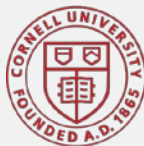


# Fabricating 2D Silica using Atomic Layer Deposition

***Reed Yalisove  
Materials Science and Engineering  
University of Michigan***

***Professor James Engstrom and Taewon Suh,  
Chemical and Biomolecular Engineering, Cornell  
University***



**Cornell  
University**



# Silicatene

- 2D layer of silicon dioxide ( $\text{SiO}_2$ )
  - Silicatene

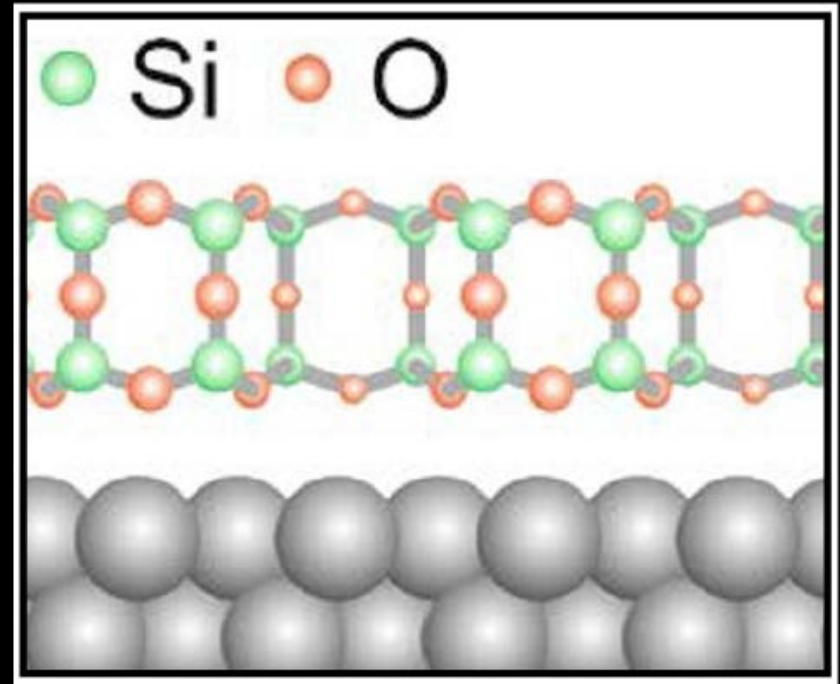


Figure from Büchner et al.

# Silicatene

- 2D layer of silicon dioxide ( $\text{SiO}_2$ )
  - Silicatene
- Attachment via van der Waals forces

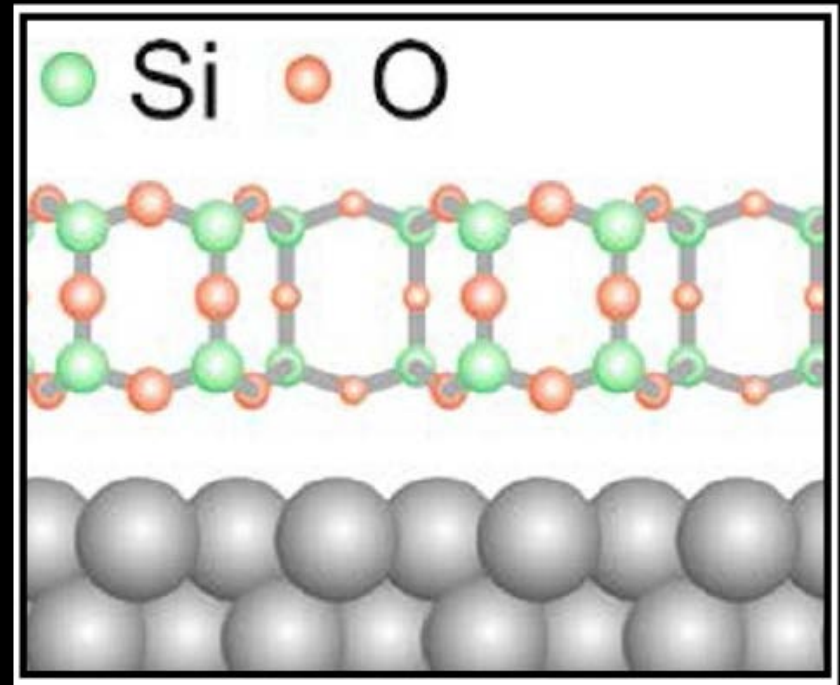


Figure from Büchner et al.

# History

- **Silicatene has been fabricated**



# History

- **Silicatene has been fabricated**
  - **Single crystal Pd, Pt, Ru**



# History

- Silicatene has been fabricated
  - Single crystal Pd, Pt, Ru
- Evaporation of Si



<https://www.cnfusers.cornell.edu/sites/default/files/tool-images/Even%20Hour%20CVC%20Evaporator.jpg>



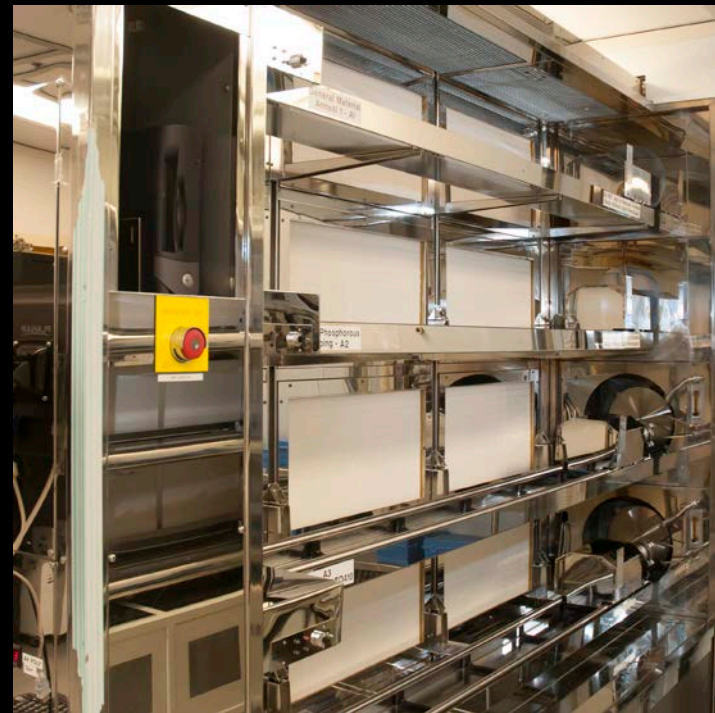
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Cornell NanoScale Science  
and Technology Facility



Engstrom  
Research Group

# History

- **Silicatene has been fabricated**
  - Single crystal Pd, Pt, Ru
- **Evaporation of Si**
- **Annealing**



<https://www.cnfusers.cornell.edu/sites/default/files/tool-images/Furnace%20Bank%20A.jpg>



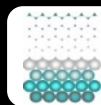
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# Motivation

- **Engstrom group specializes in Atomic Layer Deposition (ALD)**



# Motivation

- Engstrom group specializes in Atomic Layer Deposition (ALD)
- **ALD of silicatene not yet reported**



# Fabricating Silicatene

- **Deposition tools**



# Fabricating Silicatene

- Deposition tools
  - Evaporator



<https://www.cnfusers.cornell.edu/sites/default/files/tool-images/Even%20Hour%20CVC%20Evaporator.jpg>



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# Fabricating Silicatene

- Deposition tools
  - Evaporator
  - ALD



<https://www.cnfusers.cornell.edu/sites/default/files/tool-images/Oxford%20Flexal%20ALD.jpg>



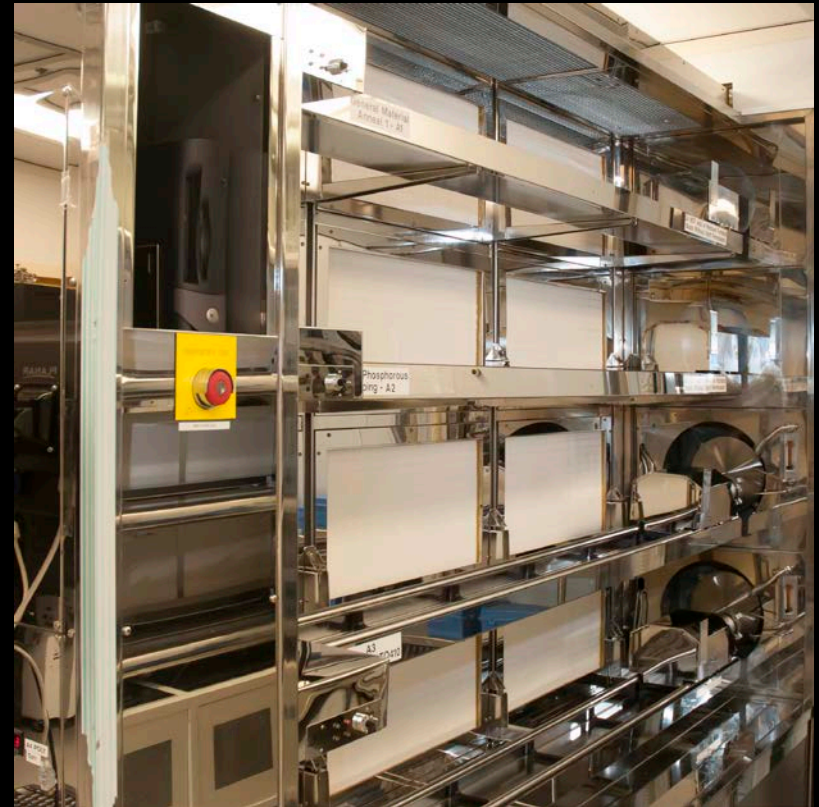
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# Fabricating Silicatene

- **Deposition tools**
  - Evaporator
  - ALD
  - Furnace



<https://www.cnfusers.cornell.edu/sites/default/files/tool-images/Furnace%20Bank%20A.jpg>



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# Studying Silicatene

- **Characterization Tools**



# Studying Silicatene

- **Characterization Tools**
  - **Contact Angle**



<https://www.cnfusers.cornell.edu/sites/default/files/tool-images/Woollam.jpeg>



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# Studying Silicatene

- **Characterization Tools**
  - **Contact Angle**
  - **Ellipsometry**



<https://www.cnfusers.cornell.edu/sites/default/files/tool-images/VCA%20Optima%20Contact%20Angle%20Goniometer.jpg>



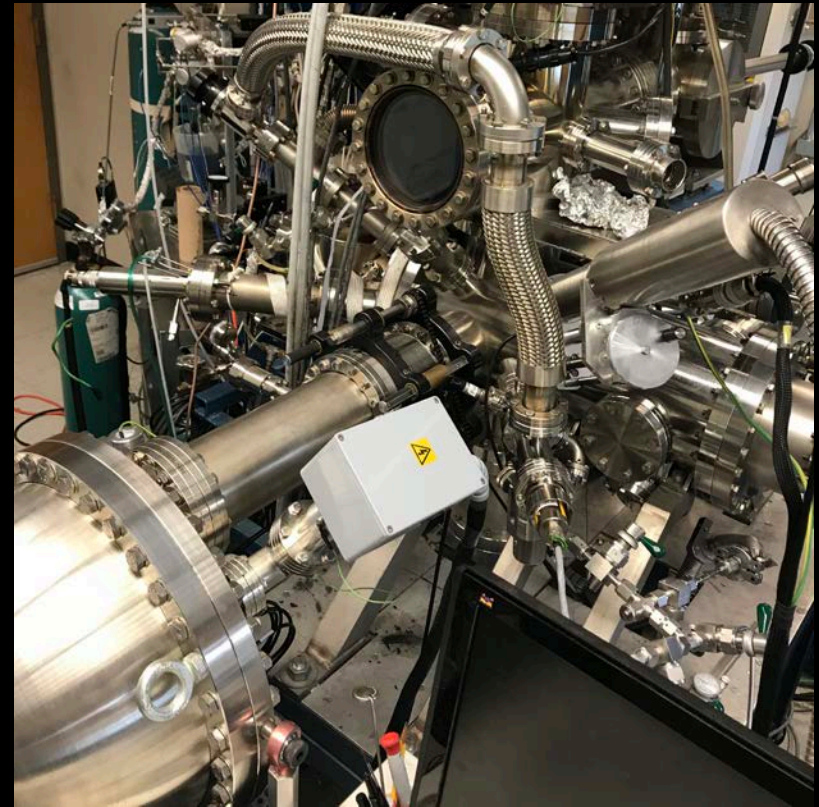
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# Studying Silicatene

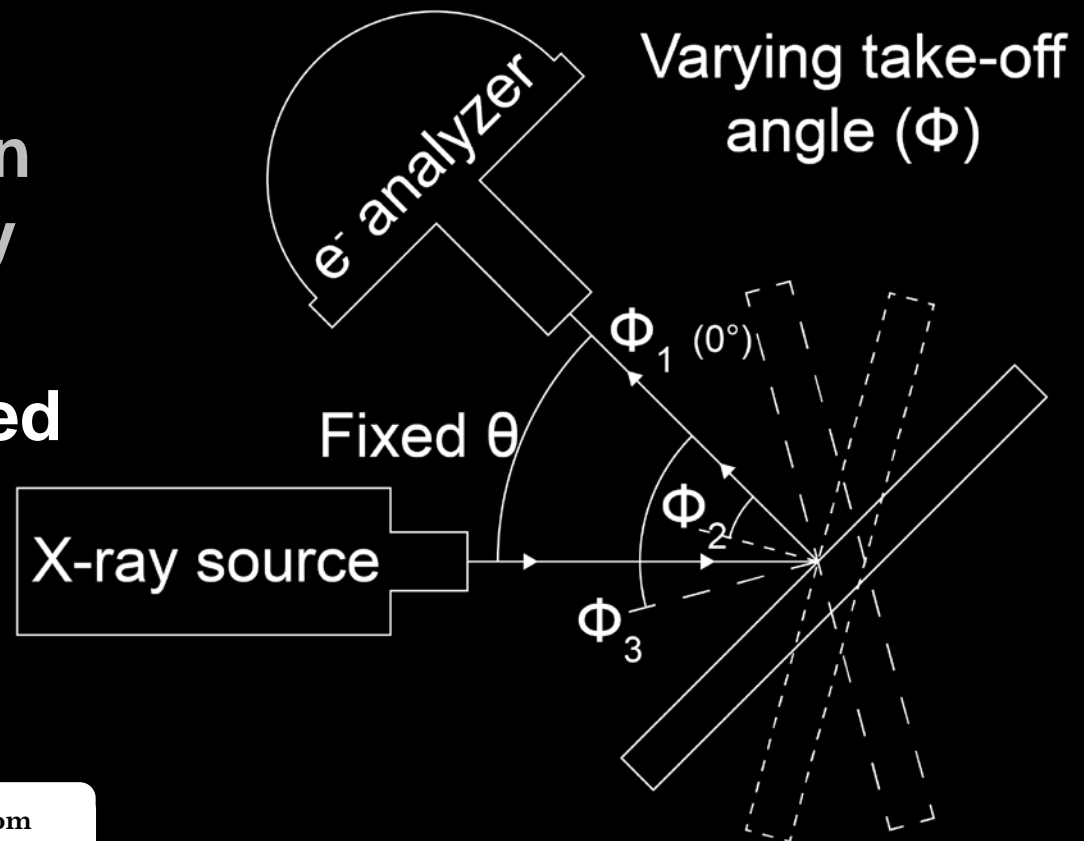
- **Characterization Tools**
  - Contact Angle
  - Ellipsometry
  - X-ray Photoelectron spectroscopy (XPS)



# Studying Silicatene

- **Characterization Tools**

- **Contact Angle**
- **Ellipsometry**
- **X-ray Photoelectron spectroscopy (XPS)**
- **Angle resolved XPS**



# Depositing metal substrates

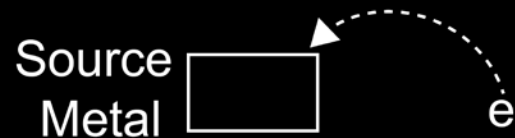
- **Metal substrates (Pd, Pt, Ru) deposited with electron beam assisted evaporator**



# Depositing metal substrates

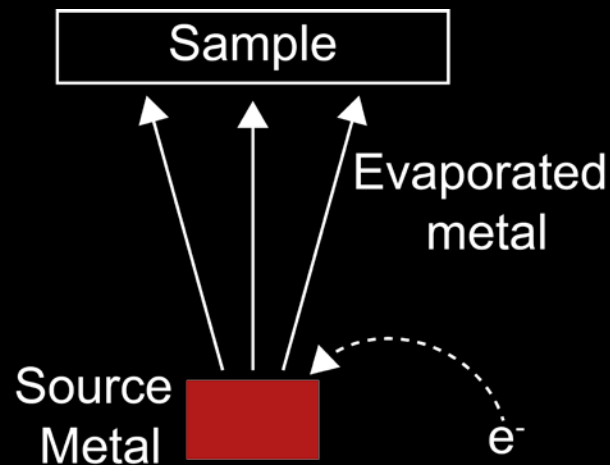
- **Metal substrates (Pd, Pt, Ru) deposited with electron beam assisted evaporator**
  - **Electron beam excites source atoms**

Sample



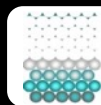
# Depositing metal substrates

- **Metal substrates (Pd, Pt, Ru) deposited with electron beam assisted evaporator**
  - **Electron beam excites source atoms**
  - **Atoms evaporate from source and deposit on substrate**



# Atomic Layer Deposition

- **SiO<sub>2</sub> deposited with ALD**



# Atomic Layer Deposition

- **SiO<sub>2</sub> deposited with ALD**
- **Two self-limiting reaction steps**

Metal  
Pd, Pt, or Ru

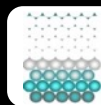
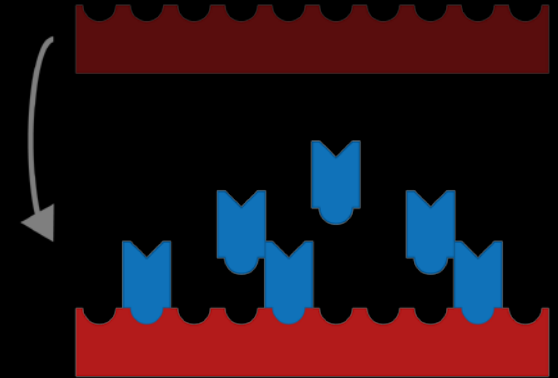


# Atomic Layer Deposition

- **SiO<sub>2</sub> deposited with ALD**
- **Two self-limiting reaction steps**

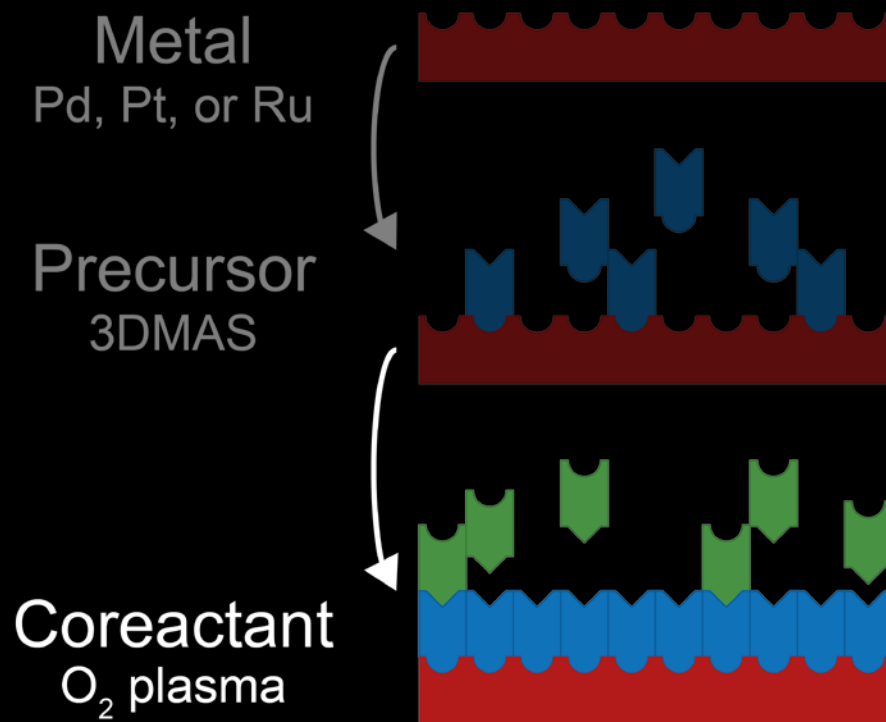
Metal  
Pd, Pt, or Ru

Precursor  
3DMAS



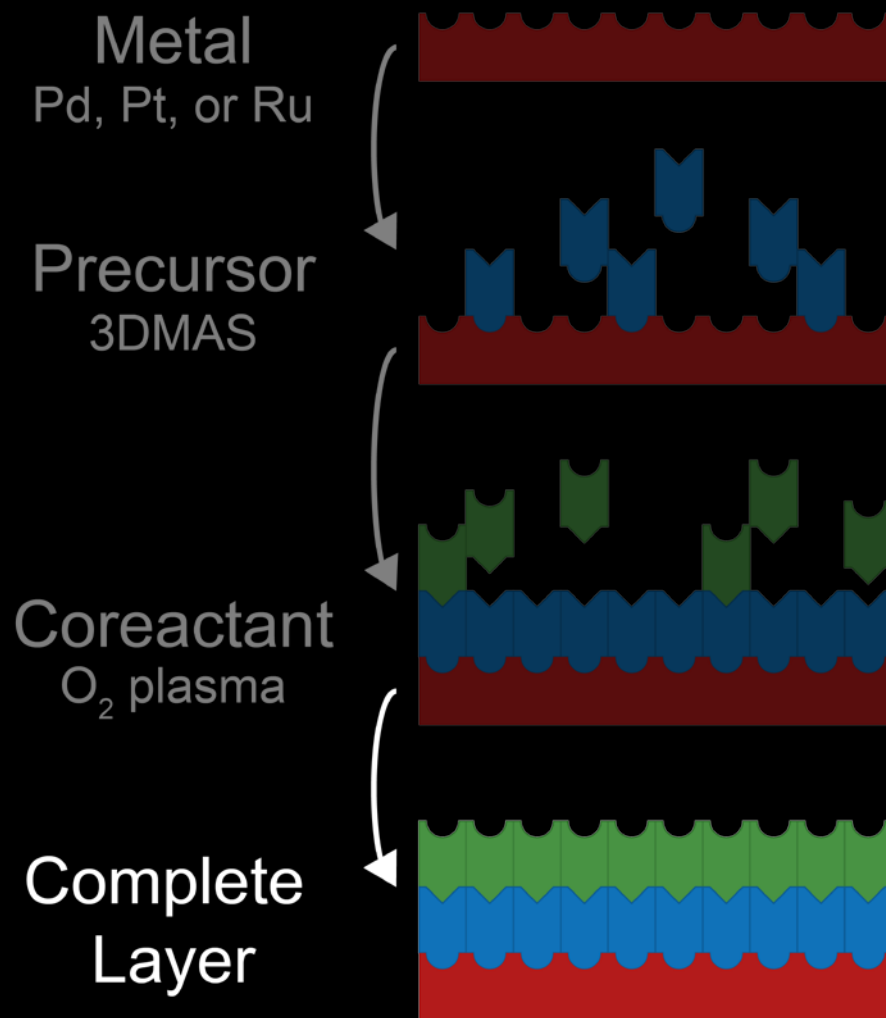
# Atomic Layer Deposition

- **SiO<sub>2</sub> deposited with ALD**
- **Two self-limiting reaction steps**



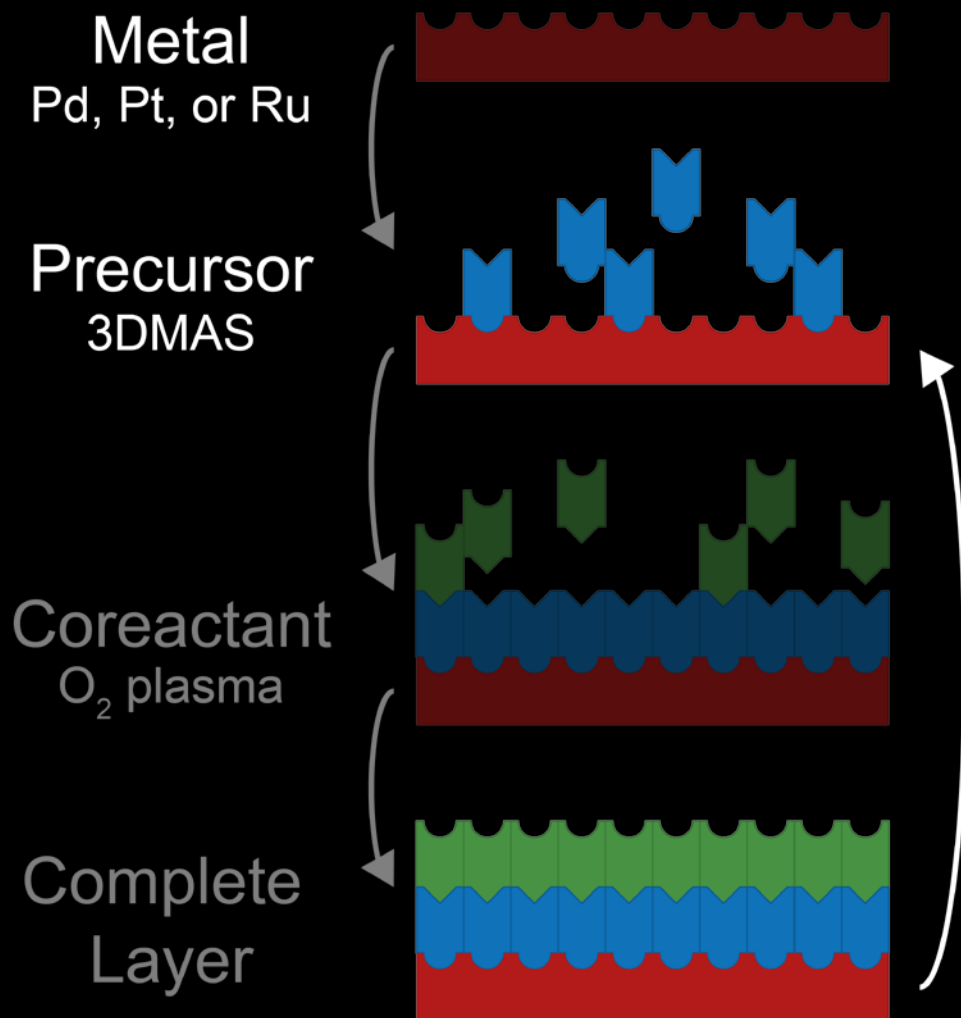
# Atomic Layer Deposition

- **SiO<sub>2</sub> deposited with ALD**
- **Two self-limiting reaction steps**



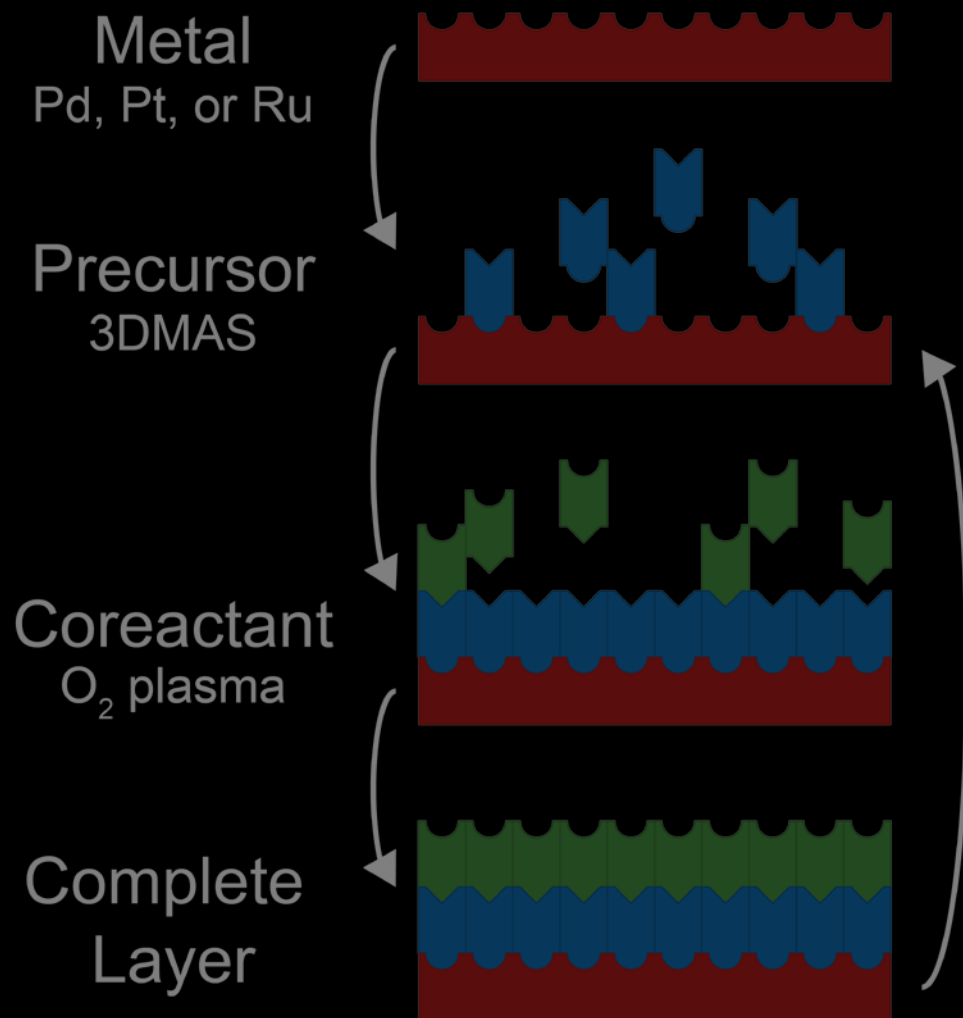
# Atomic Layer Deposition

- **SiO<sub>2</sub> deposited with ALD**
- **Two self-limiting reaction steps**



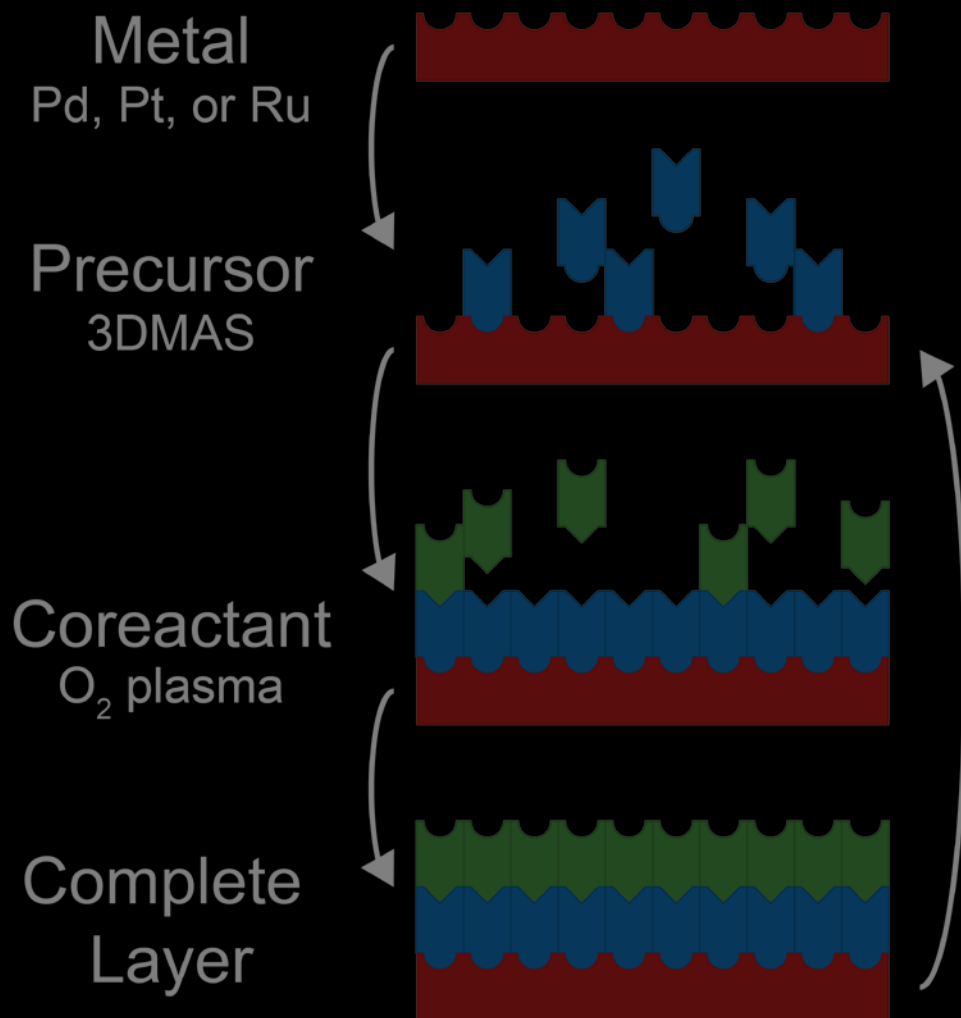
# Atomic Layer Deposition

- **SiO<sub>2</sub> deposited with ALD**
- **Two self-limiting reaction steps**
- **Å-level thickness control**



# Atomic Layer Deposition

- $\text{SiO}_2$  deposited with ALD
- Two self-limiting reaction steps
- Å-level thickness control
- High conformality
- High quality



# Contact Angle

- **Small bead of liquid placed on each sample's surface**

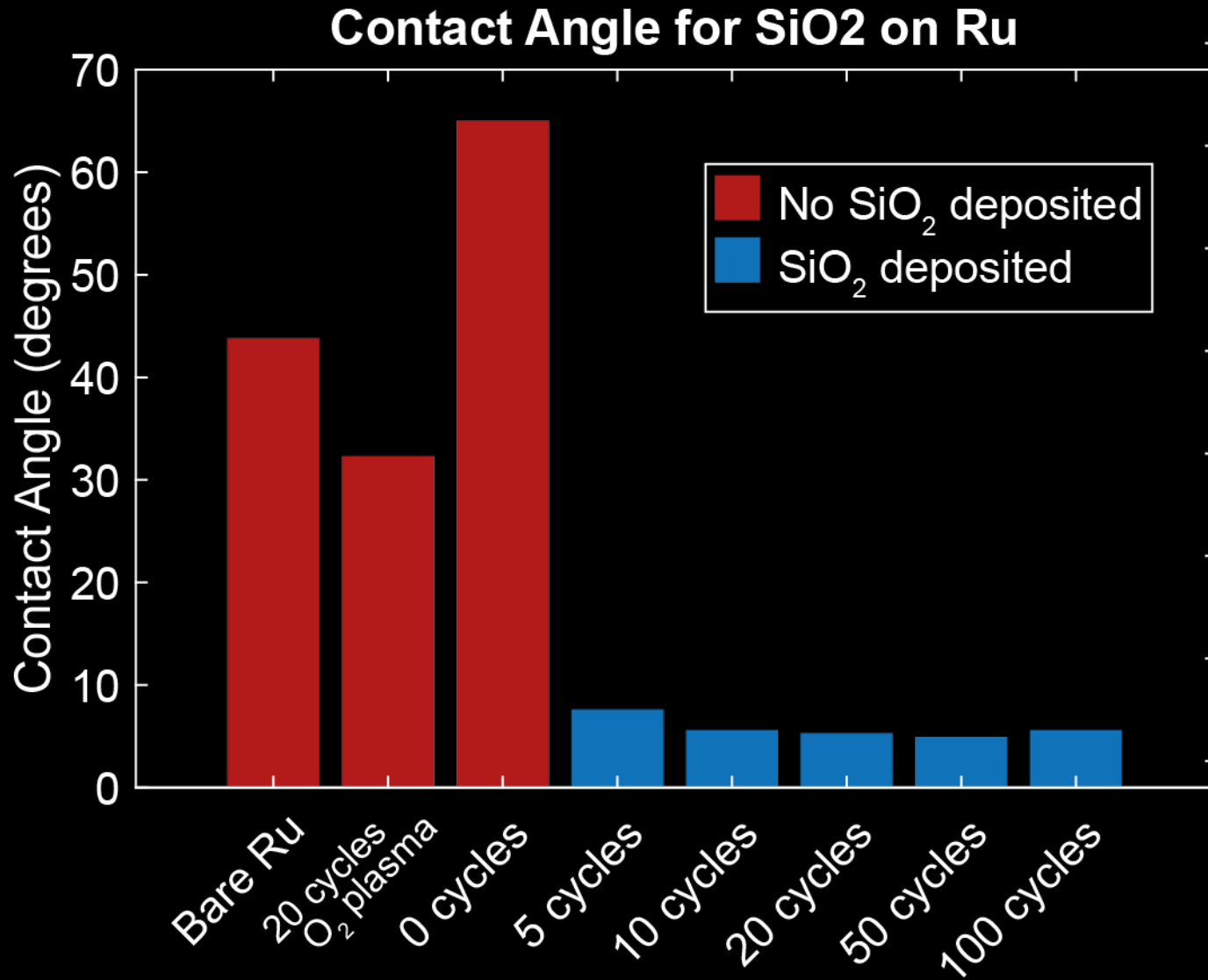


# Contact Angle

- Small bead of liquid placed on each sample's surface
- Angle of bead's contact with the sample measured



# Contact angle data



# Ellipsometry

- **Polarized light shined on sample**



# Ellipsometry

- Polarized light shined on sample
- Change in polarization measured
  - Angle and phase



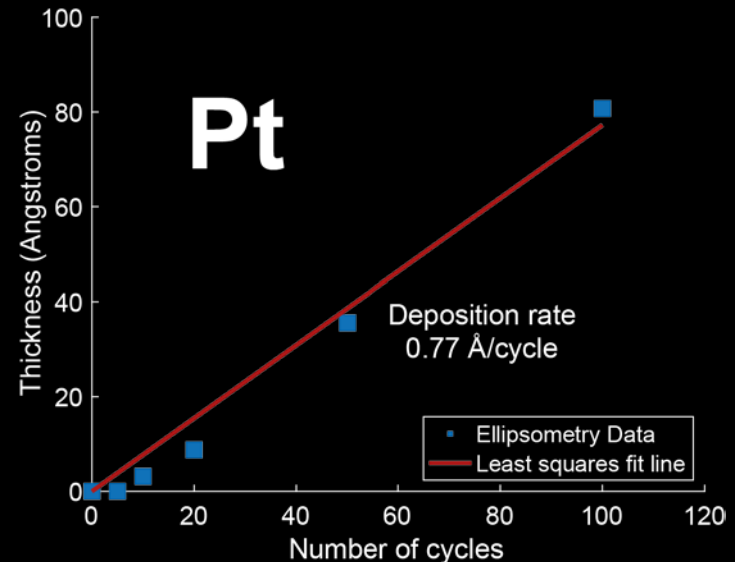
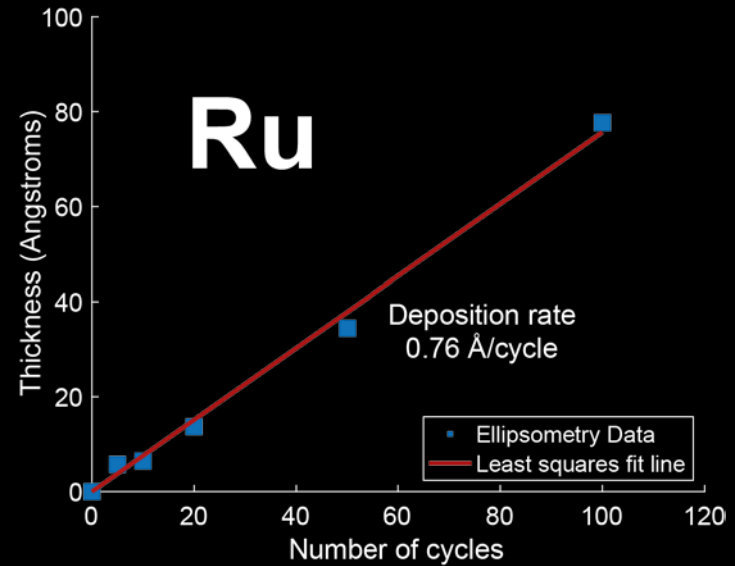
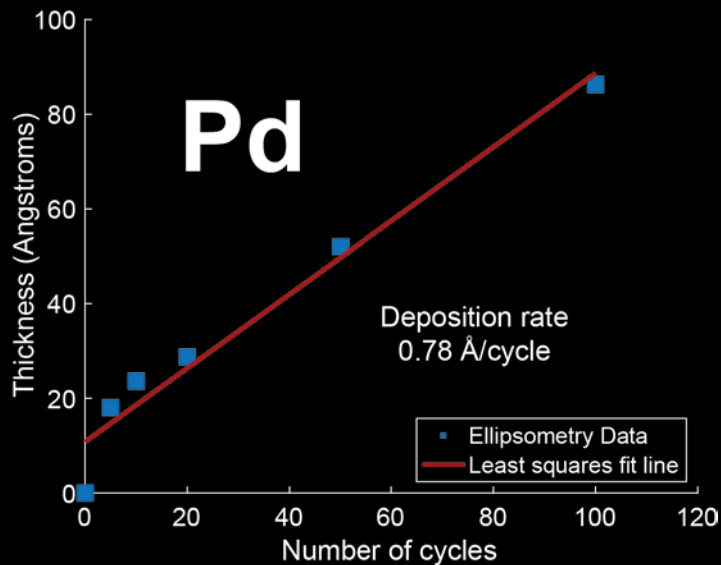
# Ellipsometry

- Polarized light shined on sample
- Change in polarization measured
  - Angle and phase
- Film thickness computed
  - Å-precision



# Ellipsometry data

- Growth rate of  $\sim 0.77 \text{ \AA/cycle}$



# Annealing

- **Normal ALD behavior observed**



# Annealing

- Normal ALD behavior observed
- **No silicatene**

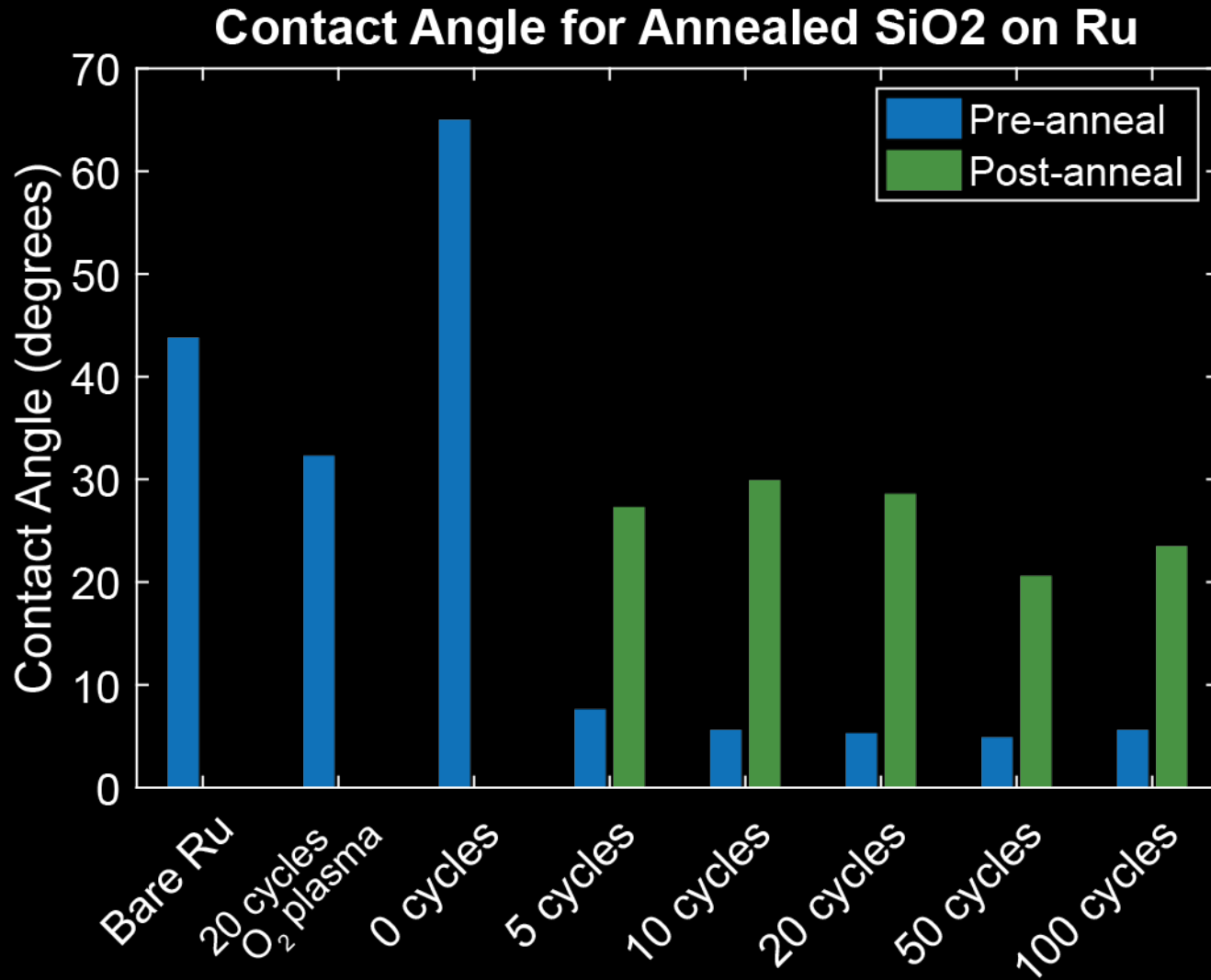


# Annealing

- Normal ALD behavior observed
- No silicatene
- To mimic established process, anneal for 10 minutes at 800° C



# Contact angle



# X-ray Photoelectron Spectroscopy

- X-ray beam incident on each sample

Incident X-rays



Sample



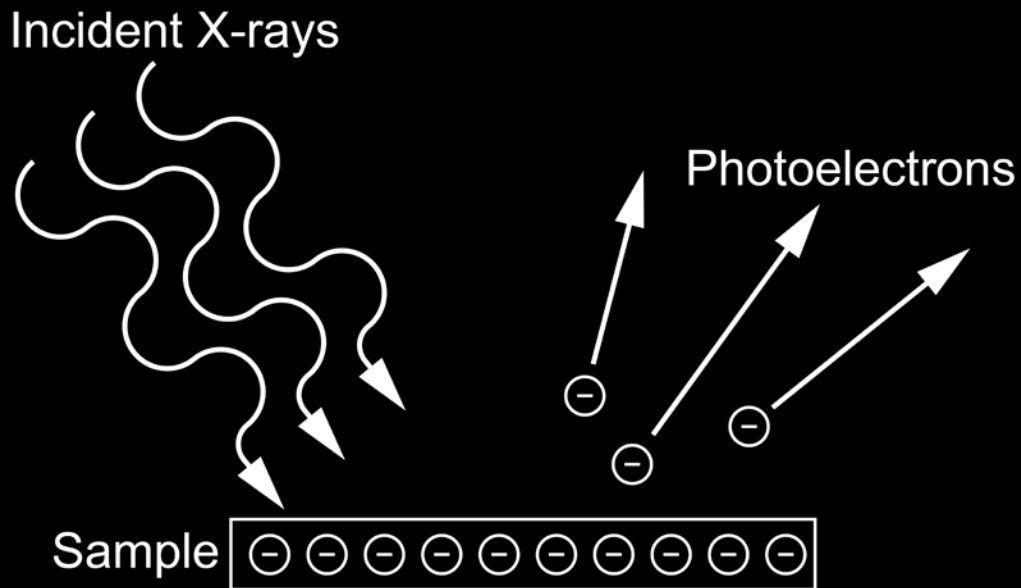
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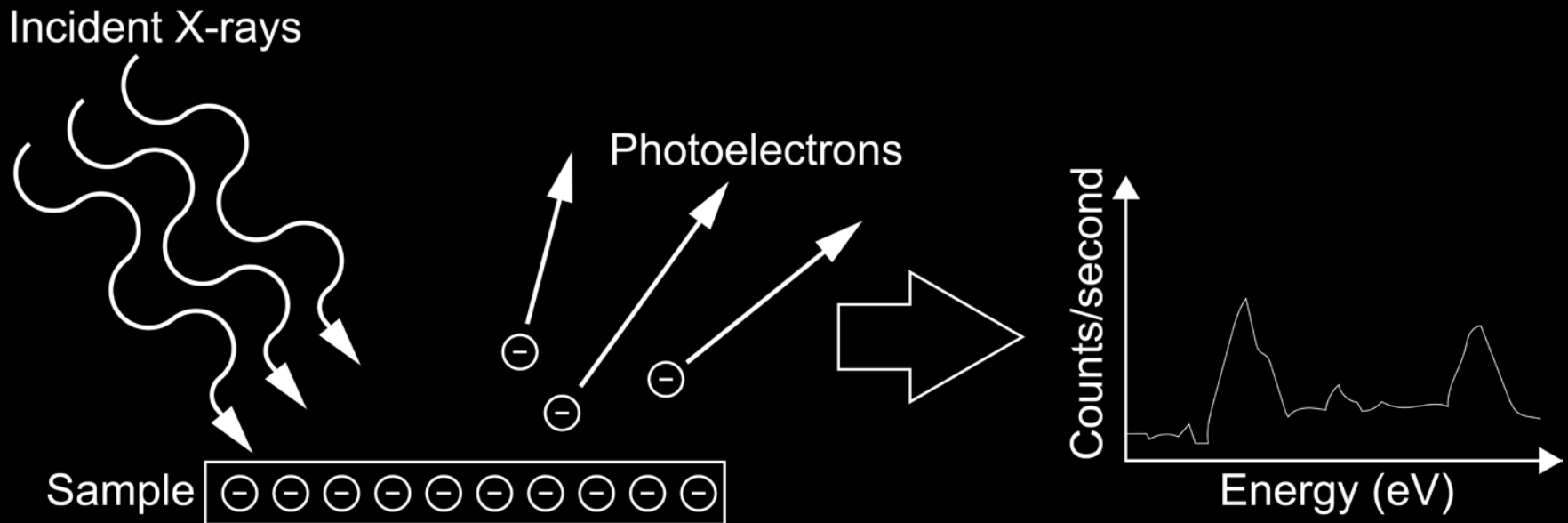
# X-ray Photoelectron Spectroscopy

- X-ray beam incident on each sample
- Electrons ejected



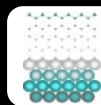
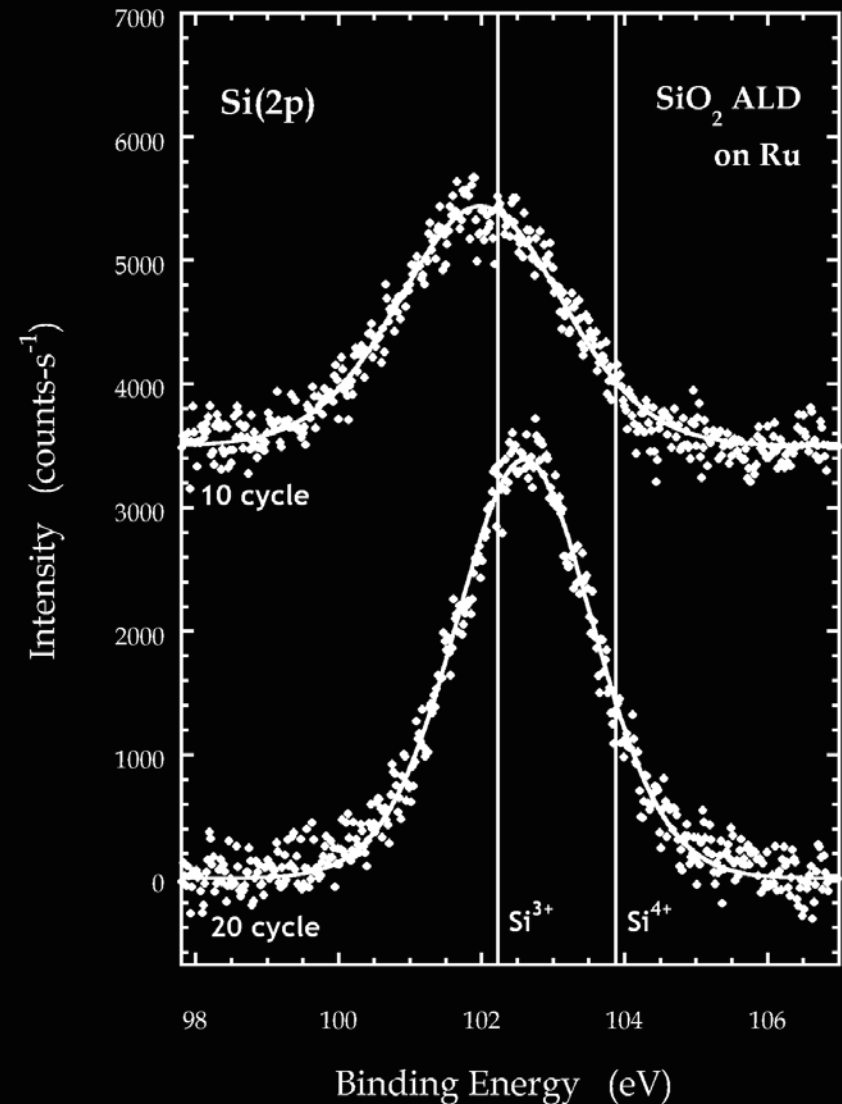
# X-ray Photoelectron Spectroscopy

- X-ray beam incident on each sample
- Electrons ejected
- Elemental composition and binding state found



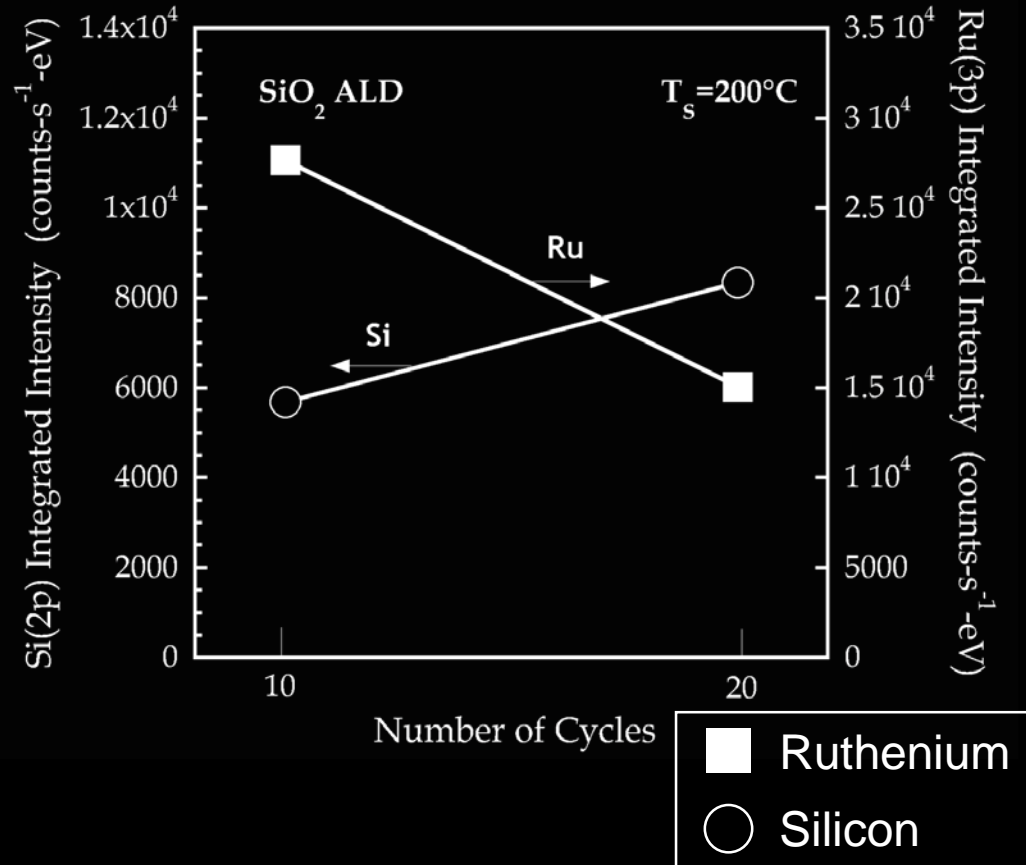
# X-ray Photoelectron Spectroscopy

- **More Si(2p) observed with more cycles**
- **Oxidation state of Si can be found**



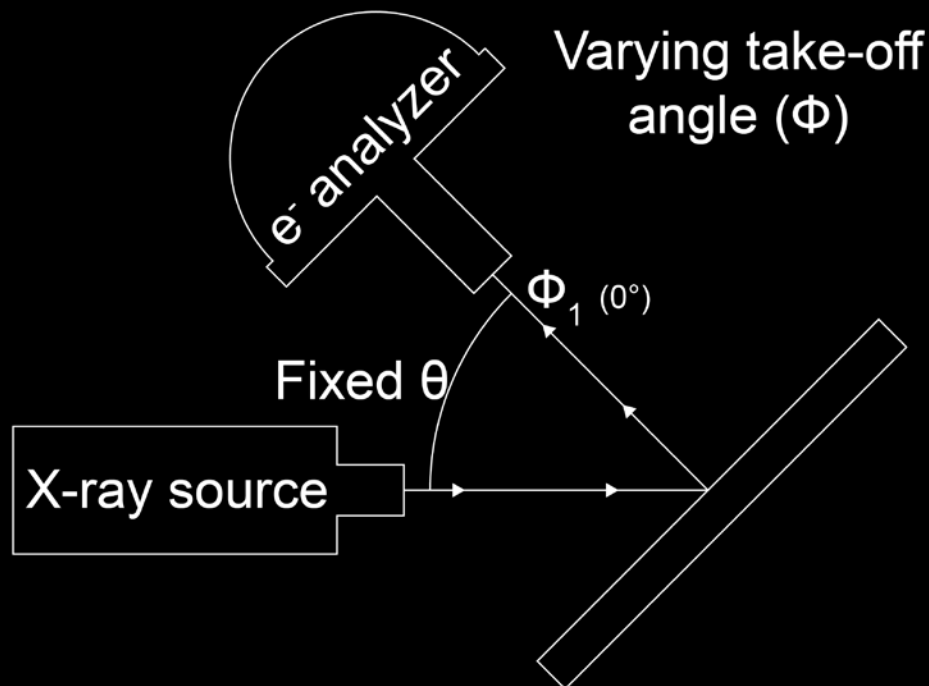
# X-ray Photoelectron Spectroscopy

- **Attenuation of substrate signal with thicker film**



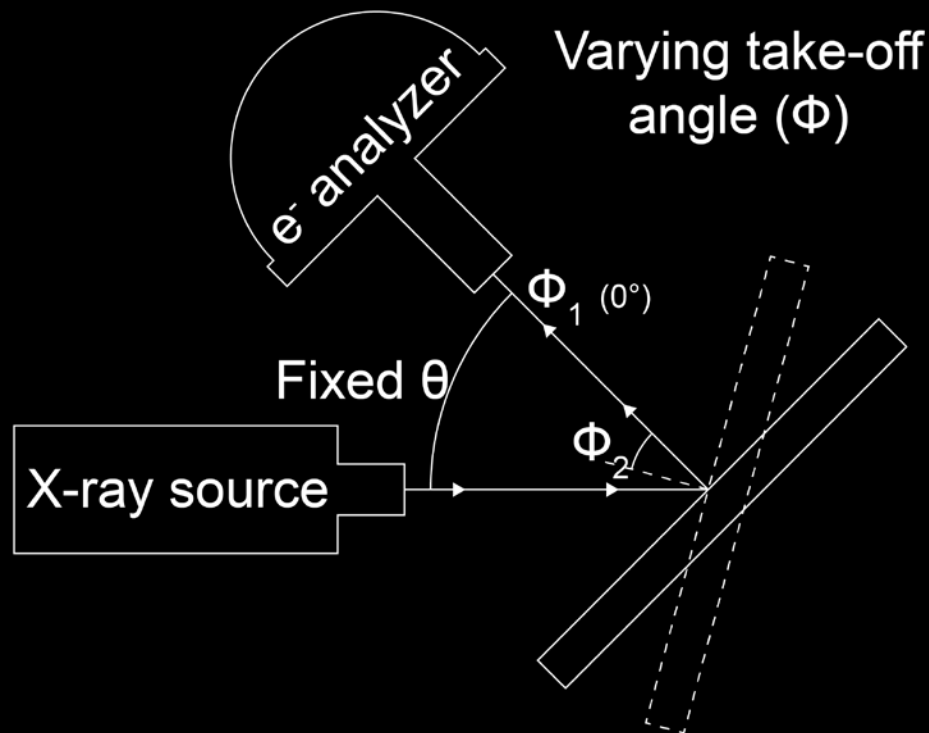
# Angle Resolved XPS

- XPS conducted at several take-off angles



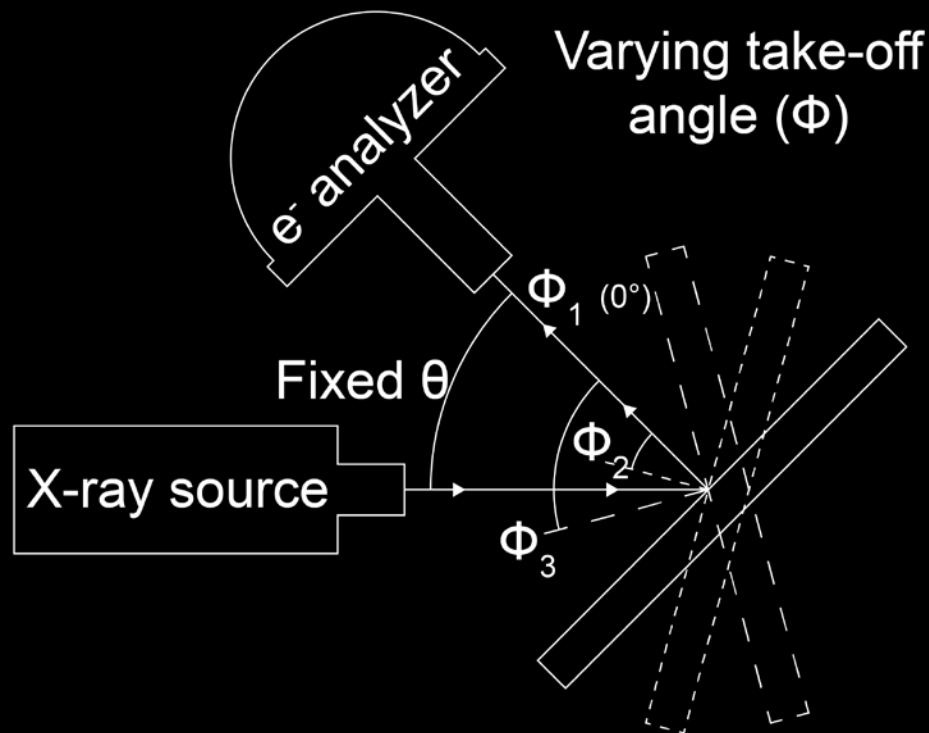
# Angle Resolved XPS

- XPS conducted at several take-off angles



# Angle Resolved XPS

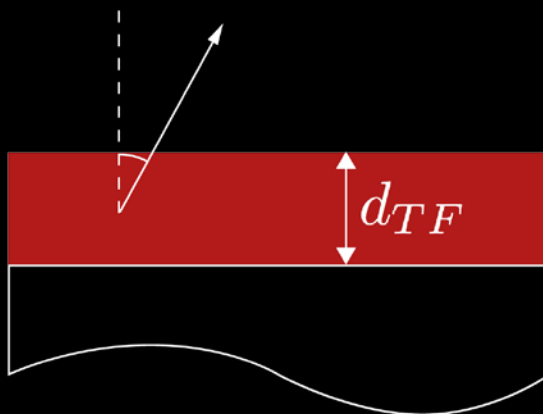
- XPS conducted at several take-off angles



# Angle Resolved XPS

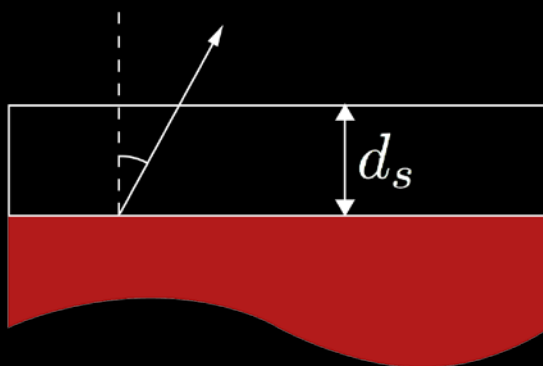
- Models based on sample structure

Thin Film



$$I(\theta) = I_0 \left[ 1 - \exp \left( -\frac{d_{TF}}{\lambda \cos \theta} \right) \right]$$

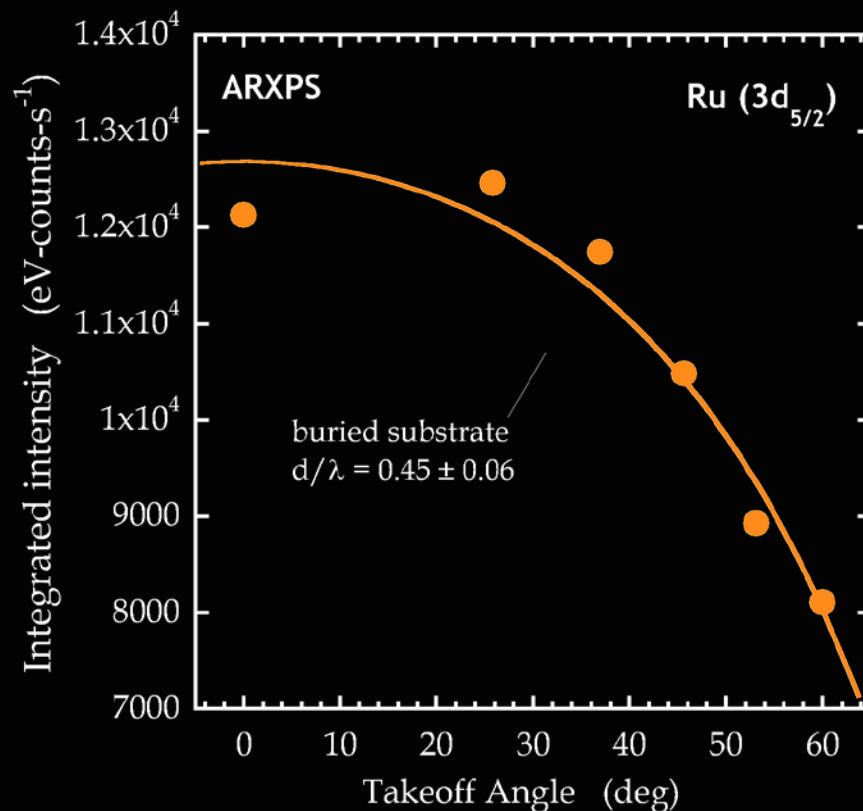
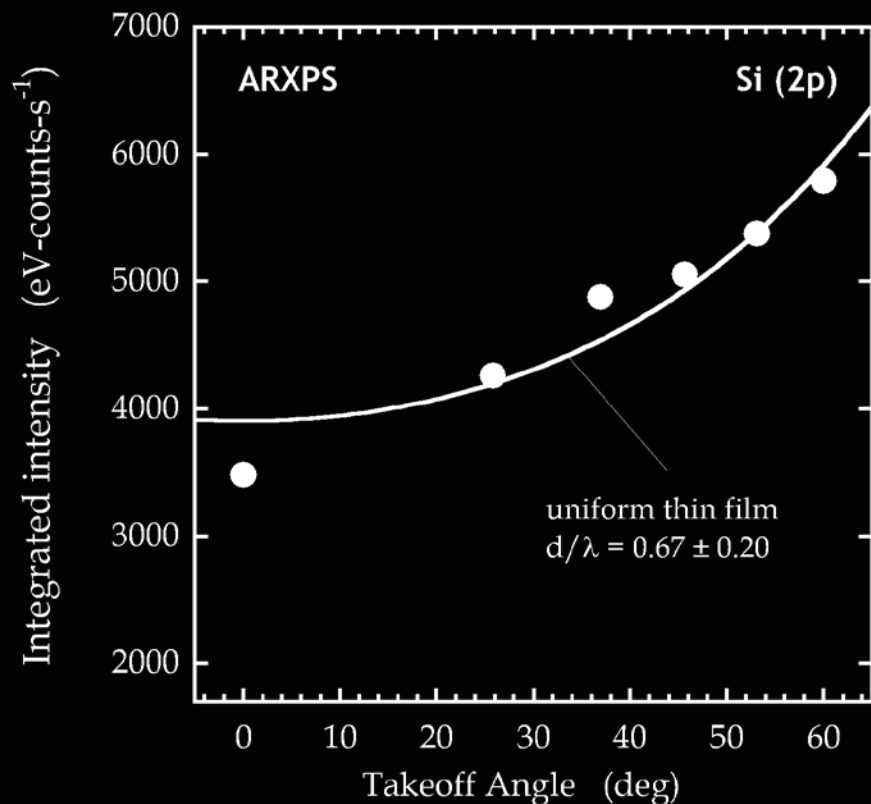
Buried  
Substrate



$$I(\theta) = I_0 \exp \left( -\frac{d_s}{\lambda \cos \theta} \right)$$

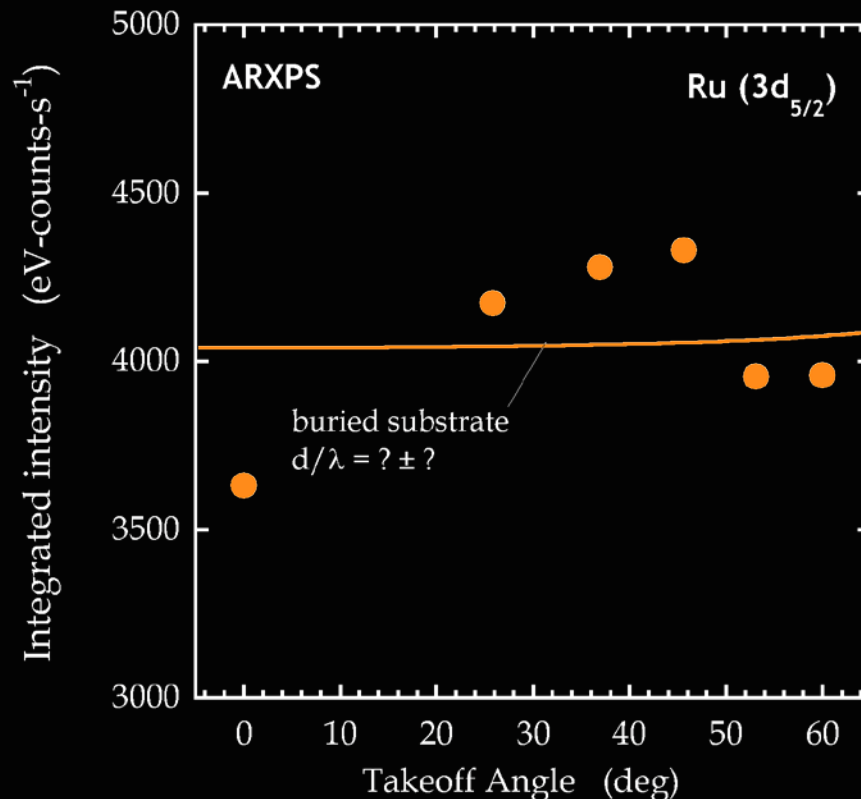
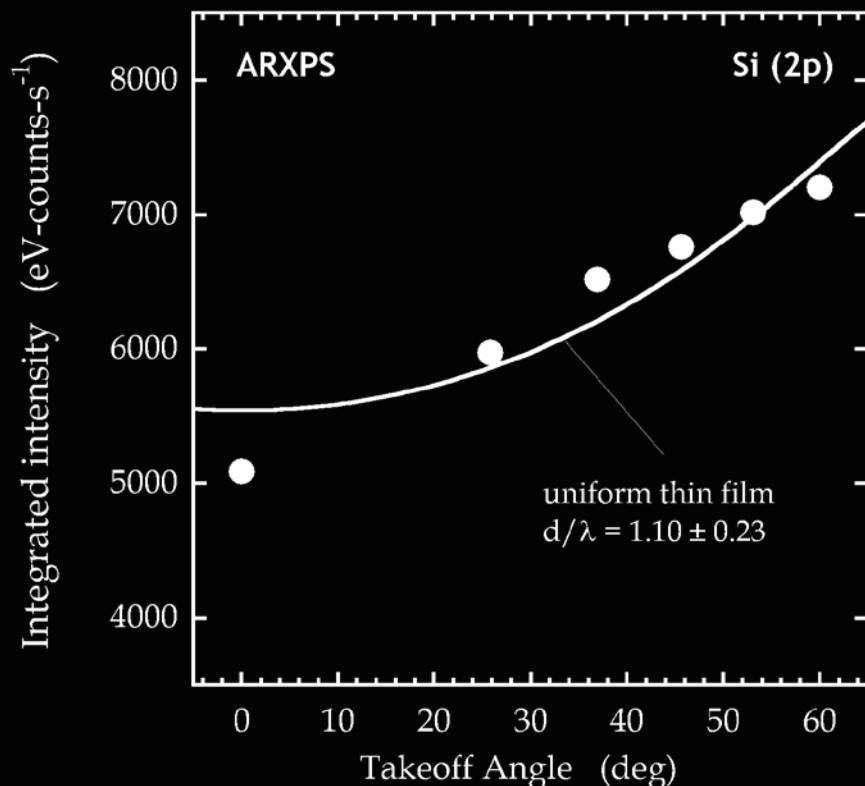
# Angle Resolved XPS

## Before annealing



# Angle Resolved XPS

## After annealing



# Conclusion

- **SiO<sub>2</sub> was deposited**



# Conclusion

- $\text{SiO}_2$  was deposited
- **Silicatene was not fabricated**



# Conclusion

- $\text{SiO}_2$  was deposited
- Silicatene was not fabricated
- **Future work**
  - Single crystal metal



# References

1. Büchner, C., & Heyde, M. (2017). Two-dimensional silica opens new perspectives. *Progress in Surface Science*, 92(4), 341-374.  
doi:10.1016/j.progsurf.2017.09.001
2. George, S. M. (2010). Atomic Layer Deposition: An Overview. *Chemical Reviews*, 110(1), 111-131.  
doi:10.1021/cr900056b
3. Petersson, C., Baglin, J., Dempsey, J., D'Heurle, F., & La Placa, S. (1985). Silicides of ruthenium and osmium: Thin film reactions, diffusion, nucleation, and stability. *Vacuum*, 35(6), 237. doi:10.1016/0042-207x(85)90531-7



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- **Cornell NanoScale Science & Technology Facility**
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- **Taewon Suh**
- **CNF REU Program Coordinator & Staff**
  - **Aaron Windsor**
  - **Jeremy Clark**
  - **Alan Bleier and Woollam staff**



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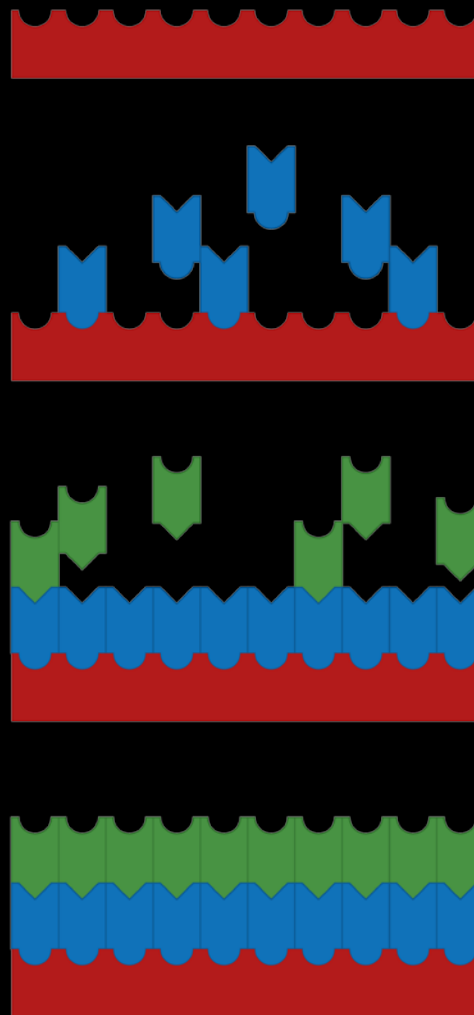


Metal  
Pd, Pt, or Ru

Precursor  
3DMAS

Coreactant  
 $O_2$  plasma

Complete  
Layer



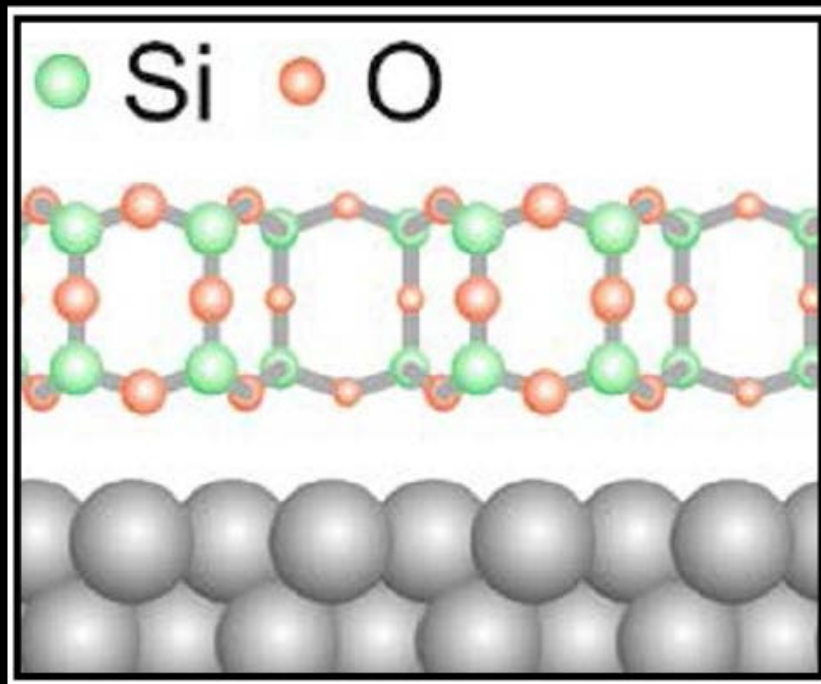


Figure from Büchner et al.