“Plasma Assisted Synthesis of Molybdenum Carbide Catalyst”

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“Novel Materials and Processing in Chemical and Biomedical Engineering”
University of Illinois at Chicago – Summer 2005

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Outline

- Introduction to Carbide and Nitride Catalysts
  - Motivation to Produce the Catalysts
  - Previous and Current Experimentation
- Apparatus and Procedures for Experimentation
- Results of Experiments
  - Tools for Analysis
  - Raman, XPS, and SEM Results
- Conclusions of Results
- Future Work for Research

Introduction to Carbide and Nitride Catalysts

- Motivation to Produce the Catalysts
  - Replace noble metal catalysts (Pt, Pd, Ru, Rh)
  - Water-Gas Shift Reaction for fuel cells:

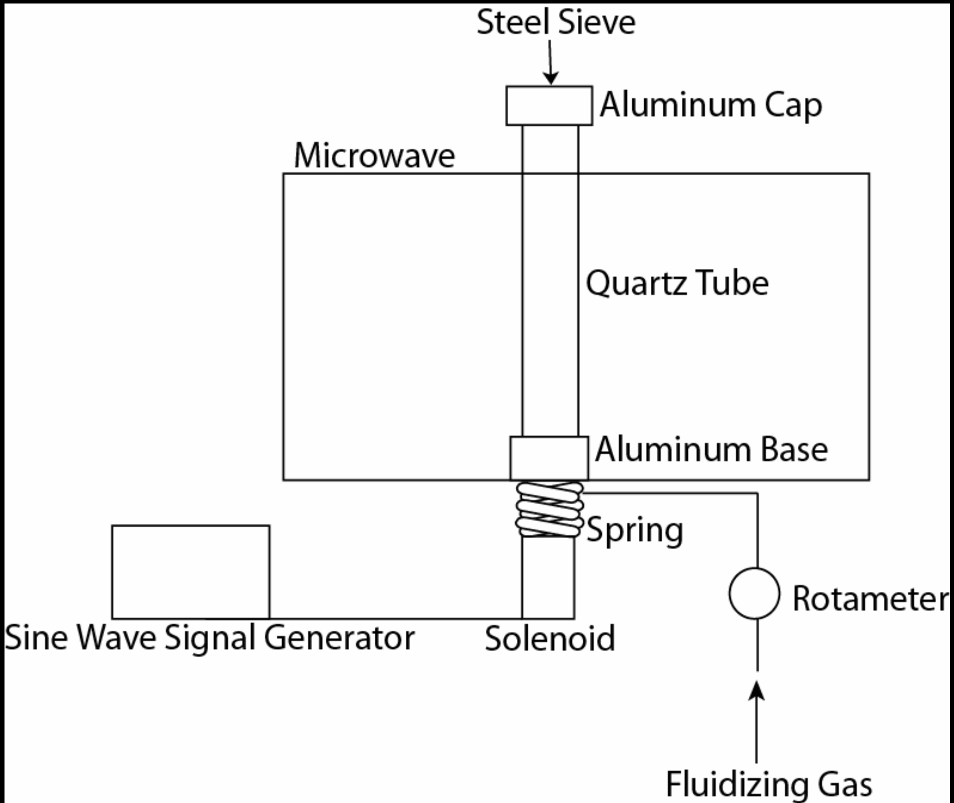
    $$CO + H_2O \leftrightarrow CO_2 + H_2$$

  - Hydroprocessing, hydrogenation, hydrogenolysis, methane activation, amination, acetone condensation, and isomerization

Introduction to Carbide and Nitride Catalysts

- Previous Experimentation
  - Industry: thermal processing
  - Our lab: Microwave Assisted Fluidized Bed Synthesis

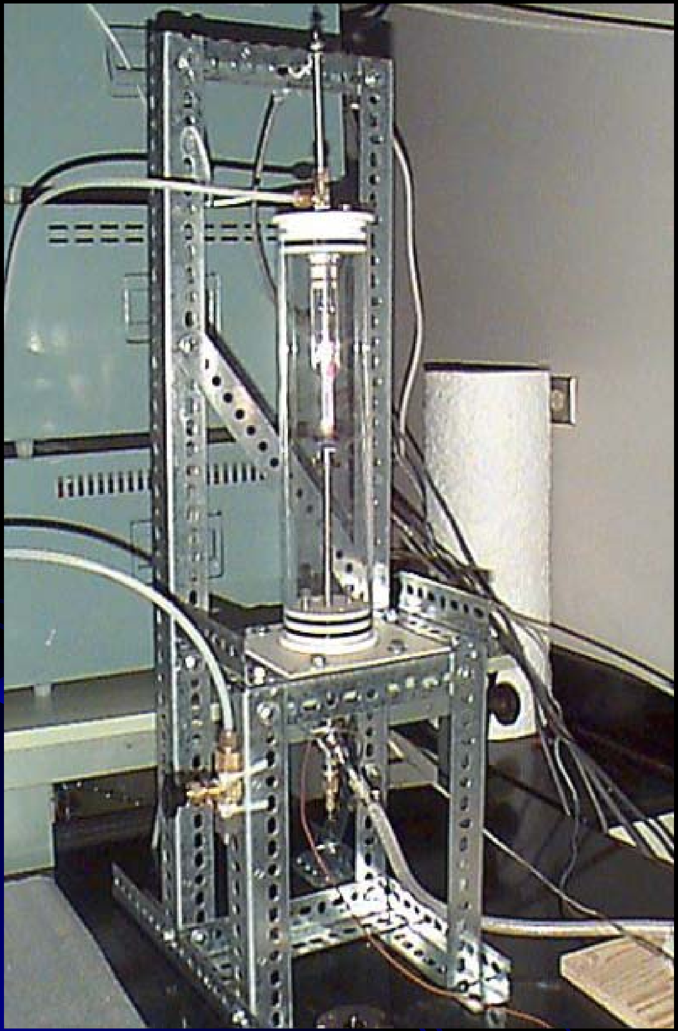
Advantages: less time, lower temperatures, lower cost



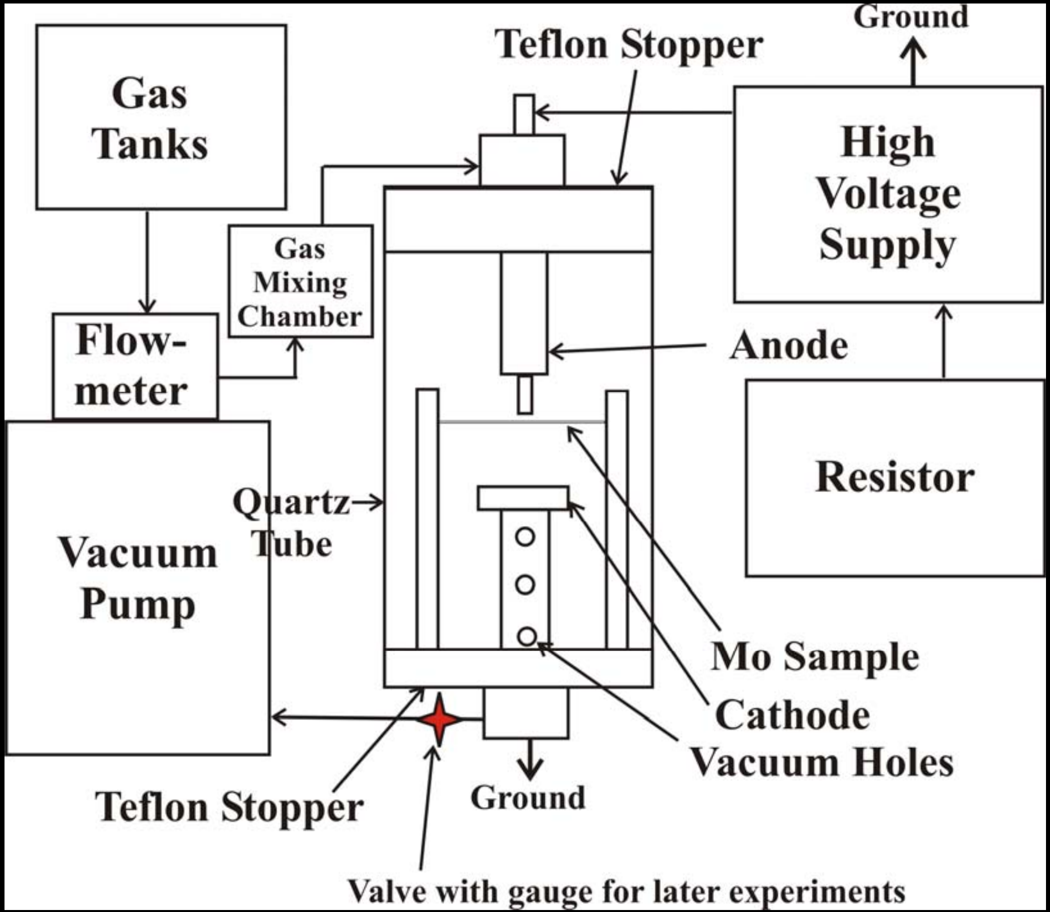
Introduction to Carbide and Nitride Catalysts

- Current Experimentation
  - Plasma Assisted Process Theory
    - Plasma-Enhanced Chemical Vapor Deposition
    - **Surface Modification** → Our goal
  - Advantages
    - Influence surface properties of metal
    - Lower temperatures and less processing time and energy than thermal chemical vapor deposition (CVD)

Apparatus and Procedures for Experiments



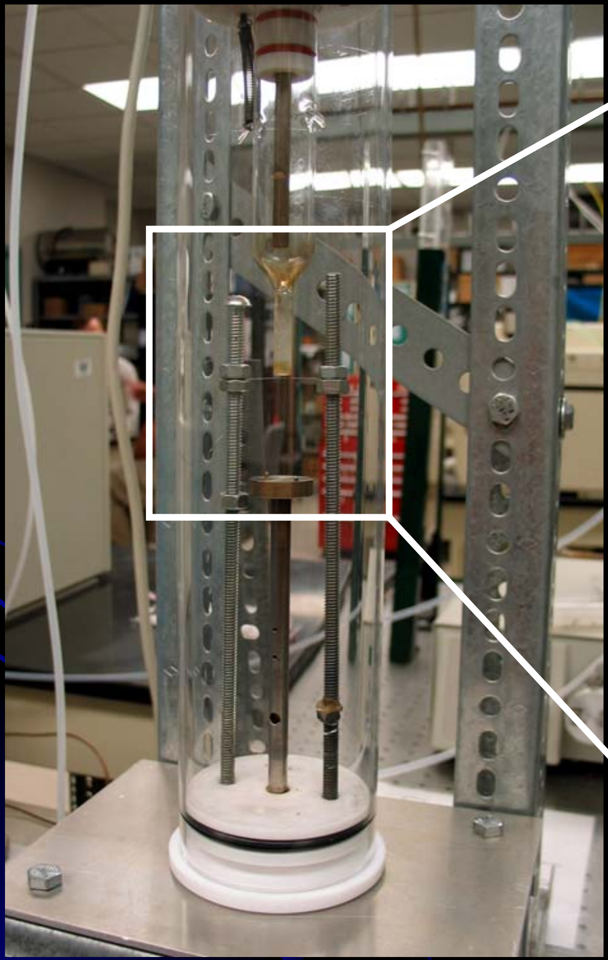
Plasma Discharge Reactor

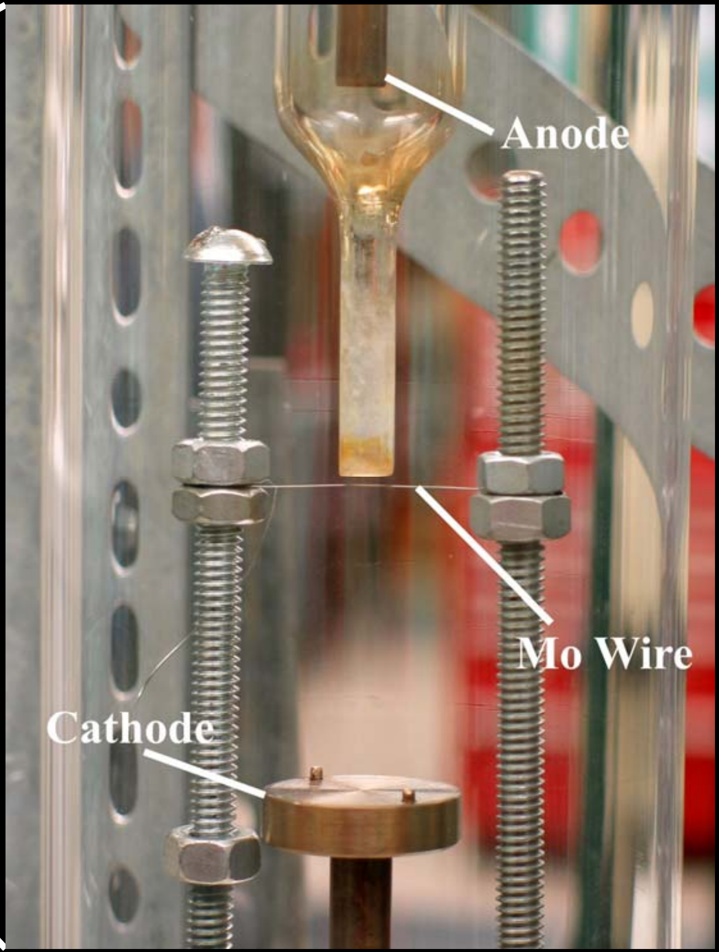


Reactor Schematic

Apparatus and Procedures for Experiments

Stretch 0.25mm diameter Mo wire or 0.025mm thick Mo foil between anode and cathode





Experimental set-up prior to plasma discharge ignition

Apparatus and Procedures for Experiments

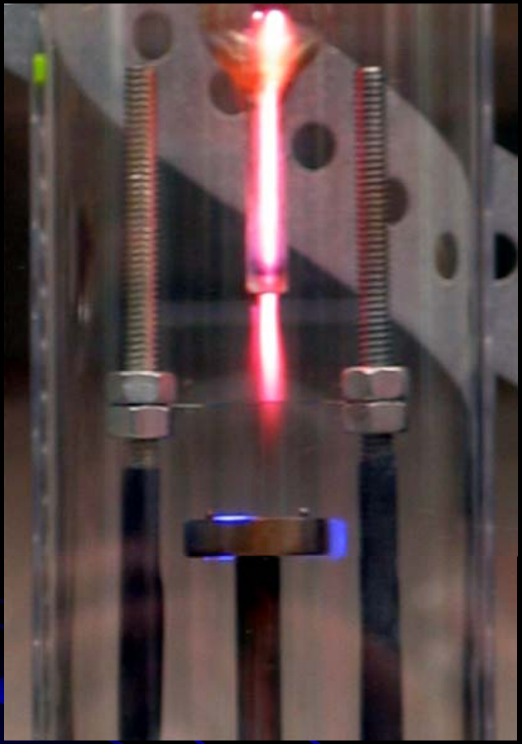
- Turn on vacuum, resistor, and high voltage source

- Set reaction chamber pressure

- Apply 8.75kV across electrode and open gas source (H_2 in Ar or C_2H_4 in Ar)

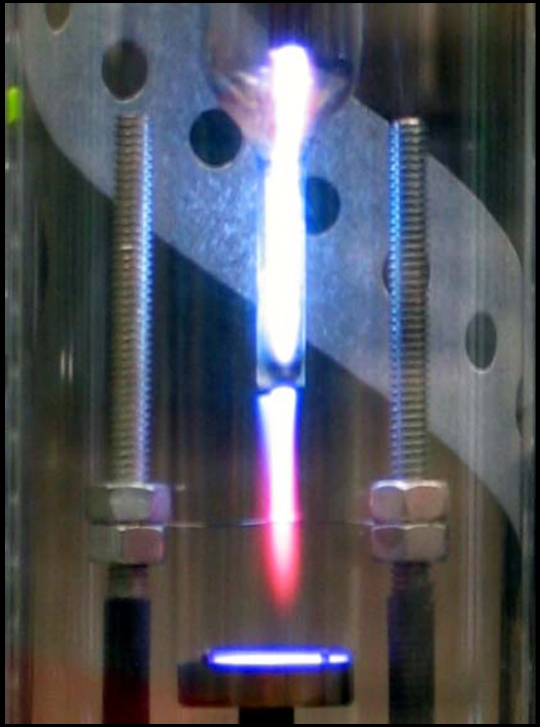
- Begin timing of reaction at introduction of gases

Apparatus and Procedures for Experiments



Discharge in Ar

Discharge in Ar + C_2H_4



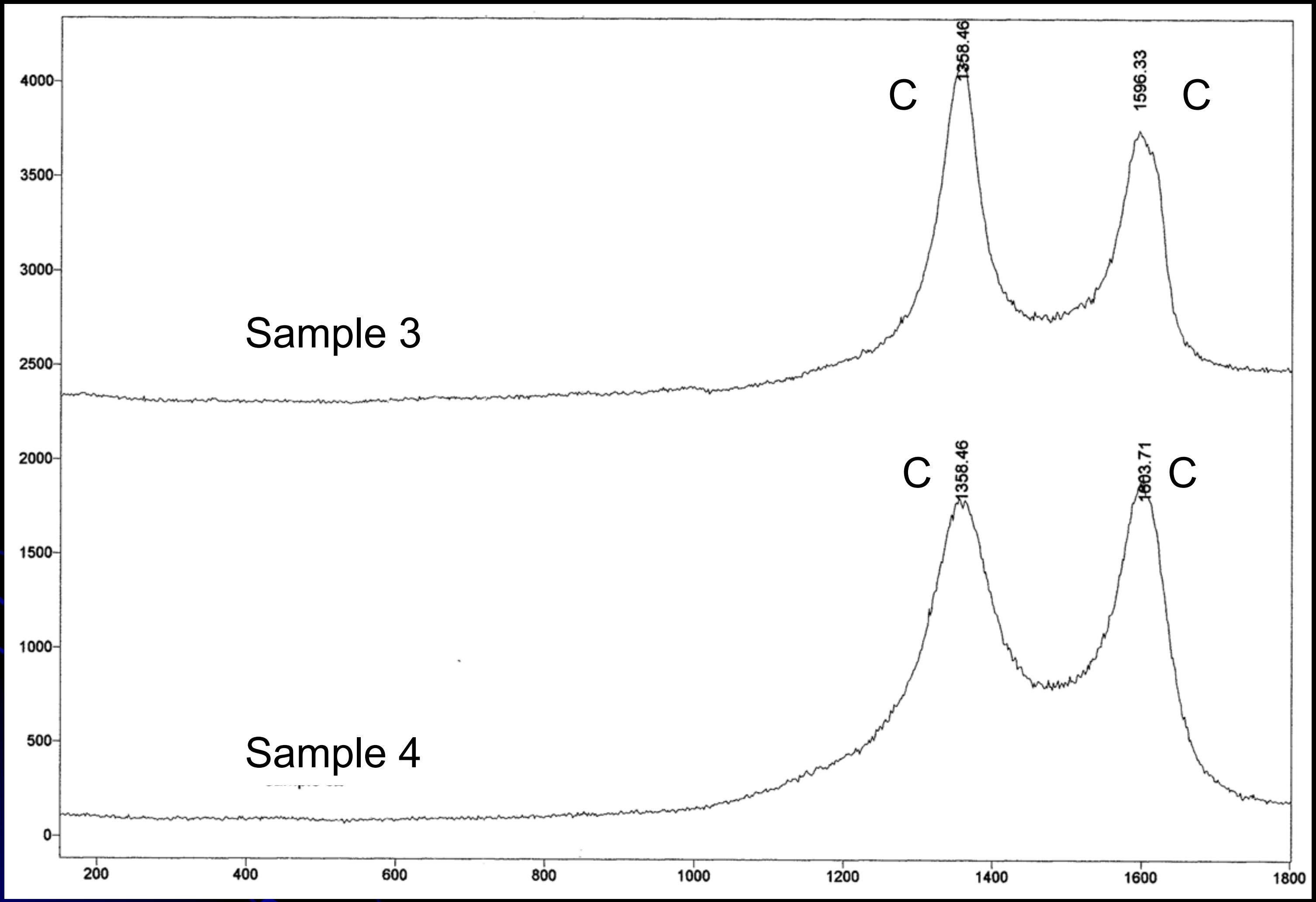
Discharge in Ar + H_2

Plasma discharges in gas

Results of Experiments

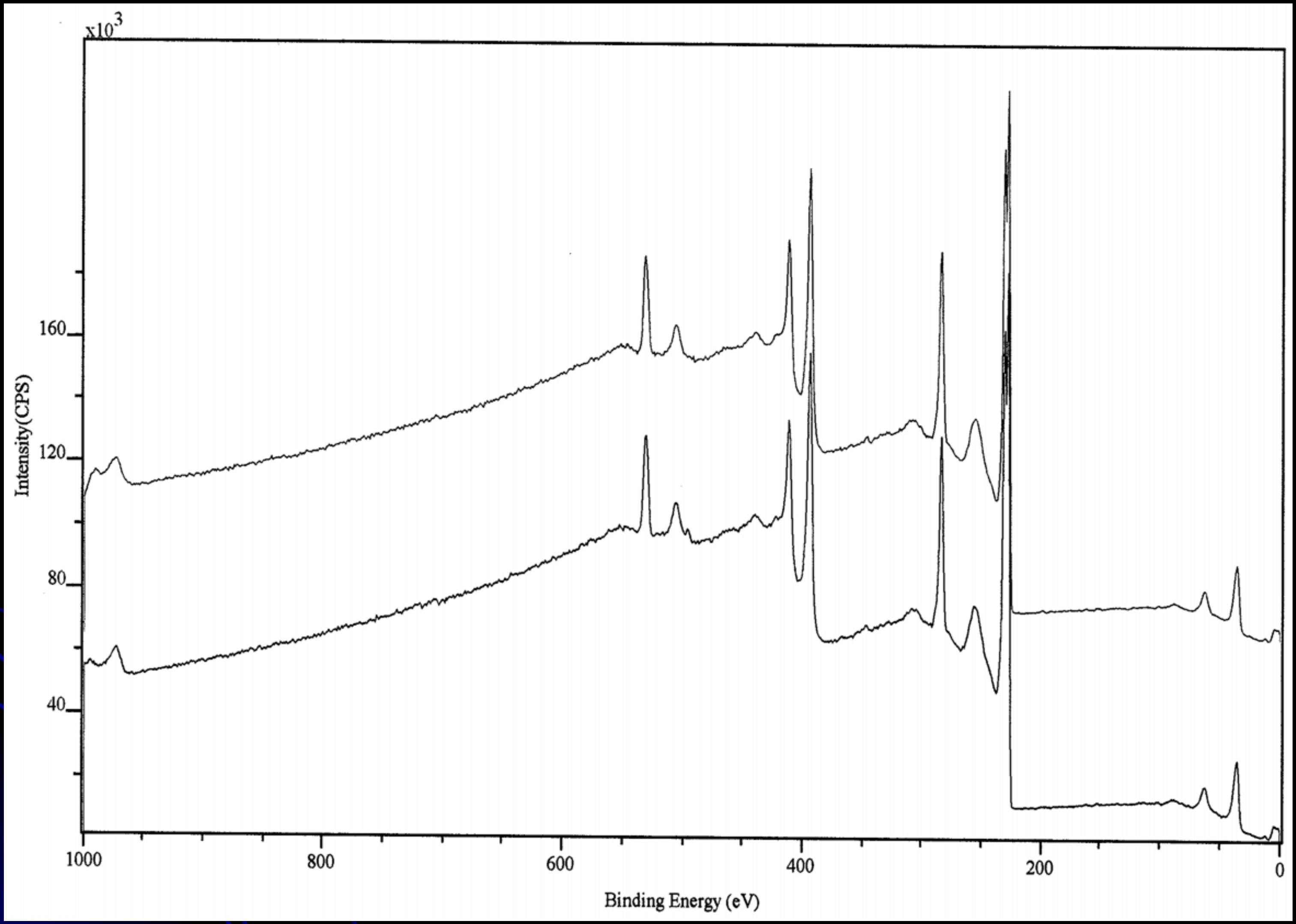
- Tools for Analysis:
  - Raman Spectroscopy - measures monochromatic light source shift
  - X-ray Photoelectron Spectroscopy – measures bond energies
  - Scanning Electron Microscopy – surface imaging

Raman Results



Raman spectra for samples 3 and 4

XPS Results



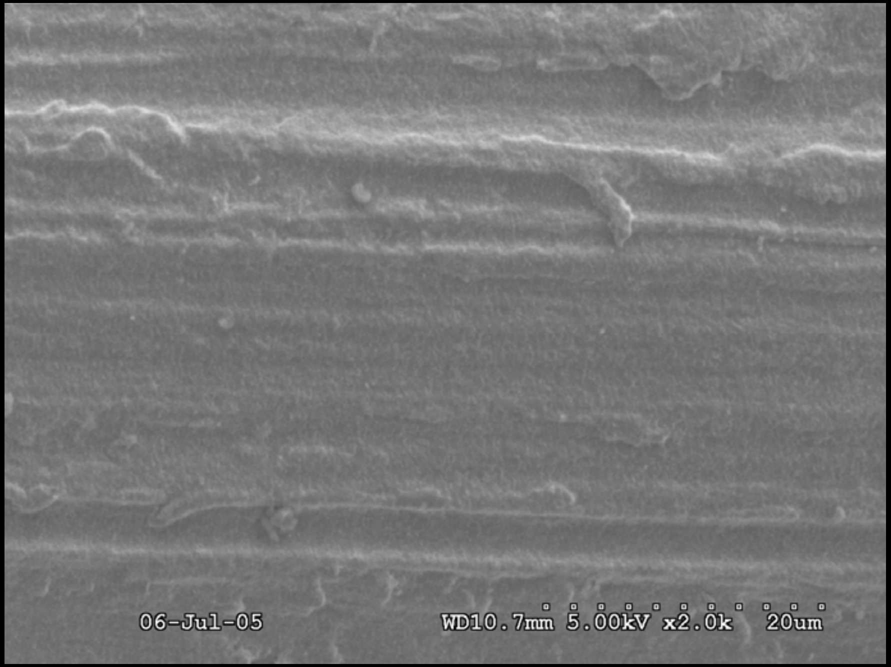
XPS for Sample 3

XPS Results

Orbital	*Sample 3*	*Assignment*	*Sample 4*	*Assignment*
Mo 3d	228	Mo	227.8	Mo/ Mo_2C
	231.3	MoO_xC_y	232.1	MoO_3
			235.5	MoO_x
C 1s	283	Mo_2C	284.3	C
	283.9	C	288.5	C-O/C=O
O 1s	530.2	MoO_x	532.1	MoO_x
	531.3	MoO_3		

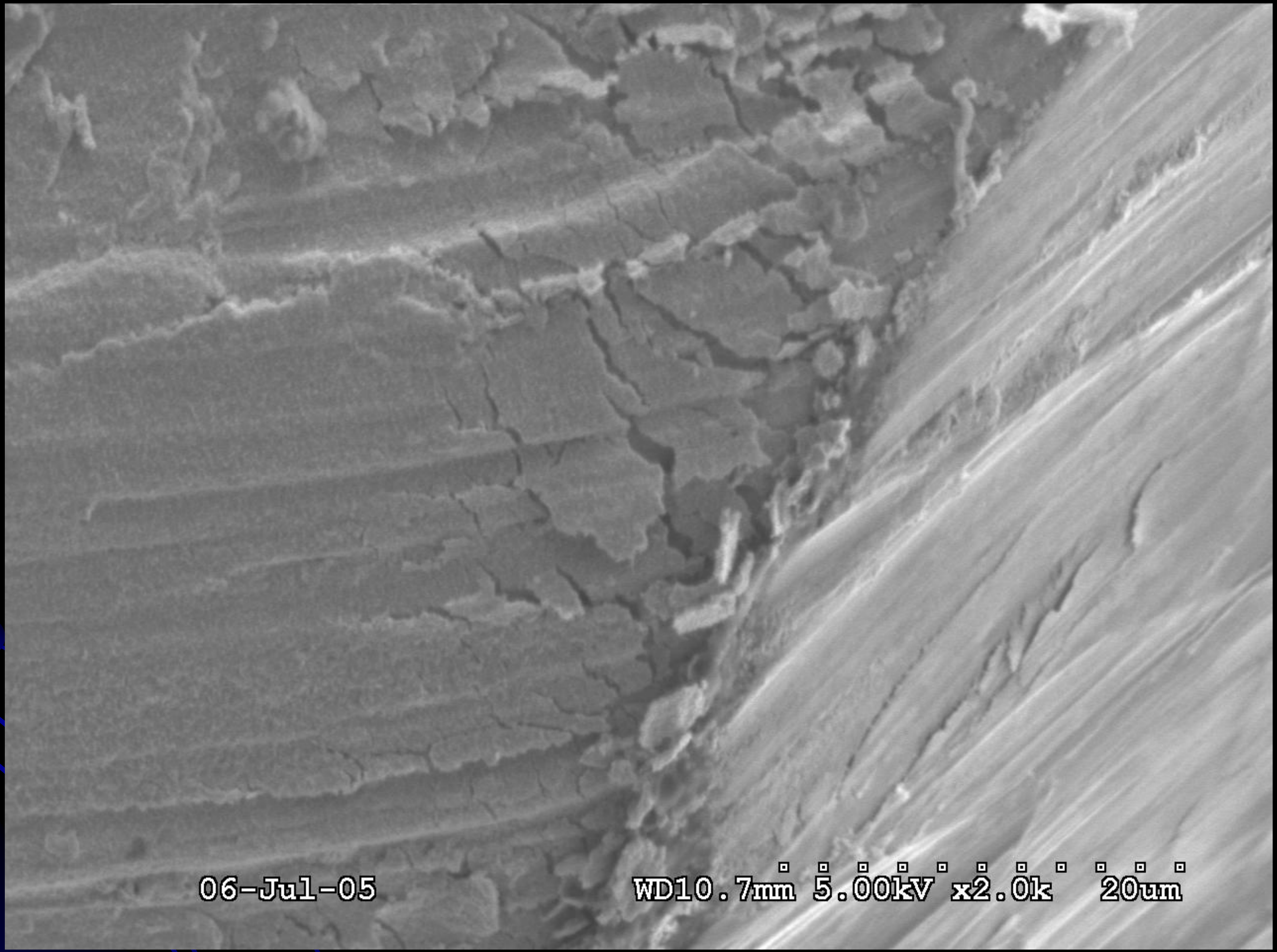
SEM Results

SEM of Mo Wire Reference at 2000x and 5.00 kV



SEM of 30 second sample at 2000x and 5.00 kV

SEM Results



SEM of 30 second sample at 2000x and 5.00 kV

Conclusions of Results

- Raman shows carbon deposits and oxides at nonuniform compositions across samples
- XPS suggests we could have carbide, oxycarbide, and oxides
- SEM shows uniform film formation
- Mo_2C confirmation requires further tools of analysis; Transmission Electron Microscopy (TEM)
- Molybdenum oxides suggest surface reaction with oxygen

Future Work for Research

- Use TEM to confirm film identity as carbide or otherwise
- Synthesize catalysts in powder form
- Test for catalytic activity in Water-Gas Shift Reaction
- Develop control process for reactor in forming carbide *and* nitride films (pressure, time, voltage, temperature, metal base identity, gas composition, etc.)

Acknowledgments

- National Science Foundation for EEC-0453432 Grant that funded the "Novel Materials in Processing in Chemical and Biomedical Engineering" for Summer 2005 at UIC
- Directors: Dr. Christos Takoudis (REU) and Dr. Andreas Linninger (RET)
- Faculty Advisor: Dr. Kenneth Brezinsky
- Graduate Student Advisor: Gabriel Duran
- Co-workers: Timothy Walsh (RET) and Charlie Robinson (Upward Bound)
- Plasma Expert: Dr. Alexei Saveliev
- Laboratory: Dr. Lawrence Kennedy
- Microscopy Expert at RRC: Dr. John Roth
- Glassblower: Brian Schwandt
- Post-Doctorate Advisor: Dr. Jennifer Dunn
- Dr. Lawrence Kennedy students
- MARC U*STAR PUCPR Honor Program

Questions?