



Precursor-Derived Nanostructured SiC-Based Materials for MHD Electrode Applications

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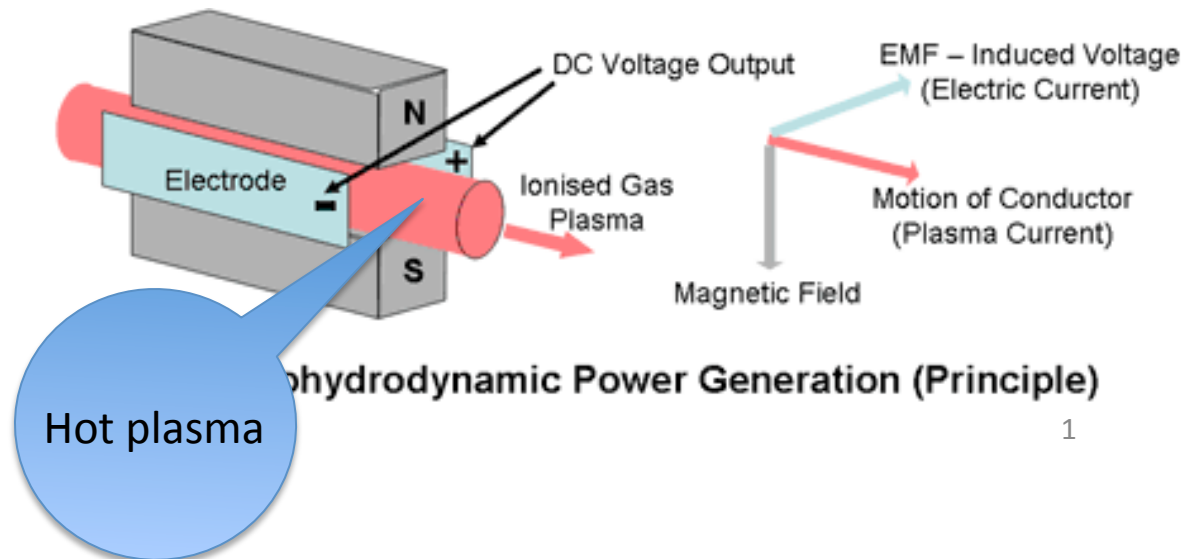


Grant No. DE-FE0023142
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Major Obstacle in MHD Power Generation

Issues in electrode materials

- Must withstand in harsh MHD environments
 - Extremely high temperature
 - Exposure to hot plasma
 - Severe ion bombardment
 - Mechanical stress via thermal expansion
 - Thermal, electrical, mechanical and chemical stability of materials for all above.
- Conformal process
- Low resistivity



Long Term Goal

To develop a novel class of SiC based ceramic composite materials with tailored compositions for channel applications in MHD generators.

Our Focus

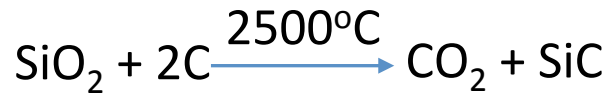
Controlling and understanding the effect of the nature of the excess carbon on the structural and electrical properties.

Role of excess carbon

- ✓ Polymer based synthesis
- ✓ Structural information
- ✓ Electrical resistivity
- ✓ Work function from thermionic emission

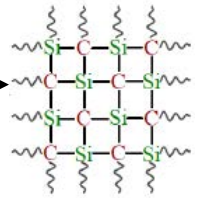
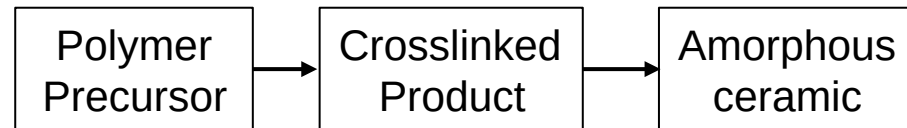
Process of Making Silicon Carbide

- Conventional method (Acheson method):



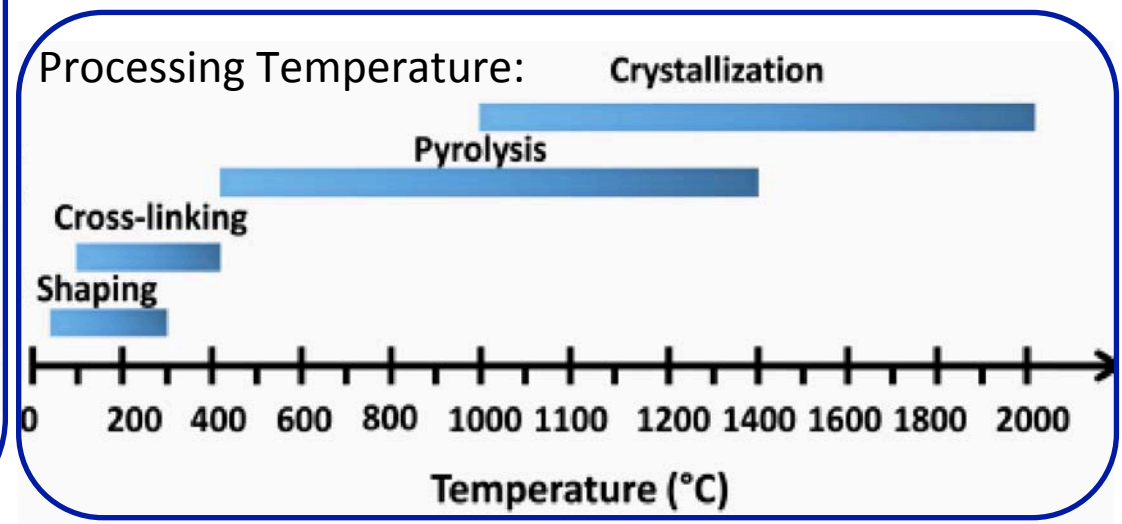
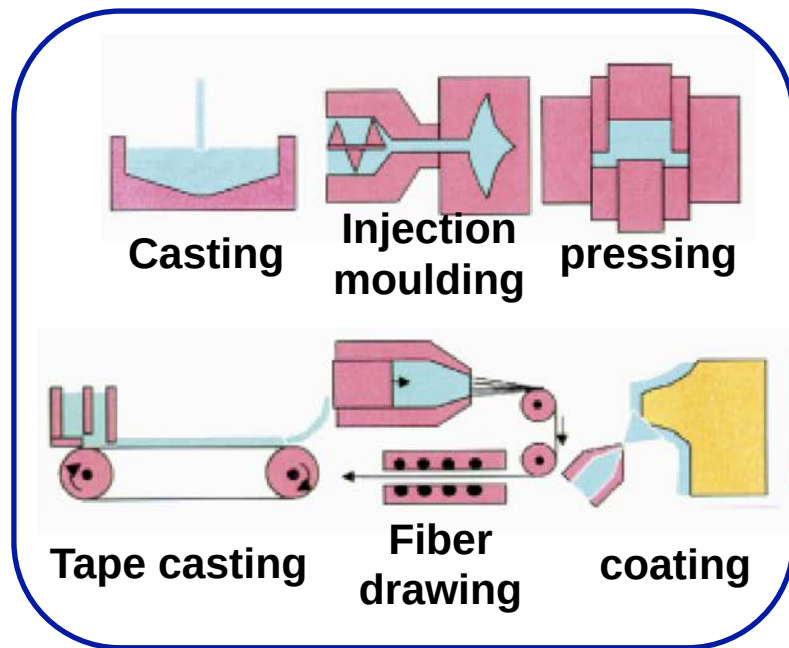
- High processing T
- Difficult to dope uniformly

- Polymer Derived Process:



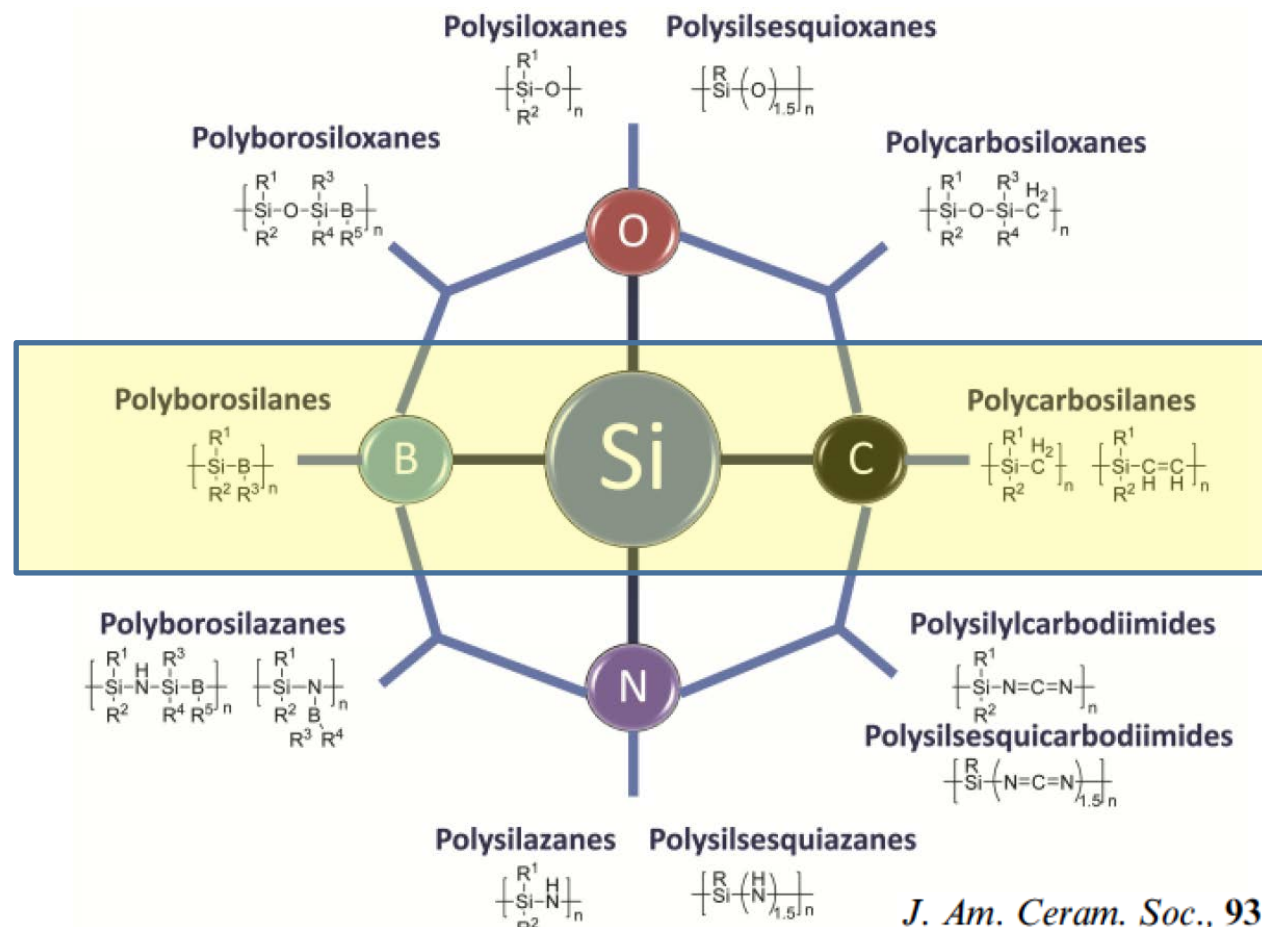
1. Conformal process
2. Possible low processing temperature
3. Uniform composition
4. Versatile choice of dopant

- Chemical Vapor Deposition

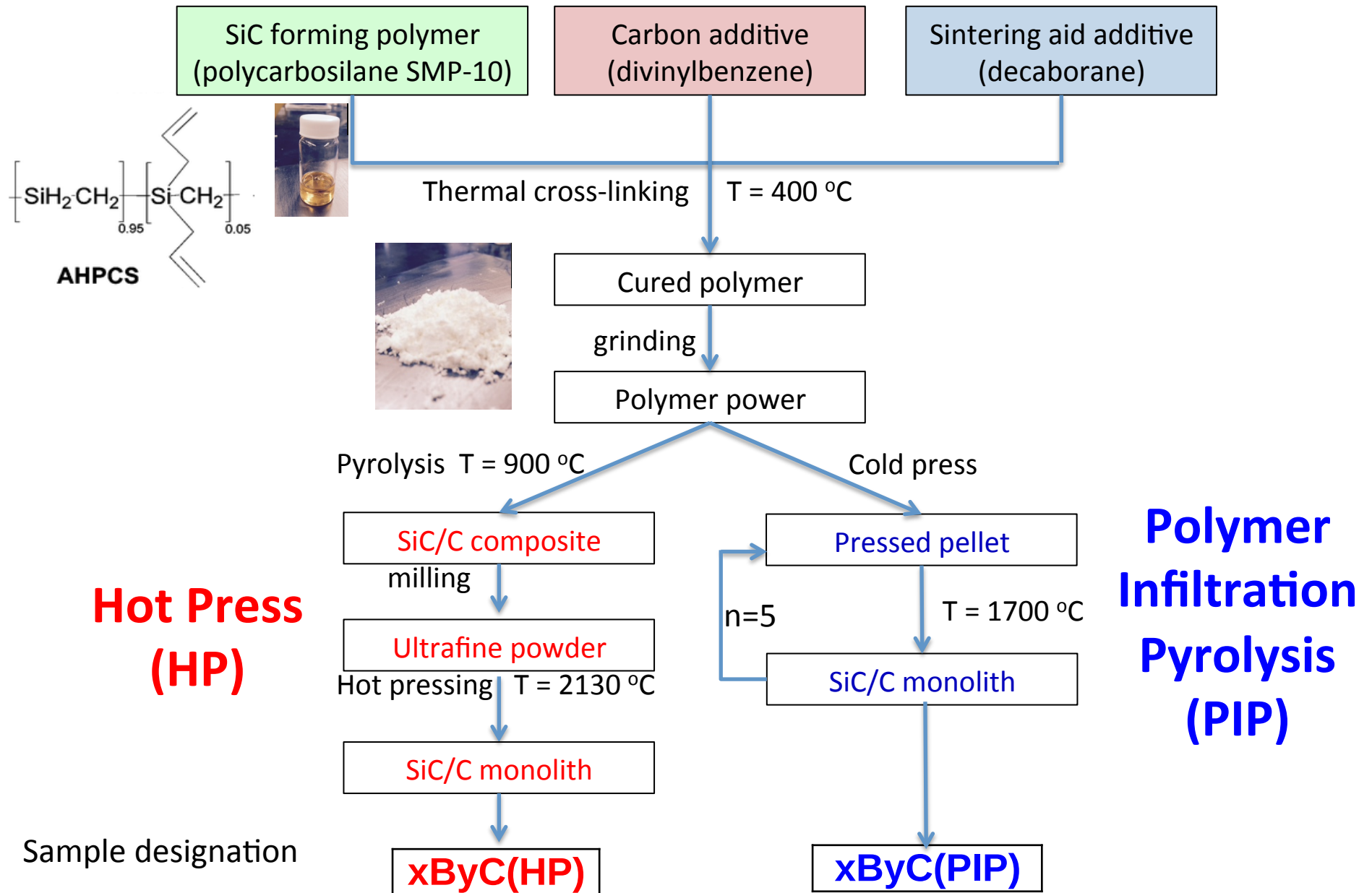


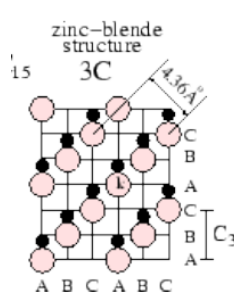
Diversity of Silicon Based Precursors

- Liquid form polymer can be mixed homogeneously
- Silicon / carbide ratio could be adjusted through precursors
- Boron, nitrogen, oxygen and other element like K can be incorporated
- Relatively low process temperature



Selection of Polymer and Processing





C:Si Atomic Ratio for SiC-C

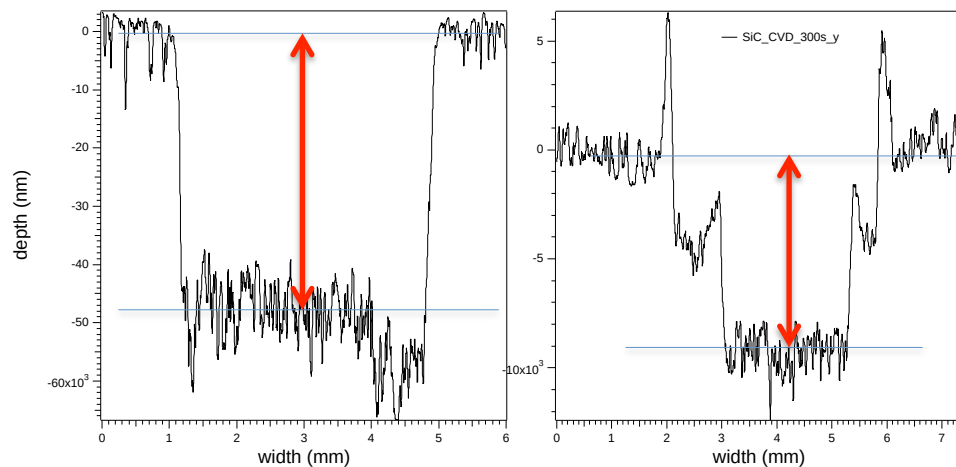
xByC(HP) vs xByC(PIP)

Glow Discharge Optical Emission Spectrometer

Horiba GD-Profilier-2

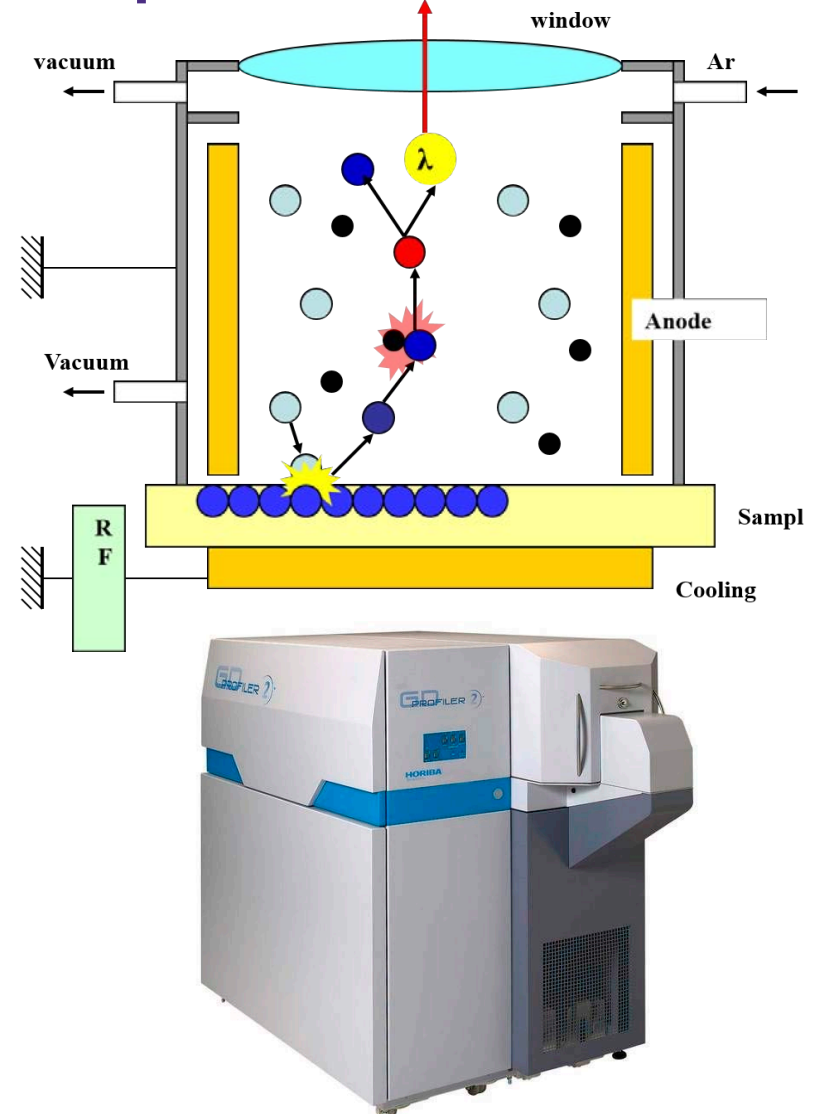
- Depth-resolved elemental analysis
- with ~10 nm depth resolution
- Semi-quantitative analysis

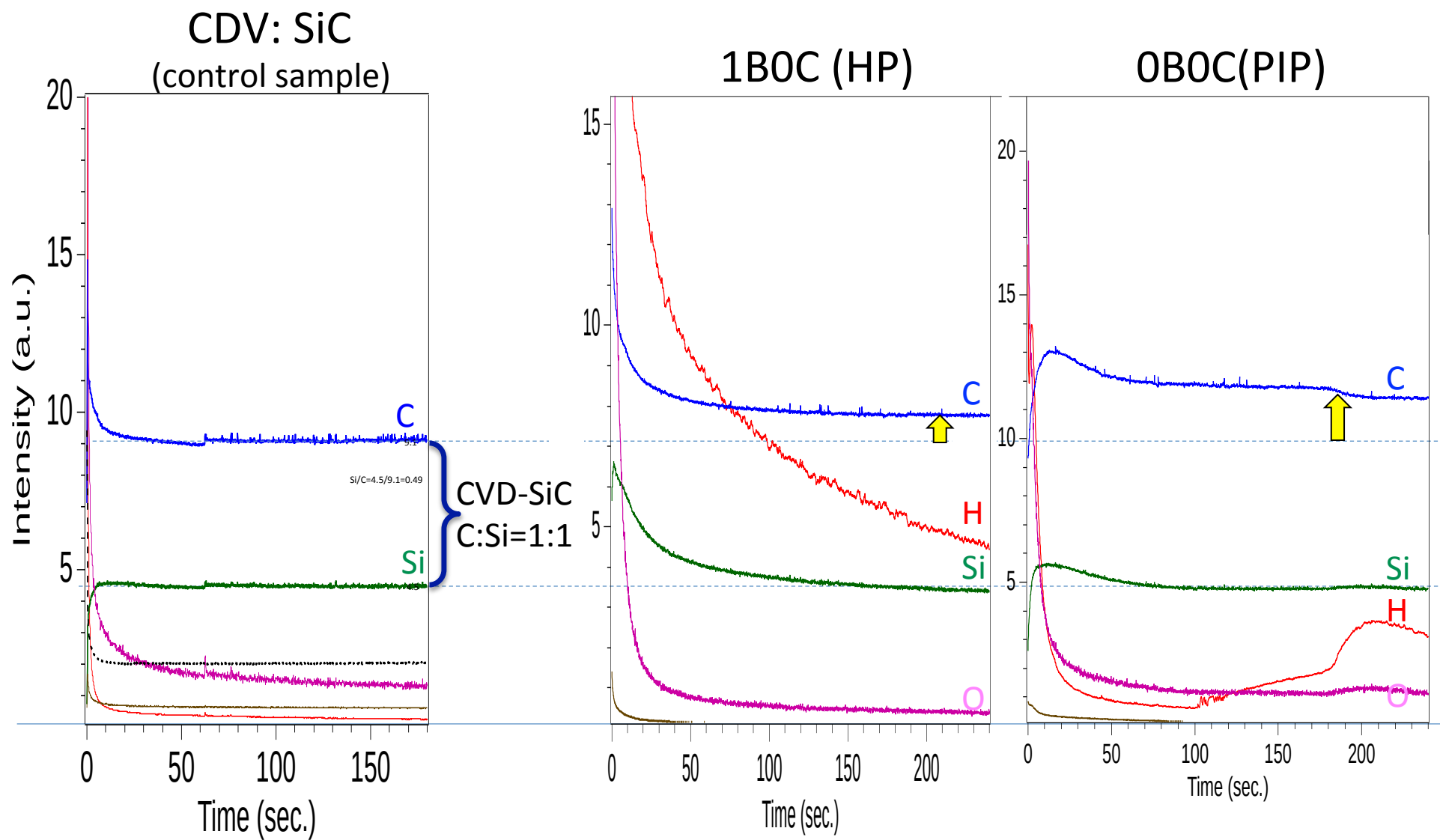
Sputter-Depth Calibration nm/sec

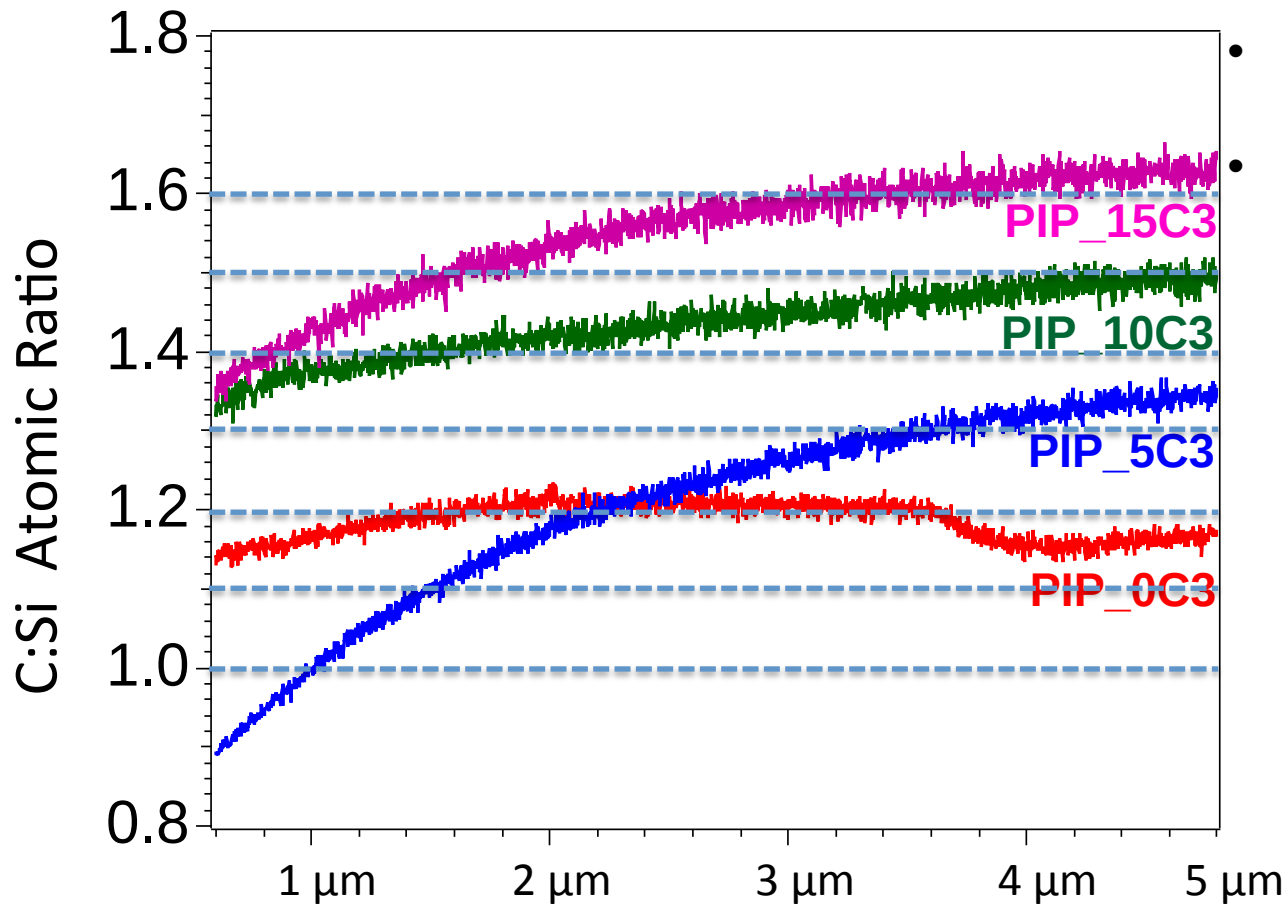


Graphite, 300s, ~50 μm

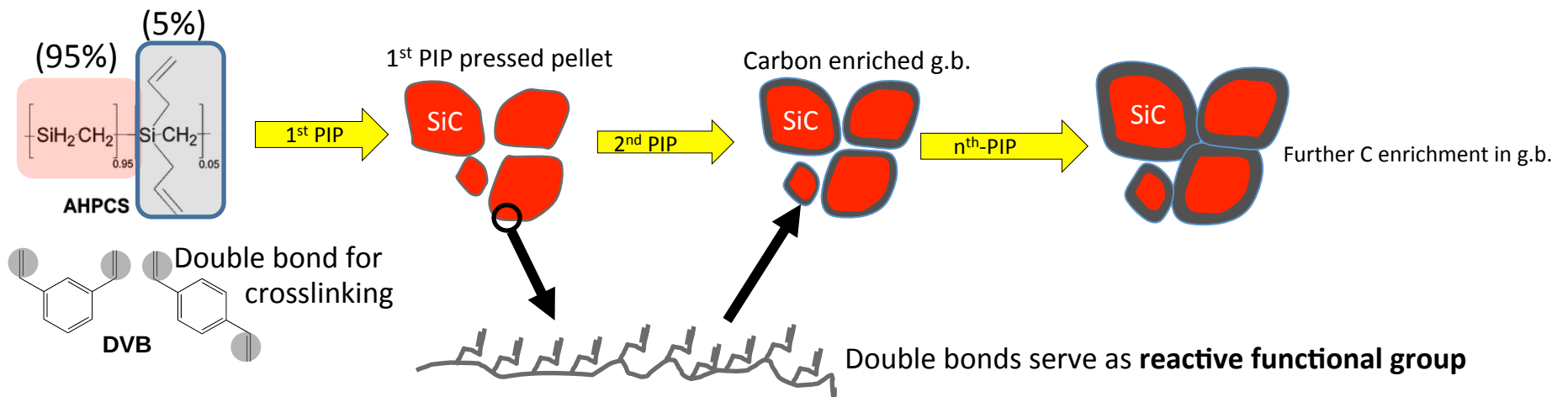
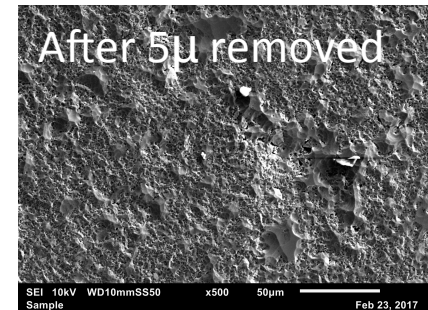
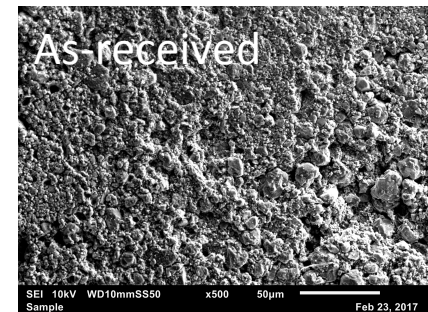
CVD-SiC, 300s, ~5 μm





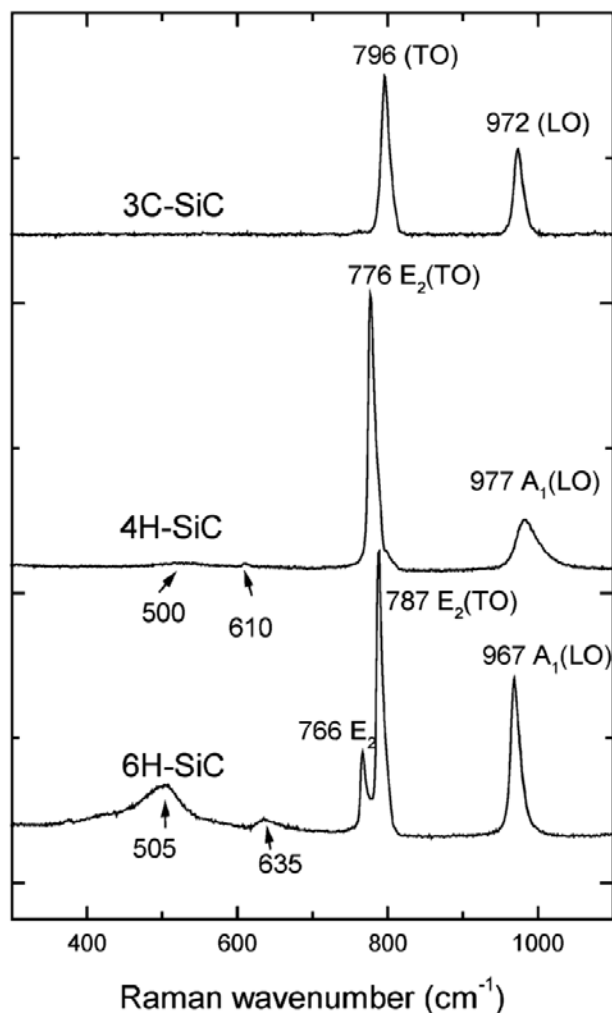


- Excess amount of carbon layers created by PIP
- Sputtering rate for C(graphite) ~ 10 times higher than SiC(3C)

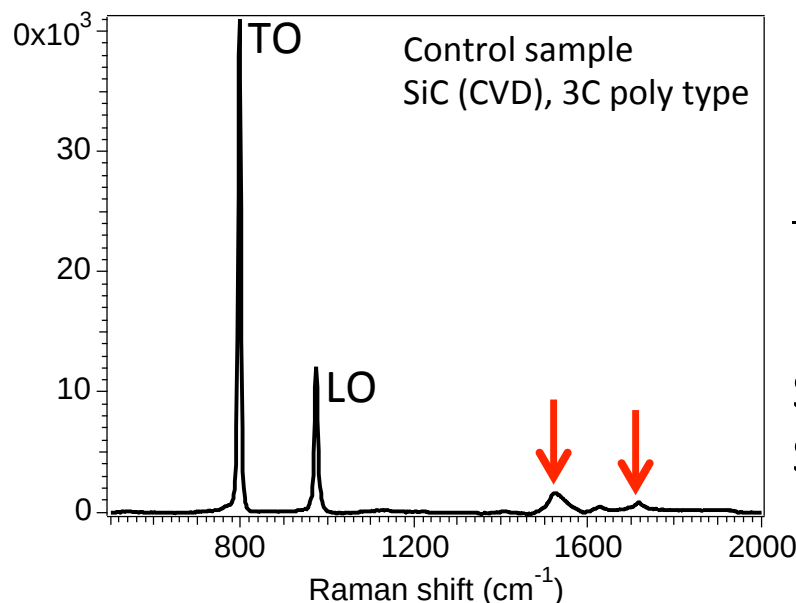


Molecular Structure of SiC-C by Raman

Excitation: Ar laser (514nm)



Y. Ward, R. J. Young, and R. A. Shatwell,
J. Mater. Sci. **39**, 6781 (2004).



Penetration depth of
514nm laser light

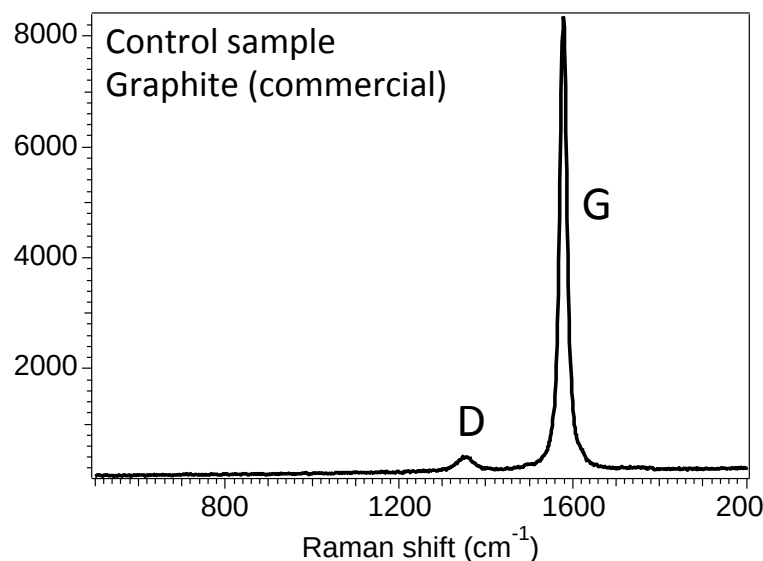
SiC(3C)~100 μ

SiC(6H)~300 μ

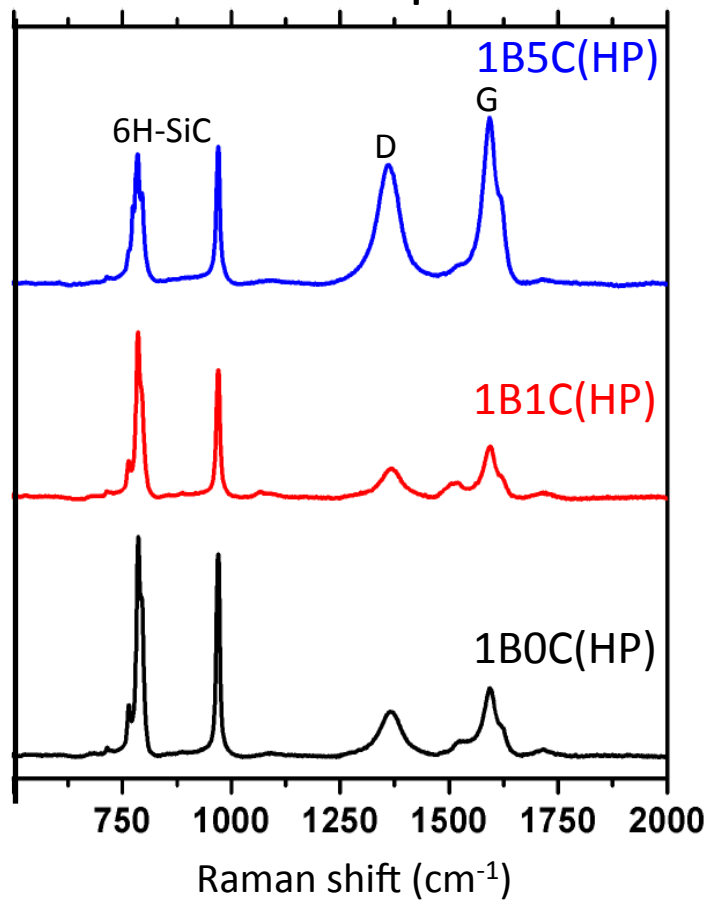
Probing bulk



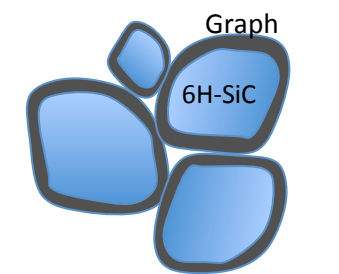
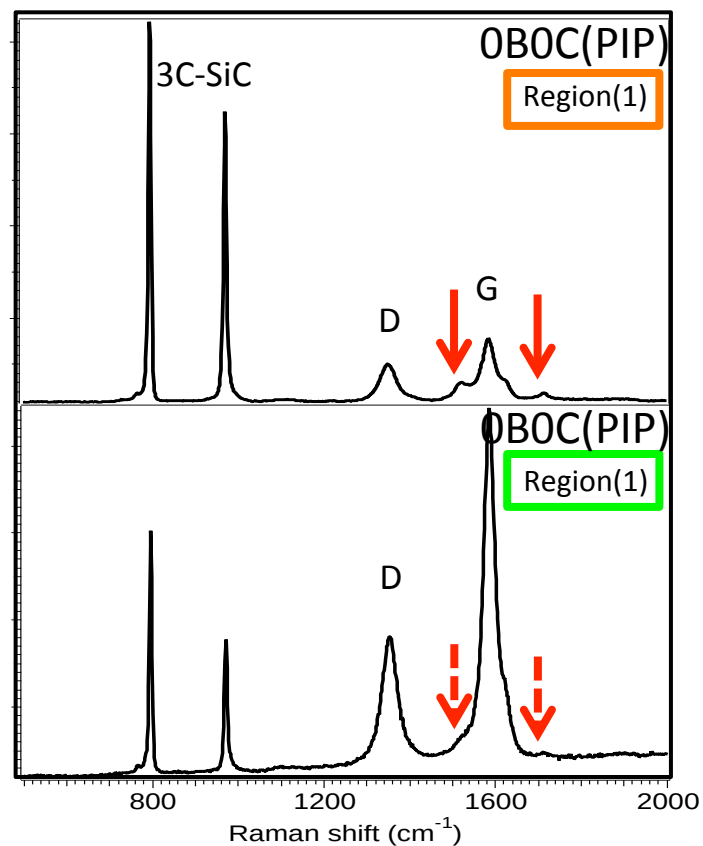
C(graph)~100-200nm
Probing surface



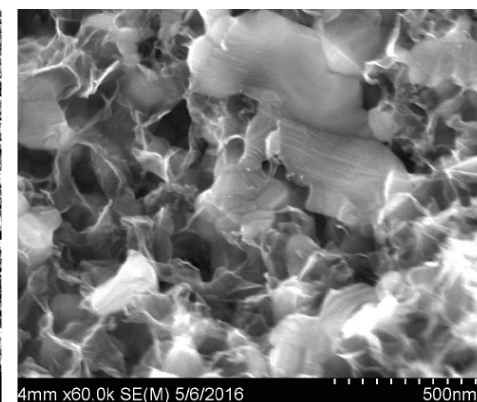
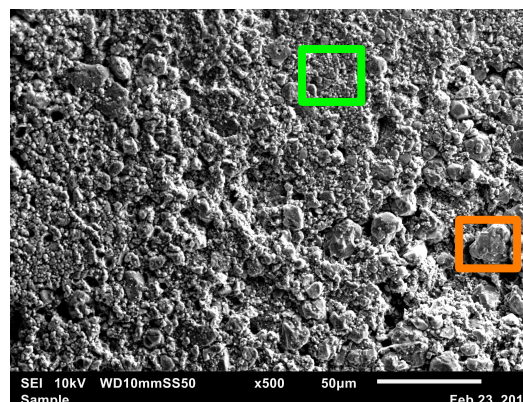
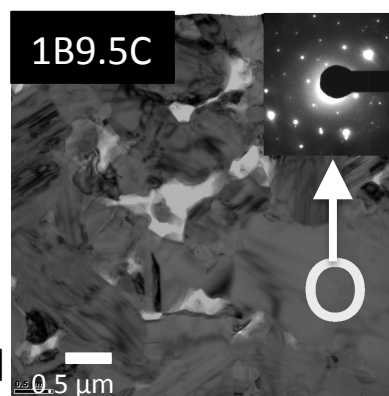
HP-samples



PIP-samples

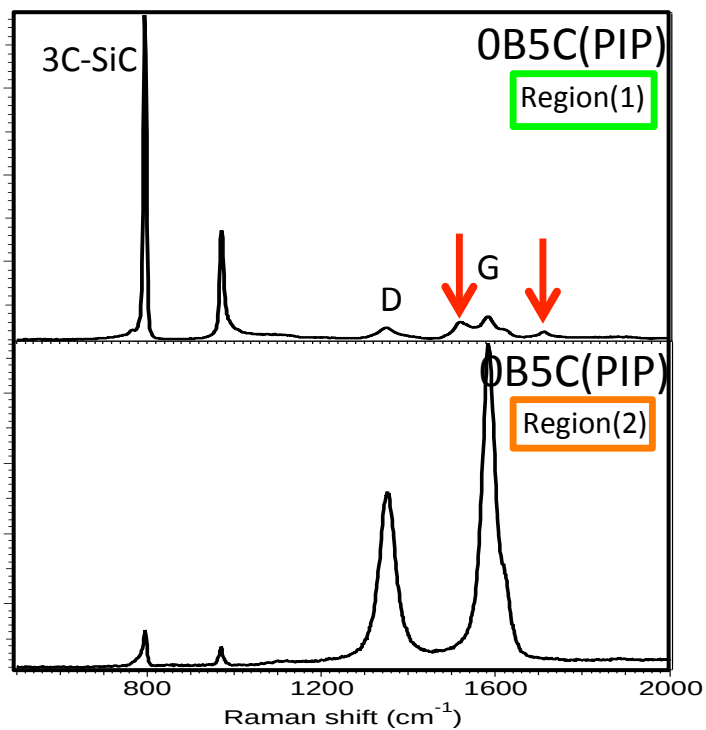


Structural model

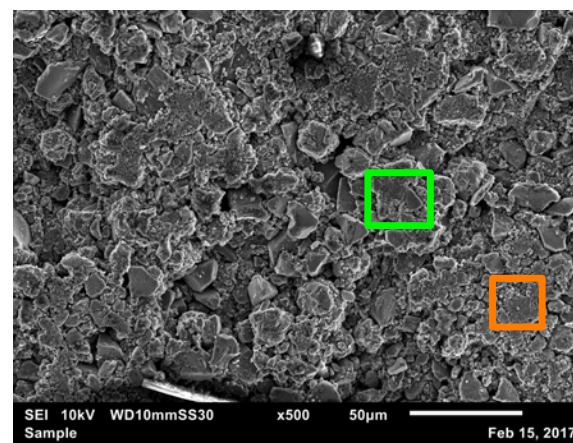
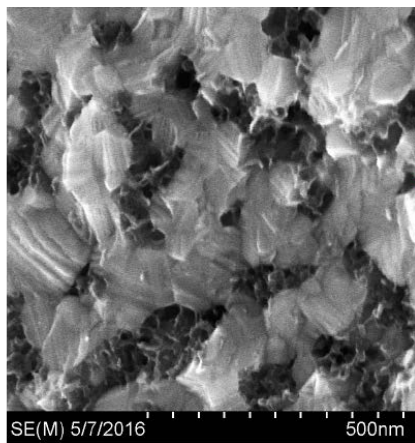
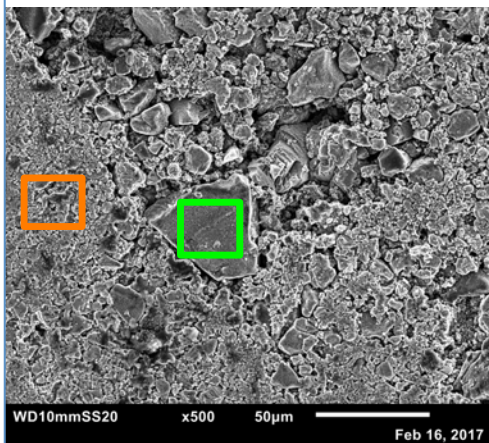
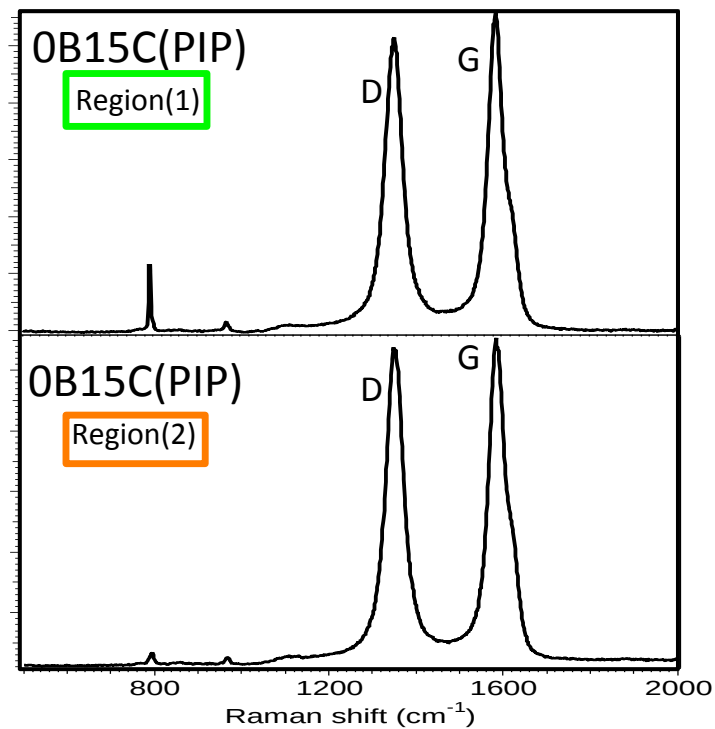


PIP-samples (cont'd)

OB5C(PIP)

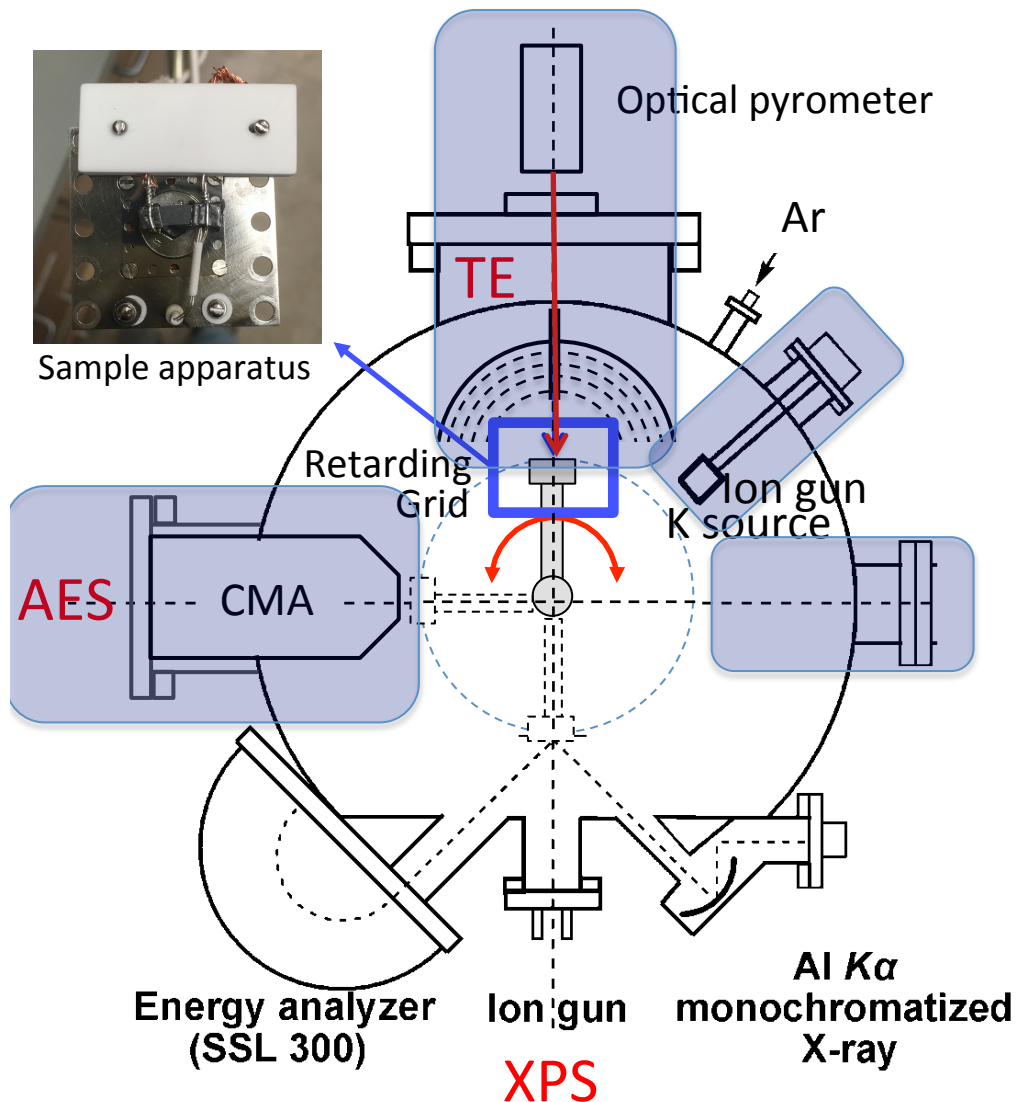


OB15C(PIP)



Temperature Induced Surface Change of SiC-C

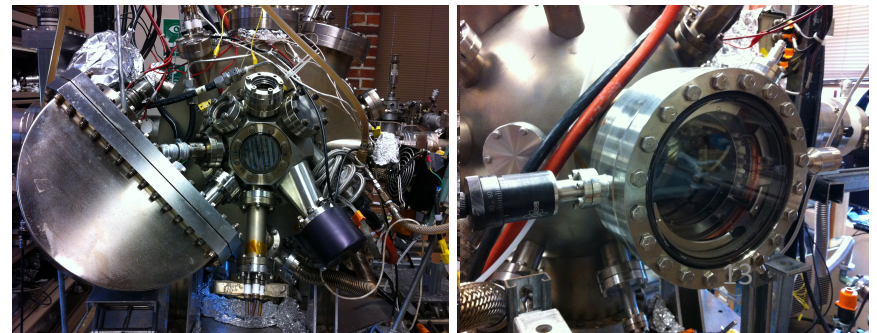
Integrated Experimental System:



- Retarding grid spectroscopy to measure thermionic emission (TE)
- X-ray photoelectron spectroscopy (XPS)
- Auger electron spectroscopy (AES)
- K deposition for work function engineering
- R-type TC and optical pyrometer for temperature measurement
- Ion gun for surface cleaning

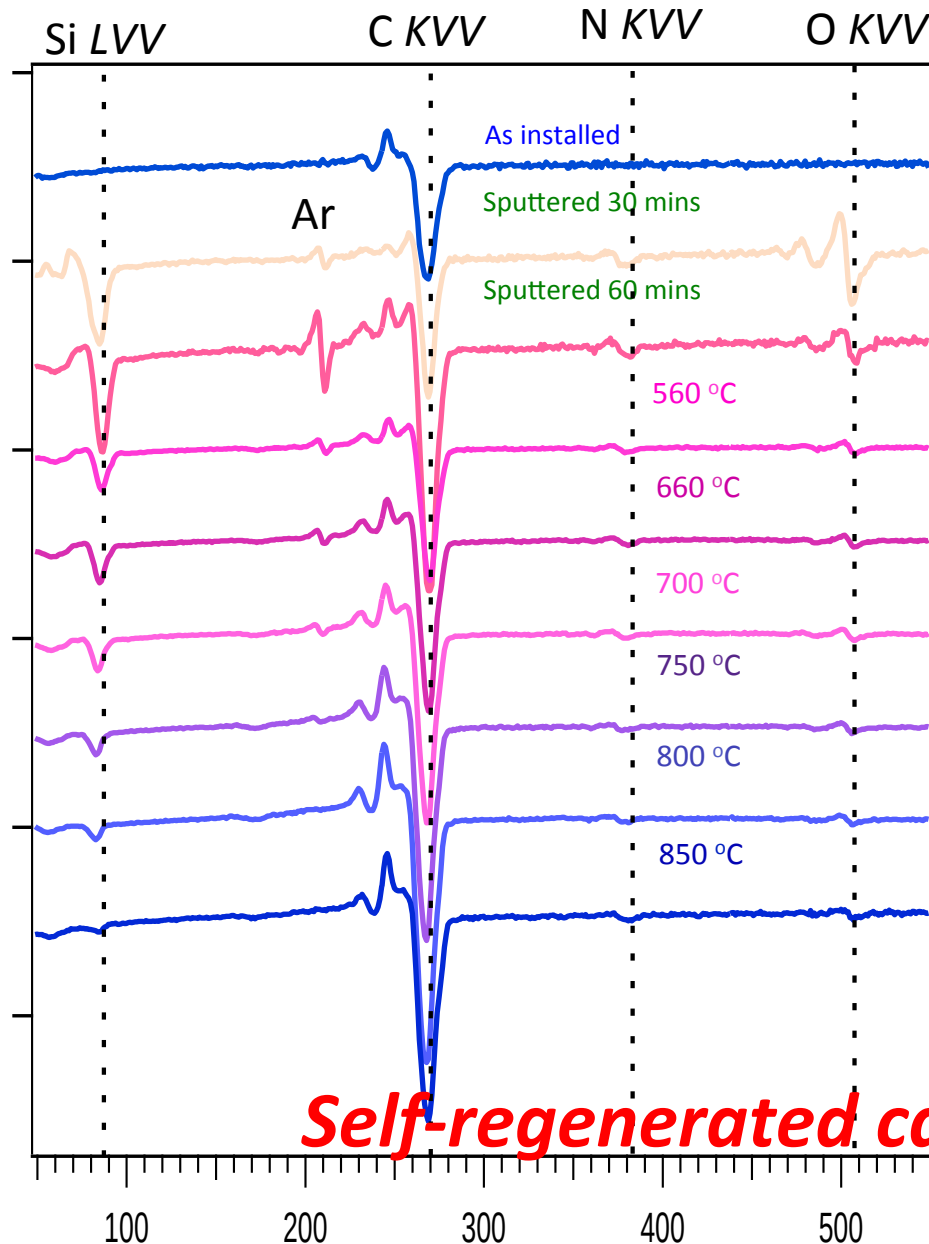
Capable of measuring:

- Surface composition at elevated temp.
- Total current and kinetic energy distribution of thermionic emission.
- Work function



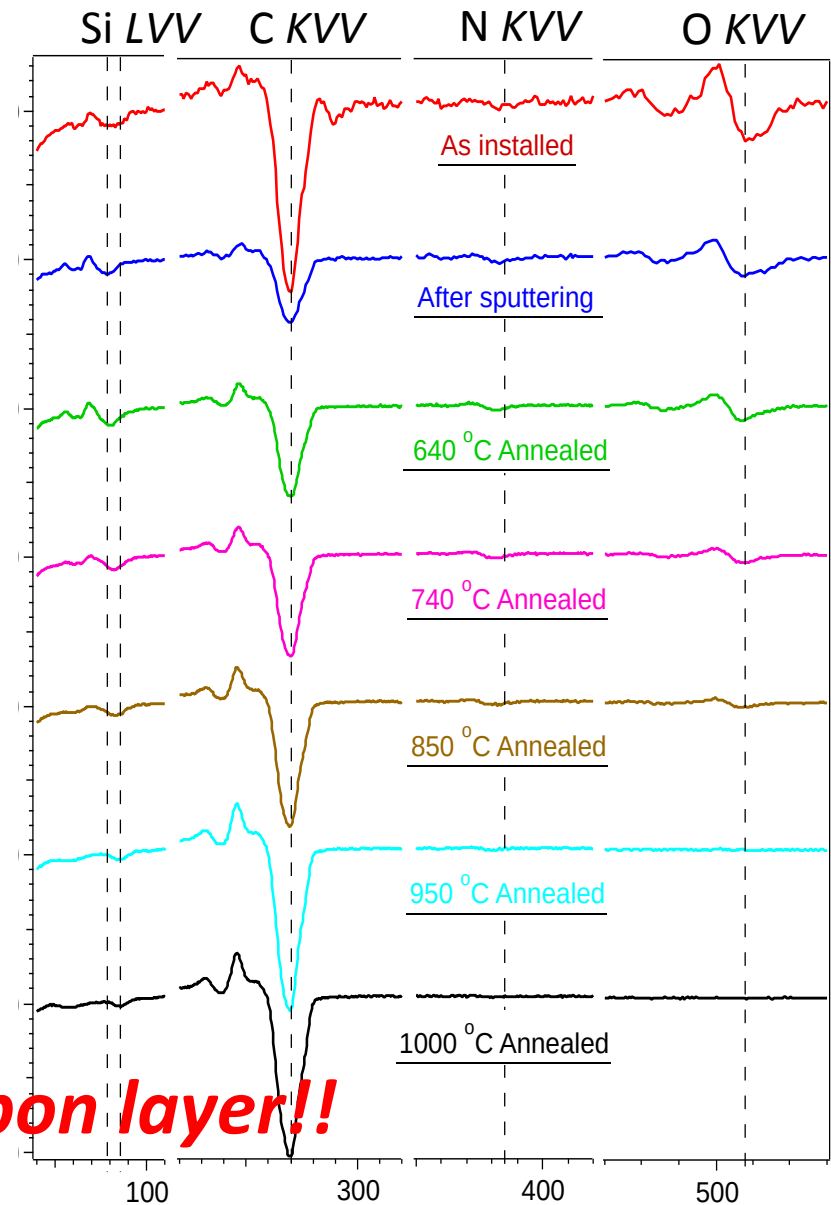
Dynamic Changes of the Surface Composition with T

Sample: **1B5C(HP)**



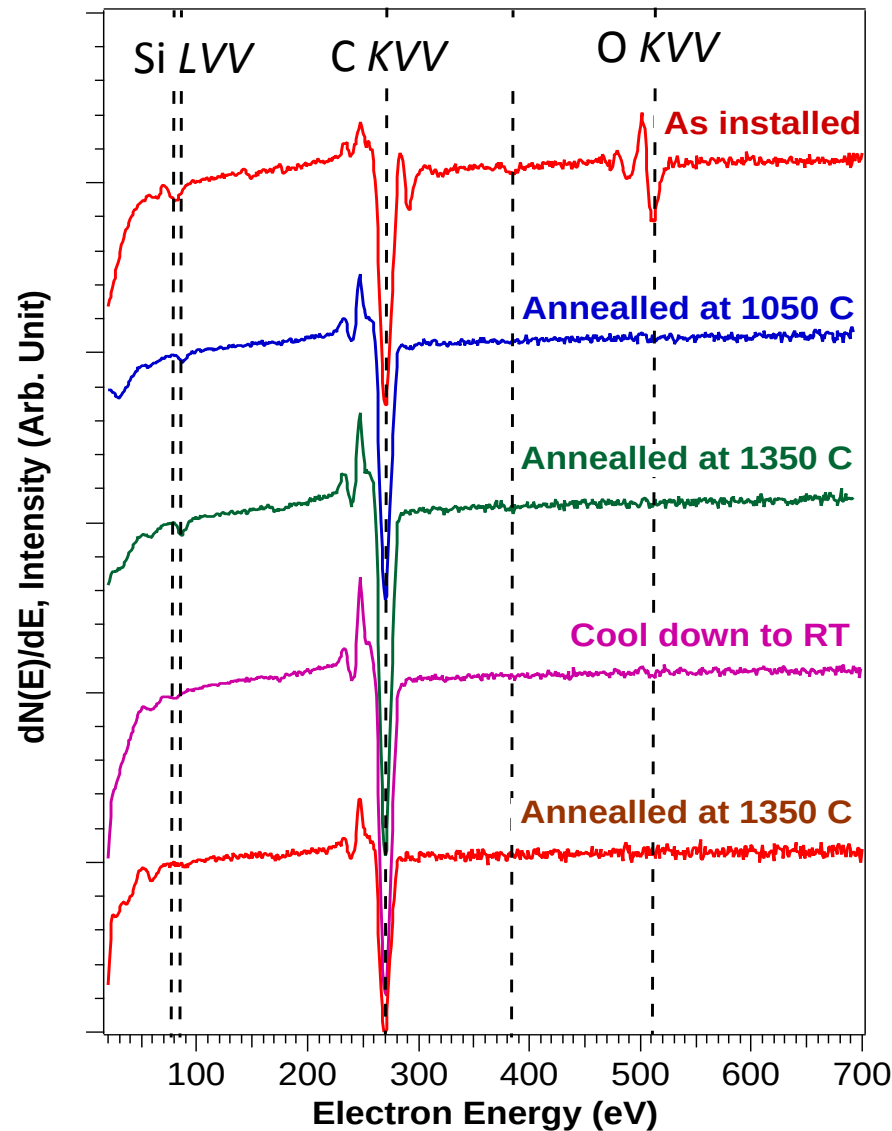
Self-regenerated carbon layer!!

Sample: **0B0C(PIP)**



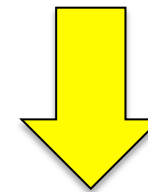
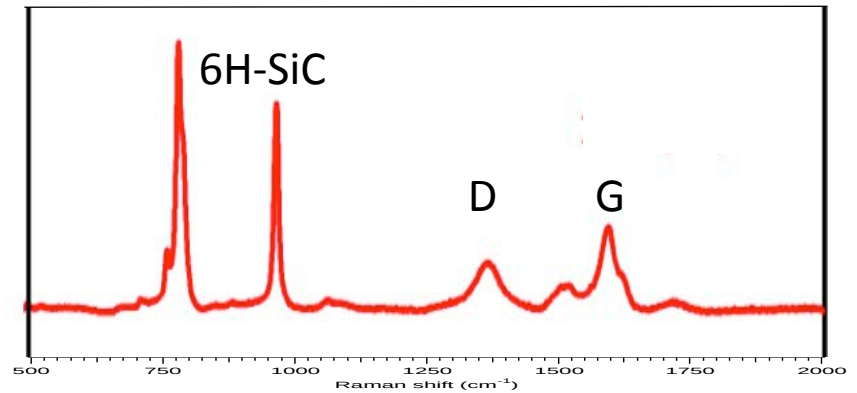
Dynamic changes of the Surface Structure with T

Auger Surface Composition with T

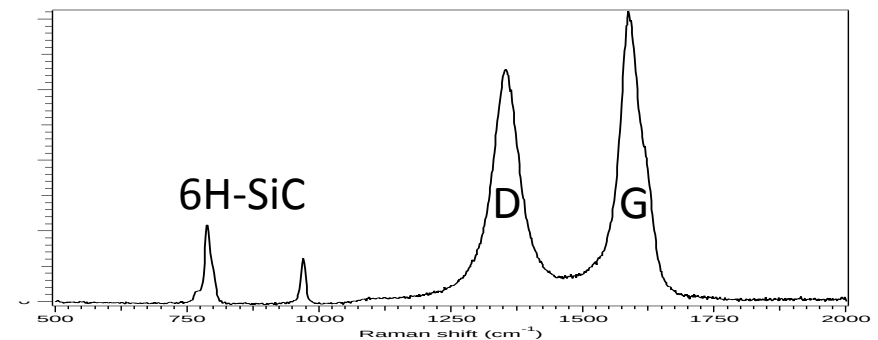


Raman Molecular Structure

As received



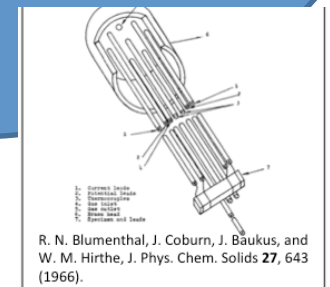
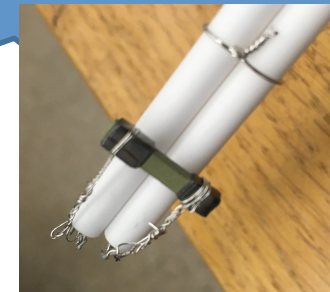
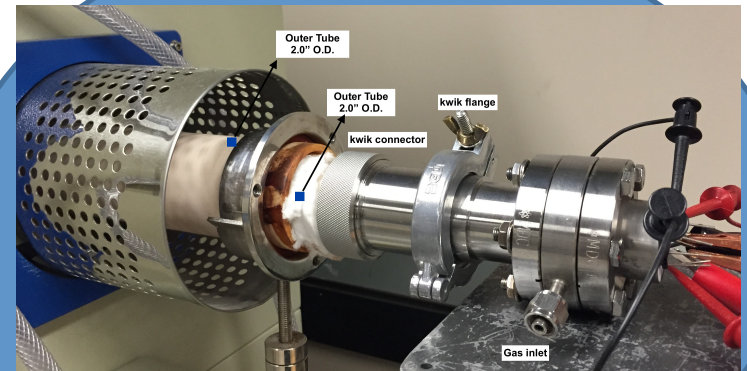
After heating to 1350°C



Sample: 1B1C (HP)

Resistivity Measurements

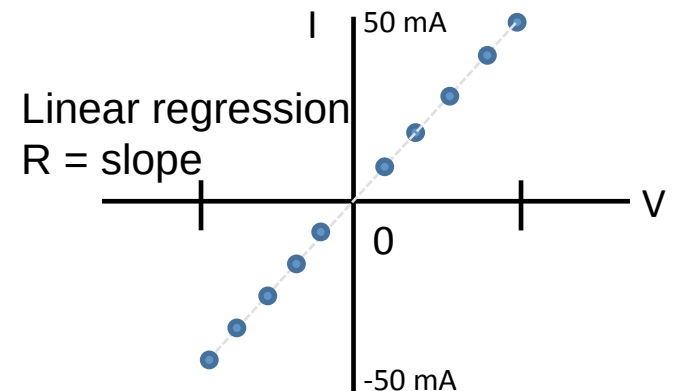
- ✓ Solutions:
 - Platinum lead, graphite contact and alumina support
 - Wrapping mechanism
- ✓ Pseudo-four-point probe (Kelvin probe)
- ✓ Alternative polarity to avoid capacity effect



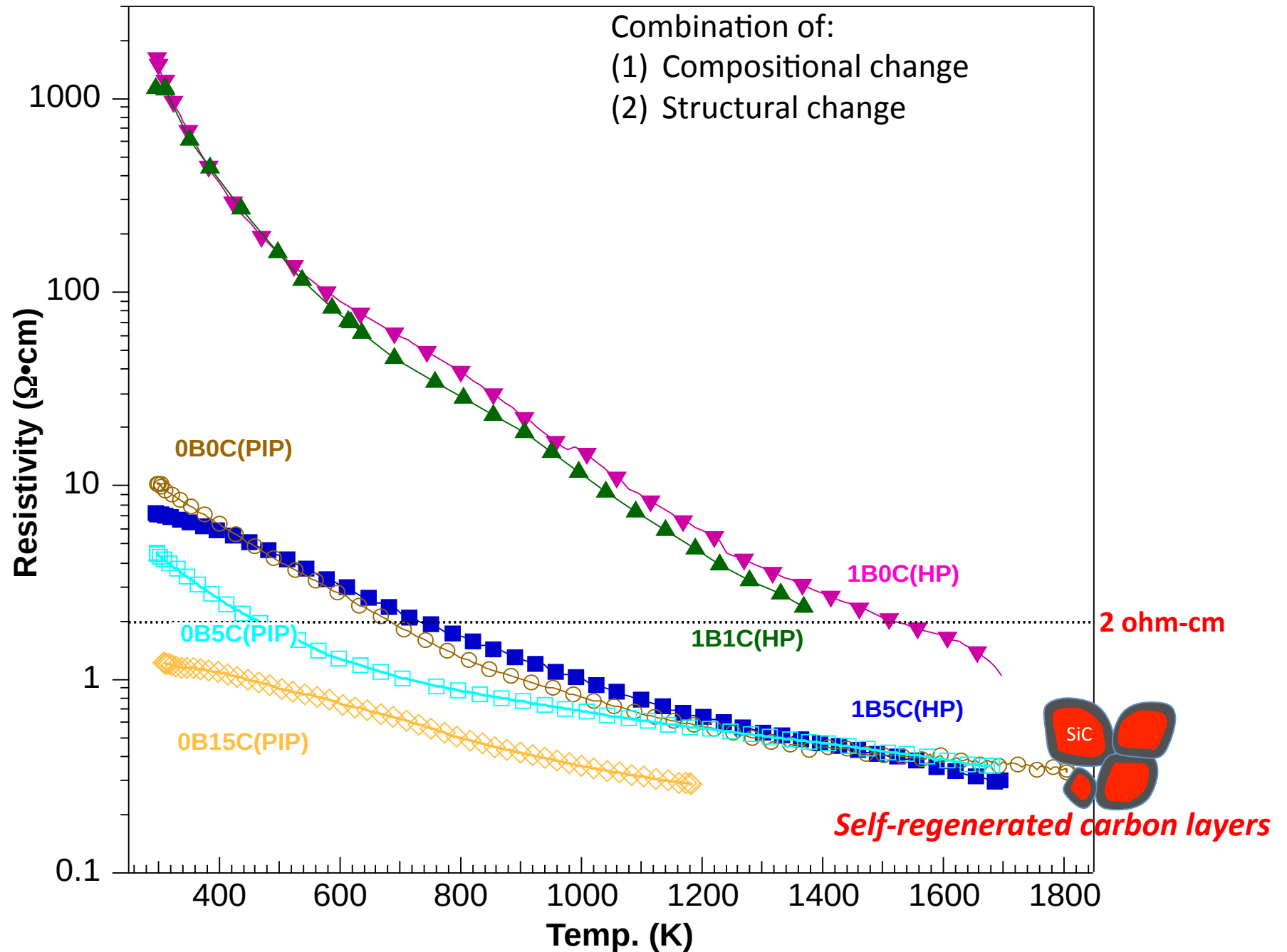
2000°K
Tube furnace

Electrometer
and current
source

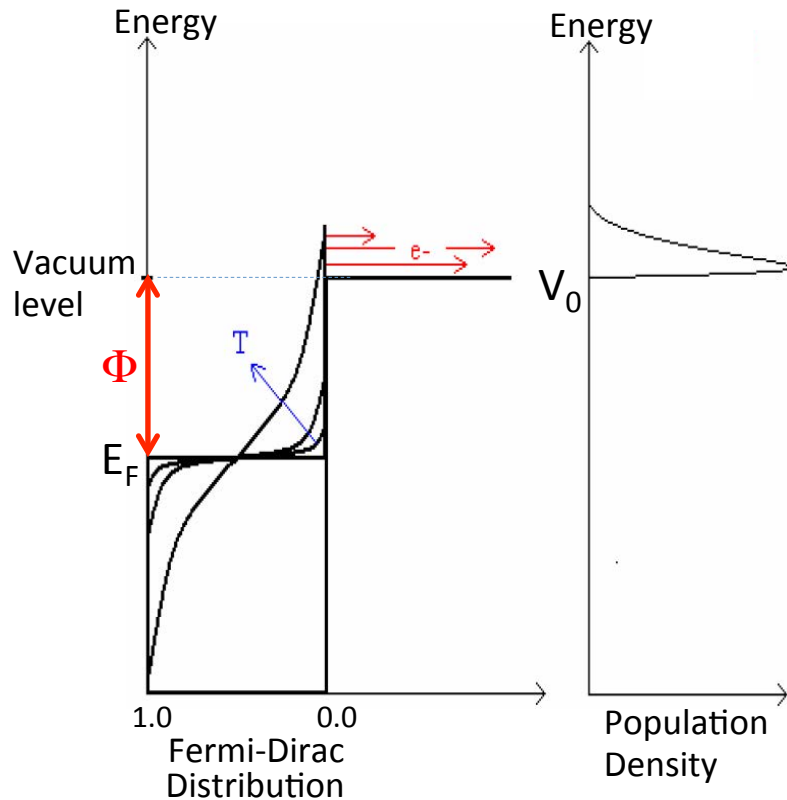
LabVIEW Work
station



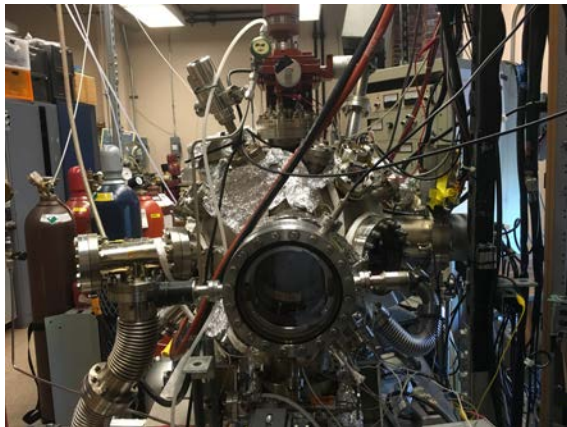
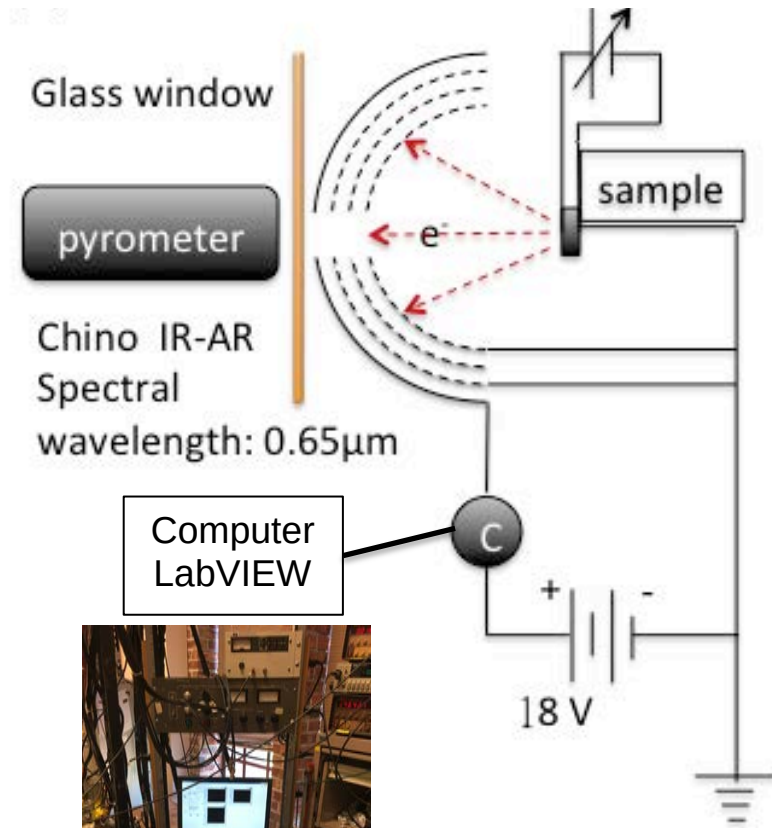
Electrical Resistivity Change with T for HP and PIP samples



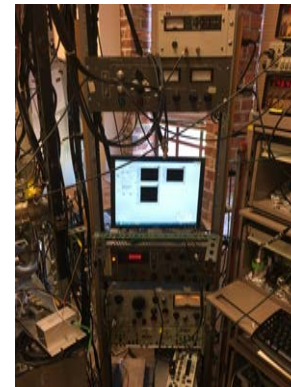
Thermionic emission (TE) Properties



$$J = -q \int_{W > \mu + \phi} v_x f(k) \frac{d\mathbf{k}}{4\pi^3} = \frac{mk_B^2 q}{2\pi^2 \hbar^3} T^2 \exp\left\{\frac{-\phi}{k_B T}\right\}$$



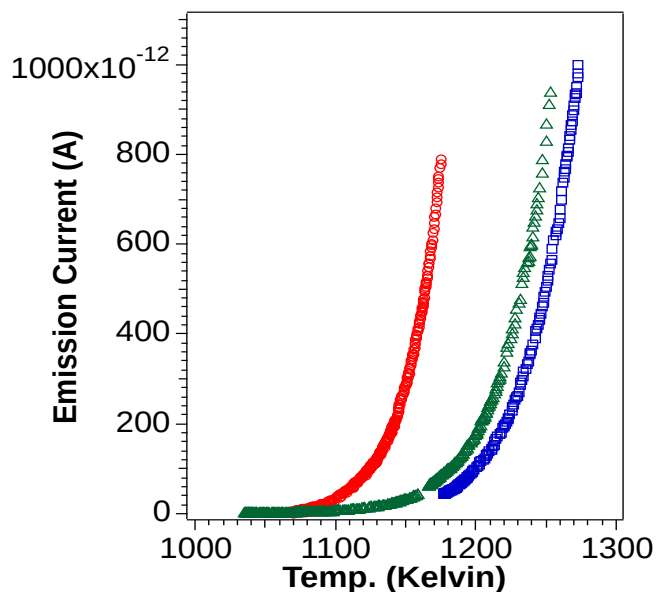
Front view of electron grid



Workstation for data acquisition

Dynamic Change of Workfunction Values

(a) Single crystal SiC



4.39 eV

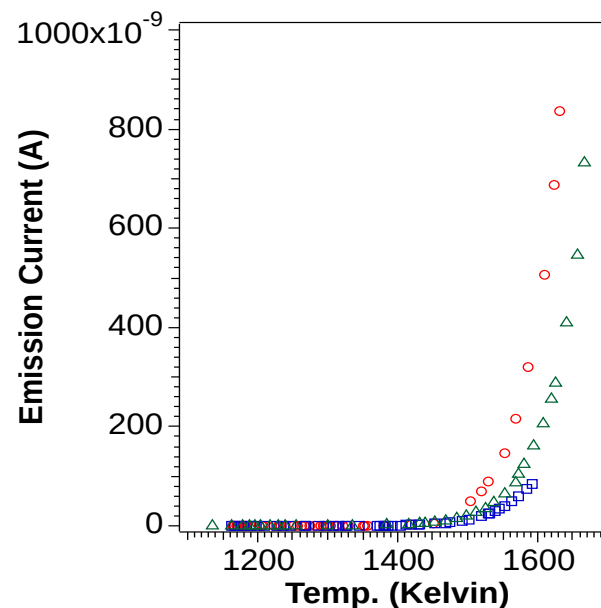


4.00 eV



4.02 eV

(b) B(OB1C)



4.8 eV

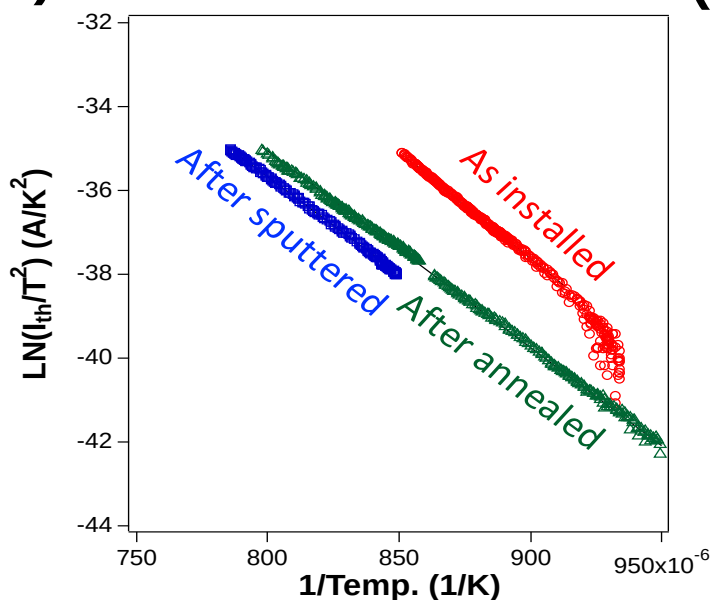


5.36 eV

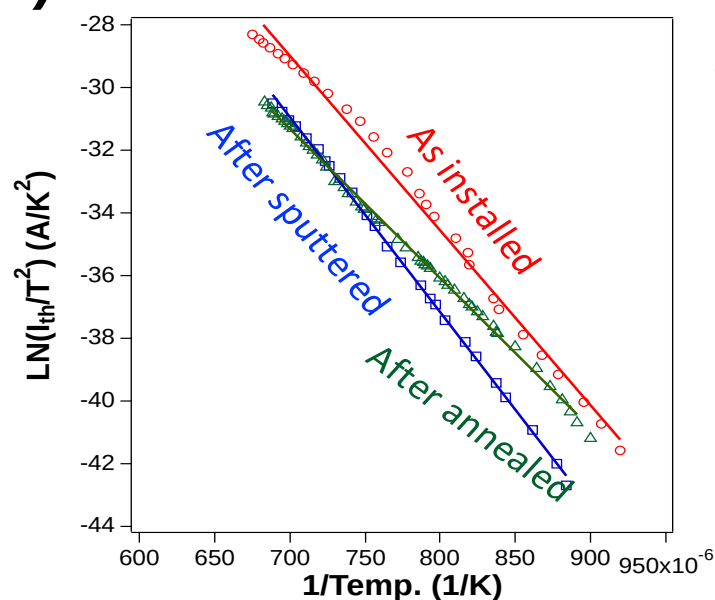


4.09 eV

(d)



(e)



Summary of this project

- A new type of silicon carbide / carbon composite synthesized from polymer derived synthesis:
Two routes: Hot-pressed and Polymer Infiltration
- Electrical properties of PDC SiC/C tailored by different carbon concentration.
- Self-regenerated surface is unique to the SiC/C composites
- Work function controlled by the self-regenerated carbon.
- K incorporation to SiC at the polymer precursor stage is planned to largely lower the workfunction of SiC-C composites.

Project Team Tasks

TASK 1: PROCESSING AND STABILITY OF NANOSTRUCTURED SI-C-X CERAMICS

Sub-Task 1.1: Effect of stoichiometry and temperature on the nanostructure

Sub-Task 1.2: Effect of temperature and stress on the stability of the nanostructure

TASK 2: MECHANICAL AND THERMAL PROPERTIES OF NANOSTRUCTURED SI-C-X CERAMICS

Sub-Task 2.1: Modulus, strength toughness, and thermal diffusivity

Sub-Task 2.2: Compressive creep

Lead: Prof. R. Bordia

TASK 3: ELECTRICAL PROPERTIES OF NANOSTRUCTURED SI-C-X CERAMICS

Sub-Task 3.1: Effect of C/Si ratio on room and elevated temperature electrical conductivity

Sub-Task 3.2: Combinatorial selection of X and effect of X on room and elevated temperature electrical conductivity

TASK 4: SURFACE ENGINEERING OF NANOSTRUCTURED SI-C-X CERAMICS

Sub-Task 4.1: Surface modification to enhance thermionic emissions

Sub-Task 4.2: Changes of surface/sub-surface structure and chemistry by high density plasma irradiation.

Sub-Task 4.3: Simulation of plasma interactions

Lead: Prof. F. Ohuchi



Thank you!

