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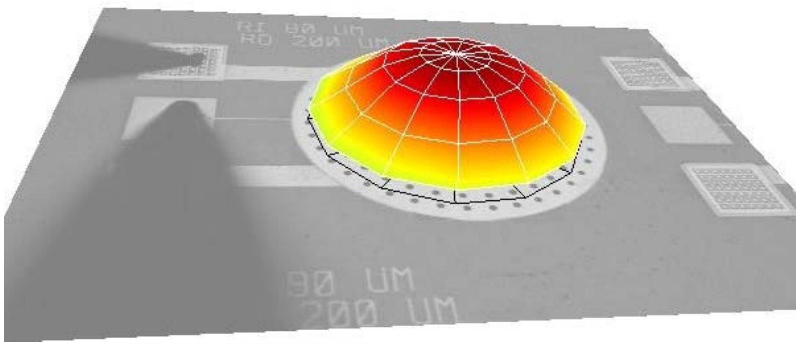

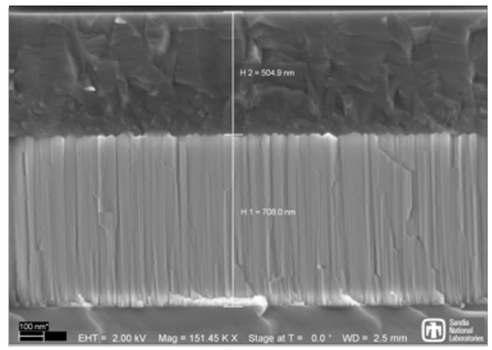

Development of an Aluminum Nitride-Silicon Carbide Material Set for High Temperature Sensor Applications

Benjamin A. Griffin, Scott D. Habermehl,
Peggy J. Clews and Chanju Fritch

9/29/2015

U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

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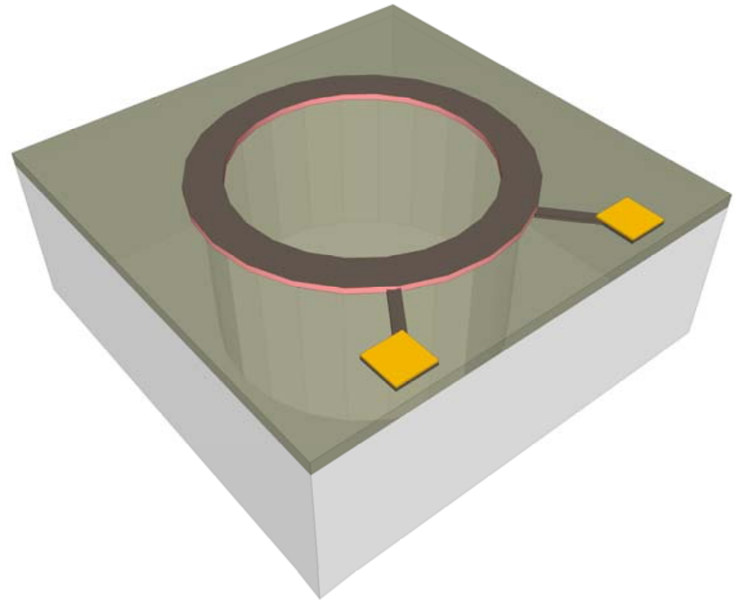
Outline

- Background
- Film Development
- Fabrication Process
- Initial Results

AlN/SiC Width Extensional Resonator

Objective and Program Relevance

- Develop material set to enable development of extreme temperature capable transducers
- Applications for transducers that can withstand extreme temperatures
  - Gas turbines (1250˚C)
  - Hypersonic flight research (755˚C)
  - Automotive engines (300-1000˚C)
  - Nuclear power plant (300˚C)
  - Coal power plants (700˚C)



*grc.nasa.gov

Current State-of-the-art

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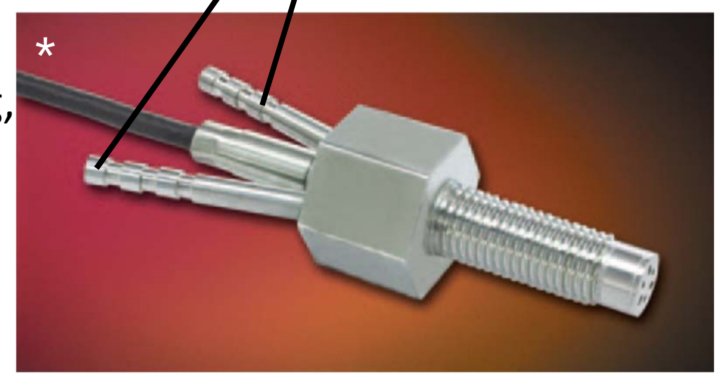
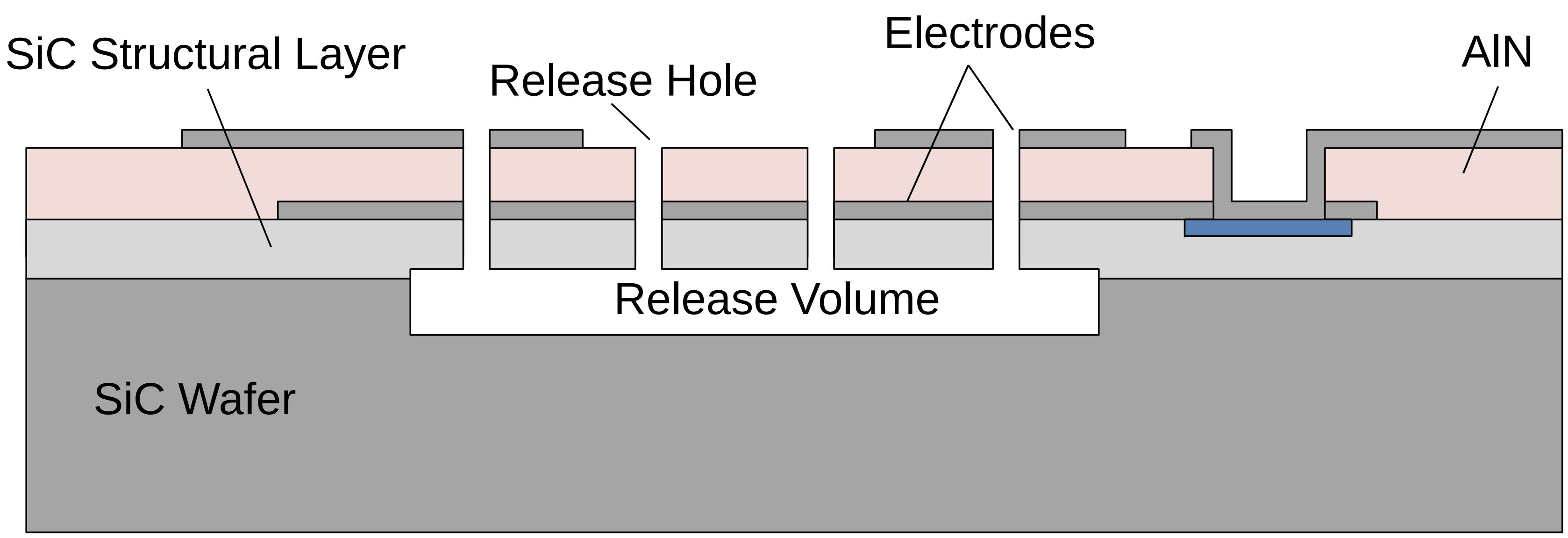
- Example: Kulite Pressure Transducers
  - MEMS Piezoresistive technology
  - XTEH-10L-190
    - Temperature limited to 540˚C
  - EWCTV-312
    - Water/air cooled
      - Inherently changing the temperature boundary condition
      - SWaP cost
    - Temperature limit: 1093˚C with water at 35 psig, 1 gal/min flow

Water/air cooling jacket

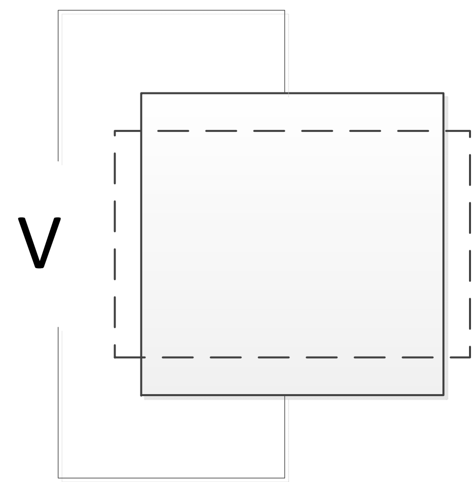


Proposed Technology

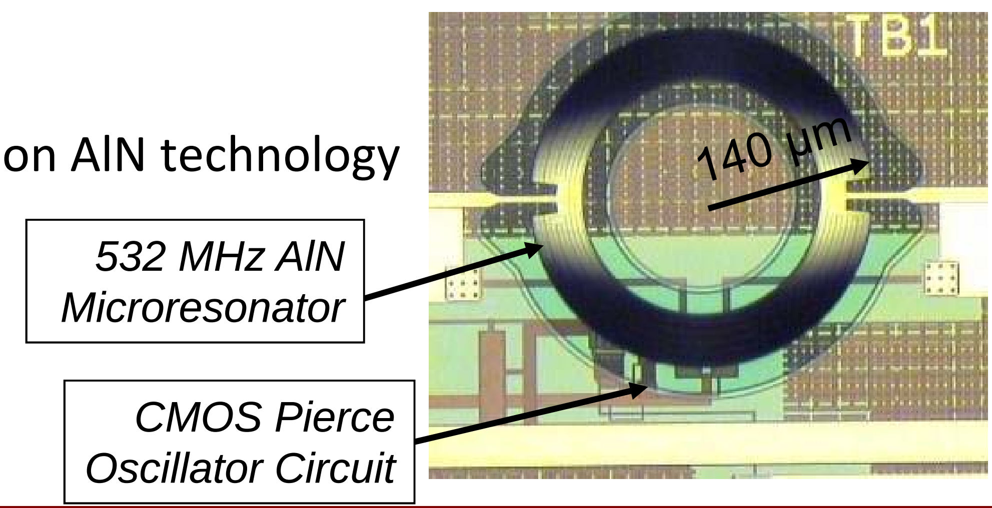
- Developing a MEMS material set that combines
  - Aluminum nitride (AlN) piezoelectric thin film
  - Silicon carbide (SiC) structural film and wafer
  - High temperature capable electrodes
    - Titanium/Titanium nitride (Ti/TiN)
    - Nitrogen doped SiC (SiC:N)



Aluminum Nitride (AlN)

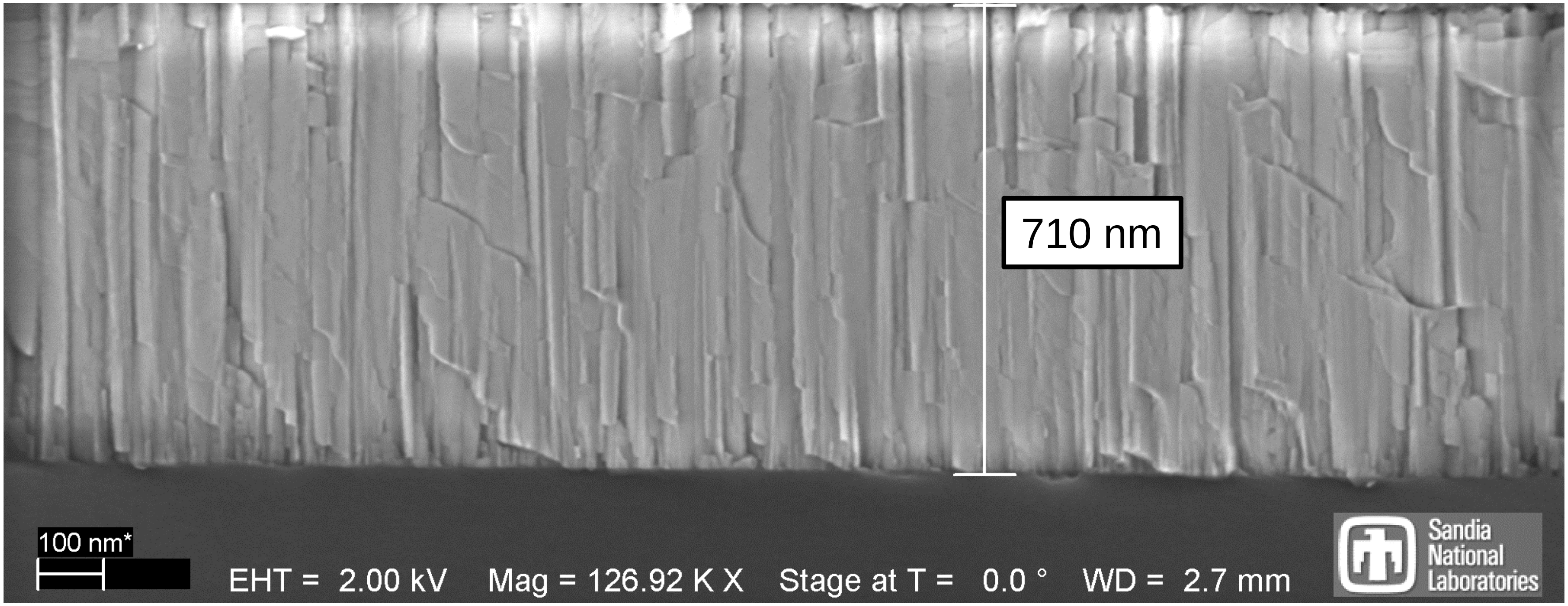


- Piezoelectric thin film
  - Non-ferroelectric
    - Dipole alignment during fabrication
    - No Curie temperature
  - Melting point of 2,200˚C
  - Piezoelectric response has been measured to 1,150˚C
  - High thermal, electrical, and mechanical strength while chemically inert
- A Sandia strength
  - 2011 R&D 100 Award based on AlN technology



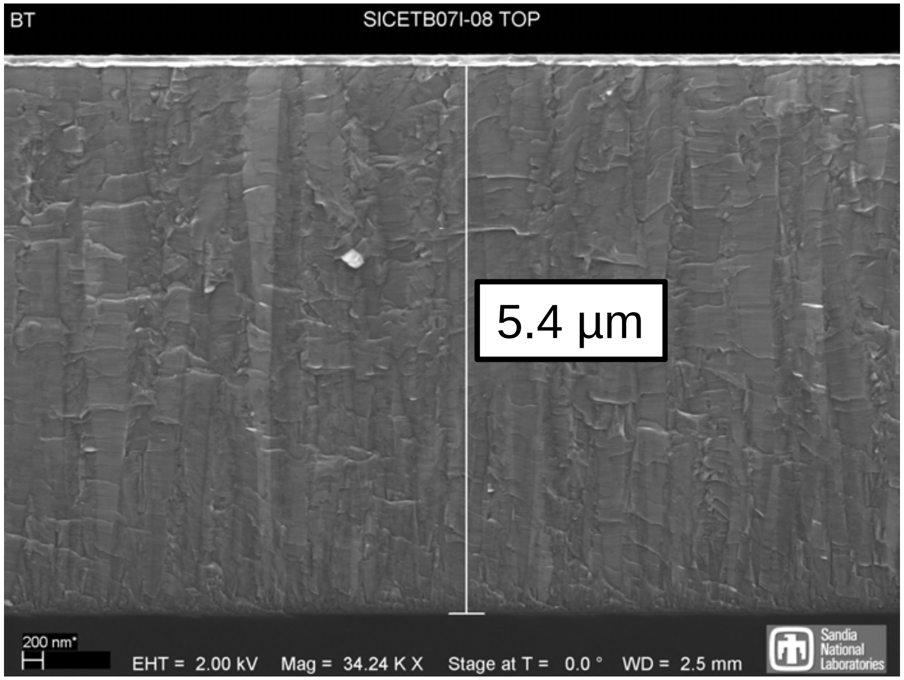
Columnar AlN

- Sputter deposited at relatively low temperature (~350˚C)
  - Dipole alignment achieved during deposition
    - Sputter parameters
    - Substrate material and roughness



Silicon Carbide (SiC)

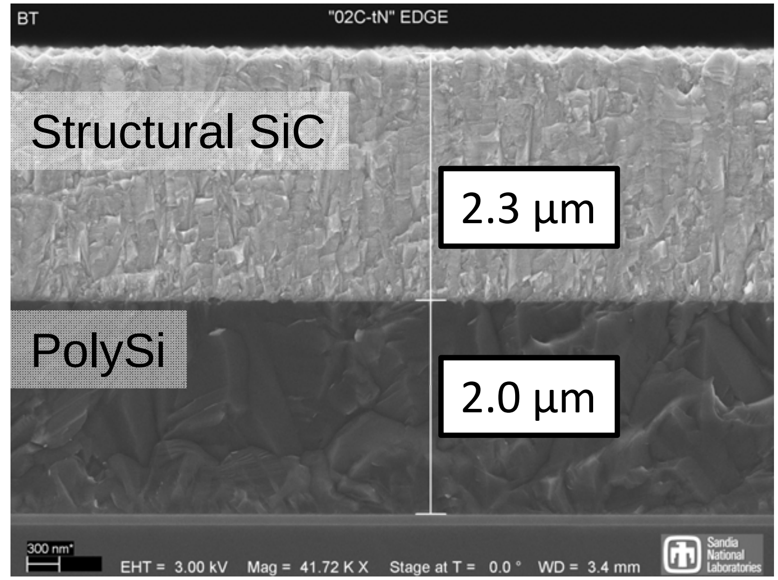
- Upper temperature limit >2500˚C, where it sublimates rather than melts
- Highest mechanical strength of semiconductors with the exception of diamond
- Coefficient of thermal expansion nearly matched to AlN
  - AlN: 4-5 ppm/K
  - SiC: 4 ppm/K

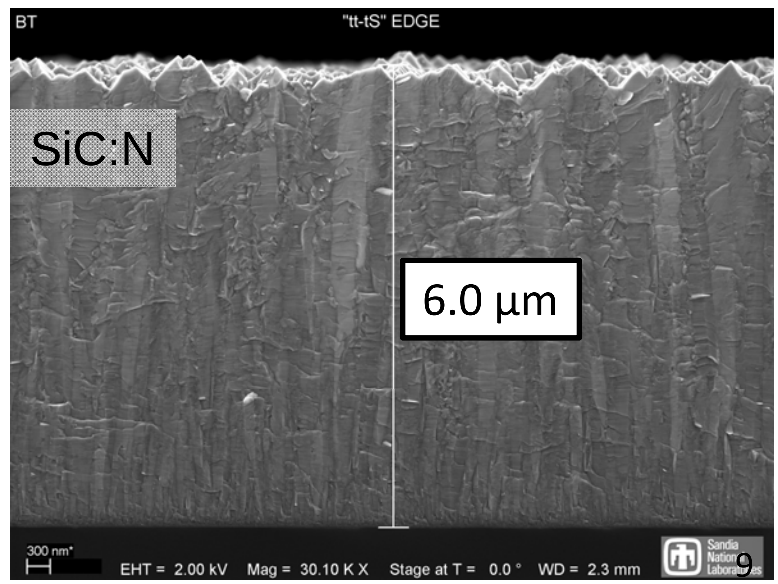


Habermehl, S., et al. (2012). "Formation of stress-controlled, highly textured, alpha-SiC thin films at 950 degrees C." Journal of Applied Physics **112** (1).

SiC Films

- Structural SiC
  - LPCVD at 850-950˚C
  - Pressure ~250 mTorr
  - Stress controlled by varying gas flow rates of the SiH_2Cl_2 (DCS) and $C_2H_2Cl_2$ (DCE)
  - Mainly hexagonal 6H-SIC
  - Surface roughness ~50 nm
- SiC:N Electrode
  - Additional flow of NH_3
  - Resistivity ~1 milliΩ·cm





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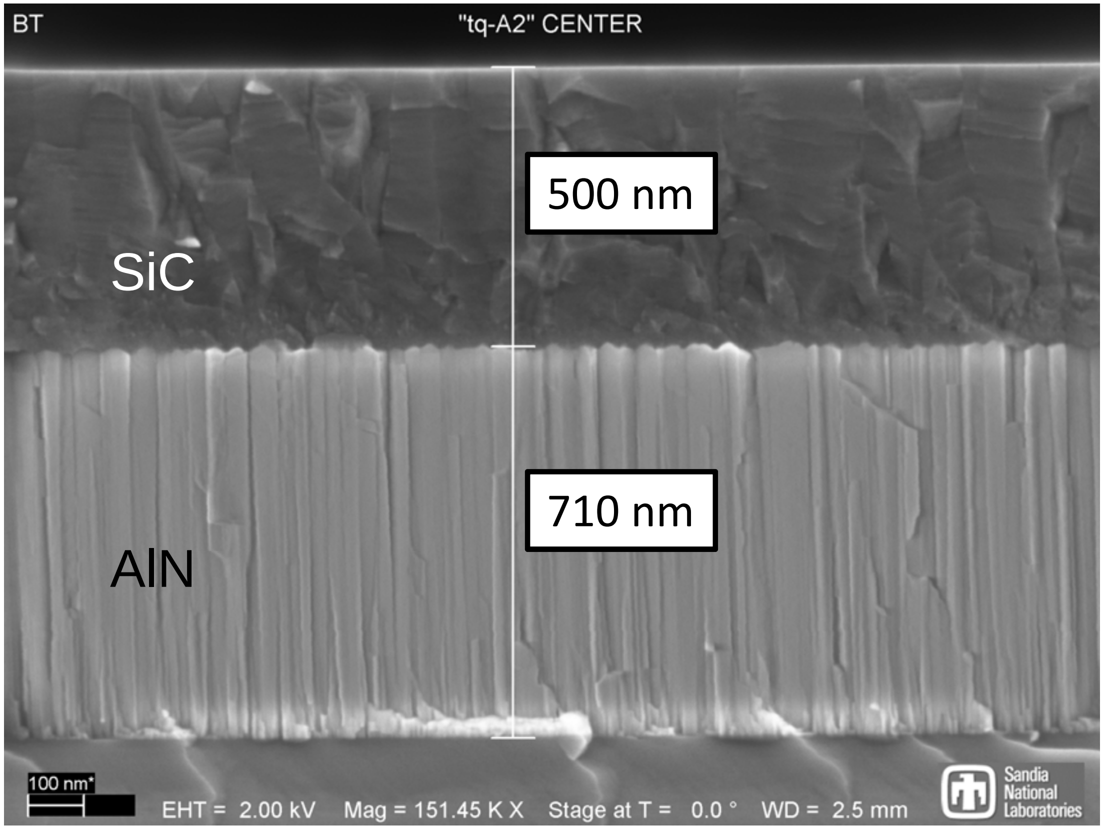
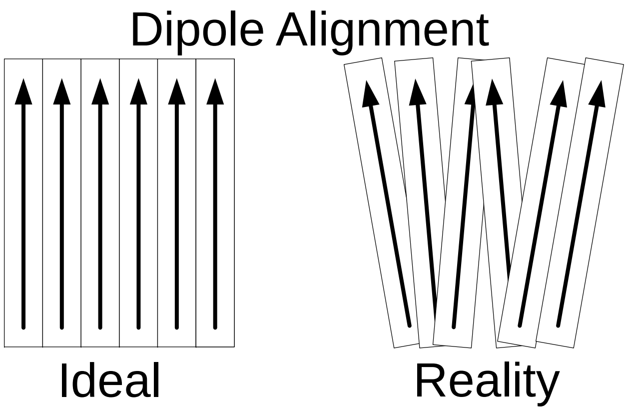
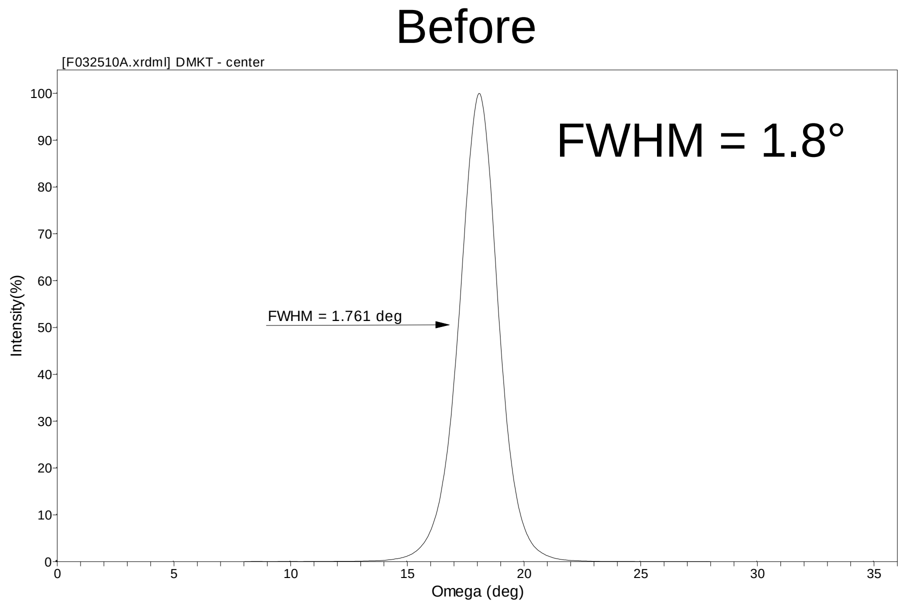
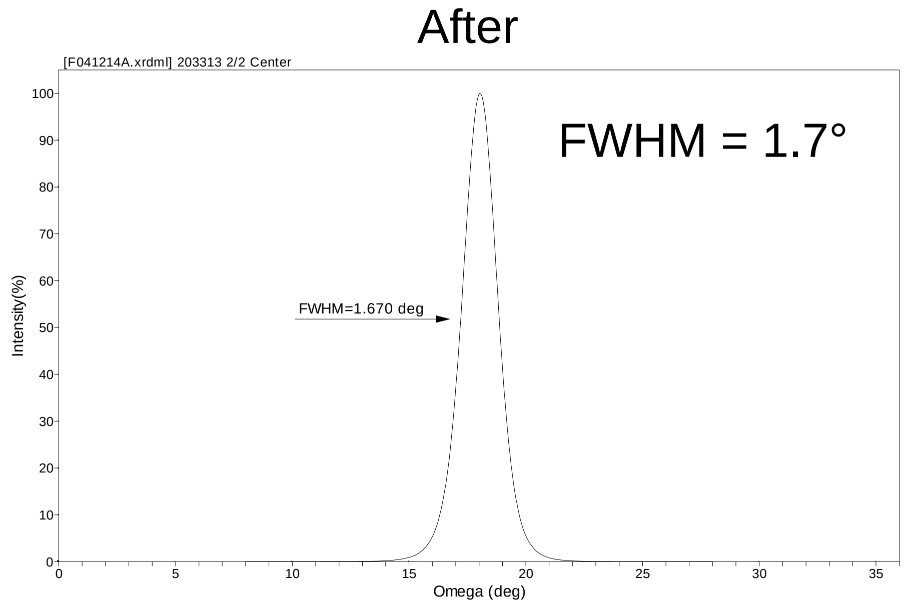


Annealed AlN Alignment

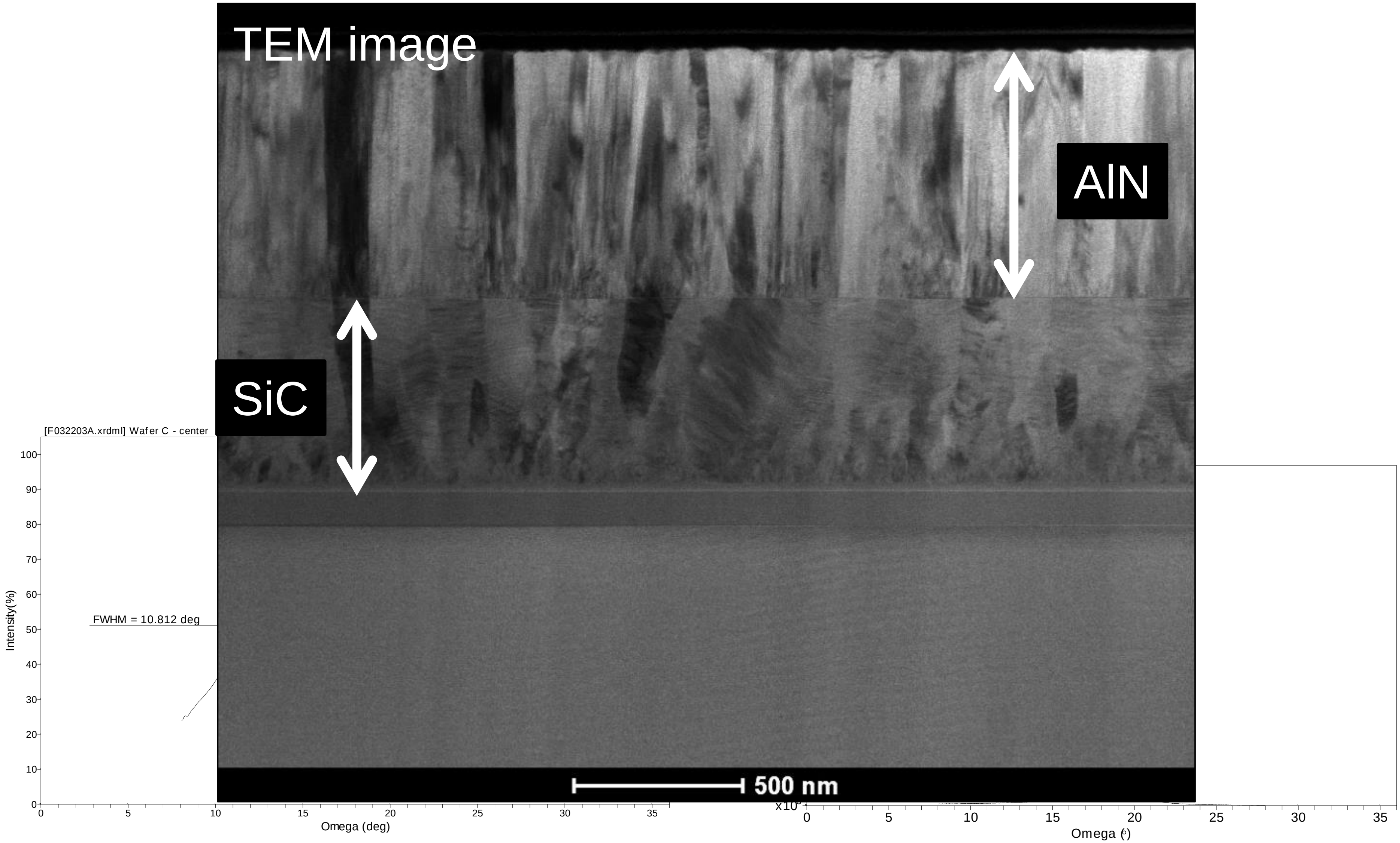
- Anneal of AlN on titanium / titanium nitride bottom metal electrode at 950˚C for 3 hr
  - X-Ray diffraction measurements to determine if AlN is still columnar
  - In general, the goal is <2˚







FWHM of AlN on SiC Electrode



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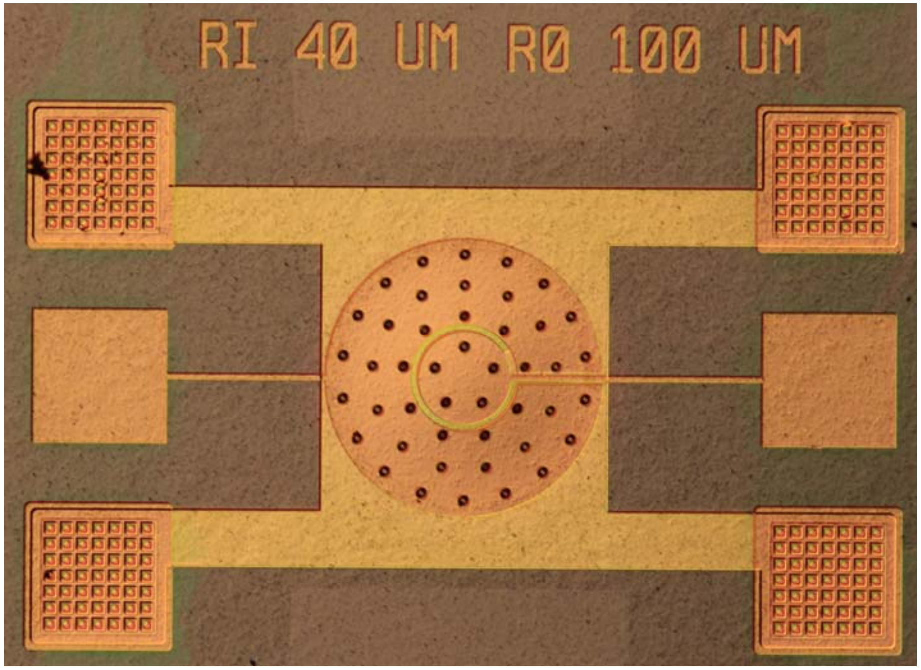
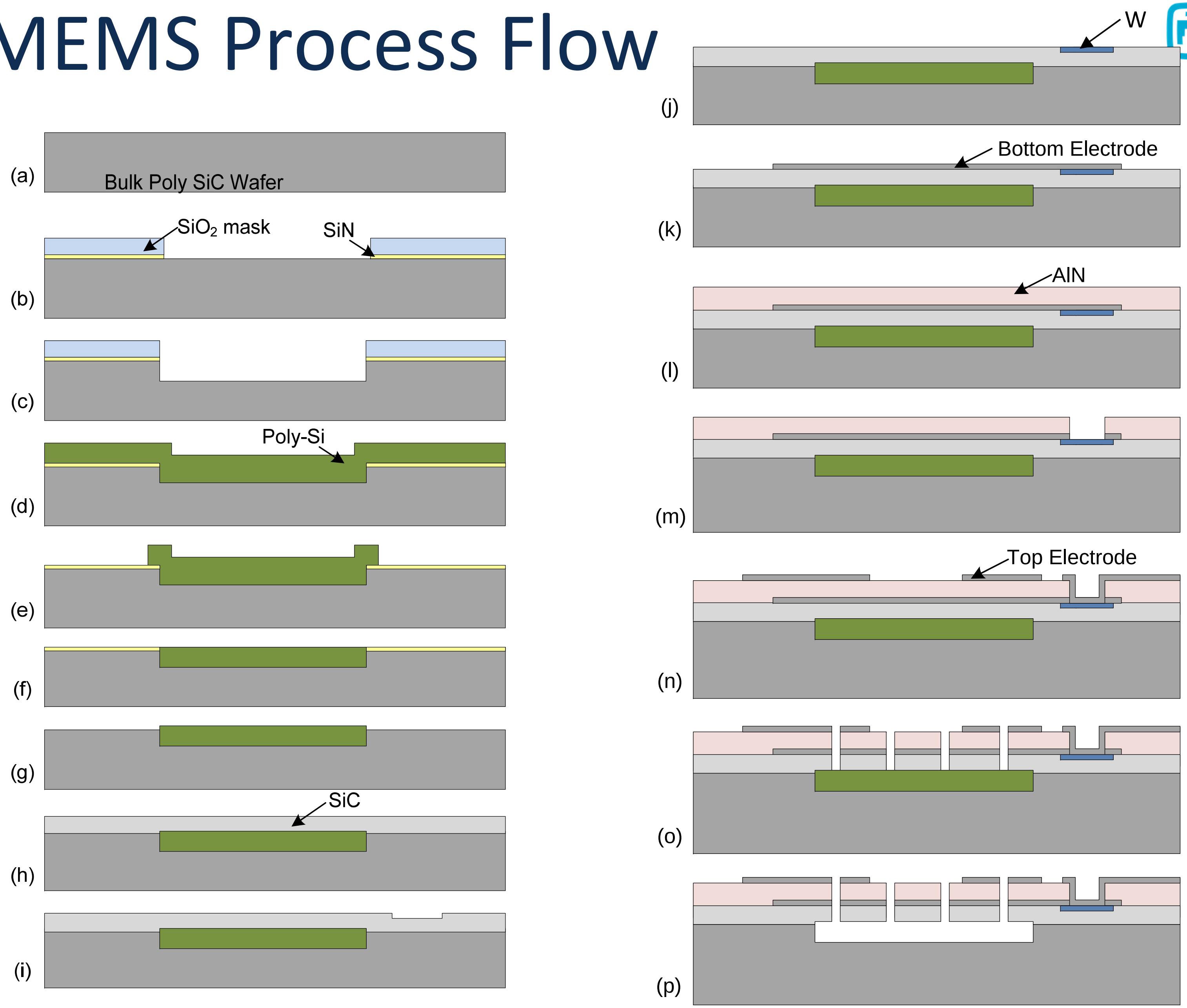


1 MHz AlN/SiC Ultrasonic Transducer

XMEMS Process Flow



Devices Fabricated

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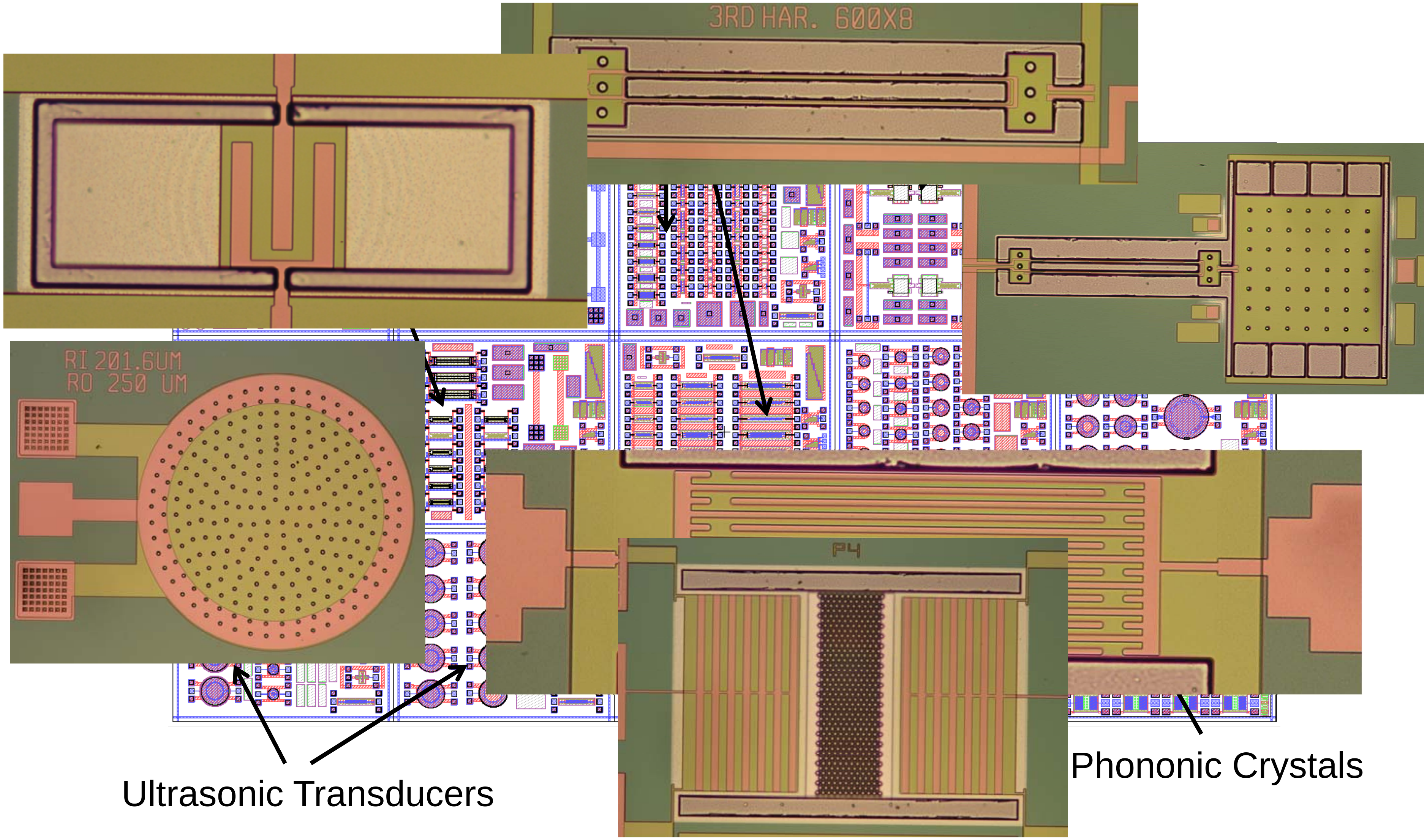

Outline

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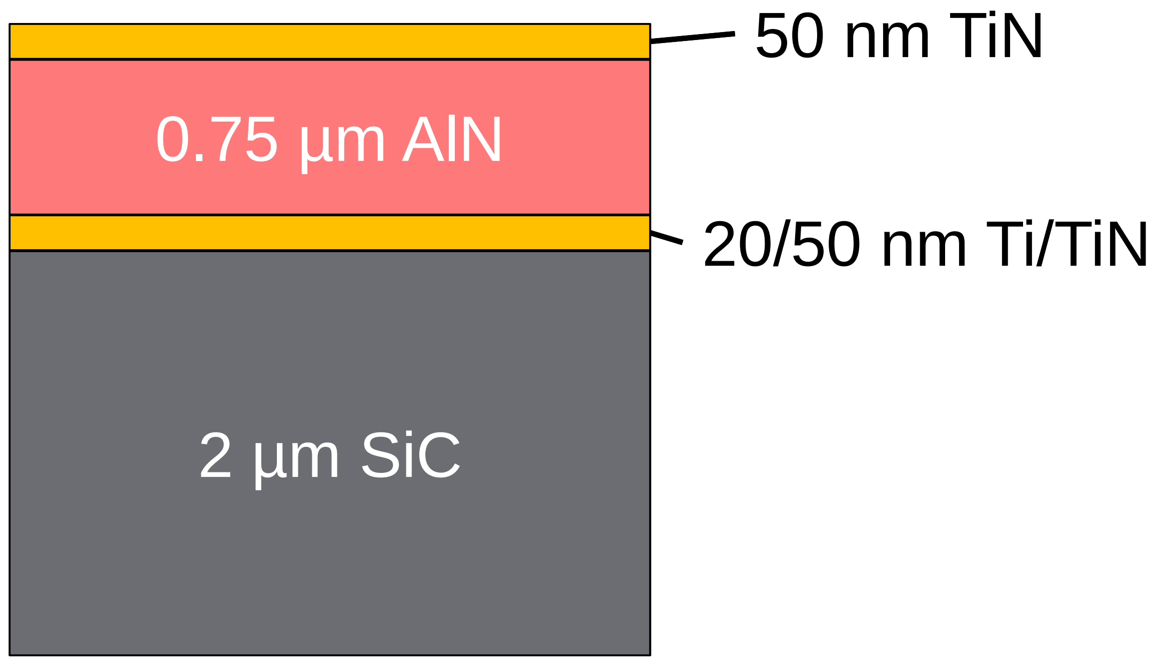

Piezoelectric Diaphragm

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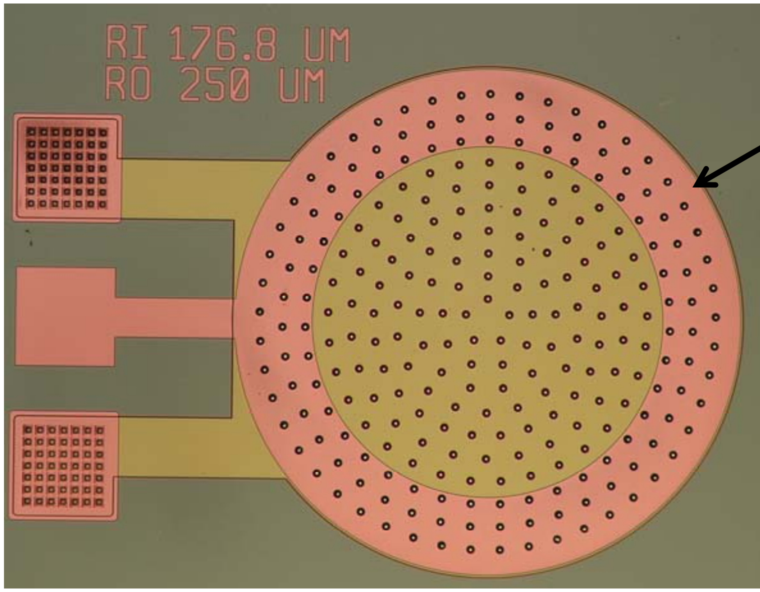
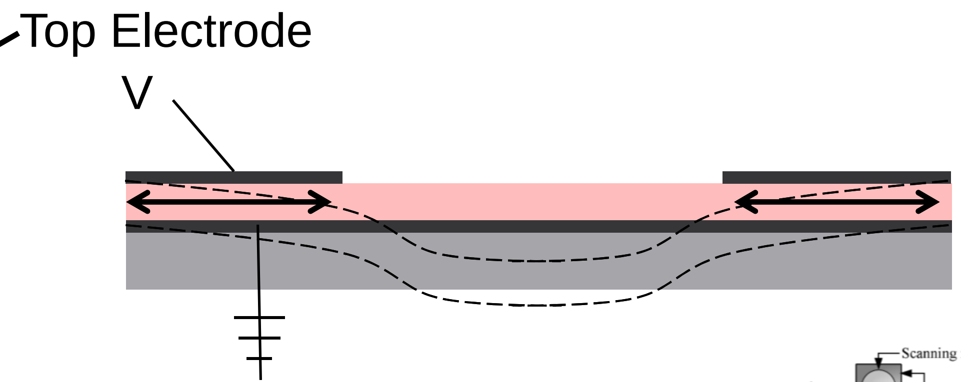
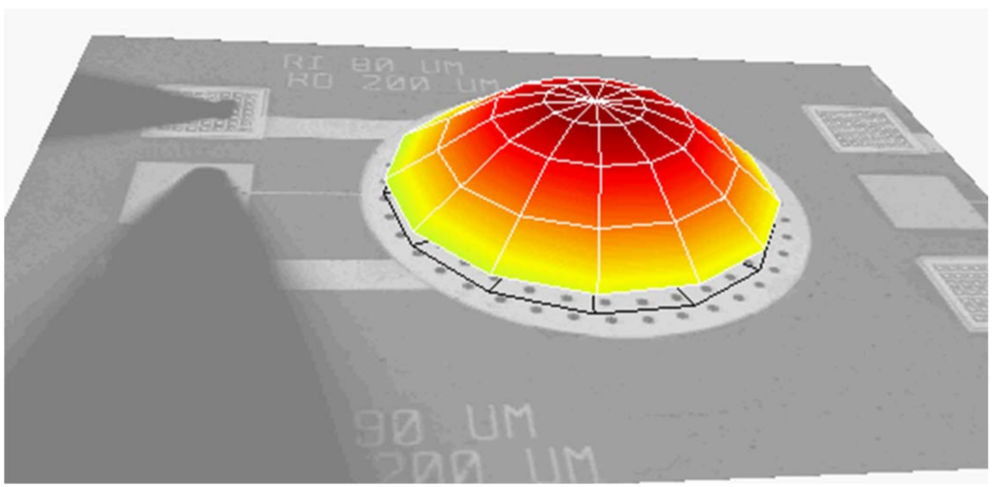
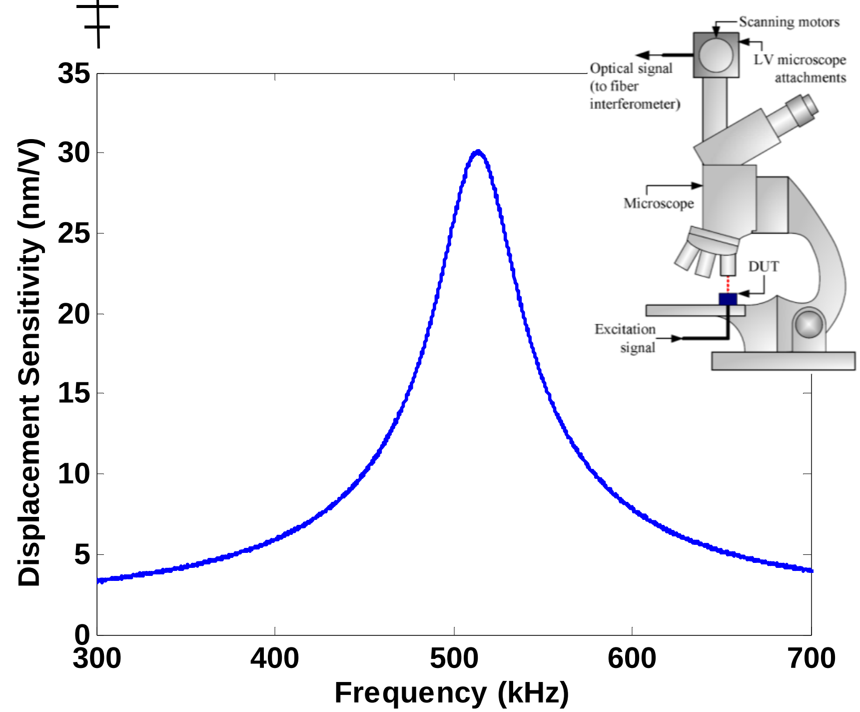


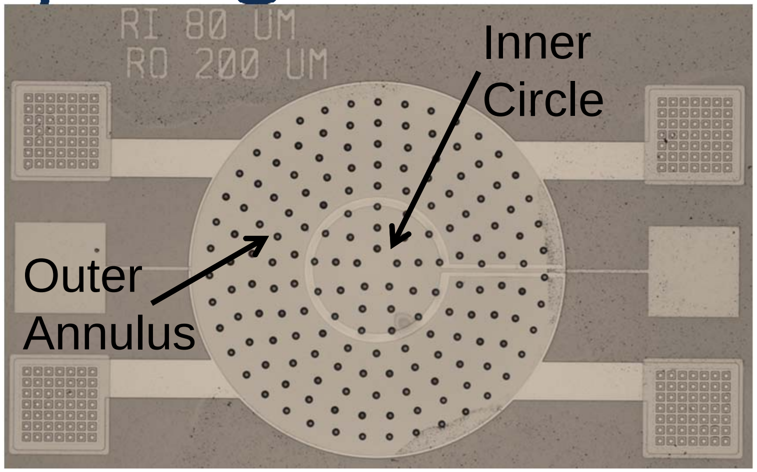


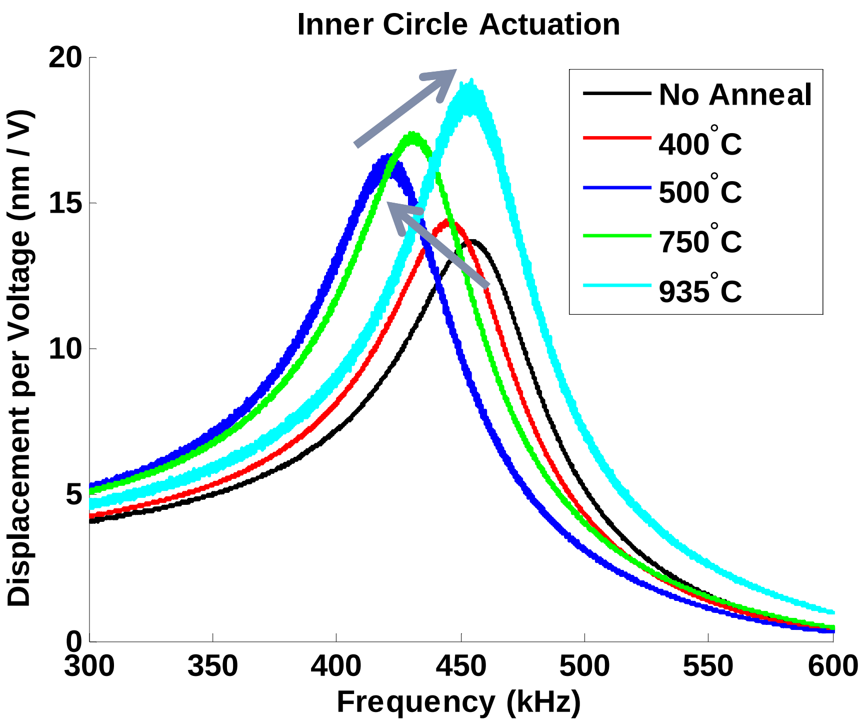


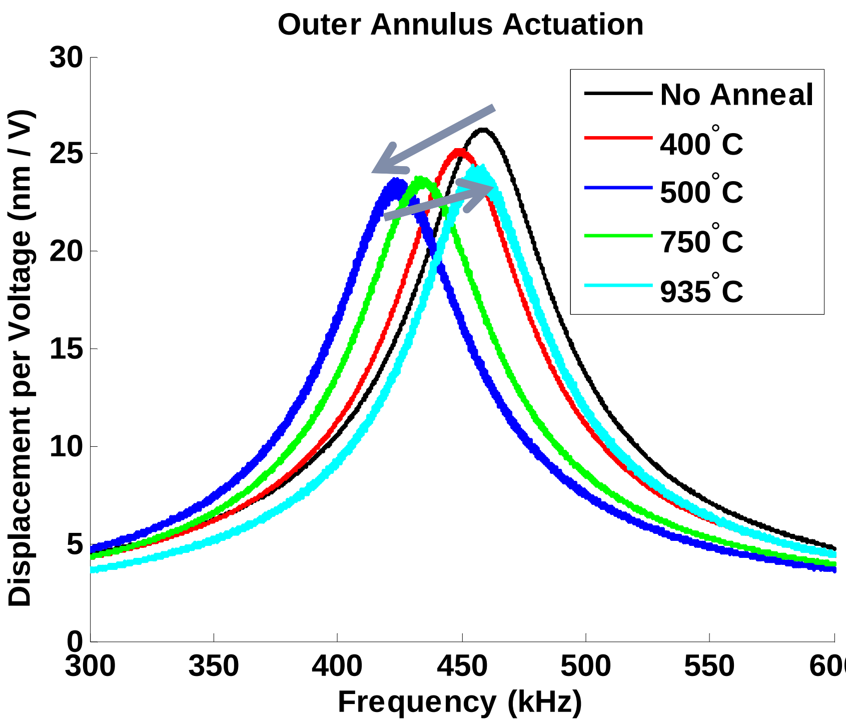


Annealing Results - Diaphragm

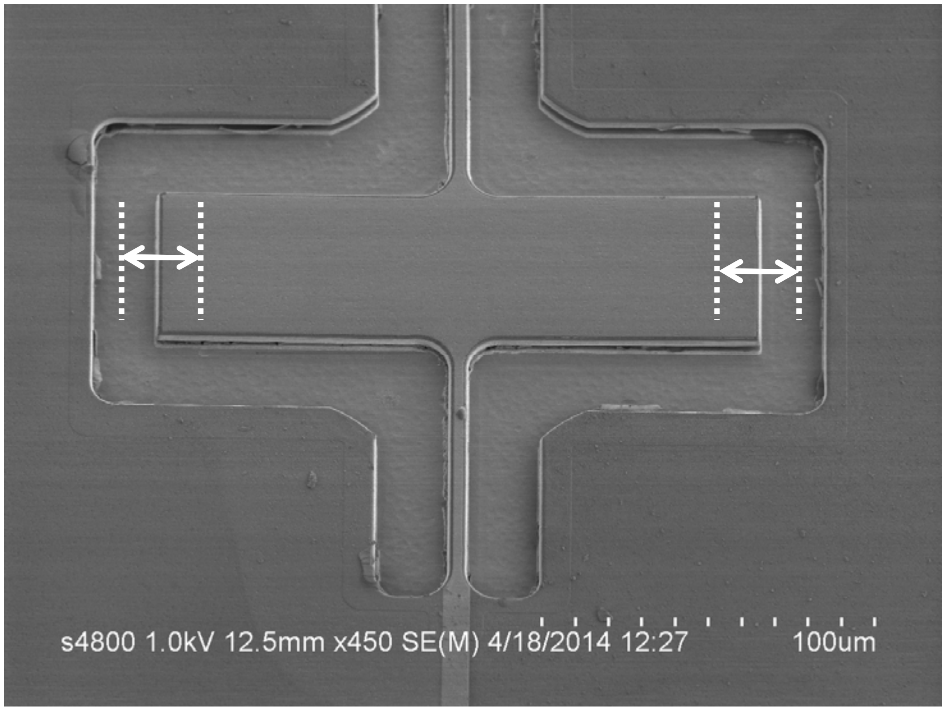
- Rapid Thermal Anneals
  - Argon purged
  - Vacuum of 1 Torr
  - Temperature held for 2-7 minutes

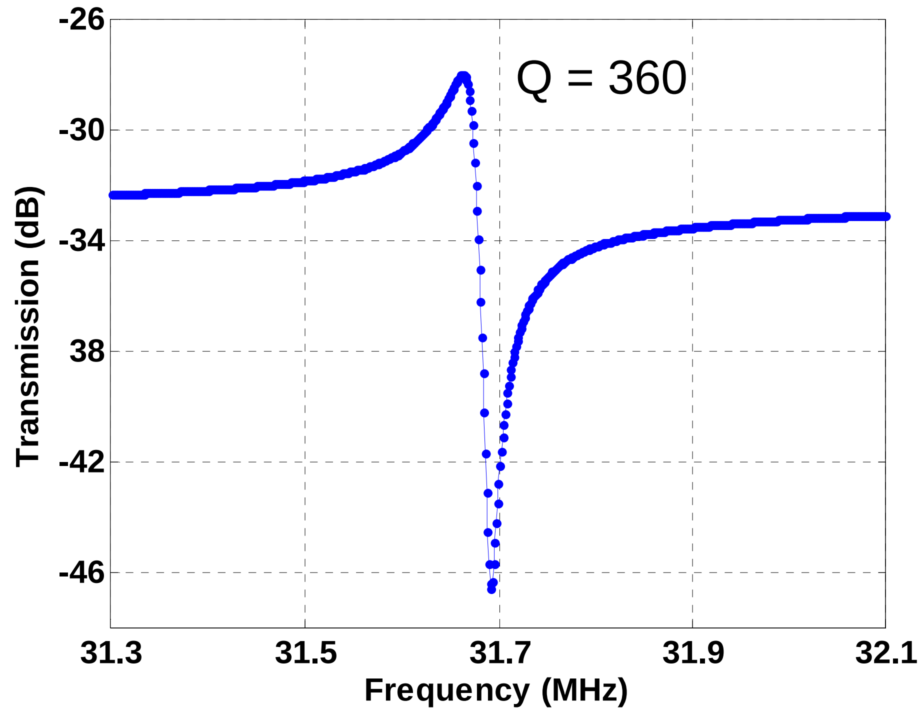




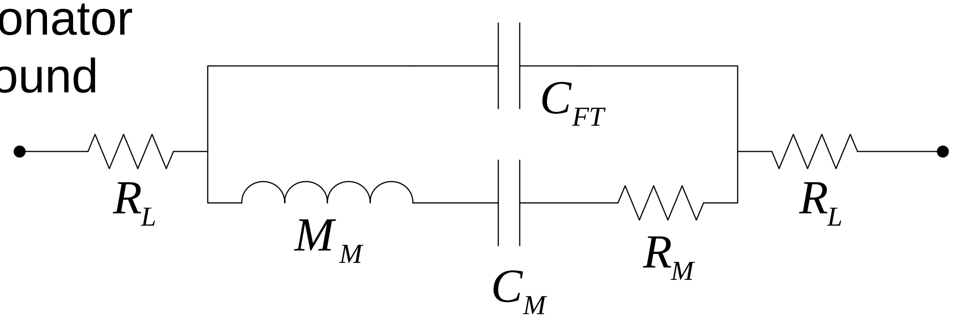


Microresonator



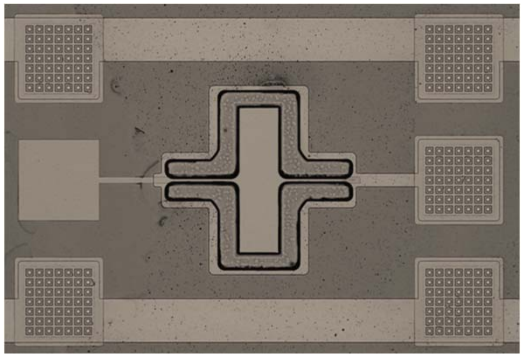


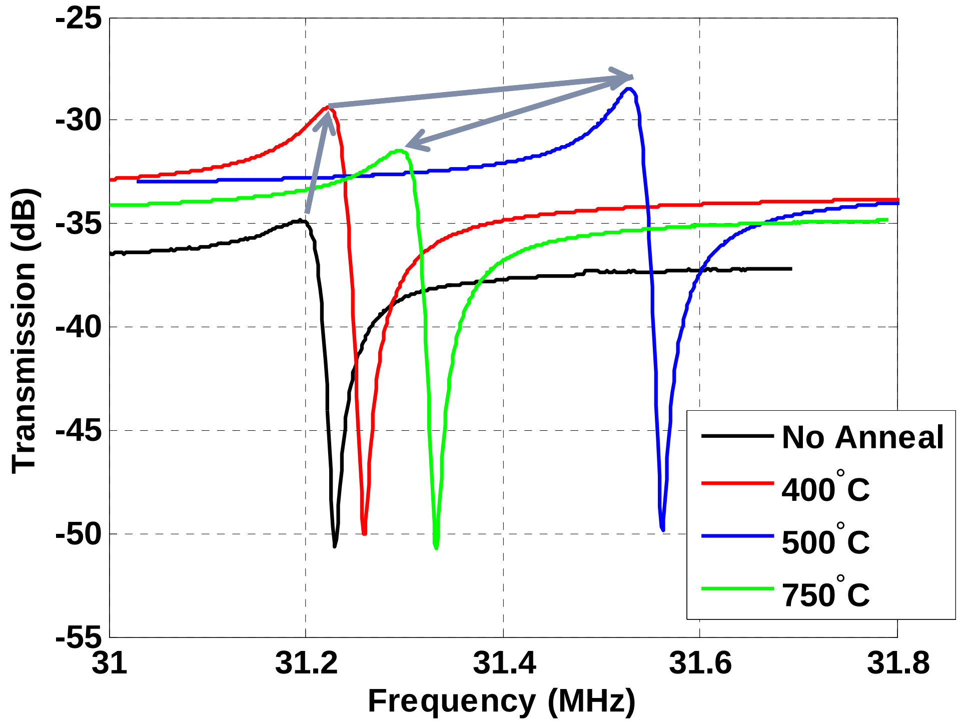
- Length extensional contour-mode resonator
- Thickness weighted estimate of the sound speed 11.1 km/s
  - Model: 30.75 MHz
  - Measured: 31.66 MHz

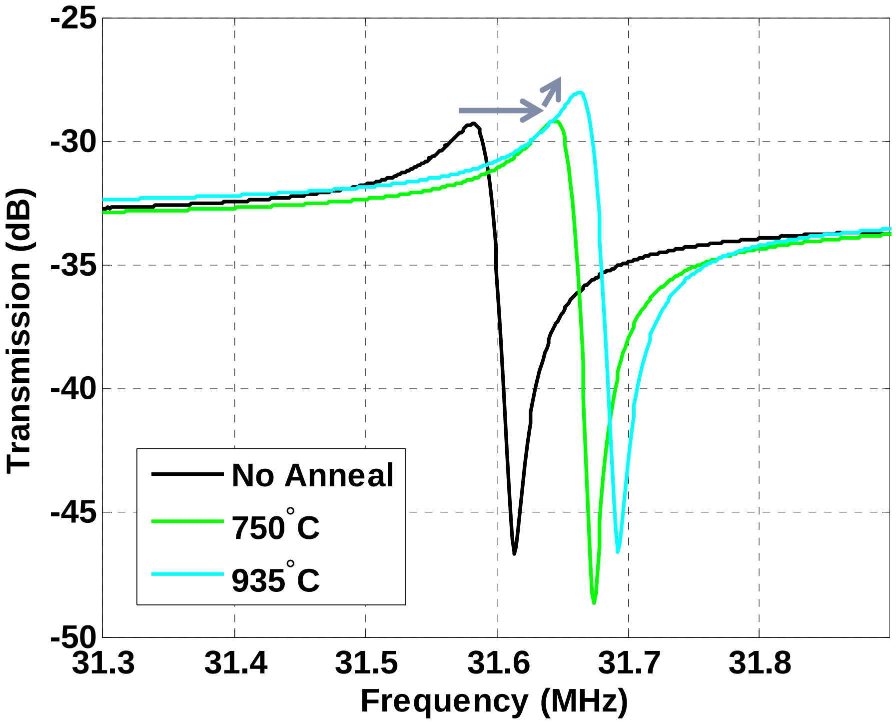


Annealing Results - Microresonator

- Rapid thermal anneals
- Inconsistent trends with annealing temperature
- Device survives and operates post 935˚C anneal
- Maximum frequency shift is less than 3%

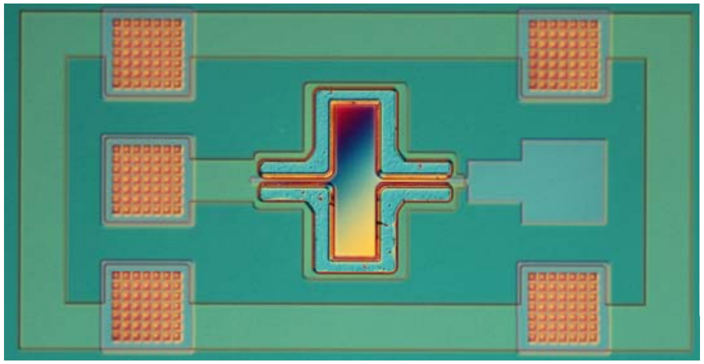


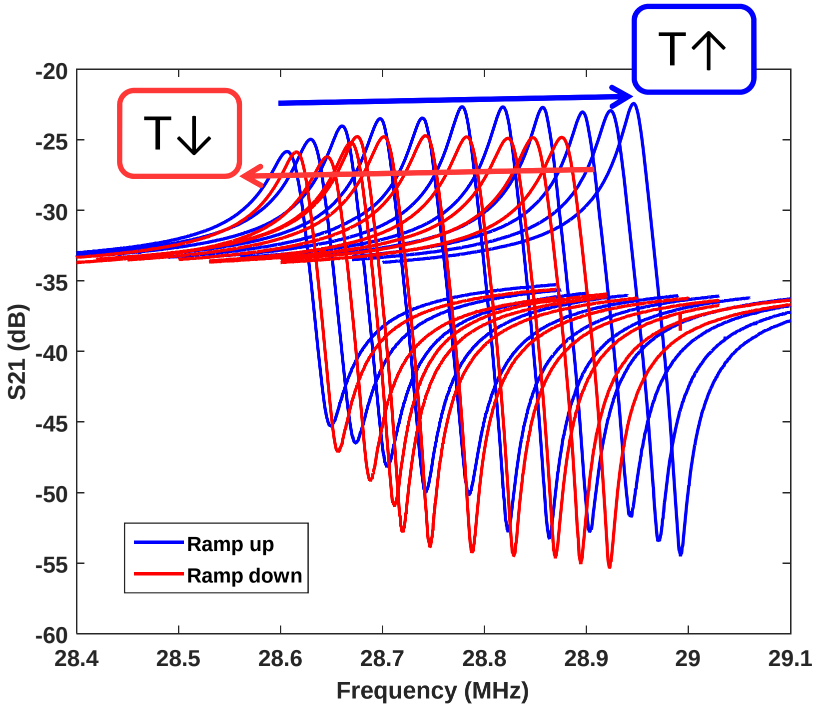


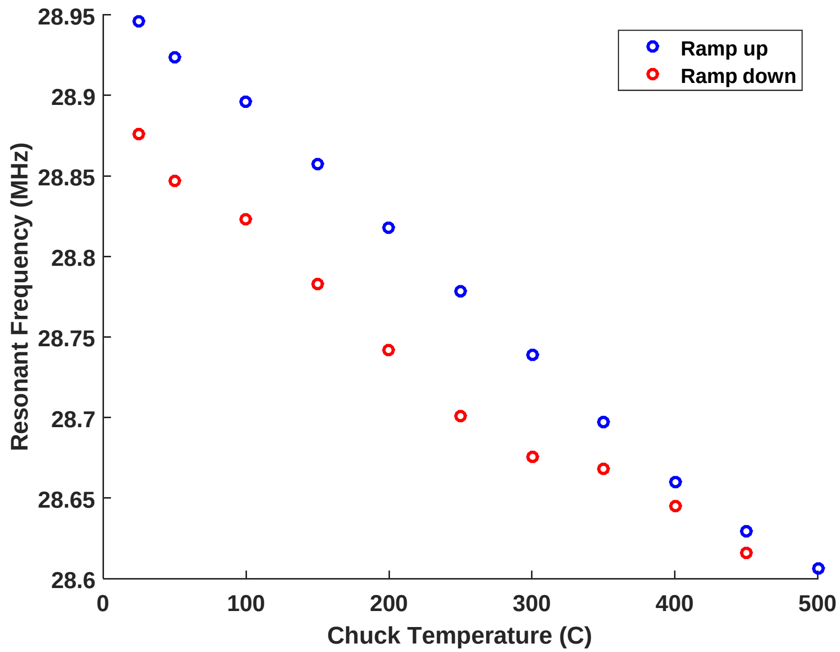


In Air Temperature Testing

- Heated chuck from to 500˚C in steps of 50˚C
- Temperature hysteresis loop results in permanent frequency shift of 2,400 ppm

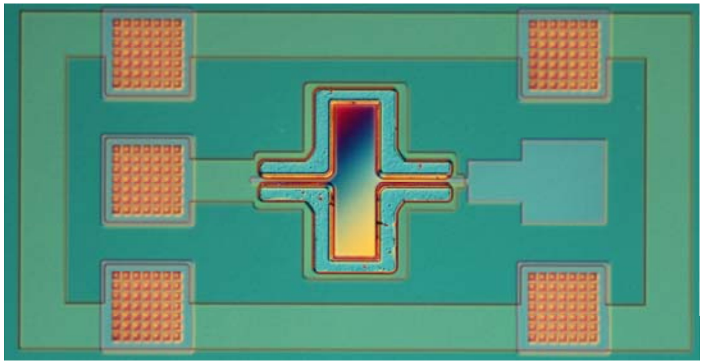


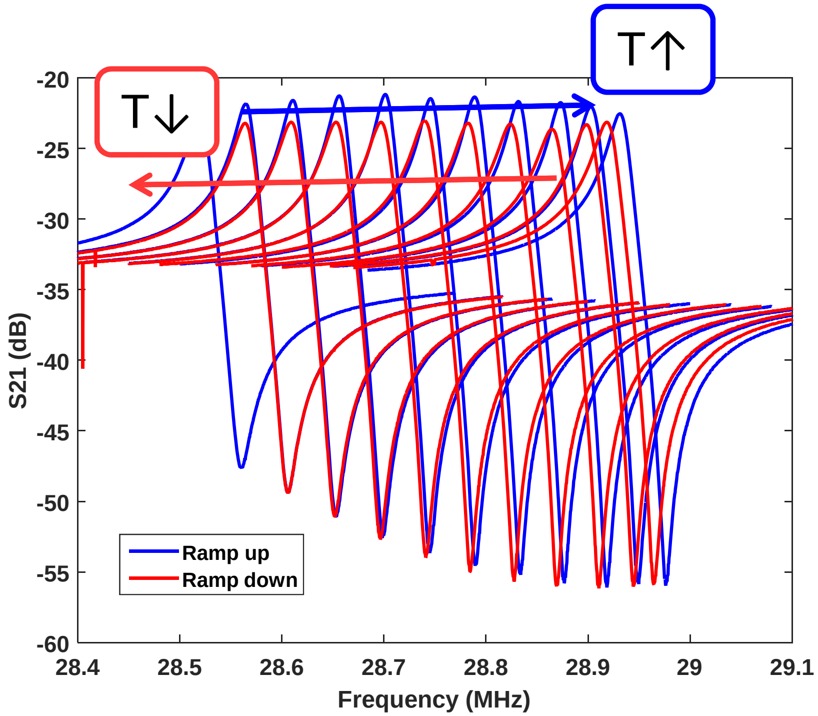


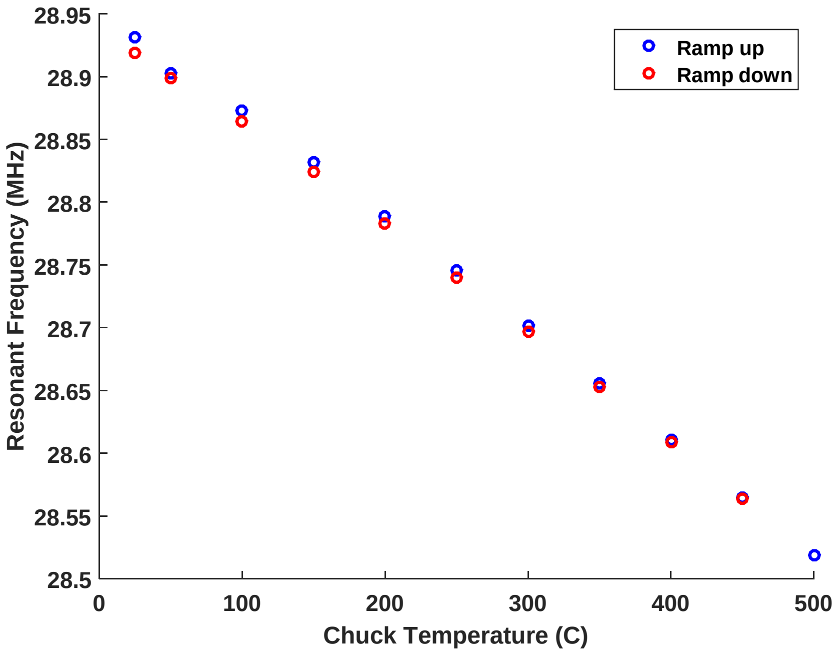


Pre-anneal Results

- RTA at 650˚C before heated chuck testing
- Frequency shift decreases from 2,400 to 400 ppm

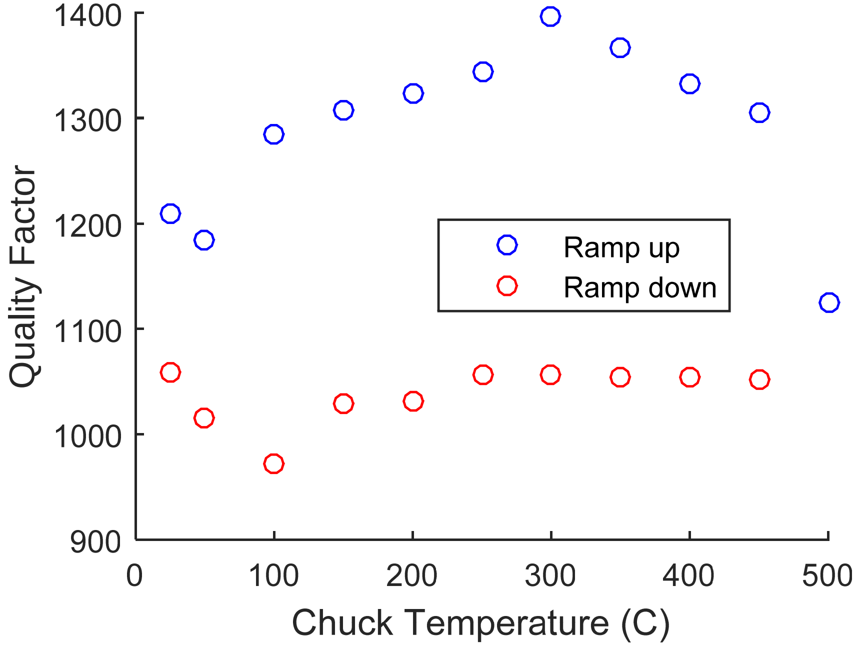


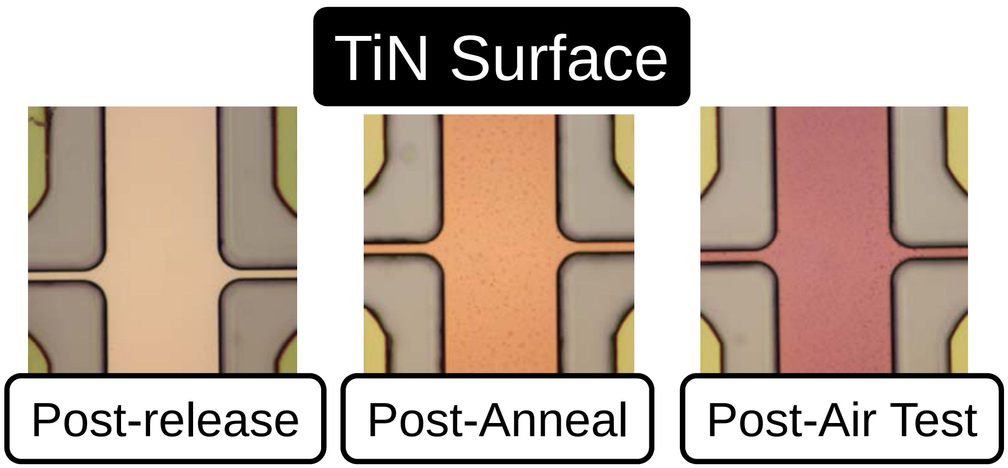


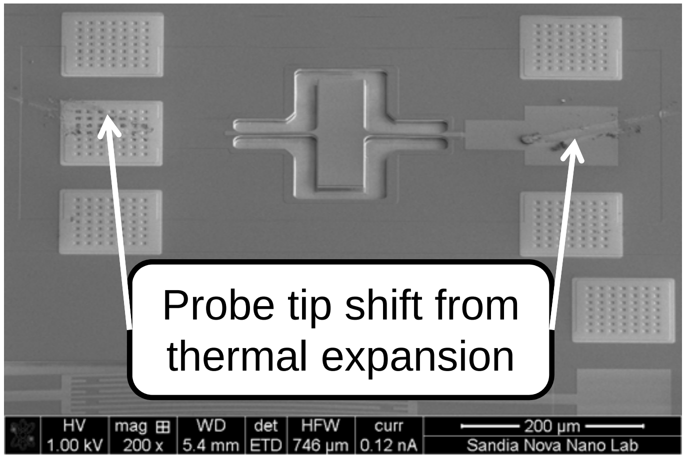


Quality Factor

- Quality factor degradation observed over temperature ramps
- Potential sources
  - TiN oxidation
  - Probe contact issues
  - Via degradation
  - AlN oxidation

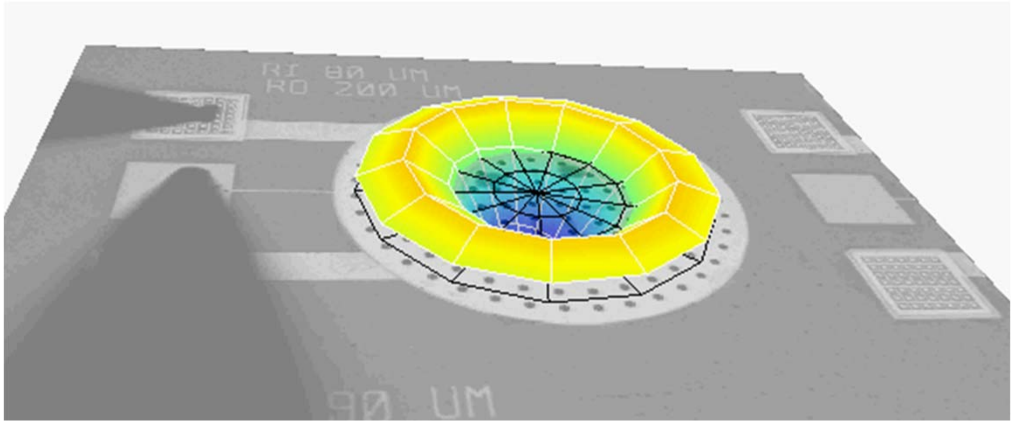






Conclusions and Future Work

- Successful development of the XMEMS process for AlN/SiC MEMS on poly-SiC wafers
- RTA results show device survival at 935˚C
- Initial in air testing shows device performance in air at 500˚C

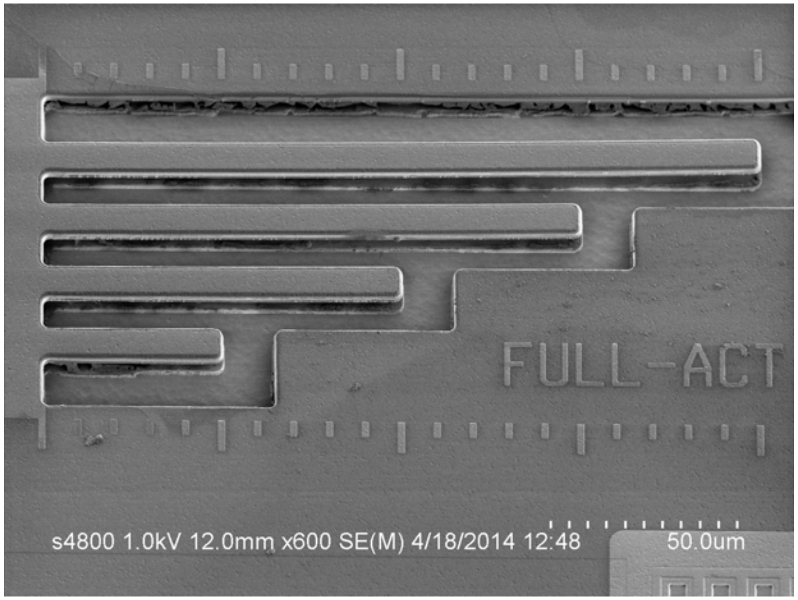


- Future Work
  - Analysis of Q degradation
  - Integration of other electrode materials

Acknowledgements

- Microelectronics Development Laboratory Staff at Sandia National Laboratories
  - M. David Henry
  - Lawrence Klebesadel
- SNL Laboratory Directed Research and Development (LDRD) Program

AlN/SiC Composite Cantilevers



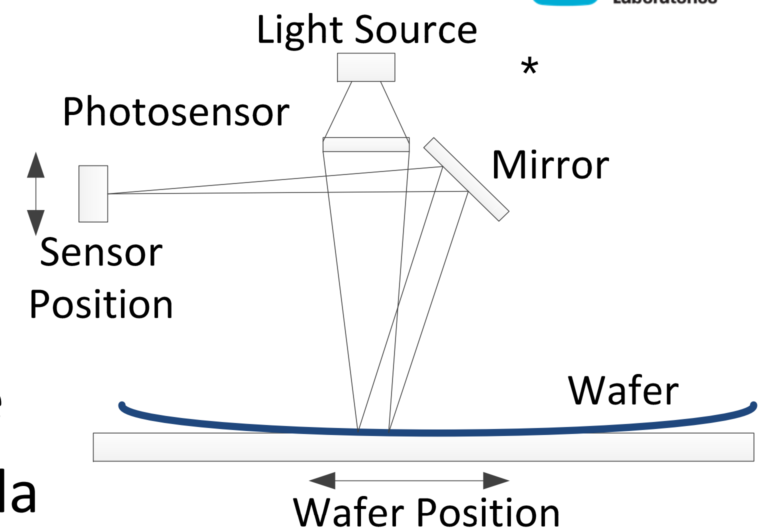
Contact Information
Ben Griffin bagriff@sandia.gov

Questions?

Backup Slides

Film Stress

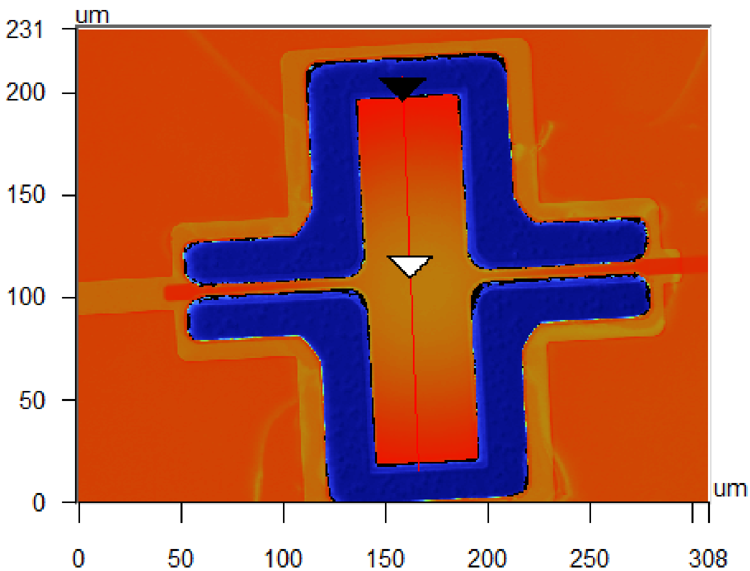
- Blanket films deposited successively
- Local wafer slope measured using non-contact wafer stress mapping system
- Linear curve fit gives radius of curvature
- Stress is extracted using Stoney's formula

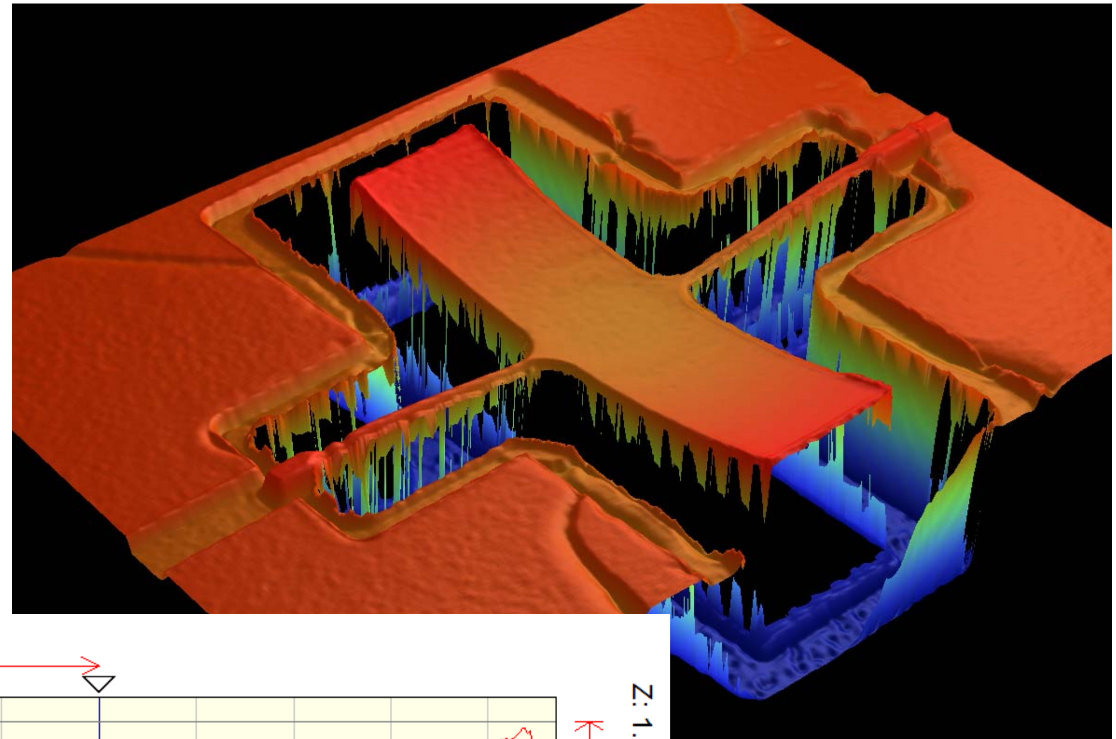


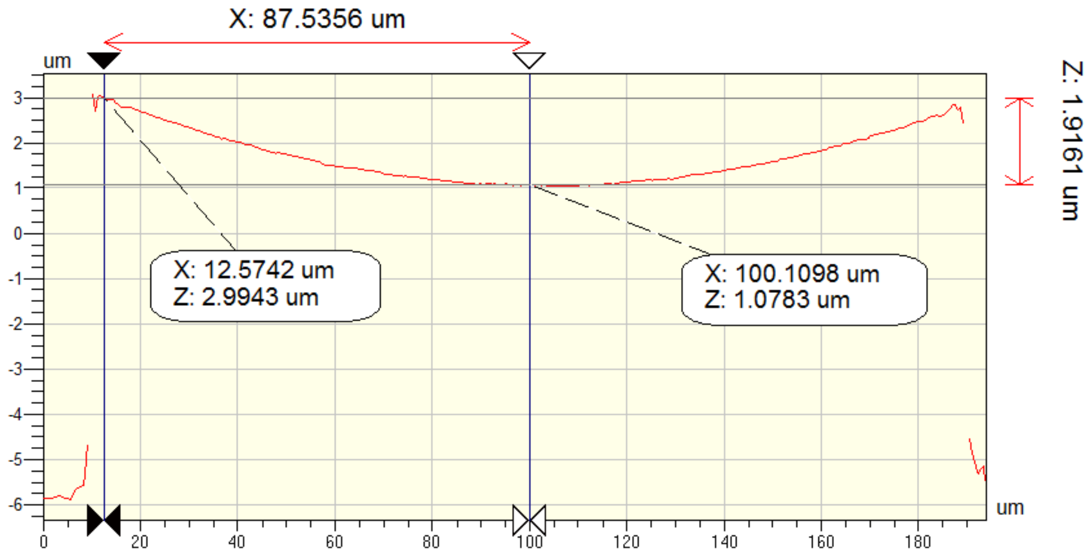
SiC:N Electrode		TiN Electrode	
SiC 2,000 nm	-80 MPa	SiC 2,000 nm	-80 MPa
SiC:N 300 nm	30 MPa	Ti/TiN 20/25 nm	-210 MPa
PETEOS 50 nm	80 MPa	AlN 750 nm	670 MPa
AlN 750 nm	40 MPa		
SiC:N 300 nm	-280 MPa		
Average Stress	-60 MPa	Average Stress	120 MPa

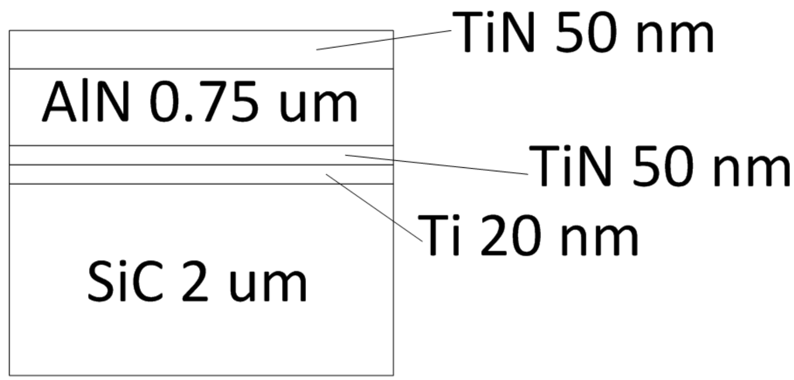
*Cheng, D., "System for measuring the curvature of a semiconductor wafer," US 5227641 (July 13 1993).

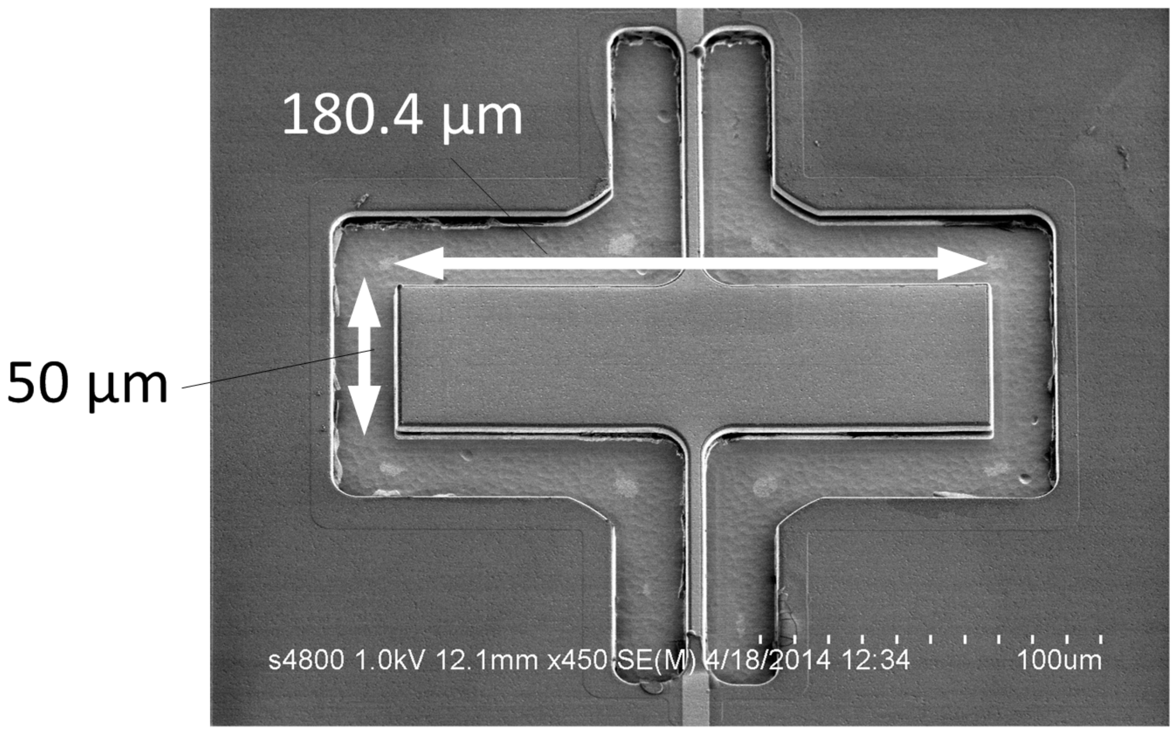
Length Extensional Static Deflection





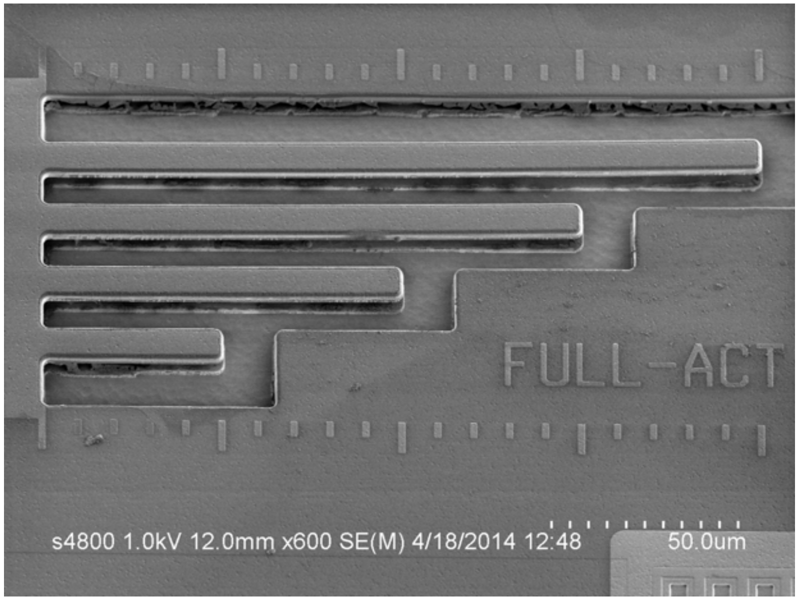


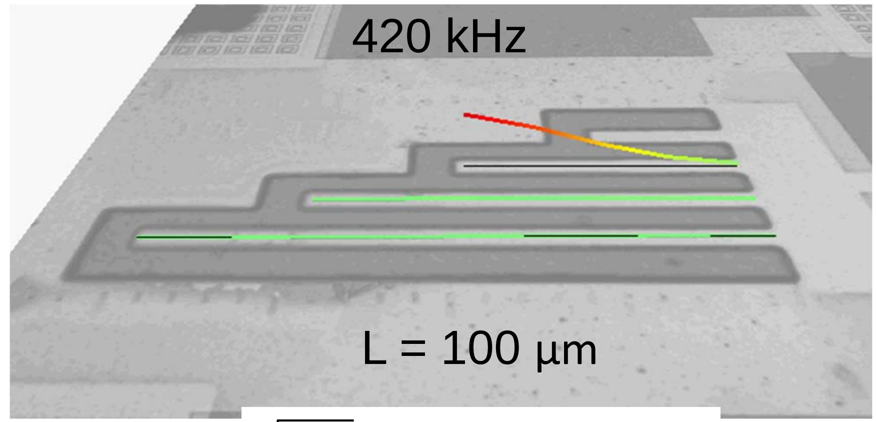

TiN 50 nm
AlN 0.75 um
TiN 50 nm
Ti 20 nm
SiC 2 um


180.4 µm
50 µm
s4800 1.0kV 12.1mm x450 SE(M) 4/18/2014 12:34
100um

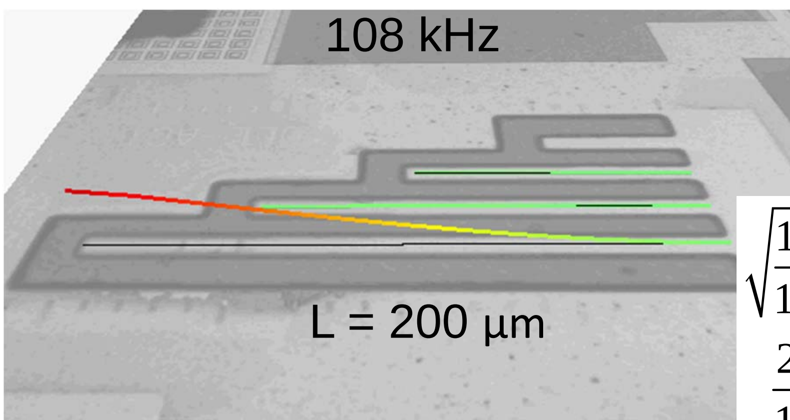
Cantilevers

$$f_{res} \propto \frac{1}{L^2}$$



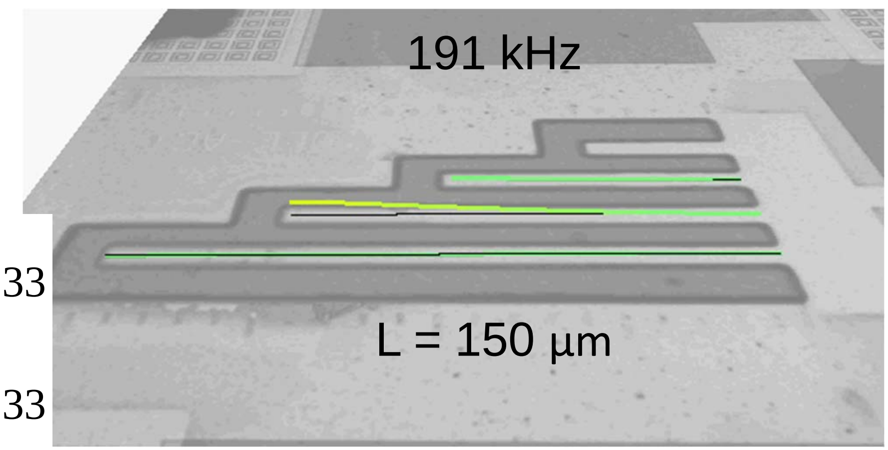


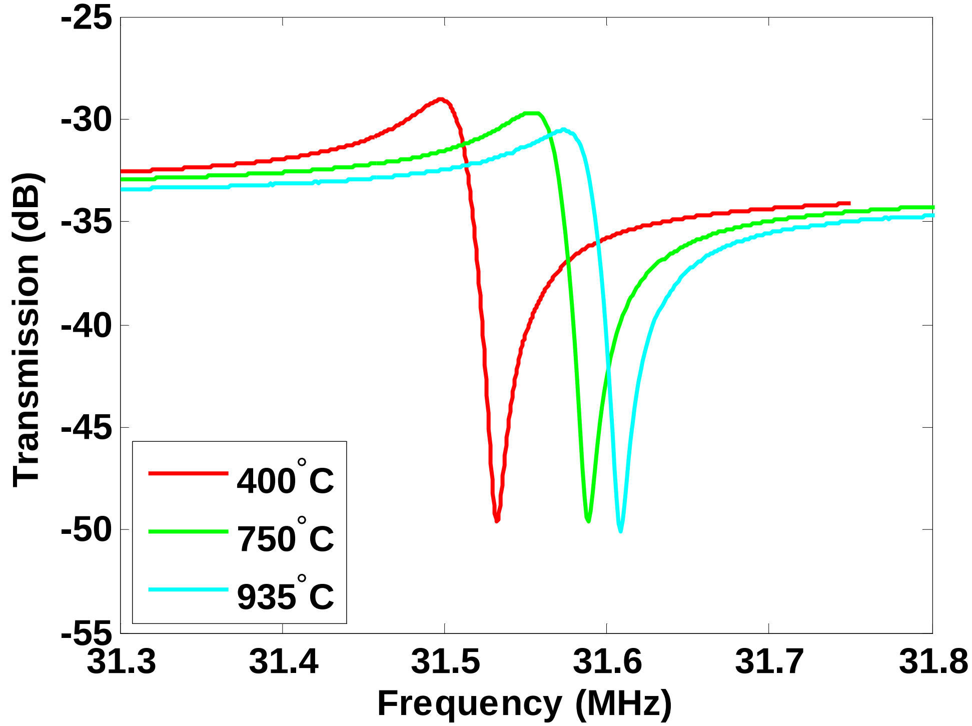
$$\sqrt{\frac{420}{191}} = 1.48 \qquad \frac{150}{100} = 1.50$$



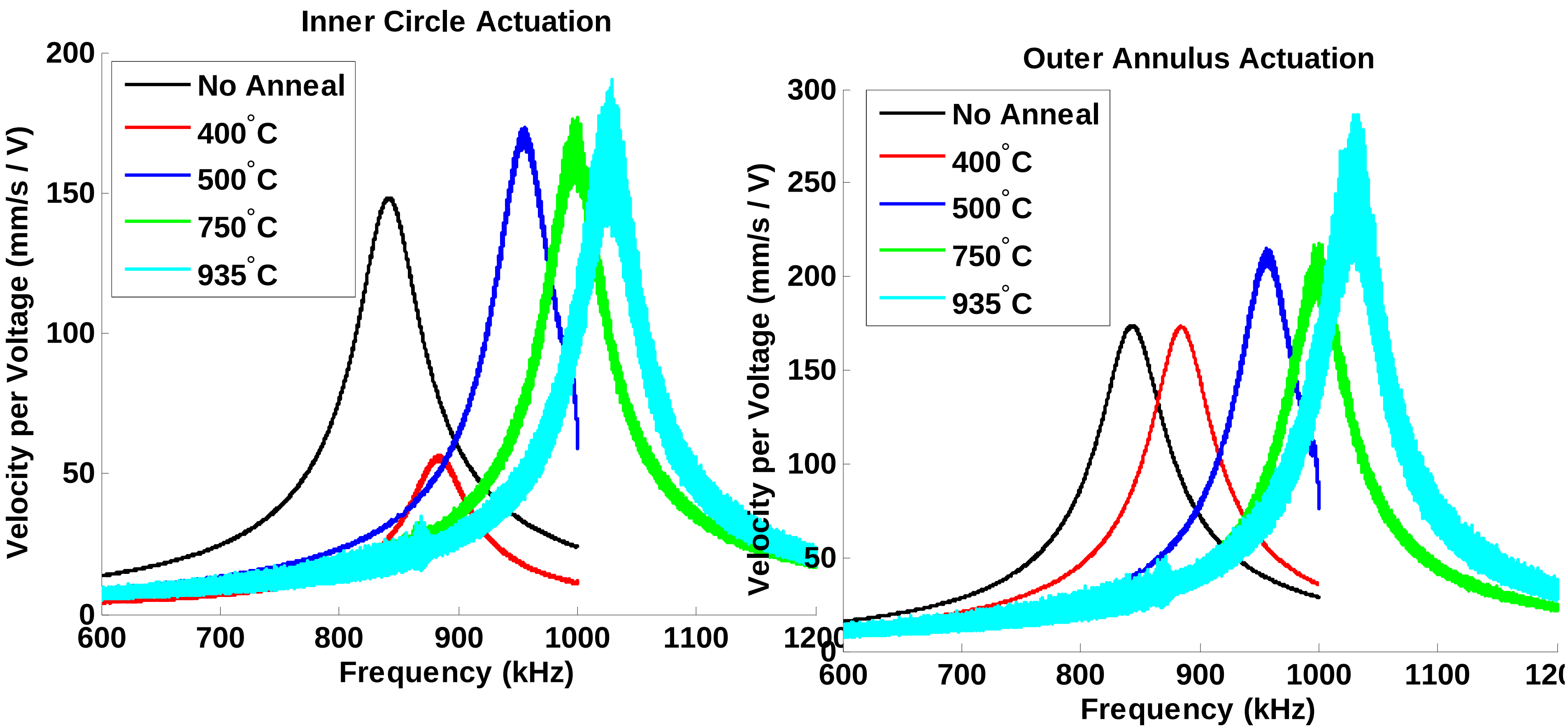
$$\sqrt{\frac{191}{108}} = 1.33$$

$$\frac{200}{150} = 1.33$$

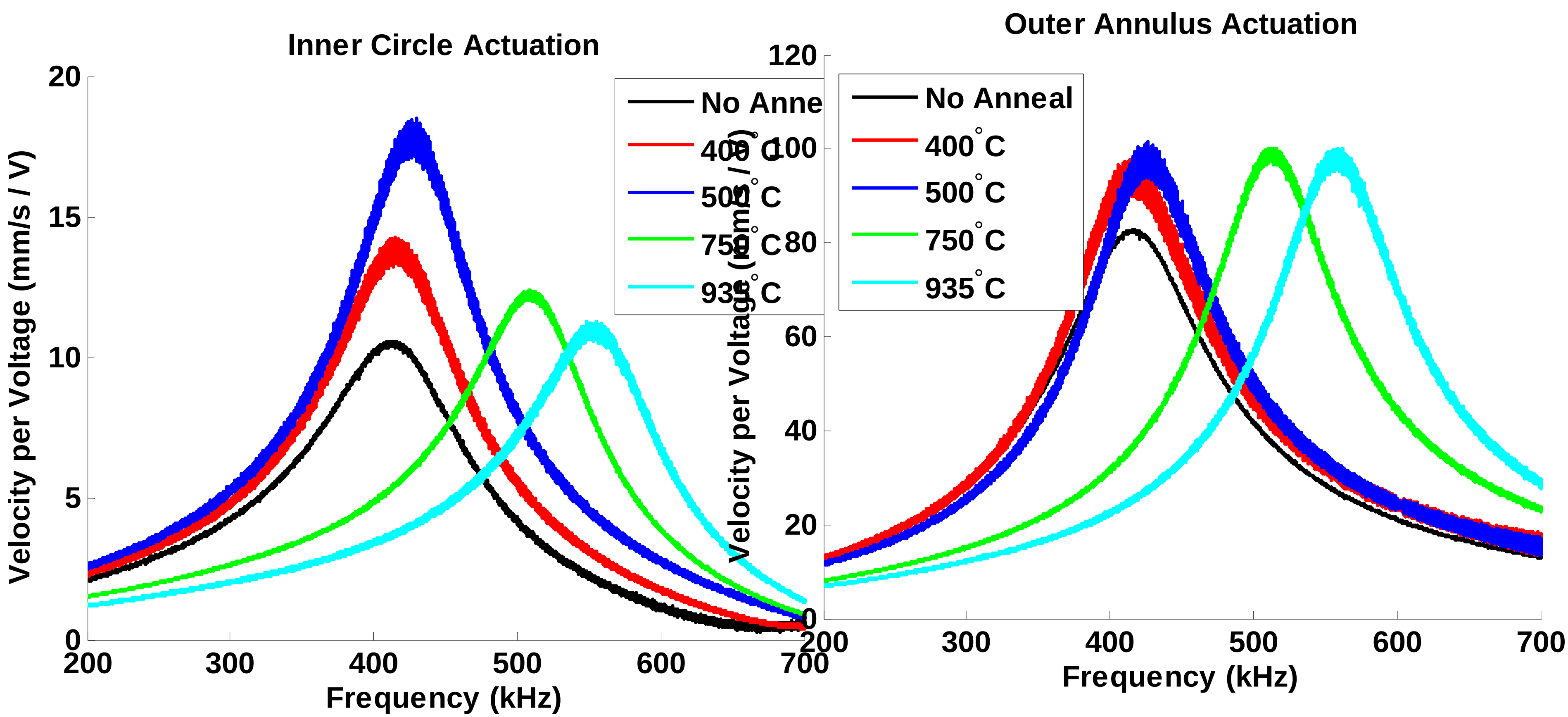



-25
-30
-35
-40
-45
-50
-55
31.3
31.4
31.5
31.6
31.7
31.8
Frequency (MHz)
Transmission (dB)
400°C
750°C
935°C

RO 100 um, RI 40 um

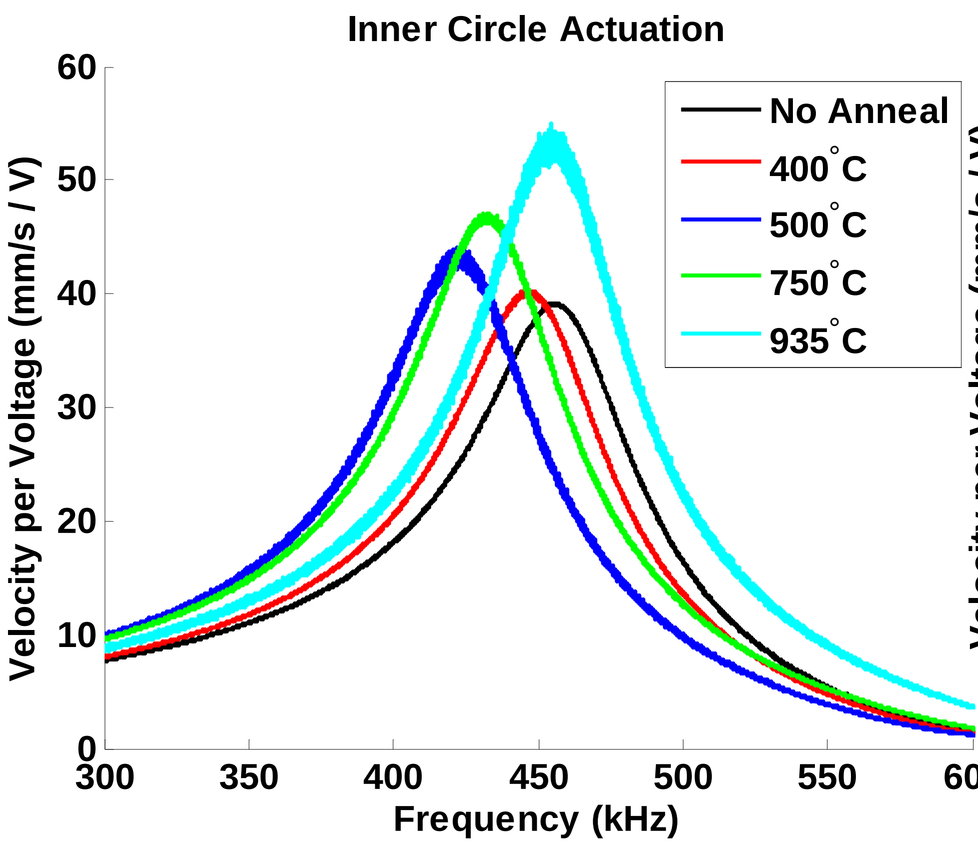


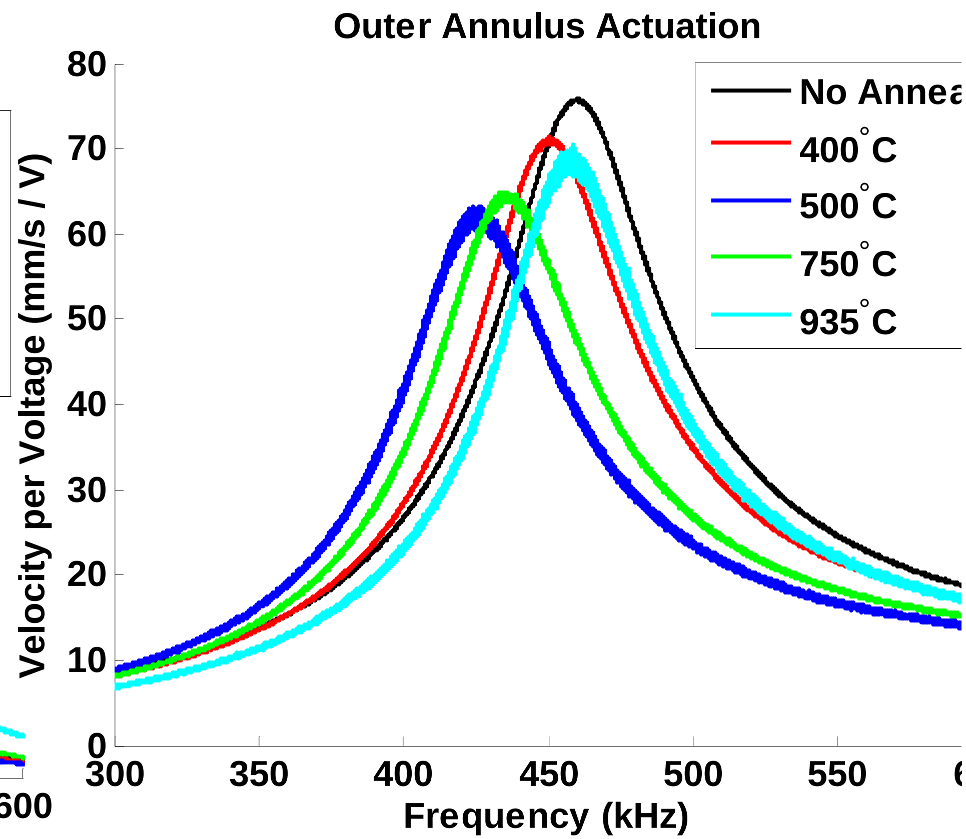
R0150, RI60



Inner Circle Actuation
Velocity per Voltage (mm/s / V)
Frequency (kHz)
No Anne
400°C
500°C
750°C
935°C
Outer Annulus Actuation
Velocity per Voltage (mm/s / V)
Frequency (kHz)
No Anneal
400°C
500°C
750°C
935°C

RI80, RO200


Inner Circle Actuation
Velocity per Voltage (mm/s / V)
Frequency (kHz)
No Anneal
400°C
500°C
750°C
935°C
0
10
20
30
40
50
60
300
350
400
450
500
550
600

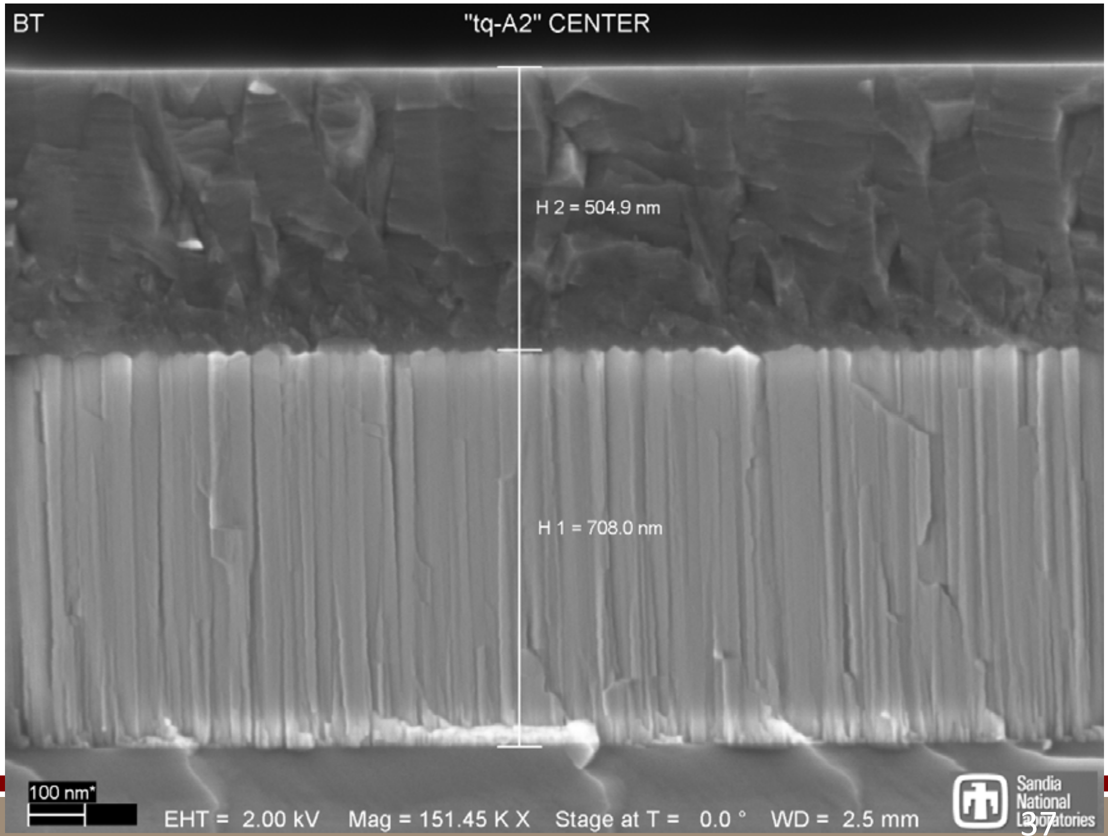

Outer Annulus Actuation
Velocity per Voltage (mm/s / V)
Frequency (kHz)
400°C
500°C
750°C
935°C
0
10
20
30
40
50
60
70
80
300
350
400
450
500
550

Research Team

- Benjamin Griffin (ECLDRD PI)
  - Research interests are focused on MEMS transducers with a specialty in acoustics, piezoelectrics, and high temperature applications
- Scott Habermehl (ECLDRD mentor)
  - 20 years of experience in chemical vapor deposition and thin film process technology and integration for rad-hard CMOS and MEMS
  - Developed low temperature techniques for deposition of silicon carbide
- Peggy Clews (fabrication integration engineer)
  - 22 years experience in fabrication and technology integration of MEMS

SiC on AlN

- Integration of doped SiC on AlN with chemical mechanical polish
  - AlN deposited at 350˚C
  - SiC deposited afterward at 950˚C



Facilities and Capabilities Used

- SiFab
  - 11,900 square foot Class 1 cleanroom in the SiFab for silicon wafer processing
  - Fabrication process up to release
- MicroFab
  - 14,900 square foot Class 10 and Class 100 cleanroom in the MicroFab for III-V compound semiconductor material processing and silicon wafer post-processing
  - Device release
- 858EL Light Labs
  - Polytec MSA 400 Scanning Laser Vibrometer
  - Wyko Scanning White-Light Interferometer
  - Cascade Microtech Microchamber with Semiconductor Parameter Analyzer

Stress Extraction Via Wafer Bow

- Stoney's Formula
  - ROC is determined by curve fitting to a wafer measurement referenced to the pre-deposition measurement
  - Film thickness depends upon the deposition step

$$\sigma = \frac{1}{R} \frac{E}{6(1-\nu)} \frac{T^2}{t}$$

$$R = \text{ROC}$$

$$E = 130 \text{ GPa}$$

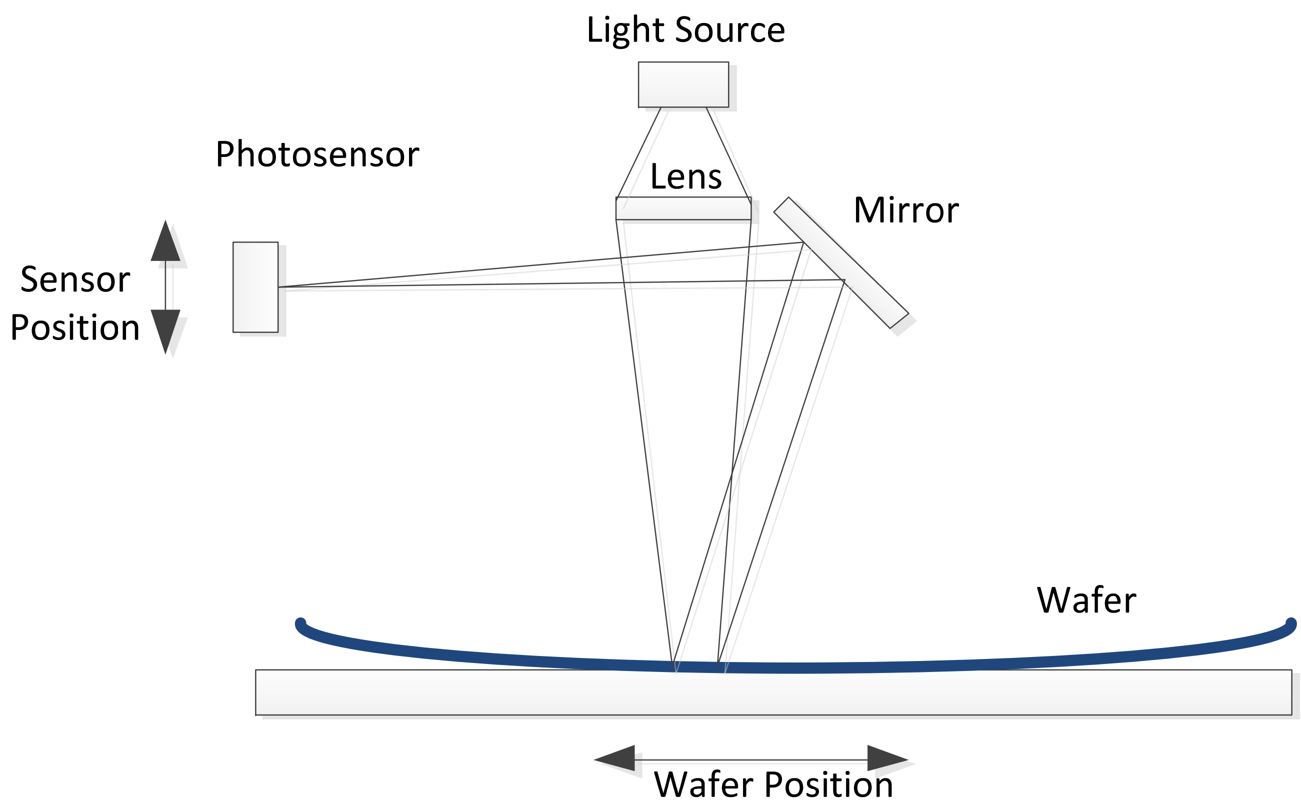
$$\nu = 0.28$$

$$T = 675\ \mu\text{m}$$

$$t = \text{film thickness}$$

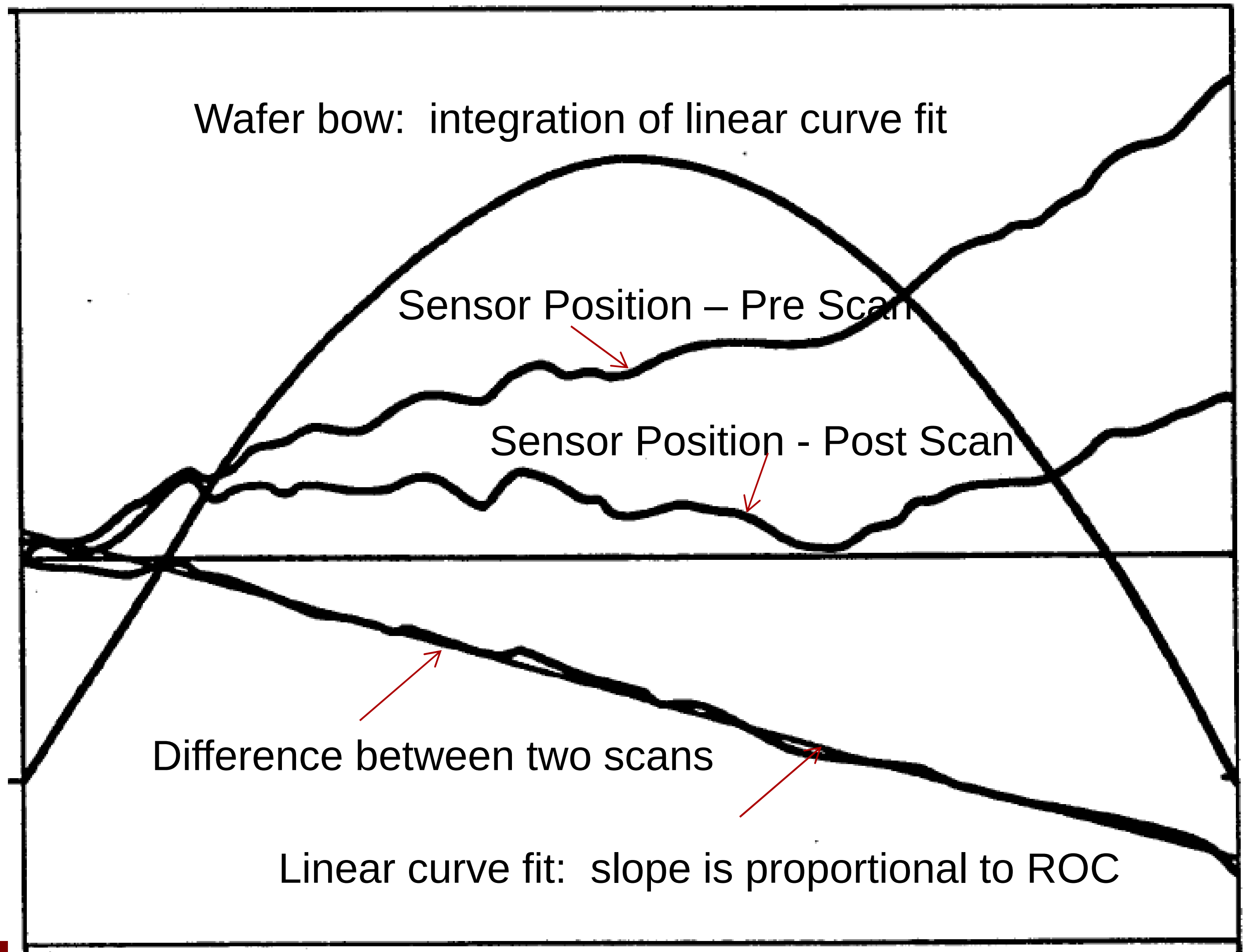
FSM Stress Measurement

- Optical Lever Sensor
  - Sensor position ~ local wafer angle

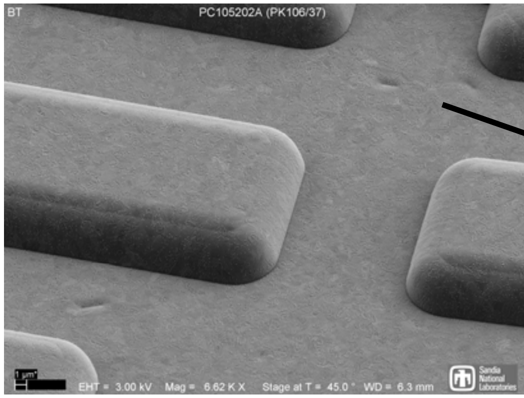


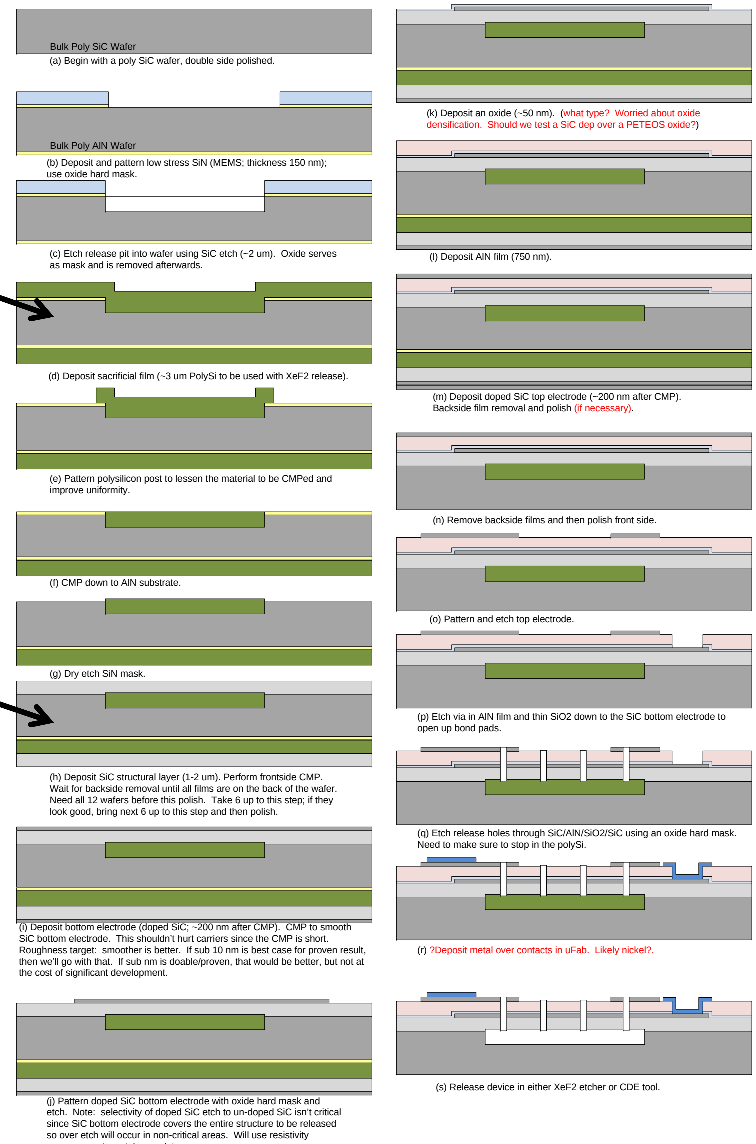
Cheng D, inventor; Frontier Semiconductor Measurement, Inc., assignee. System for measuring the curvature of a semiconductor wafer. United State 1993.

Example Data



Fabrication in Process





$$\tau = \frac{K\lambda}{\beta \cos\theta}$$

- where:
- τ is the mean size of the ordered (crystalline) domains, which may be smaller or equal to the grain size;
- K is a dimensionless **shape factor**, with a value close to unity. The shape factor has a typical value of about 0.9, but varies with the actual shape of the crystallite;
- λ is the X-ray wavelength;
- β is the line broadening at half the maximum intensity (FWHM), after subtracting the instrumental line broadening, in radians. This quantity is also sometimes denoted as $\Delta(2\vartheta)$;
- ϑ is the Bragg angle.