The Abdus Salam
International Centre for Theoretical Physics

United Nations
Educational, Scientific
and Cultural Organization

International Atomic
Energy Agency

SMR.1670 - 19

INTRODUCTION TO MICROFLUIDICS

8 - 26 August 2005

Wafer Bonding

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Wafer bonding

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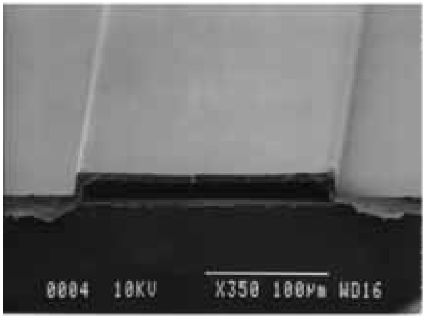
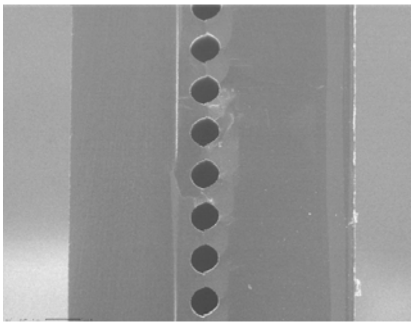
There are only 3 basic concepts for making a micro/nanochannel:

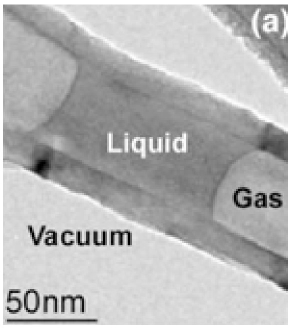
1. drilling
2. carving and <u>sealing</u>
3. forming



1. surface micromachined

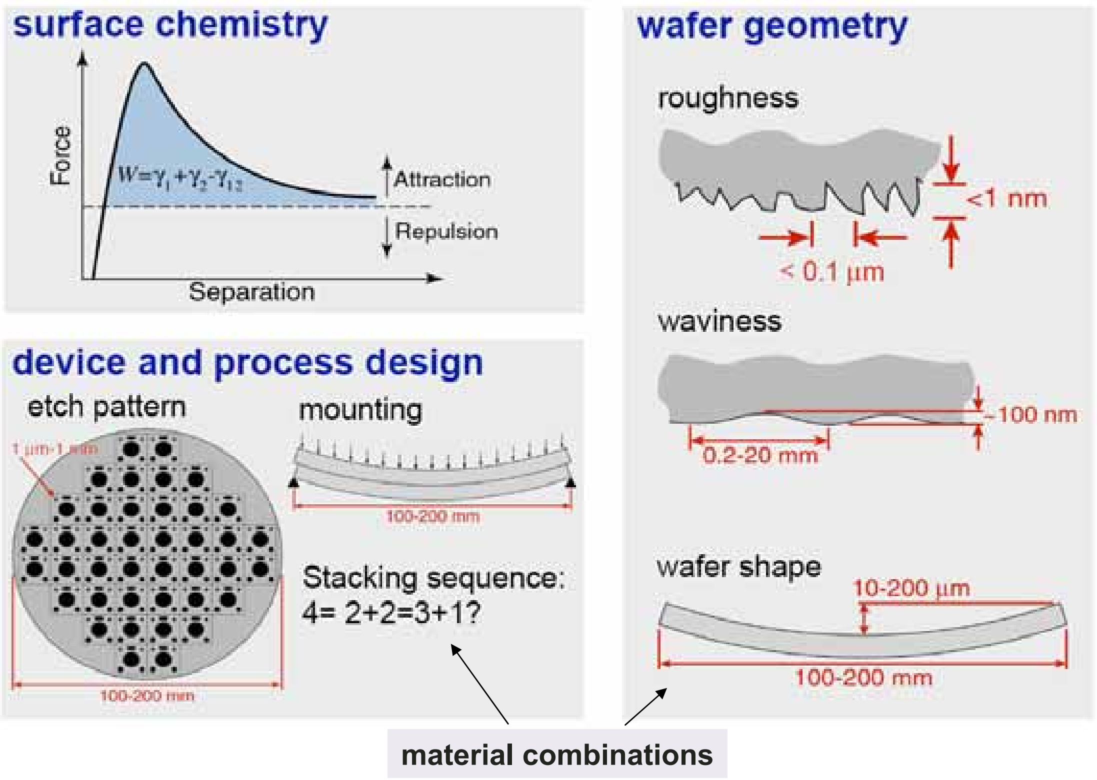


2. etched + waferbonded



3. grown carbon nanotube

Critical factors in wafer bonding



Source: M. Spearing, University of Southampton, UK

Wafer bonding (sealing at wafer scale)

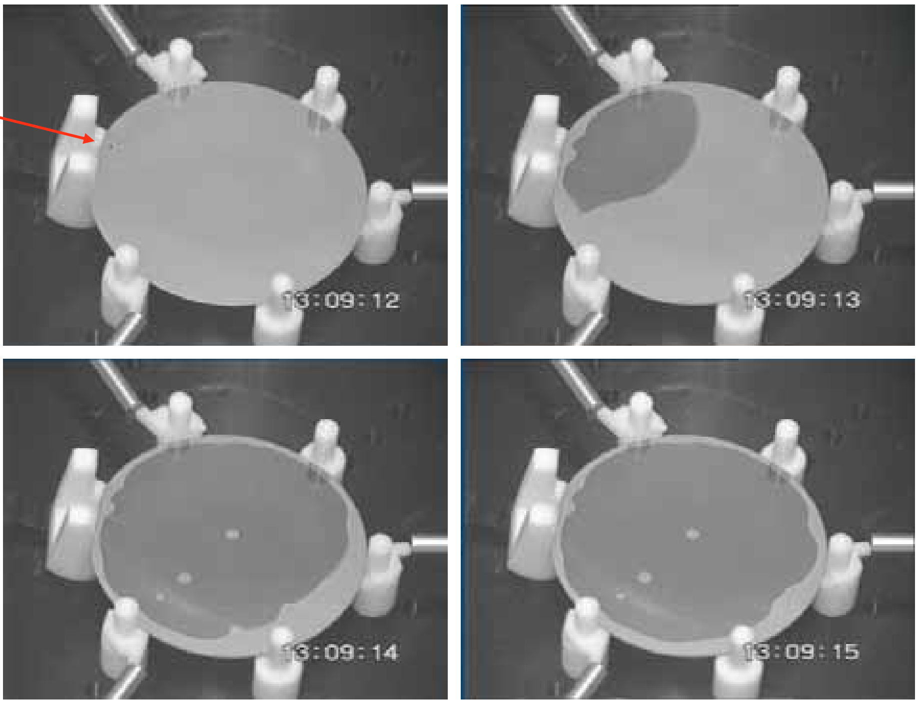
Main concepts:

1. Direct (fusion, thermocompression)

2. Anodic (electrostatic, field-assisted thermal)

3. Intermediate layer

Let's do a bonding experiment

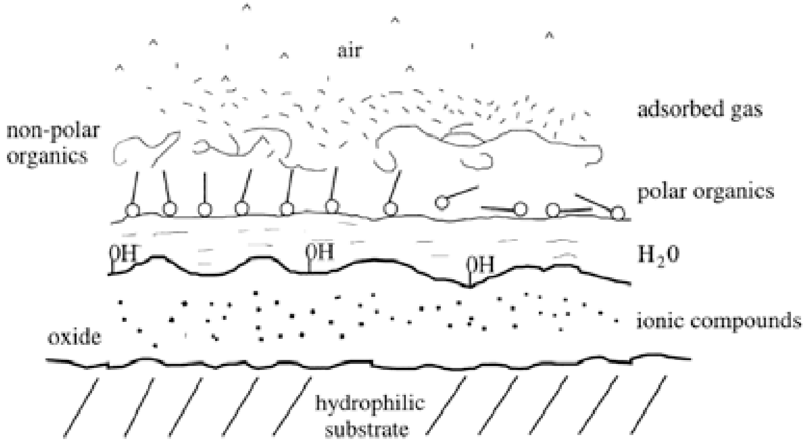


bonding starts here

Propagation of a "bonding wave" - takes seconds

Christiansen, MPI für Mikrostrukturphysik, Halle, D

Surface preparation



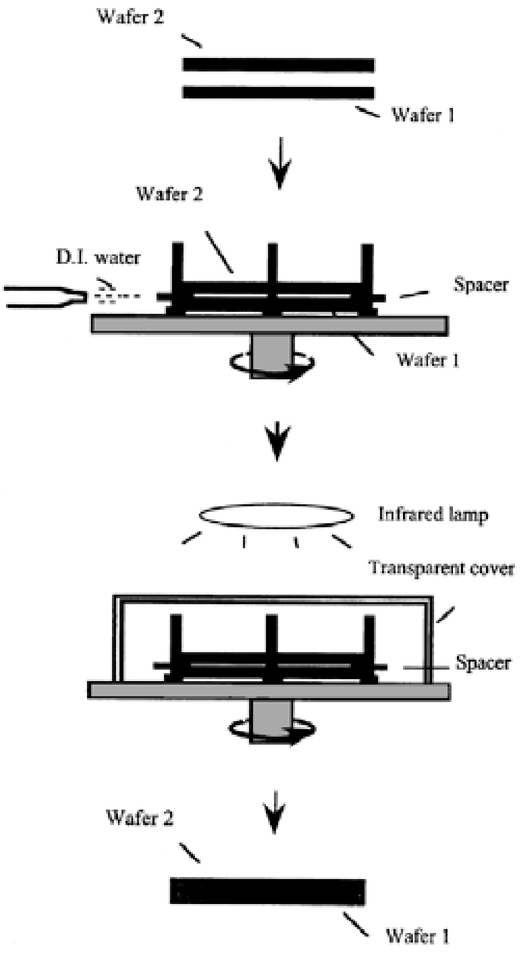
Schematic illustration of the adsorbate layers commonly expected on hydrophilic surfaces

Plössl e.a. Mat.Sci.Eng. R25, 1-88 (1999)

Surface cleaning procedures used before wafer bonding:

- RCA1 ($NH_4OH/H_2O_2/H_2O$ 1 : 1 : 5) + RCA 2 ($HCl/H_2O_2/H_2O$ 1 : 1 : 6)
- "Piranha" (H_2O_2/H_2SO_4)
- Conc. nitric acid

Mini clean rooms for wafer bonding



Tong e.a. Adv.Mat. 11, 1409-1425 (1999)

Bonding chemistry

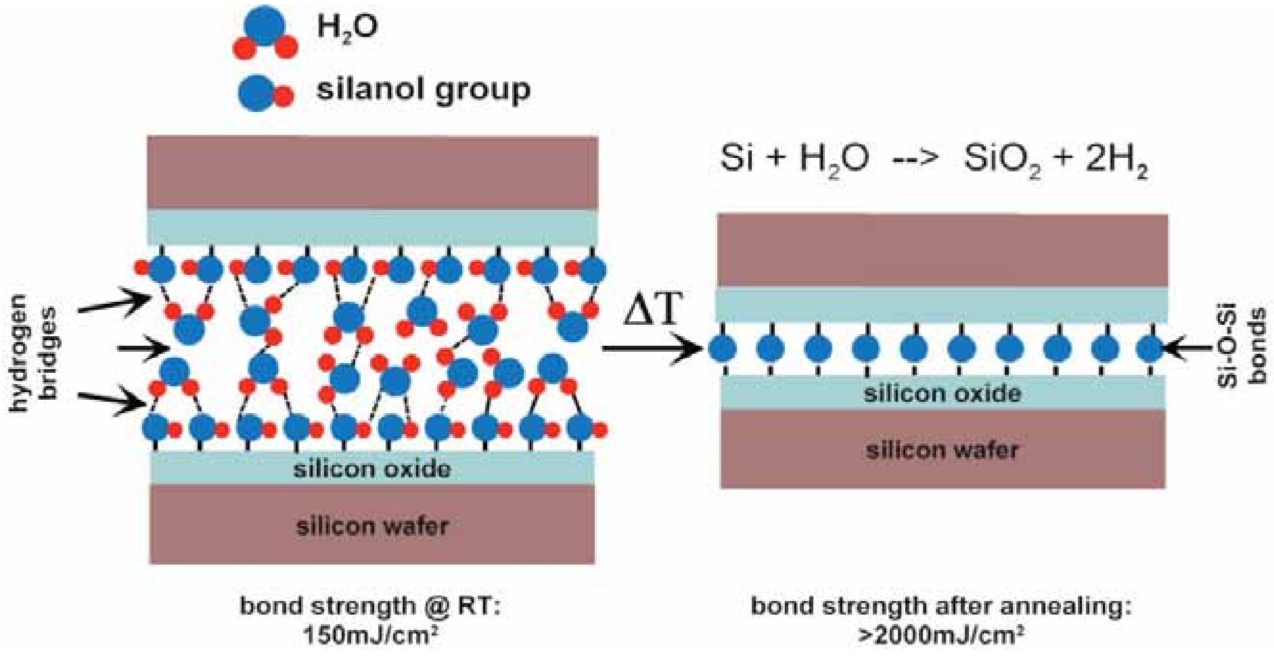
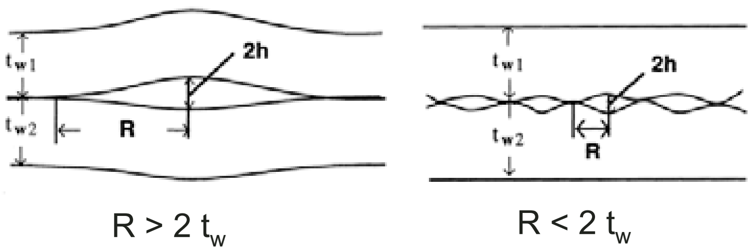


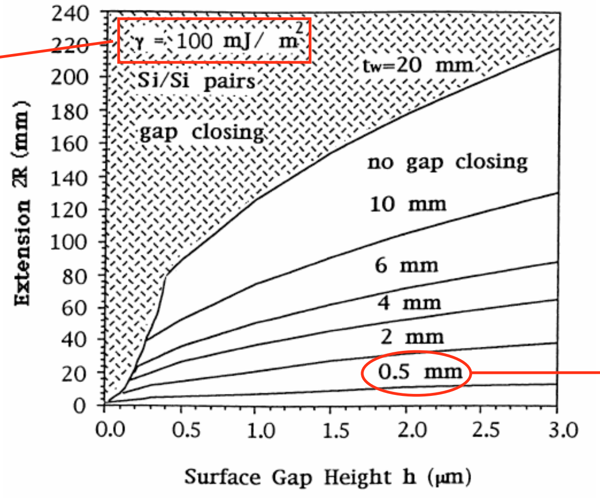
Figure from: Christiansen, MPI für Mikrostrukturphysik, Halle, D

For details see: Stengl e.a. Jpn.J.Appl.Phys. 28, 1735-1741 (1989)

Gap closing theory for waviness



$R > 2\,t_w$ $R < 2\,t_w$



room temperature bonding

thickness of 100 mm wafers

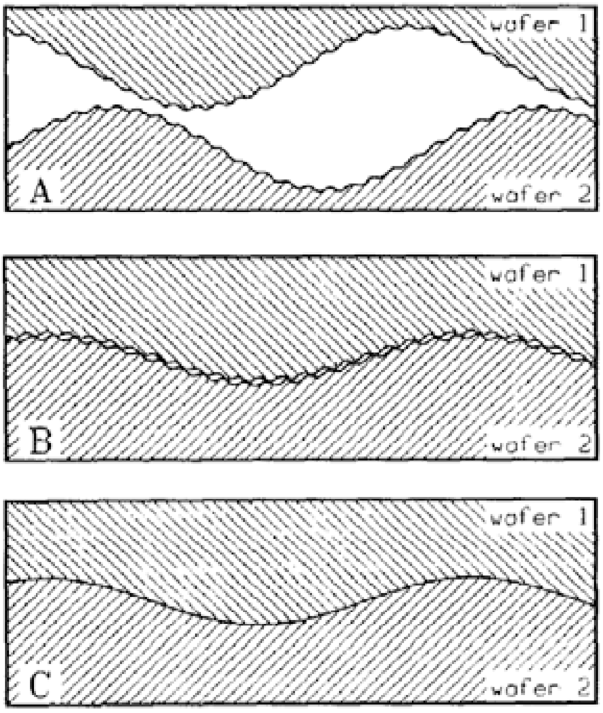
Q.-Y. Tong, U. Gösele, Mater. Chem. Phys. 37 (1994) 101

Waviness and (micro)roughness

A. before bonding

B. after room temperature bonding

C. after complete bonding at high temperature



From: Maszara e.a. J. Appl. Phys. 69, 257-260 (1991)

Bonding interface after high temperature

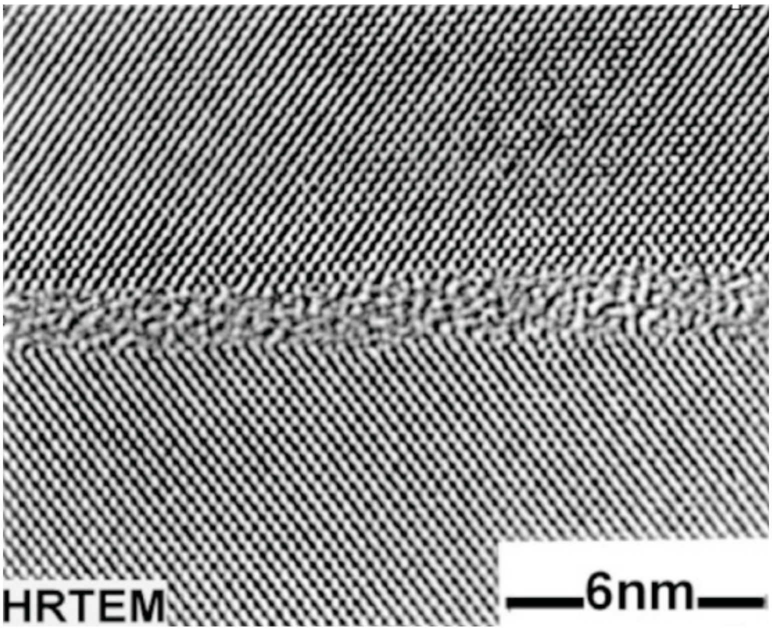


Figure from: Christiansen, MPI für Mikrostrukturphysik, Halle, D

Old school

Galileo Galilei, 1638

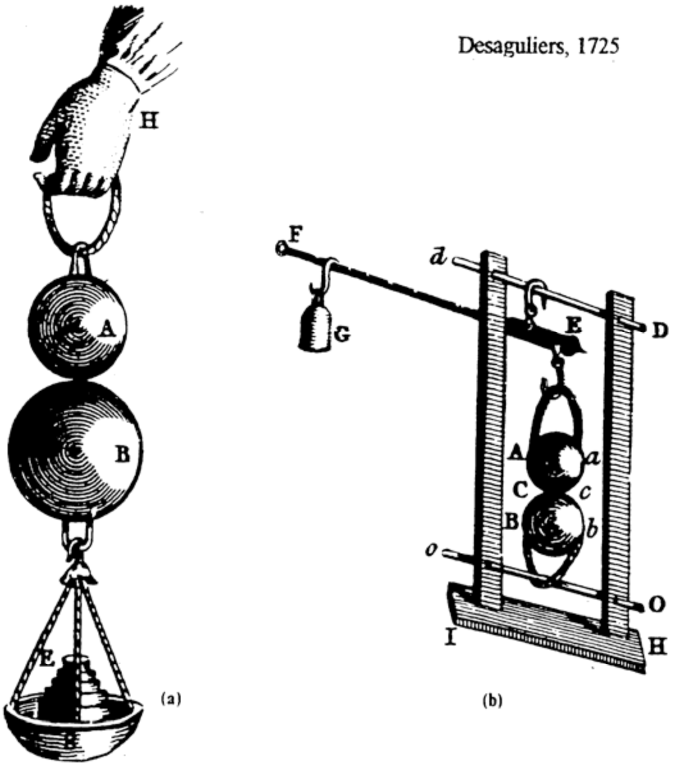
First hypothetical experiment
and discussion on
adhesion of solids
with plane surfaces

DISCORSI
E
DIMOSTRAZIONI
MATEMATICHE,
intorno à due nuoue ſcienze
Attenenti alla
MECANICA & i MOVIMENTI LOCALI,
del Signor
GALILEO GALILEI LINCEO,
Filoſofo e Matematico primario del Sereniſſimo
Grand Duca di Toſcana.
Con una Appendice del centro di grauità d'alcuni Solidi.

IN LEIDA,
Appreſſo gli Elſevirii. M. D. C. XXXVIII.

FRONTESPIZIO DEI "DISCORSI E DIMOSTRAZIONI
MATEMATICHE INTORNO A DUE NUOVE SCIENZE"
(Firenze, B. Biblioteca Nazionale)

Bonding is about contact area and contact forces

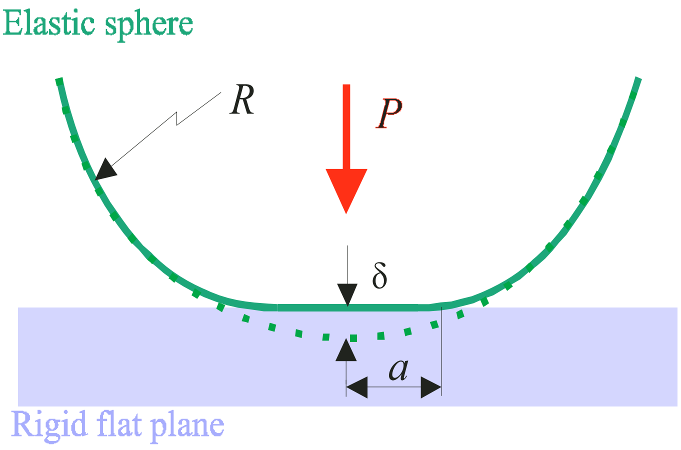


Desaguliers, 1725

Desaguliers took two lead spheres and, having from each of them cut off a segment of about 1/4 inch in diameter, pressed them together by his hand, with a little twist, to bring the flat parts to touch closer

D. Dowson, History of tribology, Longman Group Ltd. London 1979

Contact theory: Hertz



Hertz in 1880 investigated the deformation of polished glass lenses pressed together, in order to study the phenomenon of "Newton's rings"

His formulation forms the cornerstone of the theory of contact mechanics

Compression gives elastic deformation:

$$\frac{4a^3E}{3R\left(1-\nu^2\right)} = P$$

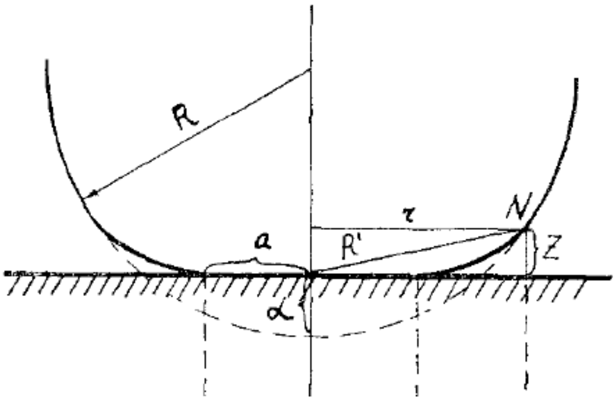
deformation:

$$\delta = \frac{a^2}{R}$$

Contact theory: Derjaguin-Muller-Toporov

Include surface adhesion energy *w*:

$$\frac{4a^3E}{3R\left(1-\nu^2\right)} = P - \boxed{2\pi wR}$$



with the adhesion energy given as:

$$w = \gamma_1 + \gamma_2 - \gamma_{12}$$

B.V. Derjaguin, V.M. Muller & Y.P. Toporov (DMT), J. Colloid Interface Sci. 53, 1975, p. 314
D. Maugis, J. Colloid Interface Sci. 150, 1991, p. 243

A stable bond is possible if:

Surface conditions allow a contact area large enough for a sufficient change in surface energy

Important parameters:

- * surface roughness
- * elasticity of materials
- * surface energy

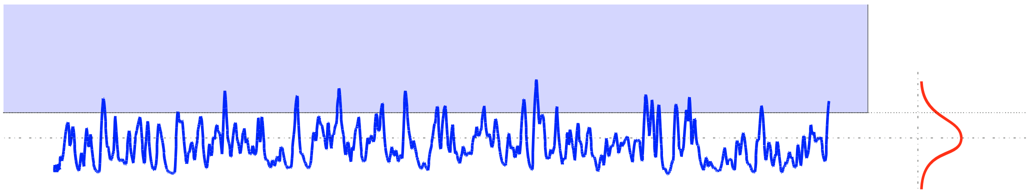
Dimensional analysis of "bondability"

- "Soft" materials are easier to bond:
  - Bondability ~ 1/E [m^3/J]
- Large interface energy leads to strong bond:
  - Bondability ~ w [J/m^2]
- Surface roughness of small height *h* or large wavelength λ is easy to deform:
  - Bondability ~ λ/*h* [m/m]

since $\lambda \sim (R.h)^{1/2}$: Bondability ~ $(w/E).(R/h^3)^{1/2}$

=> Dimensionless parameter: $(w/E).(\lambda / h^2)$

Contact between rough surface and rigid flat plane



Gaussian distribution of surface asperities

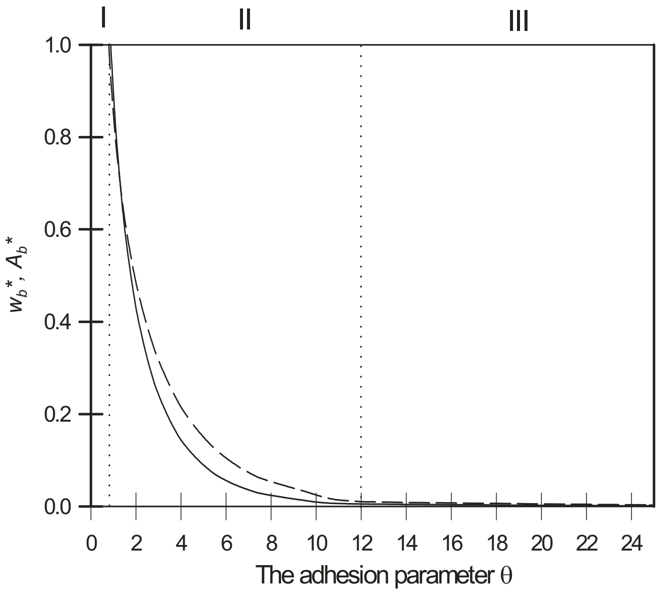
Mathematical treatment using Hertz-DMT theory leads to a characteristic parameter θ:

$$\theta = \frac{E^*}{w}\sqrt{\frac{\sigma^3}{R}}$$

with σ the standard deviation of the Gaussian curve

Gui e.a. J. Appl. Phys. 85, 7448-7454 (1999)

Bond area/energy vs. surface adhesion parameter



Bonding regime, θ<1
Non-bonding regime, θ>10
Adherence regime, 1<θ<10

Gui e.a. J. Appl. Phys. 85, 7448-7454 (1999)

Experimental results

Wafer No.	Surface modification	σ [nm]	R [µm]	η_s [µm²]	$\sigma R \eta_s$
6	CMP, Pad: IC1000 / SUBA IV Slurry: Nalco2350/ DI H_2O 1: 30	1.2	10.9±4.6	6.25	0.08
7	CMP, Pad: IC1000 / SUBA IV Slurry: Semisperse25/ DI H_2O 1: 2	1.1	13.3±5.1	4.41	0.07
8	HF (1 %) etching, 60 sec. KOH (33 %) etching 30 sec.	1.0	1.8±0.5	38.4	0.07
9	HF (1 %) etching, 60 sec. KOH (33 %) etching 10 sec.	0.9	2.0±0.8	33.6	0.06
10	No	< 0.1	76.2±43	21.2	0.07

Gui e.a. J. Appl. Phys. 85, 7448-7454 (1999)

Experimental results cont$^{\mathrm{d}}$.

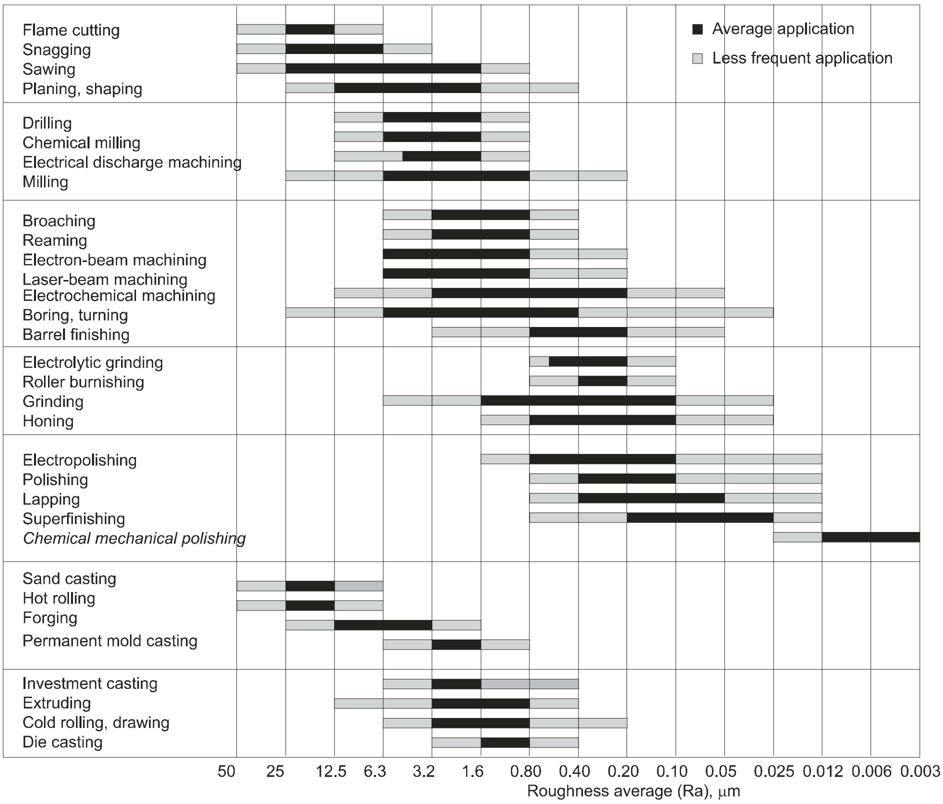
Results of direct wafer bonding at room temperature

Bonded wafer pairs	θ	Bond speed	Specific effective bonding energy [J/m^2]	Voids
Nos. 1 and 6	9.5	With pressure, slow	0.05	a few
Nos. 2 and 7	7.7	slow	0.07	a few
Nos. 3 and 8	16.8	Not bondable	-	-
Nos. 4 and 9	13.8	Not bondable	-	-
Nos.5 and 10	0.1	Spontaneously	0.10	No

Intermezzo:

Reducing surface roughness by CMP

Surface roughness after machining

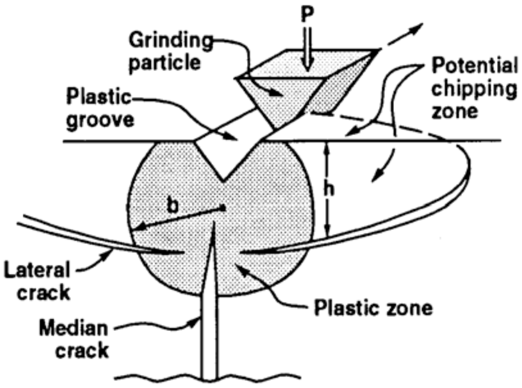


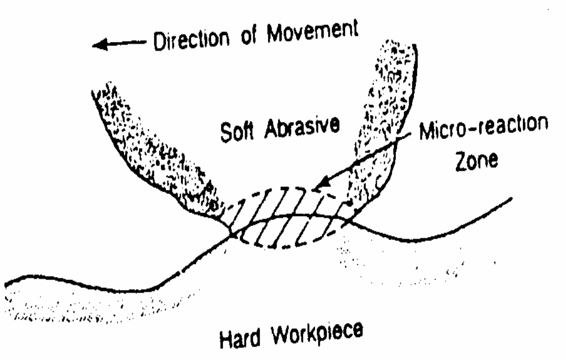
Source: *Machinery's Handbook*, 24th ed., Industrial Press, 1992

CMP vs Mechanical Polishing

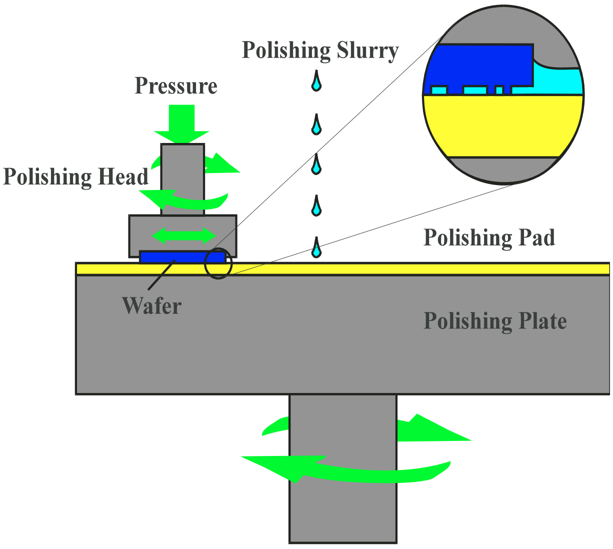
Mechanical Polishing:
Mechanical effect only

CMP: Chemical mechanical effect



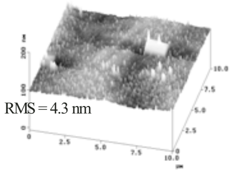


Chemical mechanical polishing, CMP

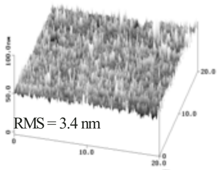


$$R_r = K * P * V$$

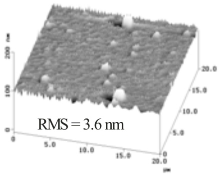
CMP and bonding of Si-based materials



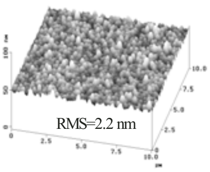
KOH etched Si surface before CMP



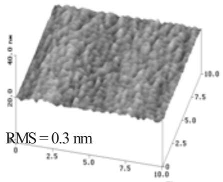
P^{++} Si surface before CMP



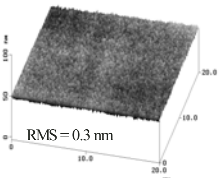
LPCVD $Si_{3+x}N_4$ surface before CMP



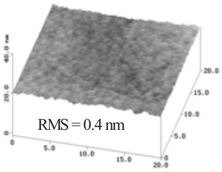
PECVD SiO_2 surface before CMP



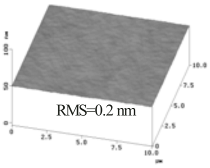
KOH etched Si surface after CMP



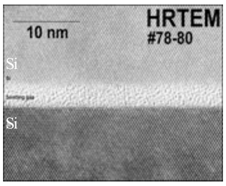
P^{++} Si surface after CMP



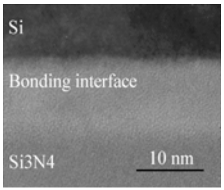
LPCVD $Si_{3+x}N_4$ surface after CMP



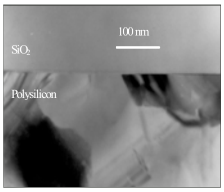
PECVD SiO_2 surface after CMP



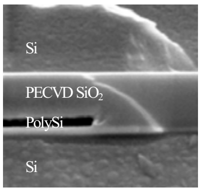
DWB between SCS and SCS after KOH etching and CMP



DWB between P^{++} Si and LPCVD $Si_{3+x}N_4$ after CMP



DWB between SiO_2 and LPCVD Polysilicon after CMP

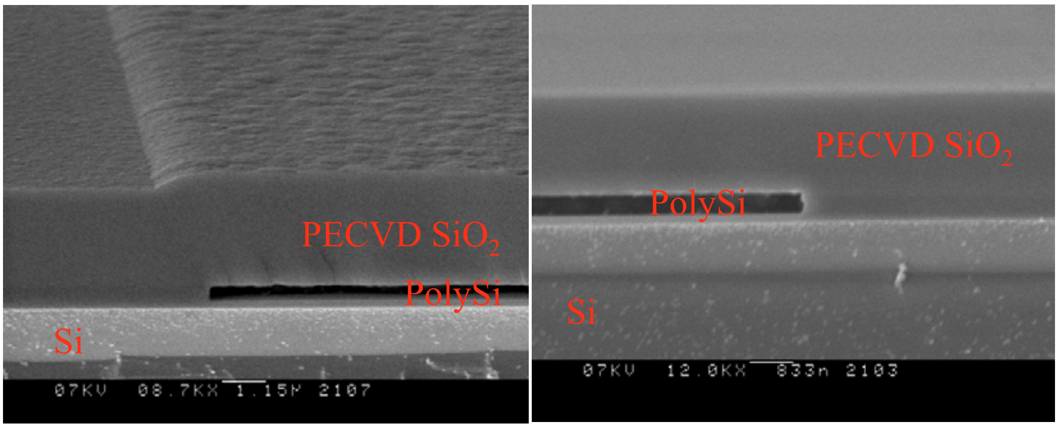


DWB between SCS and PECVD SiO_2 after CMP

Gui e.a. J. Appl. Phys. 85, 7448-7454 (1999)

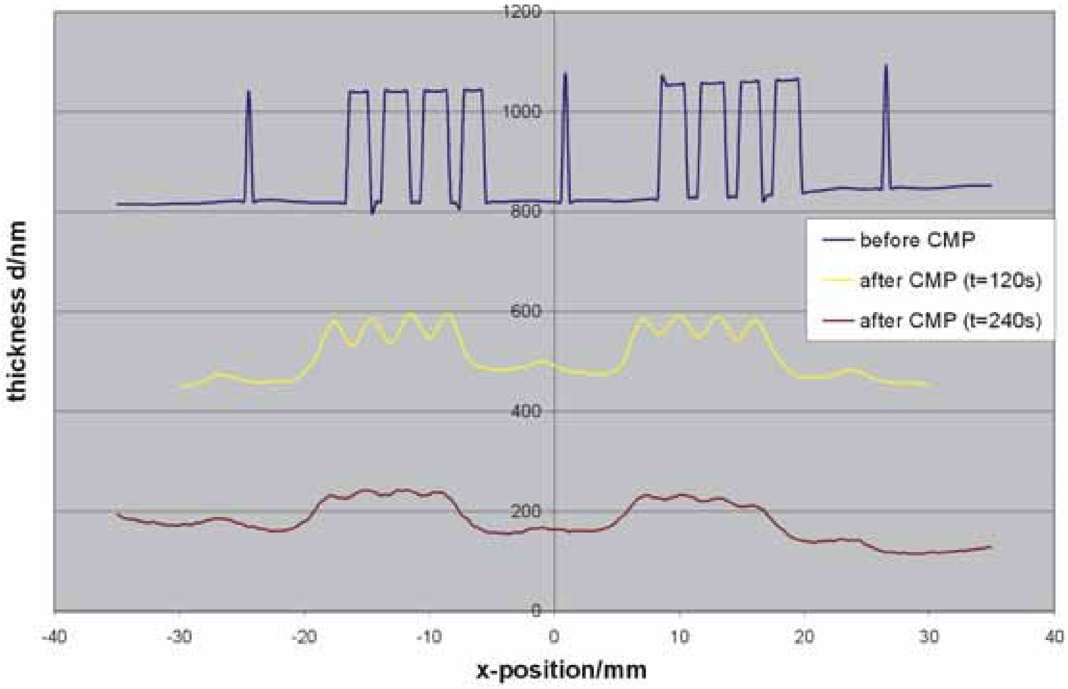
Effects of CMP

- Reduction of roughness
- Surface (chemical) conditioning
- Planarisation

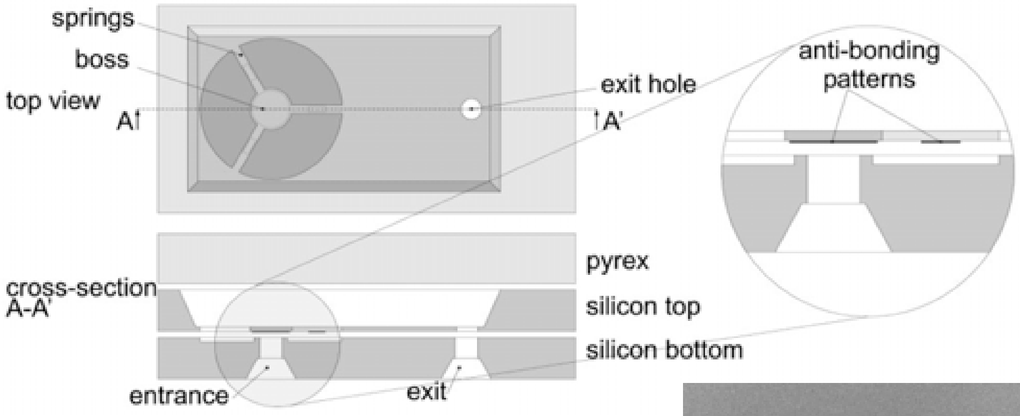


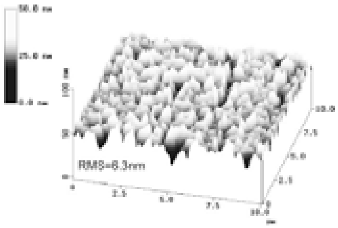
before and after planarisation by CMP

Planarisation of PECVD silicon oxide

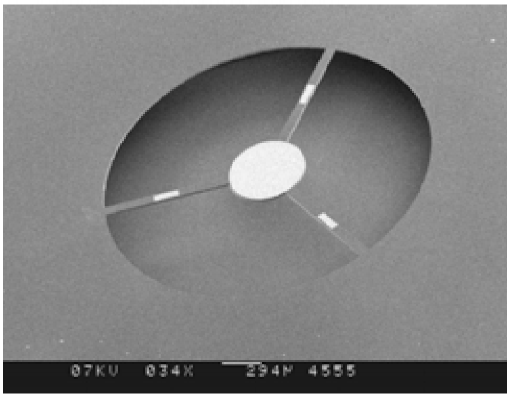


Selective direct bonding



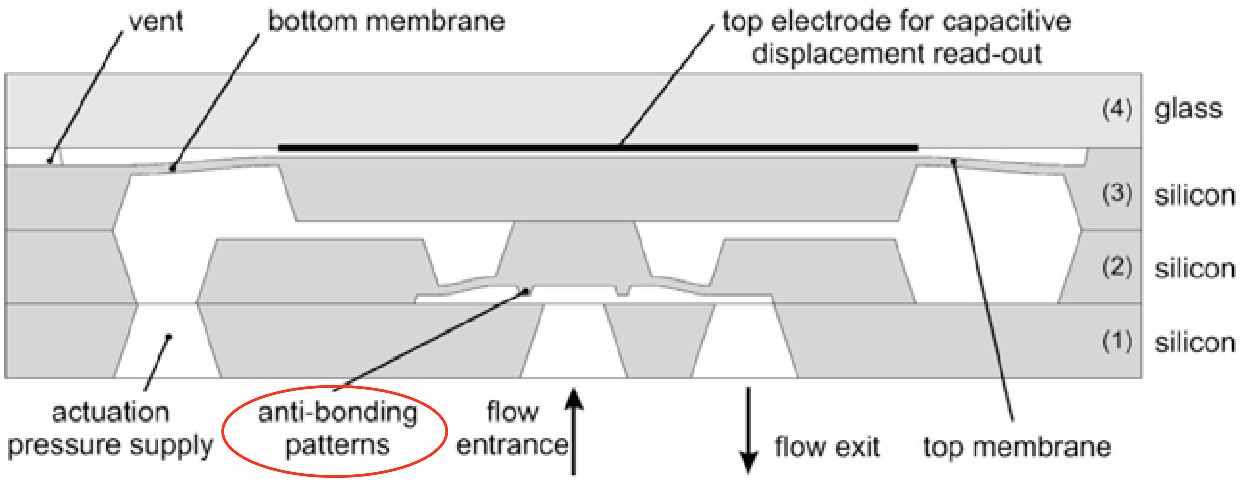


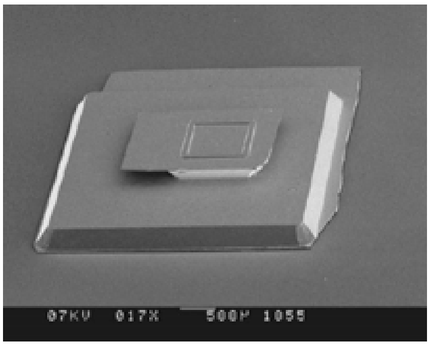
White area: rough silicon nitride
that prevents bonding



Gui e.a. J. Electrochem. Soc. 148, G225-G228 (2001)

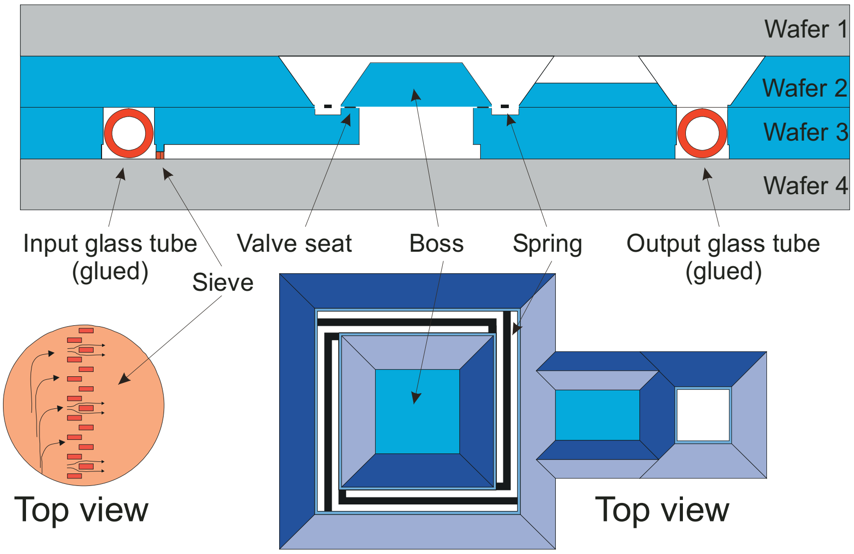
Selective bonding for NC-valve

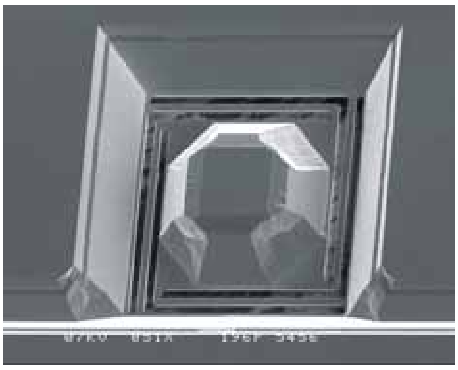


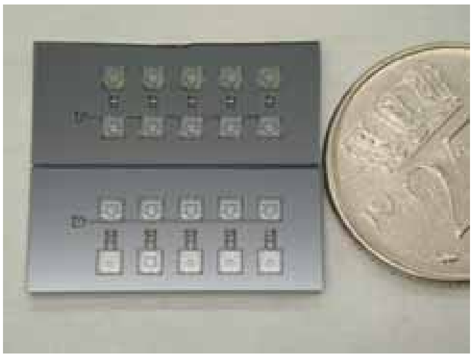


Gui e.a. J. Electrochem. Soc. 148, G225-G228 (2001)

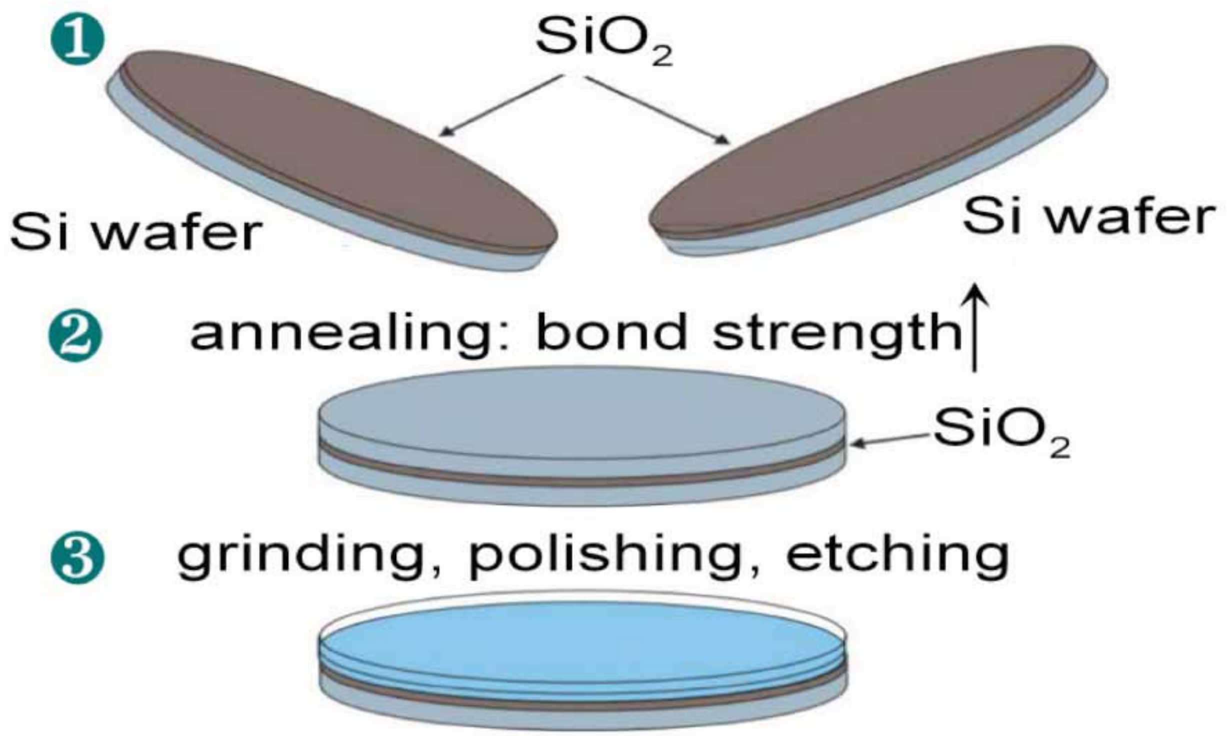
Example: Micro check valves for 50 bars of pressure







Layer transfer by direct bonding



①

② annealing: bond strength↑

③ grinding, polishing, etching

Smart cut process

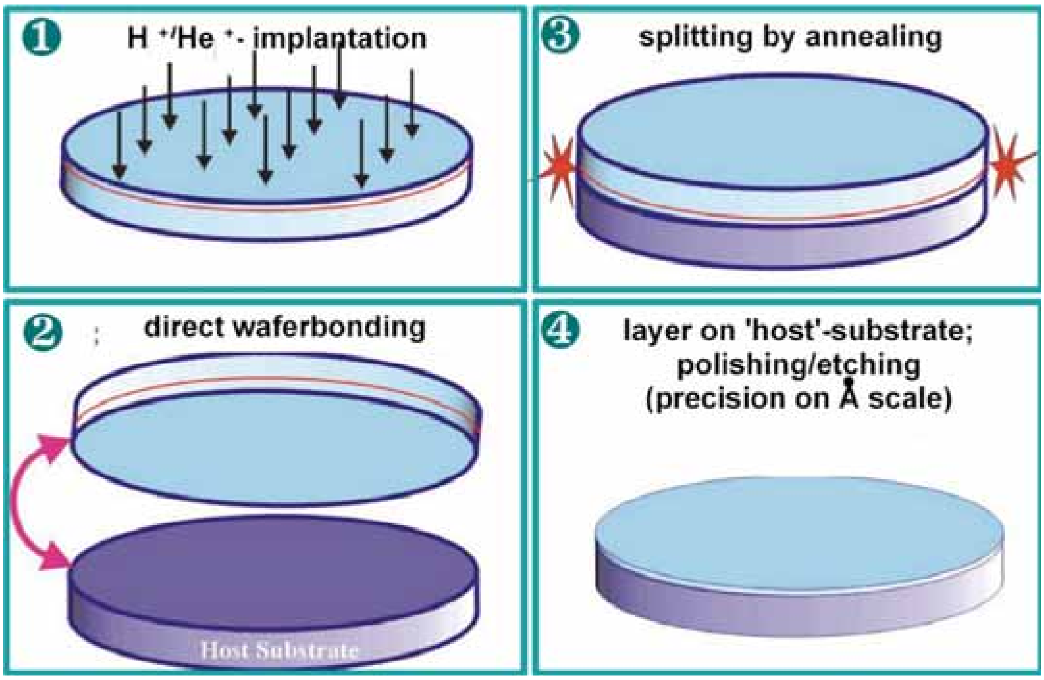


Figure from: Christiansen, MPI für Mikrostrukturphysik, Halle, D

For details see: Tong e.a. Adv.Mat. 11, 1409-1425 (1999)

Smart cut process: surface before polishing

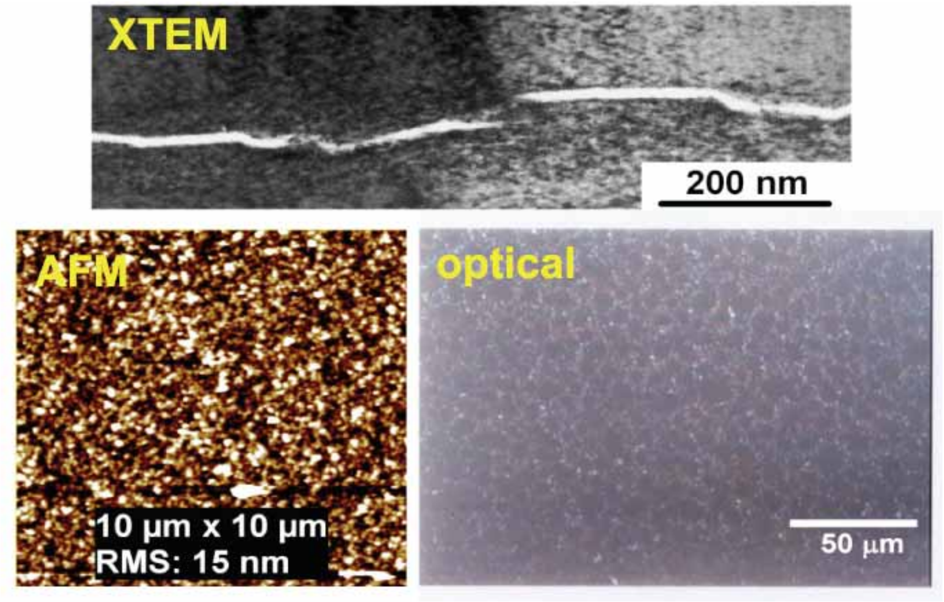
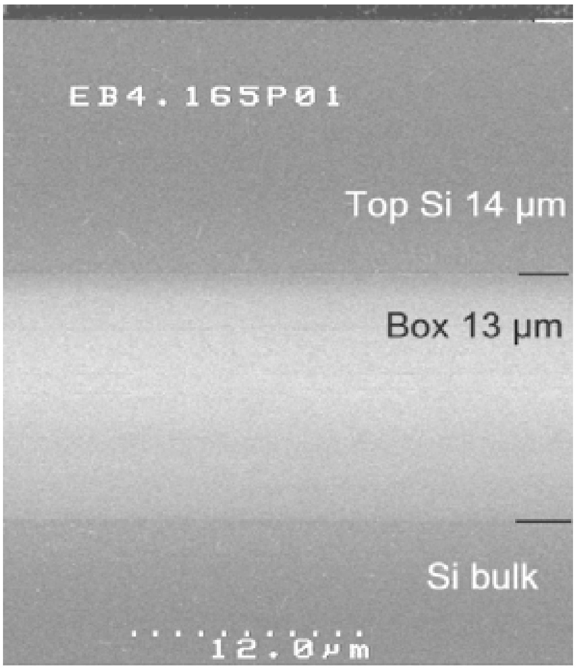
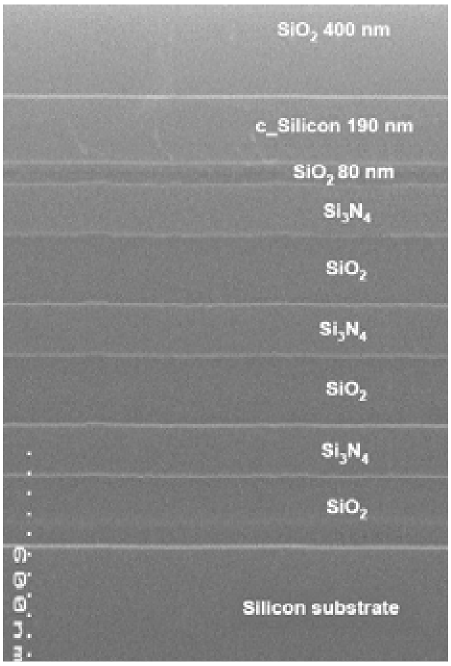


Figure from: Christiansen, MPI für Mikrostrukturphysik, Halle, D

Layer transfer by waferbonding



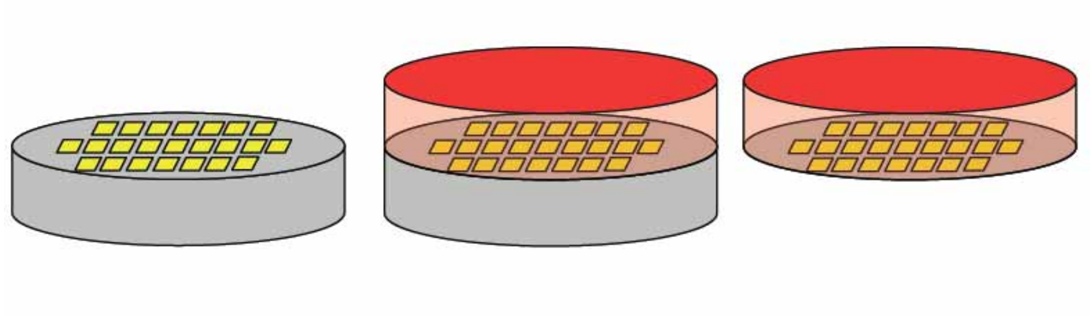
Thick buried oxide

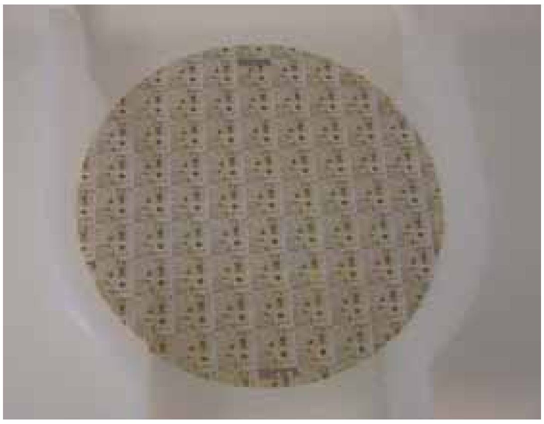


Multiple buried layer stack

Figure from: Aspar, TraciT Technologies, Grenoble, F

Pattern transfer by waferbonding

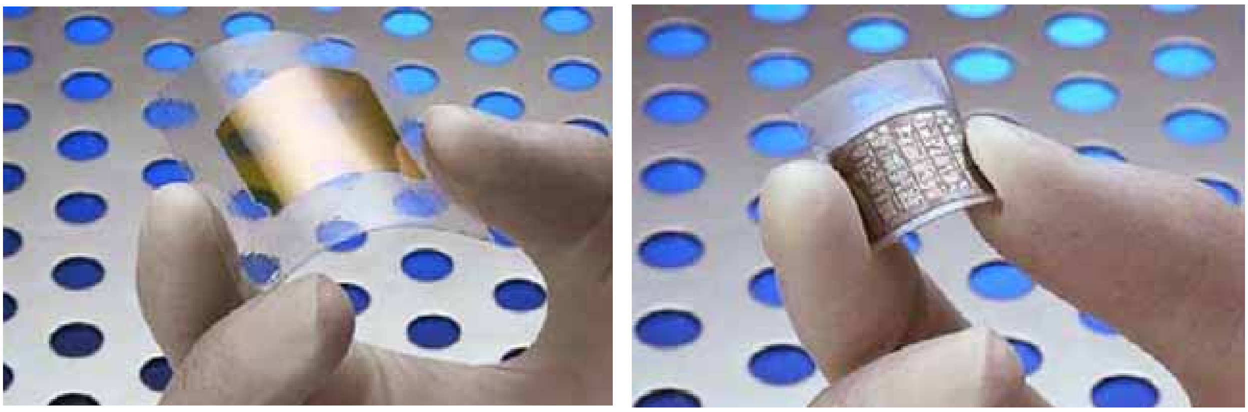




SOI devices transfered onto
200 mm fused silica wafers

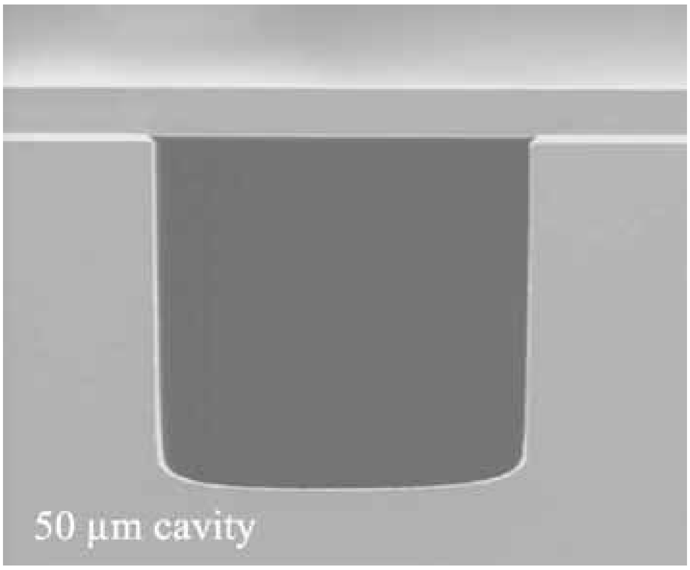
Figure from: Aspar, TraciT Technologies, Grenoble, F

Silicon on plastic



Maleville e.a. Solid-State Electr. 48, 1055-1063 (2004)

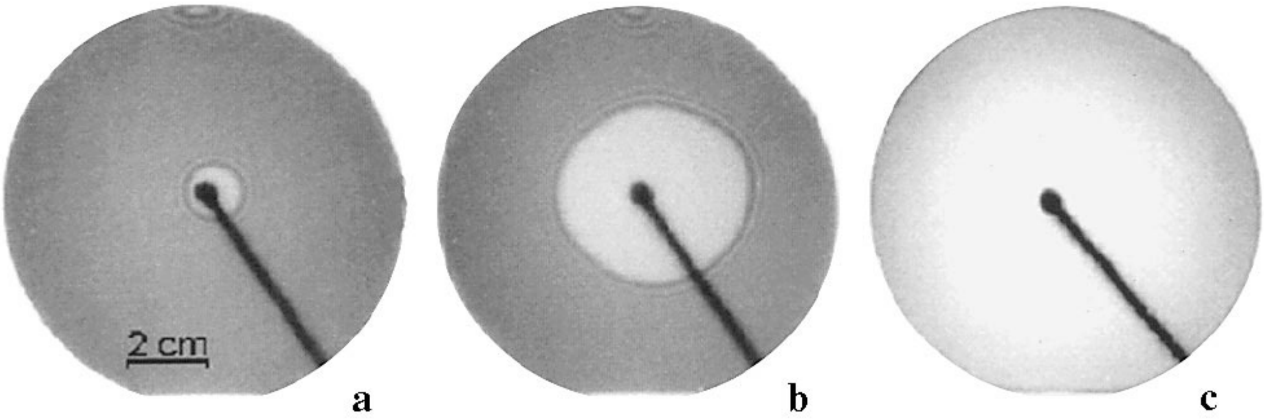
Layer transfer onto etched wafer



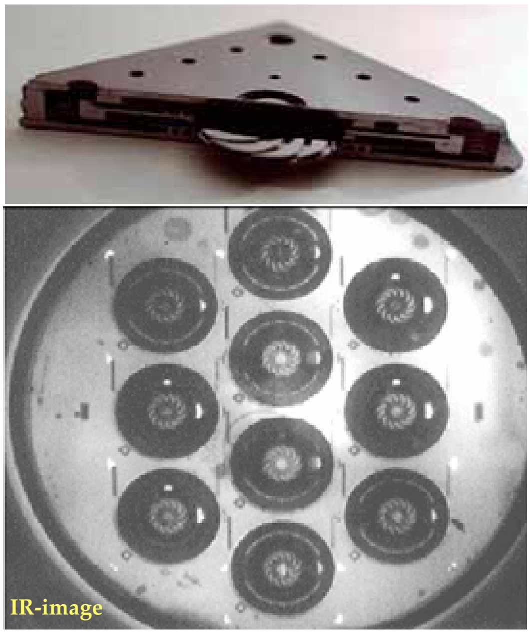
6-7 µm SOI layer

Figure from: Suni, VTT, Finland

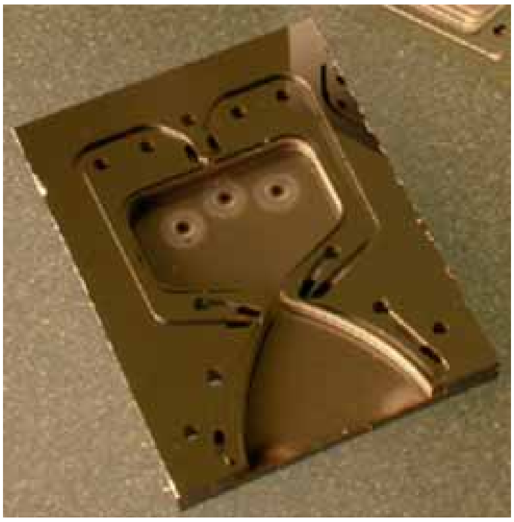
InfraRed microscopy: Newton rings



Multiple layer stacks with patterns



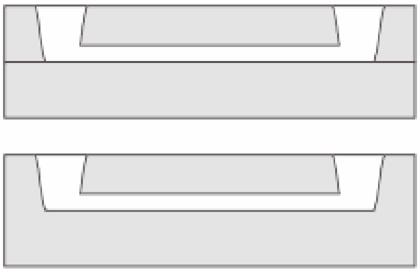
micro gas turbine, 6 or 7 Si wafer stack, deep RIE features, direct bonding, <0.5 μm alignment accuracy



Pressure fed microrocket, chamber pressures to 125 Atm (60 achieved), 6 layer stack, direct bonding. Glass frit and anodic bonding used in packaging

Source: M. Spearing, MIT, Cambridge MA, USA

- Direct Glass-to-Glass Bonding



Several glass types (BF33, AF45, fused silica, Pyrex)

Glass thickness: 1.1 mm (standard), 175 µm (conf. micr.)

Microfabricated reactors "piled up"

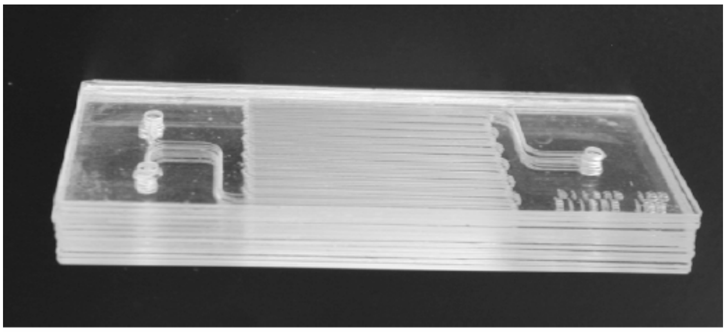
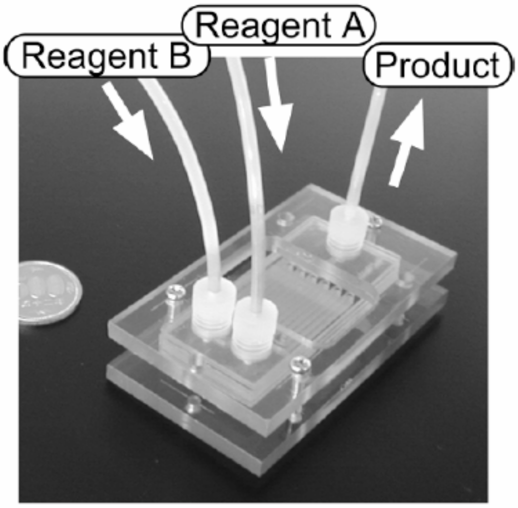
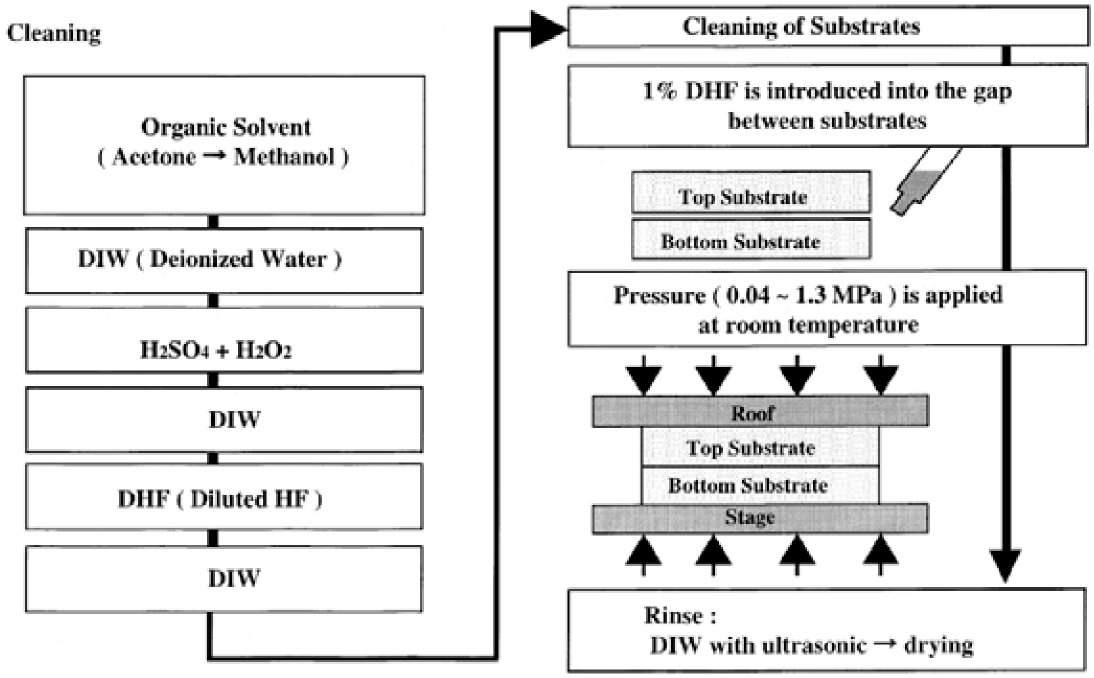


Fig. 3 A photograph of the ten-layer, pile-up microreactor.



Y. Kikutani et al., Lab Chip 2, 2002, 193

Hydrogen Fluoride bonding



Cleaning

Organic Solvent
(Acetone → Methanol)

DIW (Deionized Water)

$H_2SO_4 + H_2O_2$

DIW

DHF (Diluted HF)

DIW

Cleaning of Substrates

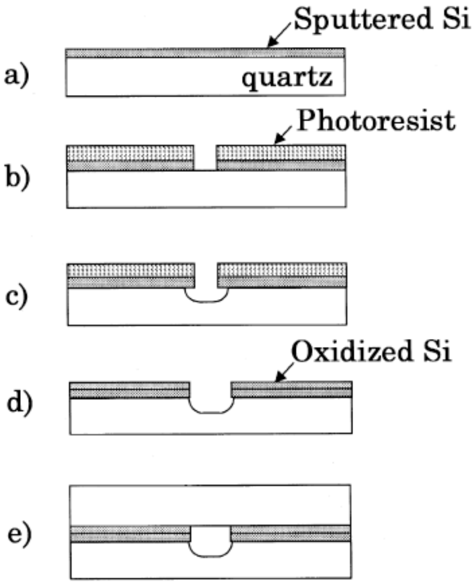
1% DHF is introduced into the gap between substrates

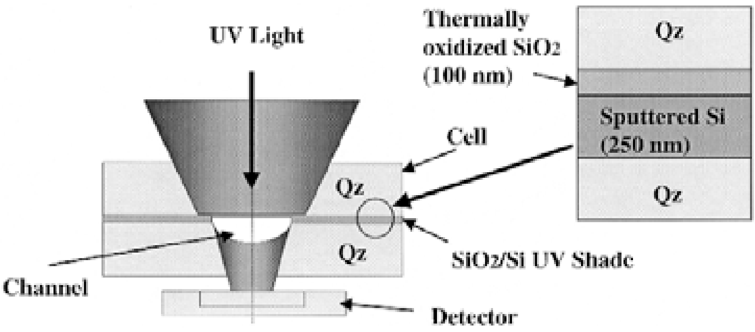
Pressure (0.04 ~ 1.3 MPa) is applied at room temperature

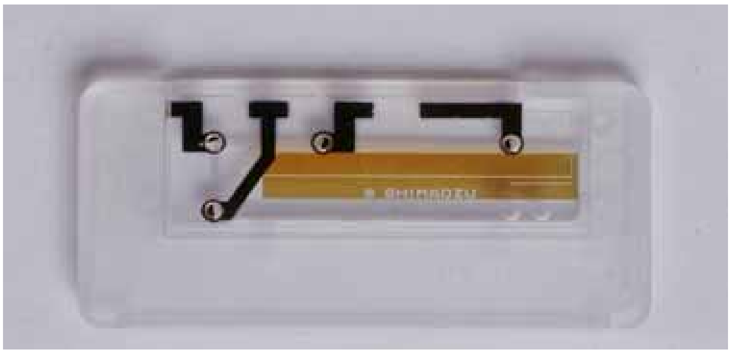
Rinse :
DIW with ultrasonic → drying

Nakanishi e.a. Sens.Act.A 79, 237-244 (2000)

HF bonding for UV flow cell

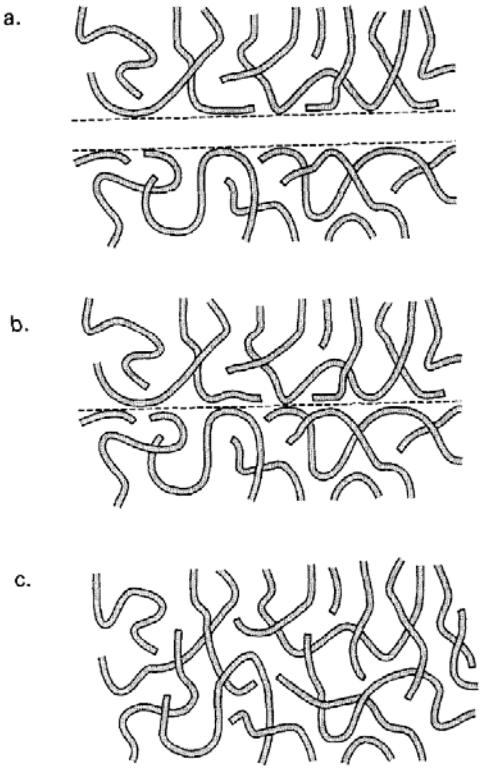






Nakanishi e.a. Sens.Act.A 83, 136-141 (2000)

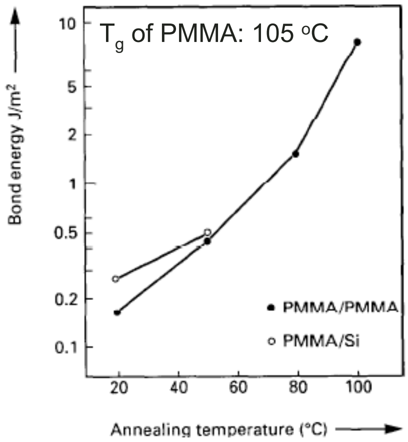
Direct polymer bonding



a. before bonding

b. after direct bonding

c. after annealing above the glass transition temperature of the polymer



Spierings e.a. 49, 139-149 (1995)

Intermezzo:

Plasma activation

Theories why plasma activation helps

- Cracks and removes hydrocarbons

- Modifies surface chemistry (terminates surface with O and OH species)

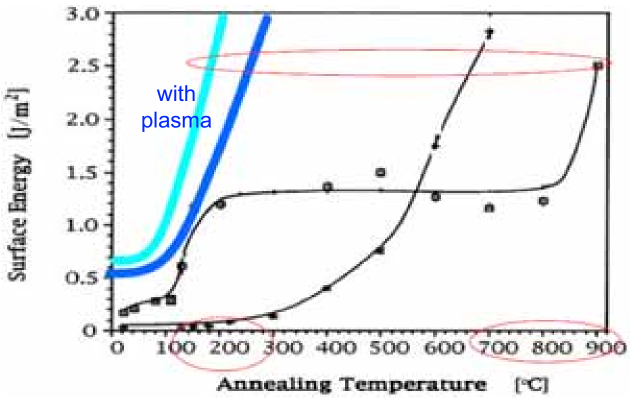
- Increases porosity of surface oxide

- Increases surface hydrophilicity

Result:

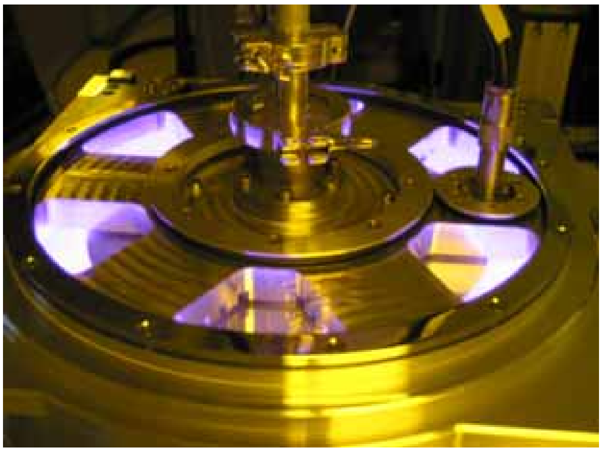
- high bond energy after lower temperature anneal

- faster bonding kinetics

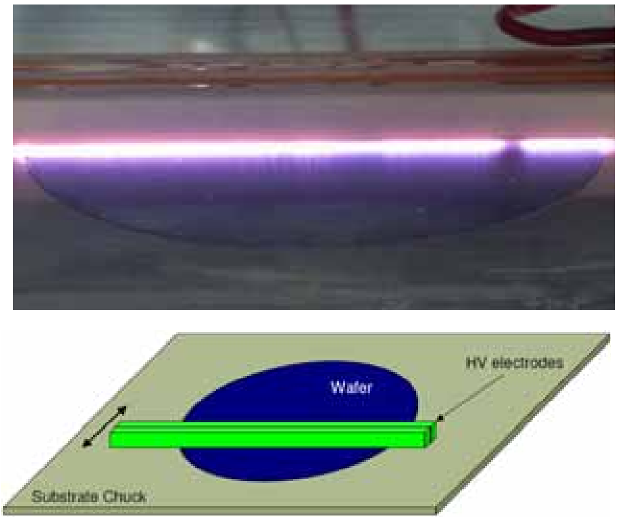


Pictures from: Gabriel, Süss MicroTec, D

Global or local plasma activation ?



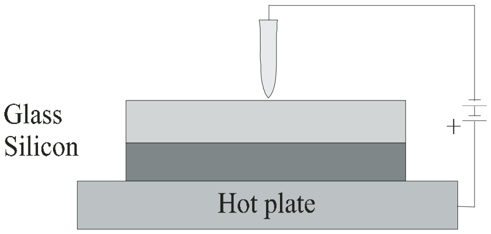
Plasma over complete wafer - EVG, AU

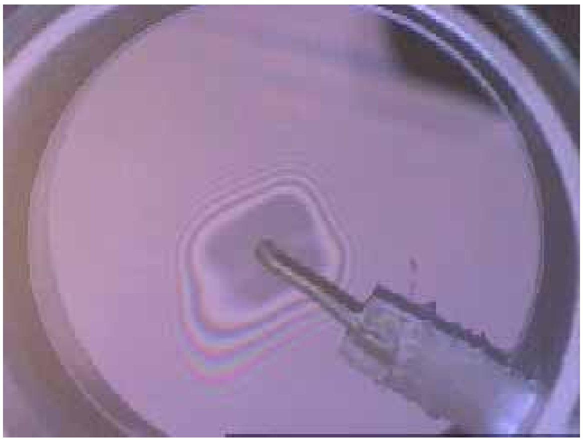


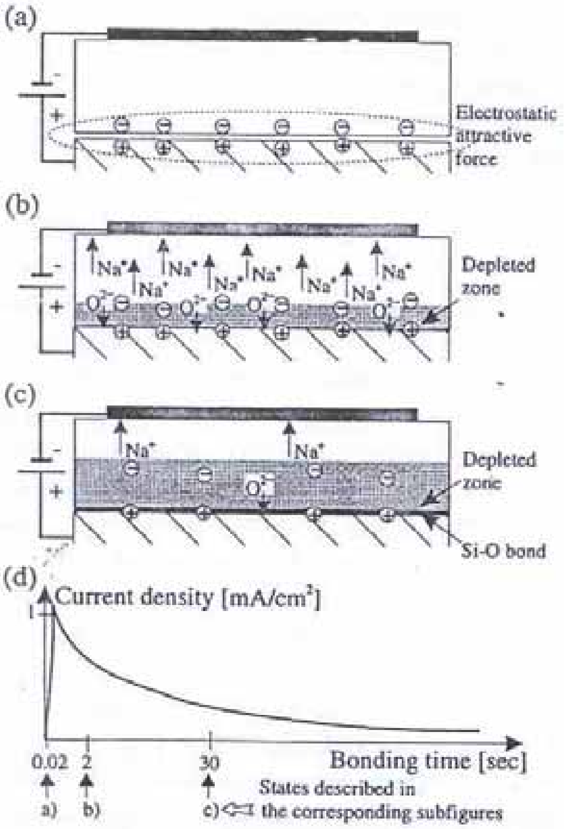
Local plasma + scanning - Süss MicroTec, D

Anodic bonding

Basic principle







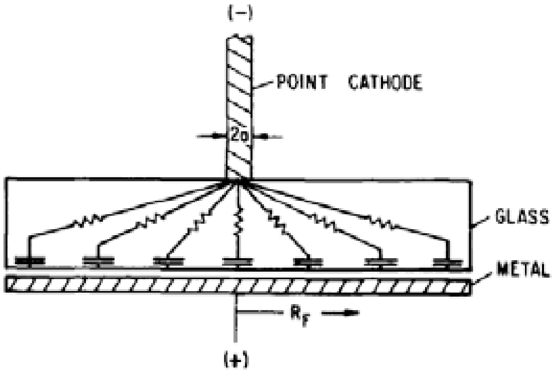
Despont e.a. Sens.Act.A 55, 219-224 (1996); Anthony J.Appl. Phys. 54, 2419-2428 (1983)

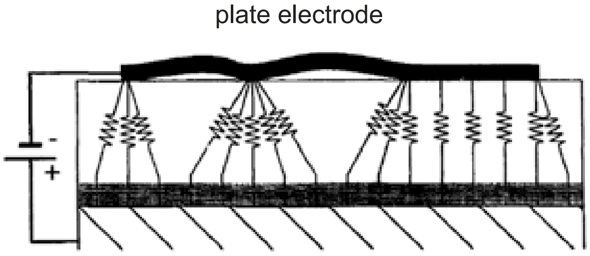
Conditions

For 0.5 mm Pyrex glass to silicon: T= 400-450 °C, V = 500V

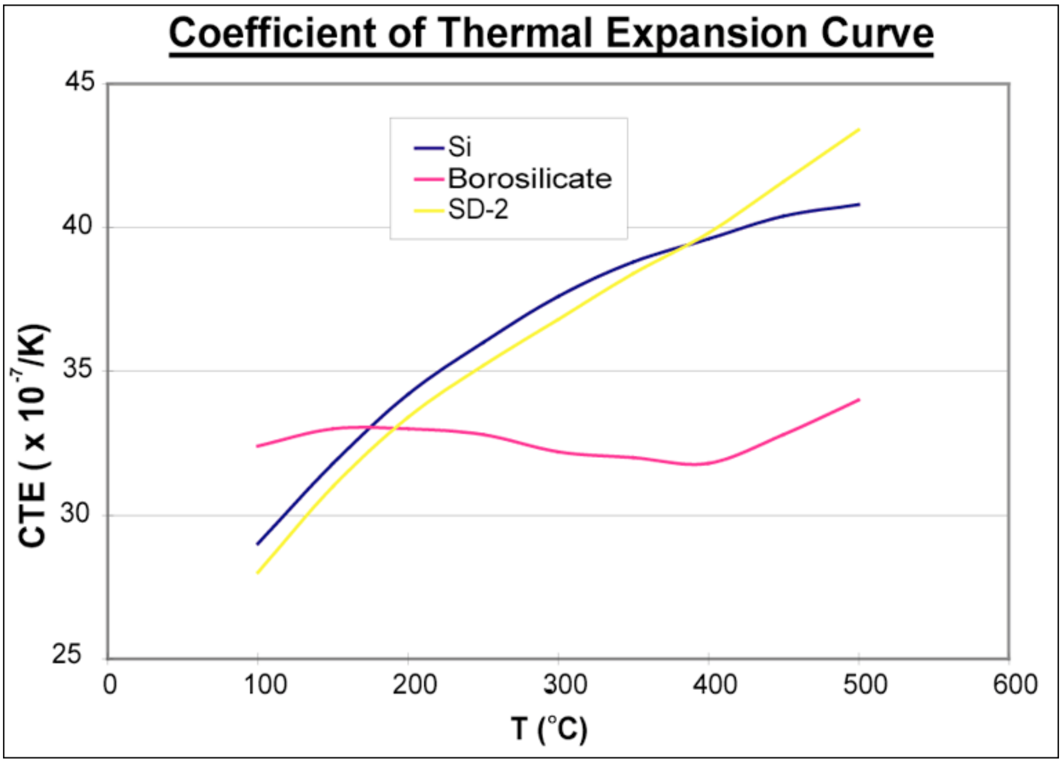
Glass types containing ions like sodium that become mobile at higher temperature, work best

A point electrode is prefered to avoid multiple bonding starting locations and improve uniformity



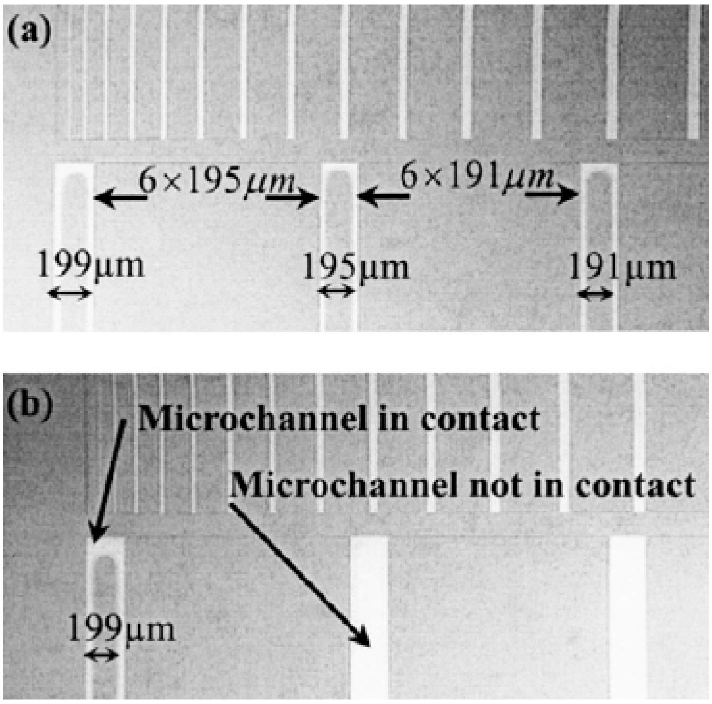


Thermal expansion matching



SD2: special glass from Hoya

Channel collapse during anodic bonding



Bonding parameters: 1027 V, 450 °C, 30 min.
Microchannel depth: 168 nm in (a), 207 nm in (b)

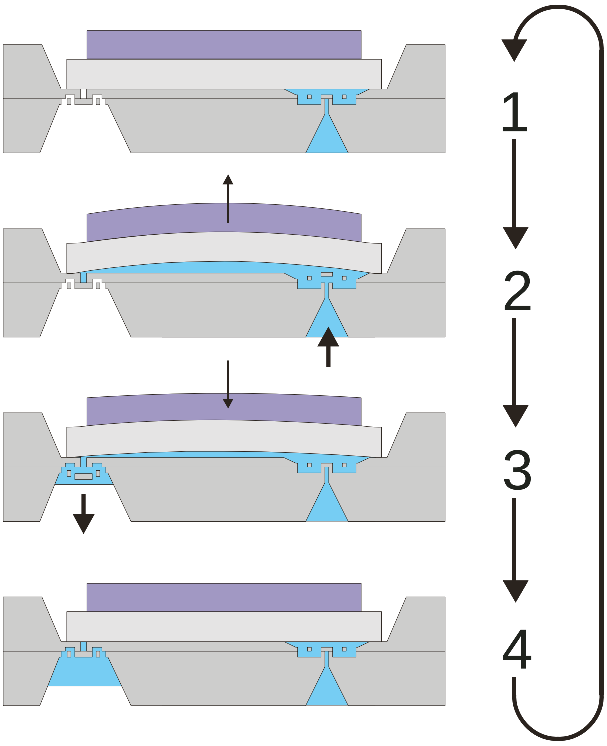
Collapse can be avoided if:

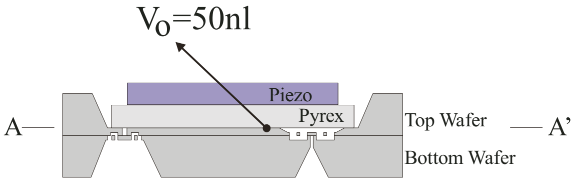
$$\frac{\varepsilon_{air} V^2 a}{E_{eff} d^3} < 1$$

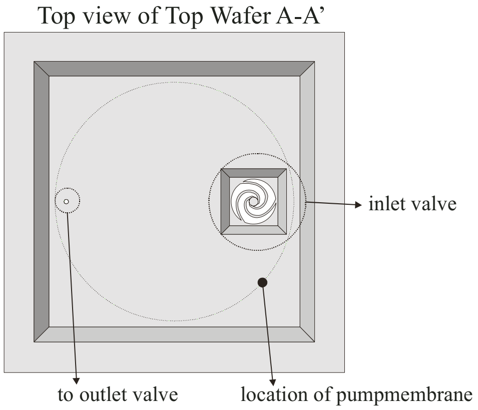
with E_{eff} characterising the stifness of the materials, a the half with of the channel, d channel depth

Shih e.a. J.Appl.Phys. 95, 2800-2808 (2004)

Case study:
peristaltic micropump with low-dead-volume pumping chambers





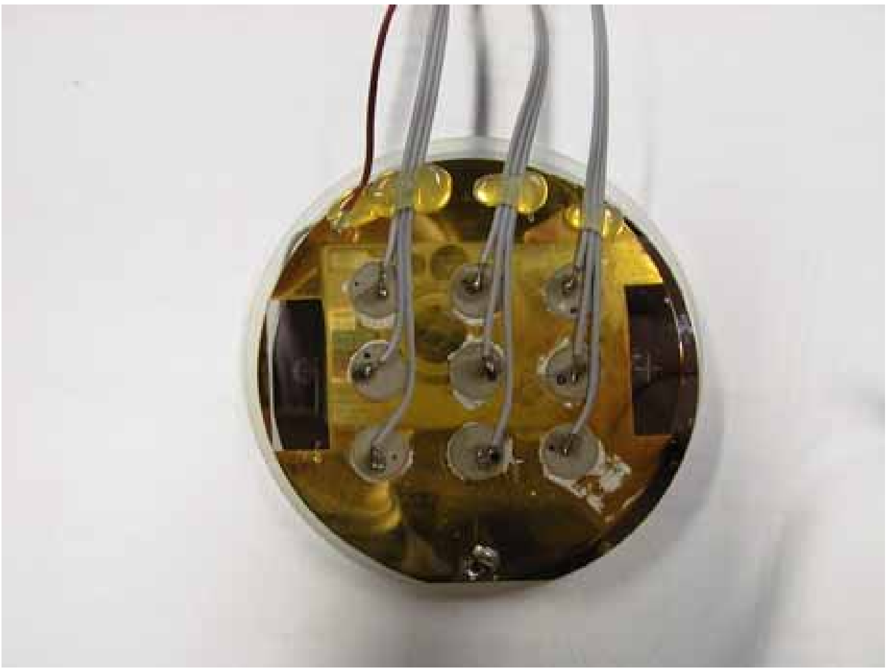


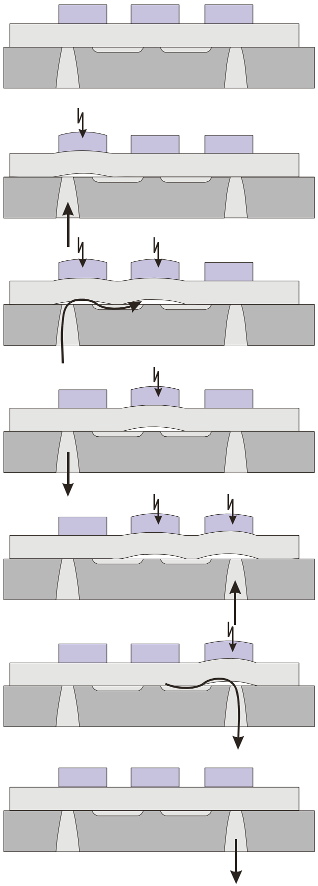
Micropump

MESA+

University of Twente
The Netherlands

Peristaltic micropump



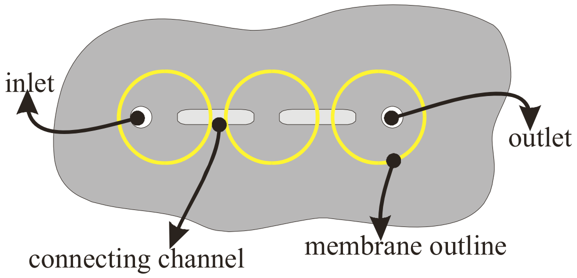


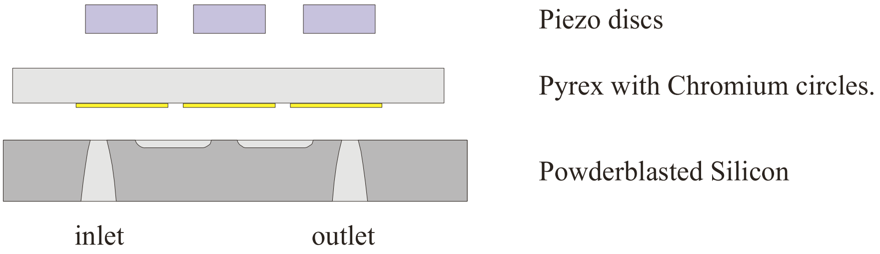
MESA+

Veenstra e.a J. Electrochem. Soc. 148, G68-G72 (2001)

University of Twente
The Netherlands

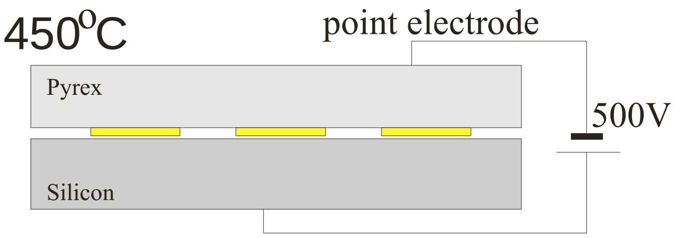
Peristaltic micropump

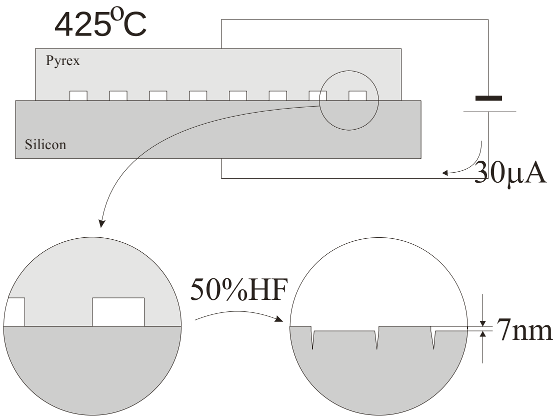


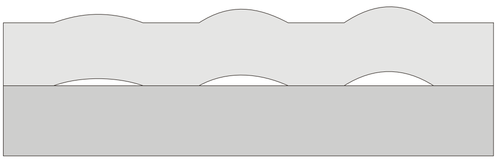


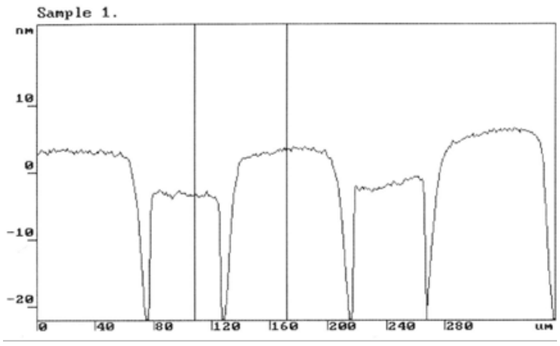
Veenstra e.a J. Electrochem. Soc. 148, G68-G72 (2001)

Selective anodic bonding



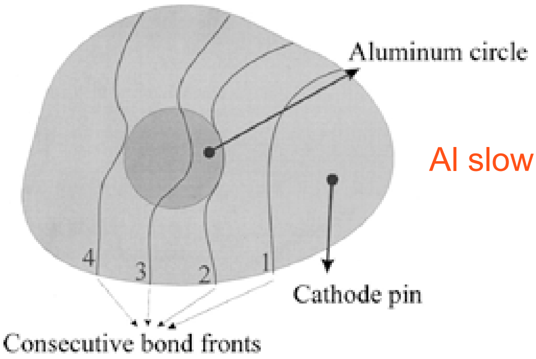




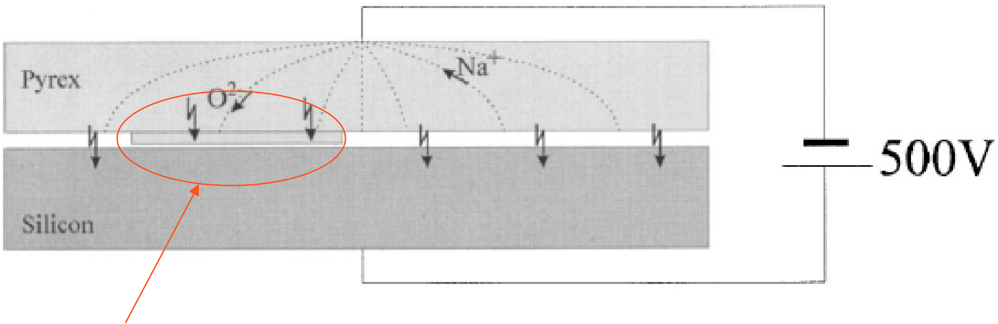


Veenstra e.a J. Electrochem. Soc. 148, G68-G72 (2001)

Cr works, Al does not



Al slowly oxidises and acts as insulator

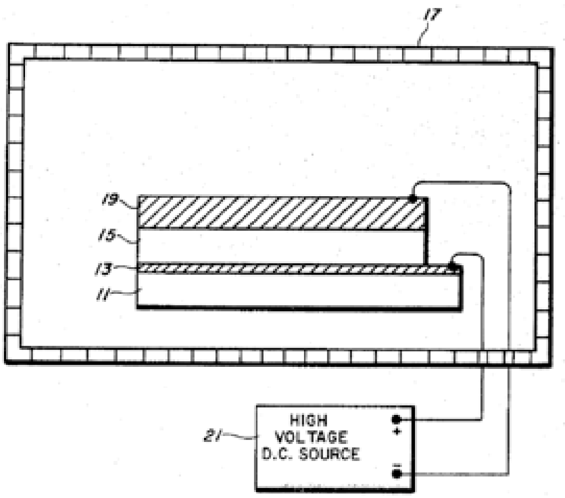


no E-field across gap between wafers

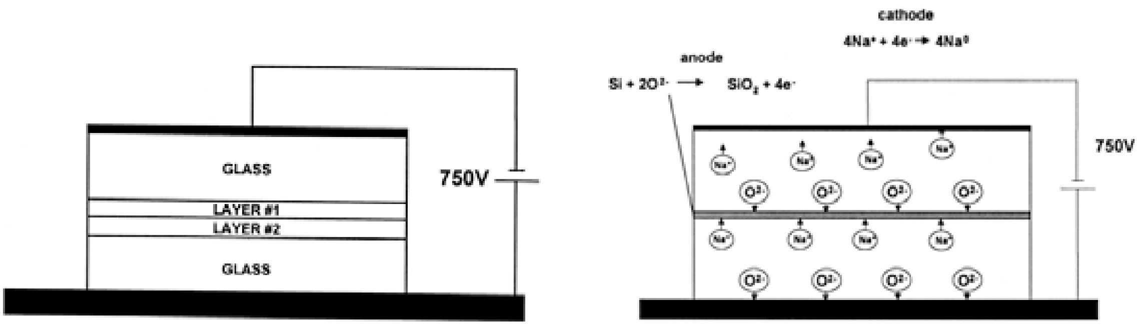
Anodic bonding with intermediate film

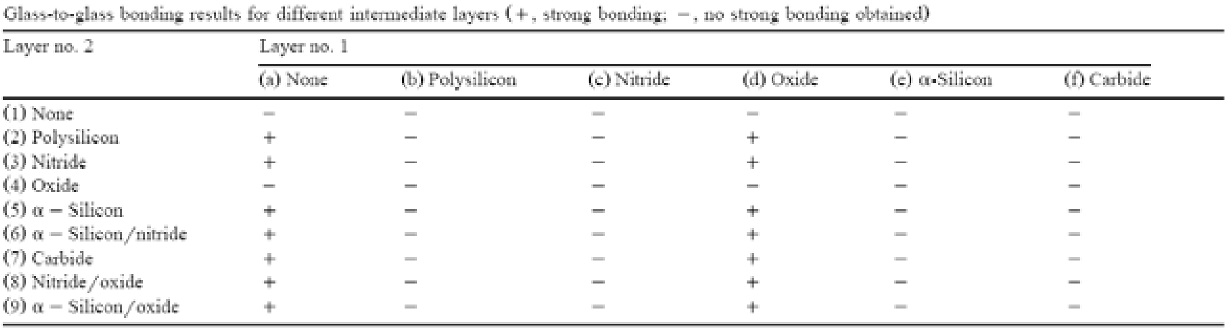
- Silicon-to-silicon with sputtered Pyrex layer; e.g. Berenschot e.a. Sens.Act.A 41/41, 338-343 (1994)
- Glass-to-glass with sputtered Si layer; e.g. Kutchoukov e.a. Proc. Transducers'03, (Boston, June 8-12, 2003) 1327-1330; shows 50 nm deep channels

Wohltjen e.a. US patent 4,452,624
"Method for bonding insulator to insulator"
Issues June 5, 1984



Anodic bonding with intermediate PECVD films





Glass-to-glass bonding results for different intermediate layers (+, strong bonding; −, no strong bonding obtained)

Layer no. 2	Layer no. 1					
	(a) None	(b) Polysilicon	(c) Nitride	(d) Oxide	(e) α-Silicon	(f) Carbide
(1) None	−	−	−	−	−	−
(2) Polysilicon	+	−	−	+	−	−
(3) Nitride	+	−	−	+	−	−
(4) Oxide	−	−	−	−	−	−
(5) α − Silicon	+	−	−	+	−	−
(6) α − Silicon/nitride	+	−	−	+	−	−
(7) Carbide	+	−	−	+	−	−
(8) Nitride/oxide	+	−	−	+	−	−
(9) α − Silicon/oxide	+	−	−	+	−	−

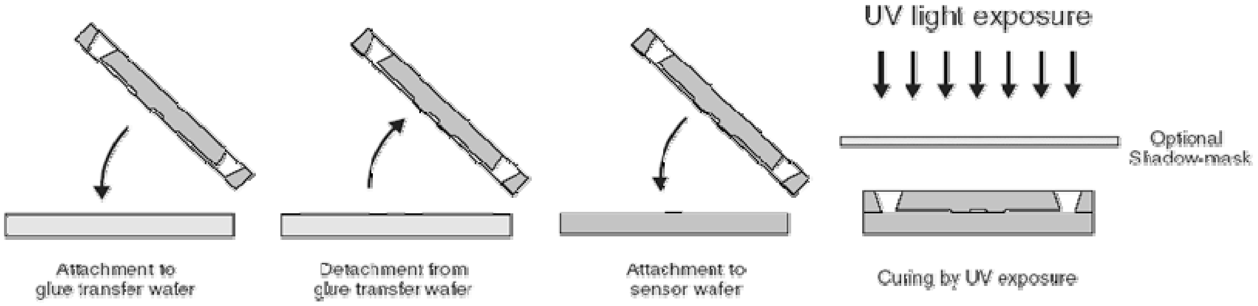
Berthold e.a. Sens.Act.A 82, 224-228 (2000)

Bonding with intermediate layers

Low melting point bonding films, e.g.:

- spin-on sodium silicate, 90 ^{o}C; Wang e.a. Sens.Act. B 45, 199-207 (1997)
- Si-Au eutectic alloying, 365 ^{o}C; Wolffenbuttel e.a. Sens.Act. A. 43, 223-229 (1994)
- In-Sn solder, 160 ^{o}C; Lee e.a. Sens.Avt. A 85, 330-334 (2000)

UV curable glue

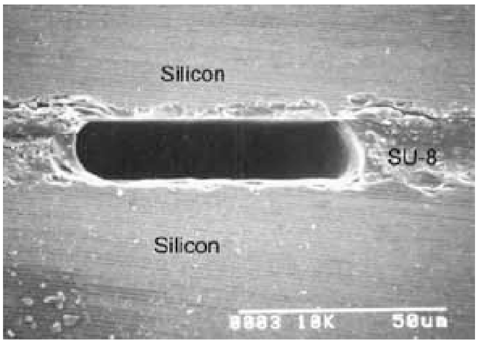


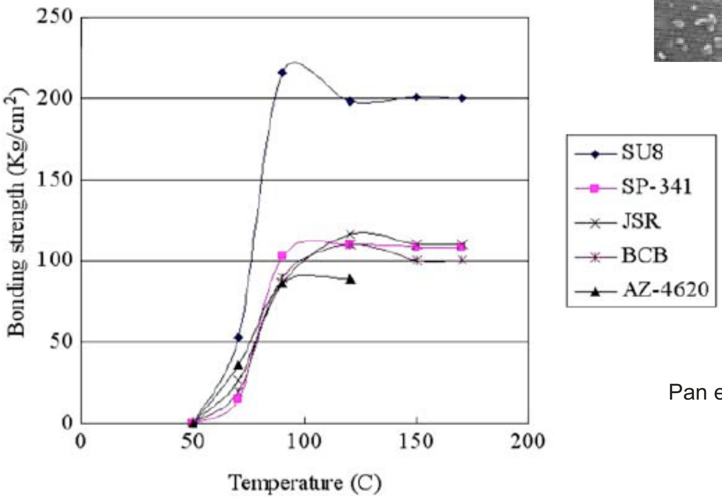
Source: S.Schlautmann, University of Twente, NL, 2001

Photoresists as bonding layers

Range of film thickness of the photosensitive used

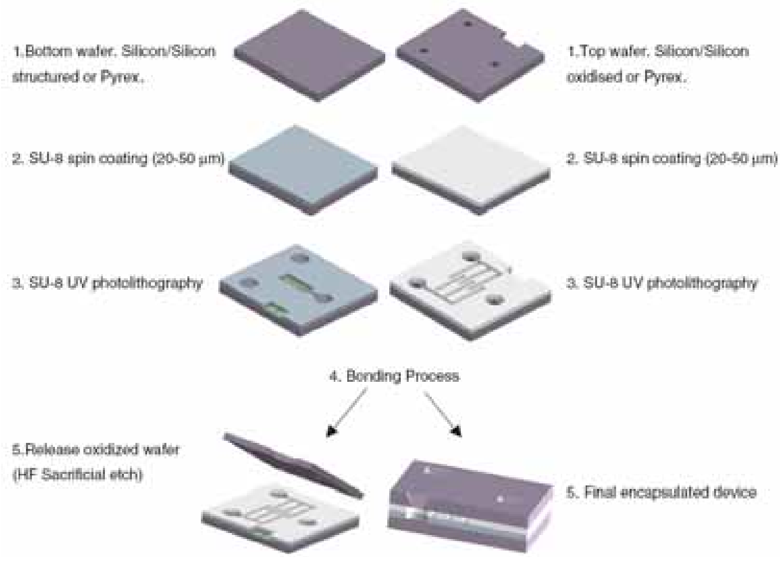
Polymer	Film thickness (μm)
SU-8	20–30
BCB	15–25
JSR-137N	20–30
AZ-4620	10–20
SP-341	15–20

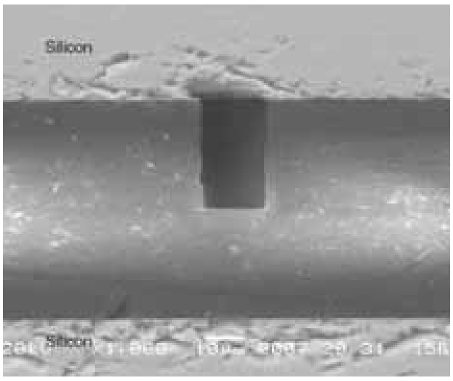


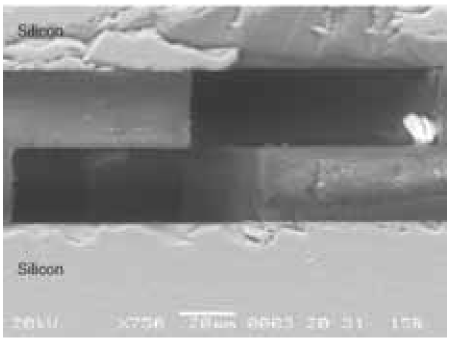


Pan e.a. Microel.Reliab. 45, 657-663 (2005)

Example: SU-8 on SU-8



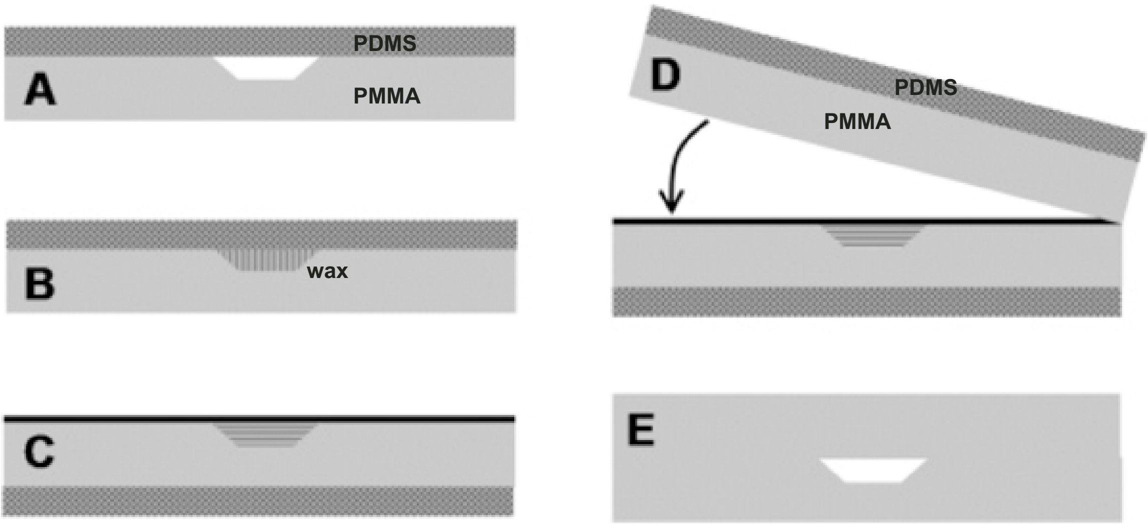




Bonding temperature 100-120 °C

Blanco e.a. J.Micromech.Microeng. 7, 1047-1056 (2004)

Solvent bonding for polymer microfluidics



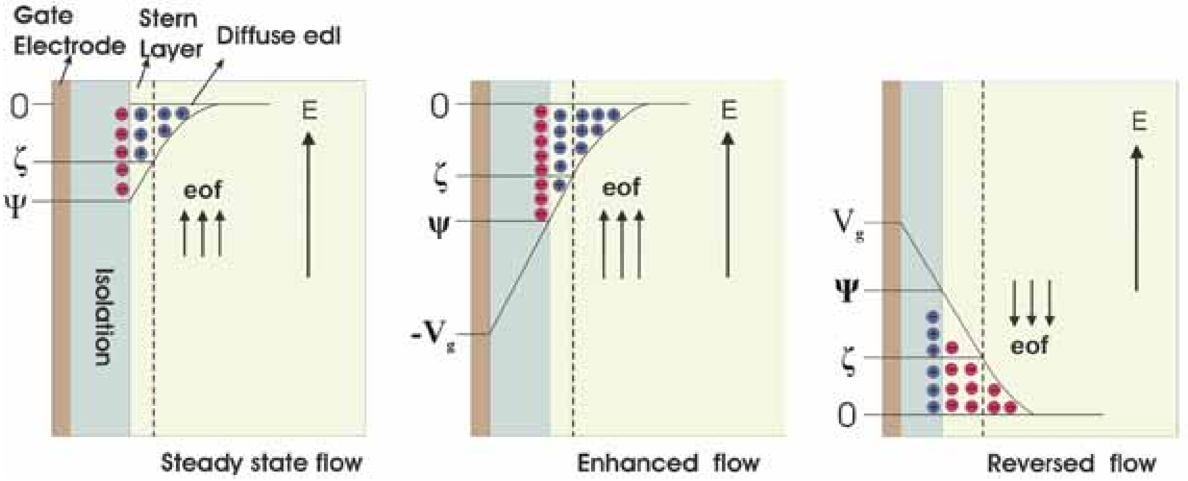
In (B) the assembly is heated, and liquid paraffin wax fills the microchannels; in (C) the device is cooled to solidify the wax, PDMS slab is removed and placed on the opposite side of PMMA to protect device exterior; patterned side of PMMA is coated with acetonitrile (black).
In (D) a second PMMA piece is pressed against first PMMA for 2 min to effect bonding.
In (E) the device is heated to melt the wax, which is removed by combination of vacuum and dissolution in cyclohexane.

Kelly e.a., Anal. Chem. 77, 3536-3541 (2005)

Case study
Field-effect flow control

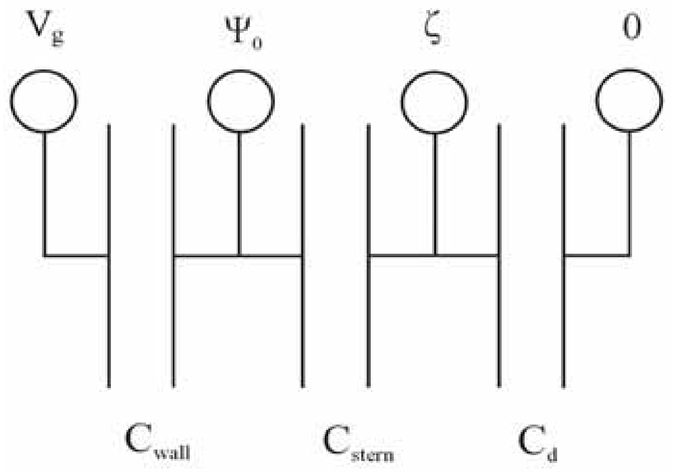
1. µTICs
2. microchannels in glass

EOF control by radial voltage



$$v_{eof} = \frac{\varepsilon \cdot \zeta}{\eta} E$$

Electrical model



$$C_{wall} = \frac{\varepsilon A}{d}$$

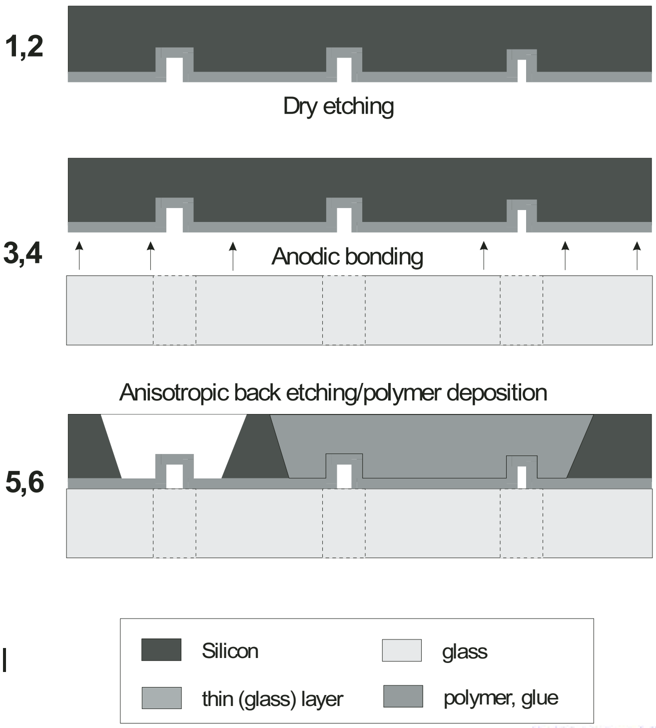
$$C_{diff} = \frac{\varepsilon A}{\lambda_d}$$

$$\Delta\zeta = \frac{C_{wall}}{C_{dl}} V_g$$

$$\lambda_d = \left(\frac{\varepsilon \cdot R \cdot T}{F^2 \cdot \frac{1}{2} \Sigma c_i z_i} \right)^{\frac{1}{2}}$$

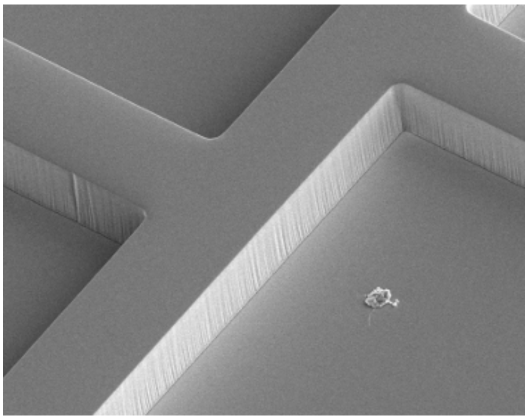
Fabrication of μTICs*

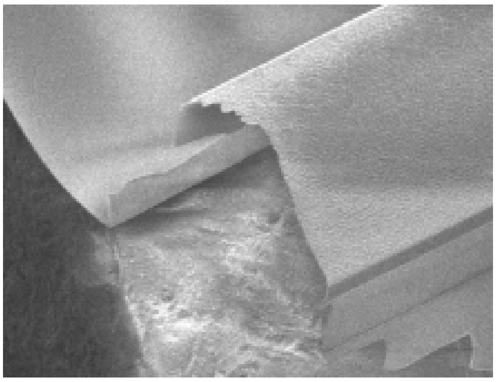
- RIE in silicon
- LPCVD 300 nm silicon nitride
- wafer bonding
- Si removal by KOH etching
- embedding in epoxy

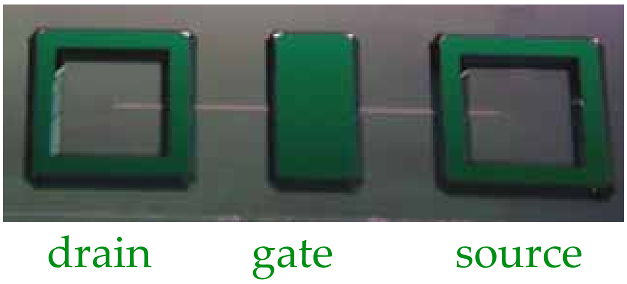


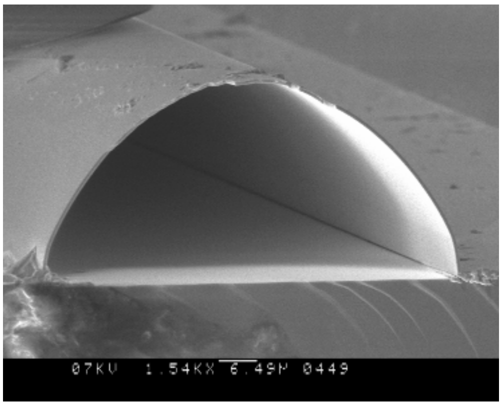
*TIC = Transparent Insulating Channel

μTIC devices

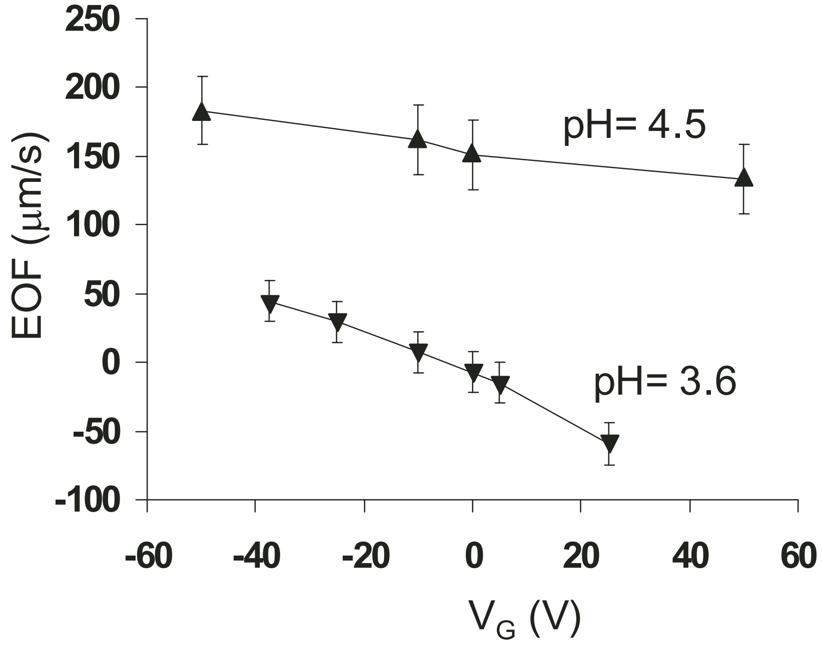






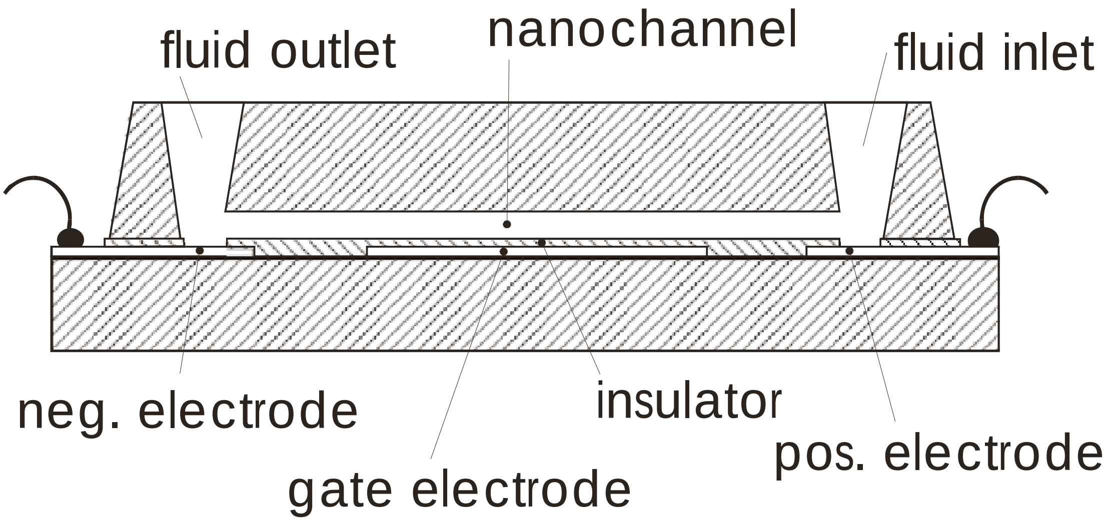


EOF as a function of Vg

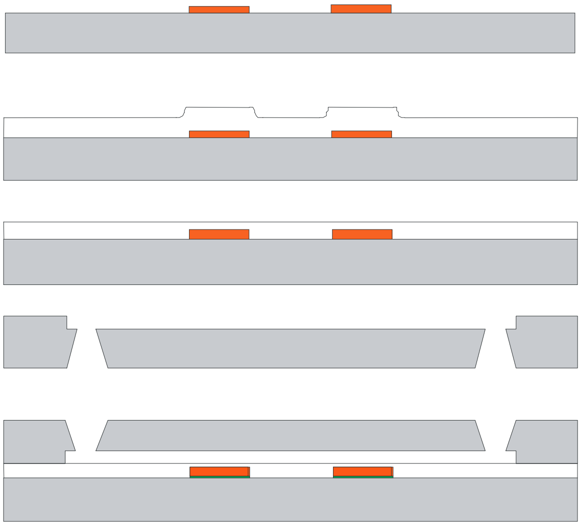


E= 30 V/cm

Field-effect flow control devices on glass



Fabrication technology



Electrode deposition

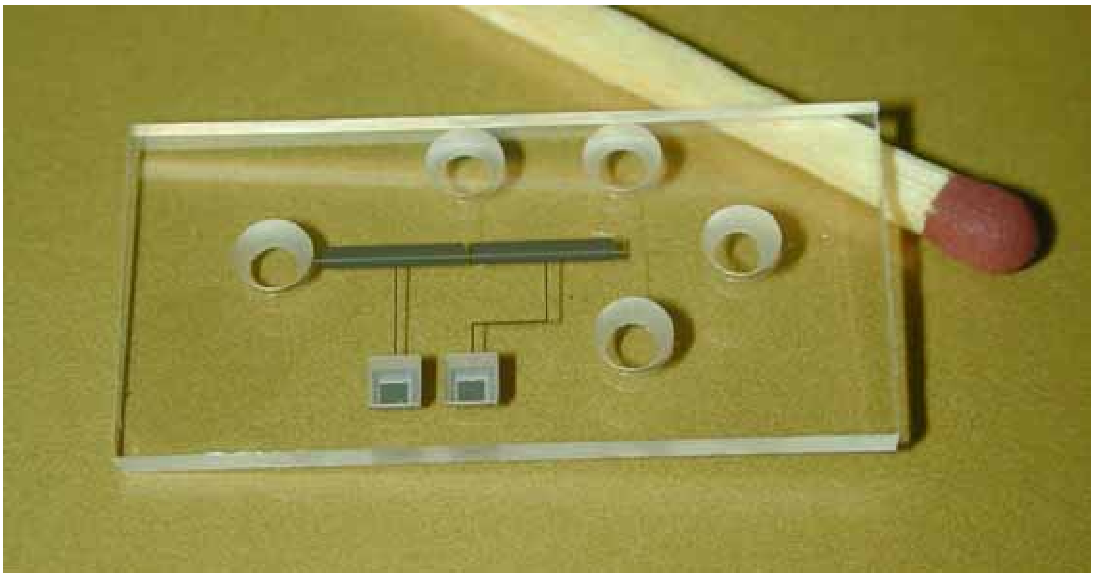
Deposition silica insulation

CMP

Channel etching

Direct bonding

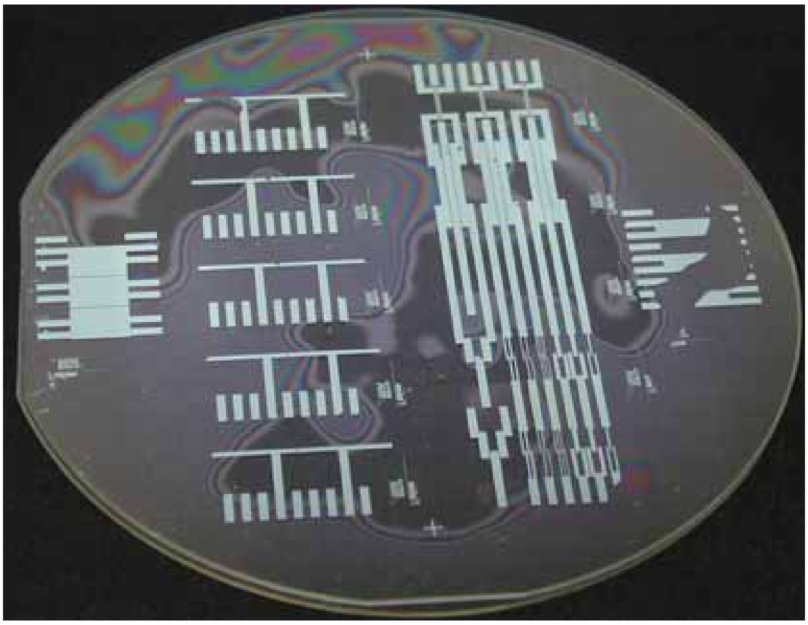
Device ok, but low yield



Main problem: dielectric breakdown of insulator film (PECVD SiO_2)

May be caused by high bonding temperature (600-650 ^{o}C)

Anodic bonding with intermediate silicon nitride layer

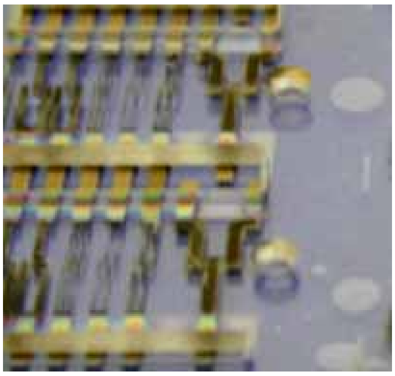


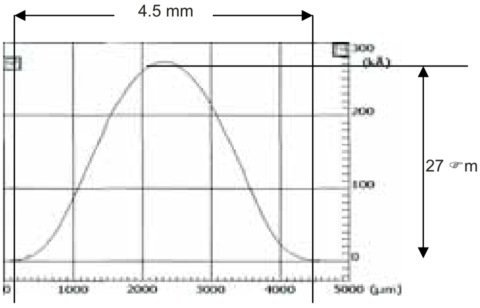
200 nm PECVD Si-nitride

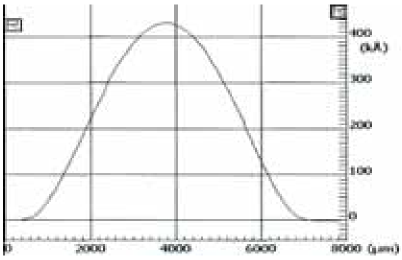
400 ºC, 1000 V, 1 hour

result: plates can be separated by knife

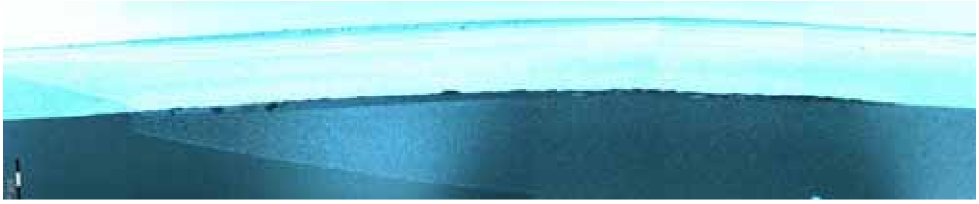
Voids





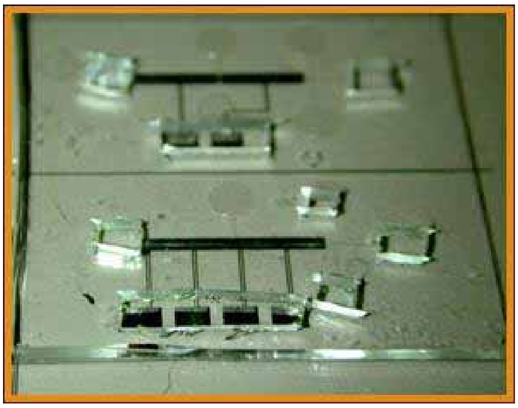


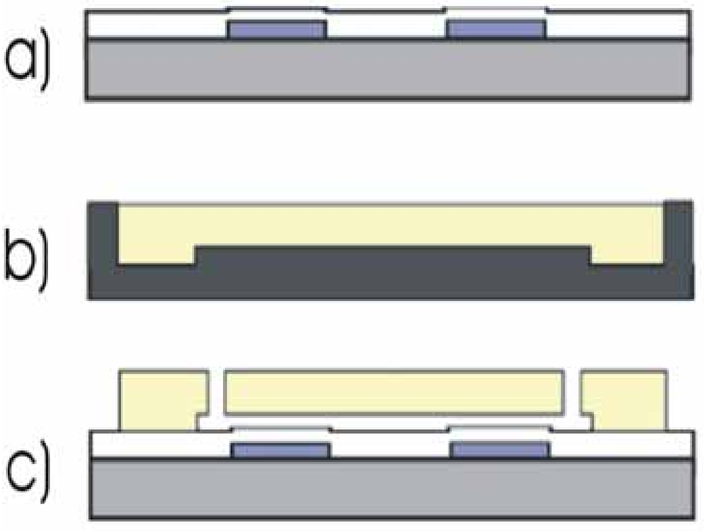
- water / air bubbles ?
- out gassing particles ?



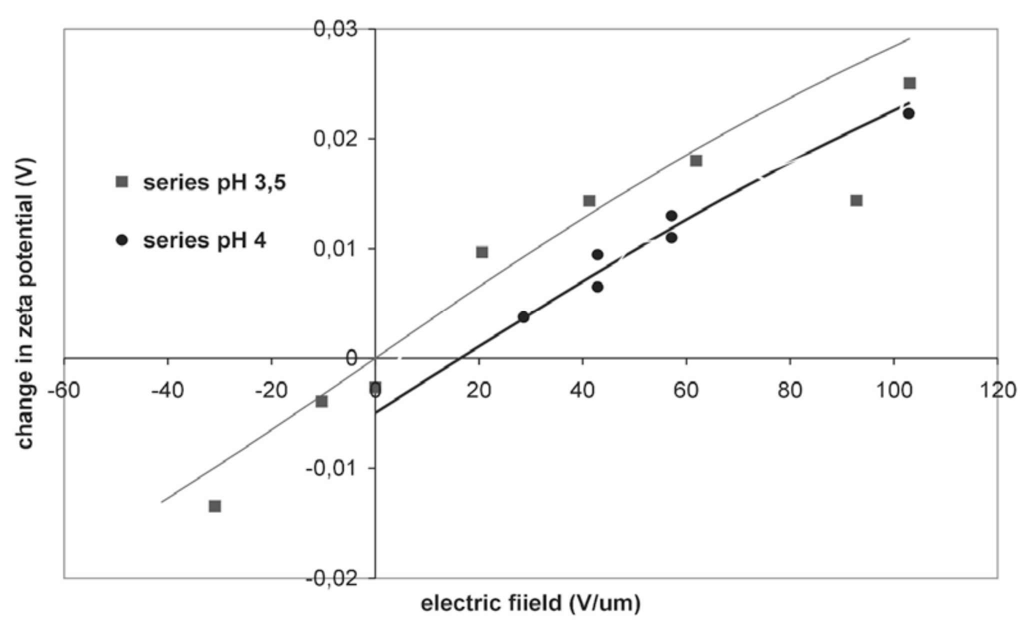
Combination of glass chip with electrodes and PDMS microchannels

PDMS was treated by oxygen plasma before bonding

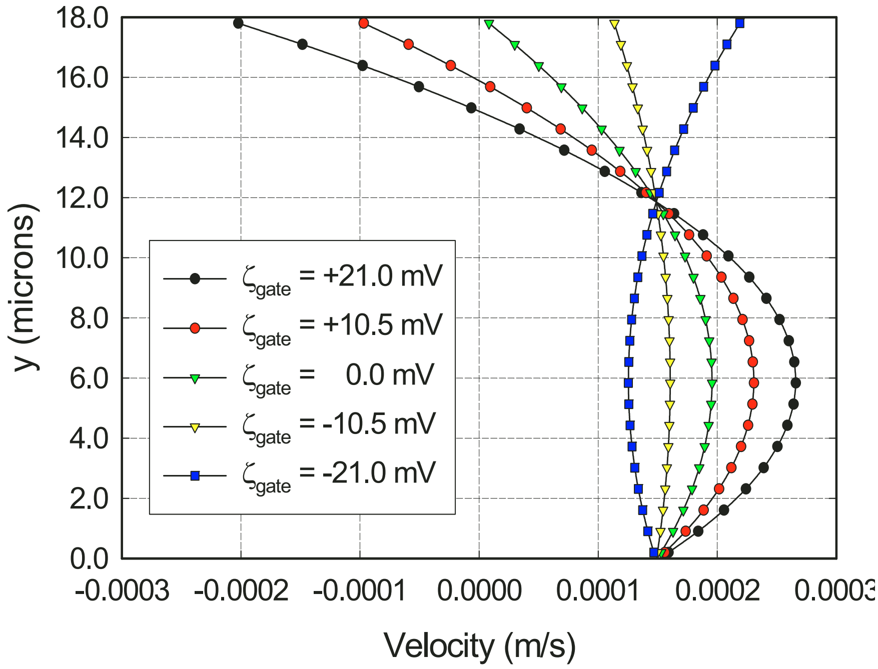




Influence gate potential on local zeta potential



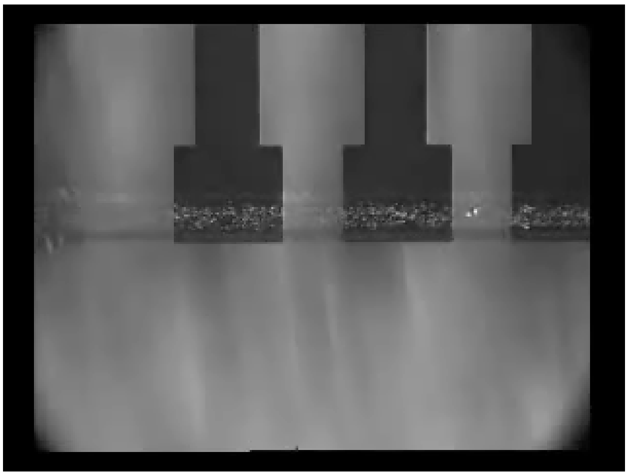
Velocity profiles under gate electrode for different zeta potentials



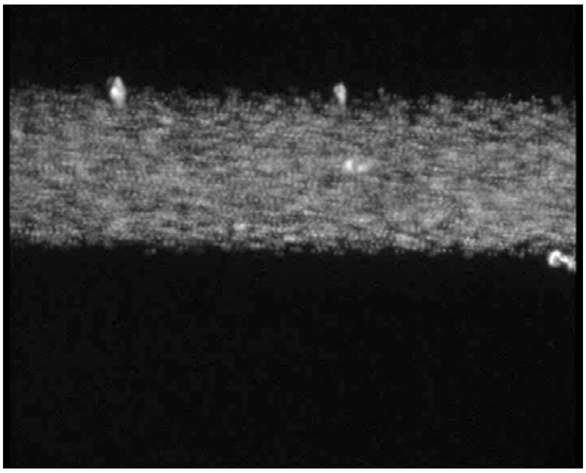
Robert Barber
and David Emerson

Centre for
Microfluidics and
Microsystems
Modelling (C3M)

Warrington

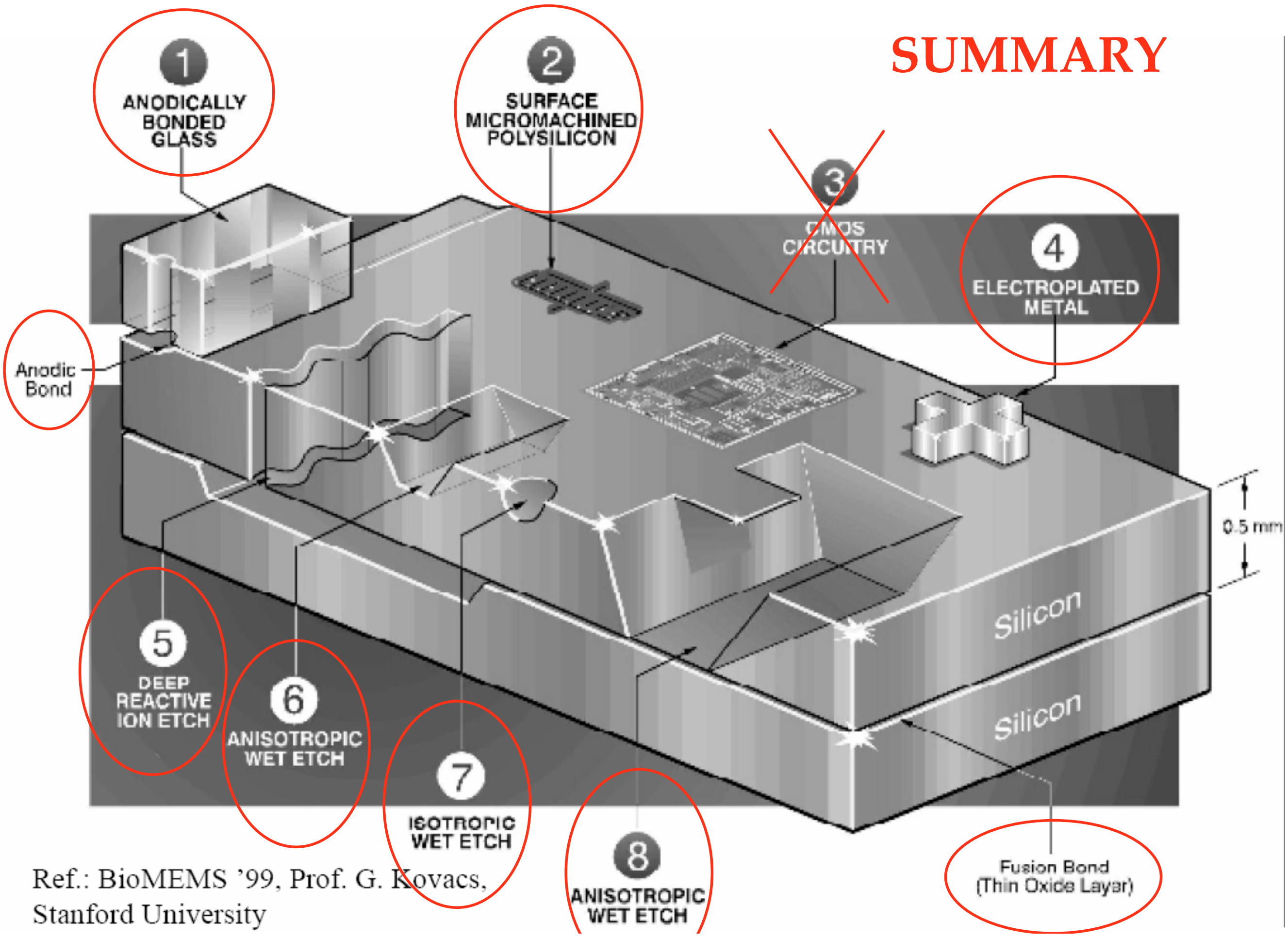


Gate DC voltage on-off



Gate AC voltage

SUMMARY



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

Recommended reading

Q.-Y. Tong and U. Gösele
Semiconductor wafer bonding: Science and Technology
VCH-Wiley, New York, 1999

Proceedings Electrochemical Society Meetings, held regularly, mainly under the title "Semiconductor Wafer Bonding"