



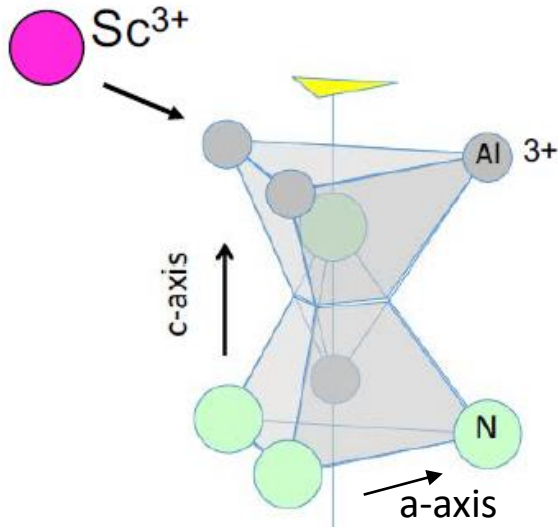
# **Etching (and Other Important Aspects) of AlScN Materials**

**Assistant Professor Troy Olsson**

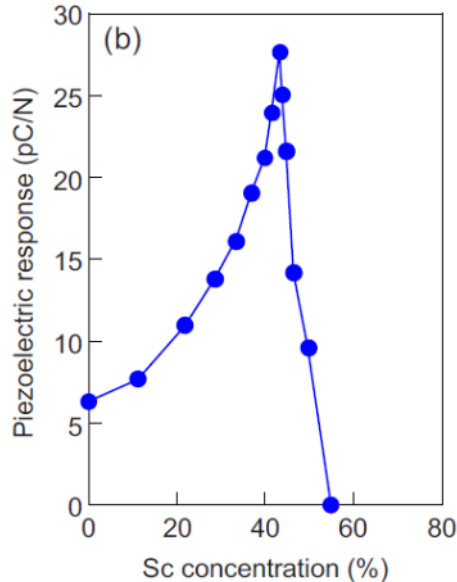
**Department of Electrical and Systems Engineering**

**University of Pennsylvania**

# Why Aluminum Scandium Nitride?



P. Murali, "Doped AlN: Materials and devices,"  
Tutorial IFCS-EFTF 2019.



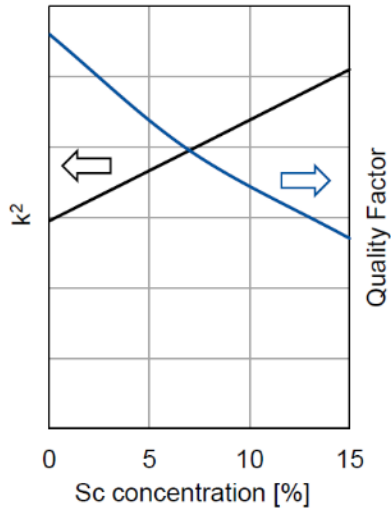
M. Akiyama, K. Kano and A. Teshigahara, "Influence of growth temperature and scandium concentration on piezoelectric response of scandium aluminum nitride alloy thin films," Appl. Phys. Lett. 95, 162107, 2009.

- AlScN

- Alters the c/a ratio of the AlN unit cell
- Large increase in the piezoelectric coefficients
- Potential to substantially widen the bandwidth of BAW filters

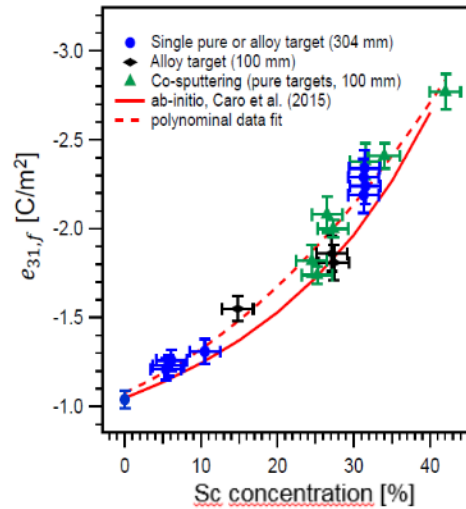
# Figure of Merit for Different Piezoelectric Applications

B. Heinz, "The growth of piezoelectric Al(1-x)ScxN thin films on Si (100) and Pt (111) using co-sputtering and compound target sputtering," IEEE NEMS, April 2018.



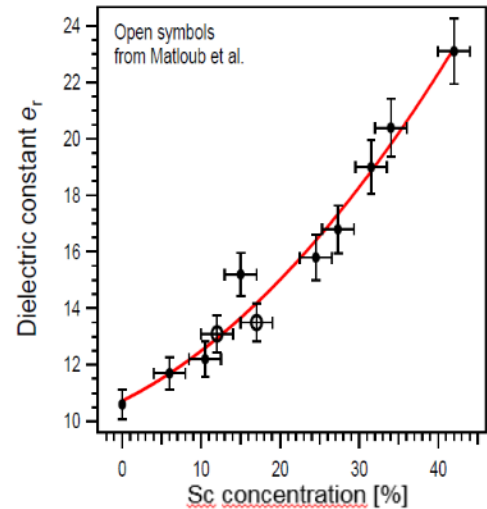
FOM for RF resonator applications

$$\frac{d_{33}^2 E}{\epsilon_0 \epsilon_r} \times Q$$



Signal to noise FOM for MEMS sensors

$$\frac{e_{31,f}}{\sqrt{\epsilon_0 \epsilon_r \cdot \tan(\delta)}}$$

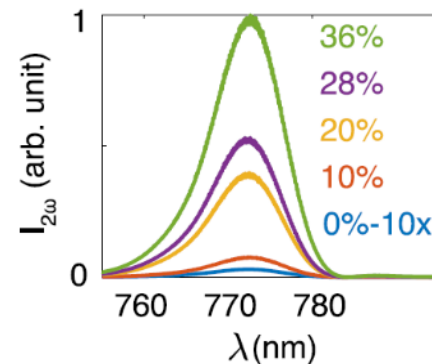
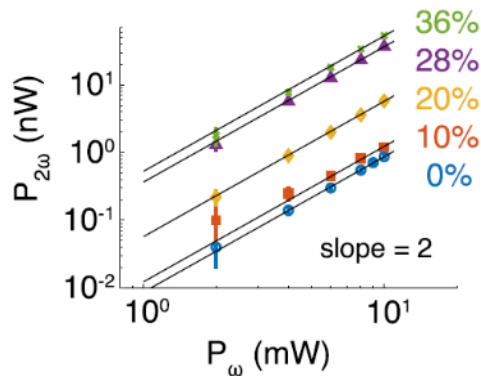


Energy FOM

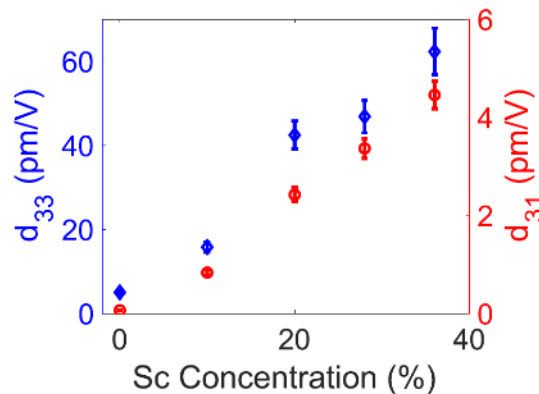
$$\frac{e_{31,f}^2}{(\epsilon_0 \epsilon_r)}$$

# AlScN Nonlinear Optics (Collaboration with Bo Zhen)

- A strongly nonlinear optical material that can be grown directly on Si
- Ferroelectric properties allowing for periodic poling
- Nonlinear optical coefficients larger than  $\text{LiNbO}_3$
- Next step is to investigate lower optical loss formulations



2<sup>nd</sup> Harmonic Generation in AlScN for different Sc alloying



Measured nonlinear optical coefficients

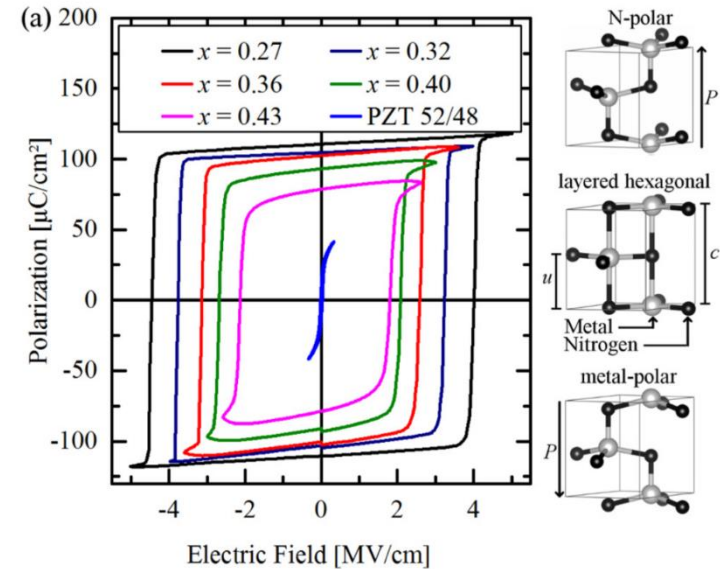
V. Yoshioka, J. Lu, Z. Tang, J. Jin, R. H. Olsson III, and B. Zhen, "Strongly Enhanced Second-Order Optical Nonlinearity in CMOS-Compatible  $\text{Al}_{1-x}\text{Sc}_x\text{N}$  Thin Films," *APL Materials*, 9, 101104, October 2021.

# Ferroelectric Properties of AlScN

[1] Phys. Status Solidi A 2020, 217, 1900840

Material	$P_r$ ( $\mu\text{C}/\text{cm}^2$ )	$E_c$ ( $\text{MV}/\text{cm}$ )	$T_{\text{MAX}}$ ( $^\circ\text{C}$ )	CMOS BEOL
AlScN	80-130	2-6.5	350	Yes
HfZrO <sub>2</sub> [1]	5-24	1.3	400	Yes
PZT	20-40	0.05	> 600	No

- Polar and ferroelectric as deposited w/ minimal wakeup
- Only two available polarization states
- Sharp ferroelectric switching
- Large remanent polarization and coercive field
- Low temperature deposition but stable polarization to extremely high temperatures (1100 °C)
- Initial reported results for films ~500 nm requiring >100V to switch



S. Fichtner, N. Wolff, F. Lofink, L. Kienle, and B. Wagner, "AlScN: A III-V semiconductor based ferroelectric", *J. Appl. Phys.* 125, 114103 (2019)

# Memory Comparison and Motivation

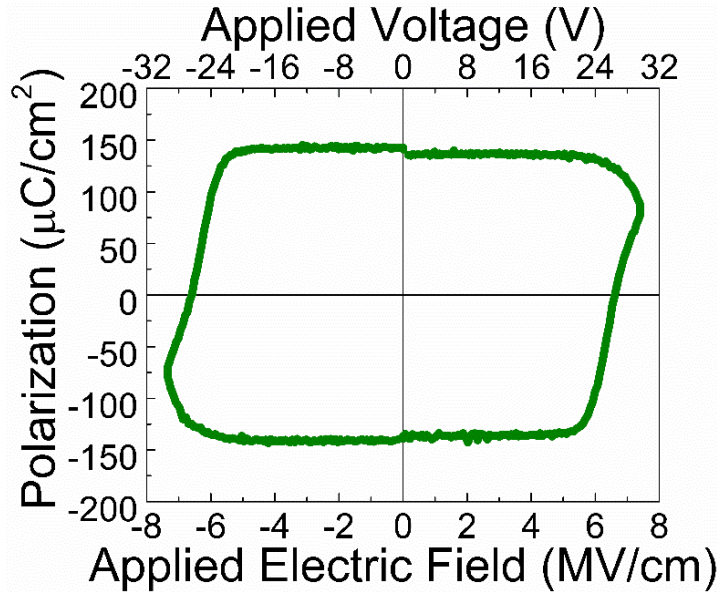
H. Cai et al., "High performance MRAM with spin-transfer-torque and voltage-controlled magnetic anisotropy effects," *Appl. Sci.*, 7, 929 (2017)

Memory	SRAM	DRAM	NOR-Flash	NAND-flash	STT-MRAM	PCRAM	ReRAM	FeRAM <sup>1</sup>
Cell area	>100 F <sup>2</sup>	6 F <sup>2</sup>	10 F <sup>2</sup>	4 F <sup>2</sup> (3D)	6~50 F <sup>2</sup>	4~30 F <sup>2</sup>	4~12 F <sup>2</sup>	15~35 F <sup>2</sup>
Multi bit	1	1	2	3	1	2	2	1
Supply	<1 V	<1 V	>10 V	>10 V	<1.5 V	<3 V	<3 V	<1.8 V
Read duration	~1 ns	~10 ns	~50 ns	~10 μs	<10 ns	<10 ns	<10 ns	<10 ns
Write latency	~1 ns	~10 ns	10 μs–1 ms	100 μs–1 ms	<10 ns	~50 ns	<10 ns	<5 ns
Retention	N/A	~64 ms	>10 y	>10 y	>10 y	>10 y	>10 y	>10 y
Endurance	>10 <sup>16</sup>	>10 <sup>16</sup>	>10 <sup>5</sup>	>10 <sup>4</sup>	>10 <sup>15</sup>	>10 <sup>9</sup>	10 <sup>6</sup> ~10 <sup>12</sup>	10 <sup>13</sup>
Write energy	~fJ/bit	~10 fJ/bit	~100 pJ/bit	~10 fJ/bit	~0.1pJ/bit	~10 pJ/bit	~0.1 pJ/bit	~10 fJ/bit

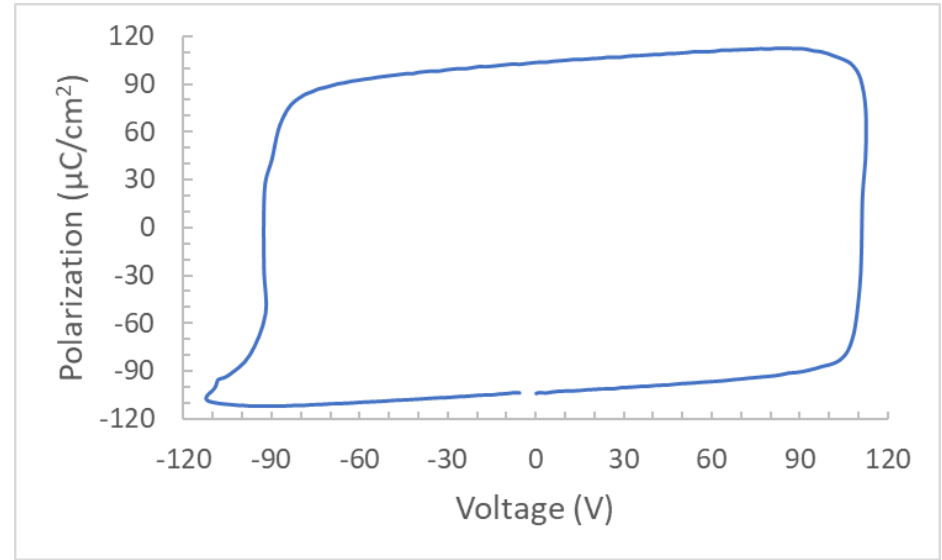
<sup>1</sup> SRAM, Static Random-Access Memory; DRAM, Dynamic Random-access memory; STT-MRAM, Spin Transfer Torque Magnetoresistive random-access memory; PCRAM, Phase-change Random-access memory; ReRAM, Resistive Random-access memory; FeRAM, Ferroelectric Random-access memory.

- Ferroelectric NVM offers the advantages of low write energy/current and fast read/write speed
- The overwhelming weakness of FE NVM is cell area or bit density
- We seek to exploit the FE properties of AlScN and its compatibility with CMOS to scale the bit density of FE NVM
- To achieve CMOS compatible write voltages will require AlScN thicknesses on order of 10 nm

# Ferroelectric AlScN Materials



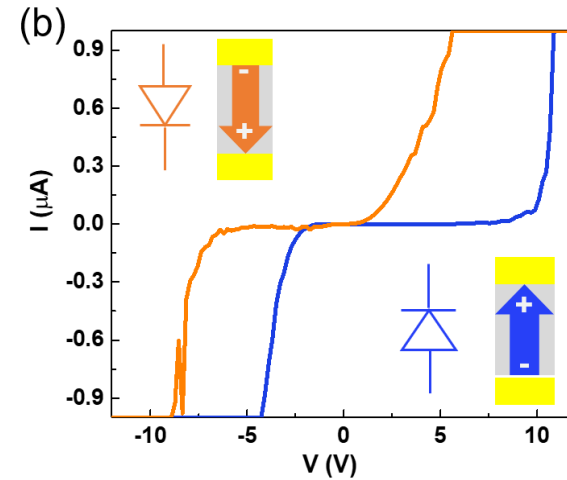
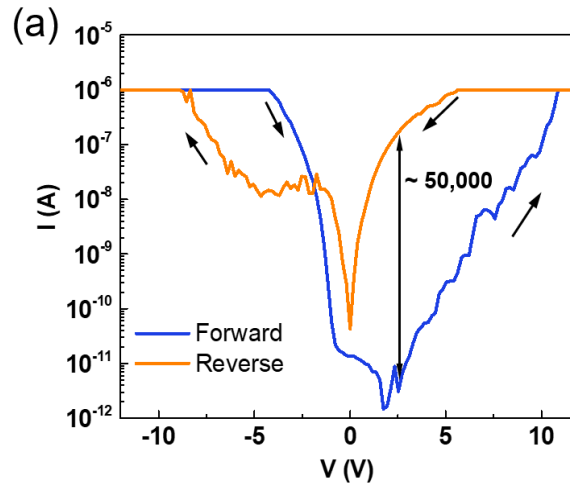
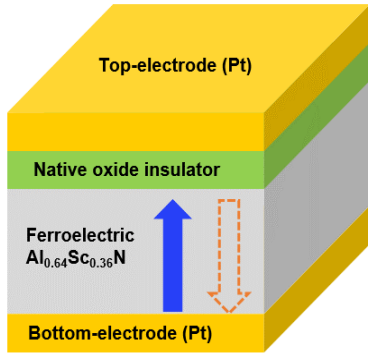
Ferroelectric P-E Loop Measured in a 20 nm Thick  $\text{Al}_{68}\text{Sc}_{32}\text{N}$  film



Ferroelectric P-E Loops Measured in a 200 nm Thick  $\text{Al}_{68}\text{Sc}_{32}\text{N}$  film

D. Wang, J. Zheng, P. S. M. Gharavi, W. Zhu, A. Foucher, S. E. Trolrier-McKinstry, E. A. Stach, and R. H. Olsson III, "Ferroelectric Switching in Sub-20 nm Aluminum Scandium Nitride Thin Films," *IEEE Electron Device Letters*, vol. 41, no. 12, pp. 1774-1777, Dec. 2020.

# AlScN Ferroelectric Diodes (Collaboration with Deep Jariwala)



I-V response of ferro-diode realized in 20 nm thick AlScN

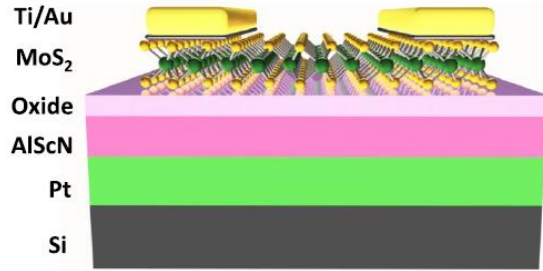
- Ratio of resistance between two different memory states is very large  $> 10^4$
- Inverting diode polarity like behavior observed in forward vs reverse sweeps
- Can be Set/Reset with 1 microamp current

X. Liu et. al, "Aluminum Scandium Nitride based Metal-Ferroelectric-Metal Diode Memory Devices with High On/Off Ratios," *Applied Physics Letters*, 118, 202901 (2021)

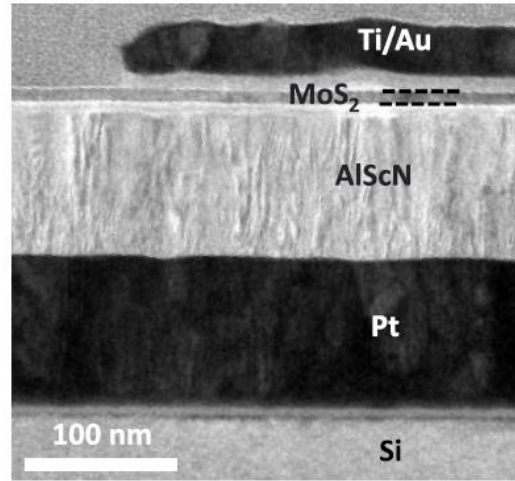


# AlScN/MOS<sub>2</sub> Ferroelectric FETs (Collaboration with Deep Jariwala)

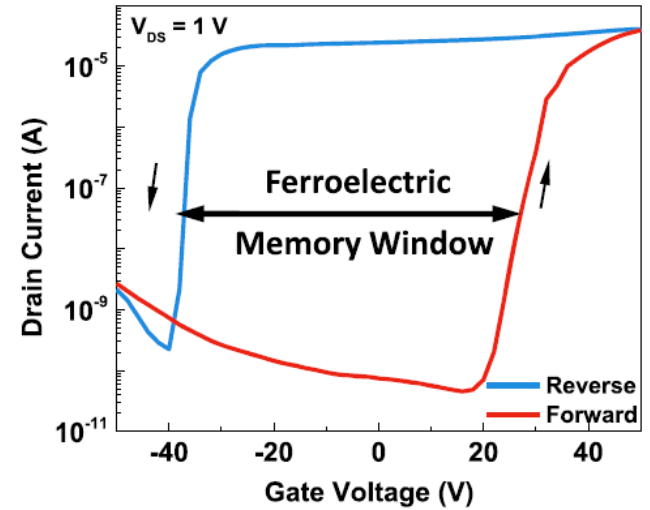
MoS<sub>2</sub>/AlScN Ferroelectric Field-Effect Transistor



FeFET device structure



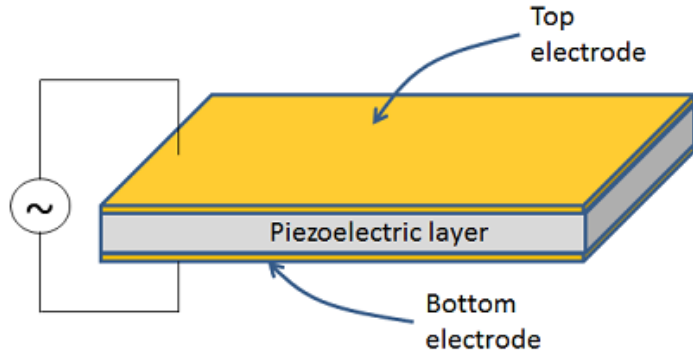
FeFET TEM image



FeFET  $I_{DS}-V_{GS}$  hysteresis loop

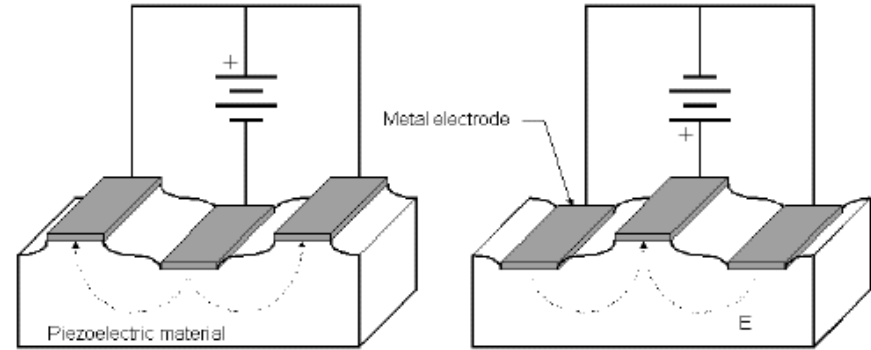
# Piezoelectric Resonators and Filters

## Bulk Acoustic Wave



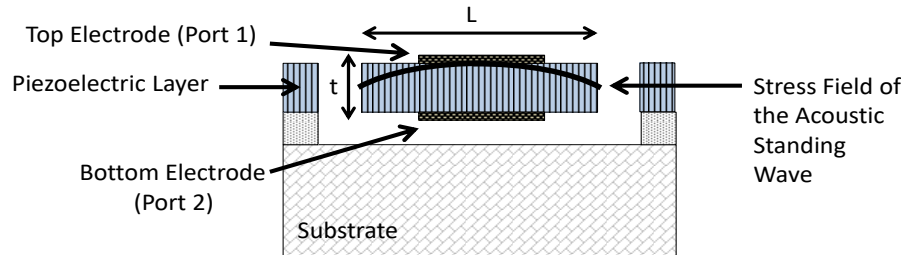
Source: C. Cassella, Course notes from ECEE7398 at NEU

## Surface Acoustic Wave

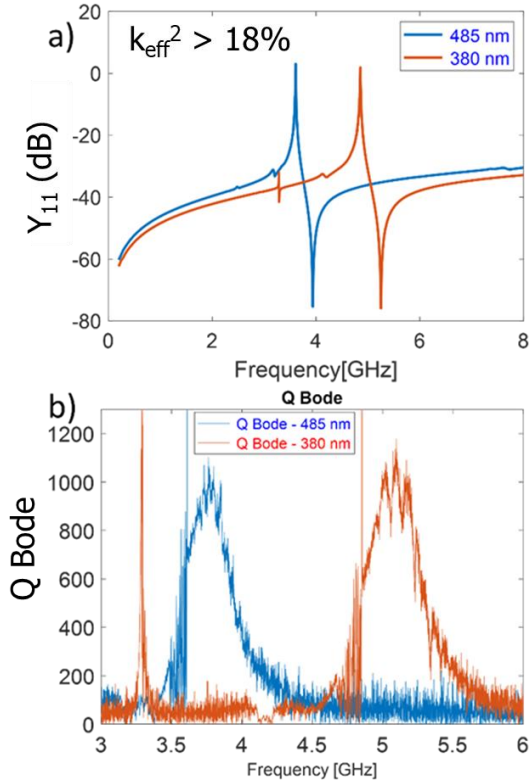


Source: D. Malocha , Intro to Surface Acoustic Wave Technology

## Lamb Wave/Contour Mode

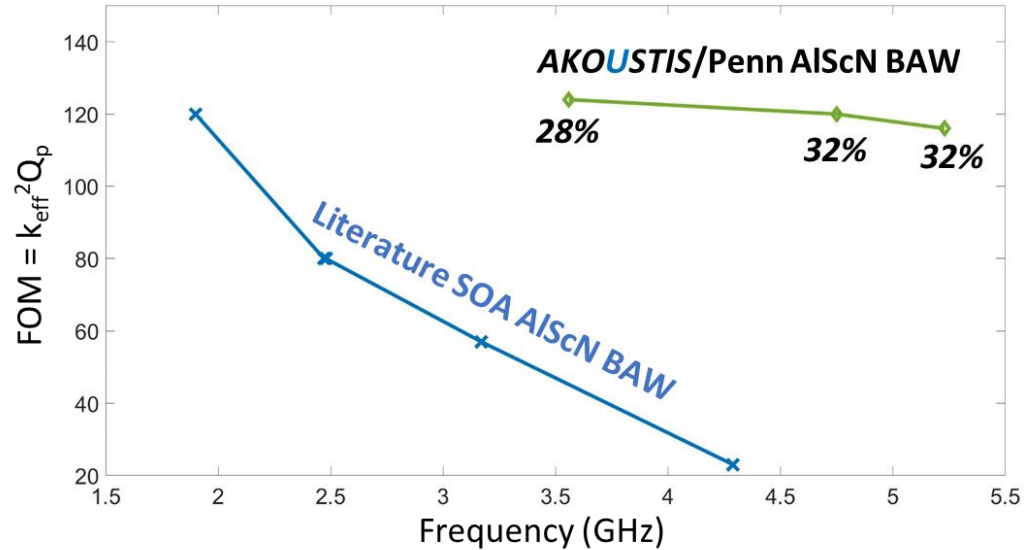


# AlScN Bulk Acoustic Wave Resonators



Al<sub>68</sub>Sc<sub>32</sub>N BAW Resonator Measurements

- Akoustis BAW resonators constructed from Penn AlScN materials exhibit SOA performancee at high frequency

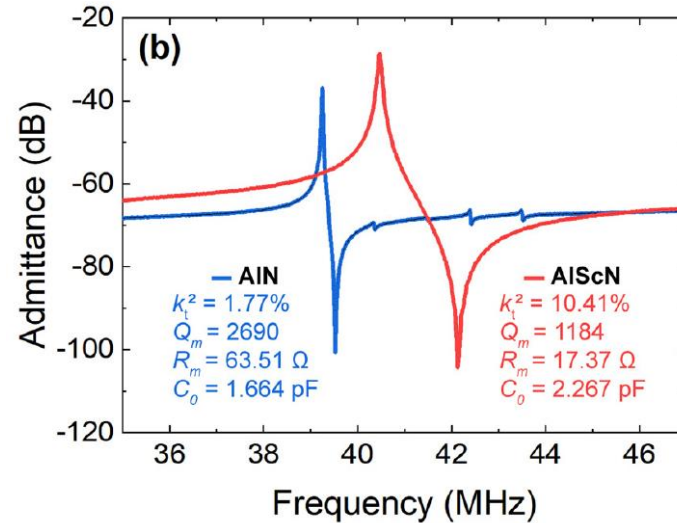
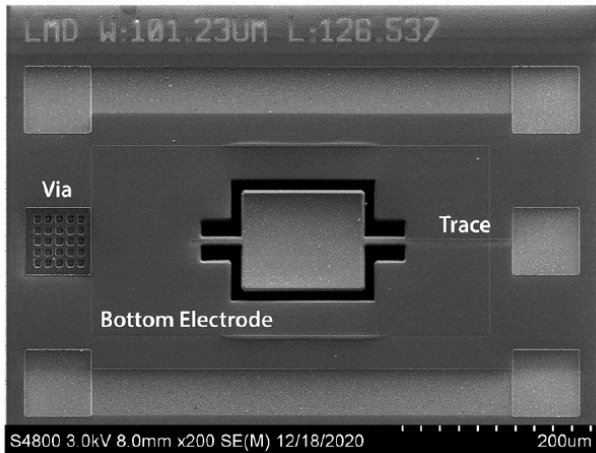


AlScN BAW Resonator Comparison

# High Performance AlScN Piezoelectric MEMS Resonators

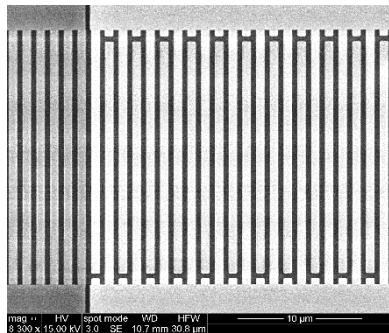
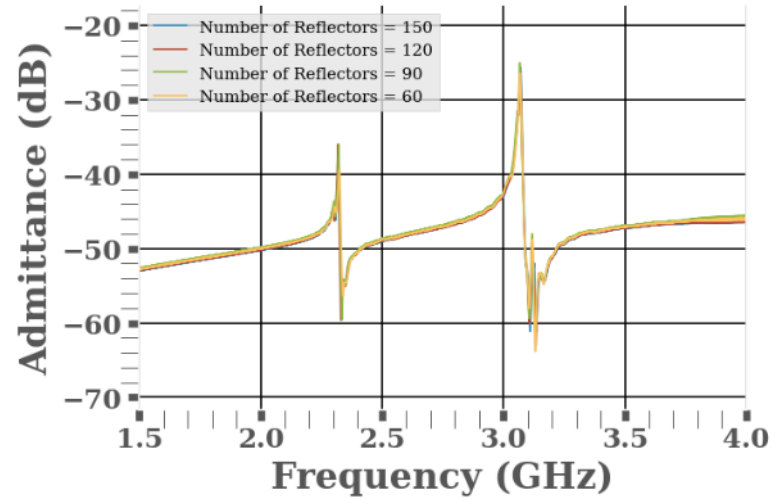
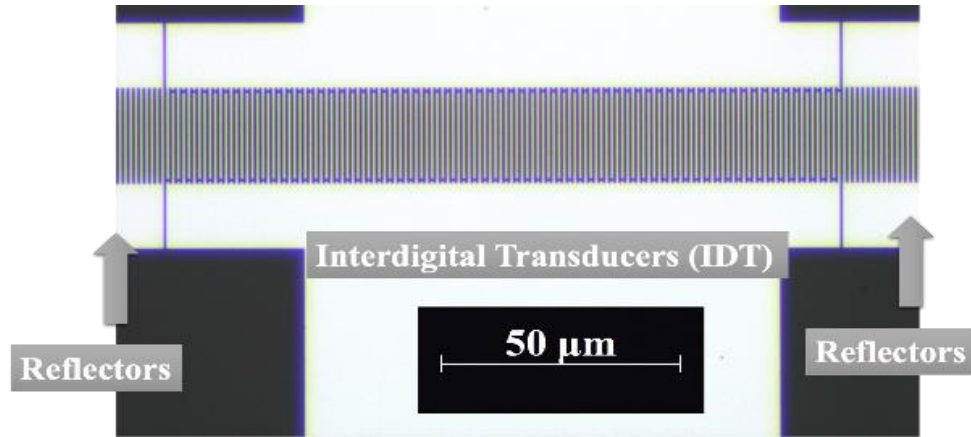
## Collaboration with Sandia

Fabricated Device



G. Esteves, T. R. Young, Z. Tang, S. Yen, T. M. Bauer, M. D. Henry, and R. H. Olsson III "Al<sub>0.68</sub>Sc<sub>0.32</sub>N Lamb Wave Resonators with Electromechanical Coupling Coefficients near 10.28%," *Applied Physics Letters*, 118 (17), 171902, April 2021.

# AlScN Surface Acoustic Wave Devices



- AlScN SAW does not require etching of the piezo material
- $k_t^2 = 5.8\%$
- Q is 600

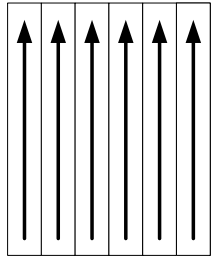
X. Du, Z. Tang, C. Leblanc, D. Jariwala and R. H. Olsson III, "High-Performance SAW Resonators at 3 GHz Using AlScN on a 4H-SiC Substrate," *IEEE Frequency Control Symposium*, Accepted

# Device Performance, Applications, and Etching Requirements

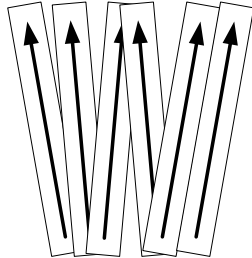
Device Type	Applications	Frequency Range	$k_t^2$	Q	Etching Requirements
BAW	<ul style="list-style-type: none"> <li>RF filters</li> <li>Oscillators</li> </ul>	2-6 GHz	23%	600 - 1100	<ul style="list-style-type: none"> <li>Thickness of 300 – 1000 nm</li> <li>Does not require vertical sidewall</li> <li>Stop on bottom electrode material</li> </ul>
MEMS	<ul style="list-style-type: none"> <li>Oscillators</li> <li>Sensors</li> <li>Energy harvesting</li> <li>RF and IF filters</li> </ul>	kHz to GHz	10%	1000 - 1500	<ul style="list-style-type: none"> <li>Thickness of 300 – 2000 nm</li> <li>Requires vertical sidewall at high frequency</li> <li>Stop on bottom electrode material</li> </ul>
SAW	<ul style="list-style-type: none"> <li>RF filters</li> </ul>	1 – 5 GHz	5.8%	600	<ul style="list-style-type: none"> <li>No etching</li> </ul>
Ferroelectric	<ul style="list-style-type: none"> <li>Memory</li> <li>Nonlinear photonics</li> <li>RF devices</li> </ul>	-	-	-	<ul style="list-style-type: none"> <li>Thickness 5 – 500 nm</li> <li>Memory requires 100 nm diameter features in thin (~5 nm) films</li> <li>Photonics requires well controlled, smooth sidewall, vertical preferred</li> </ul>

# Critical Metrics for AlScN Materials

## Dipole Alignment



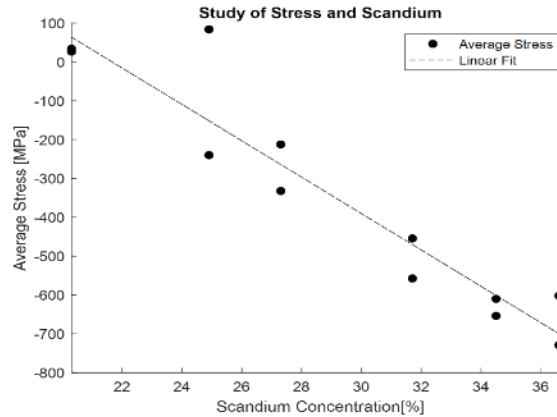
Ideal



Reality

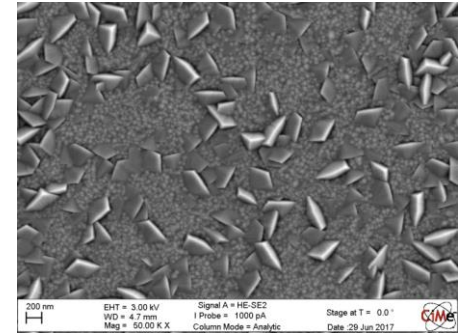
XRD FWHM is measure of film quality

## Stress



Higher Sc alloying AlScN exhibits large compressive stress

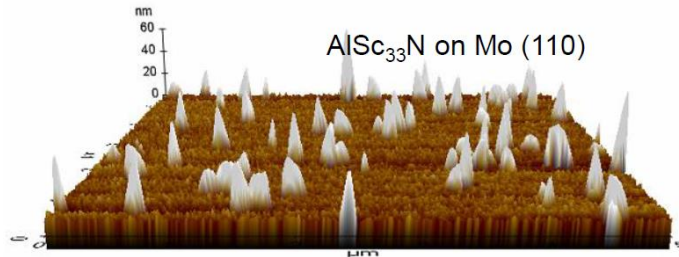
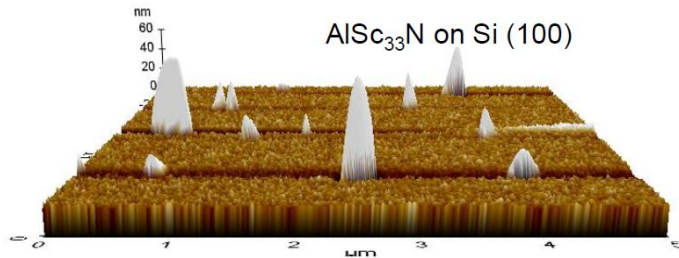
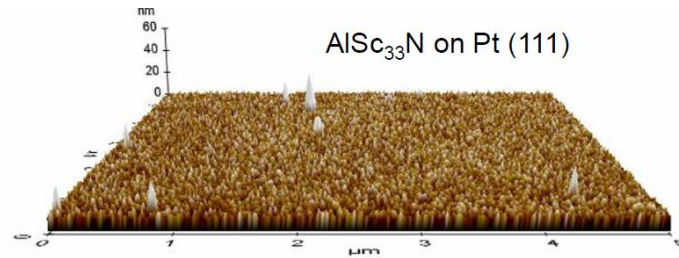
## Anomally Oriented Grain (AOG) Growth



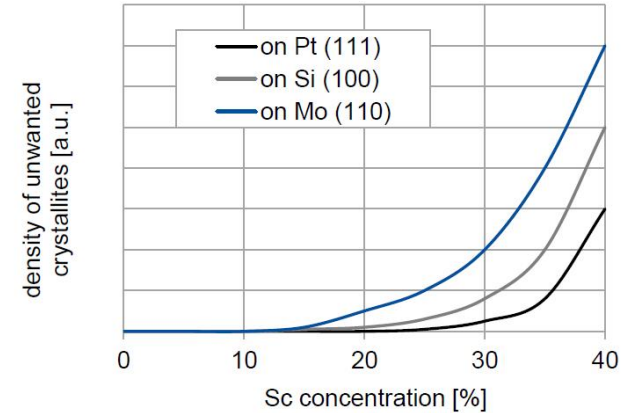
Low AOG density is key for high  $k_{\text{eff}}^2$ , low roughness, and high Q

P. Muralt, "Doped AlN: Materials and Devices," IFCS Tutorial 2019

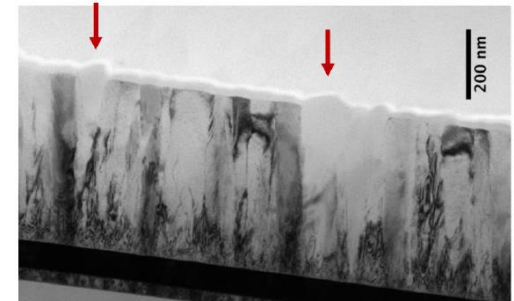
# Secondary Grain Growth in AlScN



B. Heinz, "The growth of piezoelectric Al(1-x)Sc<sub>x</sub>N thin films on Si (100) and Pt (111) using co-sputtering and compound target sputtering," IEEE NEMS, April 2018.

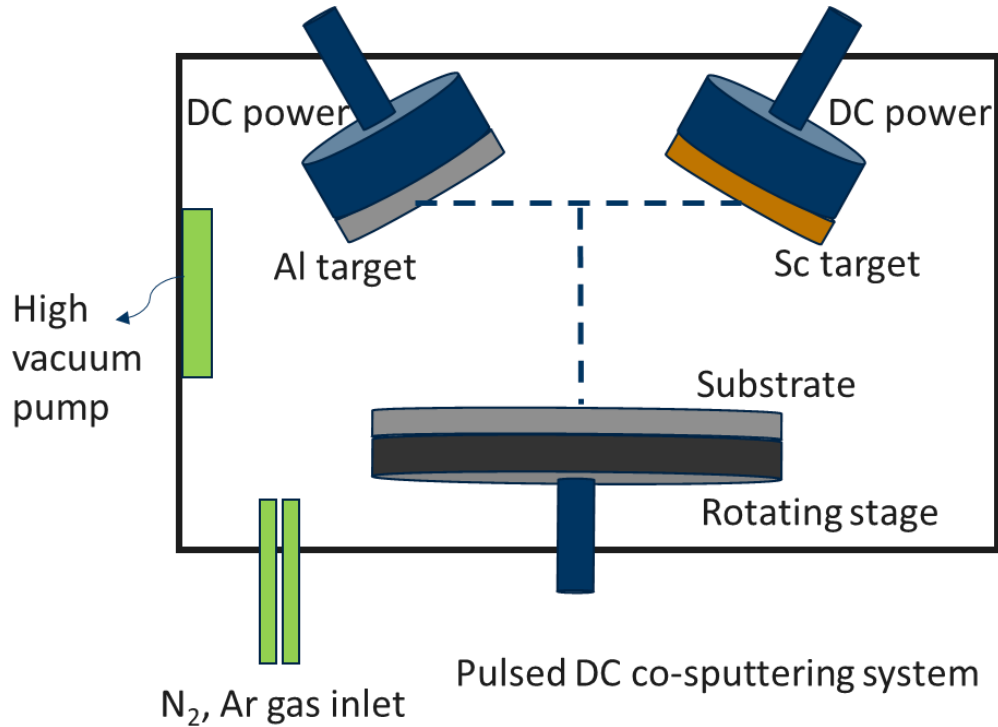


Bright field TEM picture of unwanted crystallites





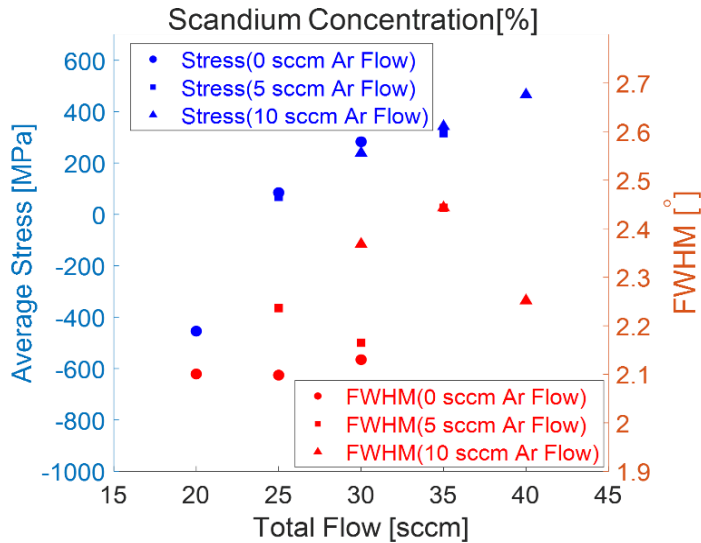
# AlScN Deposition Methods



## Ferroelectric AlScN Growth Method

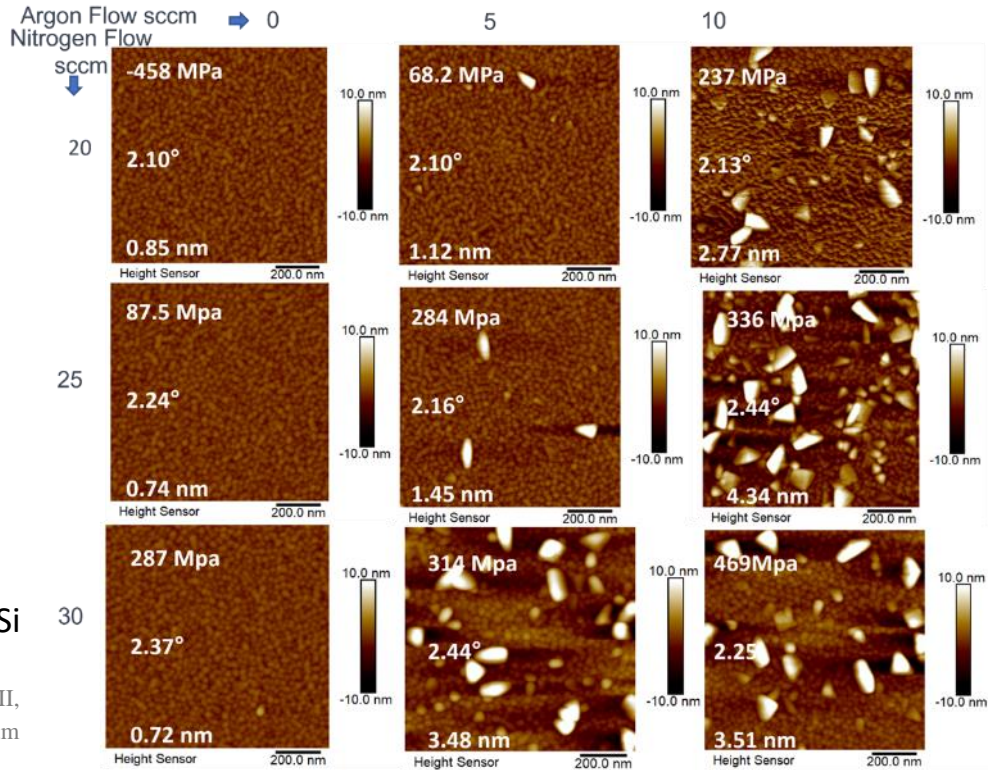
- Co-Sputtering
  - Films from 0-43% Sc
  - Deposition rate  $\sim 10$  nm/min
  - Substrates up to 150 mm

# AOG Suppression with Stress Control



Stress and crystal orientation of  $\text{Al}_{68}\text{Sc}_{32}\text{N}$  deposited on Si

R. Beaucejour, V. Roebisch, A. Kochhar, C. Moe, D. Hodge and R. H. Olsson III, "Controlling Residual Stress and Suppression of Anomalous Grains in Aluminum Scandium Nitride Films Grown Directly on Silicon," *IEEE JMEMS*, Accepted



AOG growth and stress for  $\text{Al}_{68}\text{Sc}_{32}\text{N}$  deposited on Si

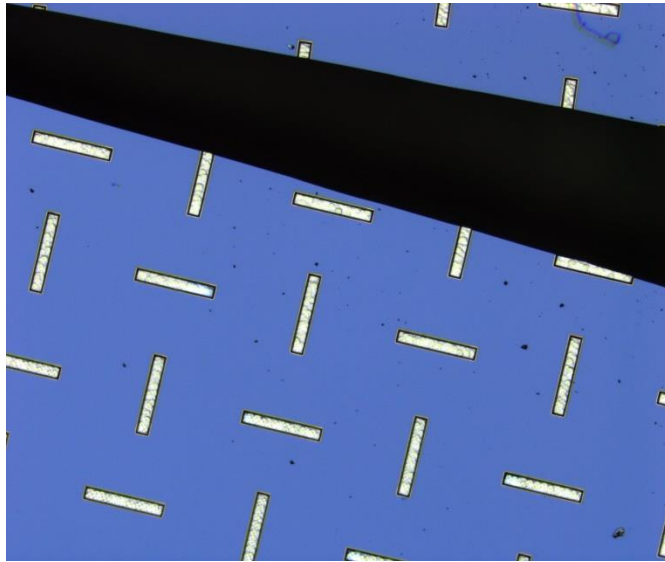
# AlScN Etching Techniques

Etch Type	Etch Chemistry	Materials with High Selectivity	Notes
Dry	Cl/BCl <sub>3</sub> , Ar Ion Mill	None	<ul style="list-style-type: none"><li>• Sidewall of 70 – 75 deg. demonstrated</li><li>• Etch rate highly dependent on Sc concentration</li><li>• Max. thickness of ~ 1 μm for high Sc doped materials</li><li>• Requires 2-5x hard mask thickness</li></ul>
Wet	Hot H <sub>3</sub> PO <sub>4</sub>	Si, SiO <sub>2</sub>	<ul style="list-style-type: none"><li>• Excellent when stopping AlScN etch on Si</li></ul>
Wet	KOH	SiN, Pt, Mo, W, SiO <sub>2</sub>	<ul style="list-style-type: none"><li>• Excellent selectivity to SiN enables low thickness mask</li><li>• Stops on a variety of metals and dielectrics</li><li>• Etch rates and sidewall angles highly dependent on Sc concentration</li><li>• Can form angled etches to simplify interconnect formation</li></ul>

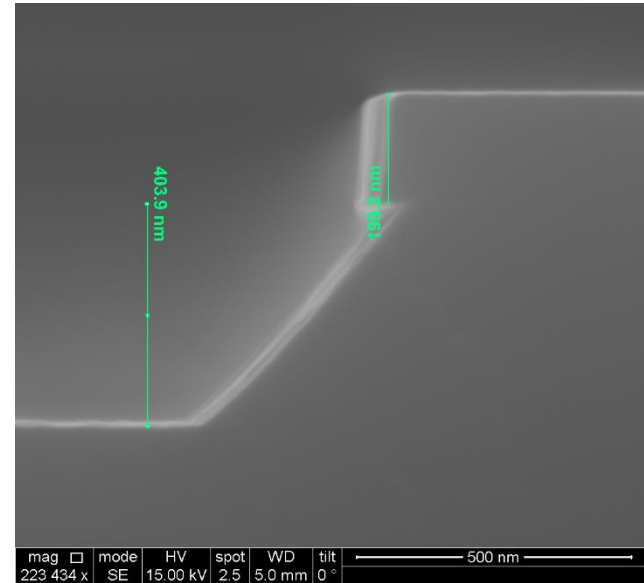
# AlScN KOH Etching Study

- $\text{Al}_{1-x}\text{Sc}_x\text{N}$  films were deposited on 4-inch wafers in the Evatec Clusterline 200 II tool with the range of  $x$  covering 0 to 40%;
- $\text{SiN}_x$  (200nm) film was PECVD deposited on top of the AlScN as a hard mask and on the backside of the wafer to protect the Si during the etch;
- SiN was patterned via dry etching using a  $\text{CHF}_3/\text{O}_2$  mixture;
- A waterbath was set up via the hotplate with temperature controlled by a feedback loop;
- 300mL KOH was poured into a beaker and then put inside the waterbath to reach a certain temperature;
- The sample was then submerged in the KOH, being etched for a given time, rinsed in DI water and blow dry by  $\text{N}_2$ ;
- The sample is finally cleaved in the middle and cross-section imaged by Quanta 600 ESEM.

# Images



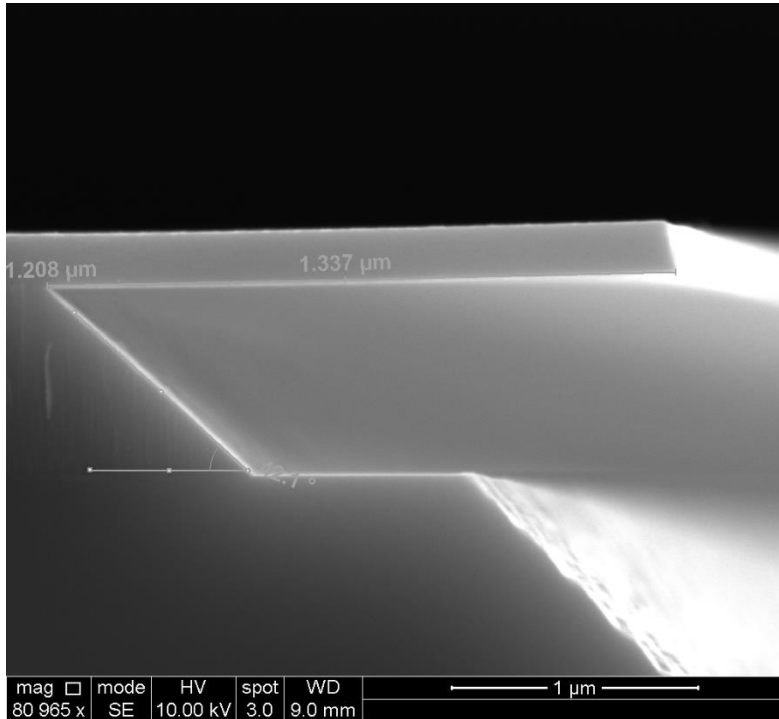
(a)



(b)

- Image showing (a) a cleaved sample after etching; (b) Cross-section SEM image of a sample etched for 30s in 30% KOH at 60°C.

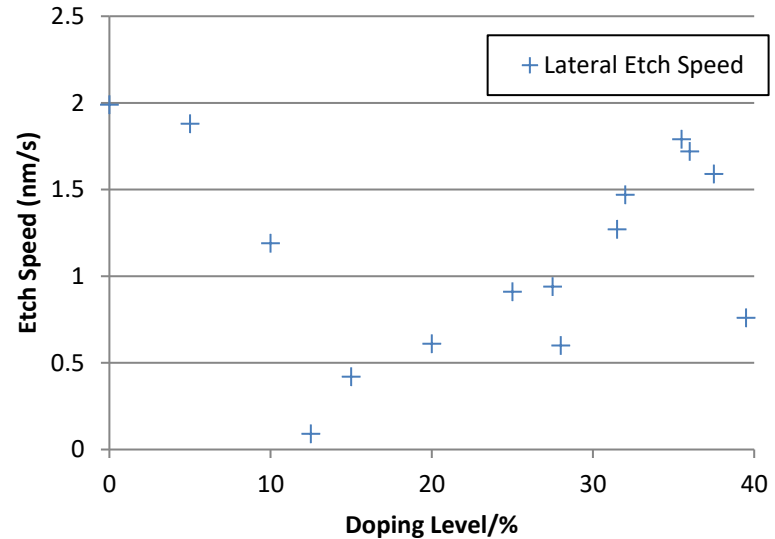
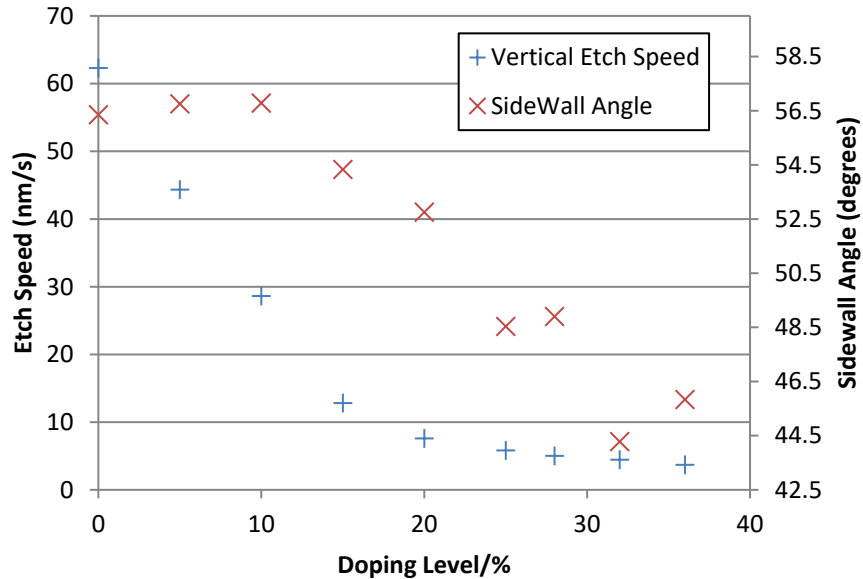
# Etch Rate Calculations



- Vertical etch rate is measured by dividing the measured etch depth by the etch time
- Lateral etch rate is measured by the undercut beneath the SiN<sub>x</sub> mask divided by the etch time
- Side wall angle is measured as shown, with 90° corresponding to a vertical sidewall

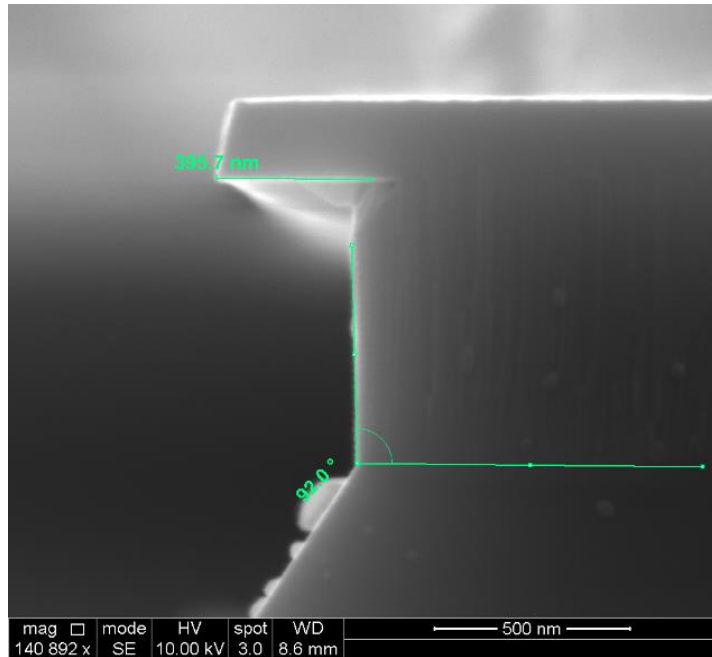
# Etch Rate Findings

Etch rates of AlScN vs. doping for 30% KOH @ 45 °C



- Etch rates vary substantially with Sc doping
- Etch rates vary with temperature and concentration but relative etch rates vs. doping are unchanged

# Controlling the Sidewall Profile



- By balancing the vertical/lateral etch rates and etching time vertical sidewalls can be formed for some Sc concentrations

Image showing a 12.5% AlScN film etched in 10% KOH at 65°C for 20 min. A vertical sidewall is achieved.



# Biomedical & IoT Integrated Communications and Sensing Laboratory (BIOTICS)

## Doctoral Students



Baha Bachnak



Rossiny Beaucejour



Michael D'Agati



Xingyu Du



Adzo Fiagbenu



Dr. Paria Gharavi



Yujia Huo



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Zichen Tang



Anne-Marie Zaccarin



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Dr. Shangyi Chen

## Masters Students



Kritank Kaylan

## Postdoctoral Scholars



Dr.  
Izhar

## Principle Investigator



Dr. Troy Olsson

# Acknowledgments

## Our Collaborators

- Jeff Shealy, Rama Vetury, Mary Winters, Craig Moe, Abhay Kochhar, and Dave Hodge of Akoustis
- Volker Roebisch, Martin Kratzer, and Bernd Heinz of Evatec
- Giovanni Esteves and David Henry of Sandia National Laboratories
- SRC liaisons at Intel and TSMC
- Labs of Prof. Deep Jariwala, Eric Stach, Mark Allen, Bo Zhen and Firooz Aflatouni

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