Advanced Solid State Sensors for Vision 21 Systems

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Prepared By

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Overview of Presentation

Background

- *Role of sensors in power generation*
- *Silicon carbide & silicon carbide sensors*
- *Summary of key related research*

WVU Silicon Carbide Sensors Program

- *Objectives, technical issues, approach, & facilities*

Results

- *Preparation of atomically flat 6H-SiC substrates*
- *Deposition & thermal stability of Pd on 6H-SiC*
- *Sensor modeling & characterization*

Summary

- *Project status & future work*

Role of Sensors in Power Generation

Future Power Plants

-Highly integrated power generation modules

-Controlled by advanced computer algorithms

-Sensors provide real-time plant-computer interface

From: Vision 21 Technology Roadmap

Sensors Measurements

-Gas species concentration

-Temperature profiles

Sensor Requirements

-Measure low concentrations

-Withstand harsh environments

Silicon Carbide

Wide Band Gap Semiconductor

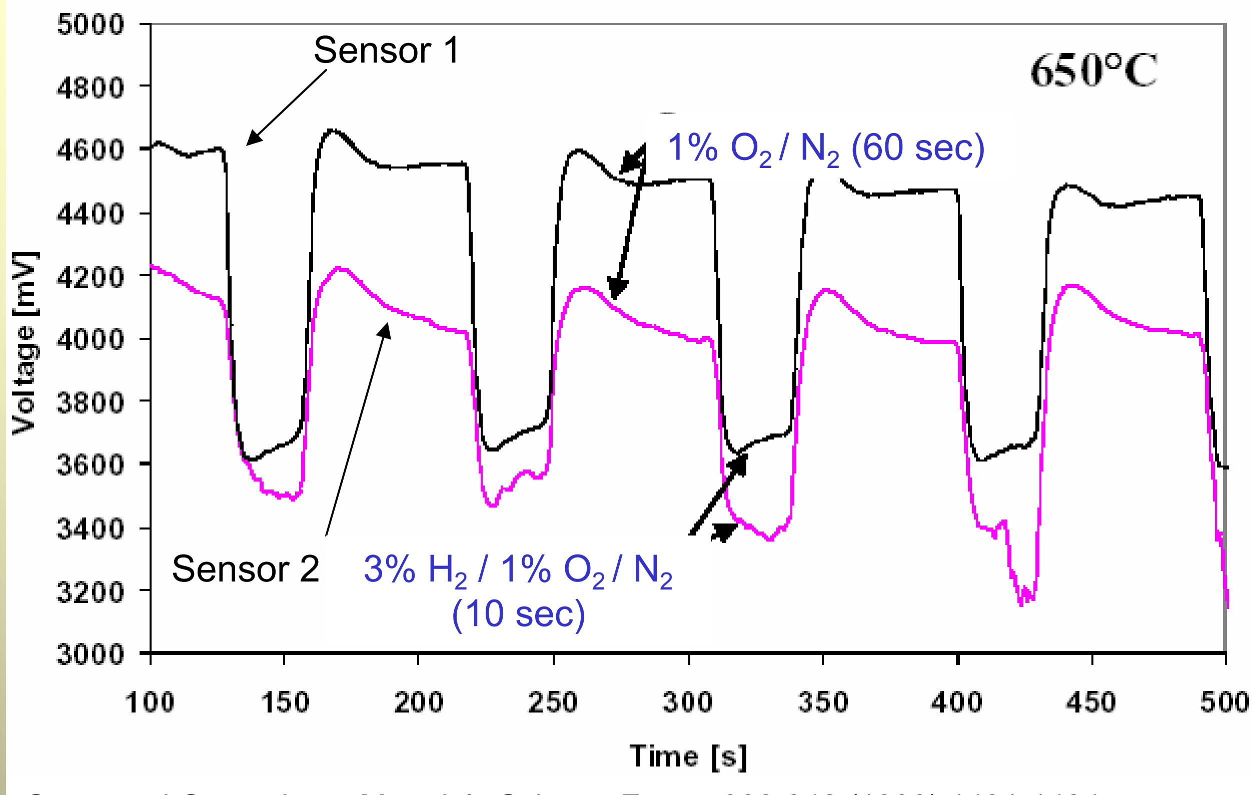
Studied Extensively Since Early 80's for High Temperature Electronics Applications

Electrical Devices & Sensors Operated at Elevated Temperatures

Signal from two sensors for gas flow switched between two gas mixtures:

1% O_2/N_2 (60 sec) and

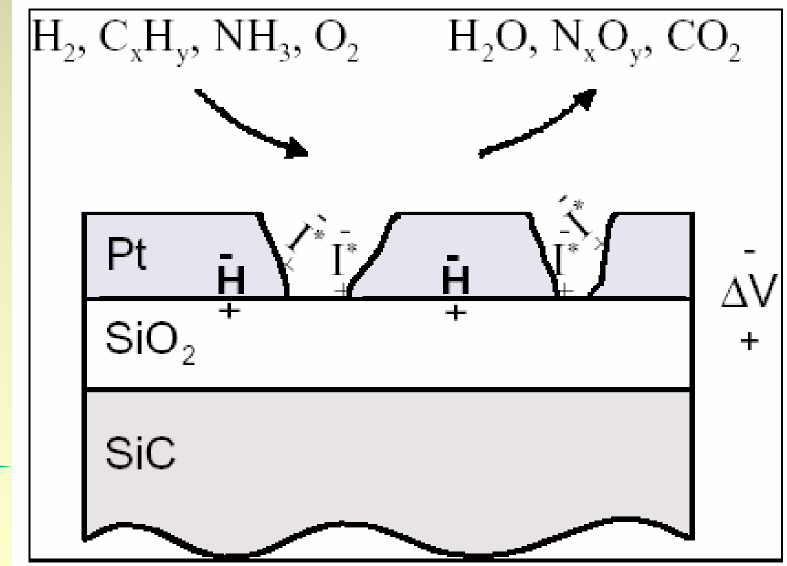
3% H_2 /1% O_2/N_2 (10 sec)



Spetz and Co-workers, Materials Science Forum 338-342 (1999) 1431-1434

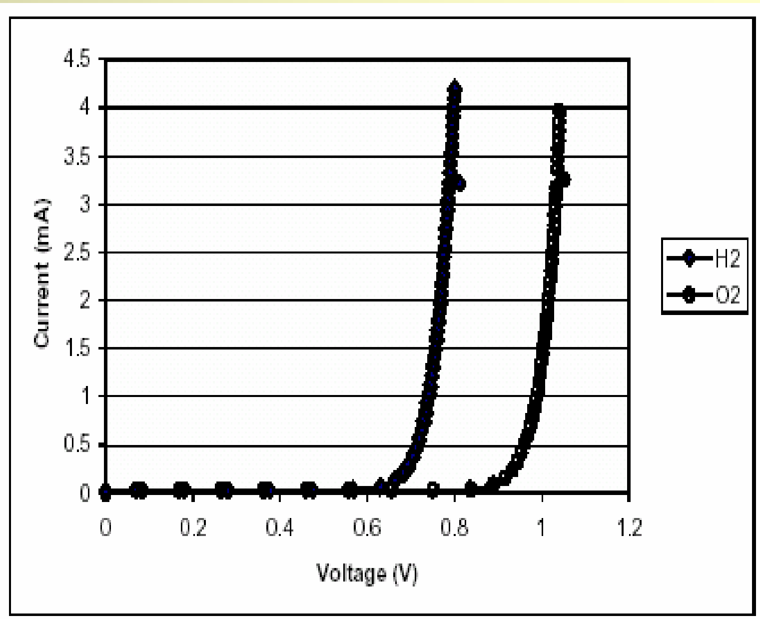
SiC - Sensor Structure and Operation

Target Molecules React with Metal Surface to Produce Steady State of Surface Intermediates



Surface Intermediates

- *Produce polarization field*
- *Alter electrical response*



A.L. Spetz, et al, Proc. Transducers´99, Sendai, Japan, June 7-10, pp. 946-949, (1999)

SiC Sensor Limitations

- *Temperature dependent response*
- *Long term thermal degradation*
- *Relatively low temperature of operation (T < 500 ^oC)*

Previous Studies

NASA Glenn Research Center – Long Standing Program

- *Chen et al., J. Vac. Sci. Technol. A15 (1997) 1228*
- *Sensors used Pd/SiC Schottky diode structure*
- *Observed thermal drift for T~425 ^{o}C for times~140 hr*
- *Thermal degradation due to interdiffusion & reaction to form Pd_xSi at interface*
- *Used $Pd/SiO_2/SiC$ structure to improve thermal stability*
- *Oxide improved stability somewhat but degraded sensor performance*

Conclusion – "Further stabilization of the diode structure is necessary for long term, high temperature sensor operation."

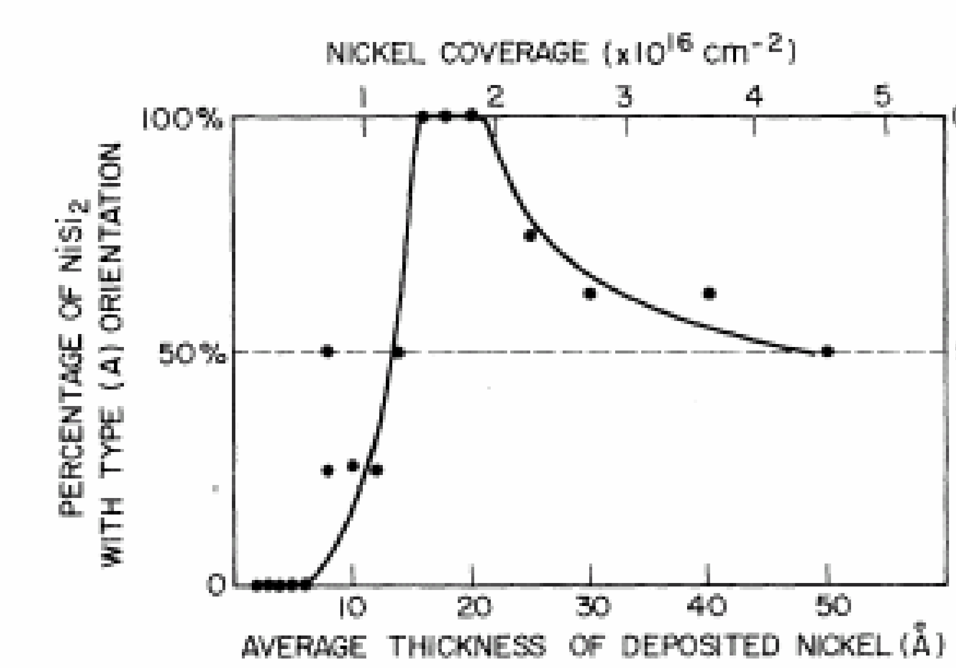
Previous Studies

Bell Labs - Early Work in Metal-Silicides

- *Poate & coworkers, Phys. Rev. Lett. 50 (1983) 429*
- *Standard method of silicide formation involves annealing thick metal films (>20 nm) on Si substrate*
- *Generally produces polycrystalline silicide films with island or columnar growth mode*

Key Result

- *Annealing thin (1.5-2 nm) Ni films produces a stable silicide epilayer*
- *Silicide epilayer serves as a template for metal film or silicide epitaxy*



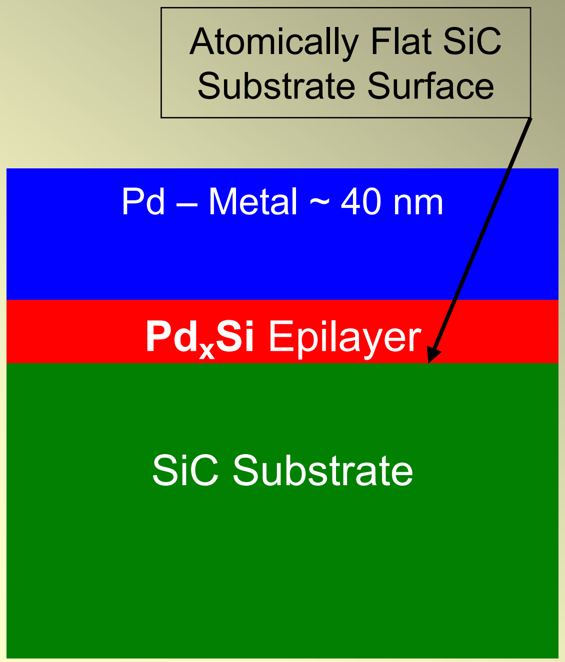
Key Question

- *Can these results be extended to Pd/SiC?*

WVU Research Issues

Increase Lifetime & Operation Temperature by Controlling Diffusion & Reaction

- *Remove surface and subsurface polishing defects from SiC substrates to produce atomically flat SiC surface*
- *Grow stable epitaxial silicides at metal-semiconductor interface*
- *Use epilayer as template for deposition of remaining device structures*



Fabricate Device Structures & Characterize Performance

- *Characterize electronic properties of stabilized device structures*
- *Lifetime & gas sensitivity testing*

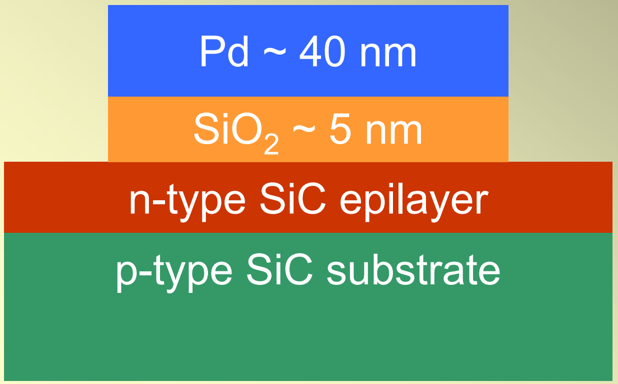
WVU Silicon Carbide Sensors Program

Objectives

- *Fabricate integrated gas & temperature sensor*
- *Increase sensor lifetime & operational temperature range*

Technical Issues

- *Integrated sensor compensates for inherent temperature dependence*
- *Control diffusion & reaction at Pd/SiC interface to increase sensor lifetime & operational temperature*

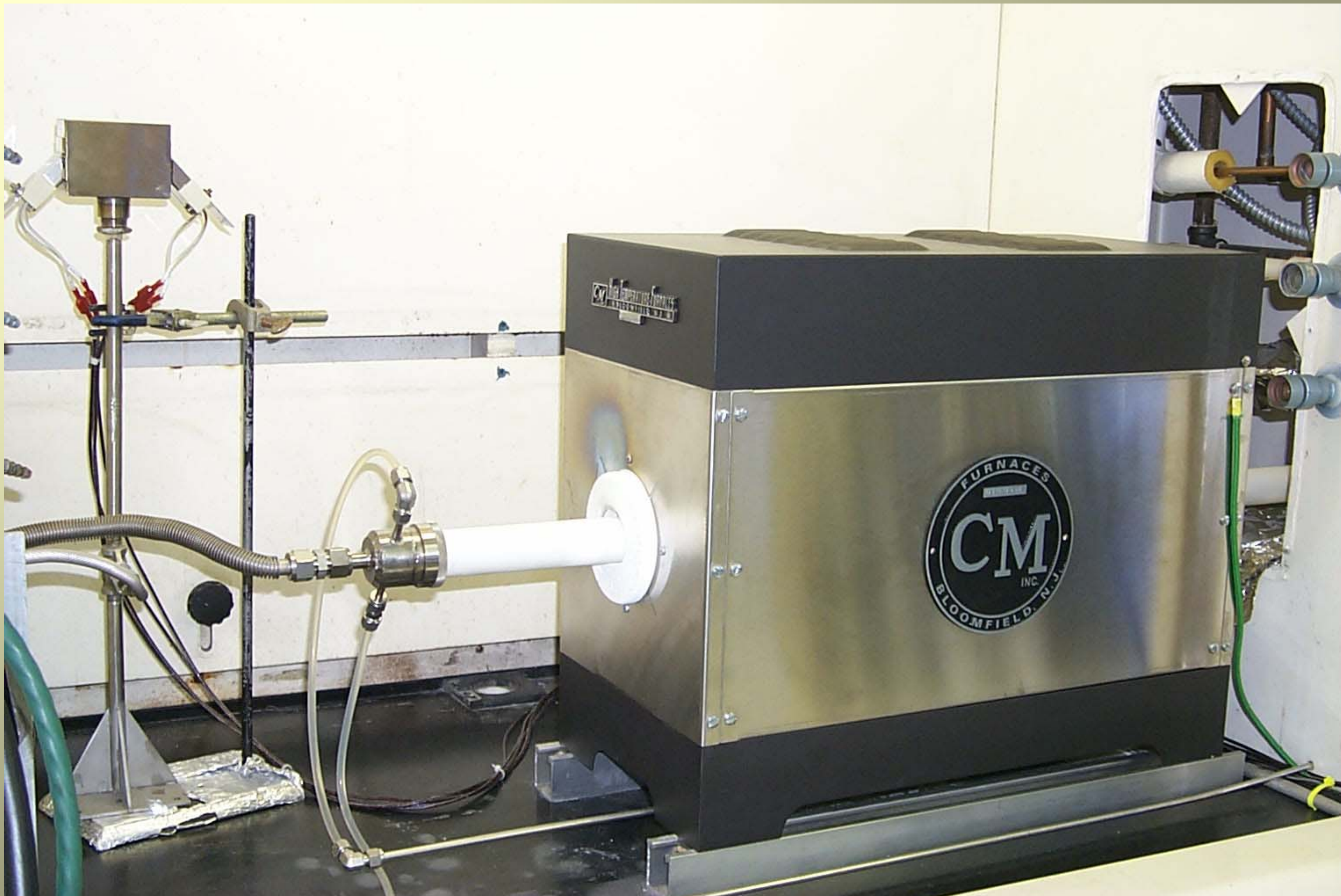


NASA Sensor Structure

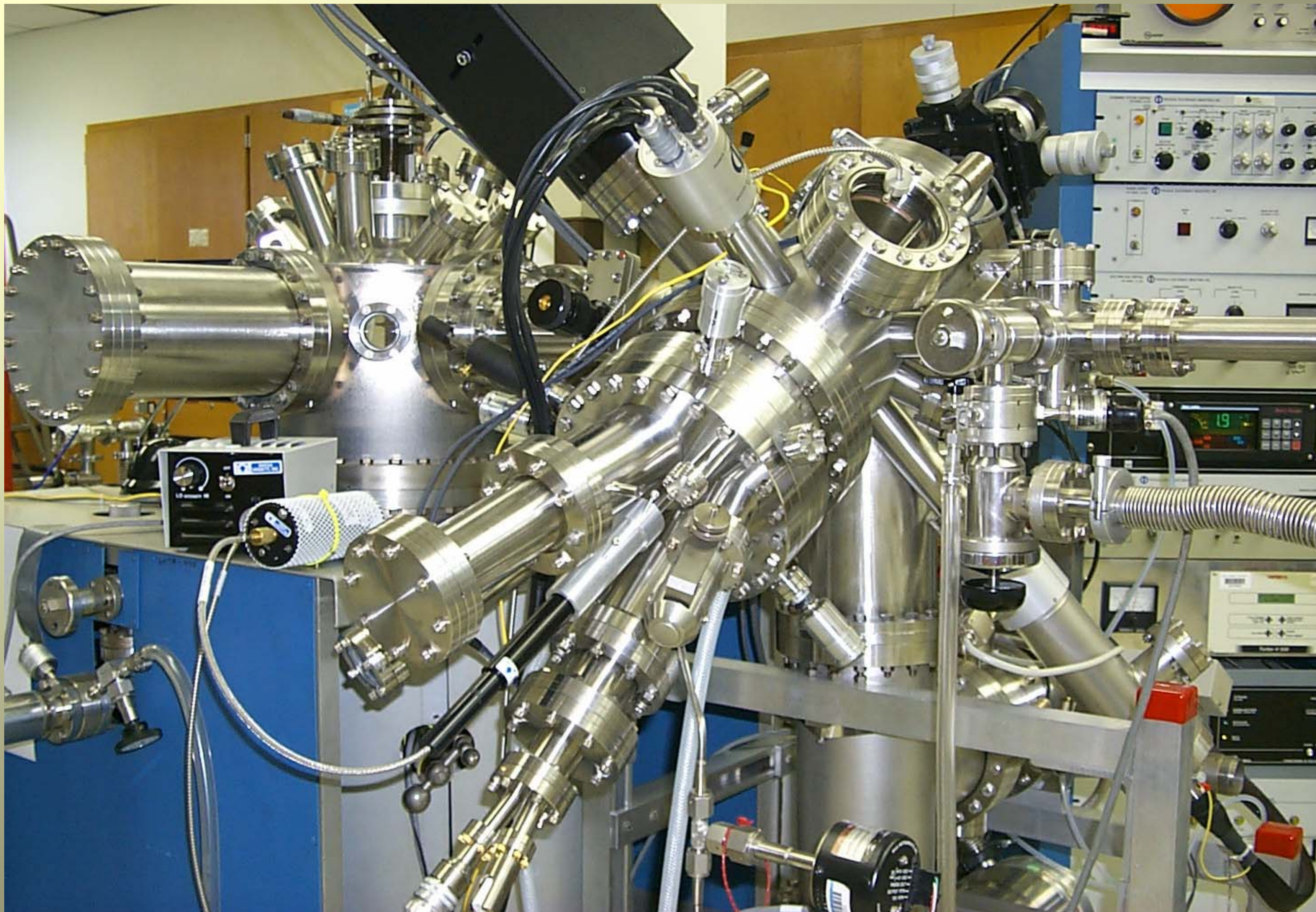
Experimental Approach

- *Remove 6H-SiC substrate damage ⇒ Atomically flat surfaces*
- *Deposit thin, thermally stable, Pd_xSi epilayer*
- *Use epilayer as template for subsequent deposition of Pd or Pd / SiO_2*
- *Fabricate & characterize sensor structures*

Experimental Facilities



High Temperature Oven for Hydrogen Etching of SiC



Gas Source Molecular Beam Epitaxy (GSMBE) System

Sputter Deposition System for Metal Thin Films

Results

Preparation of Atomically Flat 6H-SiC ✓

Deposition & Stability of Pd/SiC Interface

Sensor Modeling & Characterization

Preparation of Atomically Flat 6H-SiC

Commercial n-type 6H-SiC Substrate Wafers

- *Basal plane (0001) – Si Surface*
- *3.5^{o} off axis*
- *Resistivity ~0.03 ohm-cm*
- *CREE & Sterling (Dow Corning)*

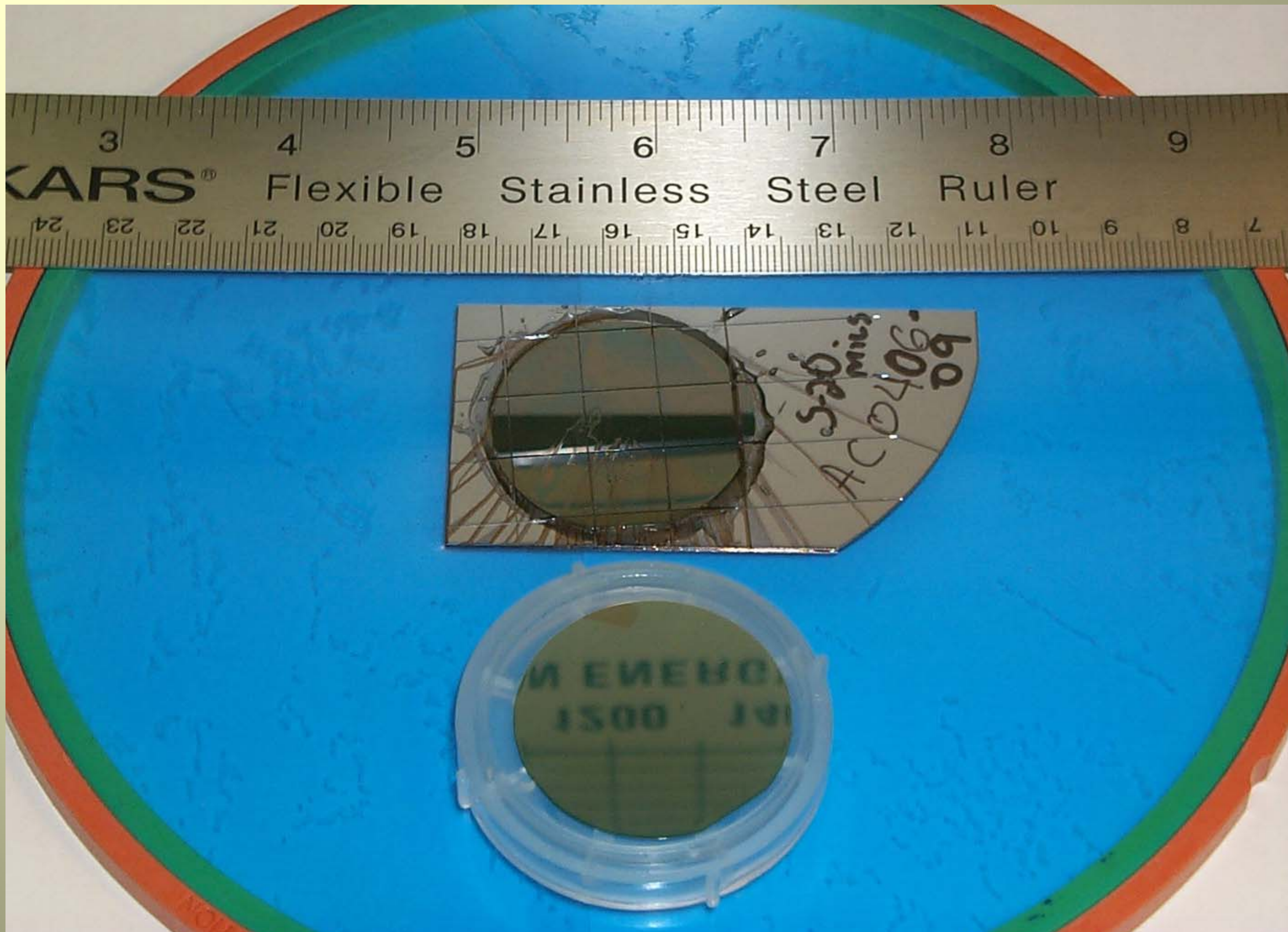
n-type 6H-SiC (0001)

Standard Method of Substrate Preparation

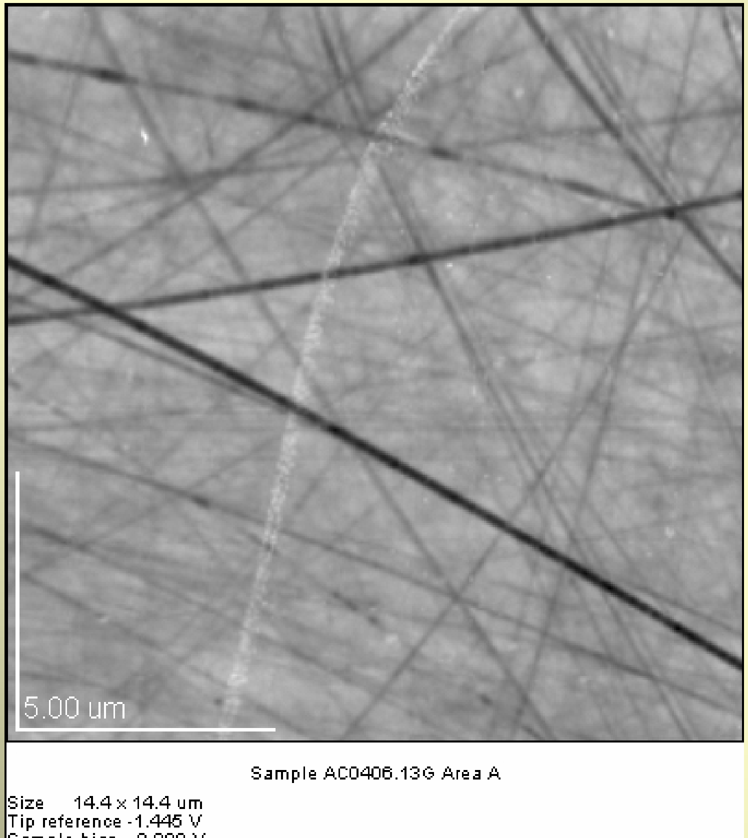
- *Mechanical Polish (Vendor)*
- *Degrease + HF acid dip + rinse (End User)*

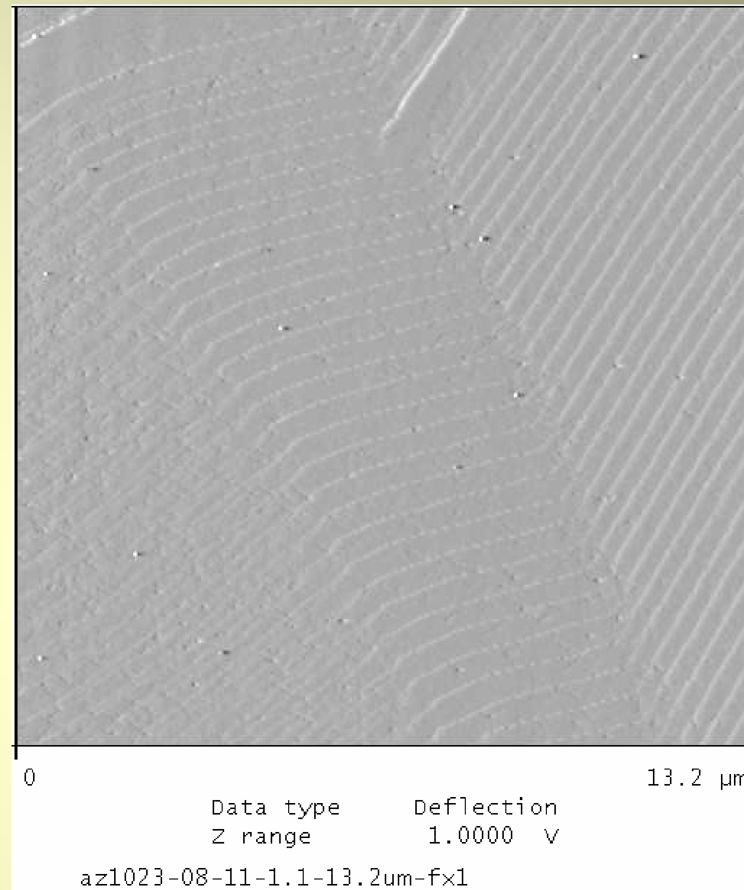
Method for Producing Atomically Flat Surfaces

- *Standard substrate preparation method*
- *Hydrogen etch (1600-1700 ^{o}C) + HF acid dip + rinse*


Flexible Stainless Steel Ruler

AFM Characterization





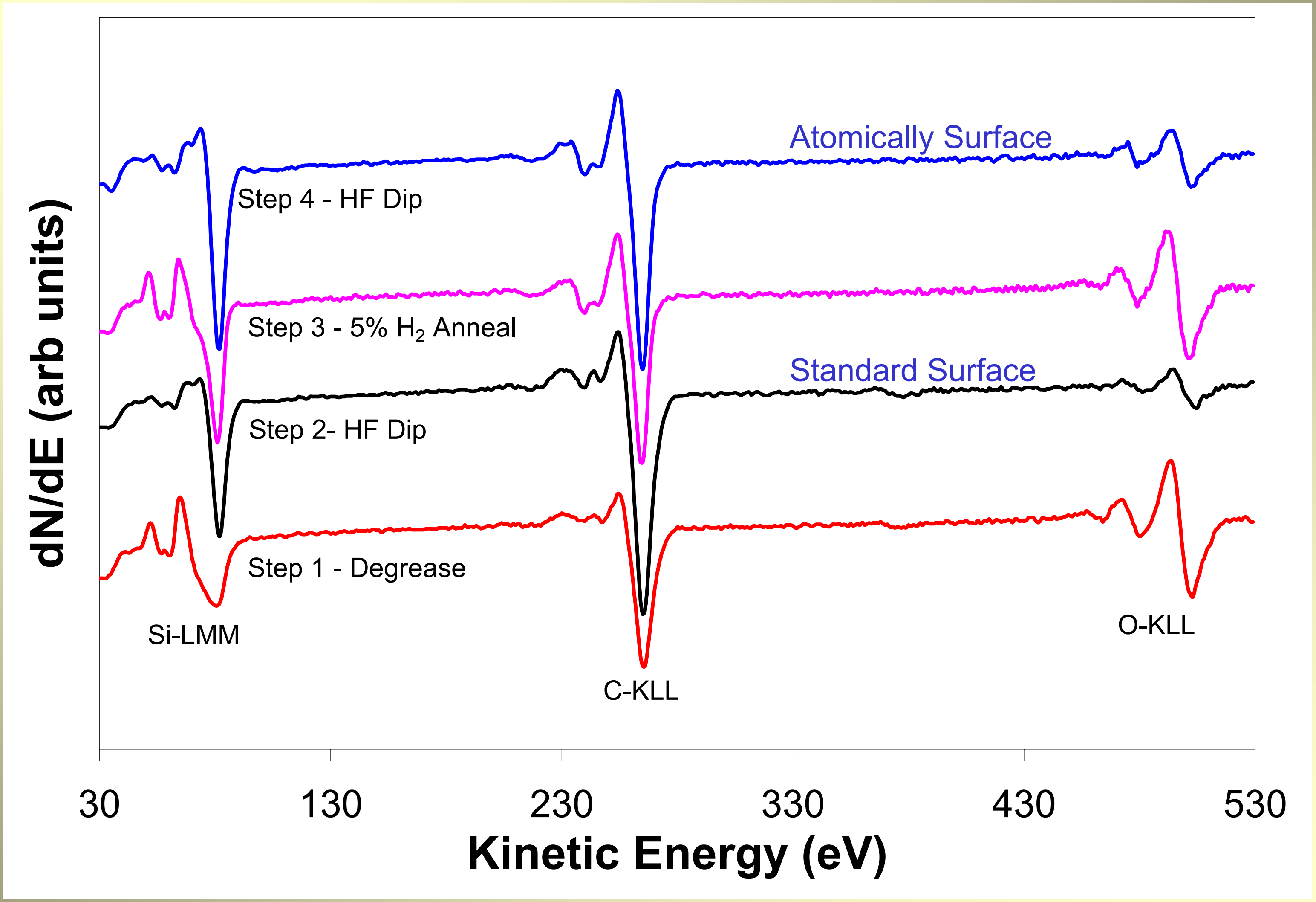
AFM of polished surface as received from vendor.

Deepest scratches ~ 20 nm deep and 0.25 µm wide.

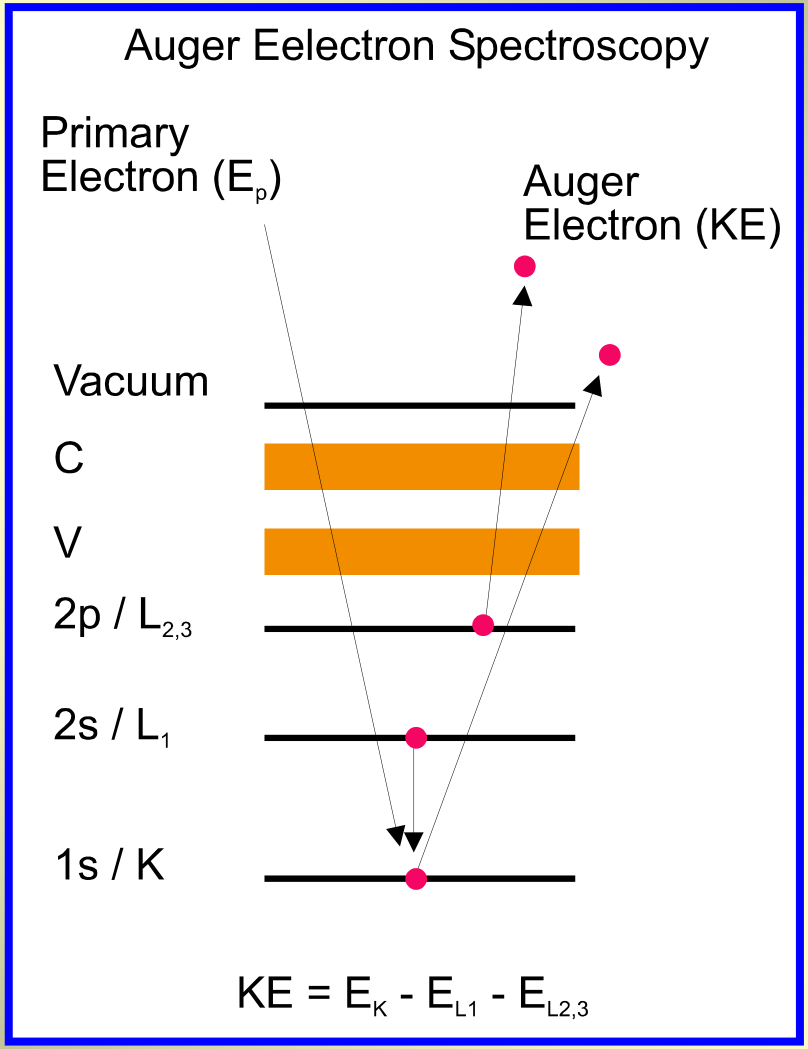
AFM of surface after hydrogen etching at 1600 °C.

Note the atomic steps and terraces at a grain boundary.

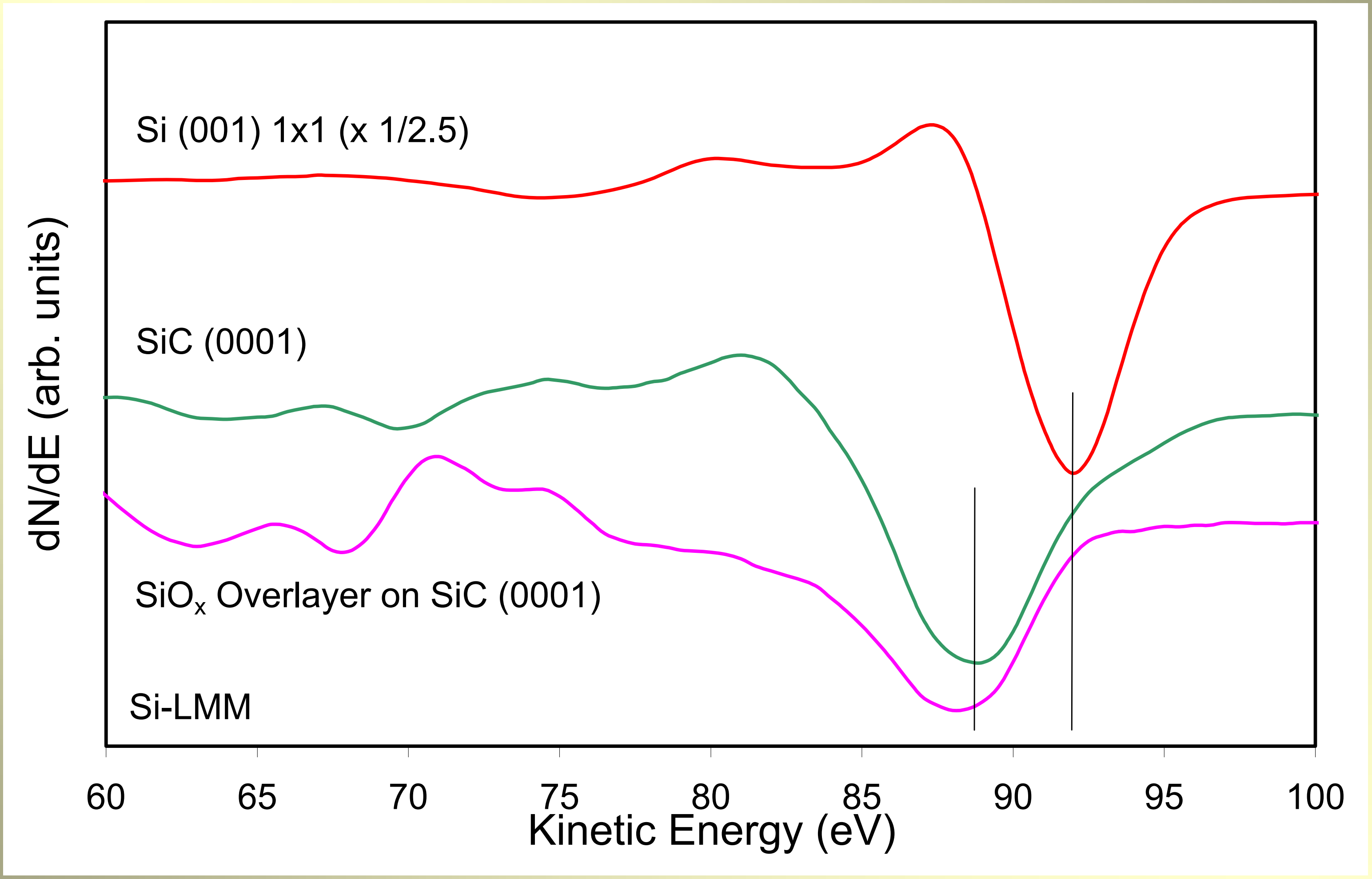
AES Characterization



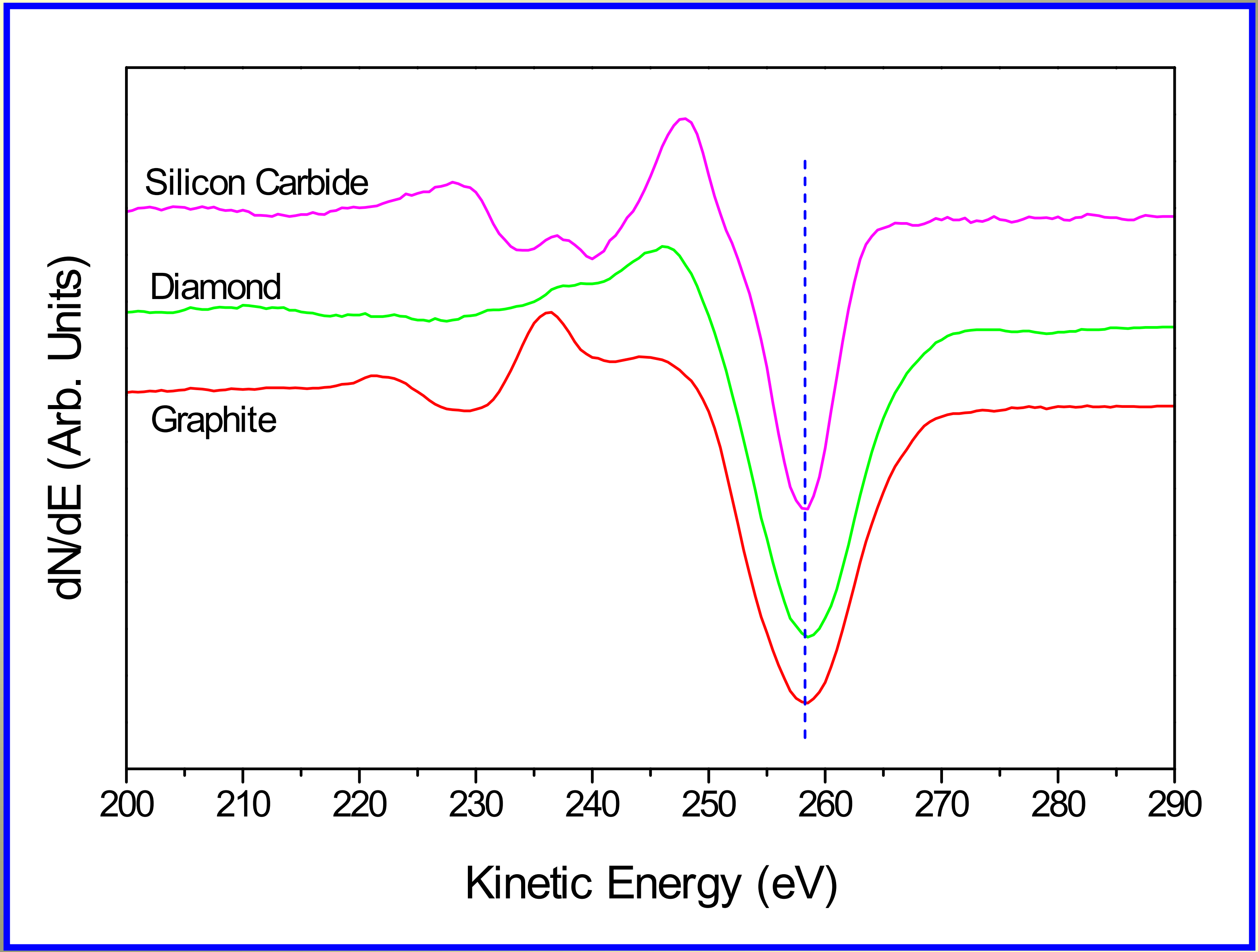
Auger Electron Spectroscopy (AES)



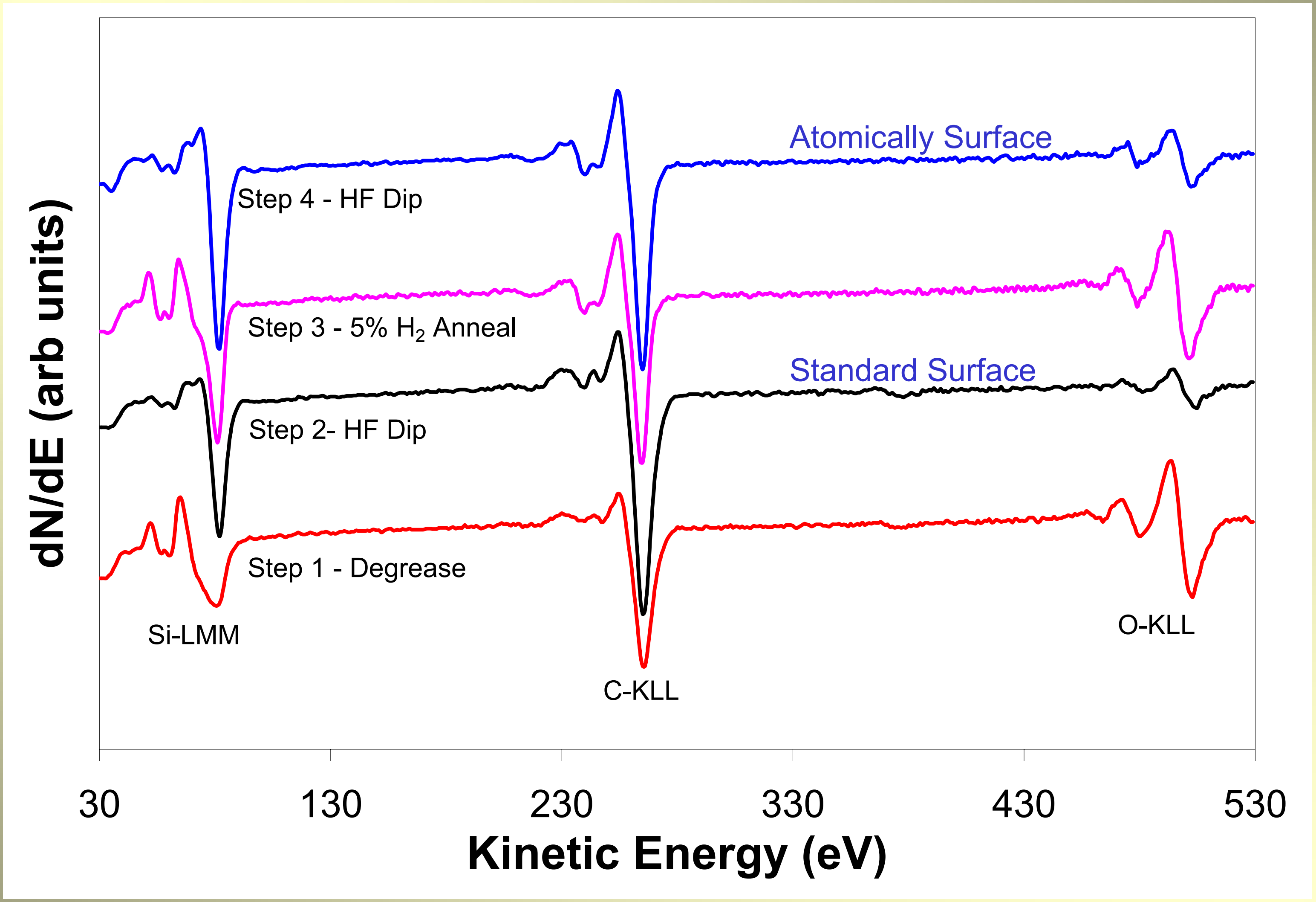
Si-Chemistry Based on Si-LMM AES



C-Chemistry Based on C-KLL AES



AES Characterization



Results

Preparation of Atomically Flat 6H-SiC

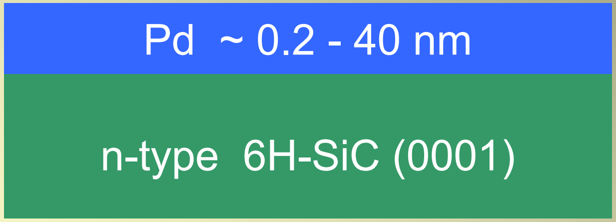
Deposition & Stability of Pd/SiC Interface ✓

Sensor Modeling & Characterization

Deposition and Stability of Pd on 6H-SiC

Substrates

-Commercial n-type 6H-SiC (0001)



Substrate Preparation

-Standard surface

-Atomically flat surface

Goal – *Compare the composition, structure, & thermal stability of thin Pd films deposited on atomically flat surfaces with those deposited on the standard surface*

Deposition Conditions

-Source material - 99.99 % pure Pd

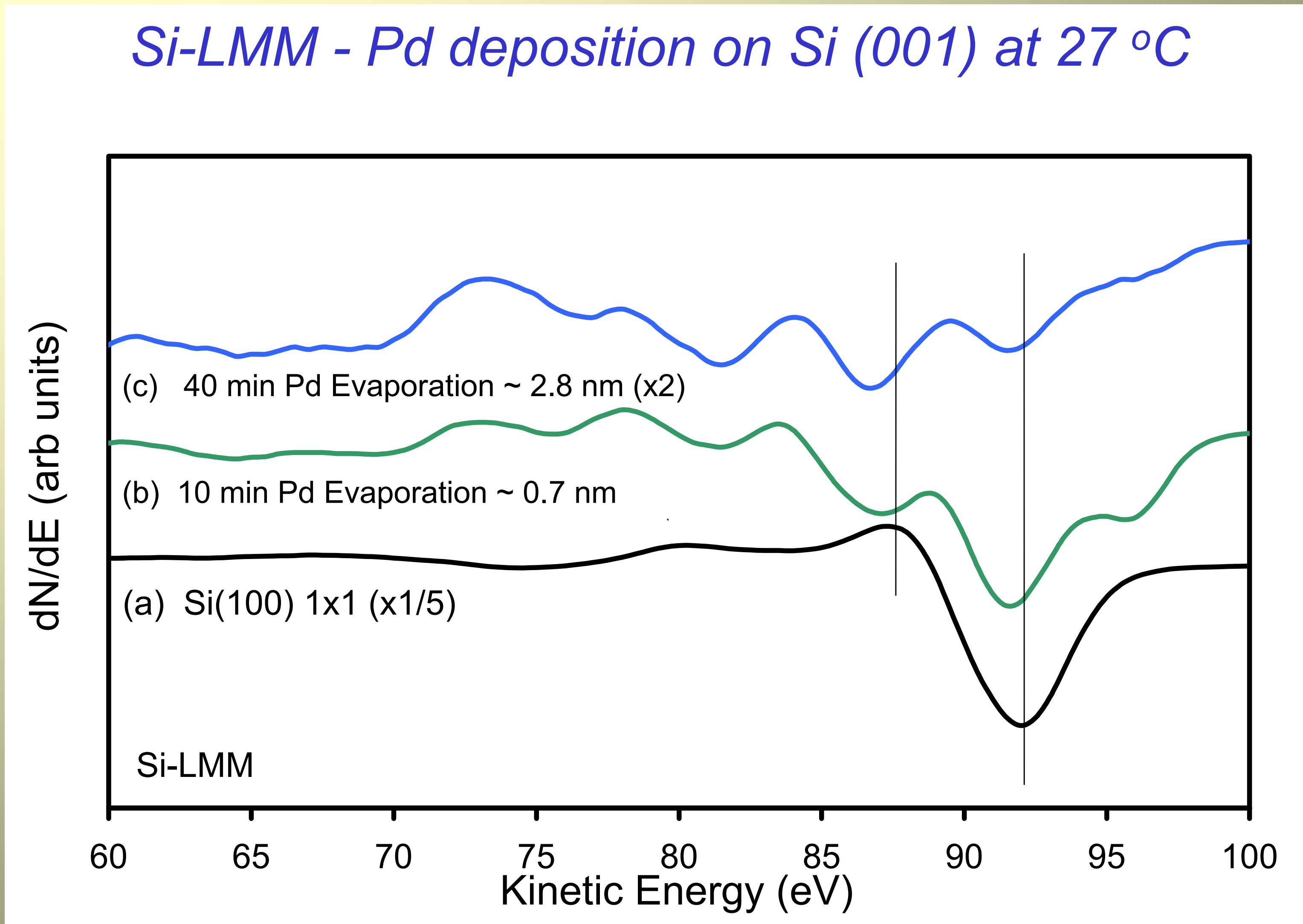
-Pd deposition rate - 0.5 – 5 ML/min (1 ML ~ 0.2 nm)

-Substrate temperature during deposition - 27 ^{o}C

-Pd/SiC annealed at 300 ^{o}C & 670 ^{o}C

-Chamber base pressure - 5x10^{-10} Torr

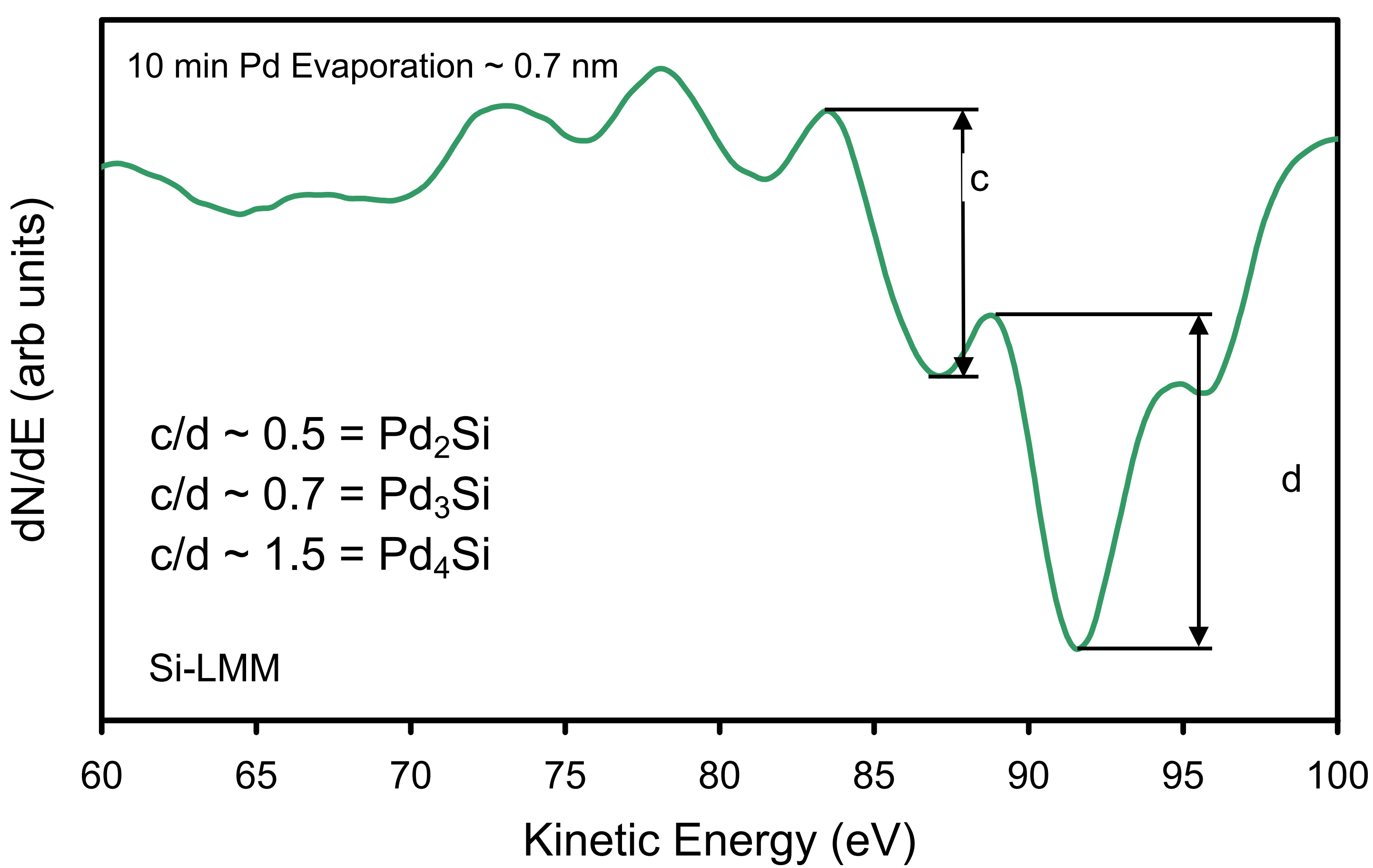
Pd-Si Chemistry Based on Si-LMM



Si & Pd-Si Chemistry Based on Si-LMM

Si-LMM for Pd_xSi

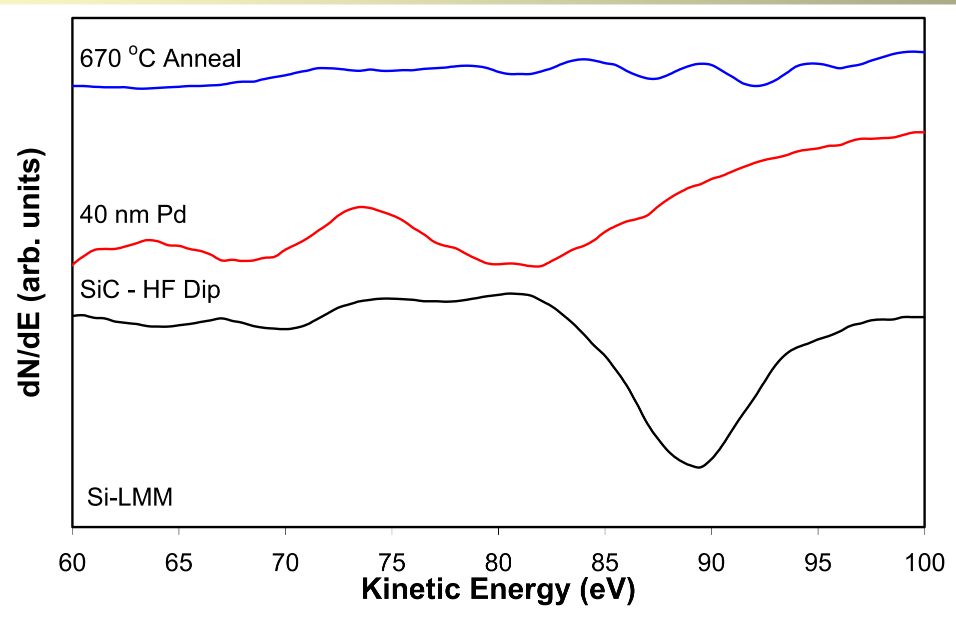
Bermudez, Appl. Surf. Sci. 17 (1983) 12-22



40 nm Pd on Standard 6H-SiC

40 nm Pd + Standard 6H-SiC at 27°C

-Pd overlayer obscures substrate Si-LMM peak



Anneal at 300°C

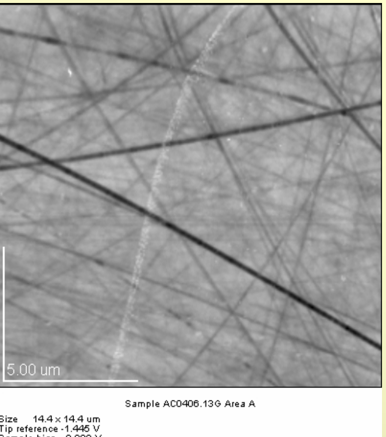
-No change in lineshape

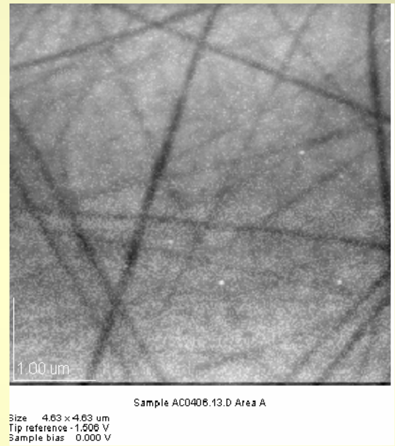
Anneal at 670°C

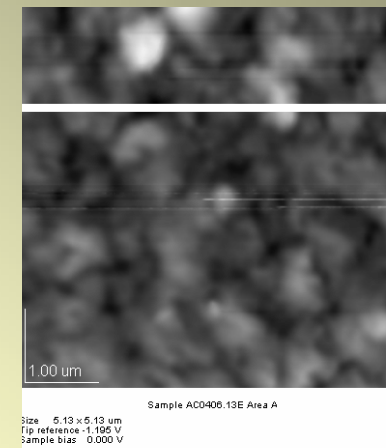
-Observe characteristic silicide lineshape

-Interdiffusion and reaction to form Pd_xSi (x>4)

40 nm Pd on Standard 6H-SiC







Standard Surface

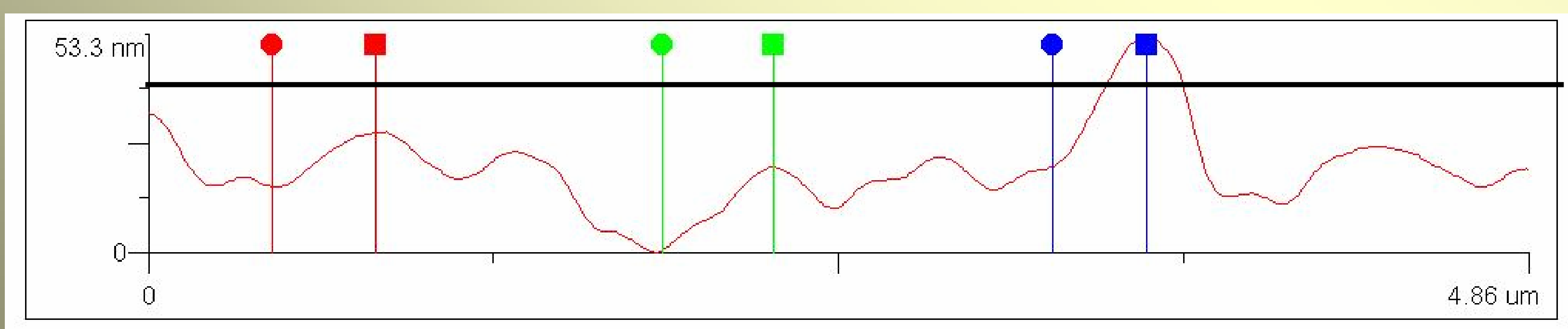
RMS = 1.7 nm

+ 40 nm Pd

RMS = 2.3 nm

+ anneal 670 °C

RMS = 8.9 nm



0.4 nm Pd on Standard 6H-SiC

0.4 nm Pd + Standard 6H-SiC at 27°C

-Pd overlayer attenuates substrate Si-LMM peak

-No reaction to between Pd-Si

-Indicates low interfacial reactivity & mobility of reactants

Annealing at 300°C

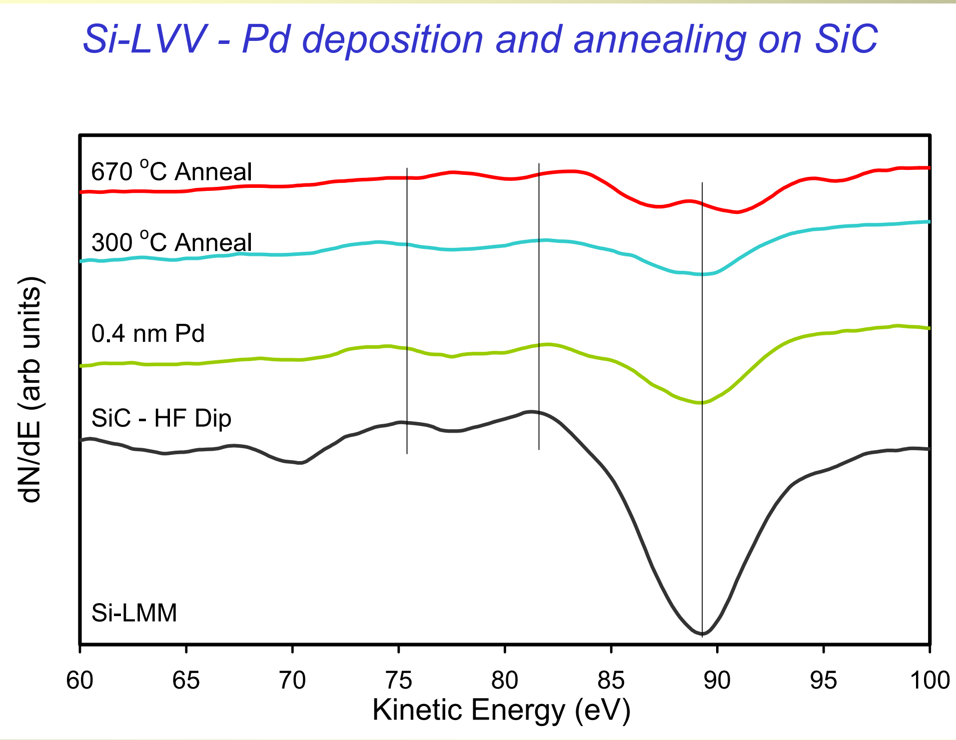
-No reaction between Pd-Si

Annealing at 670°C

-Pd_xSi (x>4) formed

AFM Characterization

-Island formation

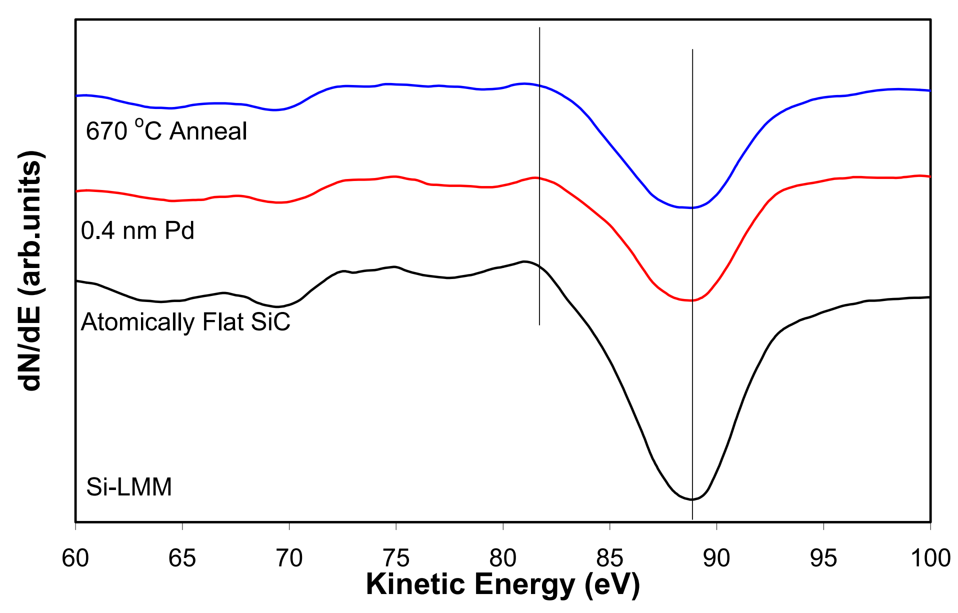


0.4 nm Pd on Atomically Flat 6H-SiC

0.4 nm Pd + Atomically Flat 6H-SiC at 27°C

-No reaction to form Pd_xSi

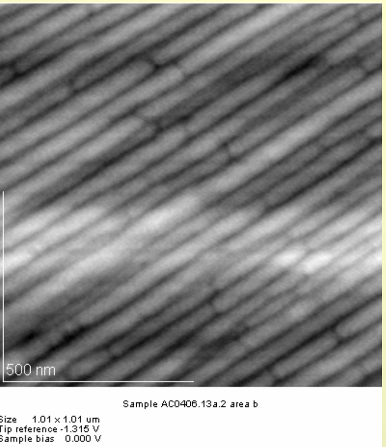
-Indicates low interfacial reactivity & mobility of reactants

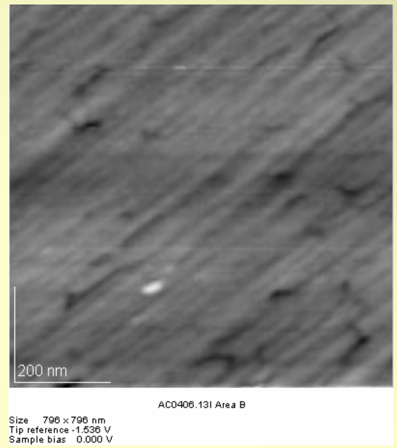


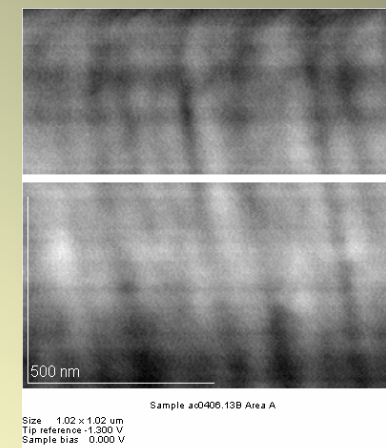
Anneal at 670°C

-No reaction to form Pd_xSi

0.4 nm Pd on Atomically Flat 6H-SiC







+ H_2 Anneal

+ HF Dip

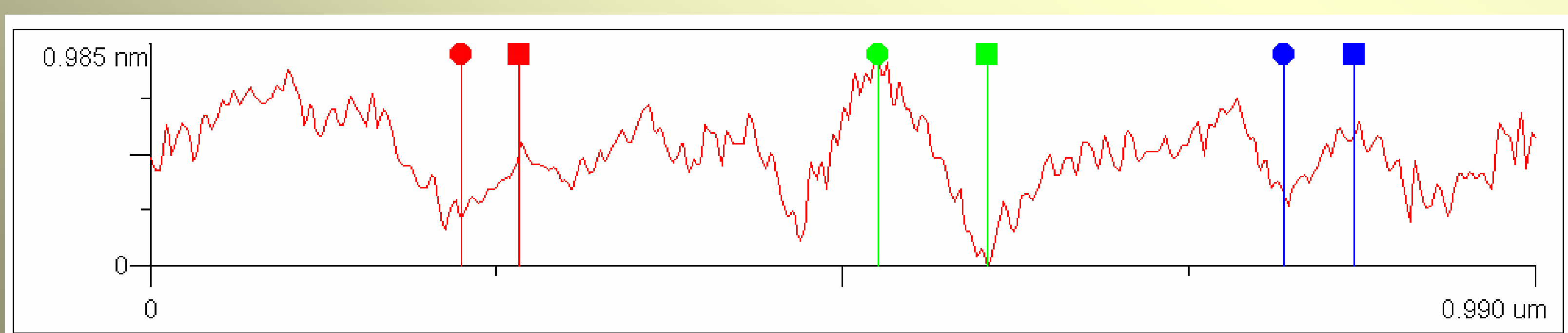
RMS = 0.7 nm

+ 0.4 nm Pd

RMS = 0.5 nm

+ anneal 670 ^oC

RMS = 0.4 nm



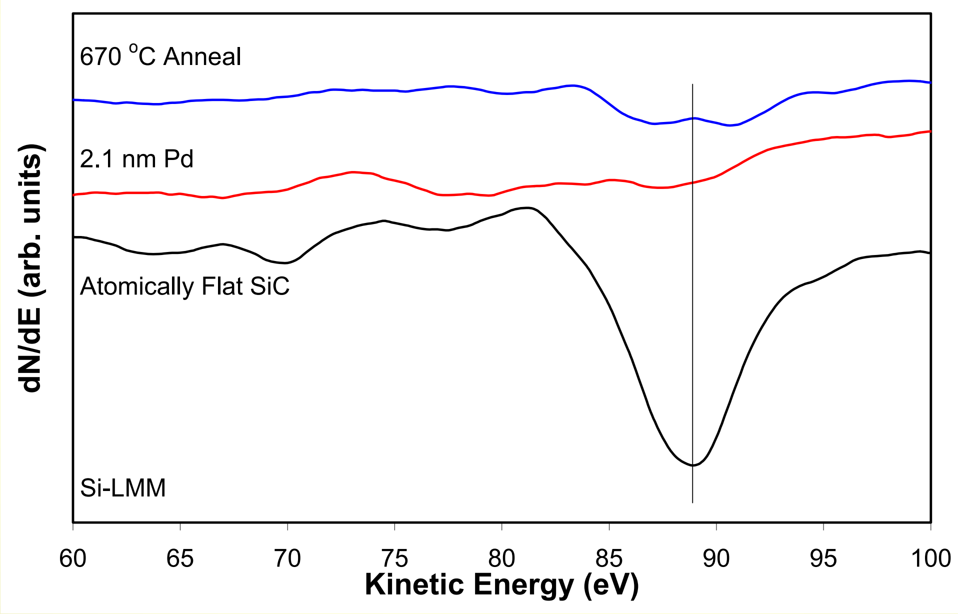
2.1 nm Pd on Atomically Flat 6H-SiC

2.1 nm Pd + Atomically Flat 6H-SiC at 27°C

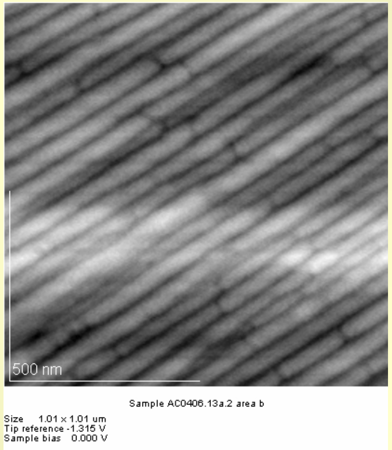
- *Pd overlayer attenuates/obscures substrate Si-LMM peak*

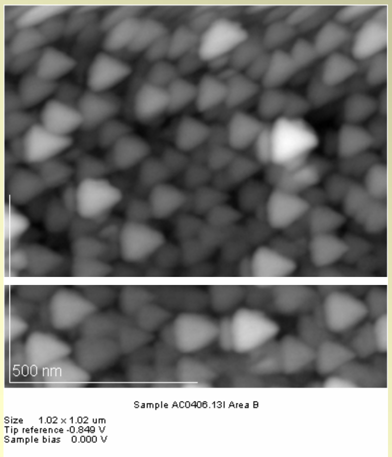
Anneal at 670°C

- *Reaction to form Pd_xSi (x >4)*



2.1 nm Pd on Atomically Flat 6H-SiC





+ H_2 Anneal

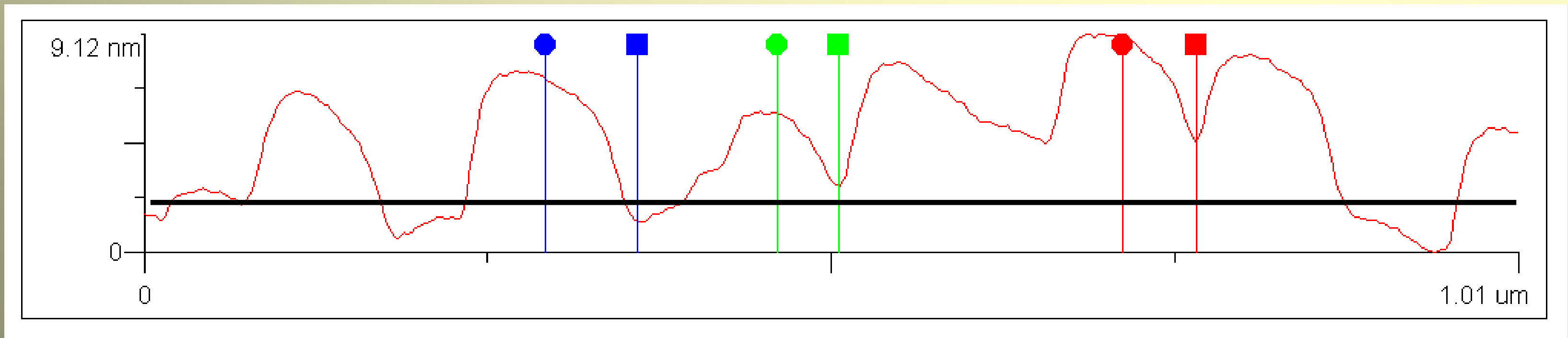
+ HF Dip

RMS = 0.7 nm p

+ 2.1 nm Pd

+ Anneal 670 ^{o}C

RMS = 2.9 nm



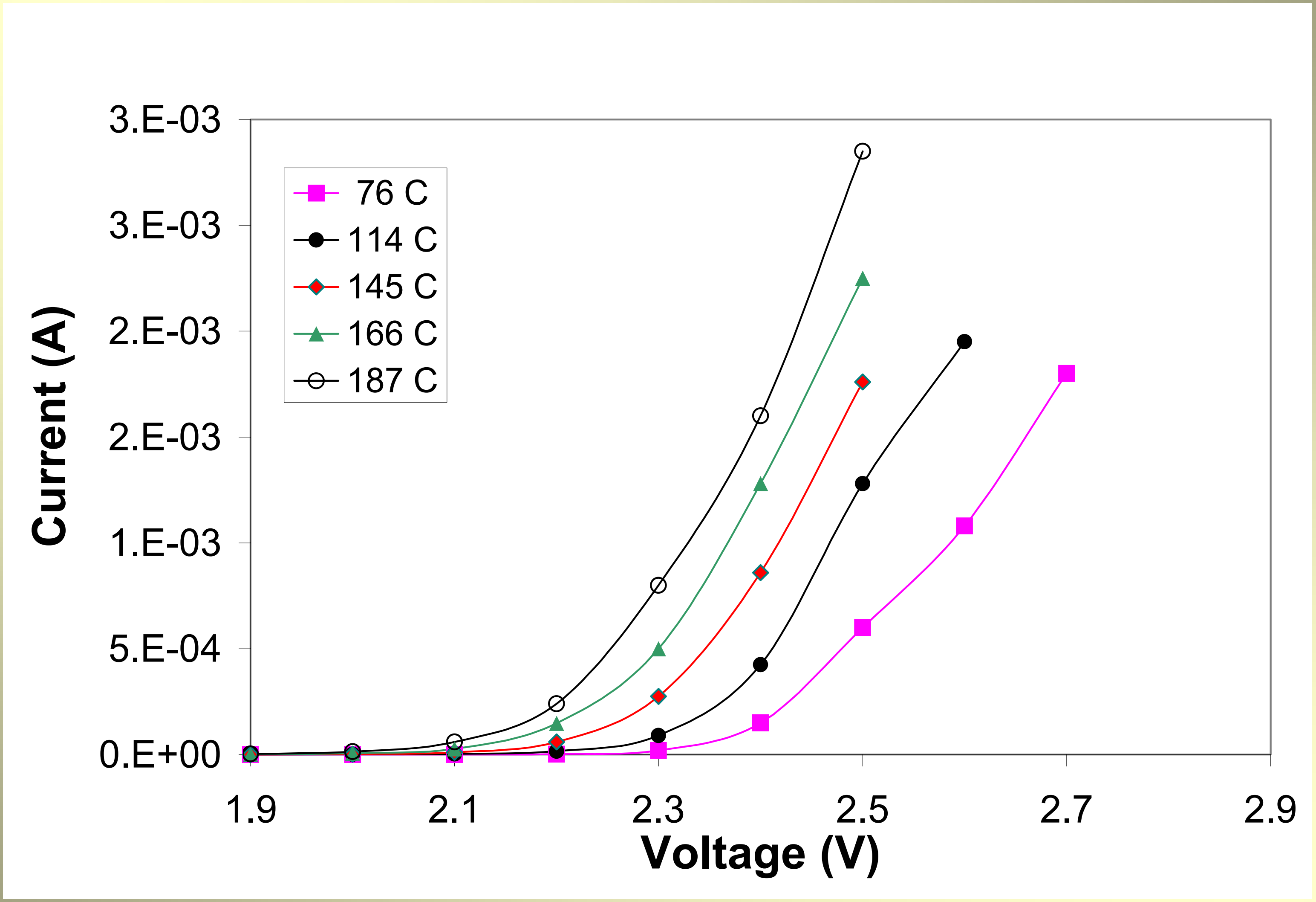
Results

Preparation of Atomically Flat 6H-SiC

Deposition & Stability of Pd/SiC Interface

Sensor Modeling & Characterization ✓

SiC p-n Junction Characterization



Project Status

Device Fabrication

- *Reproducibly produced atomically flat SiC surfaces*
- *Produced Pd/SiC interfaces stable to at least 670 ^{o}C*
- *Fabricated Pd/SiC diode structures for electrical characterization & testing*

Device Characterization

- *Characterized SiC p-n junctions for temperature sensor component*
- *Characterization of Pd/SiC diodes in progress*

Future Work

Device Fabrication

- *Determine the upper temperature limit for stability of 0.2nm Pd films*
- *Further characterize the thickness dependence of thermal stability*
- *Investigate epitaxy of Pd and Pd_xSi films on these films*
- *Fabrication of integrated gas / temperature sensor structures*

Device Modeling and Characterization

- *High temperature characterization of diode structures*
- *Integrated gas / temperature sensor characterization*