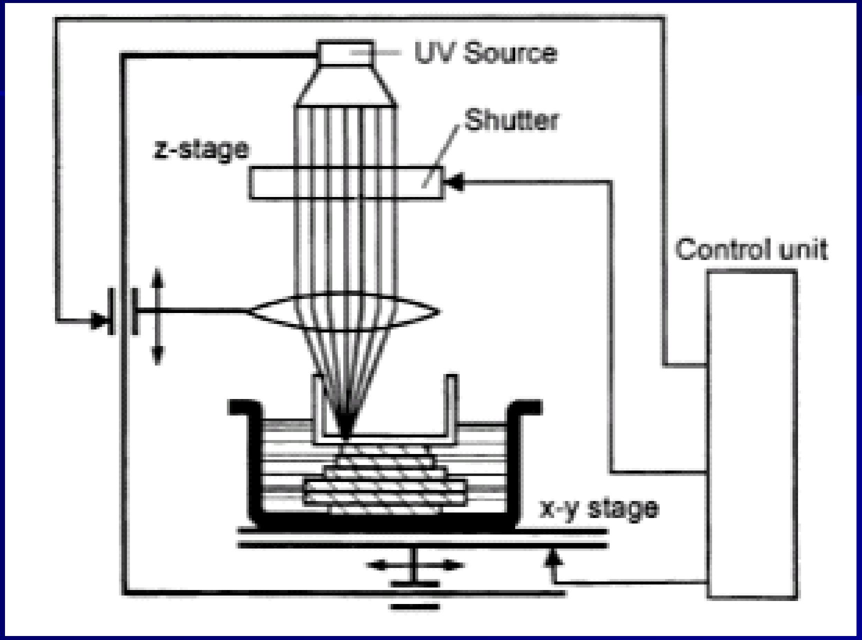
Fabrication of microchannels using soft lithography

- Advantages of PDMS:
  - Low cost
  - Transparency in VIS and NUV
  - Chemically inert



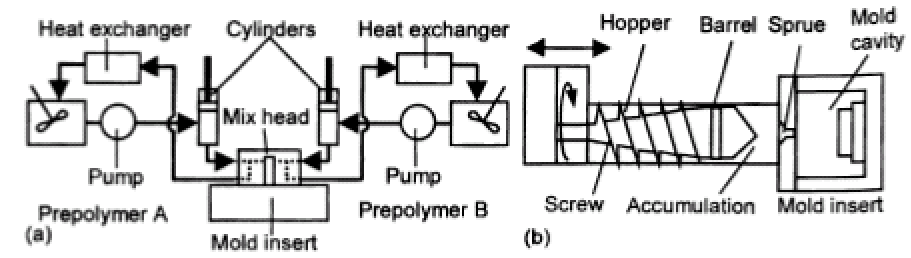
- Technology:
  - Mix prepolymer and curing agent 10:1 – 5:1
  - Pour into solid master made in SU-8 with inlets defined by glass posts
  - Cure at 60 – 80 oC for couple of hours
  - Peel off
  - treat with ozon or Oxygen plasma and attach to clean glass, silicon or another PDMS

Microstereo lithography



Single photon adsorbtion
Two photon absorbtion
Layer-by-Layer photolithography

Micromolding



- Injection molding: high pressure injection of molten PMMA, PC (polycarbonate), PSU (polysulfone) etc.

Typical Characteristics of Different Polymers for Micromolding (After [120, 121])

Resist	*PMMA*	*PC*	*PS*	*COC*	*PP*
Heat resistance (°C)	105	140	100	130	110
Density (kg/m^3)	1,190	1,200	1,050	1,020	900
Refractive index	1.42	1.58	1.59	1.53	opaque
Resistant to:					
• Aqueous solutions	yes	limited	yes	yes	yes
• Concentrated acids	no	no	yes	yes	yes
• Polar hydrocarbons	no	limited	limited	yes	yes
• Hydrocarbons	yes	yes	no	no	no
Suitable for micromolding	moderate	good	good	good	moderate
Permeability coefficients (× 10^{-17} m^2/s-Pa):					
• He	5.2	7.5	-	-	-
• O_2	0.12	1.1	-	-	-
• H_2O	480 –1,900	720 – 1,050	-	-	-
Hot-embossing parameters:					
Embossing temperature (°C)	120 –130	160 –175	-	-	-
Deembossing temperature (°C)	95	135	-	-	-
Embossing pressure (bars)	25 – 37	25 – 37	-	-	-
Hold time (s)	30 – 60	30 – 60	-	-	-

- Compression molding (hot embossing)

Other micromachining techniques

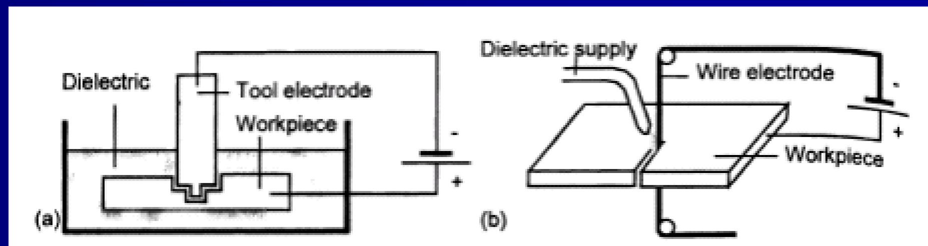
- Laser micromachinig
  (usually using an excimer lasers,
  Nd:YAG or CO_2 lasers)

Typical Ablation Depths Per Pulse of Different Material (Nanosecond Laser)

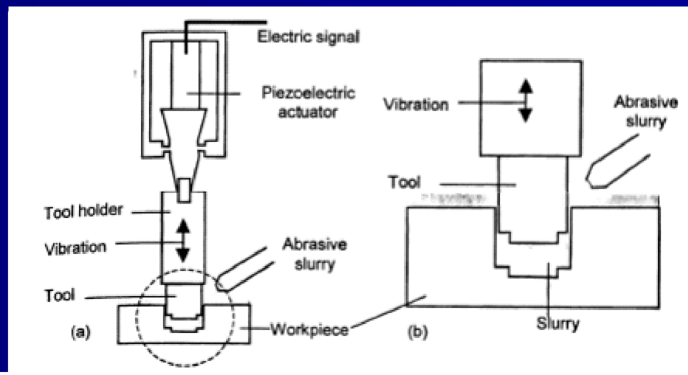
Material	*Depth Per Pulse (μm)*
Polymers	0.3 – 0.7
Ceramics and glass	0.1 – 0.2
Diamond	0.05 – 0.1
Metals	0.1 – 1.0

- Focused ion beam

- Microelectro discharge

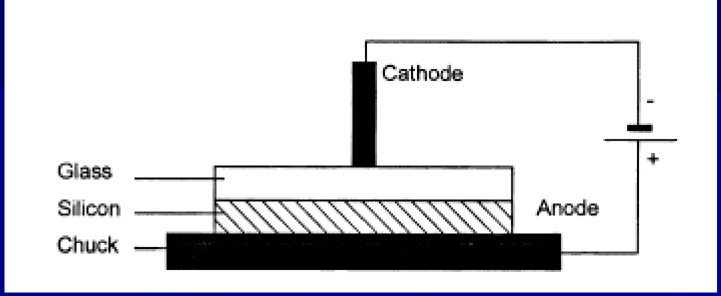


- Ultrasonic micromachining



Assembly and packaging

- Anodic bonding
  (T=400 °C, V=1kV)



- Silicon direct bonding
  reaction of OH groups on Si surfaces at T=300 – 1000 °C
- Glass direct bonding
  (T=600 °C for 6-8h)
- Polymer direct bonding
- Adhesive bonding (low melting glass (400-600°C, photoresists, UV curable epoxies, epoxies etc.)
- Eutectic bonding (e.g. gold/silicon eutectic at 363°C)

Other microfabrication issues: Biocompartibility

- Material responce to biological environments (swelling, corrosion etc.)
- Tissue and cellular responce to the material

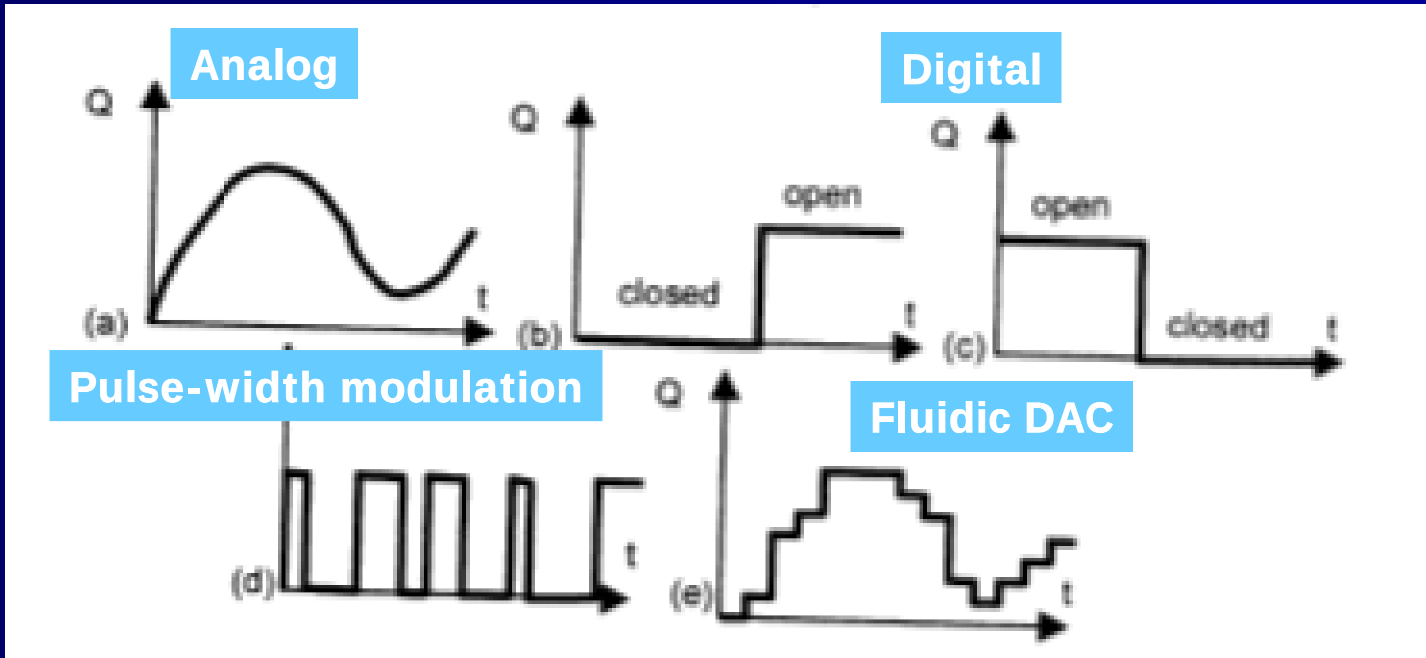
Microfluidics: Devices for Flow Control

- Valves
- Pumps
- Micromixers

Microvalve consist of closure element driven by an actuator

Active Microvalves → Normally open / Normally closed / Bistable

Active Microvalves → Analog / Digital



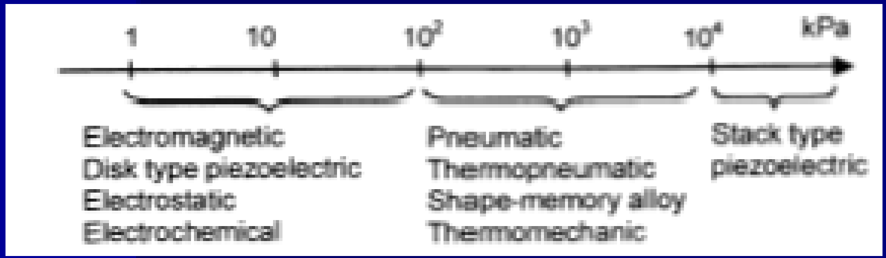
Actuation principle

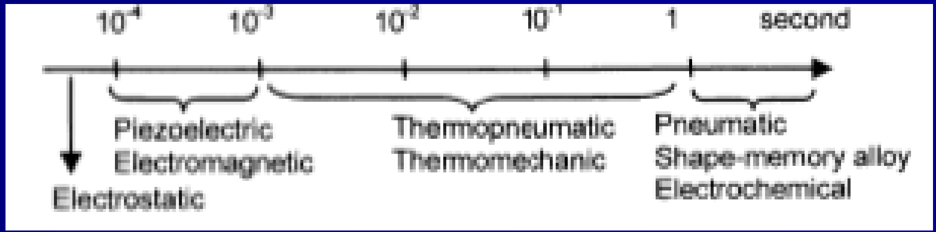
Active Microvalves

- **Pneumatic**
- **Thermopneumatic**
- **Thermomechanical**
- **Piezoelectric**
- **Electrostatic**
- **Electromagnetic**
- **Electrochemical**
- **Capillary forcel**

Valve specification:

- leakage → $L_{valve} = \frac{\dot{Q}_{closed}}{\dot{Q}_{open}}$
- valve capacity → $C_{valve} = \frac{\dot{Q}_{max}}{\sqrt{\Delta p_{max} / (\rho g)}}$
- power consumption – total power consumption in active state
- closing force (pressure range) – pressure generated by the valve
- temperature range
- responce time
- reliability
- biocompartibility
- chemical compartibility





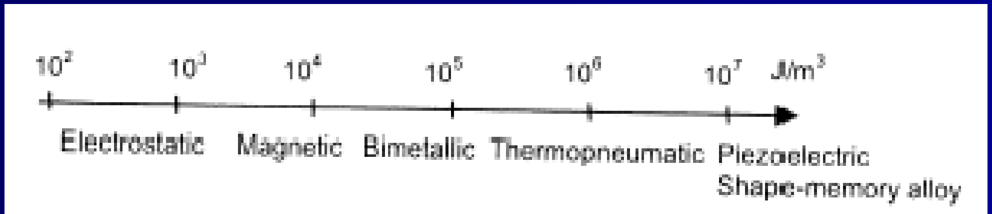
Design considerations

- Specification required
- Materials to be used
- Cost
- Suitable type of actuators
- Optimal valve spring and valve seat

Design consideration: Actuators

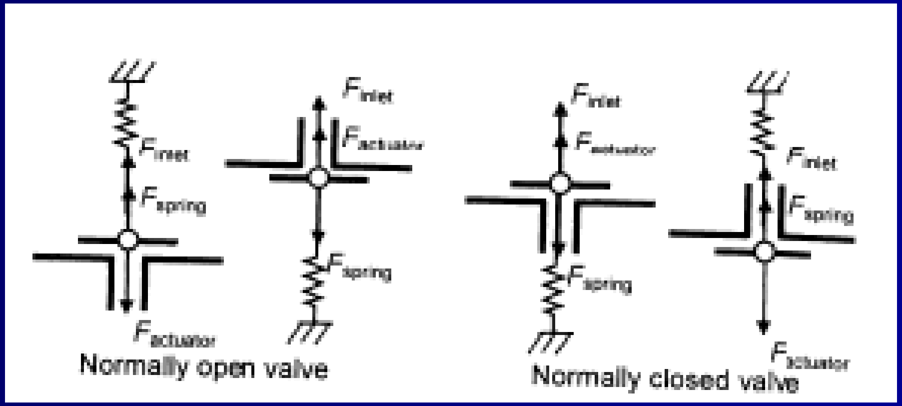
- Moving function (enough force, displacement and controllability)
- Holding function (should keep valve in a set position)
- Dynamic function (required responce time)

Energy density: $E_a^{'} = \frac{F_a s_a}{V_a}$



Design consideration: Valve spring

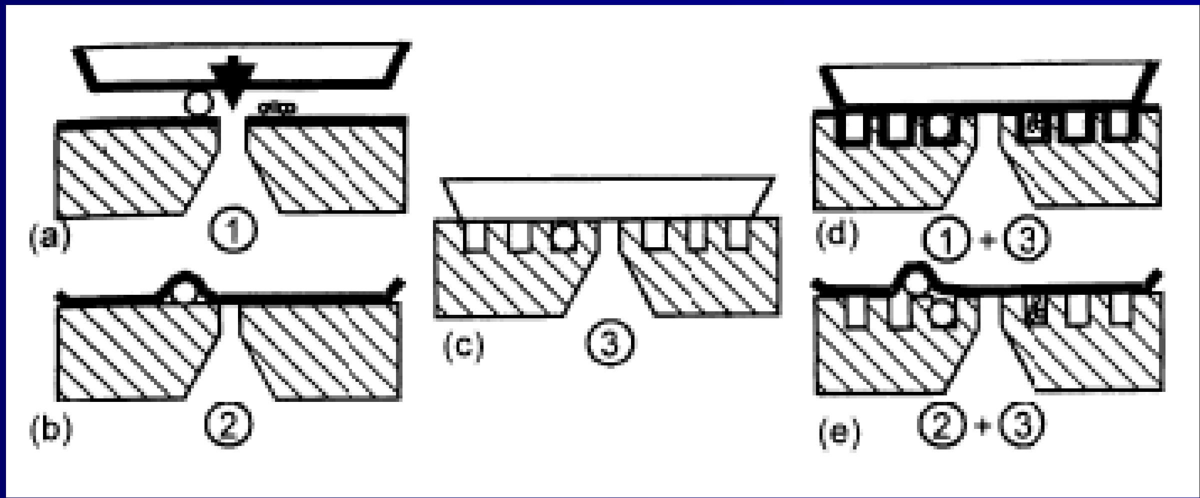
- For normally closed valves (NC) – large spring constant to resist the pressure
- For normally open valves (NO) – soft spring constant, optimized for actuator closing force



Design consideration: Valve seat

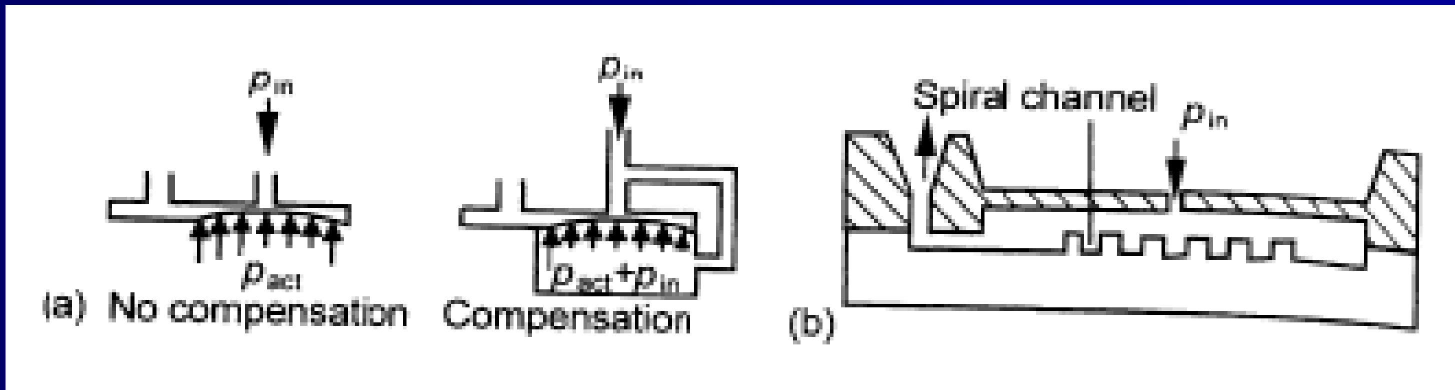
Main requirements:

- Zero leakage
- Resistance against particles trapped



Design consideration: Pressure compensation

Aim: Maintain closing force when the inlet pressure vary

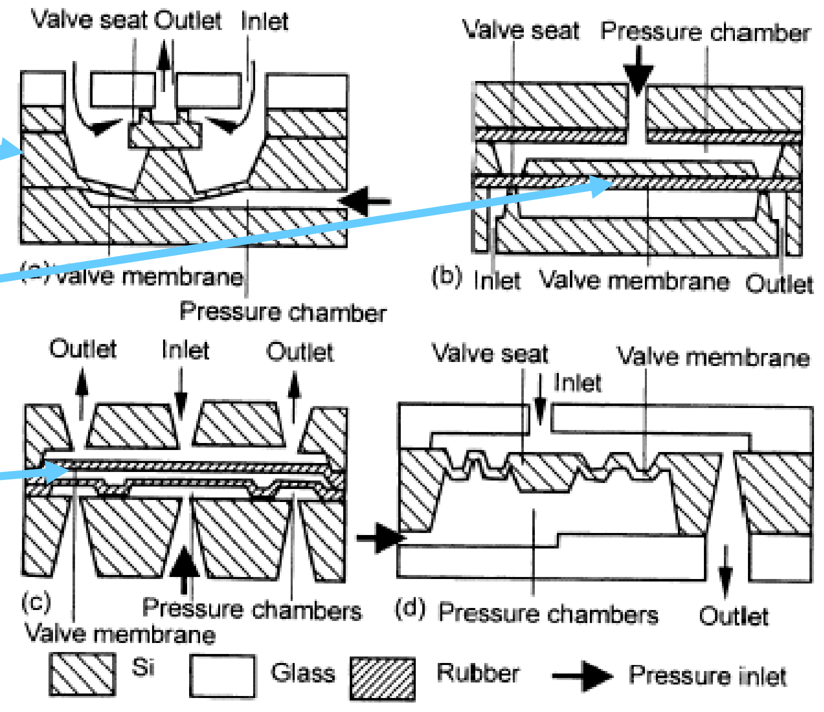


Passive flow controller

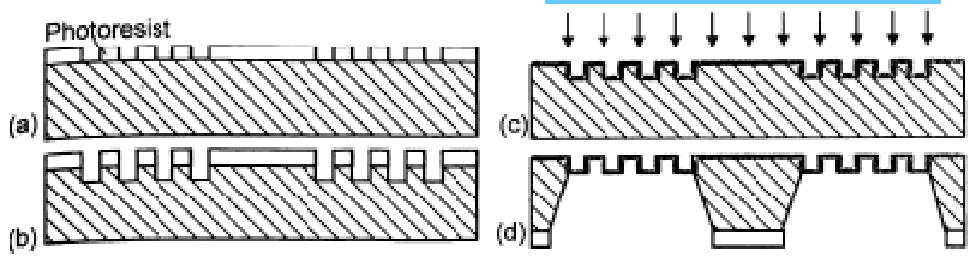
Stack of 3 directly bonded Si wafers, Si membrane 25 um thick

Silicon rubber membrane 25 um thick prepared by spin-coating, hole drilled with laser

Silicon rubber membrane 30 um thick prepared by surface micromachinig, photoresist used as a sacrificial layer.



Boron implantation



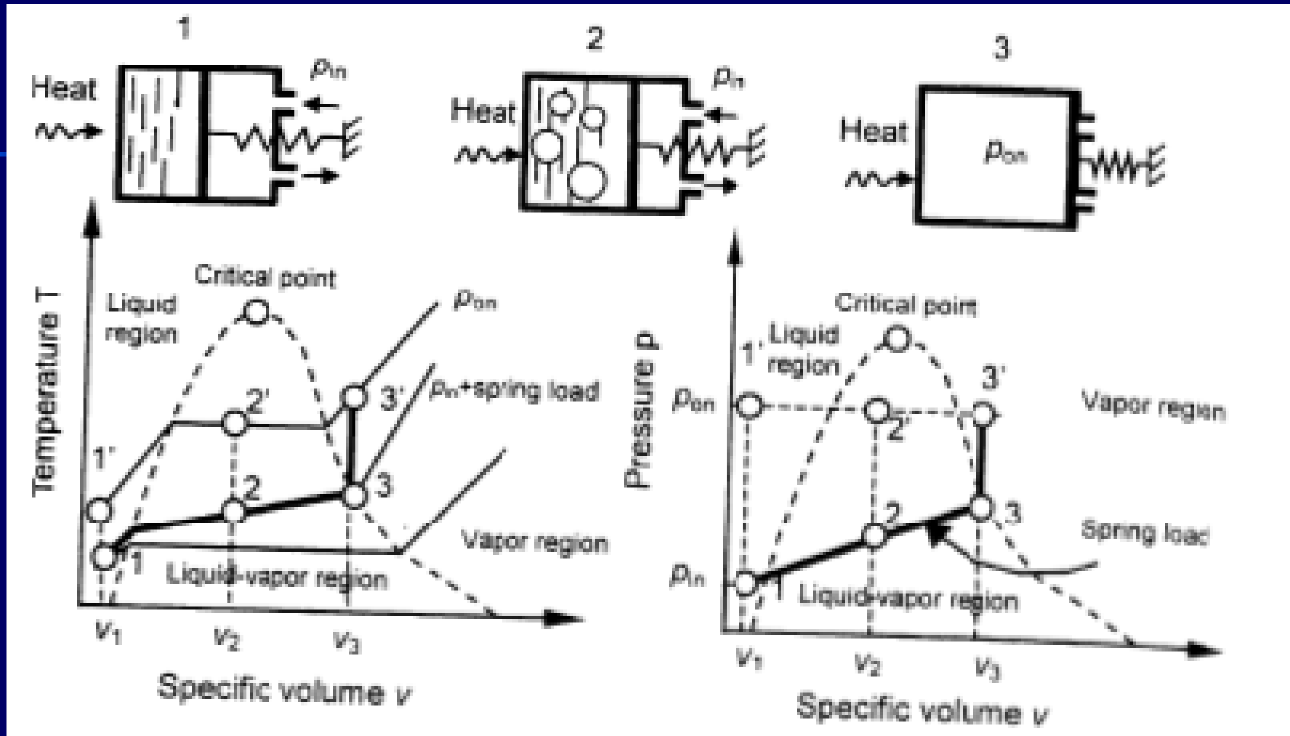
Typical parameters of pneumatic valves:

Typical Parameters of Pneumatic Valves (L_{valve}: Leakage ratio)

Refs.	*Type*	*Size (mm×mm)*	*Q_{max} (ml/min)*	*$p_{max}/p_{actuator}$ (kPa)*	*L_{valve}*	*Material*	*Technology*
[3]	NC	15×15	120 air	241/69	>300	Glass, silicon	Bulk
[4]	NO	0.225×0.225	0. 26 water	100/50	10,000	Rubber, silicon	Bulk
[5]	NC	20×20	35 N_2	65/12	35	Glass, silicon	Bulk
[6]	NO	8.5×4.2	0.5 water	60/10	10,000	Rubber, silicon	Bulk
[7]	NO	10×10	5 N_2	107/275	100,000	Glass, silicon	Bulk

Thermopneumatic valves

- Relies on the change in volume of sealed liquid or solid under thermal loading. Usually utilize solid/liquid and liquid/gas phase transition for maximum performance



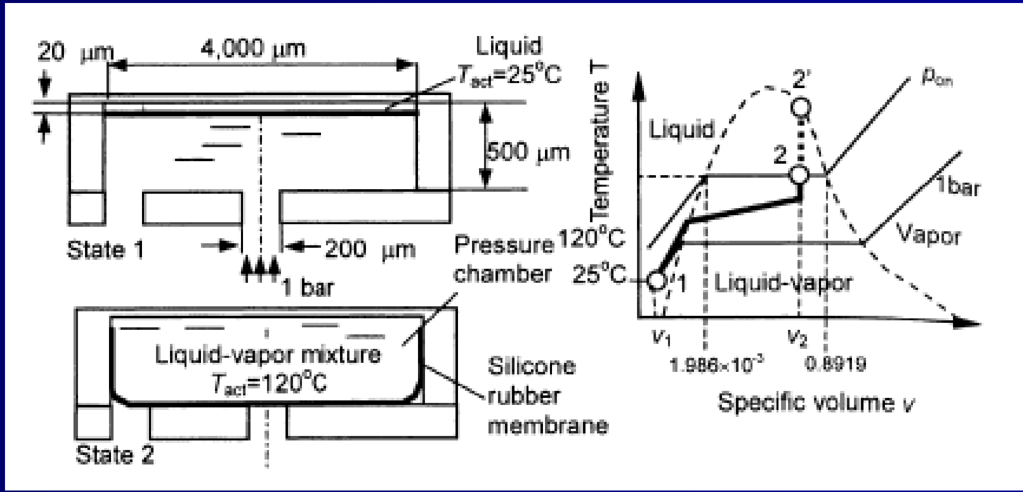
1
2
3
Heat
Heat
Heat
p_{in}
p_{in}
p_{on}
Critical point
Liquid region
p_{on}
2'
3'
p_{in}+spring load
1'
1
2
3
Vapor region
Liquid-vapor region
Temperature T
v_1
v_2
v_3
Specific volume v
Critical point
Liquid region
1'
3'
Pressure p
p_{on}
2'
Vapor region
2
3
Spring load
p_{in}
1
Liquid-vapor region
v_1
v_2
v_3
Specific volume v

Example

- Thermopneumatic valve with air. Height of the expansion cylinder 500um.
- Assuming the volume constant:

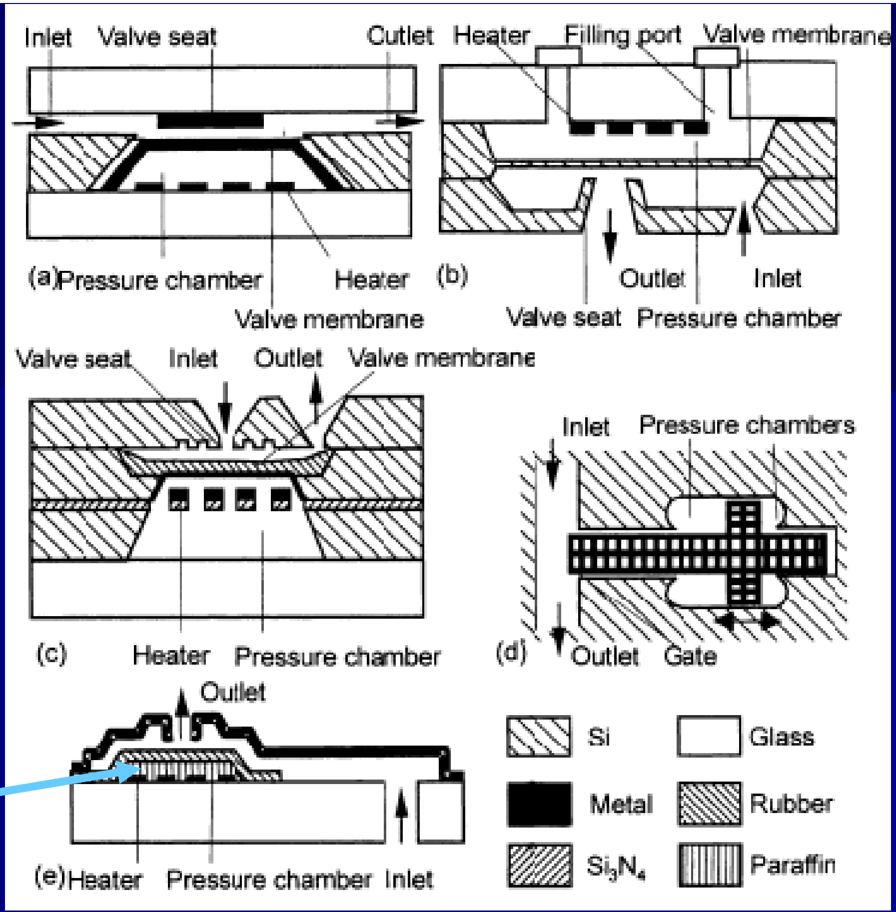
$$\frac{T_1}{T_2} = \frac{p_1}{p_2} \rightarrow T_2 = T_1 \frac{p_2}{p_1} = 300 \frac{109.67}{100} = 329K = 56C$$

Example



20 μm
4,000 μm
Liquid
T_{act}=25°C
500 μm
State 1
200 μm
1 bar
Pressure chamber
Liquid-vapor mixture
T_{act}=120°C
Silicone rubber membrane
State 2
Temperature T
Liquid
2'
2
p_{on}
1bar
Vapor
120°C
25°C
1
Liquid-vapor
v_1
v_2
1.986×10^{-3}
0.8919
Specific volume v

Design examples



Heater is placed on SiN membrane, 50um
Silicon rubber used for membrane

9um paraffin used as actuation material
2um deflection with 50mW heating

Table 6.2

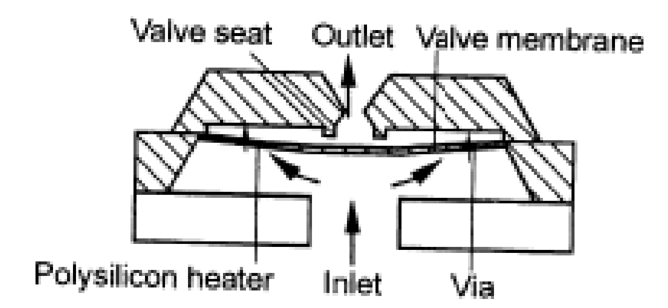
Typical Parameters of Thermopneumatic Valves

Refs.	*Type*	*Size (mm×mm)*	Q_{max} *(ml/min)*	P_{max} *(kPa)*	L_{valve}	*P (mW)*	*Membrane Material*	*Actuation Fluid*
[8]	NO	5×5	1,500 air	700	-	200	Aluminum	Methyl chloride
[9-10]	NO	8×6	10 N_2	1.3	33,000	3,500	Silicon	FC
[11,12]	NO	8×8	1,800 N_2	227	-	100	Rubber	FC
[13]	NO	0.1×0.8	0.24 water	1.4	1.15	100	-	Water
[14-15]	NO	8.5×4.2	2 N_2	100	14	50	Rubber	Paraffin

Thermomechanical valves

- Solid expansion
- Bimetallic
- Shape-memory alloys

Solid expansion valves



- Generated force: $F \propto \gamma \Delta T$
  where γ is the thermal expansion coefficient

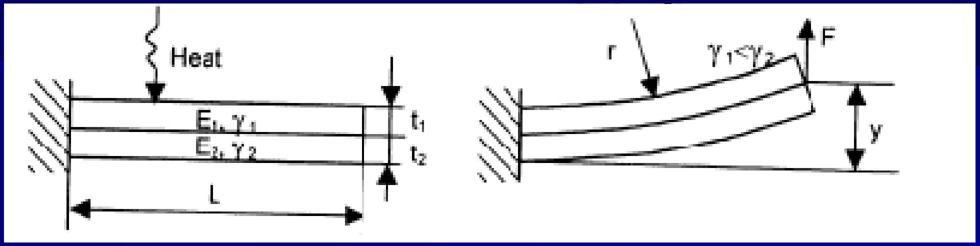
Thermal Properties of Some Materials at 300K (After [17, 28])

Material	*Density (kg/m³)*	*Heat Capacity (J/kgK)*	*Thermal Conductivity (W/mK)*	*Thermal Expansion Coefficient ($10^{-6}K^{-1}$)*
Silicon	2,330	710	156	2.3
Silicon oxide	2,660	750	1.2	0.3
Silicon nitride	3,100	750	19	2.8
Aluminum	2,700	920	230	23
Copper	8,900	390	390	17
Gold	19,300	125	314	15
Nickel	8,900	450	70	14
Chrome	6,900	440	95	6.6
Platinum	21,500	133	70	9
Parylene-N	1,110	837.4	0.12	69
Parylene-C	1,290	711.8	0.082	35
Parylene-D	1,418	-	-	30-80

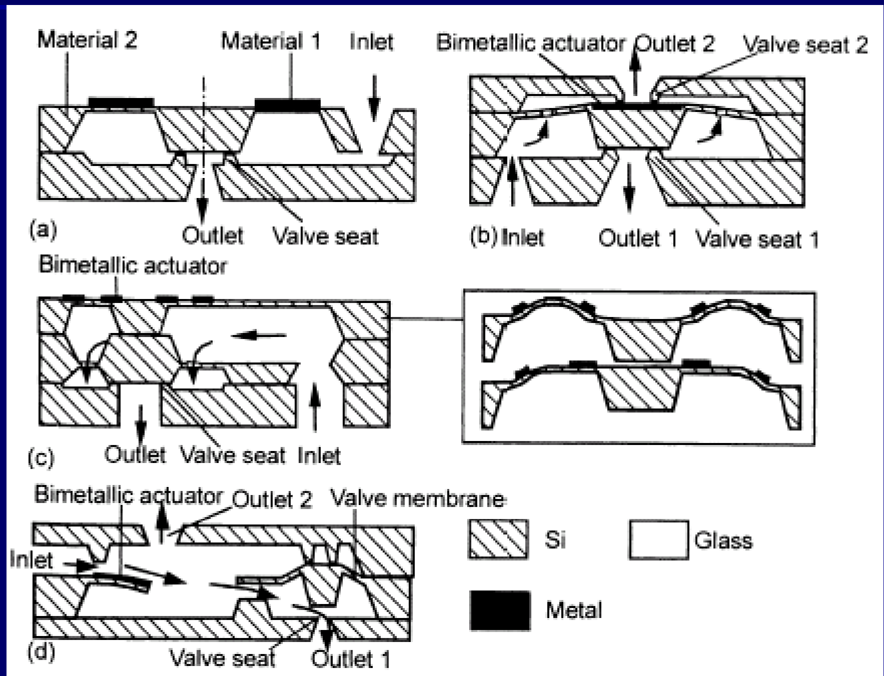
Bimetallic valves

- Uses difference in thermal expansion coefficient of two metals

$$F \propto (\gamma_2 - \gamma_1)\Delta T$$



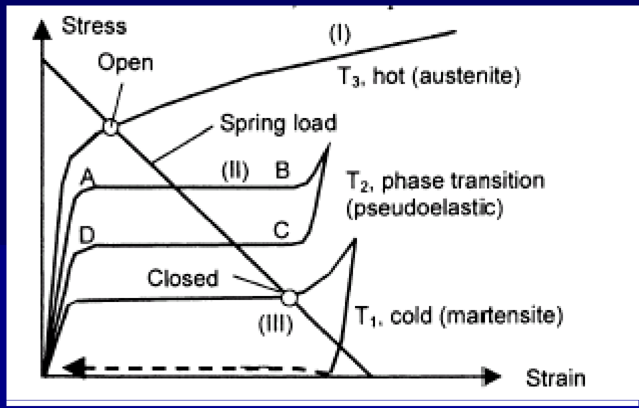
Bimetallic valves: Design examples

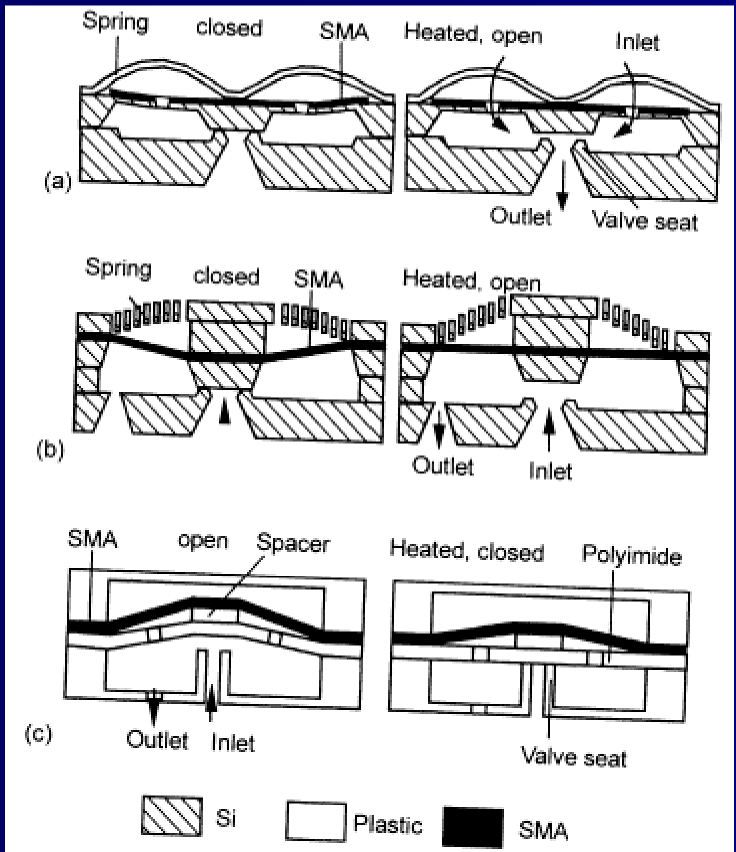


Shape memory Alloy valves

- Shape memory alloys (SMA) are materials that have property to return to their original undeformed shape upon a change of temperature
- Advantages: high force and large stroke
- Disadvantages: low efficiency, low frequency (bandwidth)

Alloy	*Composition*	*Transf. Temp. Range (°C)*	*Transf. Hysteresis (°C)*
-Cd	44/49 at.% Cd	-190 to -50	15
-Cd	46.5/50 at.% Cd	30 to 100	15
-Al-Ni	14/14.5 wt.% Al 3/4.5 wt.% Ni	-140 to 100	35
-Sn	approx. 15 at.% Sn	-120 to 30	
-Zn	38.5/41.5 wt.% Zn	-180 to -10	10
-Ti	18/23 at.% Ti	60 to 100	4
Ni-Al	36/38 at.% Al	-180 to 100	10
Ni-Ti	49/51 at.% Ni	-50 to 110	30
Fe-Pt	approx. 25 at.% Pt	approx.-130	4
Mn-Cu	5/35 at.% Cu	-250 to 180	25
Fe-Mn-Si	32 wt.% Mn, 6 wt.% Si	-200 to 150	100

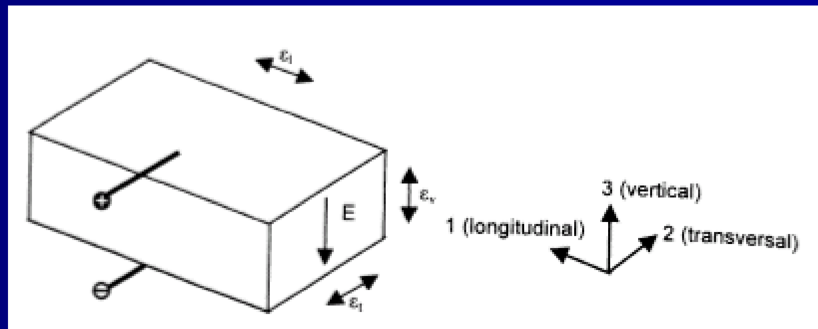

Stress
(I)
Open
T₃, hot (austenite)
Spring load
(II)
A
B
T₂, phase transition
(pseudoelastic)
D
C
Closed
(III)
T₁, cold (martensite)
Strain


Spring
closed
SMA
Heated, open
Inlet
(a)
Outlet
Valve seat
Spring
closed
SMA
Heated, open
(b)
Outlet
Inlet
SMA
open
Spacer
Heated, closed
Polyimide
(c)
Outlet
Inlet
Valve seat
Si
Plastic
SMA

Piezolelectric valves

- generate small strain (0.1%) and high stresses (MPa), therefore suitable for applications with high force and low displacement

$$\varphi_l = \varphi_t = d_{31} E_{el}$$
$$\varphi_v = d_{33} E_{el}$$

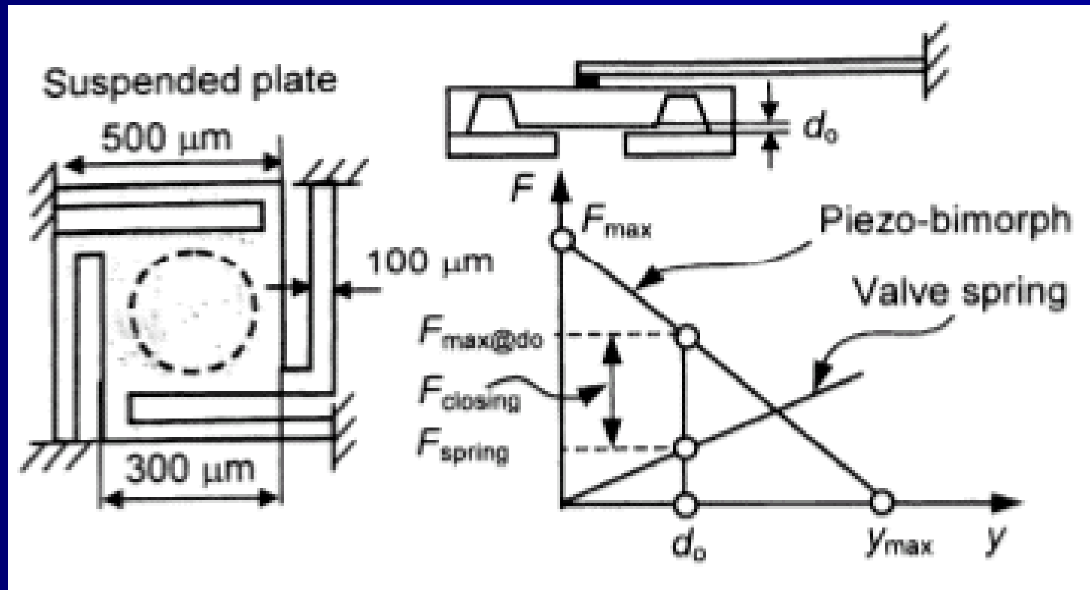


Properties of Common Piezoelectric Materials (After [17, 28])

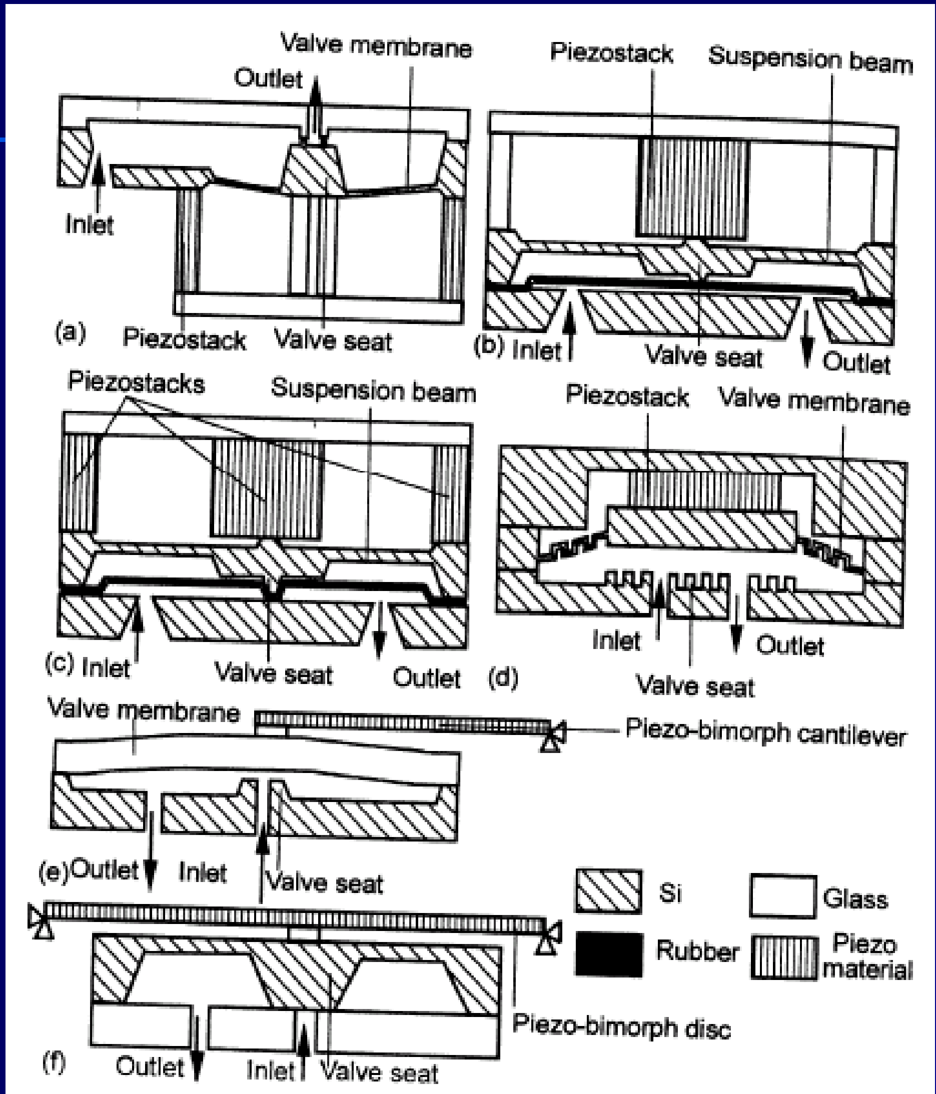
Material	d_{31} (10^{-12} C/N)	d_{33} (10^{-12} C/N)	Relative Permittivity ε_r
PZT	-60 … -270	380 … 590	1,700
ZnO	-5	12.4	1,400
PVDF	6-10	13-22	12
$BaTiO_3$	78	190	1,700
$LiNbO_3$	-0.85	6	-

Example:

Dimension (mm)	Voltage (V)	C (nF)	Y_{max} (μm)	F_{max} (N)	Frequency (Hz)
25×7.5×0.4	±70	20	±200	0.15	300



Design examples

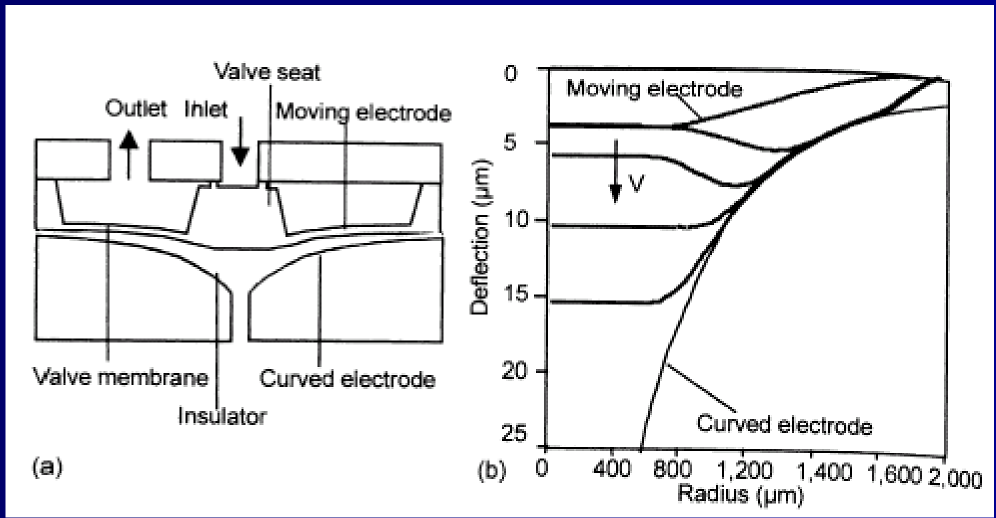


Electrostatic valve

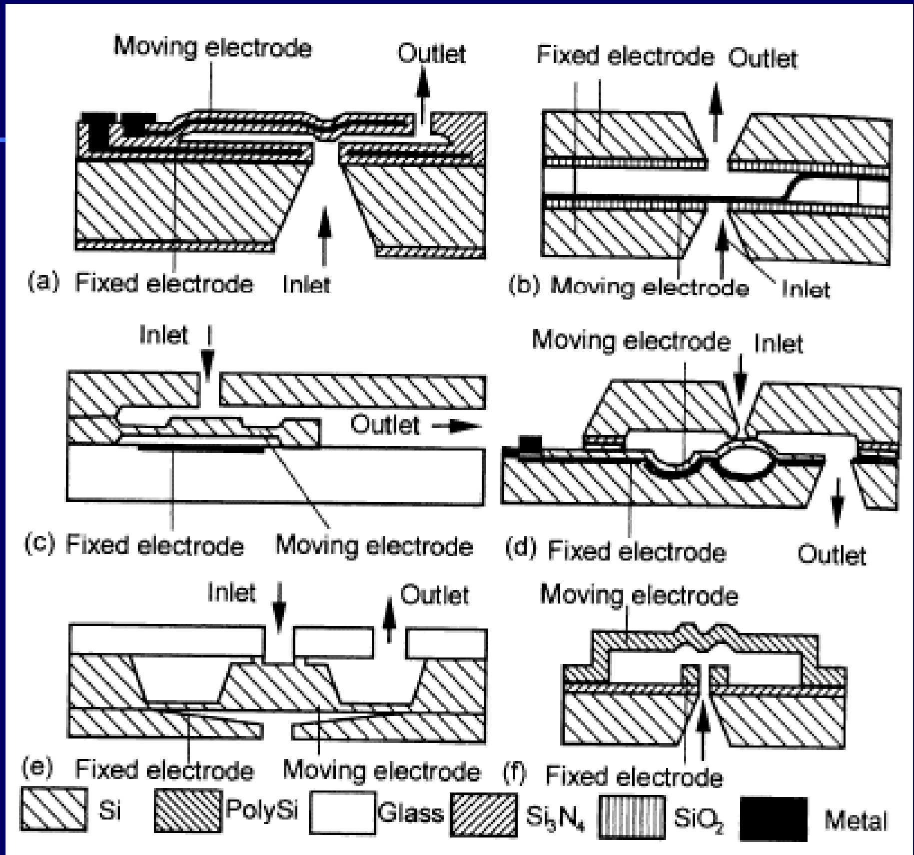
- Based on attractive force between two oppositely charged plates

$$F = \frac{1}{2}\varepsilon_r \varepsilon_0 A\left(\frac{V}{d}\right)^2, \varepsilon_0 = 8.854 * 10^{-12} F/m$$

- Advantages: fast responce
- Disadvantages: high voltage and small discplacement



Design examples



Electromagnetic valves

- Uses solenoid actuator with a magnetic core and a coil

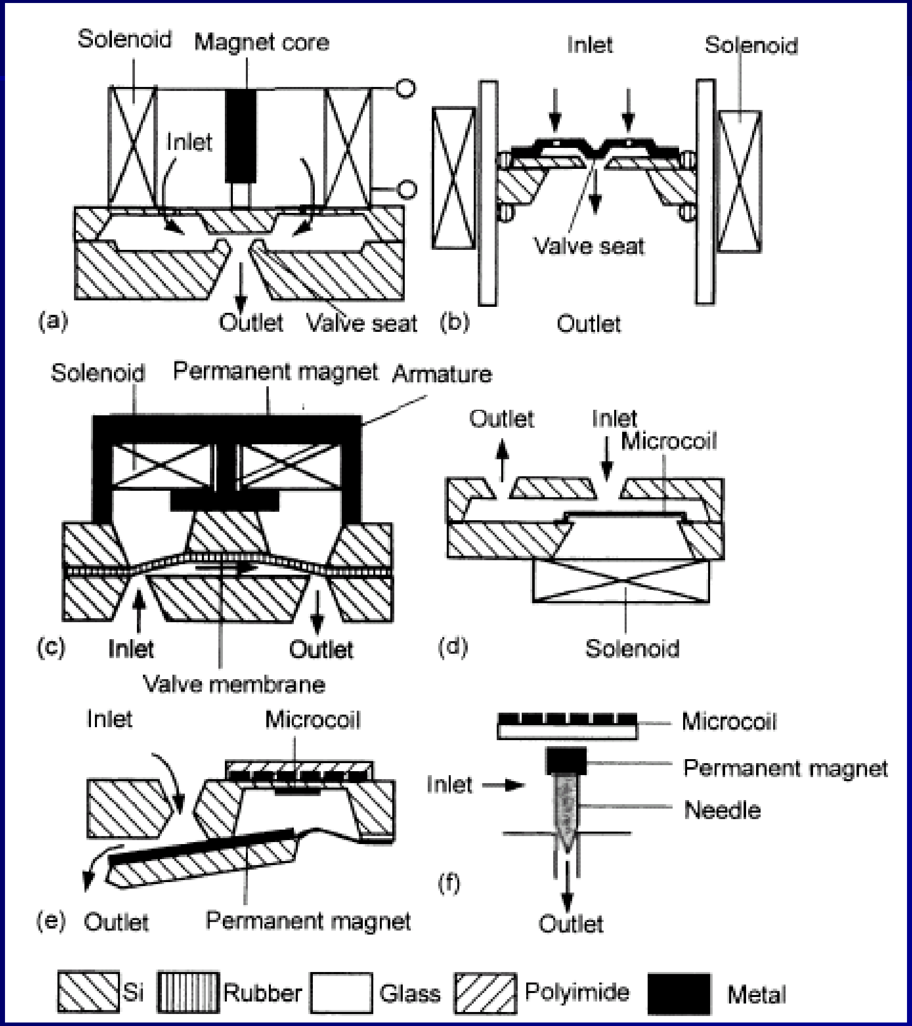
$$F = M_m \int \frac{dB}{dz} dV \; .$$

- Advantage: large deflection
- Disadvantage: low efficiency

Magnetization of Common Materials at $B = 0$ T (After [17])

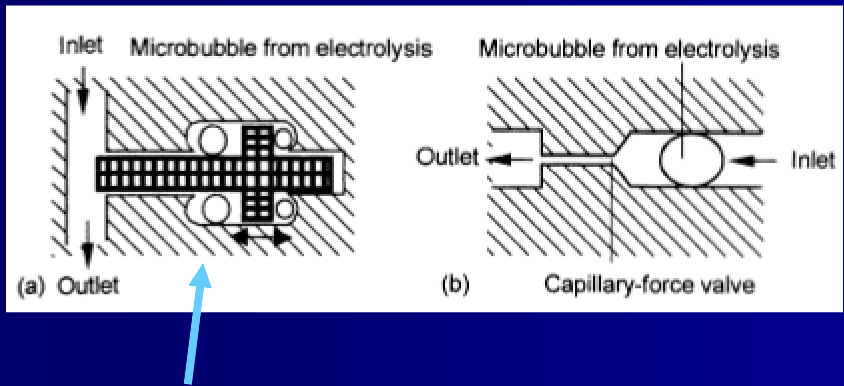
Material	*Magnetization M_m (A/m)*	*Note*
Nickel	3,000	Electroplated, annealed
Iron	320	—
Fe-Ni78	<80	Electroplated, annealed
Fe-Ta-Ni	46	Sputtered
Fe-Al-Si	40	Sputtered

Design examples



Electrochemical valves

- Actuated by a bubble created by water electrolysis



Consumes 4.3uW (10000 smaller that thermopneumatic !!!)
2.5 V operational voltage

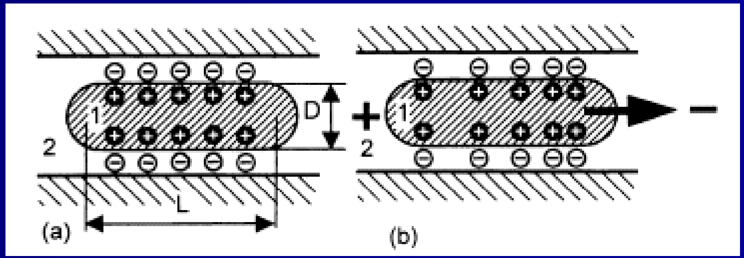
Capillary force valves

- Electrocapillary

Capacity of double layer.

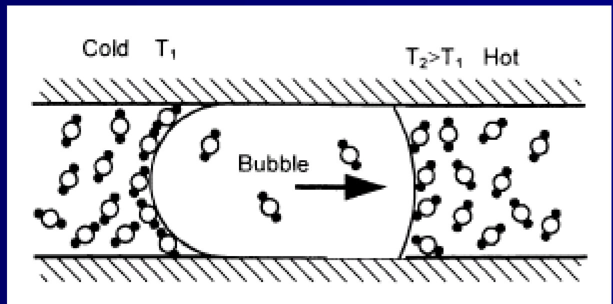
$$\sigma = \sigma_0 - \frac{C}{2}(V - V_0)^2$$

Maximum value of surface tension at V_0.

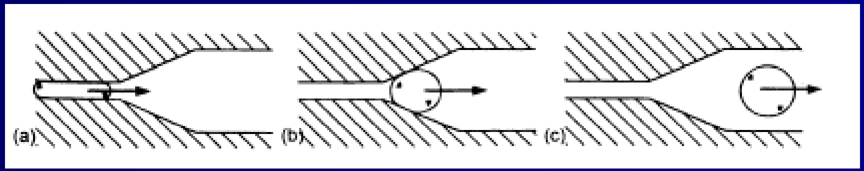


Thermocapillary effect

- Caused by temperature dependence of surface tension: viscosity and surface tension both drop with the temperature increase



Passive capillary effect



Example: a bubble valve is designed with a vapour bubble between channel sections 50um and 200um sections. What pressure can it withstand?

$\sigma_w = 72*10^{-3}$ N/m

$$\Delta p = 2\sigma\left(\frac{1}{r_1} - \frac{1}{r_2}\right) = 2 \times 72 \times 10^{-3}\left(\frac{1}{50 \times 10^{-6}} - \frac{1}{200 \times 10^{-6}}\right) = 2{,}160 \text{ Pa} = 21.6 \text{ mbar}$$

Micropumps

- micropumps → mechanical
- micropumps → Non-mechanical

Mechanical Pumping Principles

Displacement Pumps	*Dynamic Pumps*
• Check-valve pumps	• Ultrasonic pumps
• Peristaltic pumps	• Centrifugal pumps
• Valve-less rectification pumps	
• Rotary pumps	

Nonmechanical Pumping Principles

	Pressure Gradient	*Concentration Gradient*	*Electrical Potential Gradient*	*Magnetic Potential*
Fluid flow	Surface tension driven flow (electrowetting, Marangoni-effect, surface modification)	Osmosis (semipermeable membrane, surfactants)	Electro-osmosis (electrolyte) Electrohydrodynamic (dielectric fluid)	Ferrofluidic
Solute flux	Ultrafiltration	Diffusion	Electrophoresis Dielectrophoresis	Magneto-hydrodynamic flow

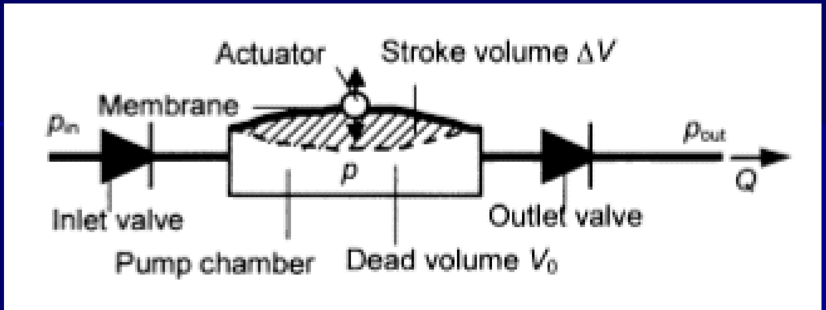
Mechanical pumps

- Require an electromechanical actuator, either external or integrated
- External actuators: drawback: large size, advantages: large force and displacement.
- Integrated actuators: fast responce and reliability

Parameters of micropumps

- Maximum flow rate (determined at 0 back pressure)
- Maximum back pressure (at which flow becomes 0), also described as pump head
- Pump efficiency

Check-valve pump



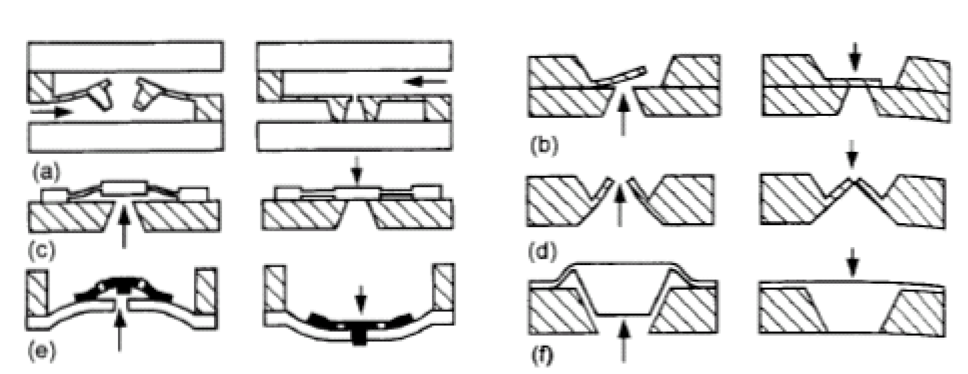
- Function under conditions of small compression ration and high pump pressure
  - Compression ratio $\psi = \frac{\Delta V}{V_0}$
  - High pump pressure $\begin{cases} |p - p_{out}| > \Delta p_{crit} \\ |p - p_{in}| > \Delta p_{crit} \end{cases}$

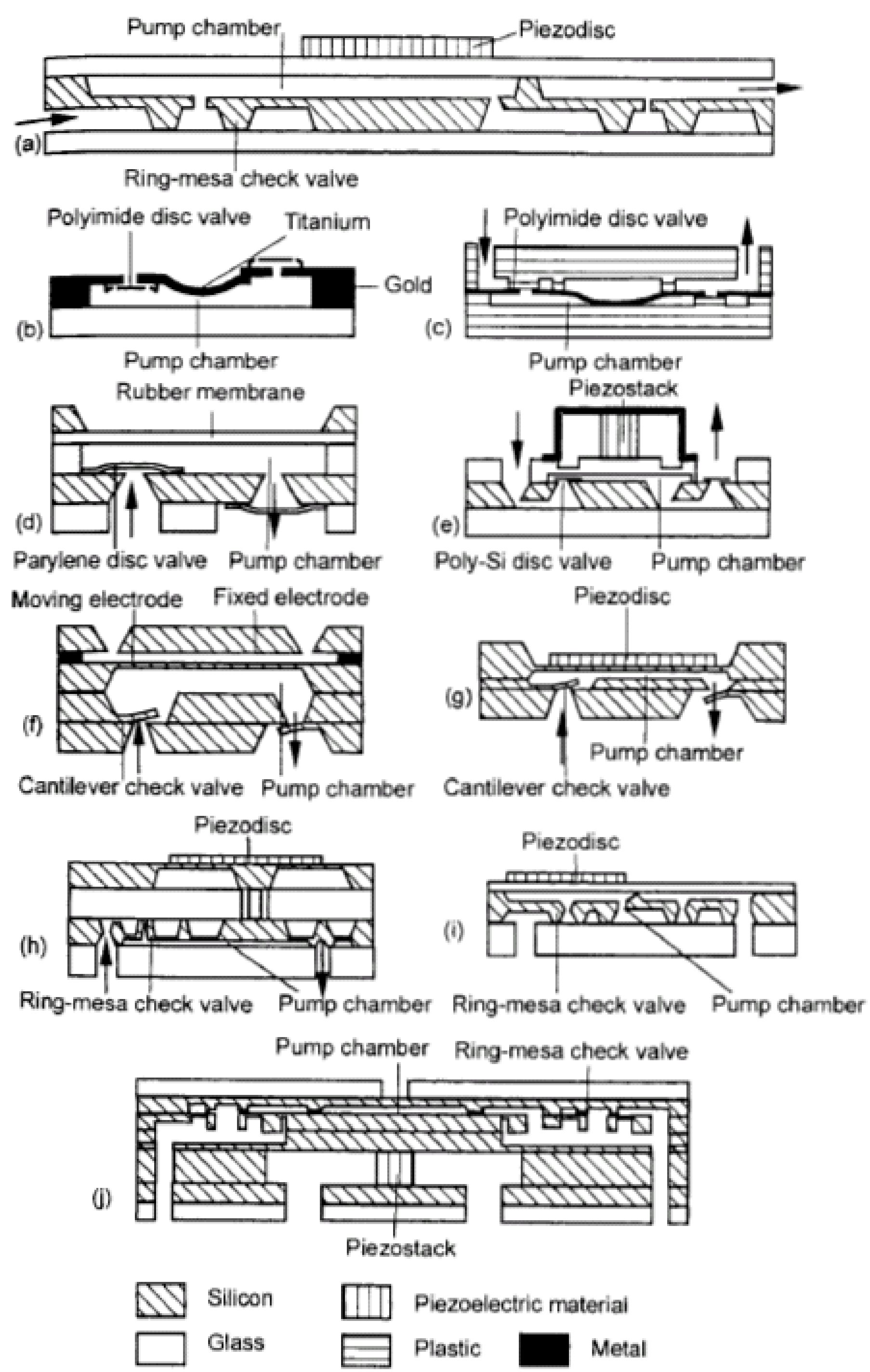
<u>Design rules</u>

- Minimize critical pressure
- Maximize stroke volume
- Minimize the dead volume
- Maximize the pump pressure using large force actuators

Design examples

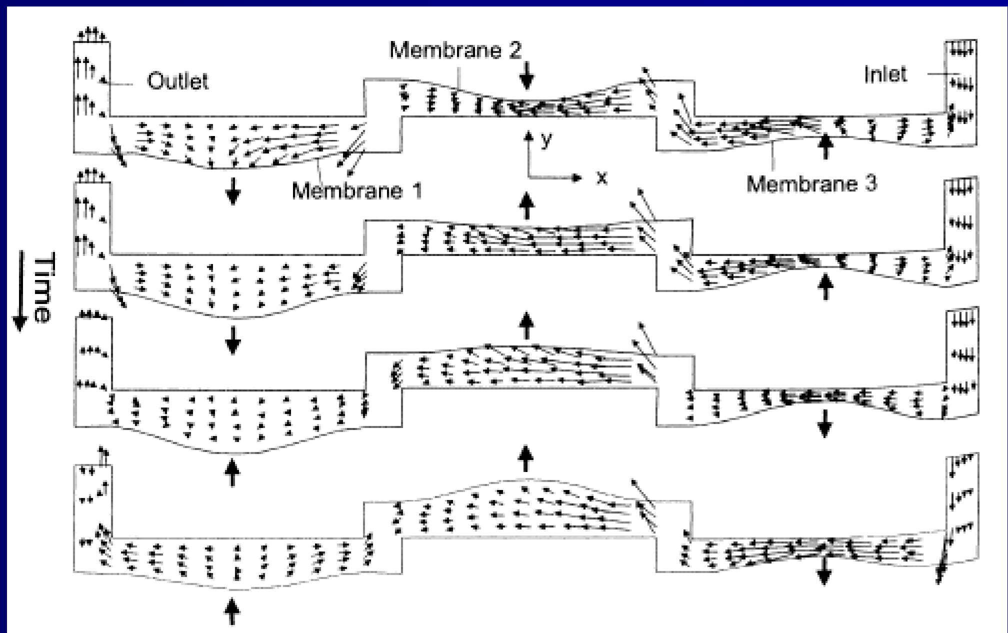
- Typical micro check valves




Pump chamber
Piezodisc
(a)
Ring-mesa check valve
Polyimide disc valve
Titanium
Gold
(b)
Pump chamber
Polyimide disc valve
(c)
Pump chamber
Rubber membrane
(d)
Parylene disc valve
Pump chamber
Piezostack
(e)
Poly-Si disc valve
Pump chamber
Moving electrode
Fixed electrode
(f)
Cantilever check valve
Pump chamber
Piezodisc
(g)
Pump chamber
Cantilever check valve
Piezodisc
(h)
Ring-mesa check valve
Pump chamber
Piezodisc
(i)
Ring-mesa check valve
Pump chamber
Pump chamber
Ring-mesa check valve
(j)
Piezostack
Silicon
Glass
Piezoelectric material
Plastic
Metal

Peristaltic pumps

- Do not require passive valves to set flow direction
- Require 3 or more chambers (actually just valves connected in seria)
- Drawbacks: leakage and small pressure difference, require a one check valve to prevent back flow
- Design rules: large stroke and large compression ratio.



Example

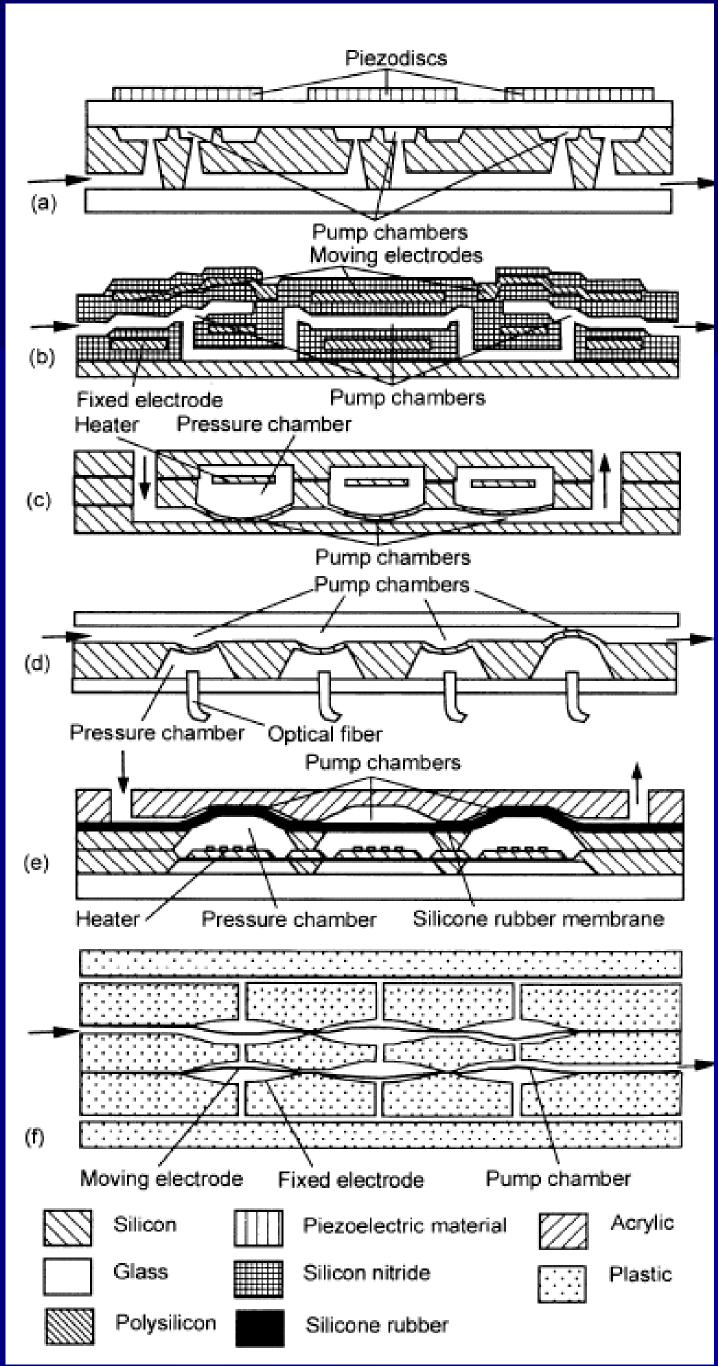
- A peristaltic pump has 3 chambers and 3 circular unimorph piezodiscs, membrane diameter 4mm, frequency 100Hz, maximum deflection 40um.

$$d(r) = d_{max}\left[1-\left(\frac{r}{R}\right)^2\right]^2$$

$$\Delta V = 2\times\int_0^{2\pi}\int_0^{R} d_{max}\left[1-\left(\frac{r}{R}\right)^2\right]^2 r\,dr\,d\varphi = \frac{2\pi}{3}d_{max}R^2 = \frac{2\pi}{3}4\times10^{-5}\times(2\times10^{-3})^2 = 3.35\times10^{-10}\,\mathrm{m}^3$$

$$Q = \Delta V f = 3.35\times10^{-10}\times100 = 3.35\times10^{-8}\,\frac{\mathrm{m}^3}{\mathrm{sec}} = 2\ \mathrm{ml/min}$$

Design examples



Valvless rectification pumps

- Diffusers/nozzles used instead of check valves for flow rectification

$$\Delta p = \xi \frac{pu^2}{2}$$

ξ - pressure loss coefficient

Fluidic diodicity:

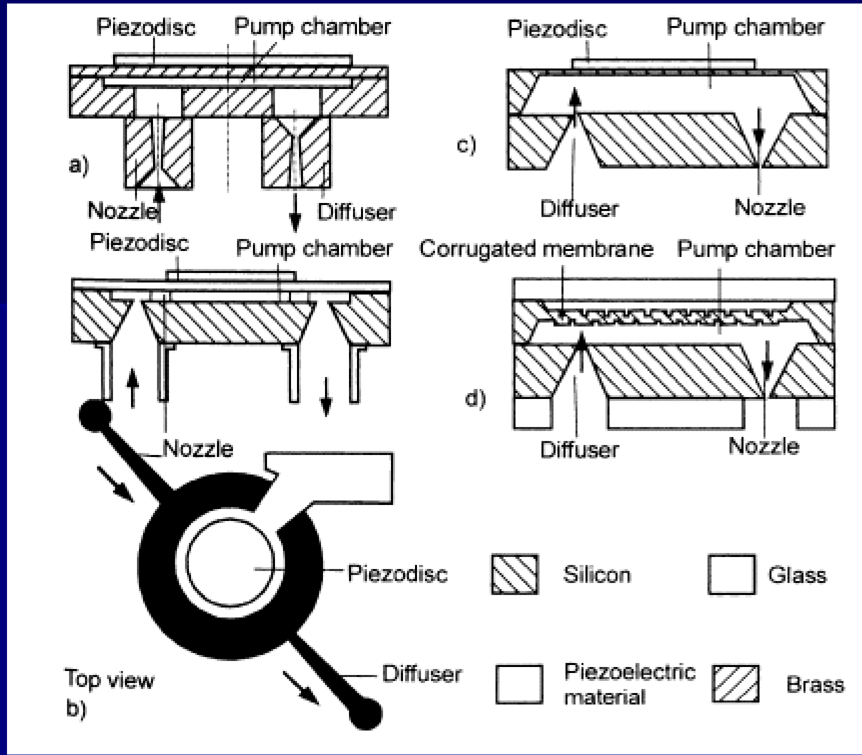
$$\eta_F = \frac{\xi_{negative}}{\xi_{positive}}$$

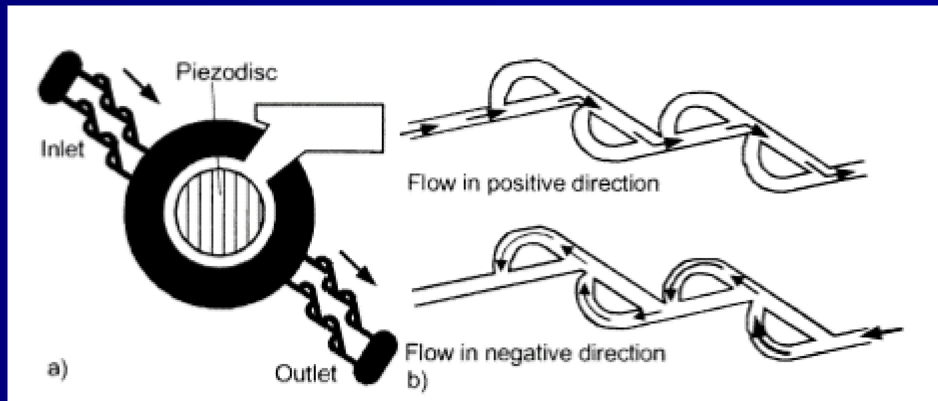
Flow rate:

$$\dot{Q} = 2\Delta V f \frac{\sqrt{\eta_F} - 1}{\sqrt{\eta_F} + 1}$$

χ - rectification efficiency

Design examples





Tesla pump:
χ=0.045, η=1.2

Micromixers

- mixing in microscale relies mainly on diffusion due to laminar flow at low Reynolds numbers

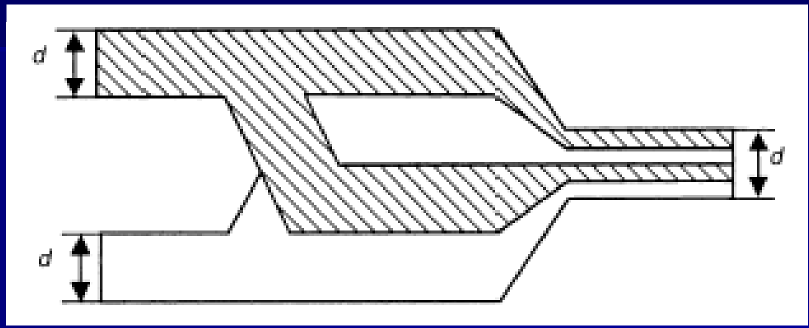
$$\tau = \frac{d^2}{2D}$$

Diffusion Coefficients in Water at 25°C [31]

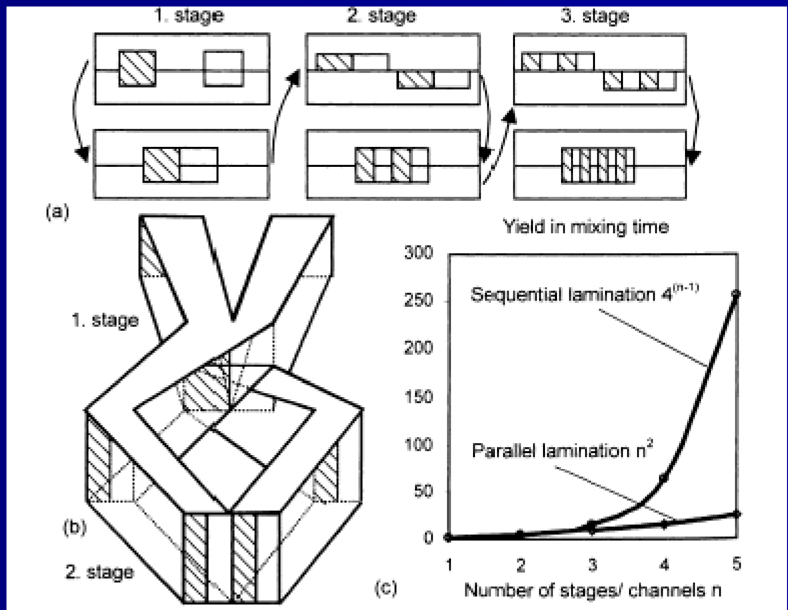
Solute	D (×10⁻⁵ cm²/s)	Solute	D (×10⁻⁵ cm²/s)
Air	2.00	Ammonia	1.64
CO_2	1.92	Benzene	1.02
Chlorine	1.25	Sulfuric acid	1.73
Ethane	1.20	Nitric acid	2.60
Ethylene	1.87	Acetylene	0.88
Hydrogen	4.50	Methanol	0.84
Methane	1.49	Ethanol	0.84
Nitrogen	1.88	Formic acid	1.50
Oxygen	2.10	Acetic acid	1.21
Propane	0.97	Propionic acid	1.06
Glycine	1.06	Benzoic acid	1.00
Valine	0.83	Acetone	1.16
Ovalbumin	0.078	Urease	0.035
Hemoglobin	0.069	Fibrinogen	0.020

Lamination in mixer

- parallel lamination

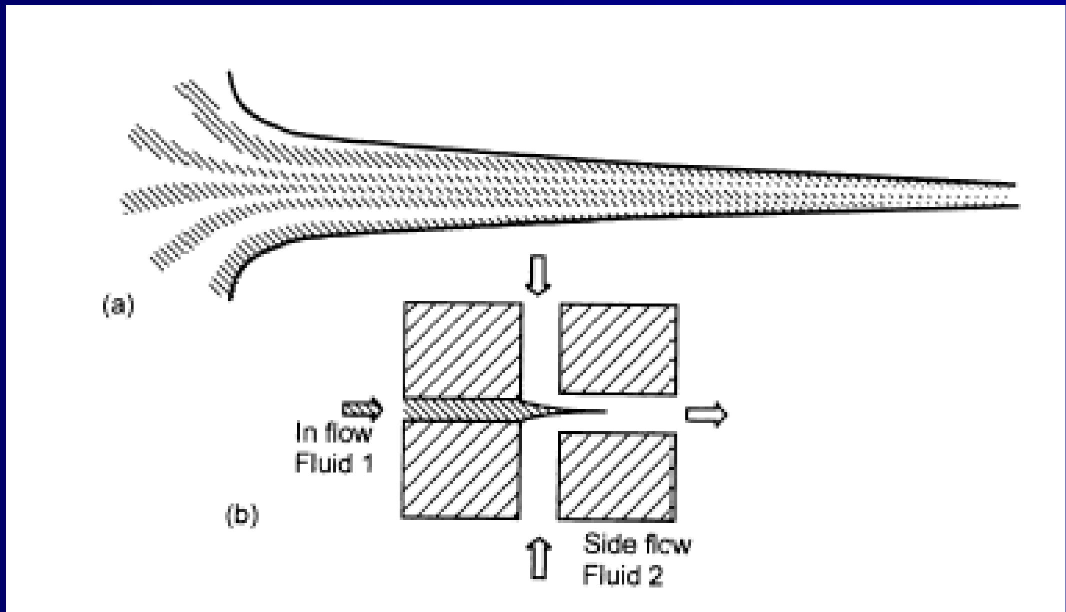


- sequential lamination



Focusing in mixer

- geometric and hydrodynamic focusing



Example:

- design a mixing for EtOH and water, if flow rates are 10ul/min and channel crossection is 100x100um
  $D=0.84 \times 10^{-5}$ cm^2/s
- how many laminates would be required to fit the design into 1mm length?