Fast Piezoresistive Probes for High Bandwidth Biology

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Talk Overview

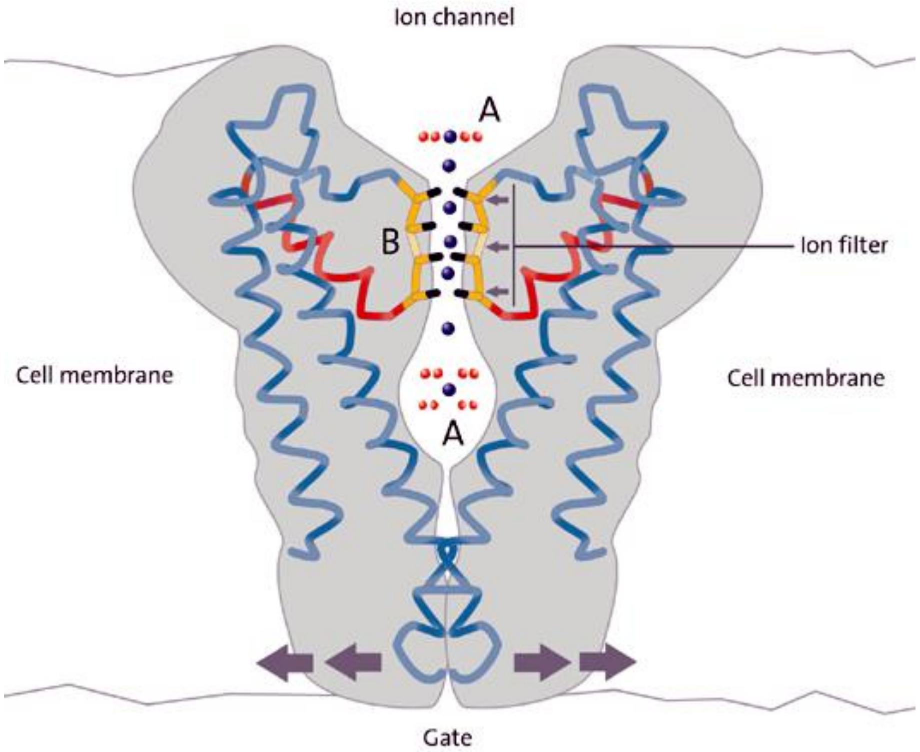
- **What is Mechanotransduction?**
- Microscale Force Sensing
- Design and Fabrication
- Aluminum nitride: review, results and applications
- Goals

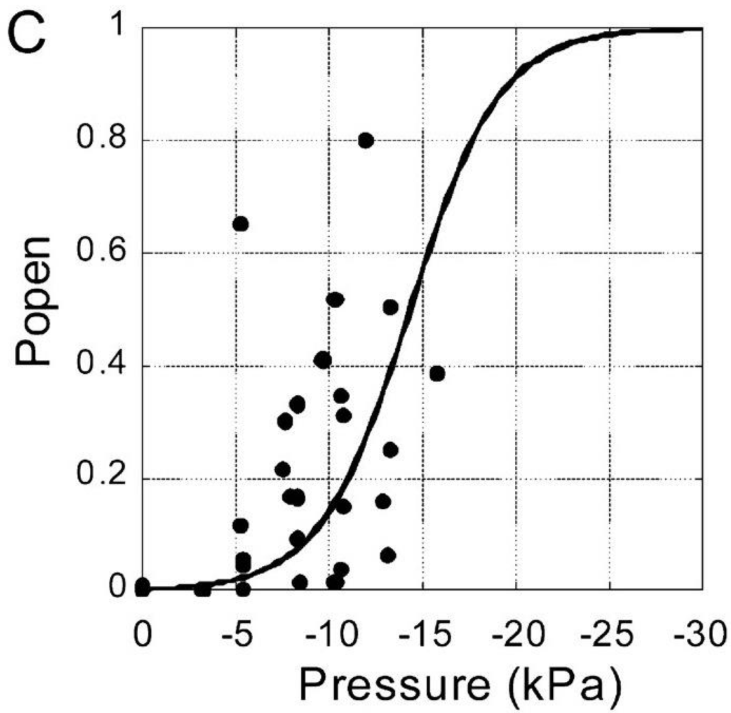
Mechanotransduction and Life

Images from images.google.com

Transformation of mechanical energy into an electrochemical signal

Cells and Ion Channels



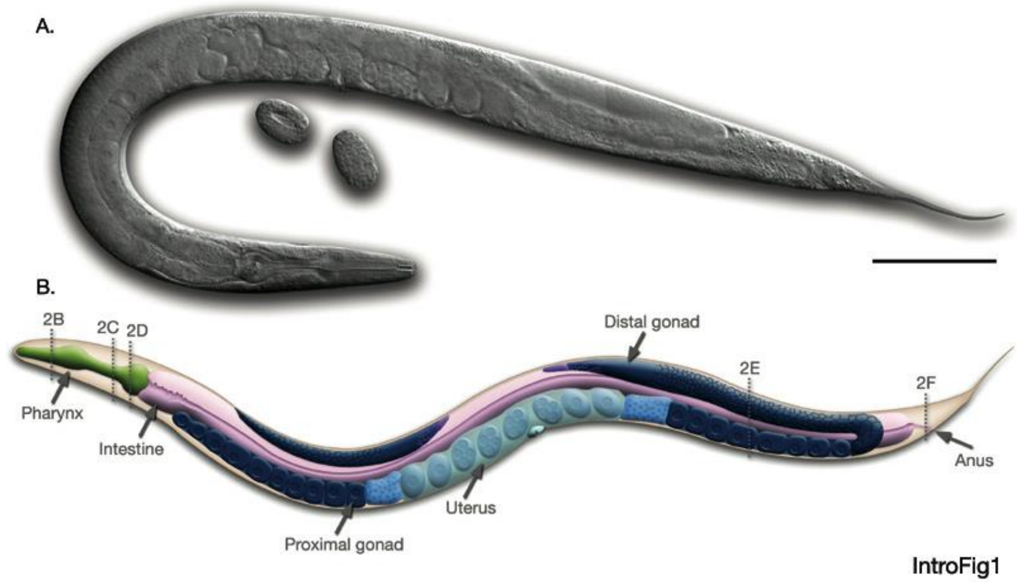


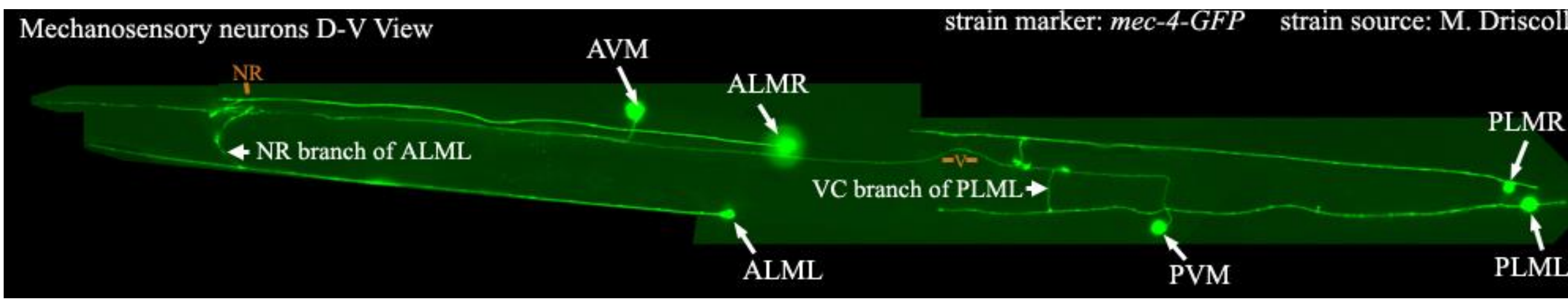
Charras et al, Biophys Journal (2004)

http://fig.cox.miami.edu/~cmallery/150/memb/ion_channel_lg1.jpg

Caenorhabditis elegans

- C. elegans
  - 1000 somatic cells
  - 302 neurons
  - 50 µm x 1 mm in size

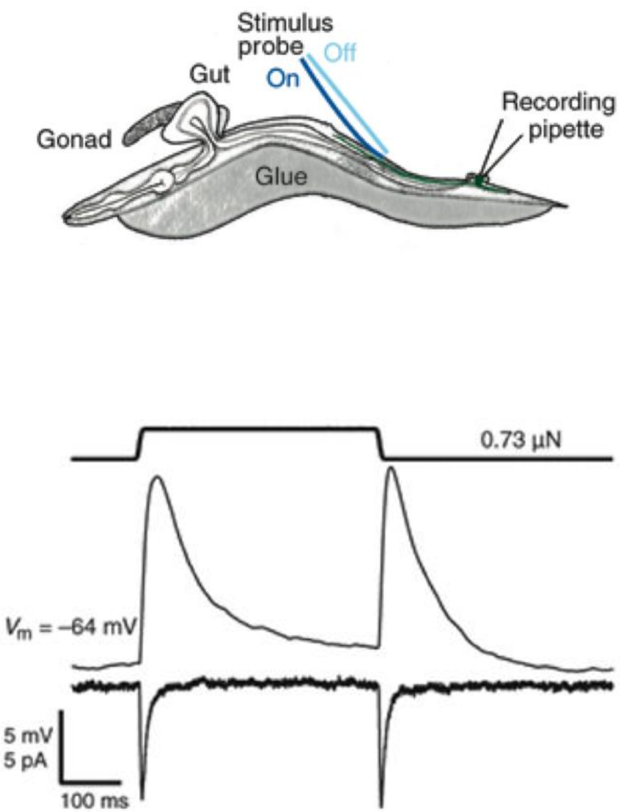


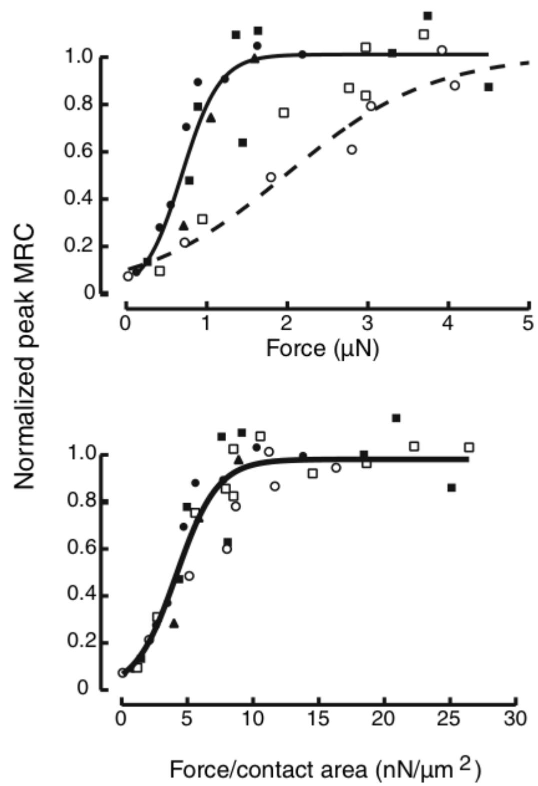


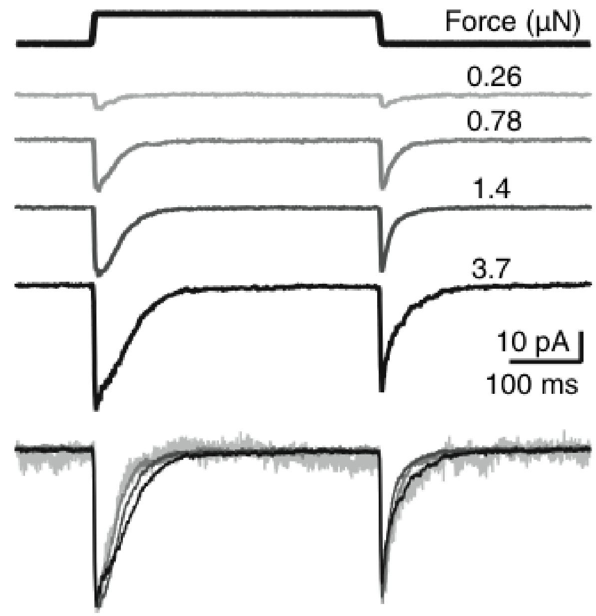
http://www.wormatlas.org/cellid/bodymechsen.htm

http://www.wormbook.org

Touch Receptor Neuron Physiology





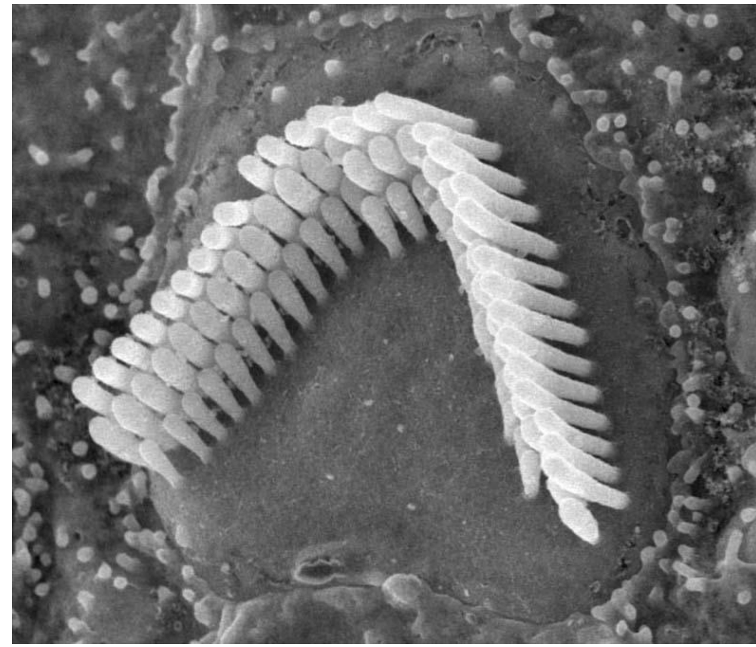


(open loop force control)

The MEC-4 DEG/ENaC channel of Caenorhabditis elegans touch receptor neurons transduces mechanical signals, O'Hagan et al, Nature Neuroscience (2005)

The Need for High Speed Sensing

- Rapid (10μs) force transduction by hair cells in the inner ear and touch receptor neurons
- Combine actuation and force sensing on-chip to increase bandwidth
- Piezoelectrically actuated cantilevers with piezoresistive force sensing
- Other applications, e.g. chemical sensing, scanning probe microscopy



Hair Cell SEM: http://www.neuroscience.cam.ac.uk

Performance Goals

- 100 pN force resolution in 1 Hz – 50 kHz bandwidth
- 10 microsecond rise time, 500nm tip displacement
- Closed loop force control
- Operation in fluid

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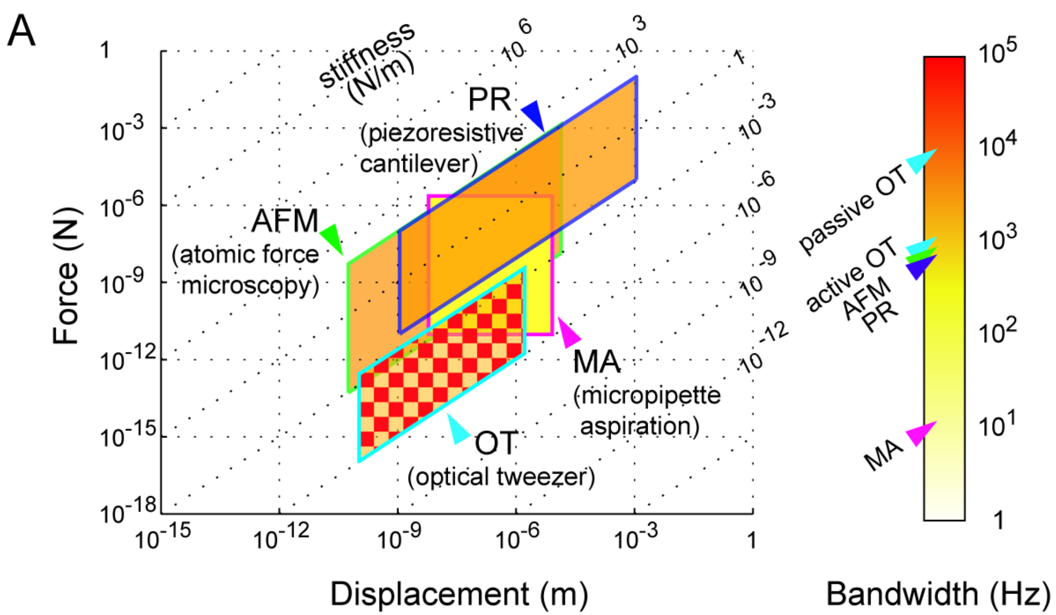
Other Force Sensing Techniques



Park et al, PNAS 2007

Piezoresistance

- Strain gauge: change in resistance with change in length
- Define gauge factor (GF)

$$GF = \frac{dR/R}{dL/L} = \frac{dR/R}{\varepsilon} = \frac{d\rho/\rho}{dL/L} + 1 + 2\nu$$

- Metal strain gauges: resistivity doesn't change so GF ~= 2
- For semiconductors…

$$\rho = \frac{1}{q(n\mu_n + p\mu_p)}$$

- Deformation of the crystal lattice under applied stress changes its energy band structure, altering carrier mobility and therefore resistivity

Slide modified from Michael Bartsch, 2004

Piezoresistivity in Silicon

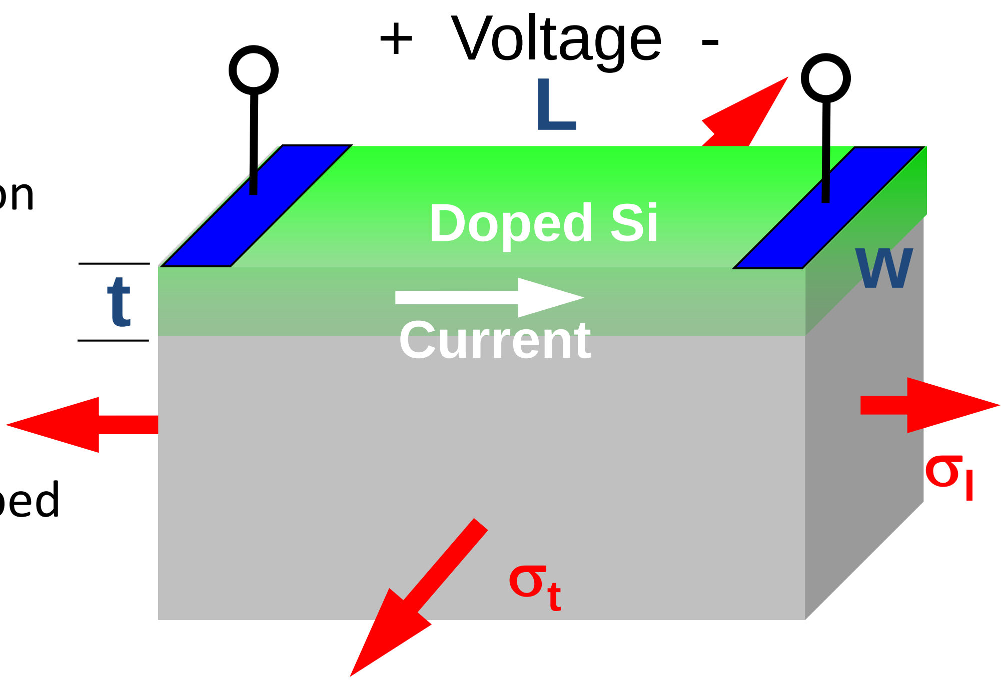
- Resistor elements fabricated by selectively introducing impurities (doping)
- Resistance of a doped silicon region is given by

$$R = \rho \frac{L}{wt}$$

- In piezoresistive materials like doped Si, ρ is stress-dependent

$$\frac{\Delta\rho}{\rho} = \pi_l \sigma_l + \pi_t \sigma_t$$

- Where π_l is the **longitudinal** piezoresistive coefficient
- And π_t is the **transverse** piezoresistive coefficient



The simplest case: piezoresistors patterned so current flows in the direction of the uniaxial stress - only longitudinal components need to be considered.

Slide modified from Michael Bartsch, 2004

Silicon Strain Gauges?

- Strain dependencies may be derived for a silicon resistor

$$GF = \frac{dR/R}{\varepsilon} = \frac{E}{\sigma}\frac{d\rho}{\rho} + 1 + 2\nu$$

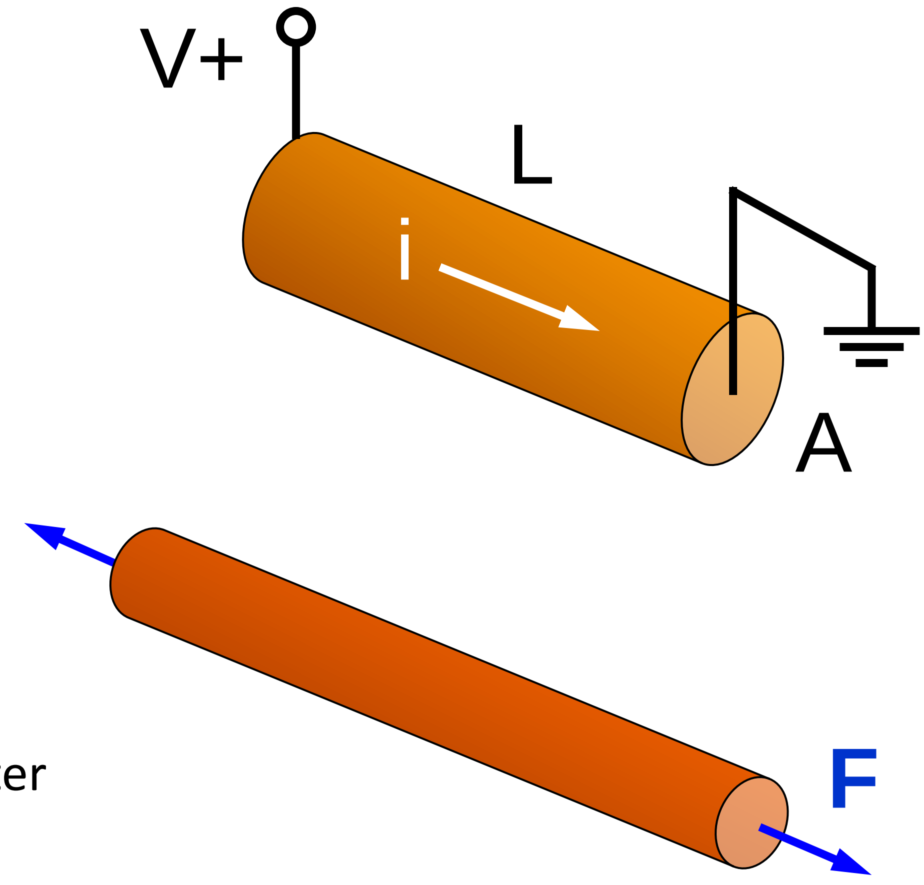
- But since $d\rho/\rho = \pi_l \sigma_l$

$$GF = E\pi_l + 1 + 2\nu$$

- With π_l of $100\text{x}10^{-11}\text{m}^2/\text{N}$ and E_{Si}= $190\text{x}10^9$Pa

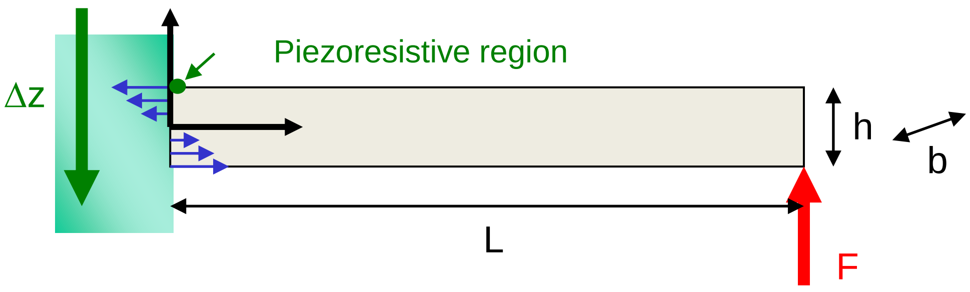
$$GF = E\pi_l + 1 + 2\nu \approx 190$$

- So the gauge factor of a Si gauge is nearly two orders of magnitude better than a metal wire!



Slide modified from Michael Bartsch, 2004

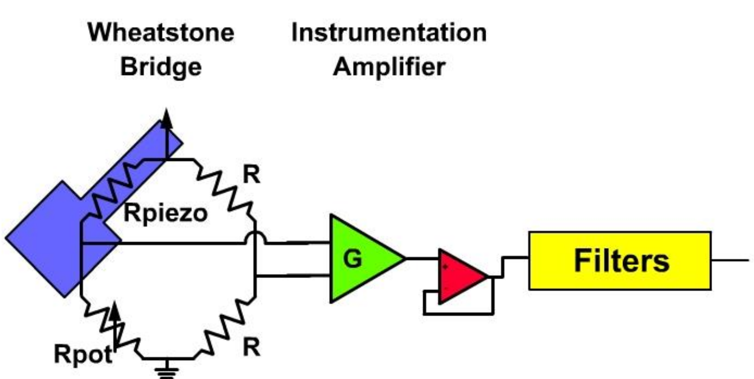
Piezoresistive Force Sensing



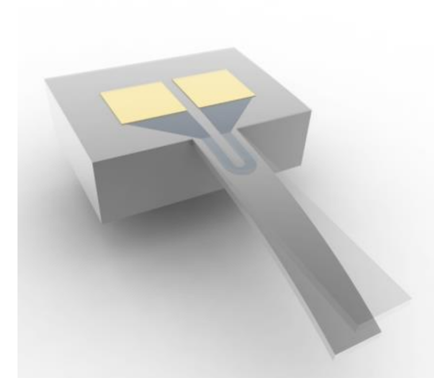
$$F = k\Delta z \qquad \left(k = \frac{3EI}{L^3} = \frac{Ebh^3}{4L^3}\right)$$

Δz → **Stiffness** → F, σ → **Piezoresistivity** → ΔR → **Wheatstone Bridge** → ΔV

$$V_{out} \cong G\frac{V_B \Delta R}{4R} \cong G\frac{3V_B \pi_L L}{2bh^2} F = GSF \qquad \left(S = \frac{3V_B \pi_L L}{2bh^2}\right)$$



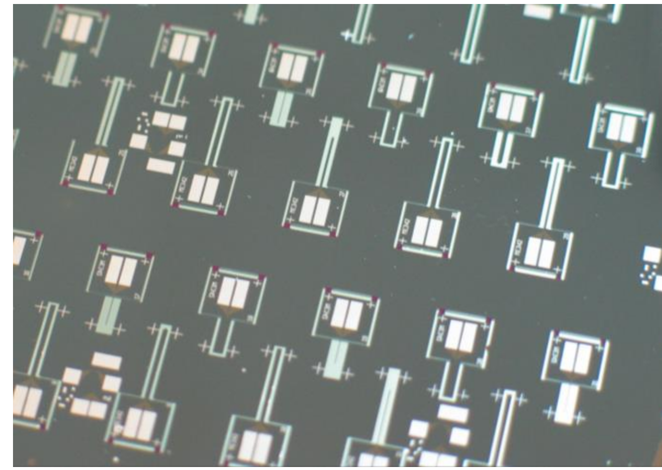
$$\frac{\Delta R}{R} \cong \pi_L \sigma \cong \frac{6\pi_L L}{bh^2} F$$



Slide modified from Sung-Jin Park, 2007

Piezoresistive Force Sensing

- Orientation is critical
- Electronic force sensing (no optics)
- Piezoresistivity varies with doping, temperature, light
- Design should maximize stress
- Small setup area + integration with other systems
- Fabrication
  - Ion implantation (thick cantilevers)
  - Epitaxy, diffusion (thin cantilevers)



Example

Width: 80~400um

Length: 2000~6000um

Thickness: 15um

k: 0.1~2N/m

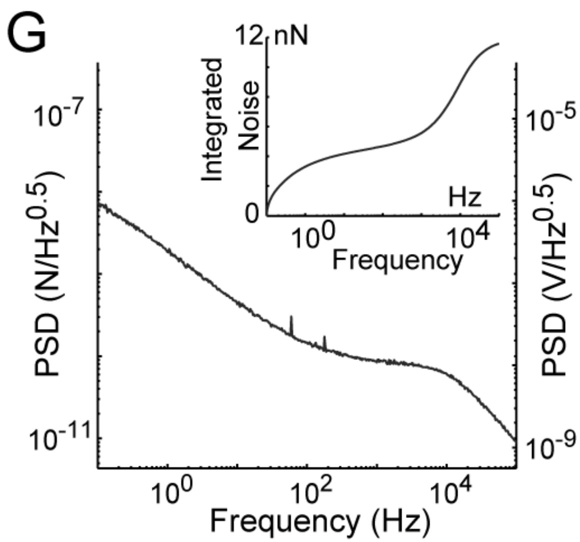
ω: 0.6~5.3 kHz

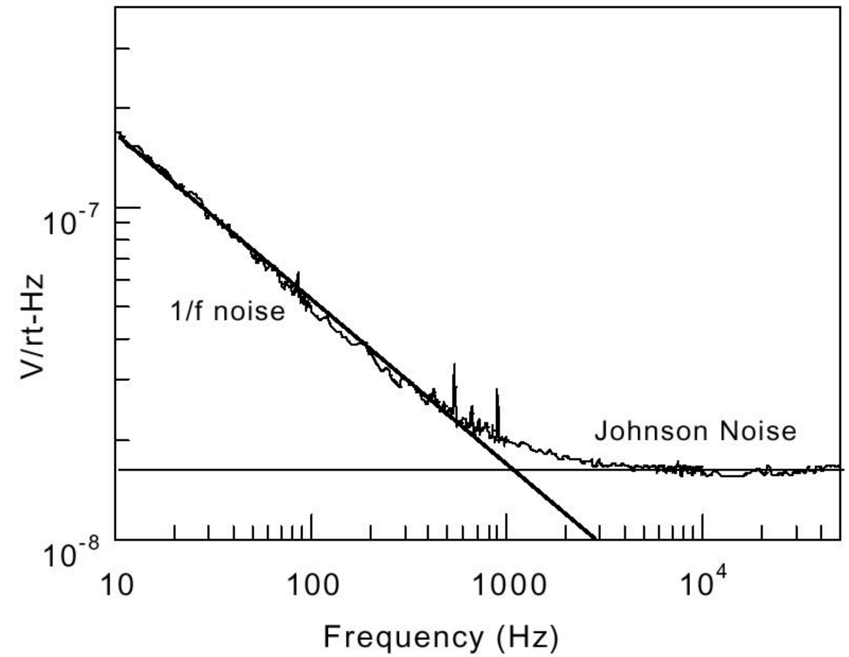
S: 3000-21000 uN/V

Slide modified from Sung-Jin Park, 2007

Force Resolution

- Sensitivity and noise trade off with process, geometry
  - Johnson noise
  - 1/f noise
  - Amplifier and background

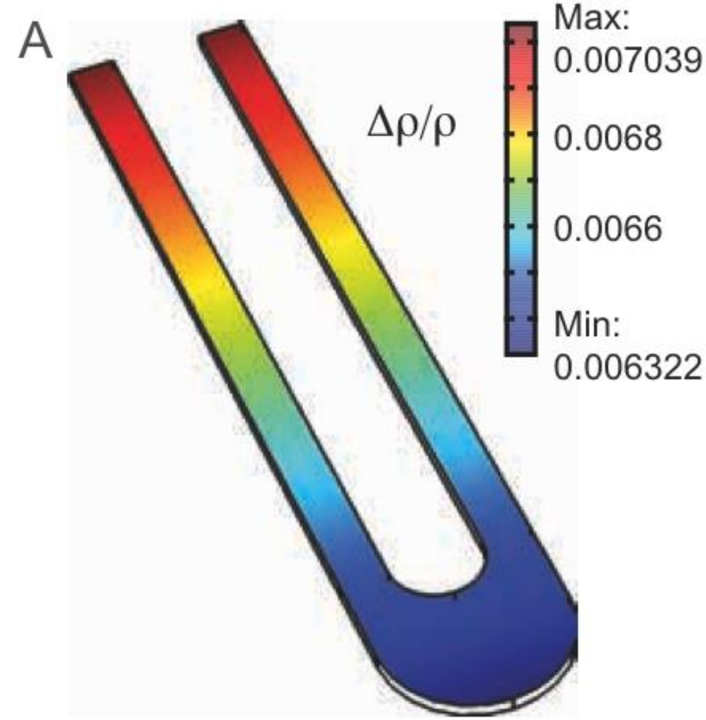




$$\sigma_{\min} = \frac{\sqrt{4kRT\left(f_{\max} - f_{\min}\right) + \frac{\alpha V_b^2}{fN}\ln\left(\frac{f_{\max}}{f_{\min}}\right) + S_b}}{\frac{V_b \bar{\pi}}{2}}$$

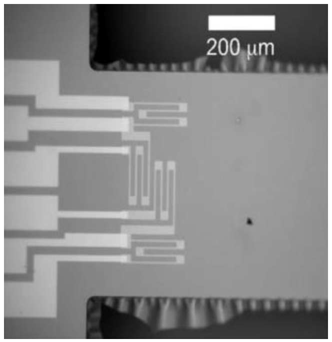
Piezoresistor Design

- Choices
  - Cantilever dimensions
  - Piezoresistor dimensions and process parameters
  - Dopant concentration
  - Bias voltage
- Given constraints
  - Frequency range
  - Measurement bandwidth

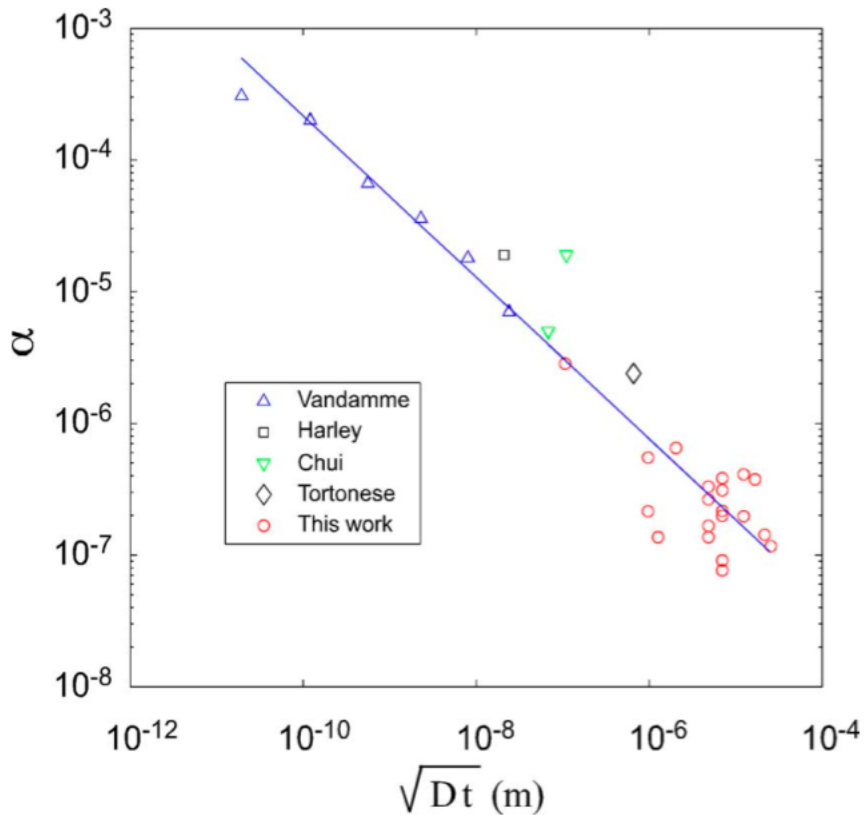


Park et al, Hilton Head 2008

Example: Low frequency design



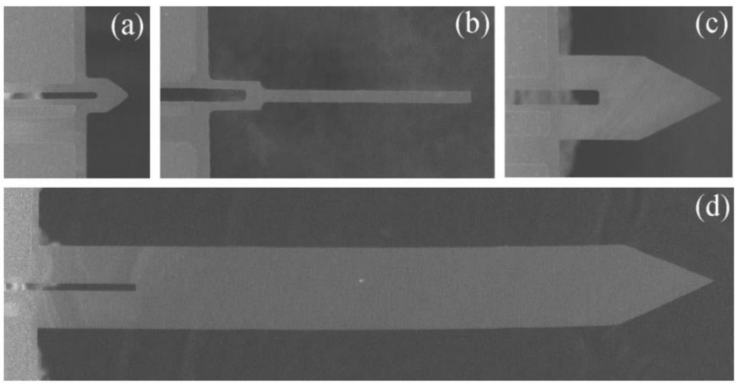
	This work	
	Full active bridge	1/4-active bridge[a]
Boron dose (cm^{-2})	5×10^{16}	1×10^{14}
Peak concentration (cm^{-3})	2.7×10^{19}	6.2×10^{17}
Anneal temperature (°C)	1100	1000
Anneal time (min)	50	52
Spring constant (N/m)	2.1	17
Sensitivity (V/N)	330	179
ω_n (kHz)	1.7	3.7
Resistance (kΩ)	1.8	16.8
Johnson noise (nV/$\sqrt{}$Hz)	5	16
Corner frequency (Hz)	0.6	20
$1/f$ noise at:		
10 Hz (nV/$\sqrt{}$Hz)	5[h]	22
10 Hz (nV/V $\sqrt{}$Hz)	0.4[h]	6
1 Hz (nV/V $\sqrt{}$Hz)	1.2[h]	20
0.1 Hz (nV/V $\sqrt{}$Hz)	3.7	NA



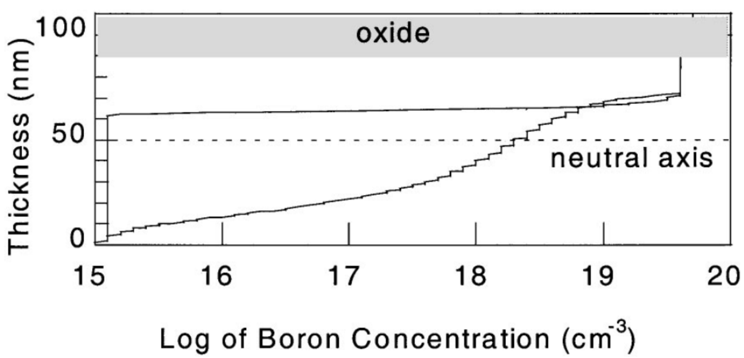
- 140dB dynamic range
- 100pN resolution from 0.1Hz to 100Hz

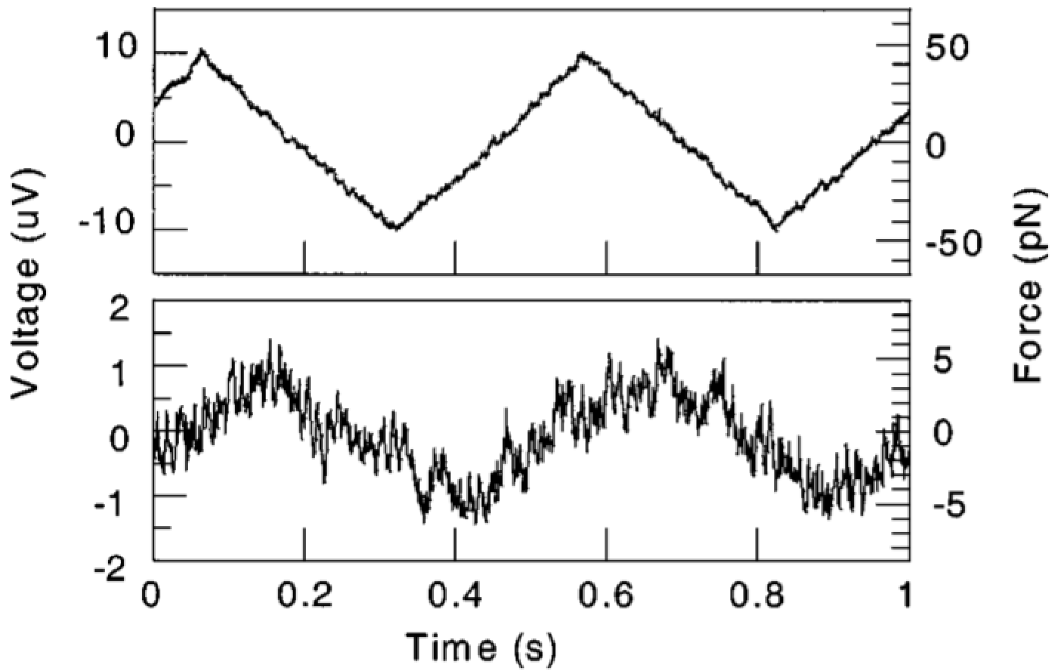
Low 1/f noise full bridge microcantilever with longitudinal and transverse piezoresistors, Mallon et al, APL (2008)

Example: High frequency design



- •89nm thick
- •500fN resolution from 10Hz to 1kHz





High-sensitivity piezoresistive cantilevers under 1000A thick, Harley and Kenny, APL (1999)

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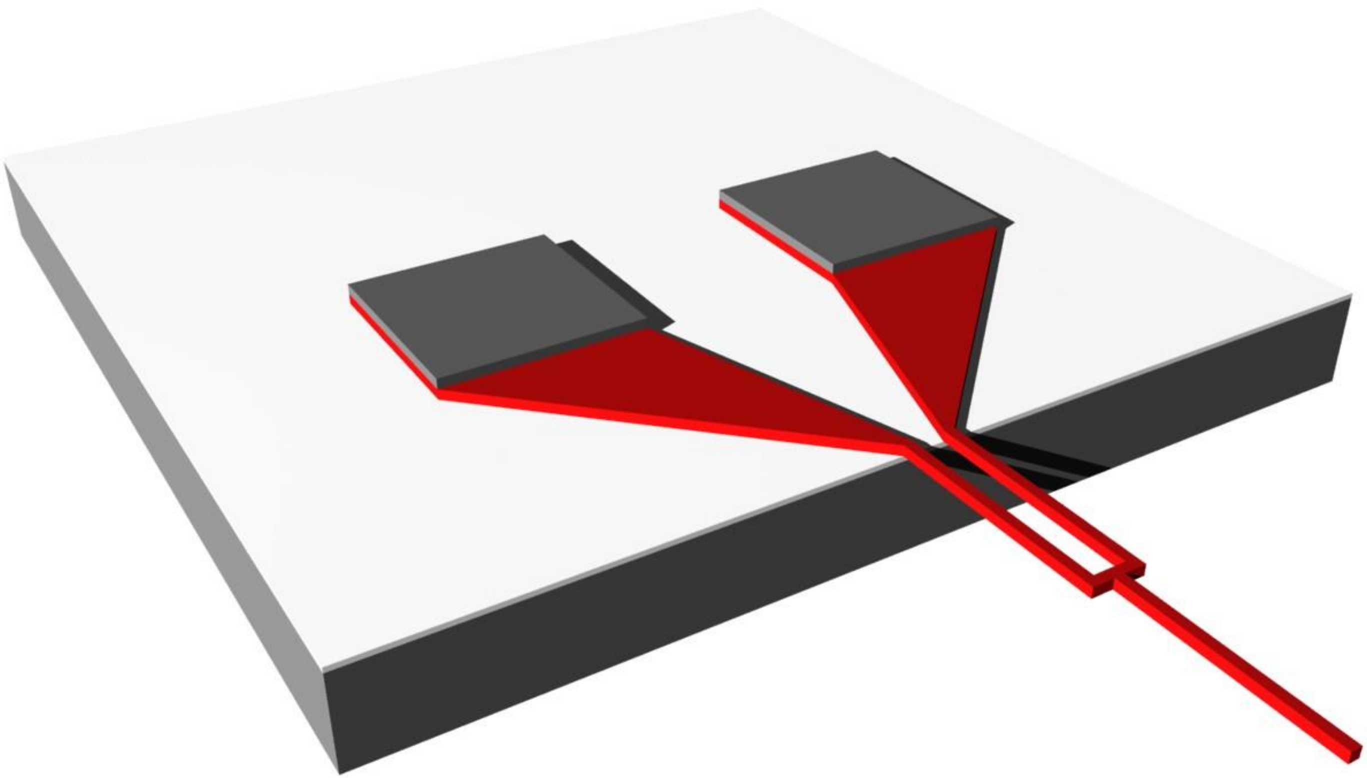
Fabrication Overview

- Force sensing: piezoresistive effect in silicon
- Actuation: aluminum nitride
- Fabrication starting from SOIs
- Device layers in 300nm – 4um range
- Parylene N for passivation
- Fabrication strategy: PR only, PE only, PR+PE

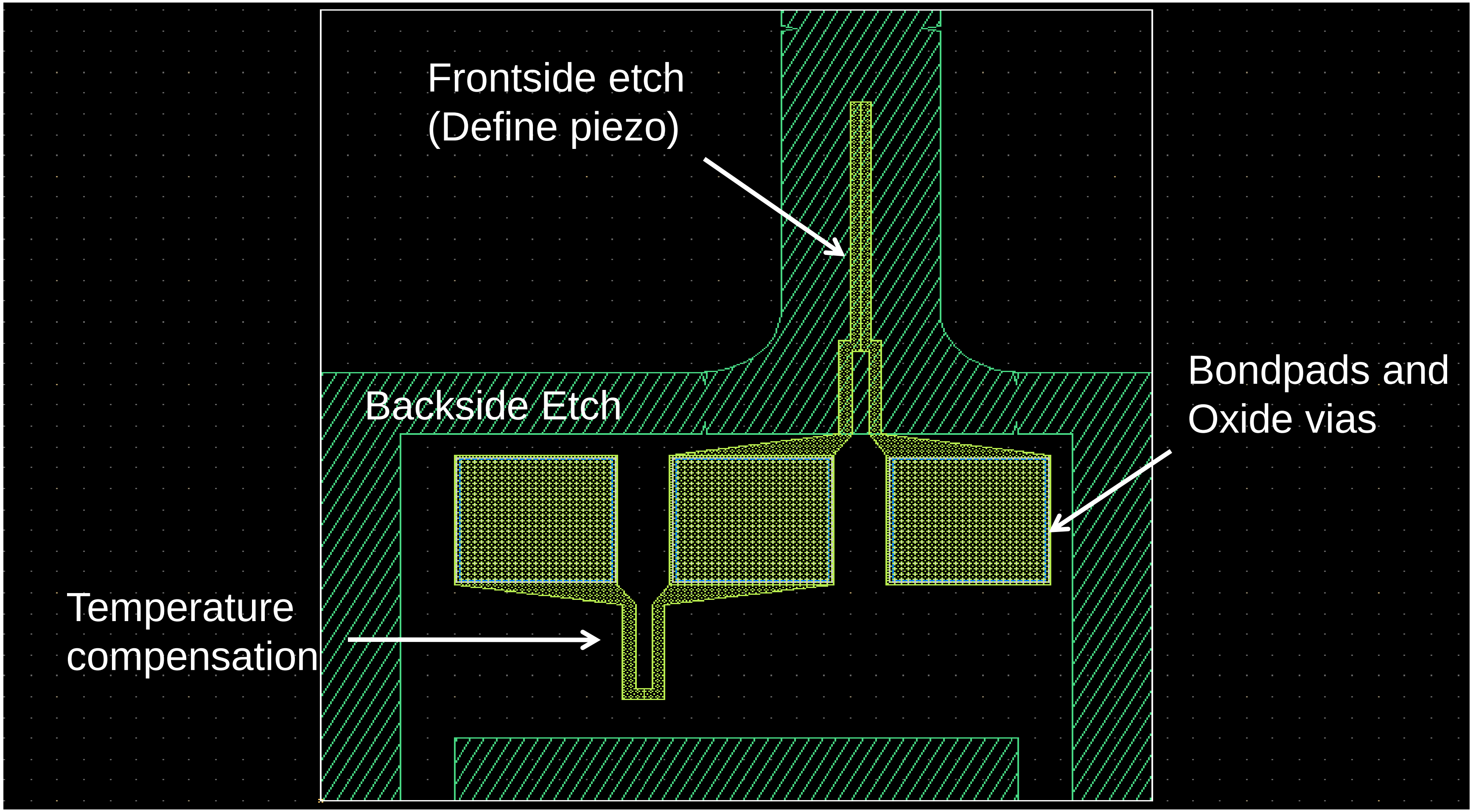
Fabrication Overview

- Piezoresistor only
  - Epitaxy or diffusion, doped near solid solubility limit
  - 5 layers
- Piezoelectric only
  - Alumium nitride unimorph on silicon cantilever
  - 6 layers
- Piezoresistor and piezoelectric
  - Combination of the two above, piezoelectric base and piezoresistor extending beyond
  - 8 layers

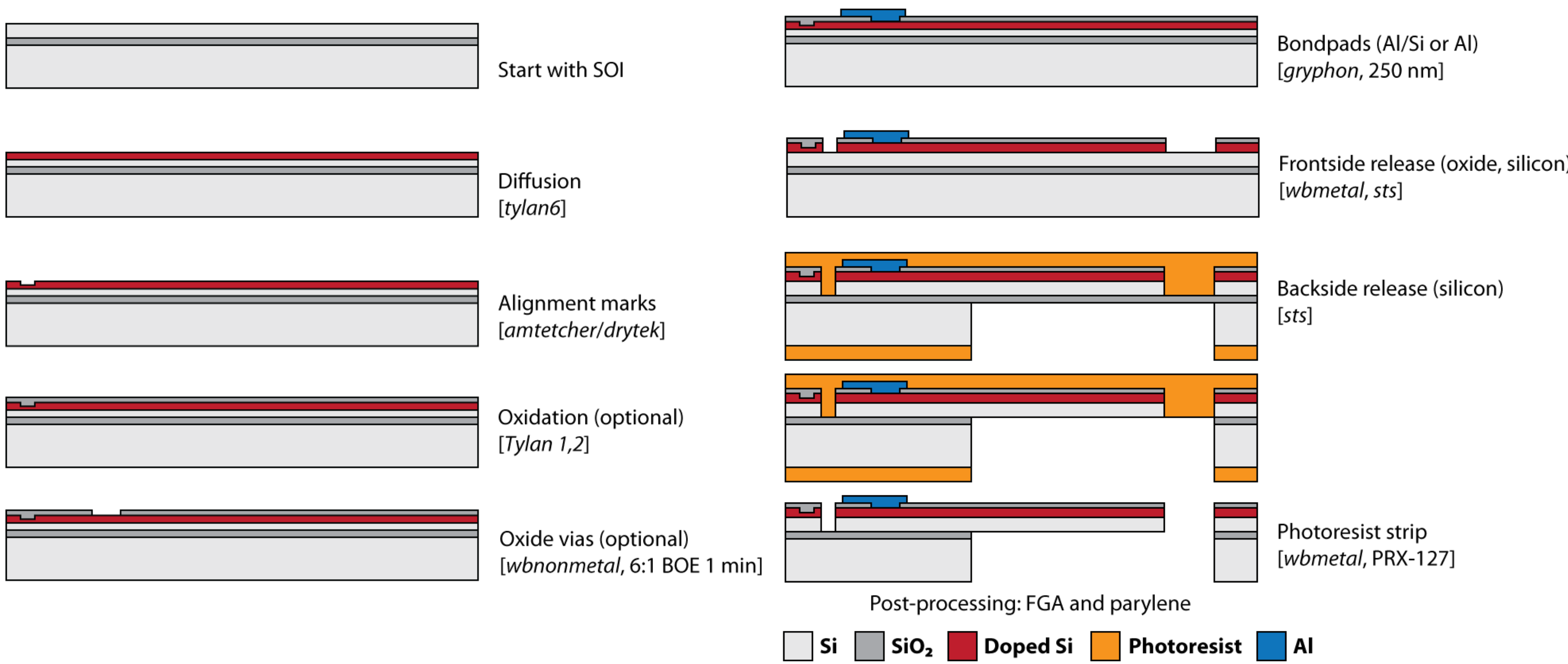
Piezoresistive Cantilever

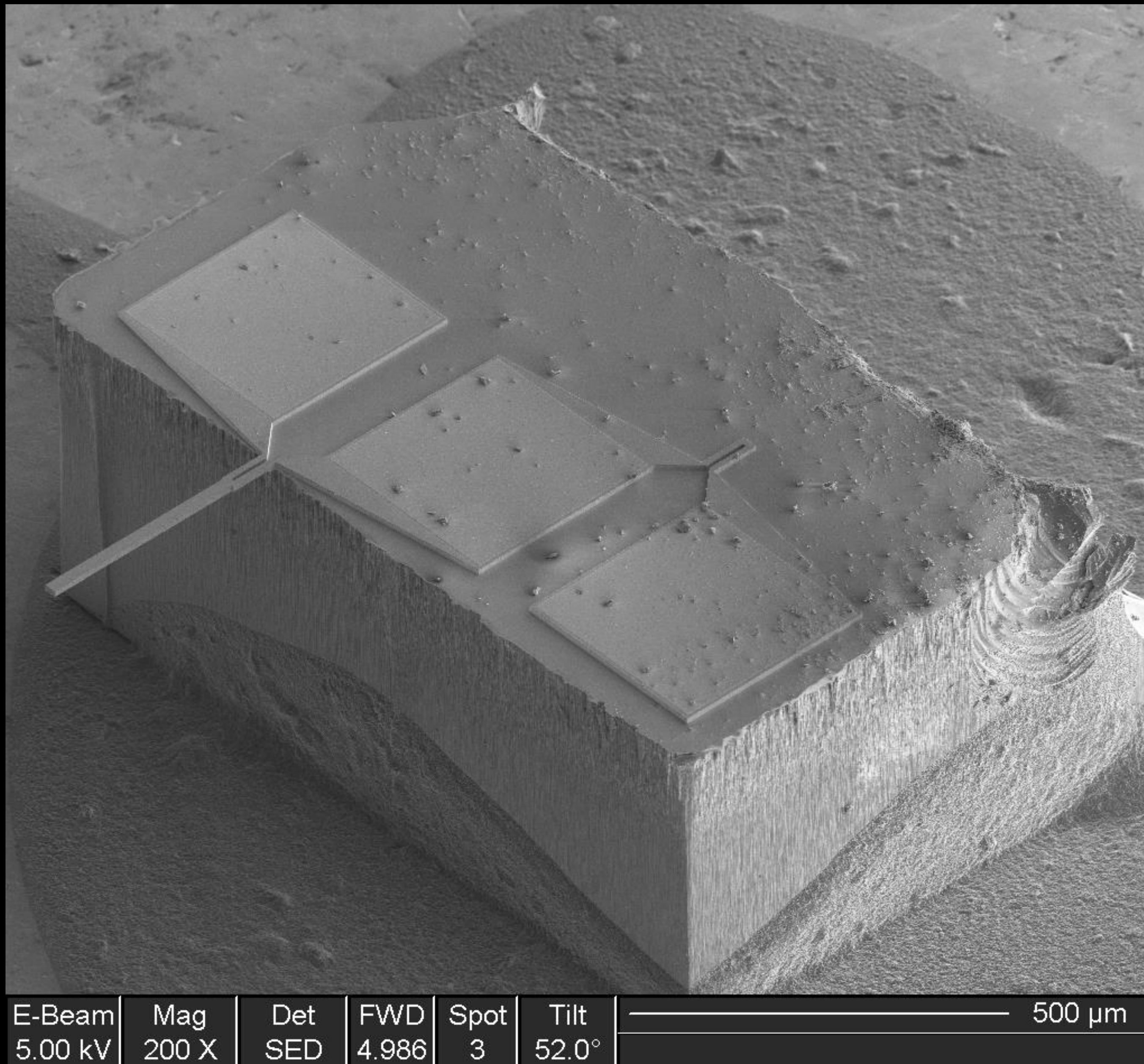


Piezoresistor Only

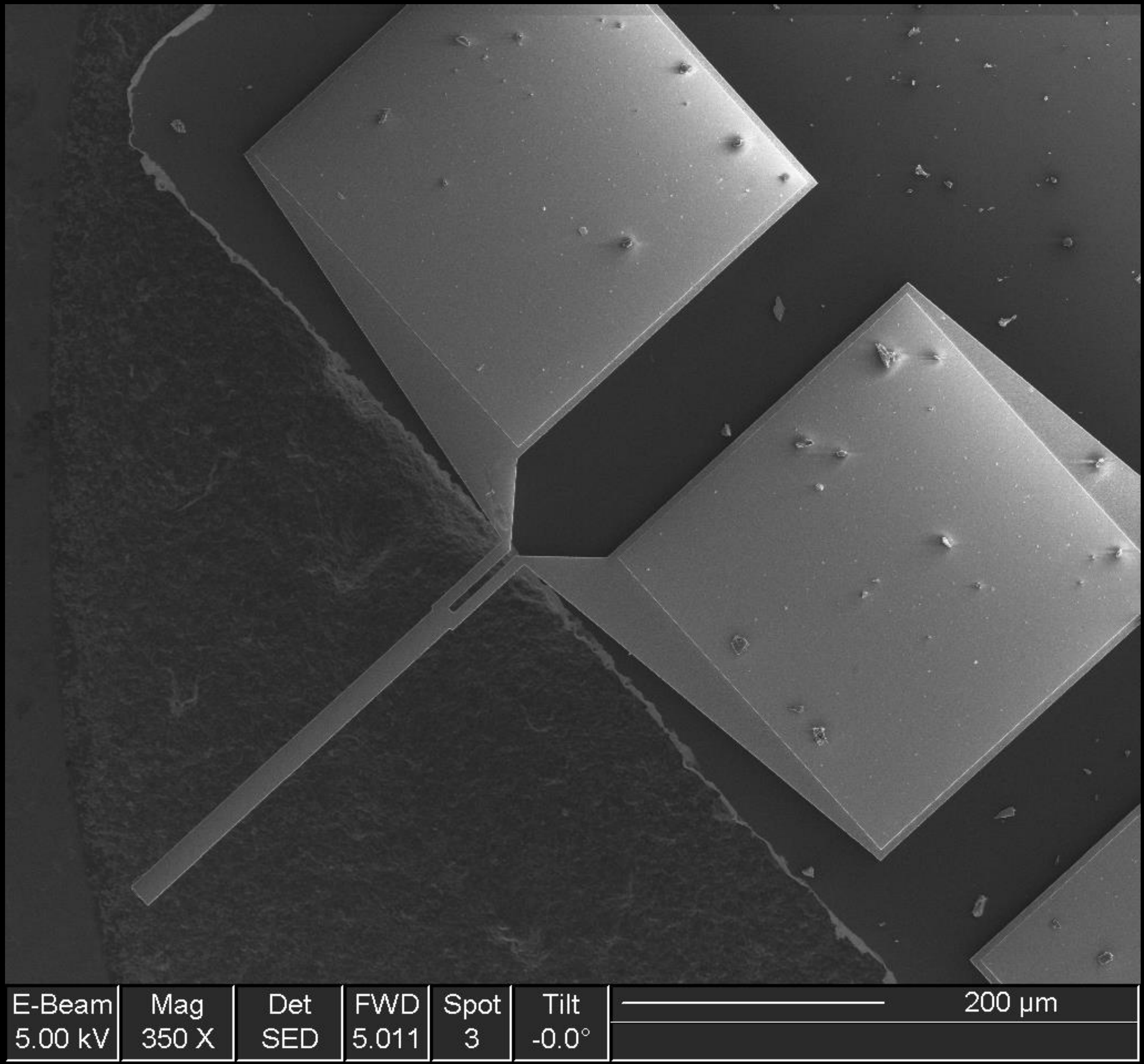


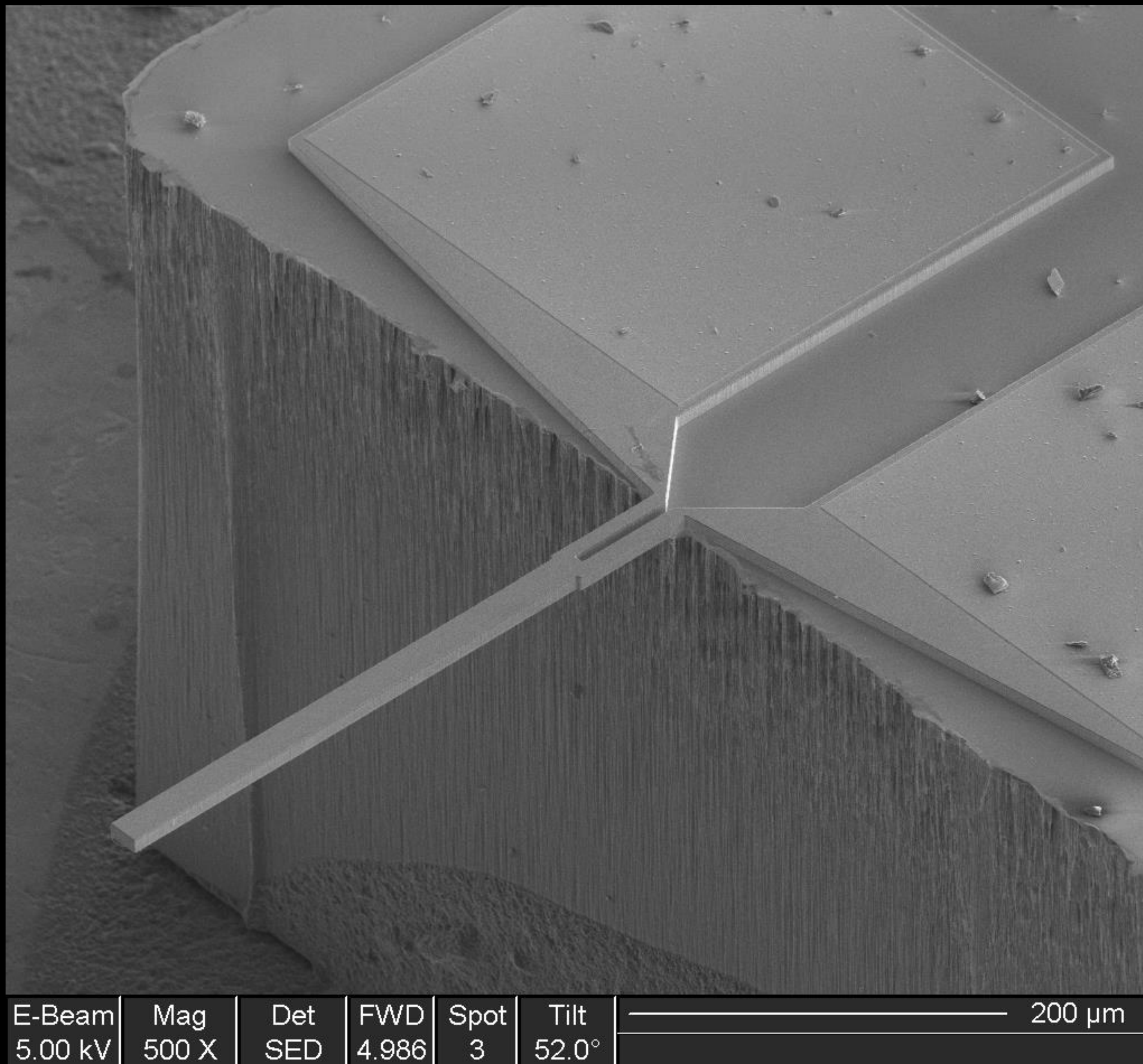
PR Wafers

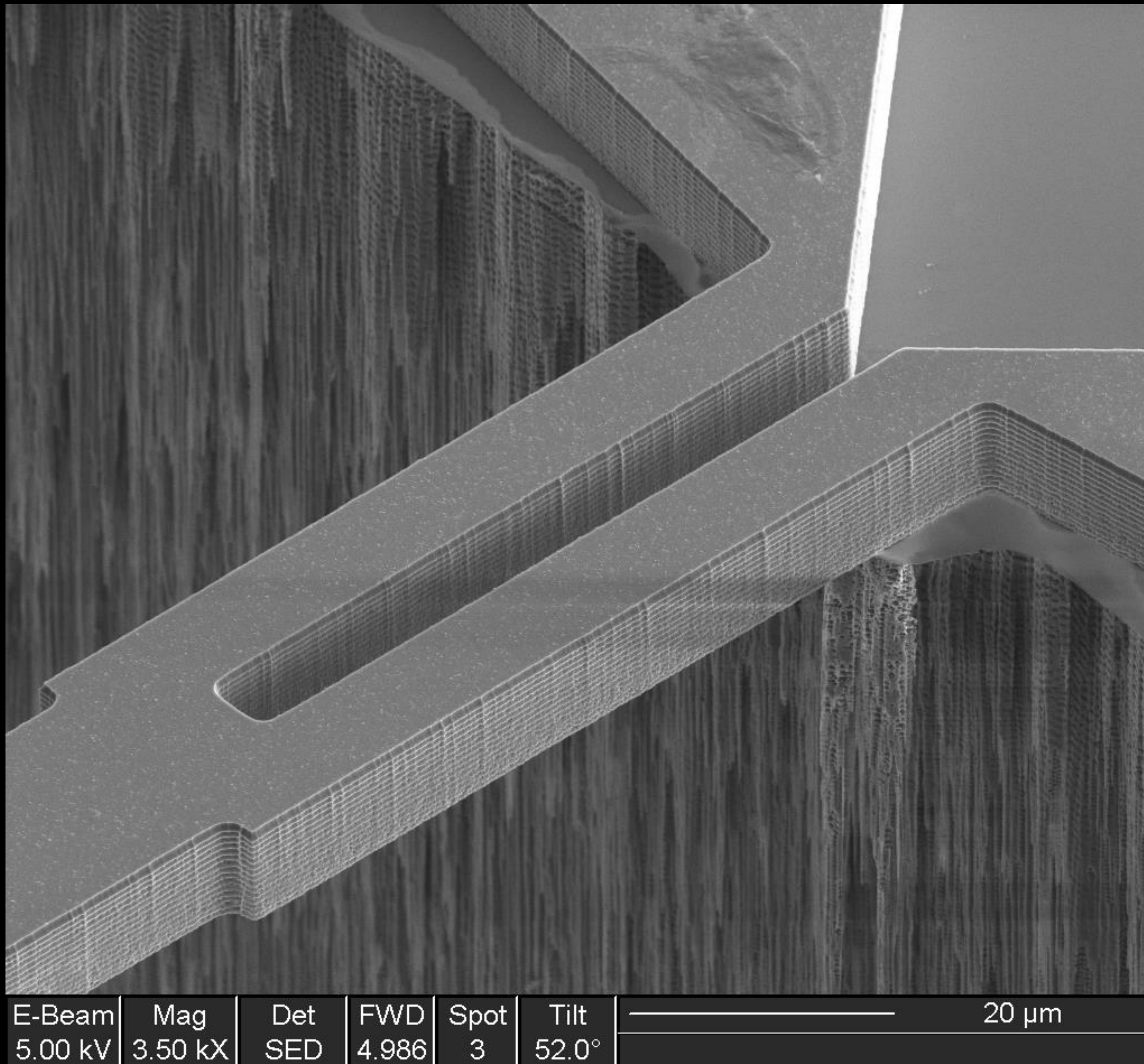

Start with SOI
Diffusion
[*tylan6*]
Alignment marks
[*amtetcher/drytek*]
Oxidation (optional)
[*Tylan 1,2*]
Oxide vias (optional)
[*wbnonmetal*, 6:1 BOE 1 min]
Bondpads (Al/Si or Al)
[*gryphon*, 250 nm]
Frontside release (oxide, silicon)
[*wbmetal*, *sts*]
Backside release (silicon)
[*sts*]
Photoresist strip
[*wbmetal*, PRX-127]
Post-processing: FGA and parylene
Si
SiO_2
Doped Si
Photoresist
Al



E-Beam
5.00 kV
Mag
200 X
Det
SED
FWD
4.986
Spot
3
Tilt
52.0°
500 µm


E-Beam
5.00 kV
Mag
350 X
Det
SED
FWD
5.011
Spot
3
Tilt
-0.0°
200 µm


E-Beam
5.00 kV
Mag
500 X
Det
SED
FWD
4.986
Spot
3
Tilt
52.0°
200 µm


E-Beam
5.00 kV
Mag
3.50 kX
Det
SED
FWD
4.986
Spot
3
Tilt
52.0°
20 µm

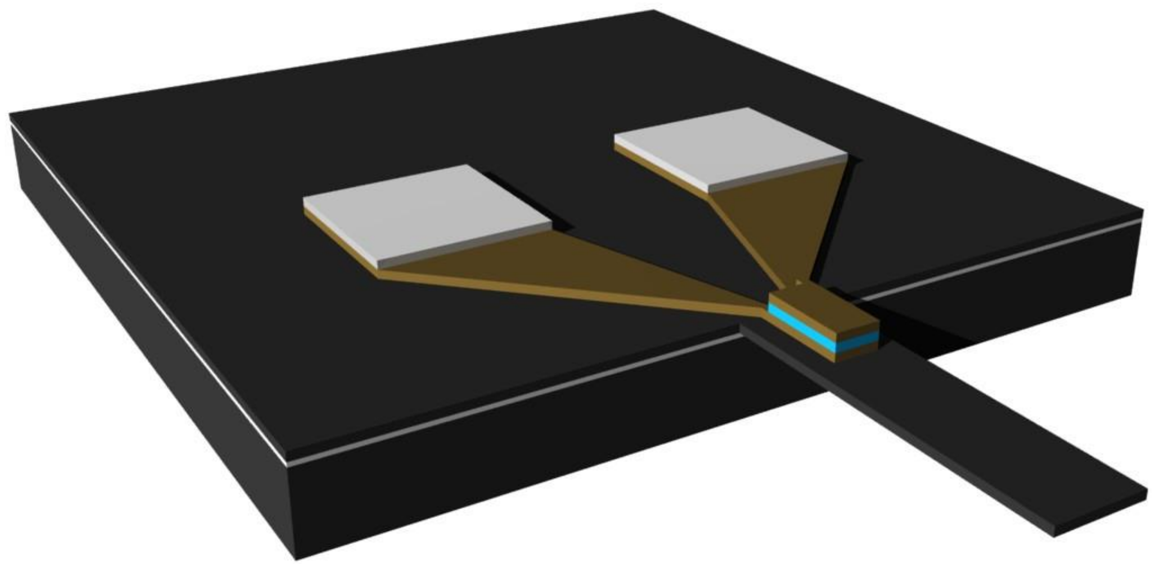
Issues that Came Up

- No frontside wafer protection on backside lithography step
  - No apparent wafer scratches, appears to be okay
- Oxide passivation
  - Oxide not required for operation or low noise (using parylene), but depositing LTO after epi/diffusion might help to protect piezoresistors
  - Decided not to use LTO
- Buried oxide etch for cantilever release
  - Metal on wafers so no HF vapor release
  - Combination BOE and AMT has been used
- Oxide stress
  - Silicon (few um) comparable to BOX (500nm)
  - Yield has been good so far, starting 340nm thick cantilevers where it will be more of an issue
- Junction Spiking
  - Bondpads on 200nm junction, but the design is insensitive to spiking
  - Have used Al/Si to date w/o freckle etch, trying pure aluminum next round

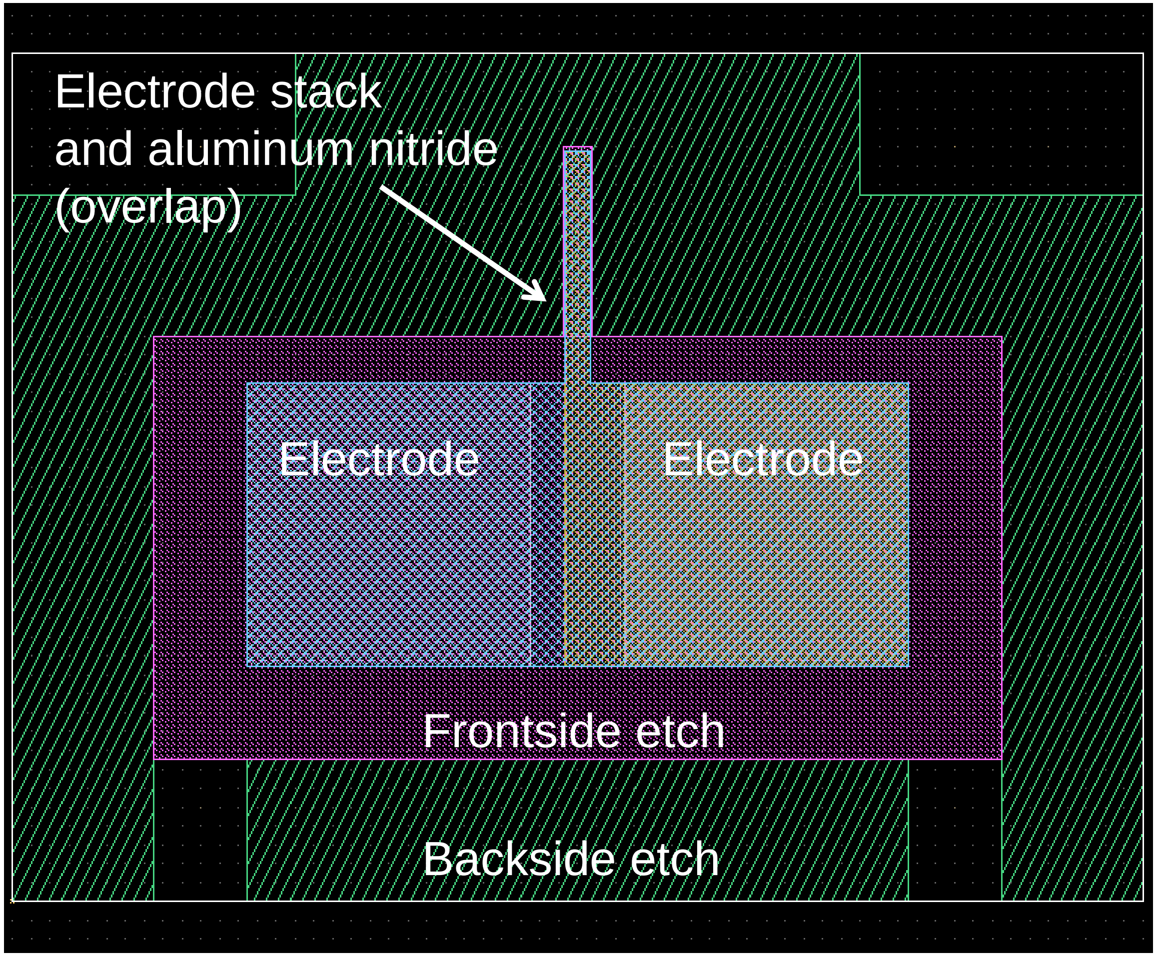
PR Cantilever Testing

- Currently in progress
  - Noise spectrum
  - Sensitivity
  - Natural frequency
  - Stiffness
  - Compare designed and actual force resolution

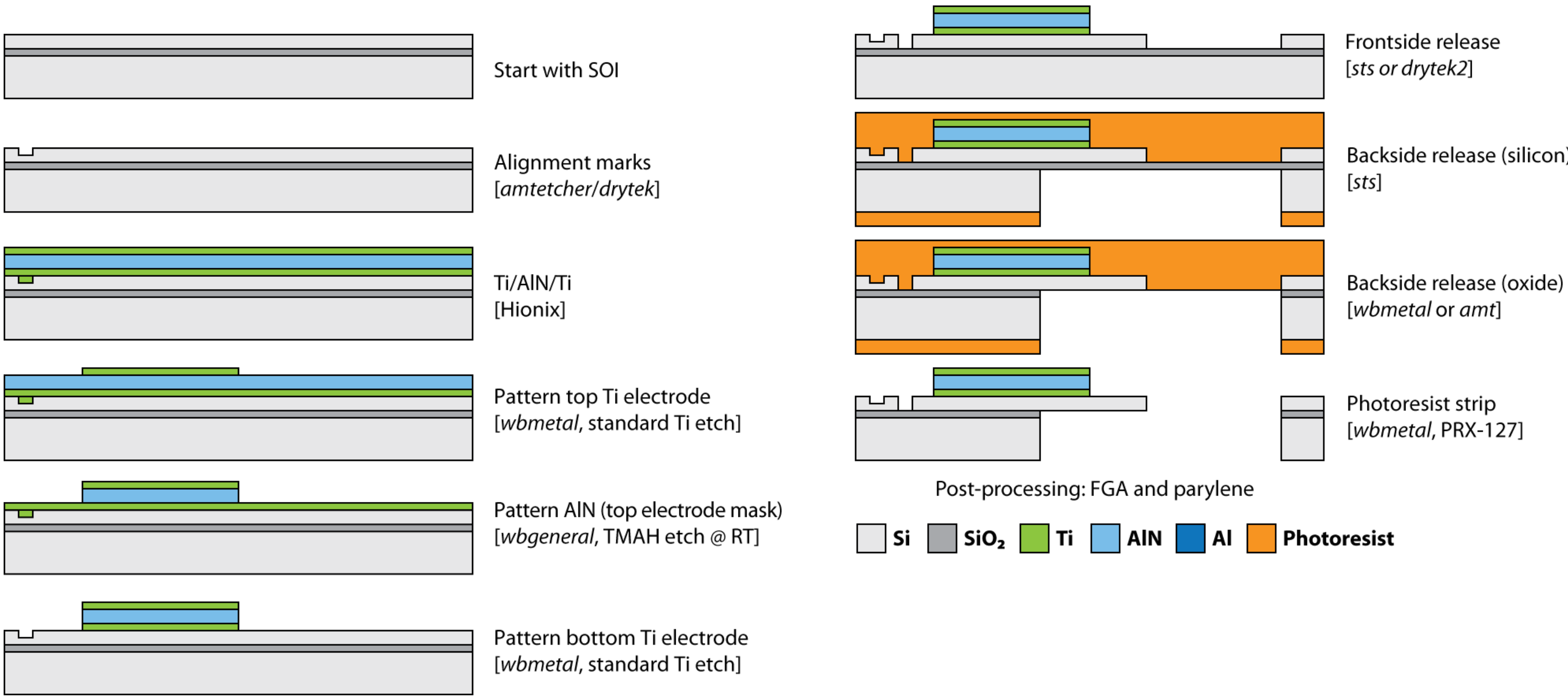
Piezoelectric Cantilever



Piezoelectric Only



PE Wafers



Start with SOI
Alignment marks
[*amtetcher/drytek*]
Ti/AlN/Ti
[Hionix]
Pattern top Ti electrode
[*wbmetal*, standard Ti etch]
Pattern AlN (top electrode mask)
[*wbgeneral*, TMAH etch @ RT]
Pattern bottom Ti electrode
[*wbmetal*, standard Ti etch]
Frontside release
[*sts or drytek2*]
Backside release (silicon)
[*sts*]
Backside release (oxide)
[*wbmetal* or *amt*]
Photoresist strip
[*wbmetal*, PRX-127]
Post-processing: FGA and parylene
Si
SiO_2
Ti
AlN
Al
Photoresist

Aluminum Nitride

- Hexagonal wurtzite crystal structure
- Reactive sputtering is one deposition technique (DC, pulsed DC, RF have been reported) for polycrystallined AlN
- Piezoelectric properties depend upon microstructure, crystal orientation of grains
- Lattice constant mismatch between substrate and AlN affects orientation
- Nitrogen poisons surface of aluminum target, which is then sputtered off

Literature Review

1) Film alignment increases with temperature
2) XRD only a rough predictor of d_{33}
3) High energy impact of AlN preferred

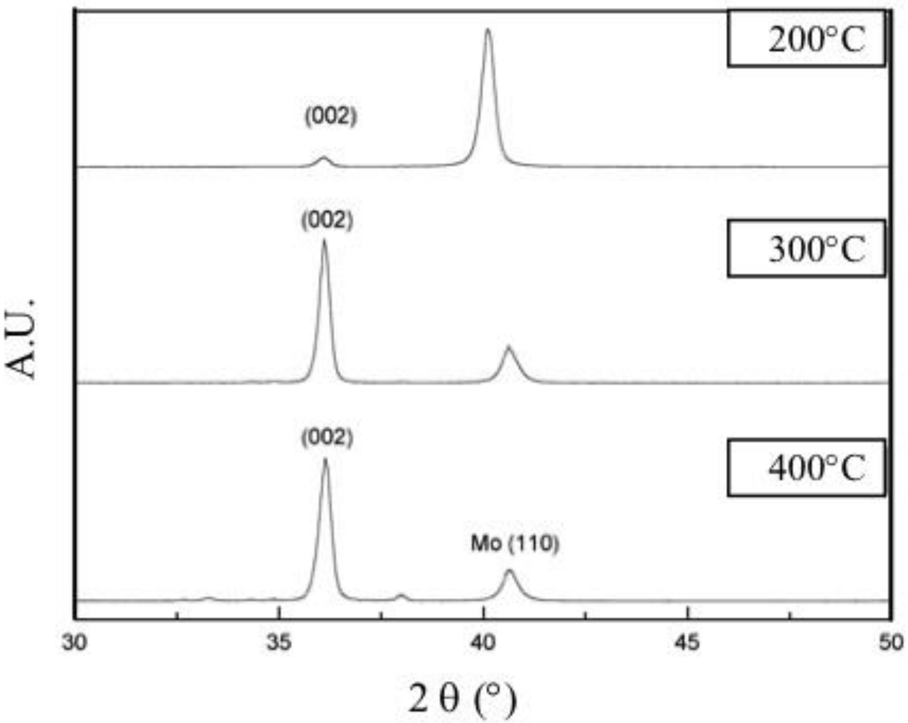


Figure 3. XRD patterns of the AlN films deposited at various substrate temperatures.

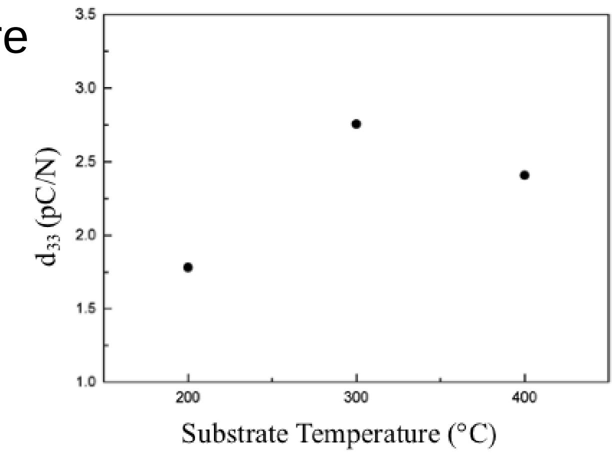


Figure 5. Piezoelectric parameter d_{33} of AlN/Mo as a function of the substrate temperatures (5 sets).

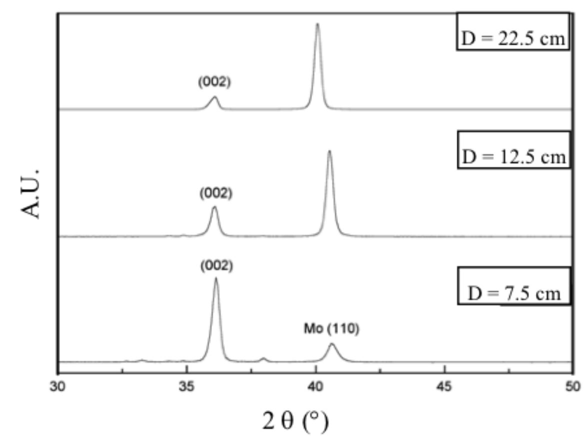


Figure 6. The XRD patterns of AlN films at different target-substrate distance.

Preferred Orientation Control and Characterization of AlN Thin Films Using Reactive Sputtering (2004)

Literature Review

1) Si, Au, Al, Mo, Ti, W have all been used as bottom electrodes for AlN
2) Optimal conditions for AlN on Si found to be 350W power, 5 mtorr, Ar/N_2 flow 10/20 sccm (RF magnetron)

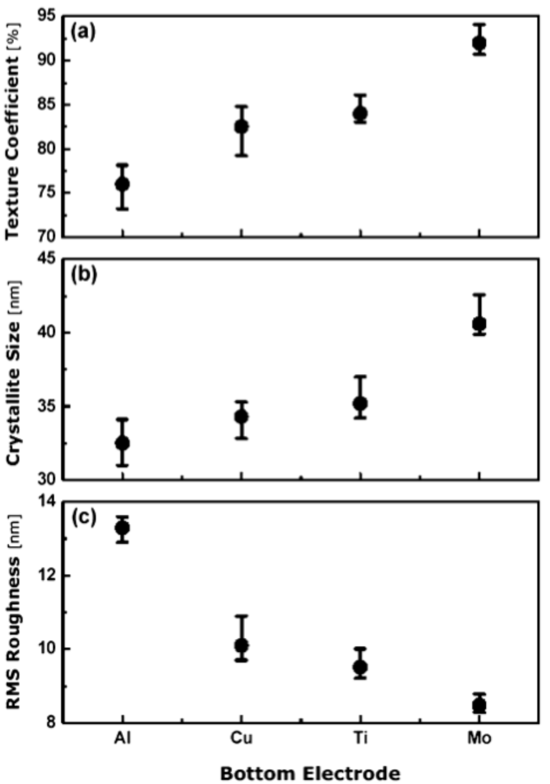


Fig. 3. Estimated values of (a) (0 0 2)-TC; (b) crystallite size and (c) RMS surface roughness of AlN films, in terms of the bottom metals used, such as Al, Cu, Ti and Mo.

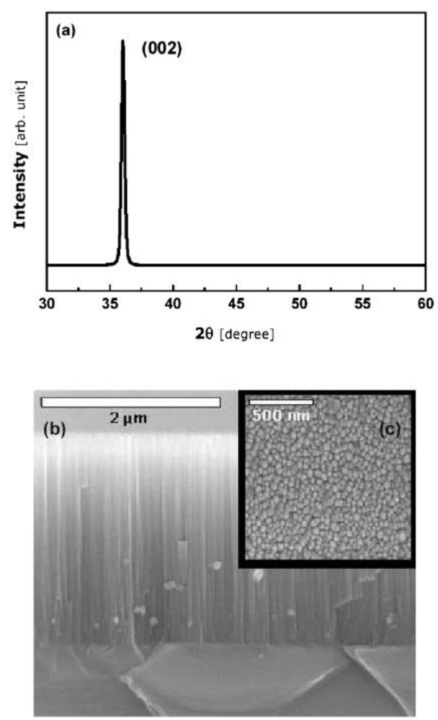


Fig. 1. AlN film deposited on Si(1 1 1): (a) XRD pattern; (b) surface morphology and (c) cross-sectional morphology.

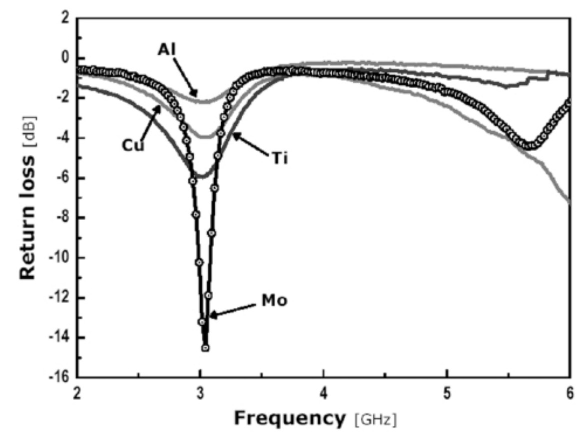


Fig. 5. Frequency response characteristics (S_{11}) of resonators with Al/AlN/metal/Si configuration, in terms of the bottom metals used, such as Al, Cu, Ti and Mo.

Table 1
Lattice mismatch between AlN and the bottom electrode metals and their thermal expansion coefficient of metals

Bottom electrode metals	Lattice mismatch (%)	Thermal expansion coefficient ($\times 10^{-6}$/K)
Al	23.15	23.1
Cu	13.8	16.5
Ti	−5.2	8.6
Mo	0.87	4.8

Plane spacing of AlN = 0.3112 nm, thermal expansion coefficient of AlN = 4.2×10^{-6}/K.

Effects of bottom electrodes on the orientation of AlN films and the frequency responses of resonators in AlN-based FBARs (2004)

Literature Review

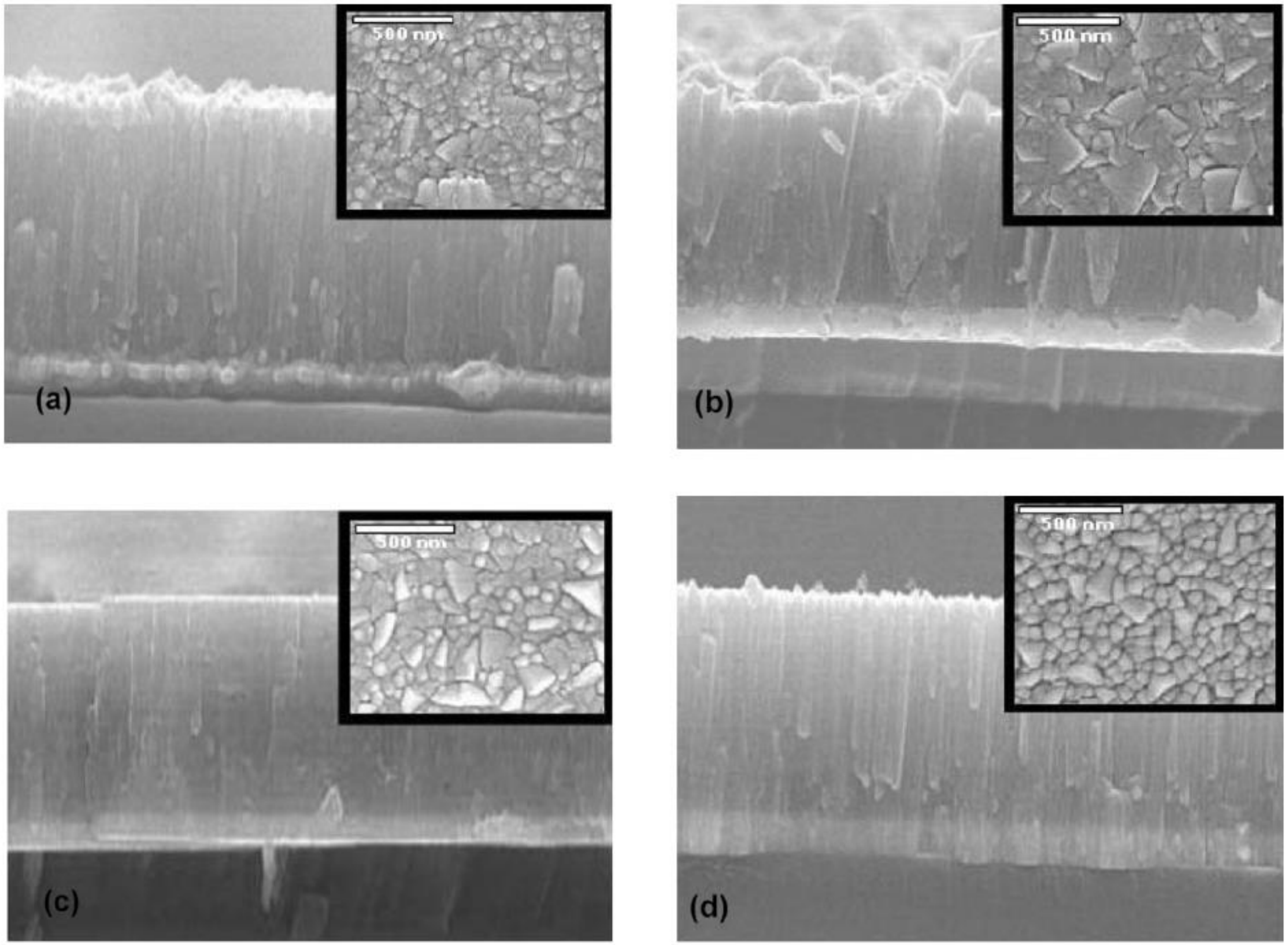


Fig. 4. FE-SEM cross-sectional and surface morphologies as shown in each insert for AlN films deposited on various bottom metals: (a) Al/Si; (b) Cu/Si; (c) Ti/Si and (d) Mo/Si.

Effects of bottom electrodes on the orientation of AlN films and the frequency responses of resonators in AlN-based FBARs (2004)

Literature Review

1) Pt is stable in nitrogen plasma, whereas nitrogen is incorporated into the Ti film but doesn't appear to form TiN
2) 7 mtorr, 1:1 Ar/N_2, 800W RF power, bias -9V to -31V.
3) Intrinsic stress evaluated by wafer bending before and after AlN dep

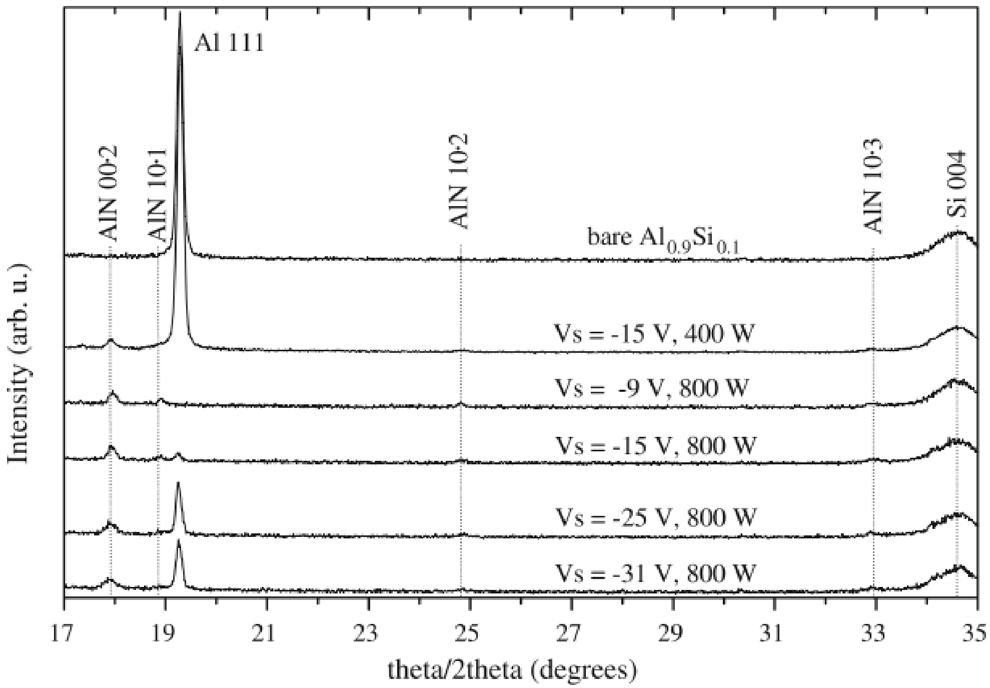


Fig. 1. XRD patterns of AlN films sputtered on $Al_{0.9}Si_{0.1}$ substrates under different process conditions.

Comparative study of c-axis AlN films sputtered on metallic surfaces (2005)

Literature Review

1) Piezoelectric properties of AlN on Ti found to be less sensitive to processing conditions than other metals, however d_{33} was smaller.
2) Explained partly by incorporation of N_2 into Ti lattice at higher deposition temperature (300C+)
3) Reducing pressure, increasing nitrogen, increasing RF power all increase bombardment energy. A minimum amount of energy is required for good piezoelectric properties (40V or larger bias between plasma and substrate)
4) Both positive and negative d_{33} values are possible due to parallel, anti-parallel growth (mechanisms not understood)
5) C-axis orientation and lattice mismatch not correlated (Pt has largest mismatch of all bottom electrodes investigated)
6) Intrinsic stress independent of electrode material, dependent upon processing conditions. Typically want 0, but for some applications some built-in stress is actually good.

Stress and piezoelectric properties of aluminum nitride thin films deposited onto metal electrodes by pulsed direct current reactive sputtering (2001)

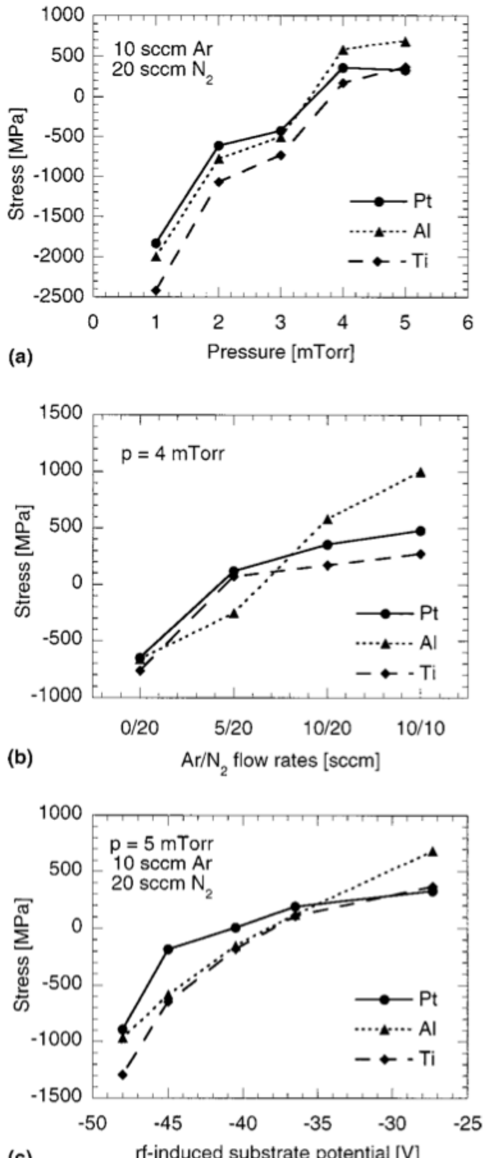


FIG. 1. Built-in stress of AlN thin films deposited on Pt, Al, and Ti as a function of (a) pressure, (b) gas flow rates, and (c) the rf bias applied on the substrate holder. For (a) and (b), the substrate holder was electrically floating.

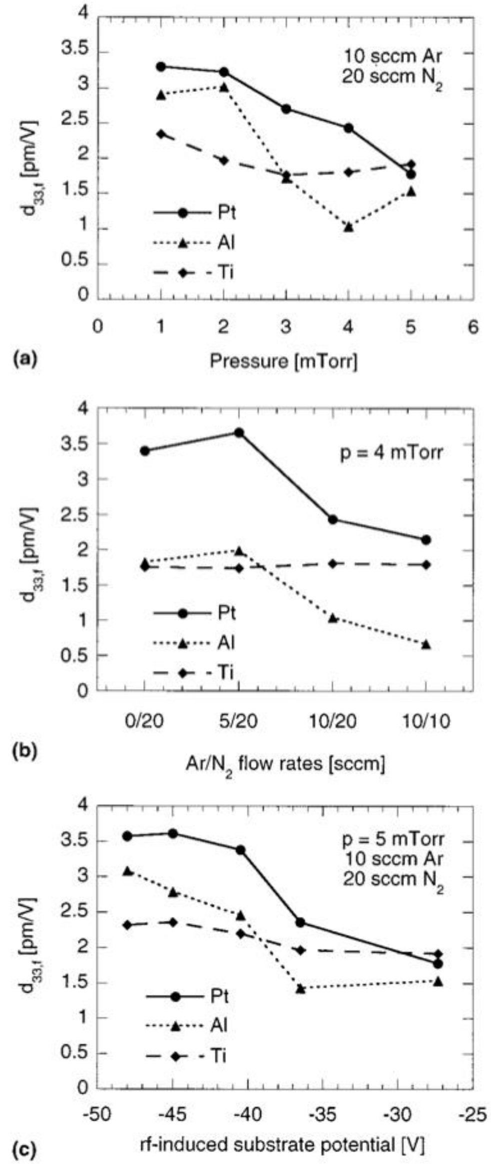


FIG. 2. Piezoelectric $d_{33,f}$ coefficient of AlN thin films deposited on Pt, Al, and Ti as a function of (a) pressure, (b) gas flow rates, and (c) the rf bias applied on the substrate holder. For (a) and (b), the substrate holder was electrically floating.

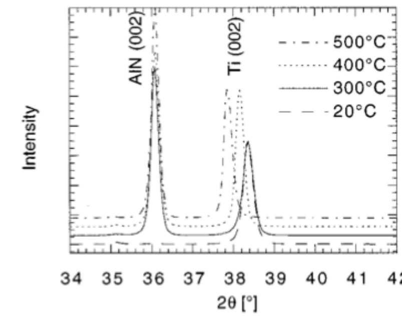


FIG. 3. X-ray spectra for AlN films deposited on Ti at different temperatures.

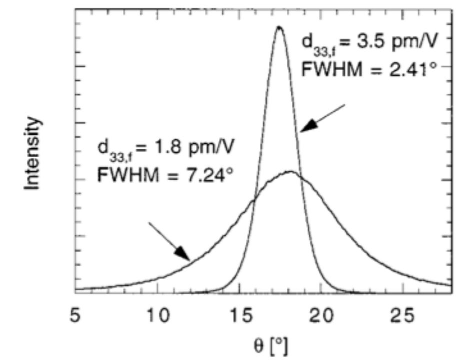


FIG. 6. X-ray rocking curves and $d_{33,f}$ coefficients of AlN films deposited on Pt with a bias of either −42 or −27 V.

Stress and piezoelectric properties of aluminum nitride thin films deposited onto metal electrodes by pulsed direct current reactive sputtering (2001)

Literature Review

1) Presputtered target for 1 hour before deposition, 5cm target to substrate, 225C, 5mtorr, 400W, 3 sccm:9 sccm Ar/N_2, 900nm/hour. Mentioned in other papers as well.
2) FWHM of AlN decreased for thicker films, no d_{33} measurement though

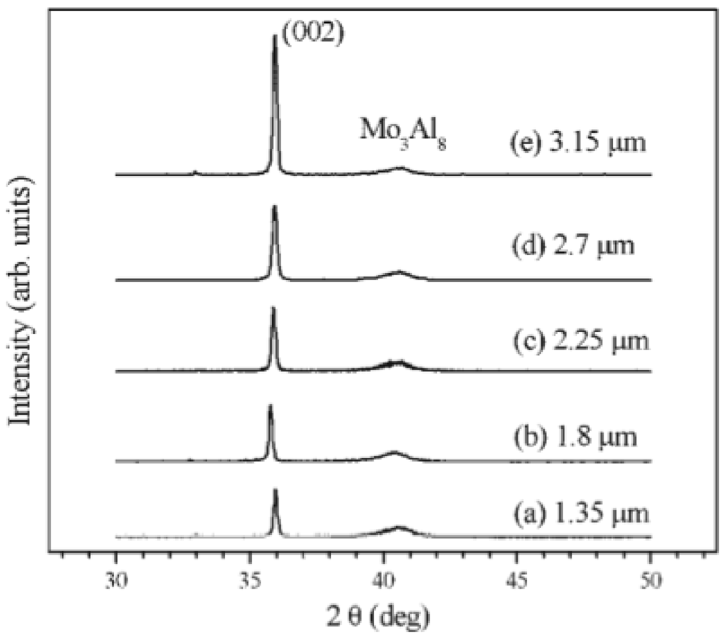


Fig. 2. XRD patterns associated with AlN films of different thicknesses: (a) 1.35 µm, (b) 1.8 µm, (c) 2.25 µm, (d) 2.7 µm, and (e) 3.15 µm.

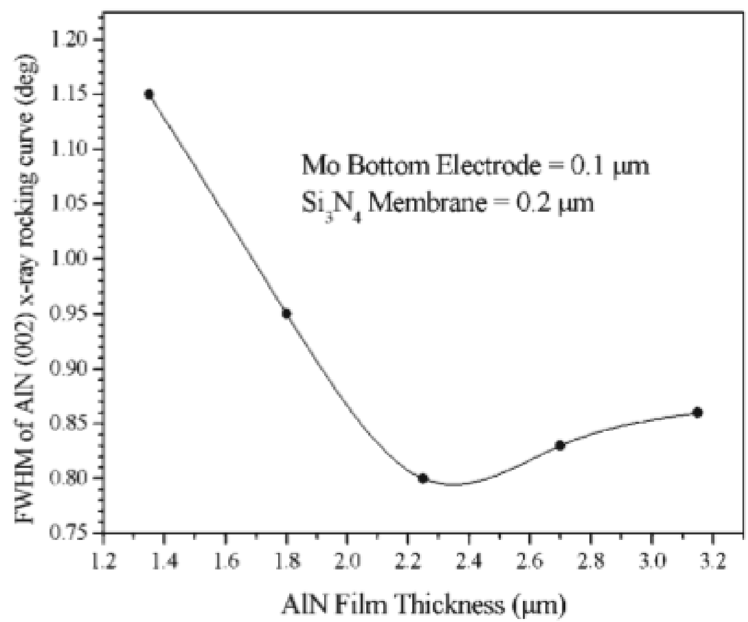


Fig. 3. Relationship between FWHM of AlN (002) X-ray rocking curve and thickness of AlN films on Mo bottom electrode.

Effect of AlN Film Thickness and Top Electrode Materials on Characteristics of Thin-Film Bulk Acoustic-Wave Resonator Devices (2005)

Literature Review

1) 300C, 3 mtorr, 1500W, Pt electrode

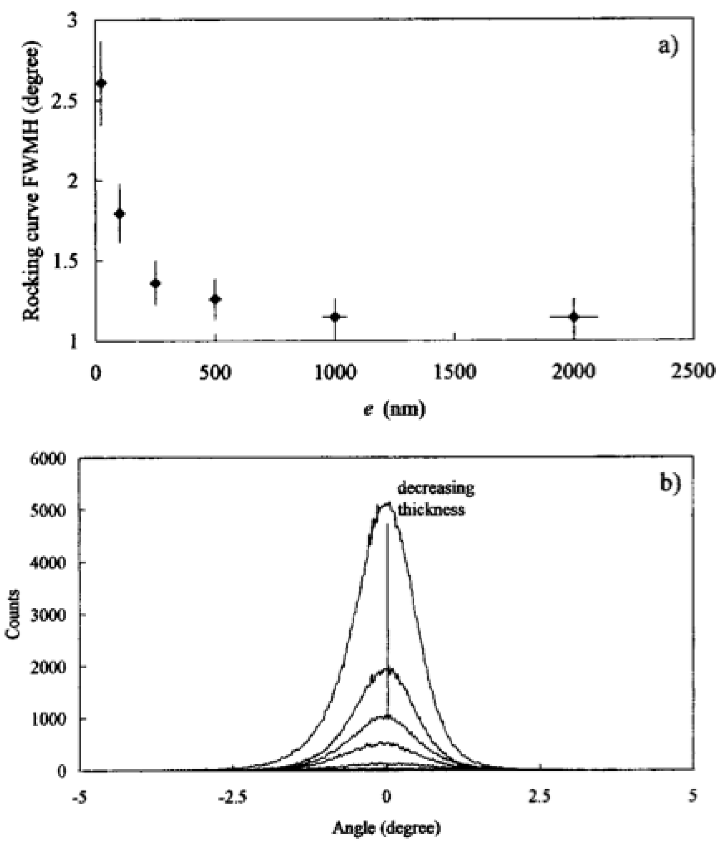


FIG. 3. (a) Rocking curve FWMH and (b) rocking curve diffraction peak magnitude as a function of AlN thin-film thickness (downwards arrow indicates decreasing AlN film thickness).

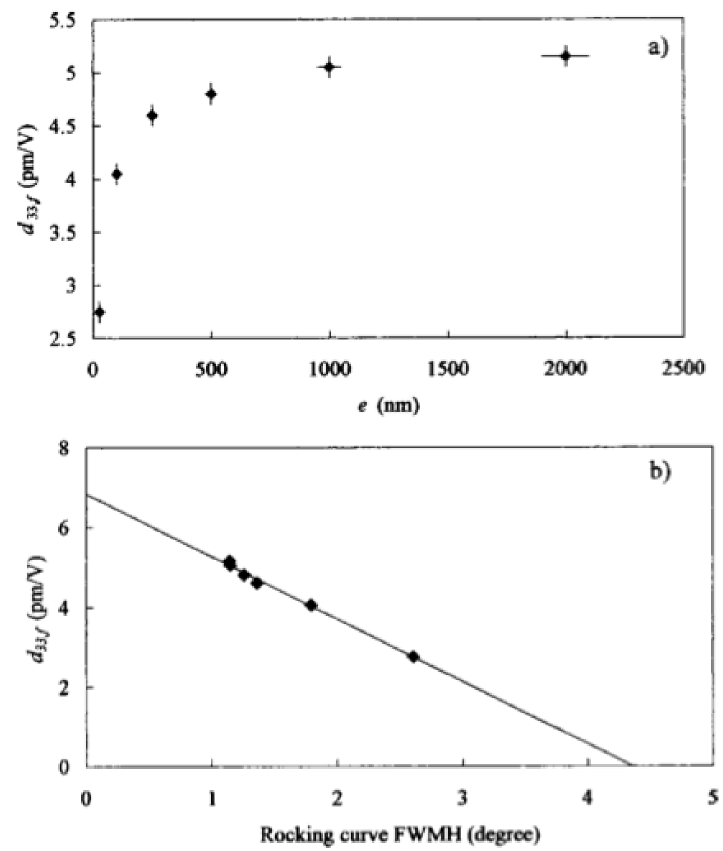


FIG. 7. Piezoelectric coefficient $d_{33,f}$ as a function of (a) AlN thin-film thickness and (b) rocking curve FWMH.

Thickness dependence of the properties of highly c-axis textured AlN thin films (2004)

Literature Review

1) A small FWHM can be achieved with little/no piezoelectric response due to antiparallel grains
2) Sputtering energy must be high enough to obtain (002) orientation, but not too high to cause lattice damage and not too low or lese it will lead to antiparallel grains (pressure and voltage bias)
3) D_{33} = 3.32 pm/V

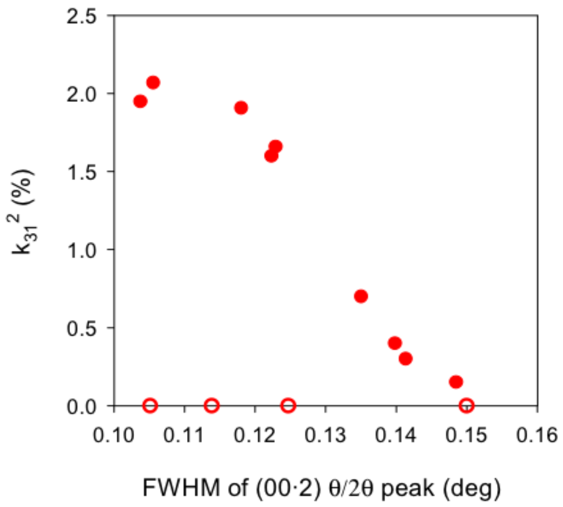


Figure 5. Transversal electromechanical coupling factor as a function of the FWHM of the 00·2 θ/2θ reflection for AlN films deposited under various sputtering conditions. Solid symbols correspond to films with pure (00·2) orientation; hollow symbols correspond to films with weak 10·2 and 10·3 peaks.

SAW and BAW Response of c-axis AlN Thin Films Sputtered on Platinum (2004)

Literature Review

1) Pressure and target-substrate distance determine orientation, low pressure and short distances favor highly oriented films in (002) direction (high energy), however growth in (100) direction is possible too
2) Energy supplied by substrate temp or bombardment with energetic particles
3) 4cm target-substrate distance used here, bare Si wafers
4) Argon primarily provides the sputtering energy; reducing its concentration reduces (002) orientation
5) Optimized parameters: 6 mTorr, 1:1 Ar/N_2, -28V self-bias, 4.2 deg FWHM on Si

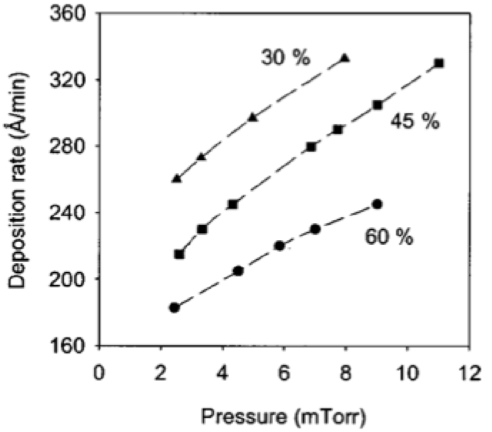


FIG. 1. Dependence of the AlN deposition rate on the total pressure for N_2 contents in the discharge gas of (▲) 30%, (■) 45%, and (●) 60%.

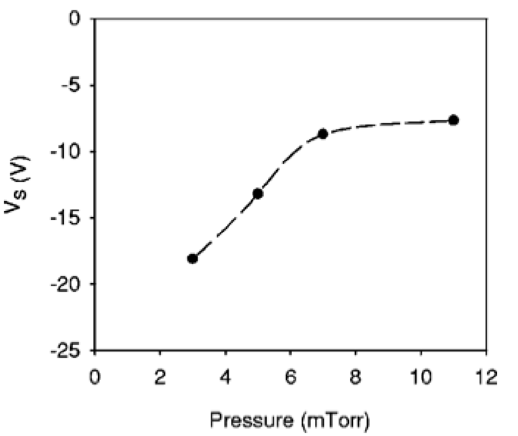


FIG. 3. Self-bias voltage of the electrically isolated substrate as a function of the total pressure (cathode voltage was 1700 V and N_2 content was 30%).

Influence of sputtering mechanisms on the preferred orientation of aluminum nitride thin films (2003)

Literature Review

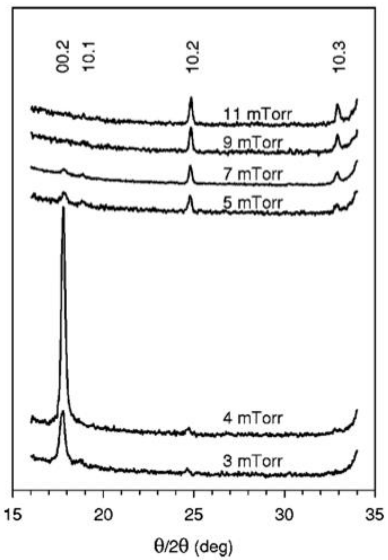


FIG. 2. X-ray diffraction patterns of AlN films deposited at different values of total pressure (cathode voltage was 1700 V and N_2 content was 30%).

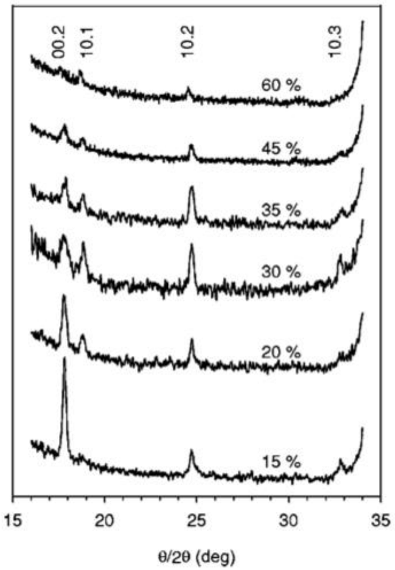


FIG. 4. X-ray diffraction patterns of AlN films deposited at different contents of N_2 in the gas (total pressure was 5 mTorr and cathode voltage was 1700 V).

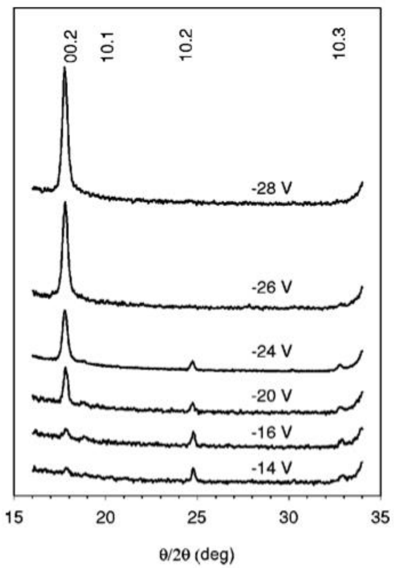


FIG. 7. X-ray diffraction patterns of AlN films deposited at different values of the substrate self-bias voltage (total pressure was 5 mTorr and N_2 content was 30%).

Influence of sputtering mechanisms on the preferred orientation of aluminum nitride thin films (2003)

Deposition parameters

- Oxygen concentration
- Temperature
- Pressure
- Power and bias
- Target-sample distance

Characterization

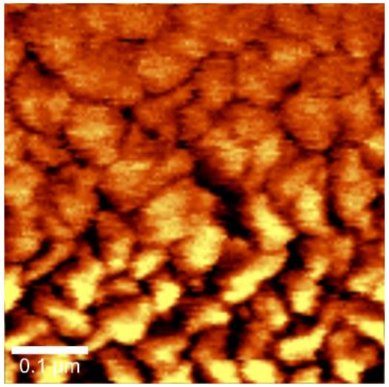
- Crystal structure (XRD)
- Surface roughness (AFM)
- Composition (XPS)
- Piezoelectric coefficient (d_{33})
  - Force-charge (direct effect)
  - Field-strain (converse effect)

Characterization Methods

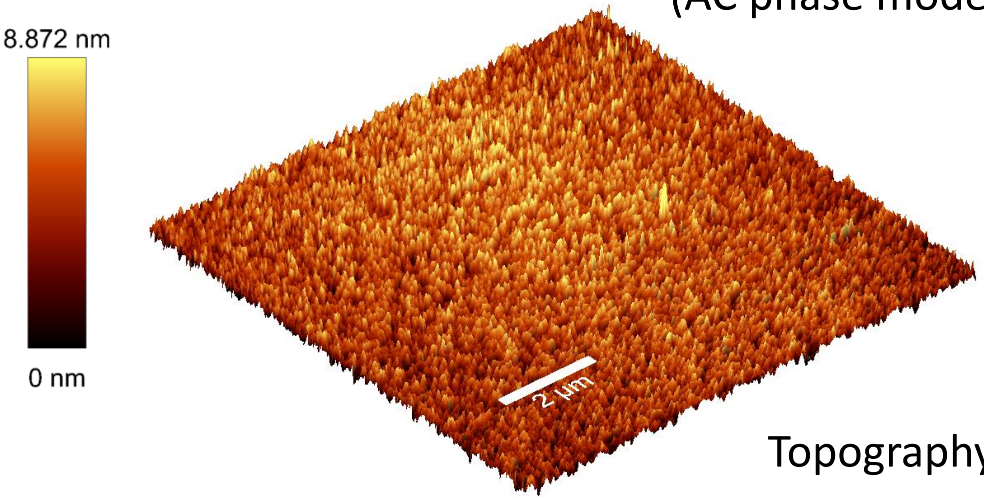
AFM Topography and Tapping Mode

XRD Rocking Curves

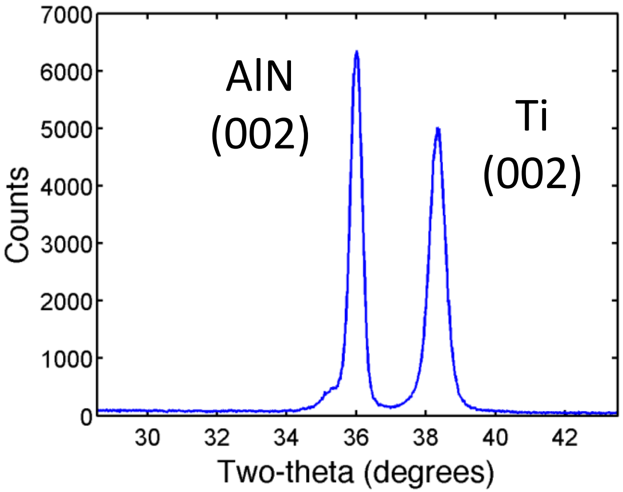
Tools for the characterization of aluminum nitride thin films. Aligned, vertical columnar grains are indicative of a high piezoelectric coefficient. XRD rocking curves, AFM scans and direct measurement are used to optimize deposition.

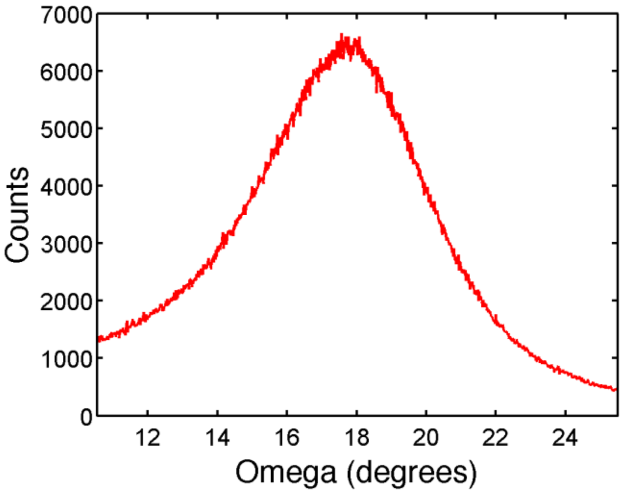


Grain structure
(AC phase mode)

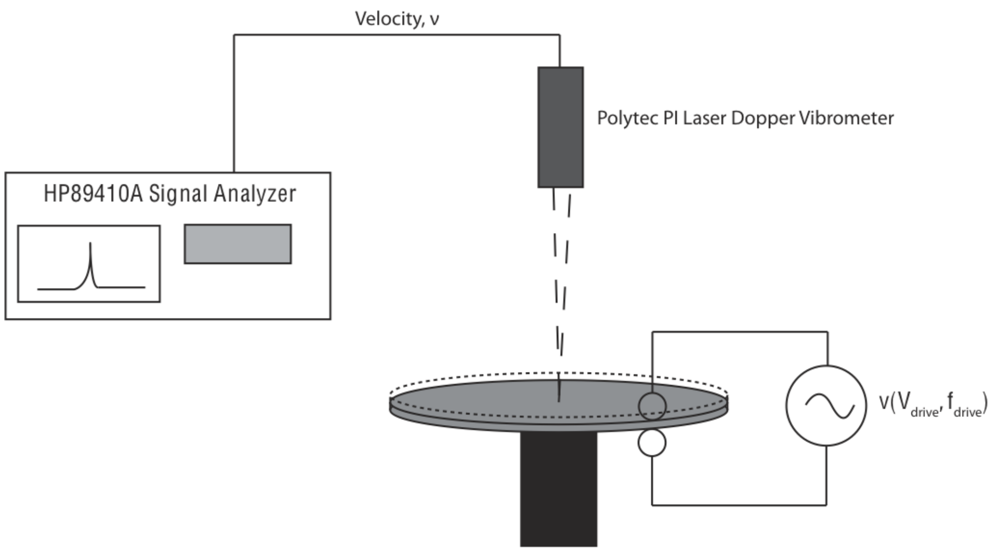


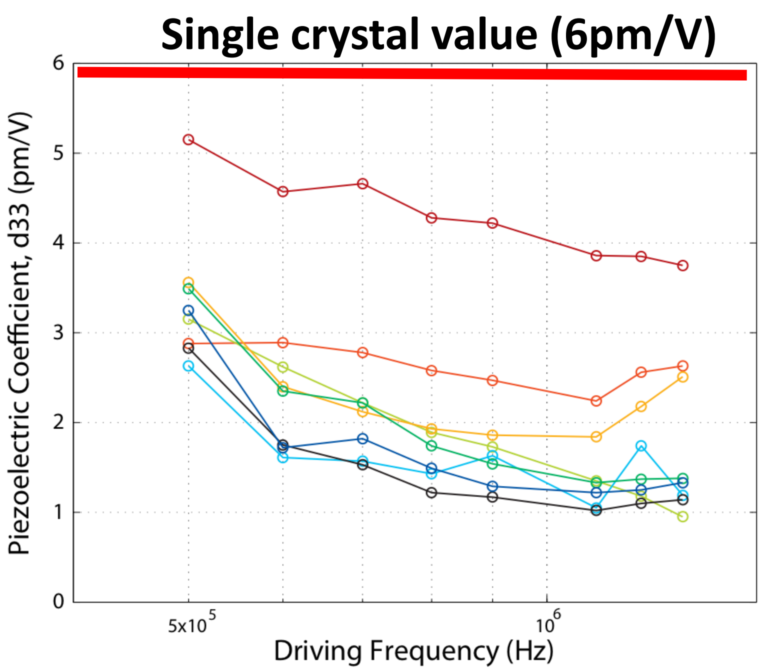
Topography





Measuring d_{33}





- 4” and 8” wafers of Ti/AlN/Ti/Si/Ti, voltage applied across film
- Deflection measured with laser doppler velocimetry (LDV)

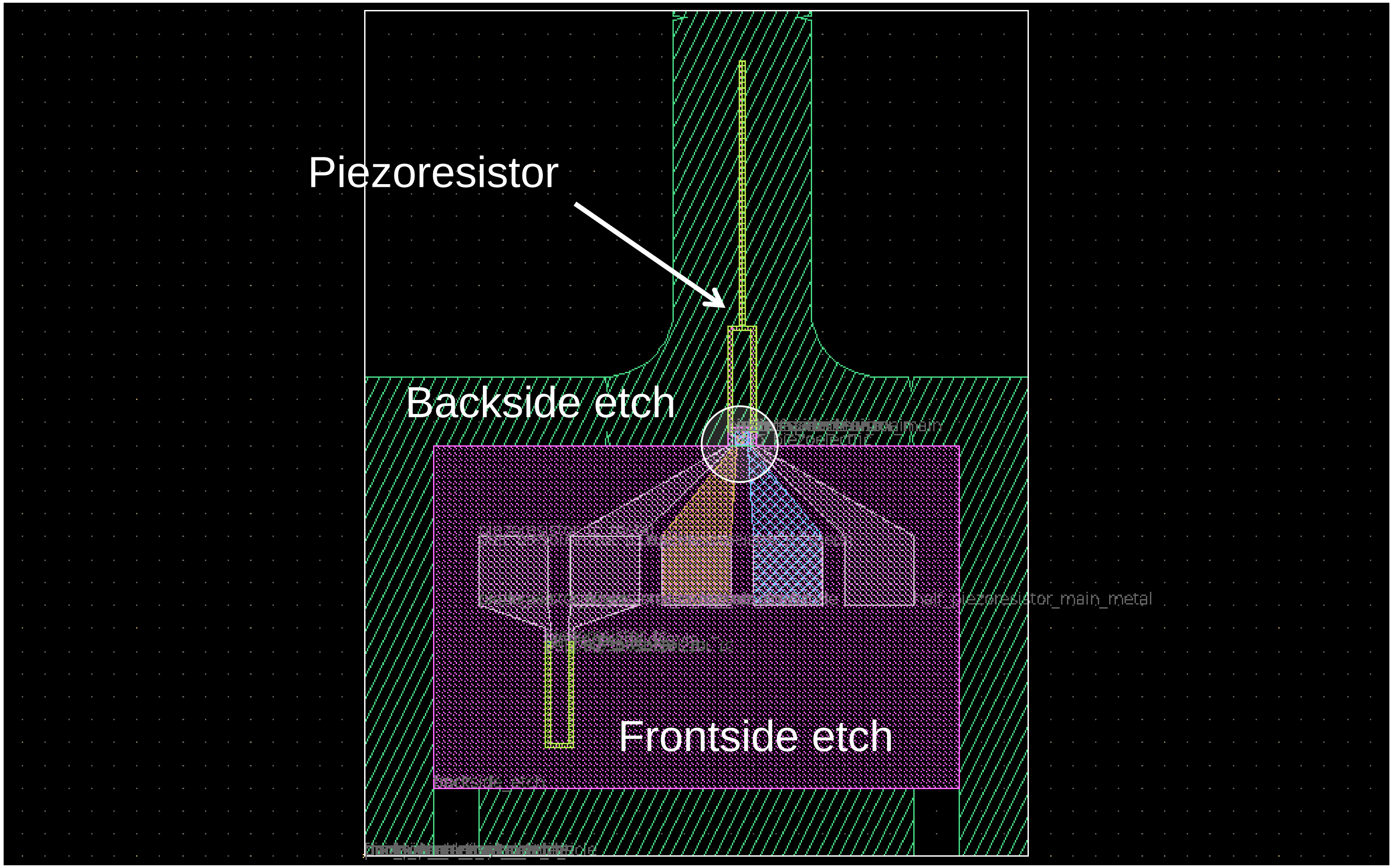
Our Results

- For Ti/AlN/Ti stacks on silicon, cleaning the substrate in-situ with ICP improves alignment
- High biases eliminated growth alignment (substrate damage?)
- First XPS analysis showed ~30% oxygen, minimizing in feature appears to be critical
- Most recent depositions showed good performance, better than best on Ti to date (up to ~4 pm/V)
- Still working on more thorough analysis (more on this later)

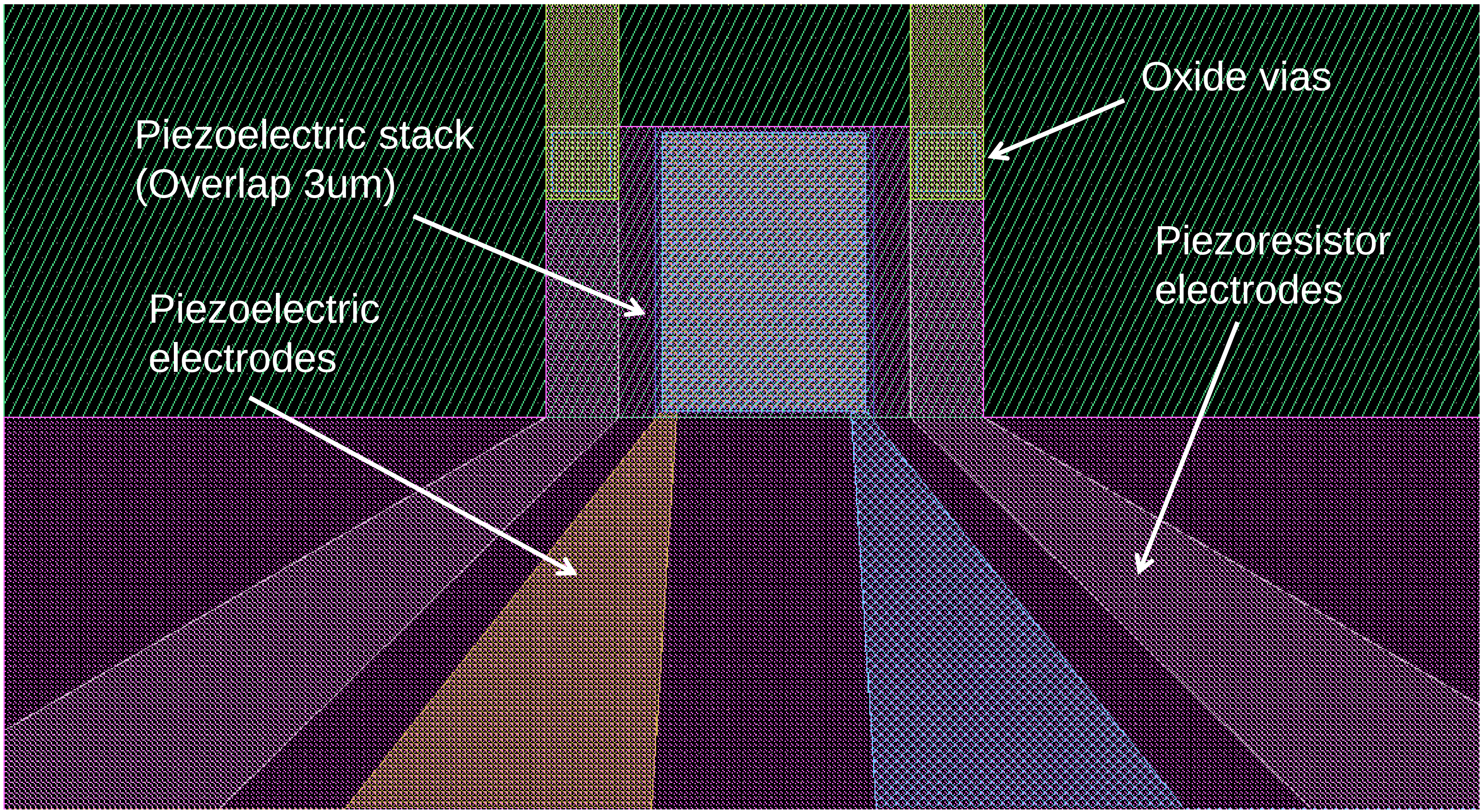
Piezoelectric Applications

- PZT, ZnO, AlN are commonly used piezoelectric thin films
  - ZnO is a fast diffuser and semiconductor (3 eV bandgap), so it is more difficult to achieve high resistivity
  - AlN has a larger bandgap (6 eV) and is CMOS compatibility. Early work had problems with intrinsic stress. Good thermal conductivity, transparent.
  - Piezoelectric properties are similar between the two
- Thin-film bulk acoustic wave resonators (FBARs)
  - AlN has high bulk wave velocity (10 km/sec), high modulus, low density
  - Operation frequency related to thickness (thinner is better)
  - Moly electrodes used for good orientation, large grain size, and low roughness
  - Critical: roughness, large grains, uniformity
- Actuators
  - High resistivity for low power
  - High d_{33}, low losses for resonant applications
  - Competitive with electrostatic actuators, lower power
  - Piezoelectrics have the reputation of being hard to fabricate in the MEMS community

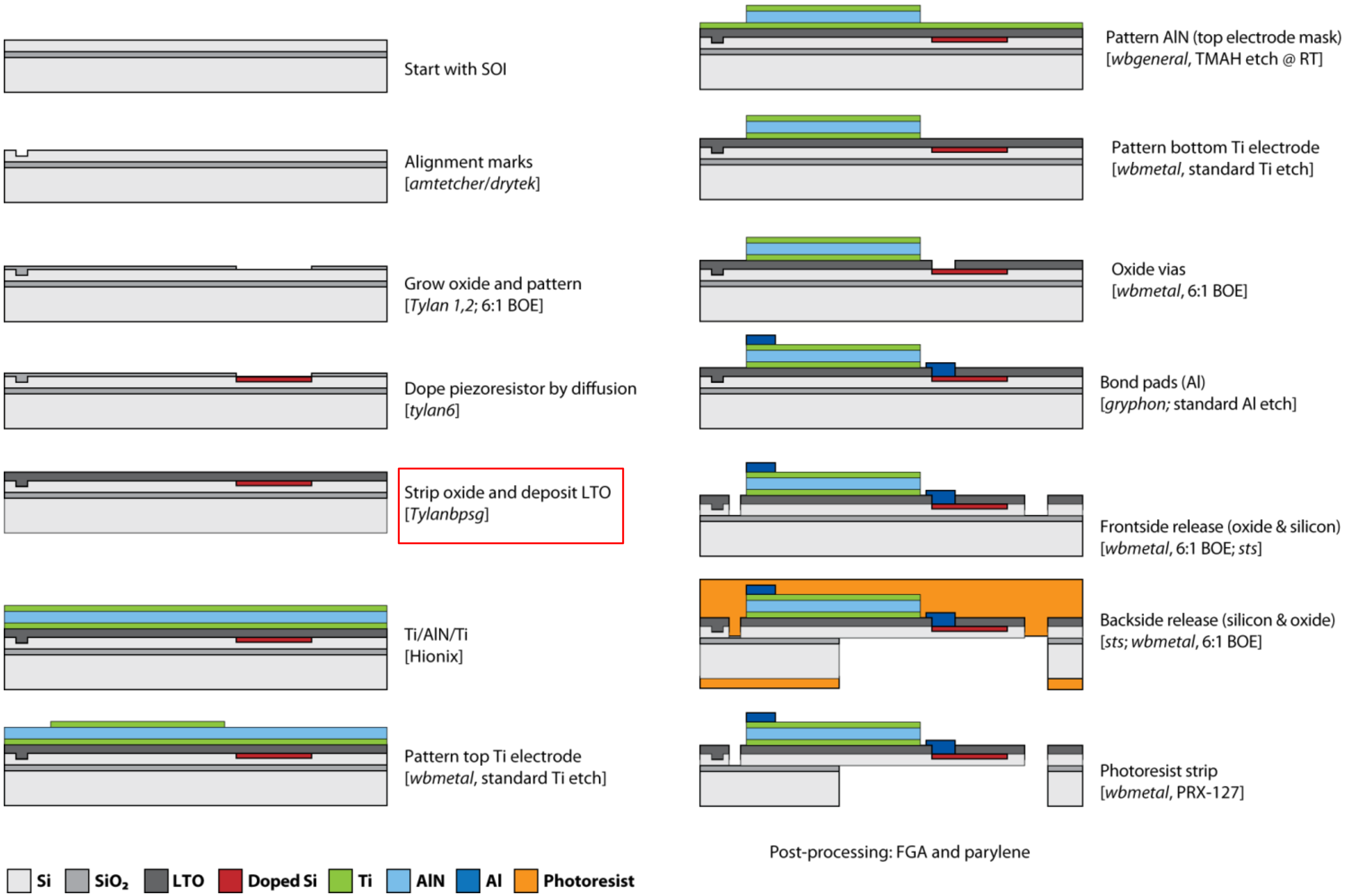
Piezoresistor and Piezoelectric



Piezoresistor and Piezoelectric



Piezoresistor and Piezoelectric



Goals

- Transducers '09 (Denver, CO)
  - Working silicon cantilevers with aluminum nitride thin films, aluminum nitride characterization, thin-film post-CMOS compatible integration
  - Todo: XPS for composition, FIB for microstructure, repeat XRD
  - Also trying to write something up for Micro & Nano Letters
- Timeline
  - Currently working on another conference abstract, will send Transducers abstract to you the middle of November
- What I need from you
  - Fast turnaround on the next round of deposition (will provide wafers approx. Nov. 10^{th})
  - Power vs. flow rate characterization
  - Determine deposition temperature

Acknowledgements

- Research help from
  - Bryan, Nahid, Sung-Jin, Piezoresistor Subgroup
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  - NSF Graduate Research Fellowship Program
- Research supported by
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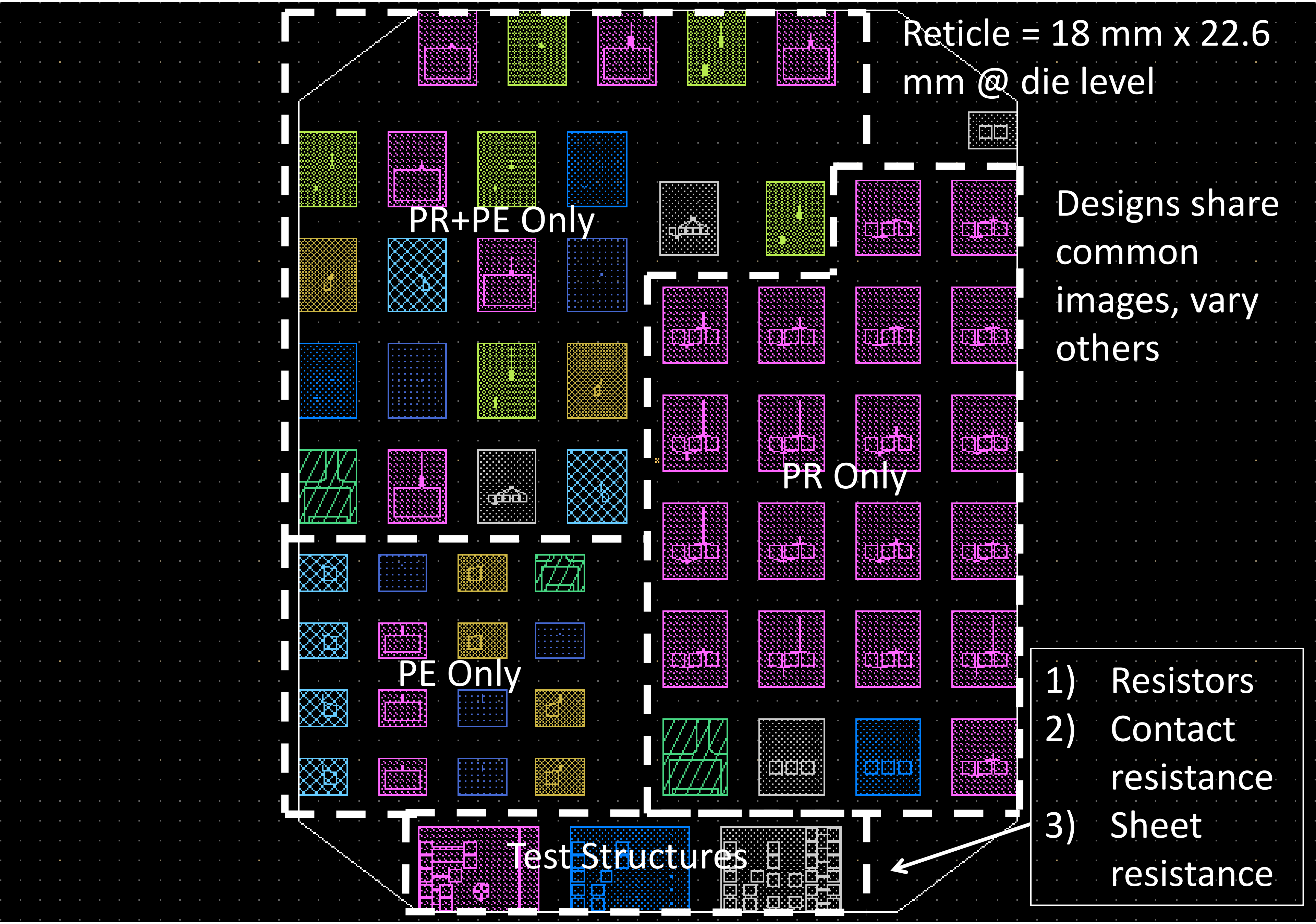
  - NSF IREE Award ECS-0449400
  - NIH R01

Stanford Microsystems group (2007)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