# Concept Demonstration of Dopant Selective Reactive Etching (DSRIE) in Silicon Carbide

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# Outline

- Motivation
- Technical Challenge
- Observed Phenomenon
- Technical Approach
- Results
- Innovation Impact on Mission if implemented
- Conclusion

# Problem Statement-Motivation

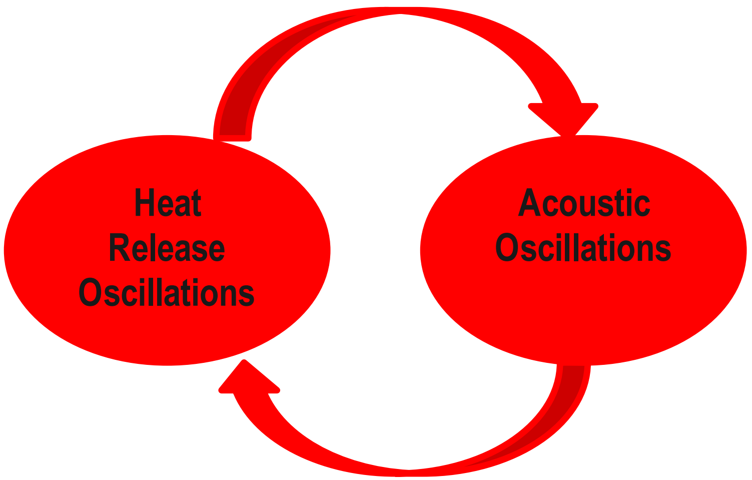
Lower emissions (LE) jet-engines are critically dependent on lean-burning (LB) operation (low fuel/air ratio).

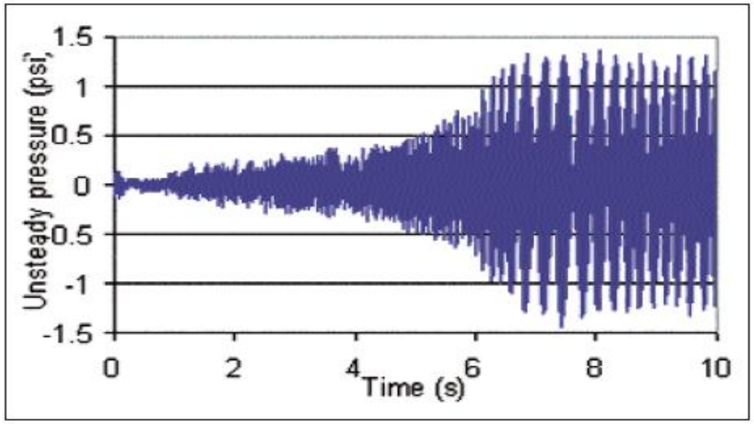
LB combustion is susceptible to thermo-acoustic instabilities, producing pressure oscillations that can reduce component life and potential engine failure.

Existing CFD instability prediction models have high uncertainties in high temperature regime.

SoA dynamic pressure sensors are placed distance away from test article-limits frequency bandwidth; Water-cooled sensors introduce “vortex noise” that corrupt signal.

Need high temperature (>500 C) pressure sensors to validate CFD models.

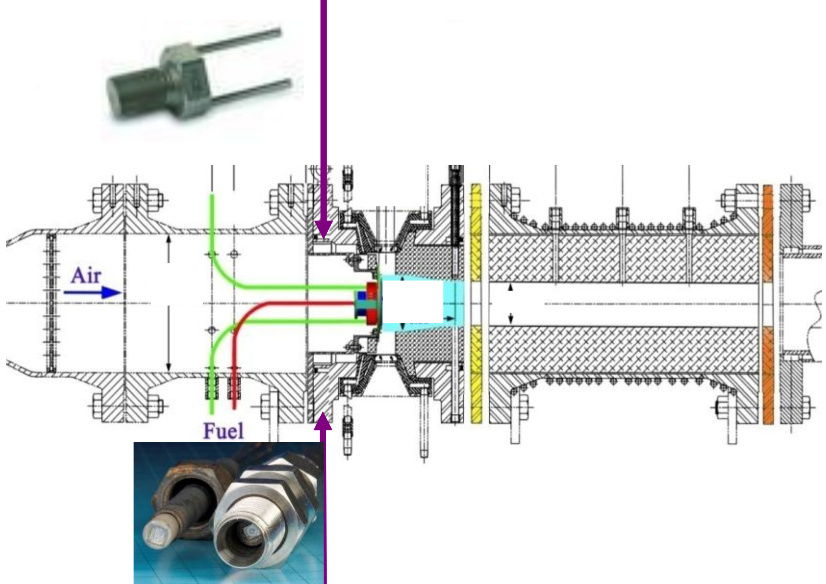


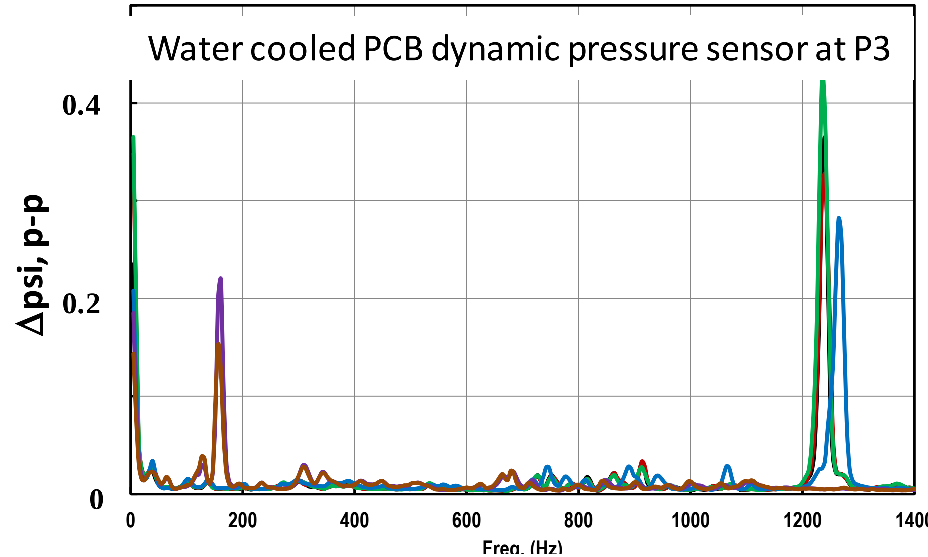
# Motivation (SoA dynamic pressure sensor versus SiC Static Sensor)

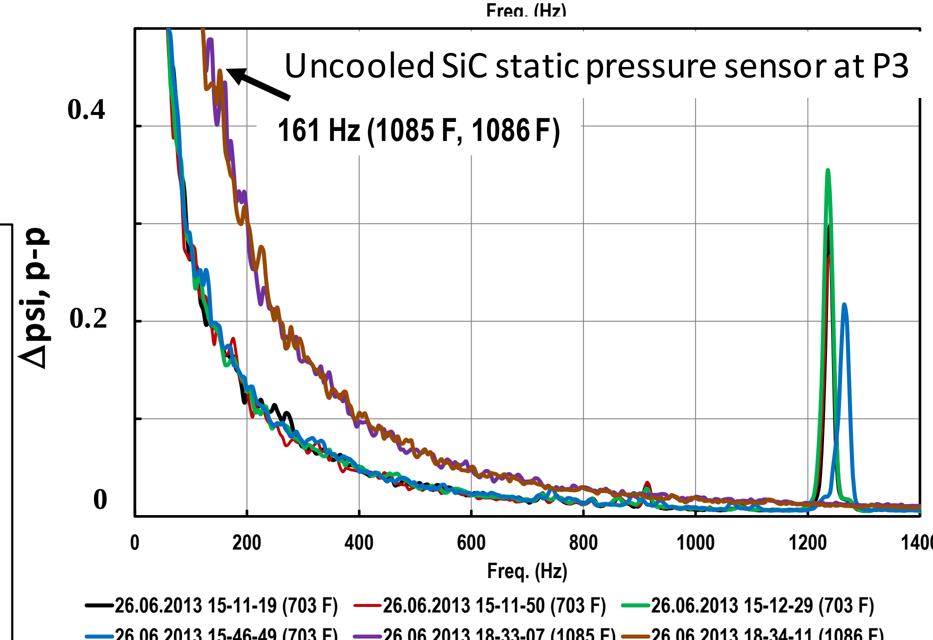
*Note: The thick diaphragm SiC sensor not designed for dynamic operation*

**PCB dynamic pressure transducers mounted on semi-infinite line**




**Flush mounted SiC dynamic pressure transducer**




Results:

- Reduced SiC data compared favorably well with the reduced PCB data.
- Low frequency noise (<500 Hz) suppressed the static SiC sensor output.

**Value Proposition**

**A real SiC dynamic pressure sensor will operate uncooled at high temperature, increase sensitivity, increase S/N ratio at lower frequencies**

# Technical Approach

## Goal: Realize ultrathin diaphragms in 4H-SiC

**Based on circular plate theory, maximum deflection (for deflections << thickness) of a clamped circular diaphragm is expressed as:**

$$d = \frac{3Pr^2}{16Eh^3}\left(1 - \upsilon^3\right)$$

**Where: d=maximum deflection (m); P=applied pressure (Pa); r=diaphragm radius (m); E=Young's Modulus (Pa); h=diaphragm thickness (m); $\upsilon$=Poisson ratio**

**E = 475 GPa for SiC; h (research goal) = 5 µm; r =1 mm; $\upsilon$ = 0.212**
**Calculated maximum stresses on diaphragm: Center=6.27 MPa; Clamped edge=-10 MPa. Both well below the fracture strength of SiC**

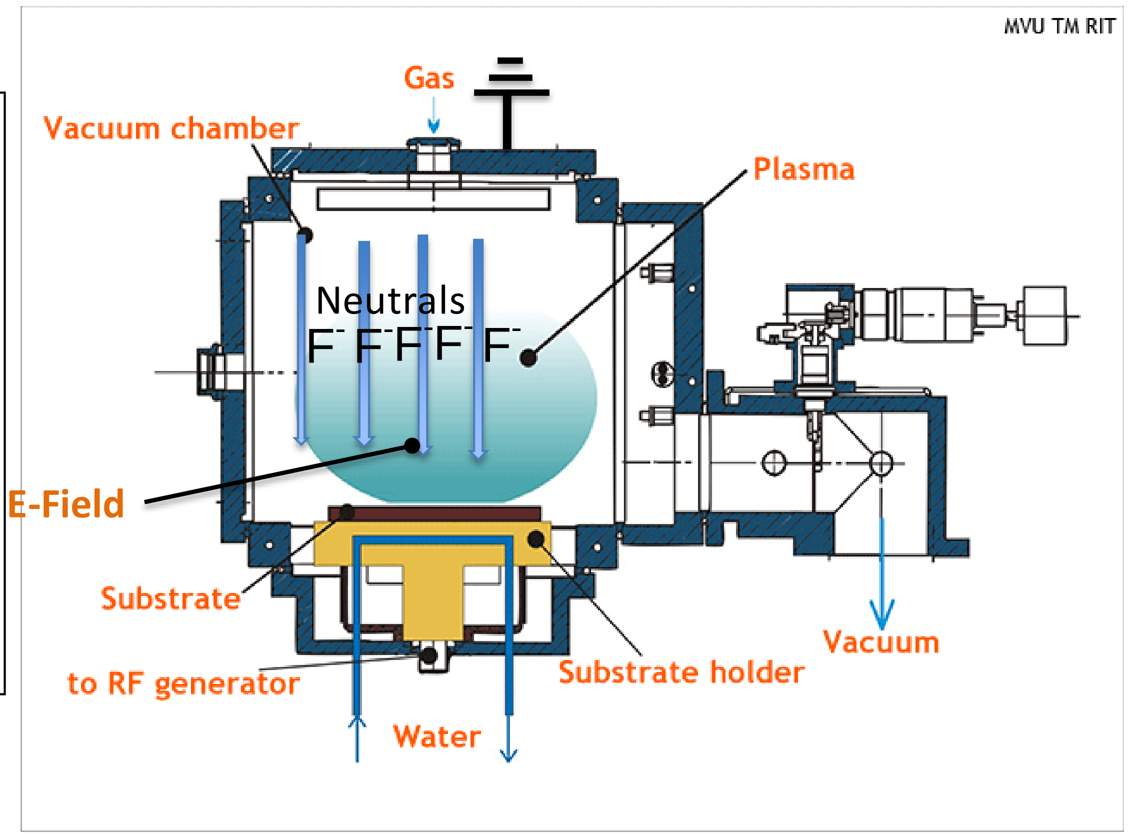
**The predicted applied pressure is ~345 Pa (0.05 psi)**

**Goal: Obtain SiC diaphragm thickness 5 µm or less!**

# Reactive Ion Etching (RIE) for Diaphragm Fabrication: The Basics

Halogen-based gas (i.e., $SF_6$) is ionized to create F ions and neutrals. Neutrals react chemically with target → low etch rates.

With argon added and ionized, $Ar^+$ physical bombardment of target increases surface area → increased chemical reaction and higher etch rates.

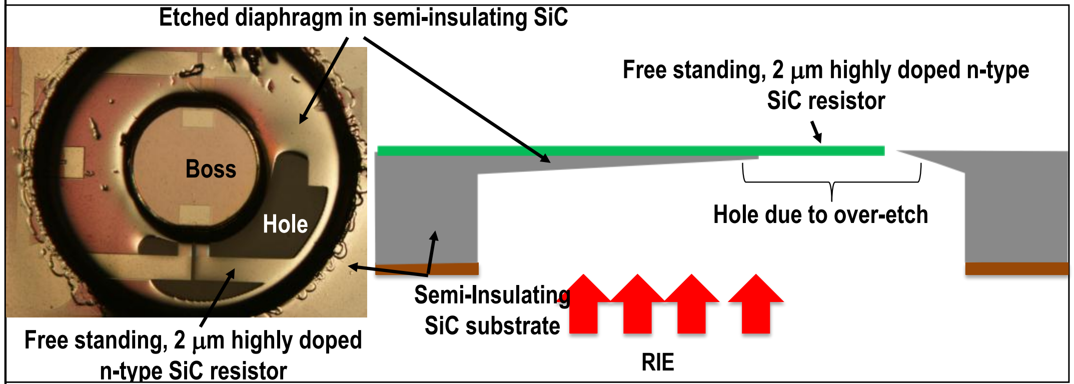


# Conventional SiC RIE

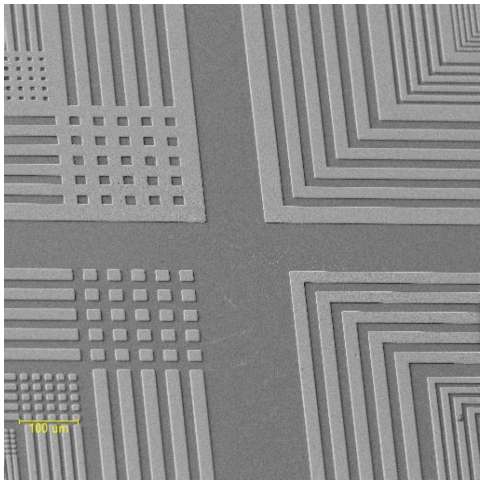
Conventional RIE (with $SF_6$ + Ar) of semi-insulated (SI) 4H-SiC substrate to create thinner diaphragms resulted partial etch-through of the micro diaphragms.

Observed free standing, 2-µm thick "cantilevers" of the patterned highly doped n-type homoepitaxially grown 4H-SiC piezoresistor layer on the SI SiC.

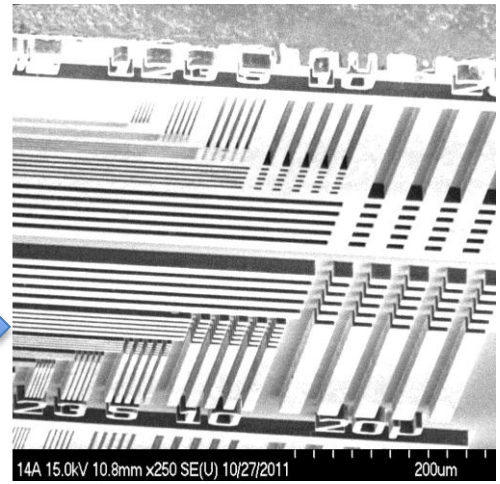
**Etch selectivity between SI and highly doped n-type SiC is proposed.**
**If controlled and reproducible, could lead to the realization of ultrathin diaphragms by RIE.**



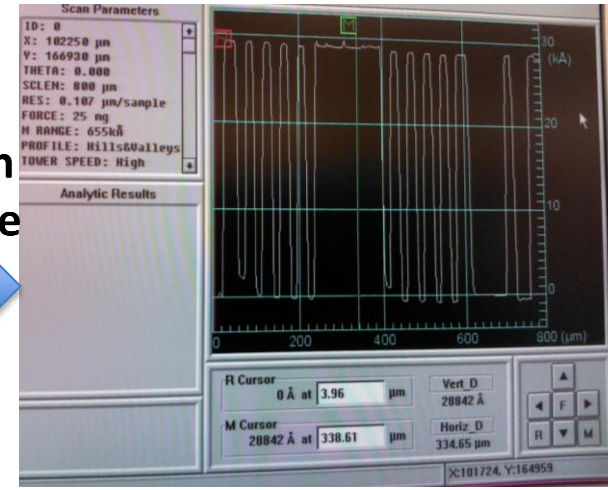
# Experiment: RIE Etching of SI and N-type 4H-SiC with $SF_6$




RIE




Depth Profile




Photolithography and Al pattern definition with calibration mask

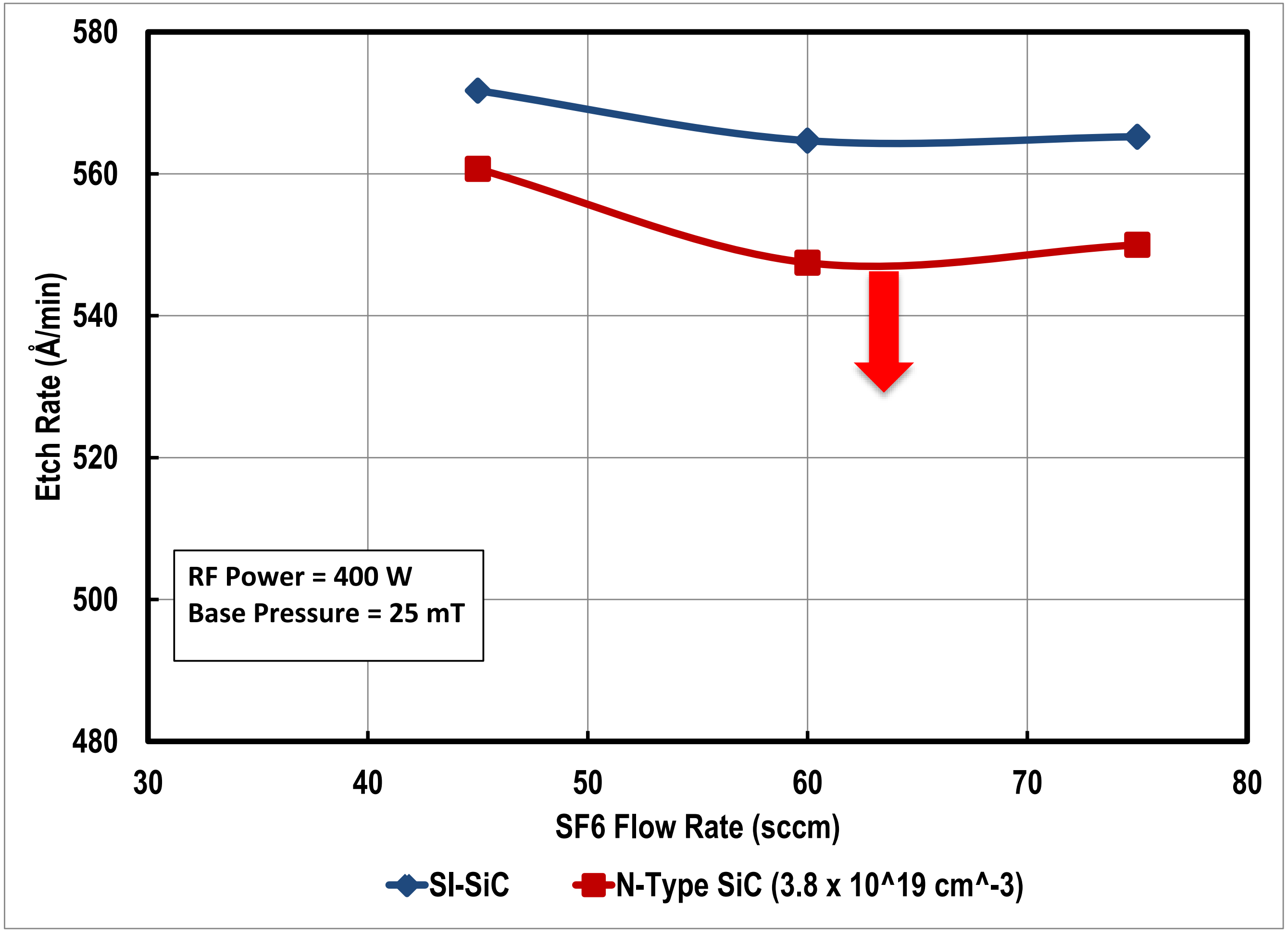
Calibration patterns etched in n-type and semi-insulating SiC samples.

Al mask stripped off and performed depth profile measurement

<u>Fixed process conditions:</u>
RF Power=400 W;
Base Pressure = 25 mT;
Etch Time=2 hours

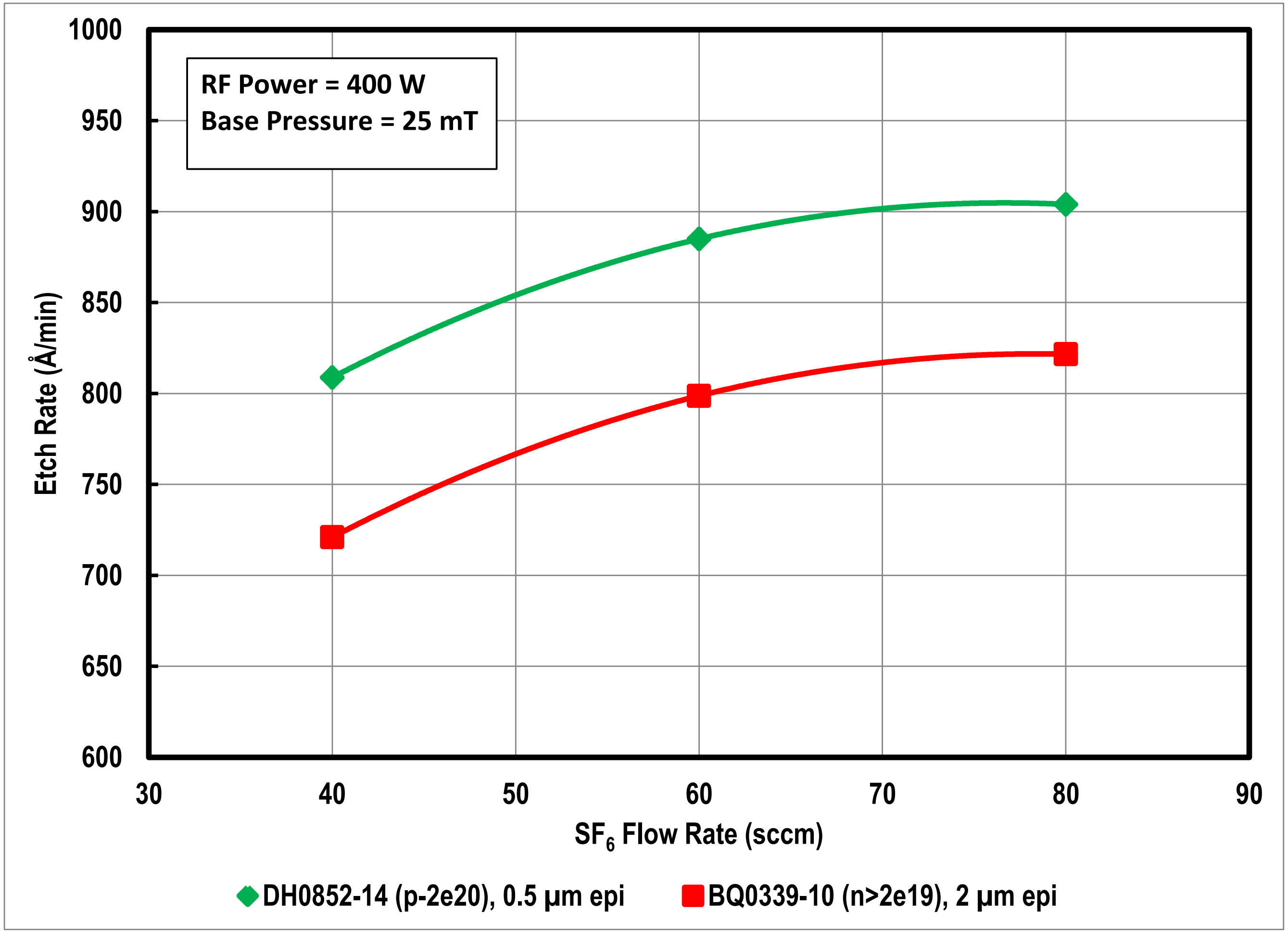
| $SF_6$= Flow Rate (sccm) | SI | N-Type($N_d$=3.8 x $10^{19}$ $cm^{-3}$) |
|---|---|---|
| 45 | 3 | 8 |
| 60 | 4 | 9 |
| 75 | 5 | 10 |

# Selectivity Between SI and N-Type 4H-SiC




Etching selectivity between SI and doped n-type substrates in $SF_6$ gas only.

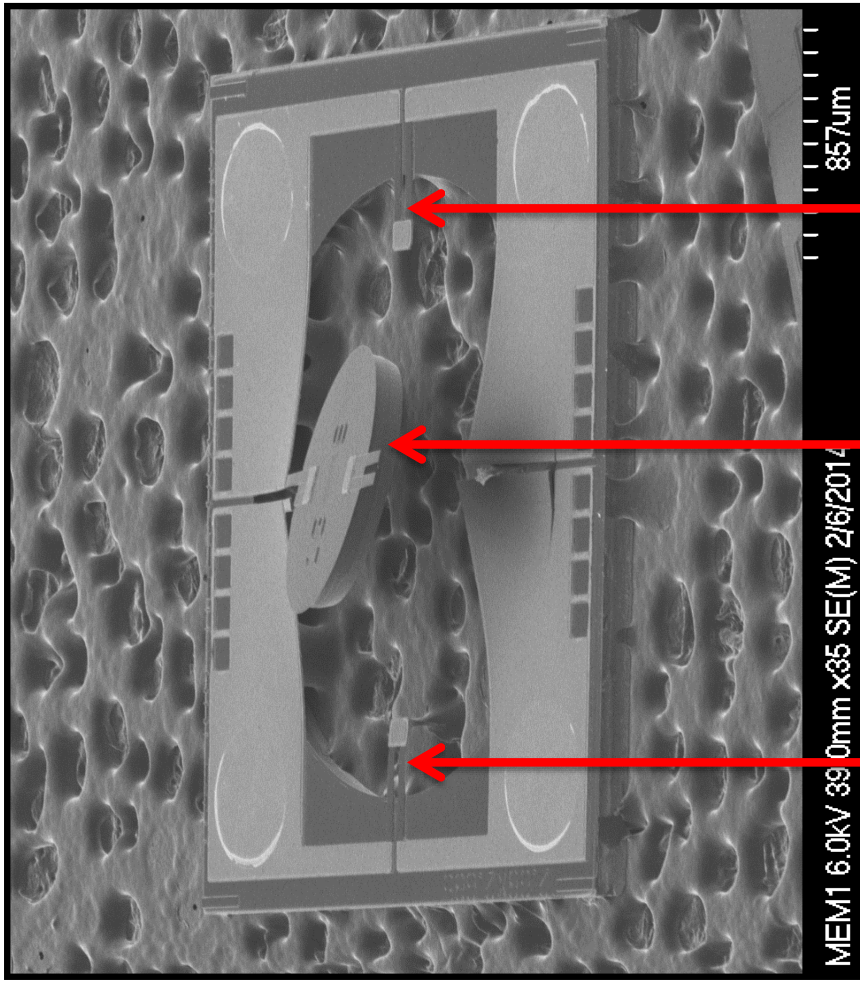
# Selectivity Between N- and P-Type 4H-SiC




**Etching selectivity between highly doped p- and n-type 4H-SiC substrates in $SF_6$ gas only.**

# DSRIE Release of N-Type Cantilevers from SI Substrate

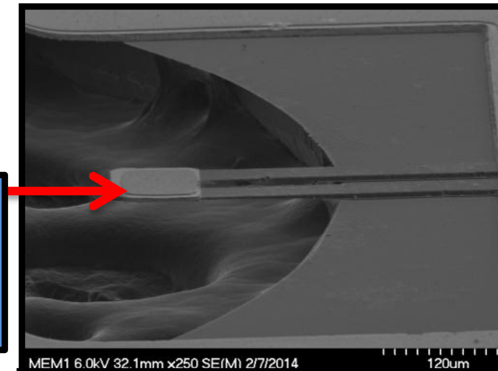
## First proof of concept

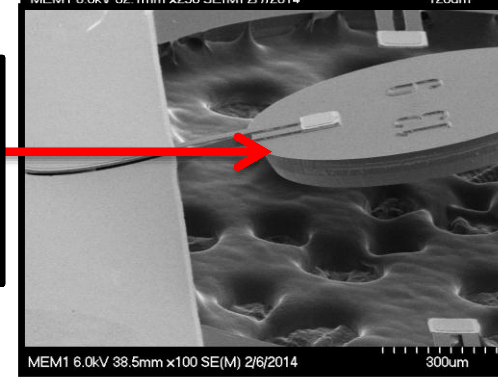


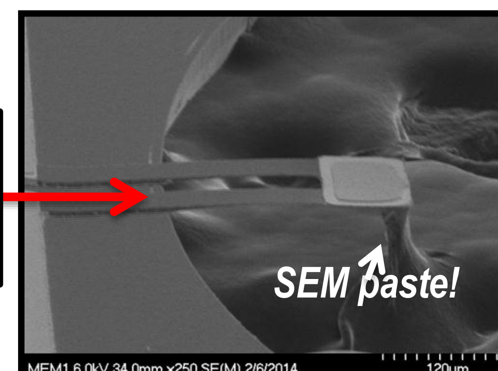

Suspended 2 µm thick single crystal SiC piezoresistor

Proof mass suspended by 2 µm thick single crystal SiC piezoresistor

Suspended 2 µm thick single crystal SiC piezoresistor



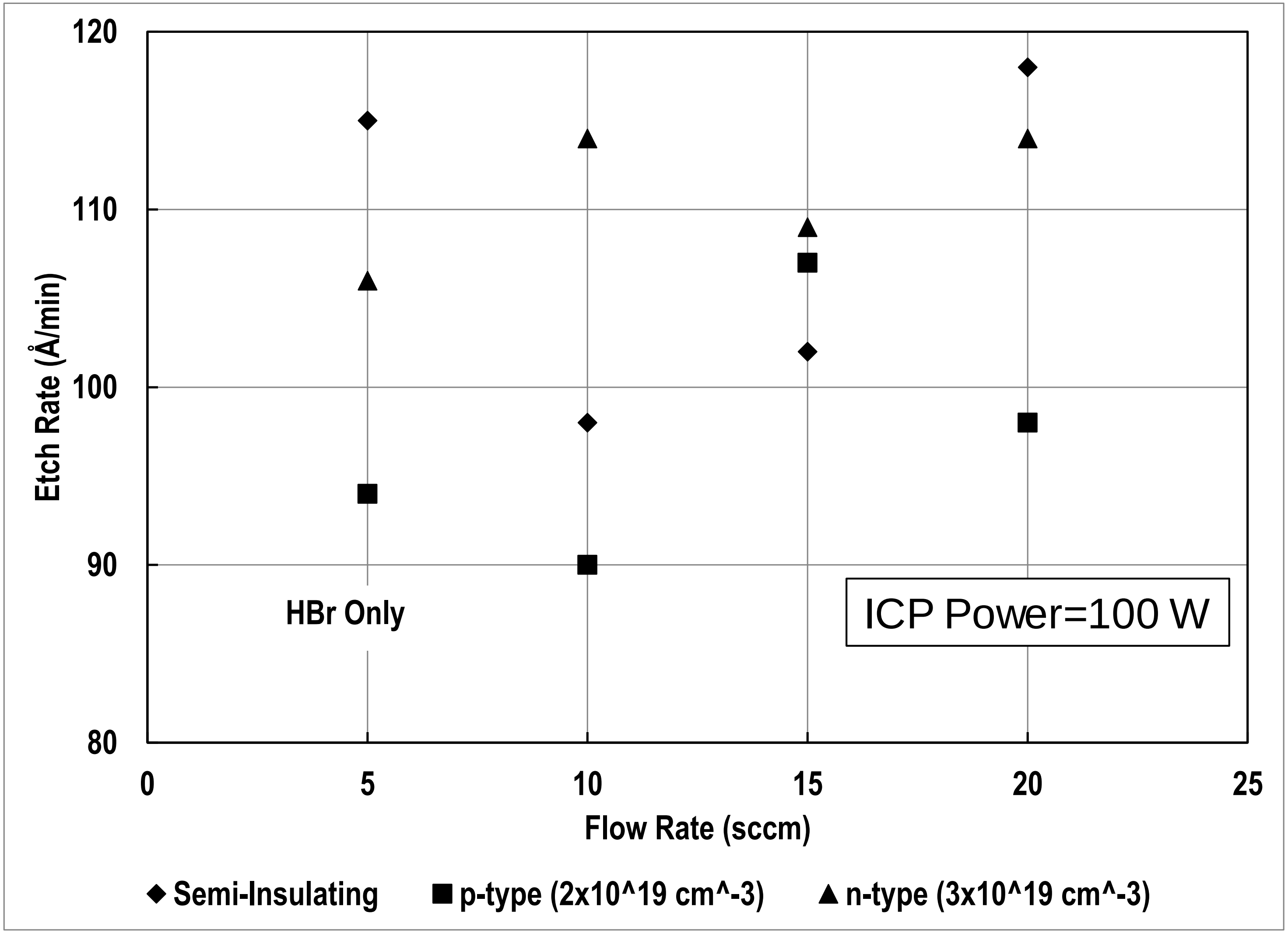



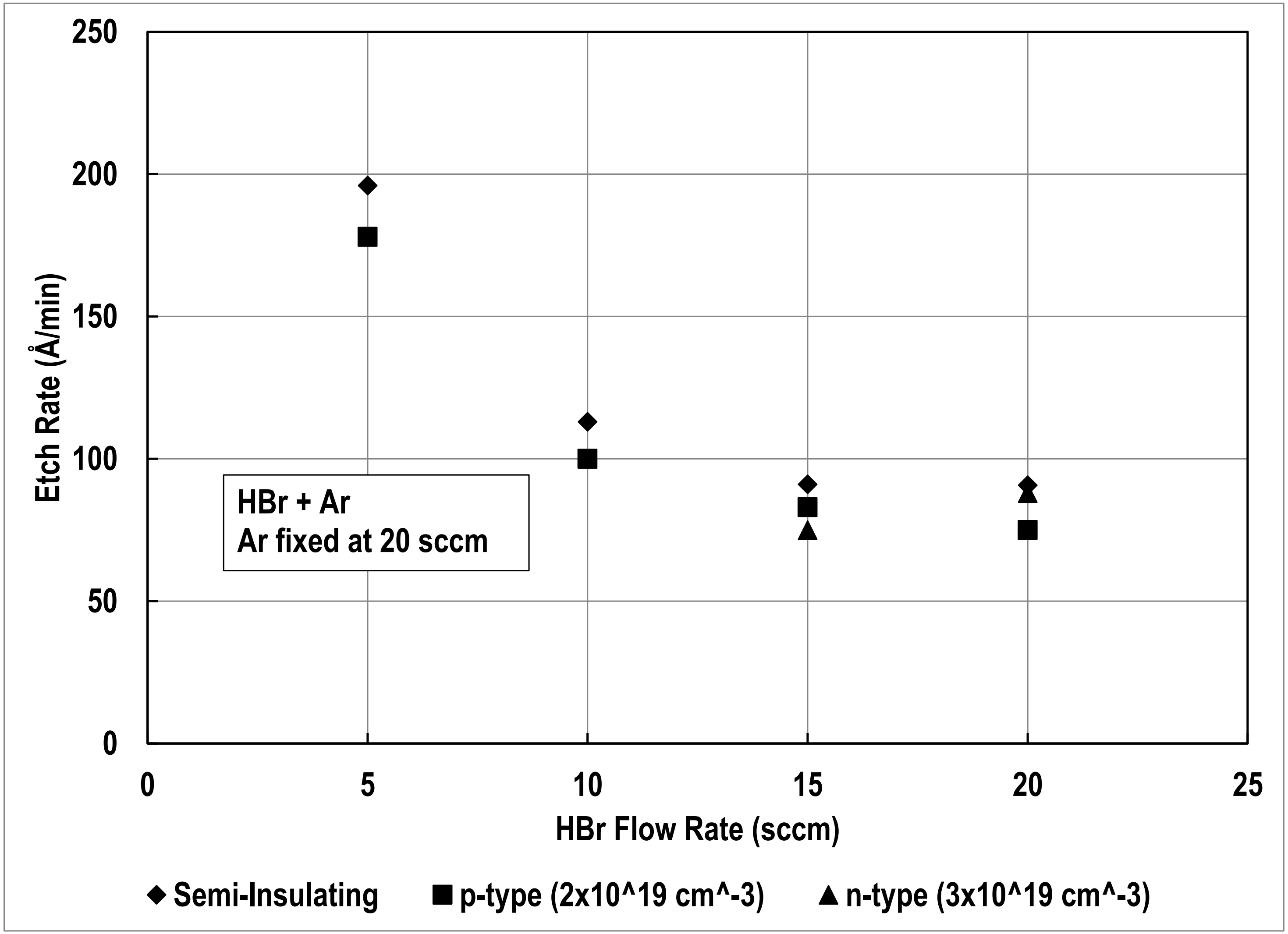
# Investigation of Selected Halides
# $BCl_3$, HBr, and $Cl_2$

- **Wafer Clean**
  10 minutes in Acetone and 10 minutes in IPA
- **Nickel Deposition**
  230 nm thick nickel in Cooke E-beam Evaporator
- **Optical lithography**
  AZ 5214 E 4000 RPM about 1.3 um thick; Bake at 110 degrees for 1 minute; Expose with NASA mask for 40 seconds at Karl Suss MJB Aligner; Develop in AZ327 for 4 minutes.
- **Wet Etch**
  4 minutes in Nickel Etchant (Type TFB from Transene Company); 10 minutes in Acetone to remove photoresists
- **ICP-RIE Etch**
  100 W for RIE and ICP power settings; Gas flow rates from 5 sccm, 10 sccm, 15 sccm, to the maximum of 20 sccm; First depth measurement with Alpha Step
- **Nickel Removal**
  4 minutes in Nickel Etchant; Second depth measurement

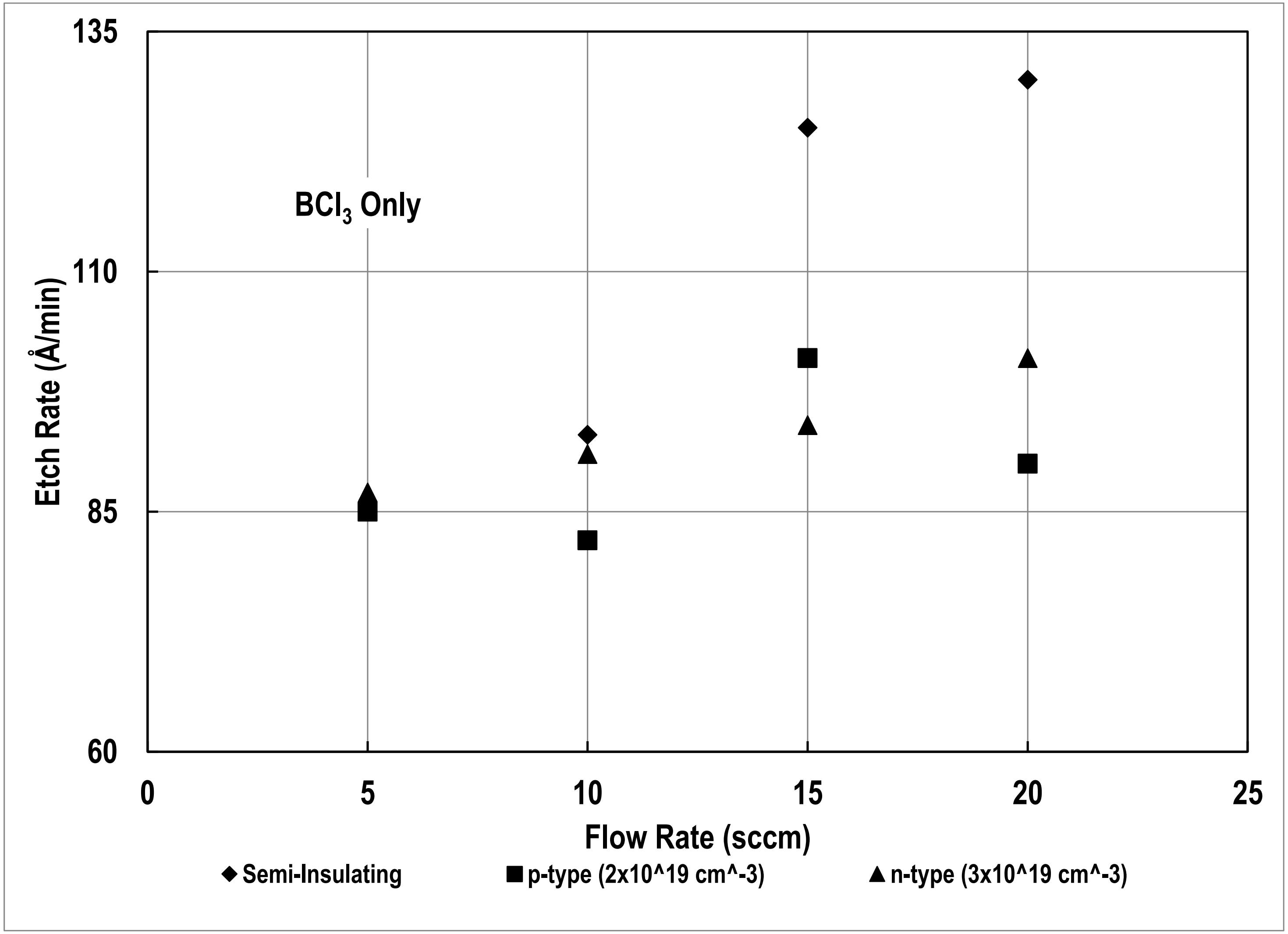
# Etching with HBr



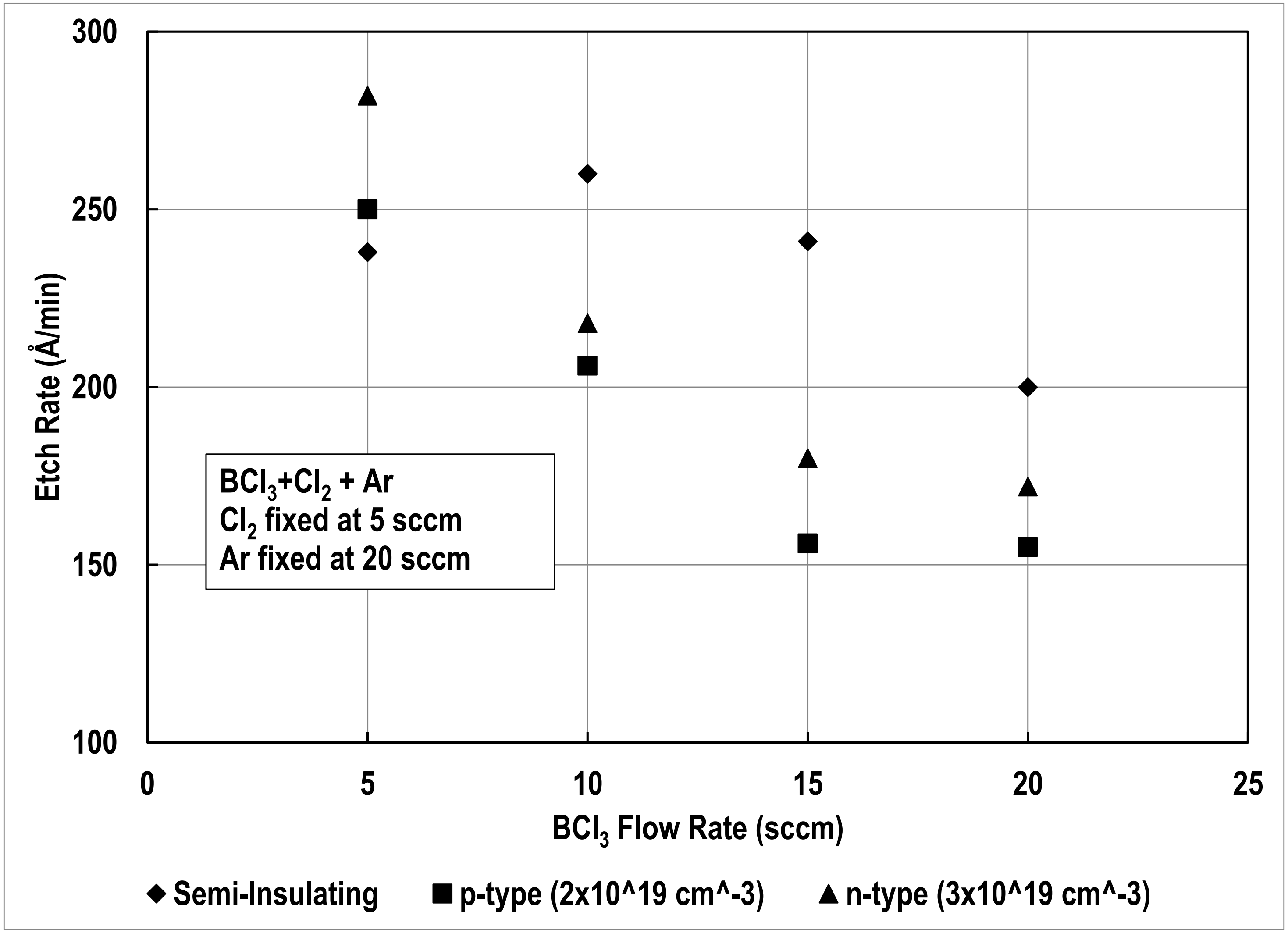
# Etching with HBr + Ar



# Etching with $BCl_3$



# Etching with $BCl_3 + Cl_2 + Ar$



# Summary of ER Selectivity Between SI and N-Type 4H-SiC

| Gases | Power (W) | Flow rate (sccm) | ER |
|---|---|---|---|
| SF6 | 400 | 60 | 1.03 |
| HBr | 100 | 10-20 | ~1 |
| $BCl_3$ | 100 | 15-20 | 1.33 |
| $BCl_3$ + $Cl_2$+ Ar | 100 | $BCl_3$ (15), $Cl_2$ (5), Ar (20) | 1.37 |

Facility limitation prevented investigation of higher power and flow rates

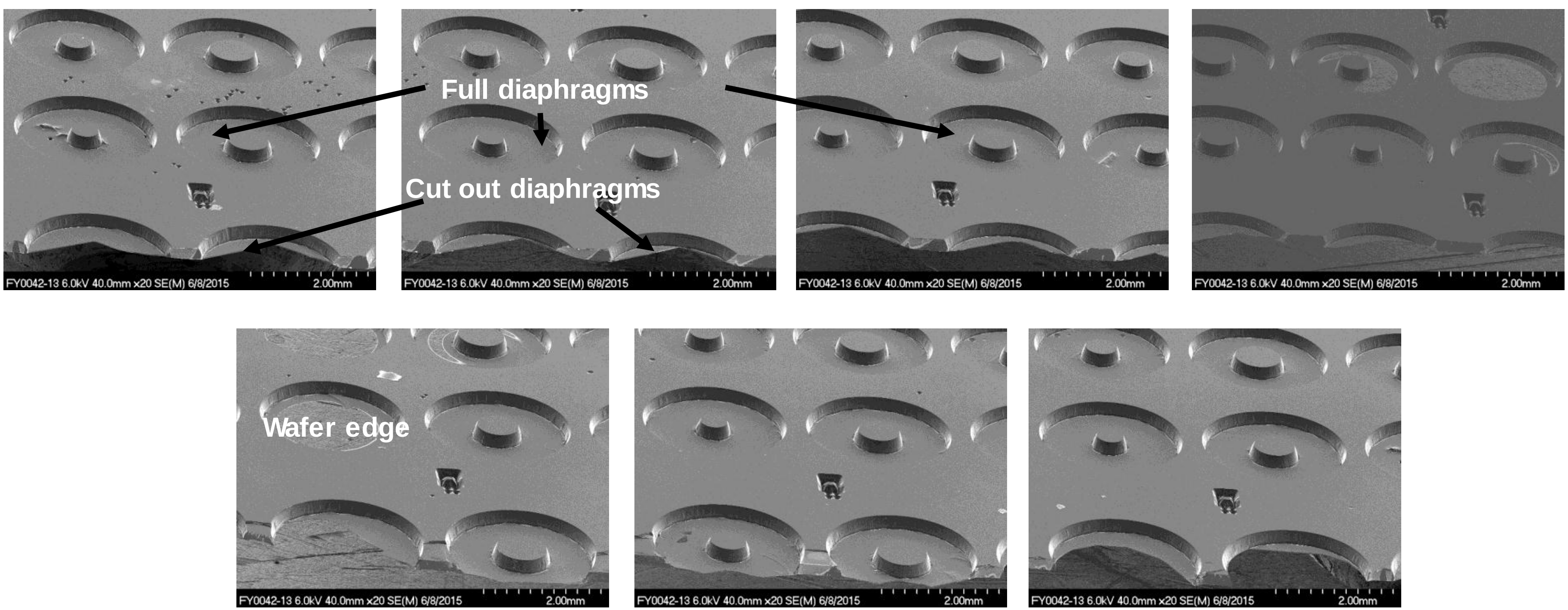
# Ultrathin Diaphragm with $BCl_3$ + $Cl_2$ + Ar etching


3 µm 4HSiC n-type epilayer

150 µm 4HSiC SI substrate

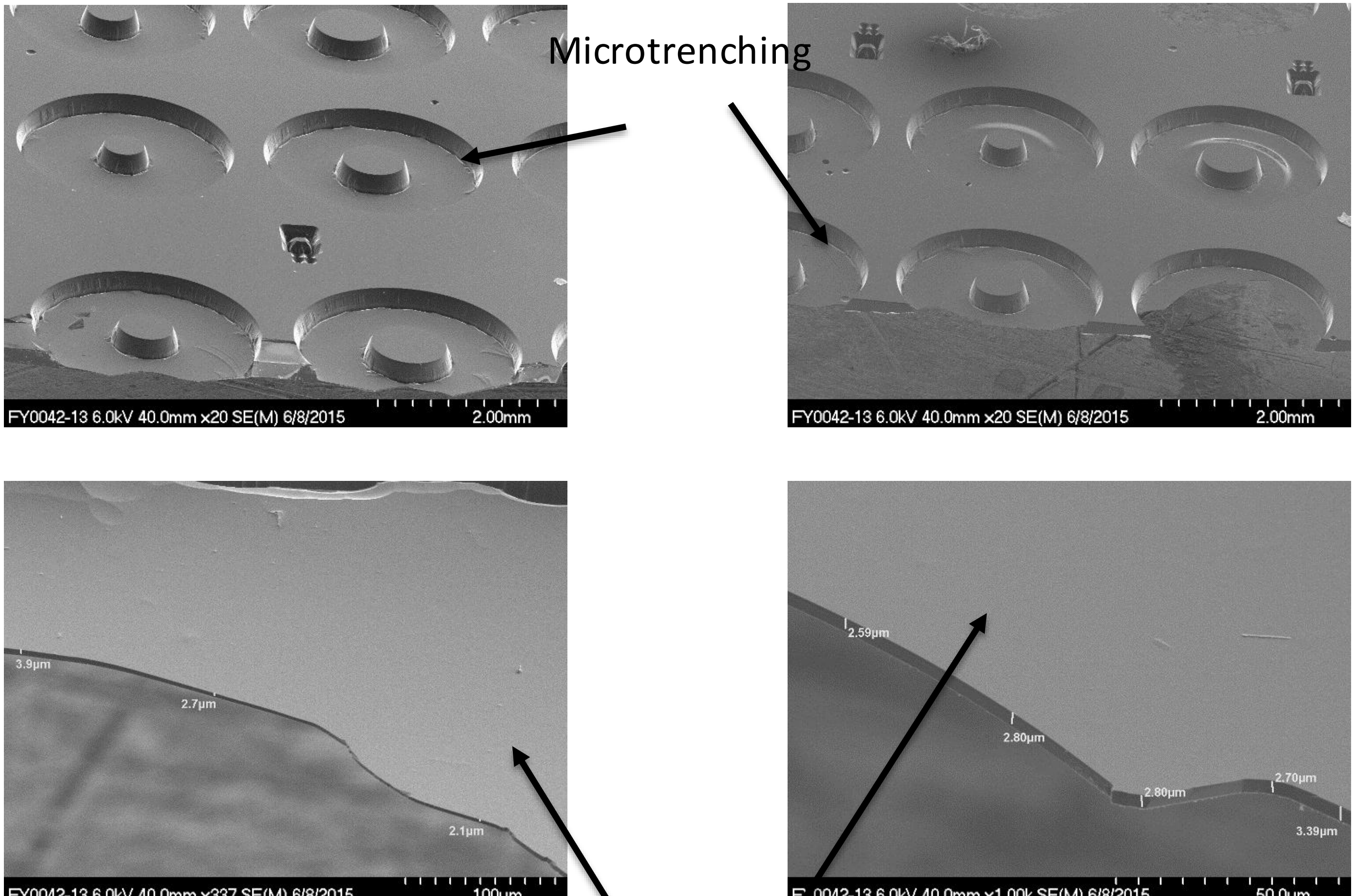
Start with fast ER recipe through most of SI substrate, then switch to DSRIE:
$BCl_3$ (15 sccm)
$Cl_2$ (5 sccm),
Ar (20 sccm)
Power 100 W

Results



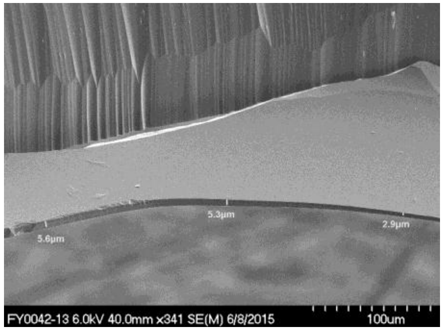
# Ultrathin Diaphragm with BCl3 + Cl2 + Ar etching

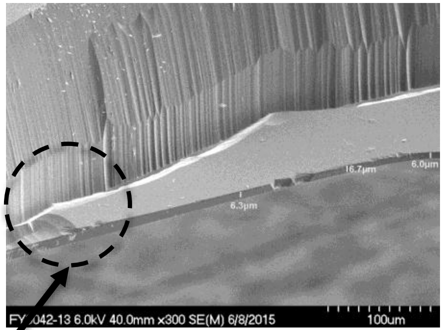
Microtrenching

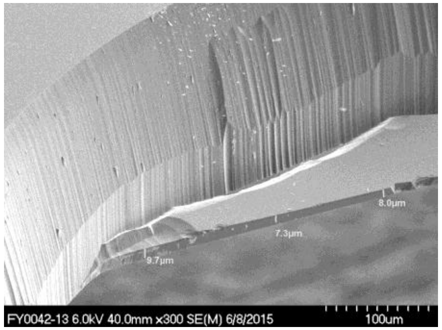



Diaphragm thickness between 2 and 3.5 µm

# Ultrathin Diaphragm with BCl3 + Cl2 + Ar etching

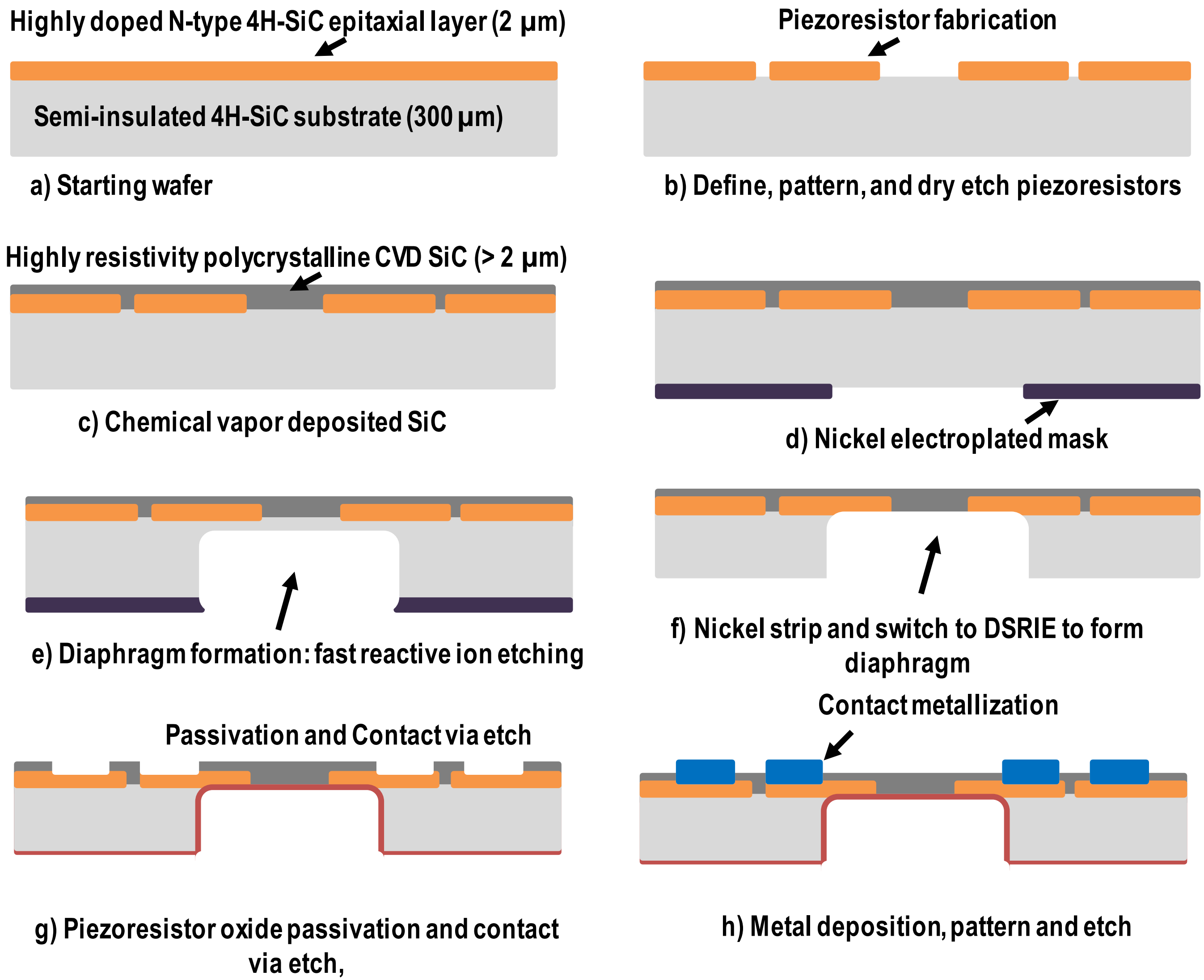



Observed microtrenching and etch non-uniformity across wafer causes reduced yield. Etch conditions and chamber optimization are required to increase yield

# Fabrication Process Steps of Ultrathin Diaphragm Sensors Using DSRIE



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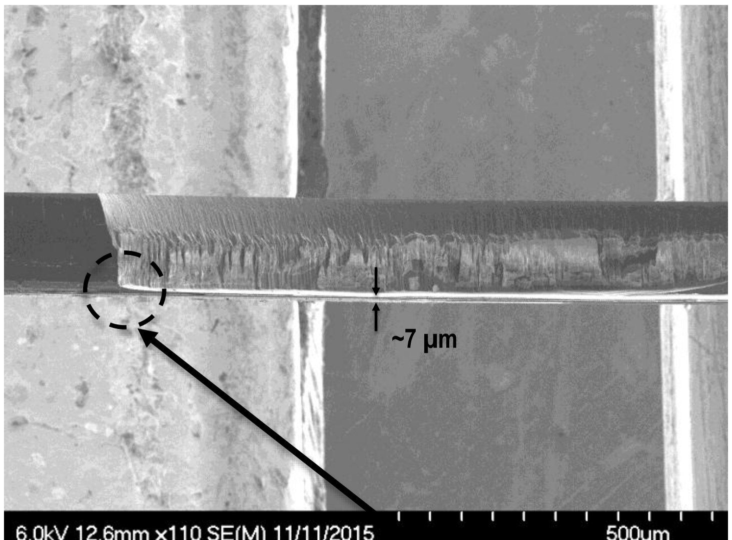
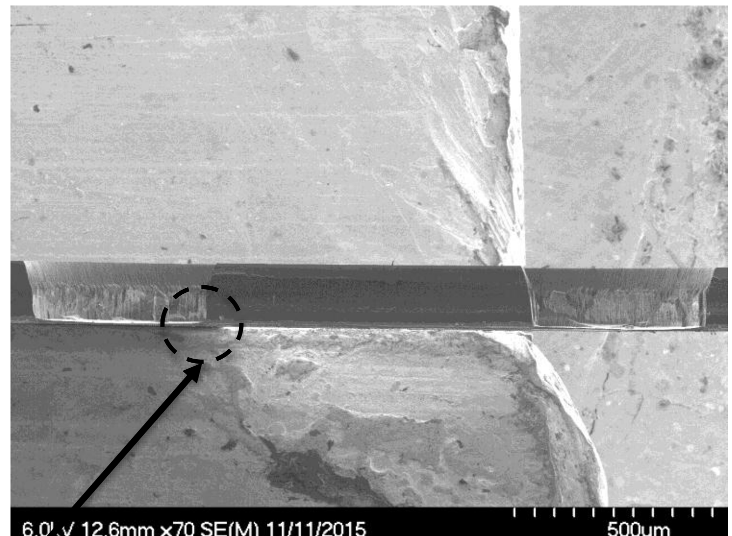
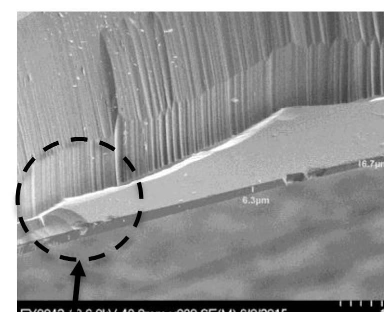


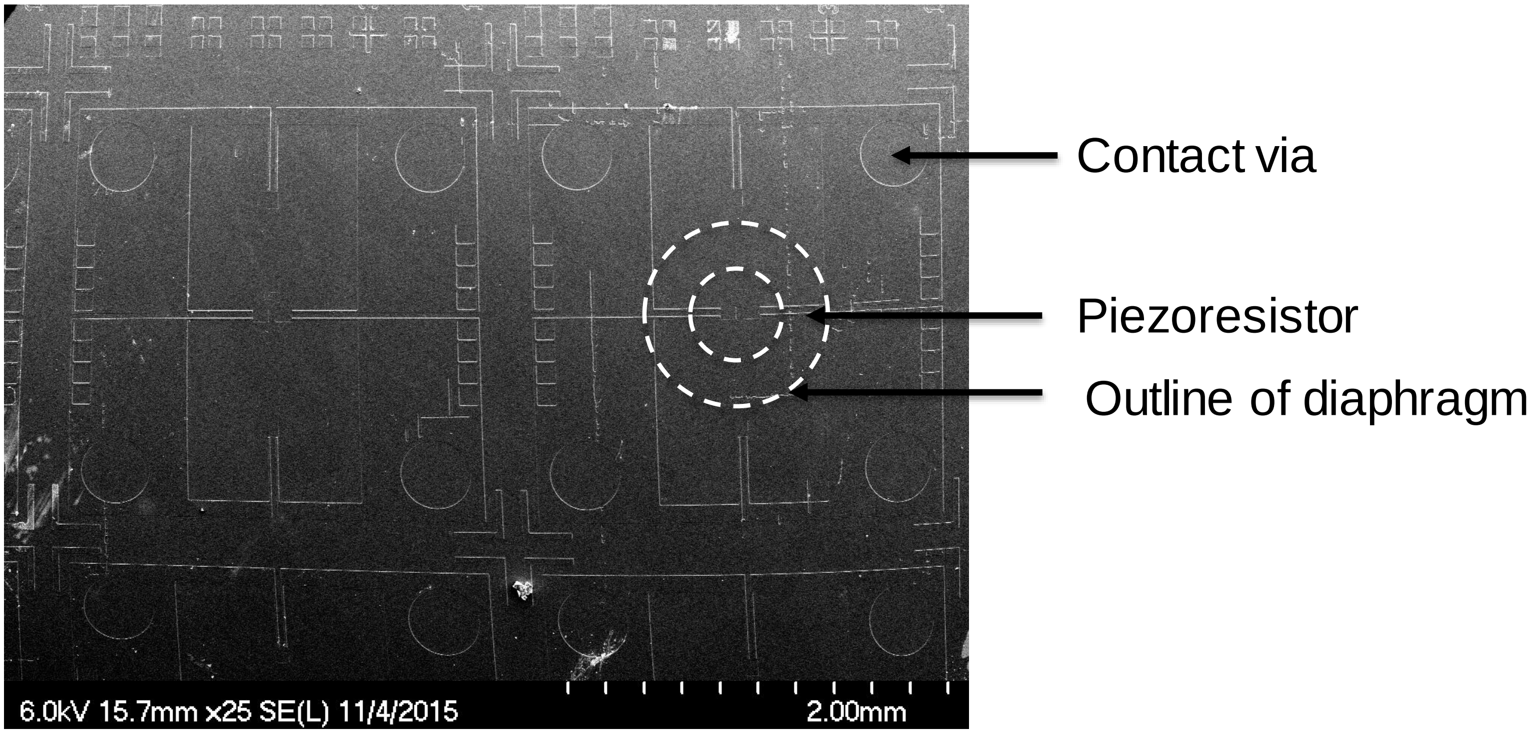




Diaphragm microtrench eliminated
Diaphragm thickness between 4 and 7 µm



Before

# Fabrication Process Steps of Ultrathin Diaphragms-Sensor Side




**Next Step: Complete fabrication, package and test SiC dynamic pressure sensor with ultrathin diaphragms**

# Innovation and Impact on Mission

**Mission Challenges:** Thermoacoustic instabilities in combustors are known to be precursor to flame-out or damage to engine components.

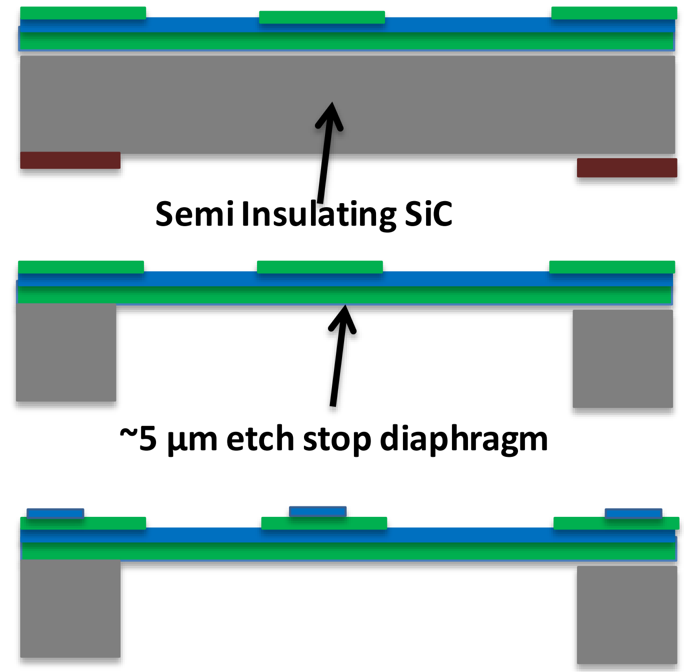
Existing instability prediction models have high uncertainty margins. Need environmentally robust and reliable pressure sensors for model validation and improvement.

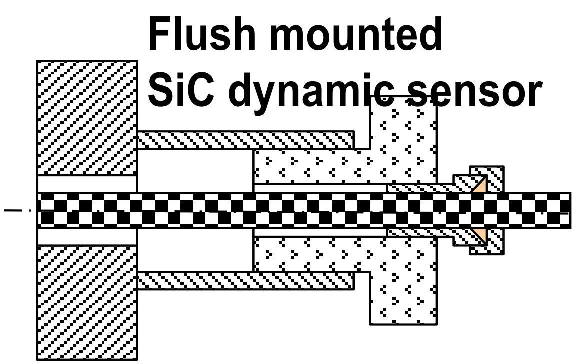
**Existing Technology Gap:** SoA sensors placed feet from test article-limits frequency bandwidth; Water cooling adds “vortex noise” to corrupt signal.
Robust and reliable sensors needed for direct (no water cooling) measurement of sub-psi dynamics at >500 °C.

600 °C SiC pressure sensor technology currently exists, but SiC fabrication technology cannot produce the ultra-thin diaphragms to achieve the high sensitivity needed to resolve sub-psi pressure dynamics.

**Innovation: DSRIE will result in ultra-thin (< 5 µm) SiC diaphragms to accurately resolve sub-psi dynamics at temperatures in excess of 500 °C (current capability).**

Benefit of SiC Etch Stop Mechanism



# Summary/Conclusion

- We investigated the possible existence of dopant and conductivity selectivity in SiC during reactive ion etching with selected halide gases;

- Within the limits of experimental space, the maximum etch selectivity between the SI and n-type SiC was 1.37, enough to release free standing n-type cantilevers and diaphragms of less than 10 µm thick.

- Applied process to fabricate array of ultrathin SiC diaphragms for pressure sensors.

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