
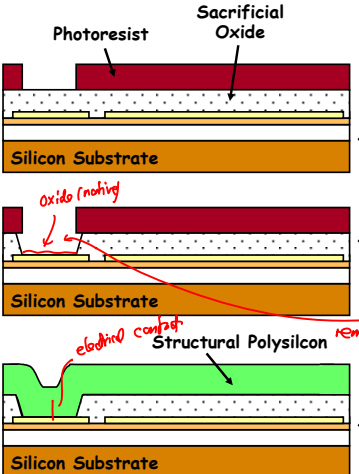



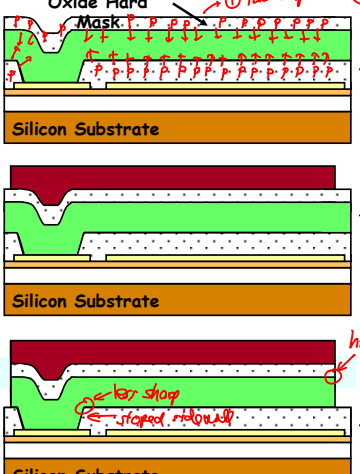
Surface-Micromachining Process Flow

- Deposit sacrificial PSG:
 - ☞ Target = 2µm
 - ☞ 1 hr. 40 min. LPCVD @450°C
- Densify the PSG
 - ☞ Anneal @950°C for 30 min.
- Lithography to define anchors using the ANCHOR(df) mask
 - ☞ Align to the poly1 layer
- Etch anchors
 - ☞ RIE using $\text{CHF}_3/\text{CF}_4/\text{He}$ @350W, 2.8Torr
 - ☞ Remove PR in PRS2000
 - ☞ Quick wet dip in 10:1 HF to remove native oxide
- Deposit structural polySi
 - ☞ Target = 2µm
 - ☞ In-situ Phosphorous-doped
 - ☞ 11 hrs. LPCVD @650°C

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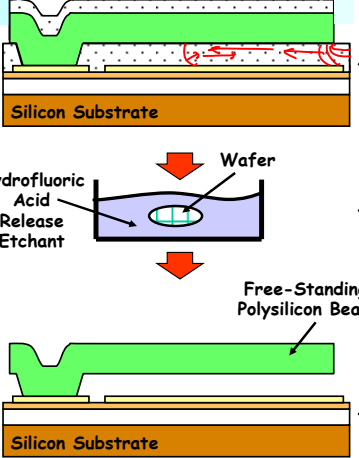
Surface-Micromachining Process Flow

- Deposit oxide hard mask
 - ☞ Target = 500nm
 - ☞ 25 min. LPCVD @450°C
- Stress Anneal
 - ☞ 1 hr. @ 1050°C
 - ☞ Or RTA for 1 min. @ 1100°C in 50 sccm N_2
- Lithography to define poly2 structure (e.g., shuttle, springs, drive & sense electrodes) using the POLY2(cf) mask
 - ☞ Align to the anchor layer
 - ☞ Hard bake the PR longer to make it stronger
- Etch oxide mask first
 - ☞ RIE using $\text{CHF}_3/\text{CF}_4/\text{He}$ @350W, 2.8Torr
- Etch structural polysilicon
 - ☞ RIE using $\text{CCl}_4/\text{He}/\text{O}_2$ @300W, 280mTorr
 - ☞ Use 1 min. etch/1 min. rest increments to prevent excessive temperature

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Surface-Micromachining Process Flow

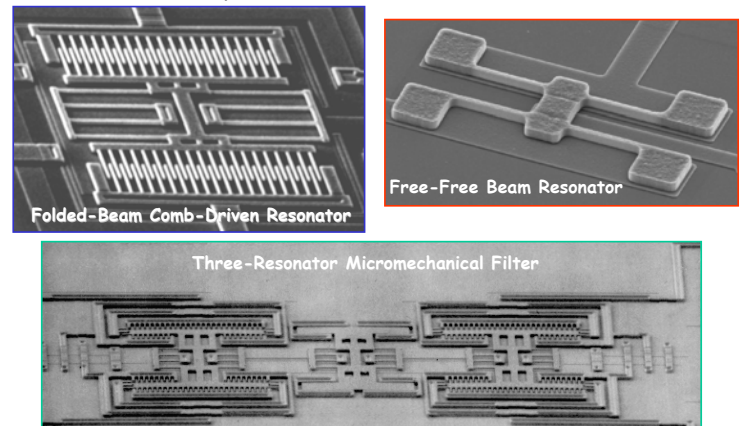
- Remove PR (more difficult)
 - ☞ Ash in O_2 plasma
 - ☞ Soak in PRS2000
- Release the structures
 - ☞ Wet etch in HF for a calculated time that insures complete undercutting
 - ☞ If 5:1 BHF, then ~ 30 min.
 - ☞ If 48.8 wt. % HF, ~ 1 min.
- Keep structures submerged in DI water after the etch
- Transfer structures to methanol
- Supercritical CO_2 dry release

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Polysilicon Surface-Micromachined Examples



• Below: All surface-micromachined in polysilicon using variants of the described process flow



Folded-Beam Comb-Driven Resonator

Free-Free Beam Resonator

Three-Resonator Micromechanical Filter

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Structural/Sacrificial Material Combinations

Structural Material	Sacrificial Material	Etchant
Poly-Si	SiO ₂ , PSG, LTO	HF, BHF
Al	Photoresist	O ₂ plasma
SiO ₂	Poly-Si	XeF ₂
Al	Si	TMAH, XeF ₂
Poly-SiGe	Poly-Ge	H ₂ O ₂ , hot H ₂ O

- Must consider other layers, too, as release etchants generally have a finite E.R. on any material
- Ex: concentrated HF (48.8 wt. %)
 - ↳ Polysilicon E.R. ~ 0
 - ↳ Silicon nitride E.R. ~ 1-14 nm/min
 - ↳ Wet thermal SiO₂ ~ 1.8-2.3 μm/min
 - ↳ Annealed PSG ~ 3.6 μm/min
 - ↳ Aluminum (Si rich) ~ 4 nm/min (much faster in other Al)

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Wet Etch Rates (f/ K. Williams)

Wet-Etch Rates for Micromachining and IC Processing (Acheson)

The top etch rate was measured by the authors with fresh solutions, etc. The center and bottom values are the low and high etch rates observed by the authors and others in our lab under less carefully controlled conditions.

MATERIALS

ETCHANT	TEMPERATURE	SUBSTRATE	Etch Rate [nm/min]	Etch Rate [nm/min]	Etch Rate [nm/min]	Etch Rate [nm/min]	Etch Rate [nm/min]	Etch Rate [nm/min]	Etch Rate [nm/min]	Etch Rate [nm/min]	Etch Rate [nm/min]	Etch Rate [nm/min]	Etch Rate [nm/min]	Etch Rate [nm/min]	Etch Rate [nm/min]
Concentrated HF (48.8%)	Room Temperature	Silicon nitride	~ 0	~ 230	100	70	150	100	100	100	100	100	100	100	100
10:1 HF	Room Temperature	Silicon nitride	~ 7	0	230	230	340	150	4700	11	3	2000	2000	126	
28:1 HF	Room Temperature	Silicon nitride	~ 0	0	91	95	130	W	1500	6	1	W	0		
5:1 HF	Room Temperature	Silicon nitride	~ 9	2	1000	1000	1200	600	4400	9	4	1400	<20	F	1000
Phosphoric Acid (85%)	Room Temperature	Silicon nitride	~ 7	~ 0.7	0.8	<1	37	24	28	19	9600	~	~	~	350
Silicon Etchant (120 HNO ₃ , 40 H ₂ O, 1 H ₂ O ₂)	Room Temperature	Silicon	1300	1100	1000	87	W	110	400	1700	2	3	4000	130	3000
KOH (1 KOH : 1 H ₂ O by weight)	Room Temperature	<100 Silicon	140	>10k	F	77	~	~	~	~	~	~	~	~	F
Aluminum Etchant Type A (56 H ₂ O, 1 HNO ₃ , 1 HCl, 1 H ₂ O ₂)	Room Temperature	Aluminum	<10	<9	0	0	0	~	<10	0	2	6000	~	~	~
Titanium Etchant (20 H ₂ O, 1 H ₂ O ₂ , 1 HF)	Room Temperature	Titanium	~ 12	~ 120	W	W	W	2100	8	4	W	0	8000	~	~
H ₂ O ₂ (30%)	Room Temperature	Tungsten	~ 0	0	0	0	0	0	0	0	0	<20	100	0	<2
Phenak (1:50 H ₂ PO ₄ : 1 H ₂ O)	Room Temperature	Cleaning off oxides and organics	~ 0	0	0	0	0	0	0	0	0	0	1000	~	~
Acidbase	Room Temperature	Phenolic	~ 0	0	0	0	0	0	0	0	0	0	0	0	>10k

Notes: ~ means not performed. W=not performed, but known to be Fast (≥ 100 nm/min); P=not performed, but known to be Fast (≥ 100 nm/min); ~ means of film profile during etch or where present. Arrows was visibly attacked and ruptured. Etch rates are all at a etch rate for the temperature listed and half of the volume for single-crystal silicon and the metals. Etch rates may vary with temperature and prior use of solution, area of exposure of film, other materials present (e.g., photoresist), film impurities and microstructures, etc. Some variation should be expected.

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Film Etch Chemistries


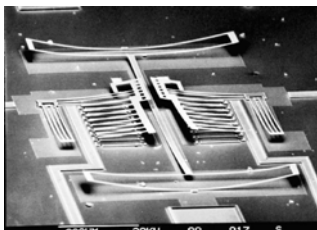

- For some popular films:

Material	Wet etchant	Etch rate [nm/min]	Dry etchant	Etch rate [nm/min]
Polysilicon	HNO ₃ :H ₂ O: NH ₄ F	120-600	SF ₆ + He	170-920
Silicon nitride	H ₃ PO ₄	5	SF ₆	150-250
Silicon dioxide	HF	20-2000	CHF ₃ + O ₂	50-150
Aluminum	H ₃ PO ₄ :HNO ₃ : CH ₃ COOH	660	Cl ₂ + SiCl ₄	100-150
Photoresist	Acetone	>4000	O ₂	35-3500
Gold	KI	40	n/a	n/a

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Issues in Surface Micromachining

- **Stiction:** sticking of released devices to the substrate or to other on-chip structures
 - ↳ Difficult to tell if a structure is stuck to substrate by just looking through a microscope
- **Residual Stress in Thin Films**
 - ↳ Causes bending or warping of microstructures
 - ↳ Limits the sizes (and sometimes geometries) of structures
- **Topography**
 - ↳ Stringers can limit the number of structural levels

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Microstructure Stiction

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Microstructure Stiction

- **Stiction: the unintended sticking of MEMS surfaces**
- **Release stiction:**
 - ↳ Occurs during drying after a wet release etch
 - ↳ Capillary forces of droplets pull surfaces into contact
 - ↳ Very strong sticking forces, e.g., like two microscope slides w/ a droplet between
- **In-use stiction: when device surfaces adhere during use due to:**
 - ↳ Capillary condensation
 - ↳ Electrostatic forces
 - ↳ Hydrogen bonding
 - ↳ Van der Waals forces

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Hydrophilic Versus Hydrophobic

contact angle

- **Hydrophilic:**
 - ↳ A surface that invites wetting by water
 - ↳ Get stiction
 - ↳ Occurs when the contact angle $\theta_{\text{water}} < 90^\circ$
- **Hydrophobic:**
 - ↳ A surface that repels wetting by water
 - ↳ Avoids stiction
 - ↳ Occurs when the contact angle $\theta_{\text{water}} > 90^\circ$

Lotus Surface [Univ. Mainz]

Hydrophilic case

Hydrophobic case

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Microstructure Stiction

Wetted Area A Force Applied to Maintain Equilibrium F Liquid Layer Thickness g Contact Angle θ_c

- Thin liquid layer between two solid plates \Rightarrow adhesive
- If the contact angle between liquid and solid $\theta_c < 90^\circ$:
 - ↳ Pressure inside the liquid is lower than outside
 - ↳ Net attractive force between the plates
- The pressure difference (i.e., force) is given by the Laplace equation

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Microstructure Stiction Modeling

Laplace Equation: Surface Tension @ the Liq.-Air Interface

$$\Delta p_{la} = \frac{2\gamma_{la}}{r}$$

r ← Radius of Curvature of the Meniscus (-) if concave

Pressure Difference @ the Liquid-Air Interface

$$[r = -\frac{(g/2)}{\cos\theta_c}] \Rightarrow F = -\Delta p_{la} A = \frac{2A\gamma_{la}\cos\theta_c}{g}$$

Force needed to keep the plates apart
 \Rightarrow (+) force means g
 \Rightarrow (-) Laplace pressure

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Avoiding Stiction

- Reduce droplet area via mechanical design approaches

- Avoid liquid-vapor meniscus formation
 - Use solvents that sublime
 - Use vapor-phase sacrificial layer etch
- Modify surfaces to change the meniscus shape from concave (small contact angle) to convex (large contact angle)
 - Use teflon-like films
 - Use hydrophobic self-assembled monolayers (SAMs)

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Supercritical CO₂ Drying

- A method for stictionless drying of released microstructures by immersing them in CO₂ at its supercritical point
- Basic Strategy:** Eliminate surface tension-derived sticking by avoiding a liquid-vapor meniscus
- Procedure:**
 - Etch oxide in solution of HF
 - Rinse thoroughly in DI water, but do not dry
 - Transfer the wafer from water to methanol
 - Displace methanol w/ liquid CO₂
 - Apply heat & pressure to take the CO₂ past its critical pt.
 - Vent to lower pressure and allow the supercritical CO₂ to revert to gas → liquid-to-gas Xsition in supercritical region means no capillary forces to cause stiction

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Hydrophilic Versus Hydrophobic

- Hydrophilic:**
 - A surface that invites wetting by water
 - Get stiction
 - Occurs when the contact angle $\theta_{water} < 90^\circ$
- Hydrophobic:**
 - A surface that repels wetting by water
 - Avoids stiction
 - Occurs when the contact angle $\theta_{water} > 90^\circ$

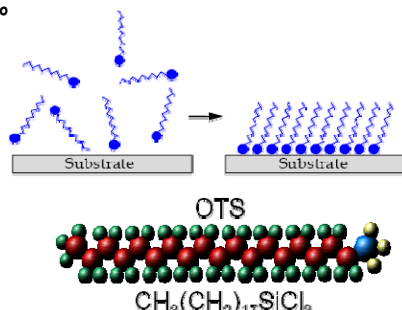
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Tailoring Contact Angle Via SAM's

- Can reduce stiction by tailoring surfaces so that they induce a water contact angle $> 90^\circ$

Self-Assembled Monolayers (SAM's):

- Monolayers of "stringy" molecules covalently bonded to the surface that then raise the contact angle
- Beneficial characteristics:
 - Conformal, ultrathin
 - Low surface energy
 - Covalent bonding makes them wear resistant
 - Thermally stable (to a point)



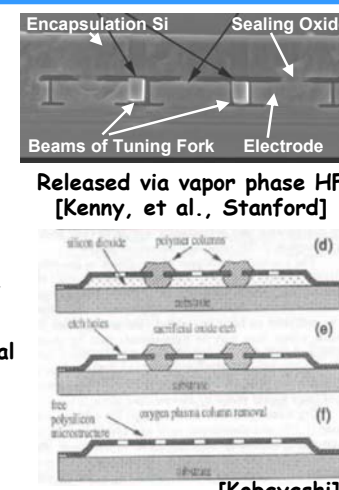
OTS
 $\text{CH}_3(\text{CH}_2)_{17}\text{SiCl}_3$

	θ_{water}
ODT SAM	$112 \pm 0.7^\circ$
SiO_2	$< 10^\circ$

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Dry Release

- Another way to avoid stiction is to use a dry sacrificial layer etch
- For an oxide sacrificial layer
 - use HF vapor phase etch
 - Additional advantage: gas can more easily get into tiny gaps
 - Issue: not always completely dry \rightarrow moisture can still condense \rightarrow stiction \rightarrow soln: add alcohol
- For a polymer sacrificial layer
 - Use an O_2 plasma etch (isotropic, so it can undercut well)
 - Issues:
 - Cannot be used when structural material requires high temperature for deposition
 - If all the polymer is not removed, polymer under the suspended structure can still promote stiction



Released via vapor phase HF [Kenny, et al., Stanford]

[Kobayashi]

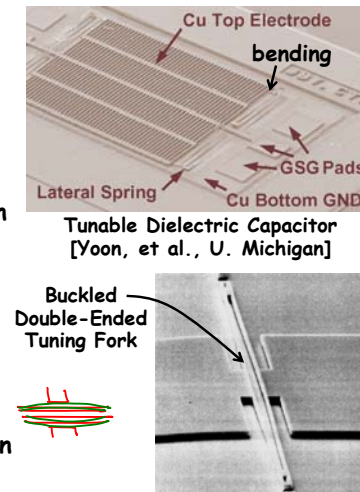
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Residual Stress

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Residual Stress in Thin Films

- After release, poorly designed microstructures might buckle, bend, or warp \rightarrow often caused by residual film stress
- Origins of residual stress, σ
 - Growth processes
 - Non-equilibrium deposition
 - Grain morphology change
 - Gas entrapment
 - Doping
 - Thermal stresses
 - Thermal expansion mismatch of materials \rightarrow introduce stress during cool-down after deposition
 - Annealing



Tunable Dielectric Capacitor [Yoon, et al., U. Michigan]

Buckled Double-Ended Tuning Fork

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Need to Control Film Stress

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- Resonance frequency expression for a lateral resonator:

$$f_0 \approx \frac{1}{2\pi} \sqrt{\frac{4E_y t W^3}{ML^3} + \frac{24\sigma_r t W}{5ML}}$$

↑ Basic term ↑ Stress term

Since $W \ll L$, the stress term will dominate if $\sigma_r \sim E_y$

E_y = Young's modulus
 σ_r = stress
 t = thickness
 W = beam width
 L = beam length
 M = mass

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Tensile Versus Compressive Stress

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- Under tensile stress, a film wants to shrink w/r to its substrate
 - Caused, e.g., by differences in film vs. substrate thermal expansion coefficients
 - If suspended above a substrate and anchored to it at two points, the film will be "stretched" by the substrate
- Under compressive stress, a film wants to expand w/r to its substrate
 - If suspended above a substrate and anchored to it at two points, the film will buckle over the substrate

tensile film

compressive film buckled

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Vertical Stress Gradients

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- Variation of residual stress in the direction of film growth
- Can warp released structures in z-direction

200µm 20KV 00 017 S

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Stress in Polysilicon Films

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- Stress depends on crystal structure, which in turn depends upon the deposition temperature
- Temperature $\leq 600^\circ\text{C}$
 - Films are initially amorphous, then crystallize
 - Get equiaxed crystals, largely isotropic
 - Crystals have higher density \rightarrow tensile stress
 - Small stress gradient
- Temperature $\geq 600^\circ\text{C}$
 - Columnar crystals grow during deposition
 - As crystals grow vertically and in-plane they push on neighbors \rightarrow compressive stress
 - Positive stress gradient

high T

low T

high T

low T

same film (but the substrate shrinks)

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Annealing Out Polysilicon Stress

- Control polySi stress by annealing at high temperatures
 - ↳ Typical anneal temperatures: 900-1150°C
 - ↳ Grain boundaries move, relax
 - ↳ Can dope while annealing by sandwiching the polysilicon between similarly doped oxides (symmetric dopant drive-in), e.g. using 10-15 wt. % PSG
- Rapid thermal anneal (RTA) also effective (surprisingly)

The left graph, titled 'Anneal Curves for Polysilicon', plots Tensile and Compression (strain) against Time in minutes. It shows four curves for different annealing temperatures: 700°C, 850°C, 950°C, and 1550°C. The 1550°C curve shows a sharp drop in tensile strain and a corresponding increase in compression strain over time. The right graph plots Stress in MPa against the PH₃ / SiH₄ Mole Ratio on a logarithmic scale. It shows three curves for different temperatures: 560°C, 590°C, and 610°C. The stress decreases as the mole ratio increases and as the temperature increases. A label 'RTA 900°C / 60 s' is present in the upper right of the graph.

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