Techniques and Instrumentation at the Materials Research Lab Central Research Facilities

Kathy Walsh, Senior Research Scientist

(with many contributions from the rest of the MRL staff)

ILLINOIS
Materials Research Laboratory
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Illinois Materials Research Lab

- ~ 50,000 sq.ft. labs
- ~ 150 tools
- 24 technical staff members
- User facility

(next door to Loomis)

Tours and general questions:
mrl-facilities@illinois.edu

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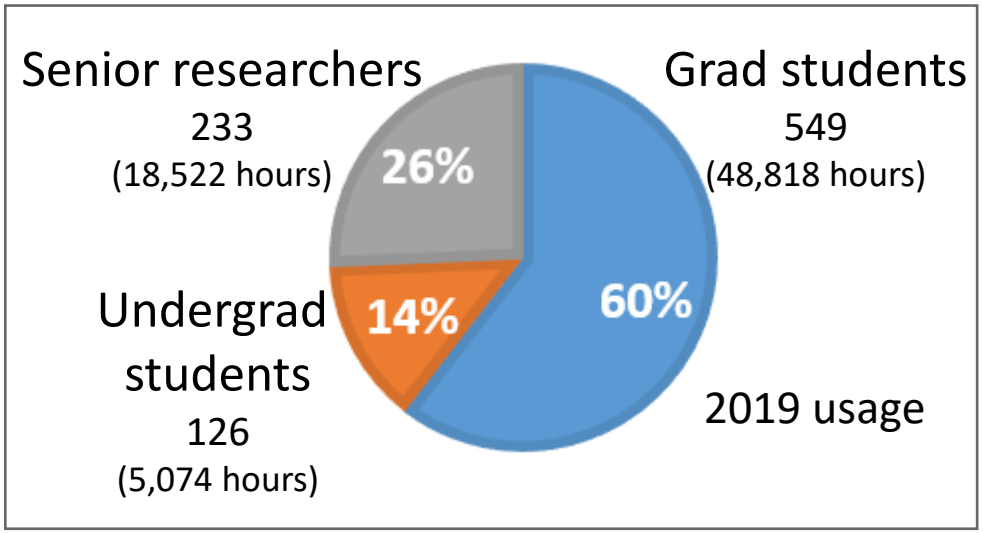
Illinois Materials Research Lab

- Open 24/7 to researchers at all levels
- Academic, government, and industrial



Submit proposal → be trained → do science
go.illinois.edu/MRLorientation

Tours and general questions:
mrl-facilities@illinois.edu

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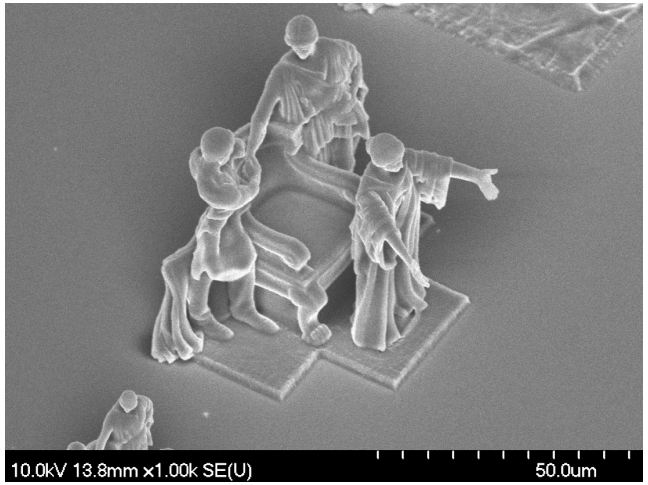
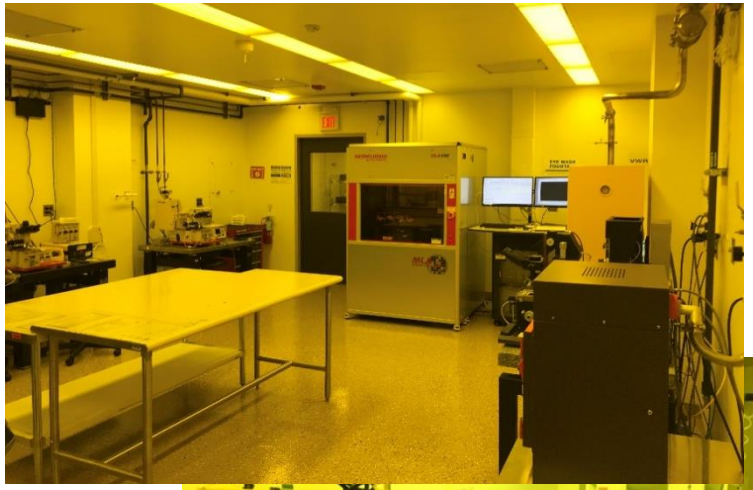
Materials Research

- Fabrication
  - Microfabrication (cleanroom lithography)
  - Nanoscale 3D printing
- Characterization
  - Atomic scale to centimeter scale
  - Morphology of samples
  - Composition of samples
  - Behavior of samples



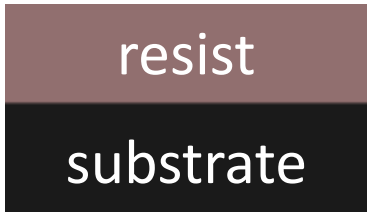
Microfabrication

- Often used for devices
- Cleanroom (not dusty)
- Deposit light- or electron-sensitive resist
- Expose and develop pattern
  - UV light
  - electrons (smaller)
- Deposit material
- Remove resist
- Alternatively, etch away material



Lithography

1. Deposit resist



2. Expose a pattern; develop (remove) resist

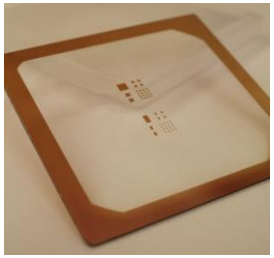
3. Deposit material (gold, etc.)

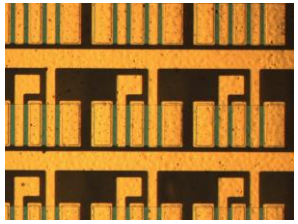
4. Liftoff (remove remaining resist)

Lithography

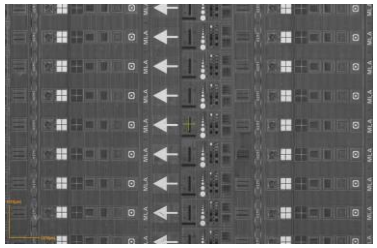
- Photolithography (UV photons)
  - Shine light through a mask

MJB4 aligner





  - Maskless photolithography (direct writing on wafers)

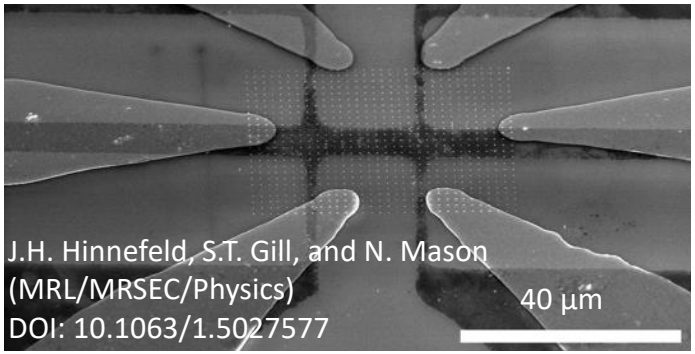


Heidelberg MLA150

- E-beam lithography (electrons)

Already have: Raith eLine
Coming soon: Raith EBPG 100 kV

Raith eLine



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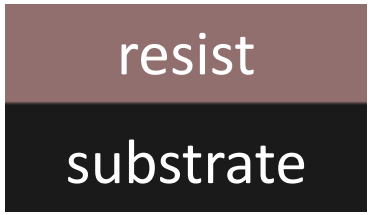
Lithography

1. Deposit resist



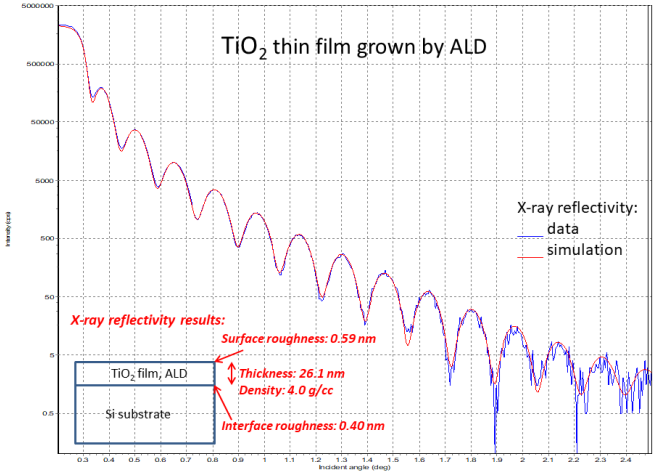
2. Expose a pattern; develop (remove) resist

3. Deposit material (gold, etc.)

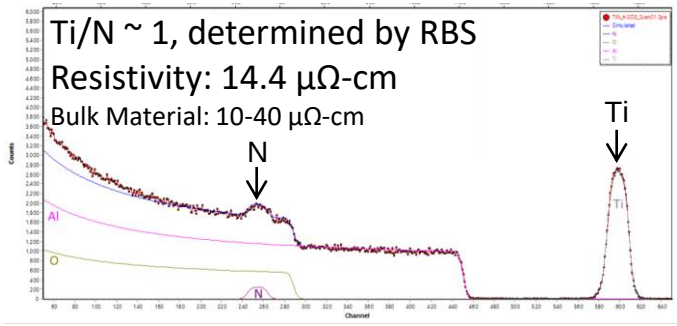
4. Liftoff (remove remaining resist)

Thin Film Deposition

- Metals, oxides, nitrides, etc.
- Atomic Layer Deposition
- Magnetron Sputtering
- Thermal Evaporation
- E-beam Evaporation
- 2D Nanomaterial Growth
- Parylene Coating



Titanium Nitride sputtered on Sapphire



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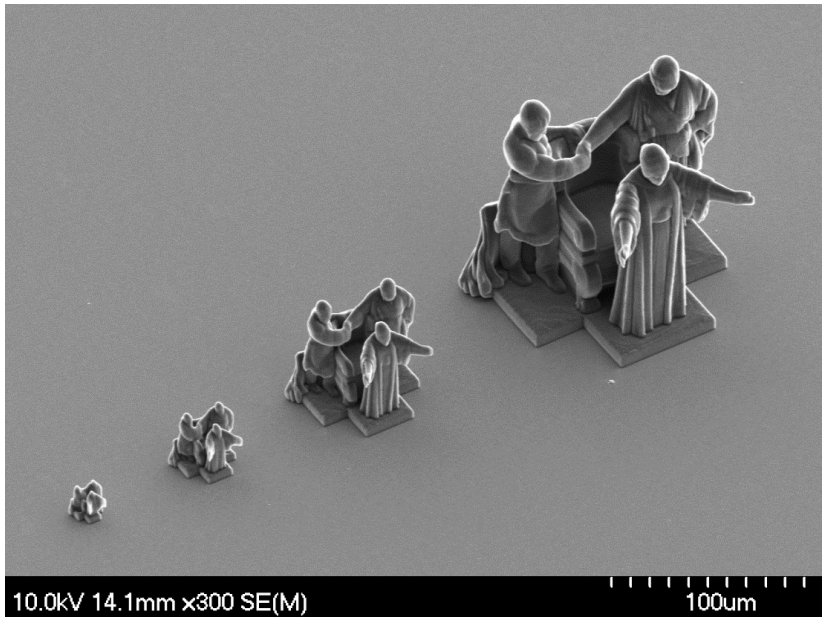
Nanoscale 3D Printing

- Feature sizes down to 250 nm
- 200 µm x 200 µm field of view for high resolution
- 400 µm x 400 µm FOV or stitching possible
- Photoresist
- Write on silicon wafers, fused silica, quartz, or sapphire



Fabricated using the Nanoscribe GT
Imaged using the Hitachi S4800 SEM

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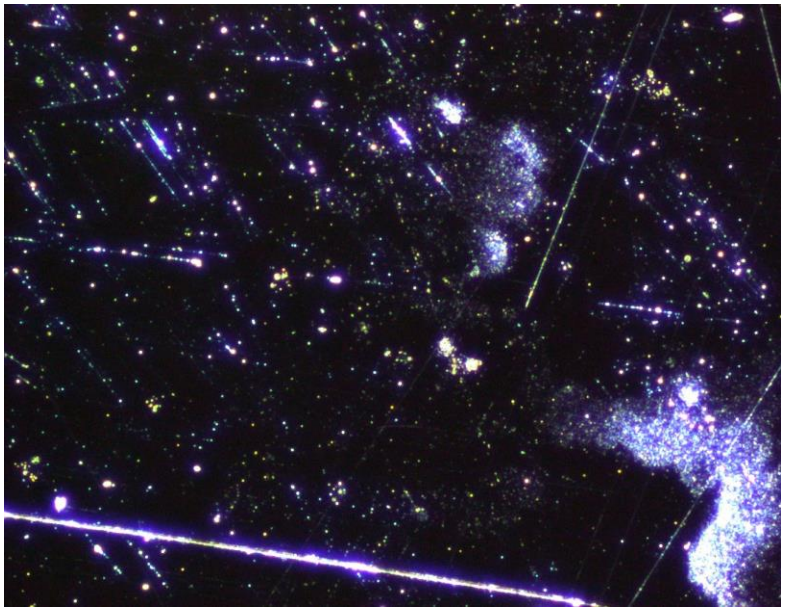
Materials Characterization

- What does it look like?
- What is it made of?
- How does it behave?

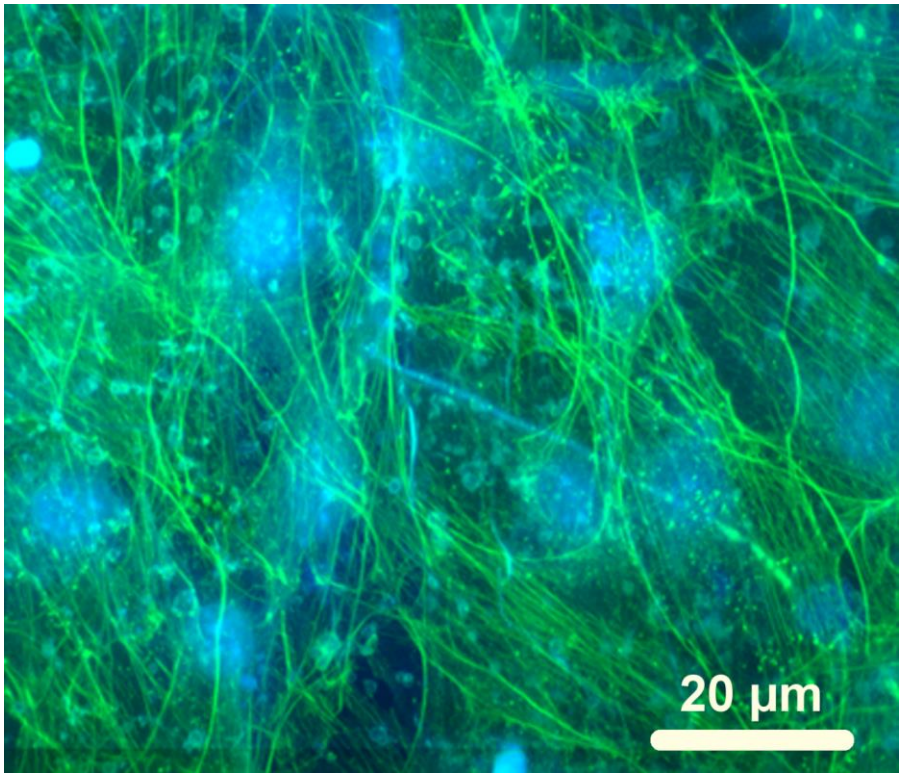
What Does It Look Like?

- Optical Microscopies
- Scanning Electron Microscopy
- Transmission Electron Microscopy
- Atomic Force Microscopy

Optical Microscopies



steel hardness calibration standard
(dark field)



fibroblasts network on epoxy
confocal fluorescence image, courtesy of
Joselle McCracken (Nuzzo Group)

bug head
(bright field)

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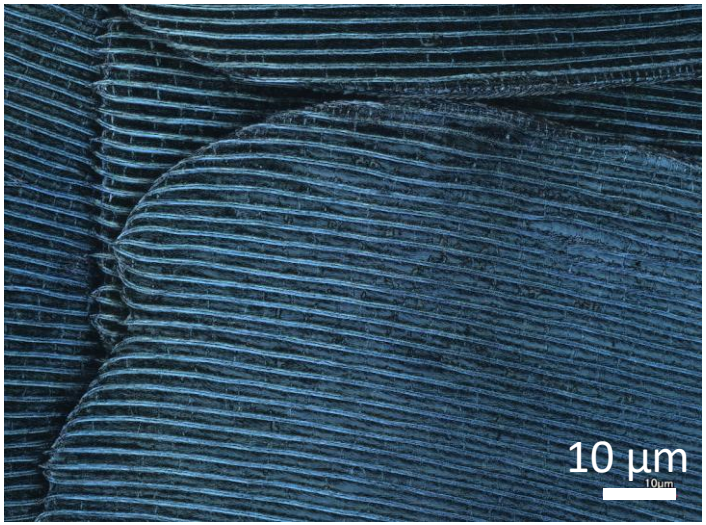
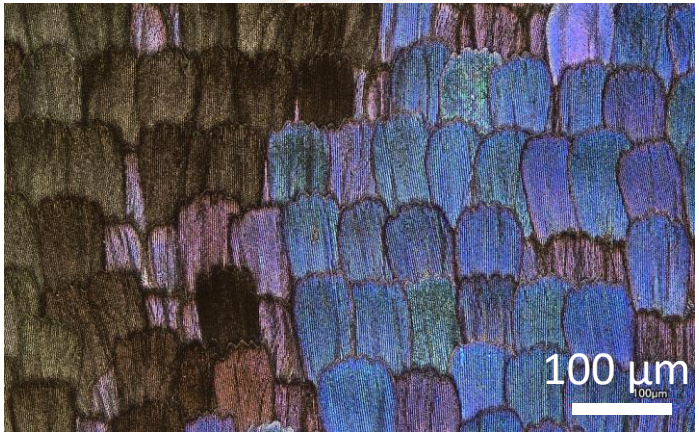
Optical Microscopies

- Ordinary optical microscopy, fluorescent tagging
- Optical sectioning
- Resolution limited by wavelength (λ/2)





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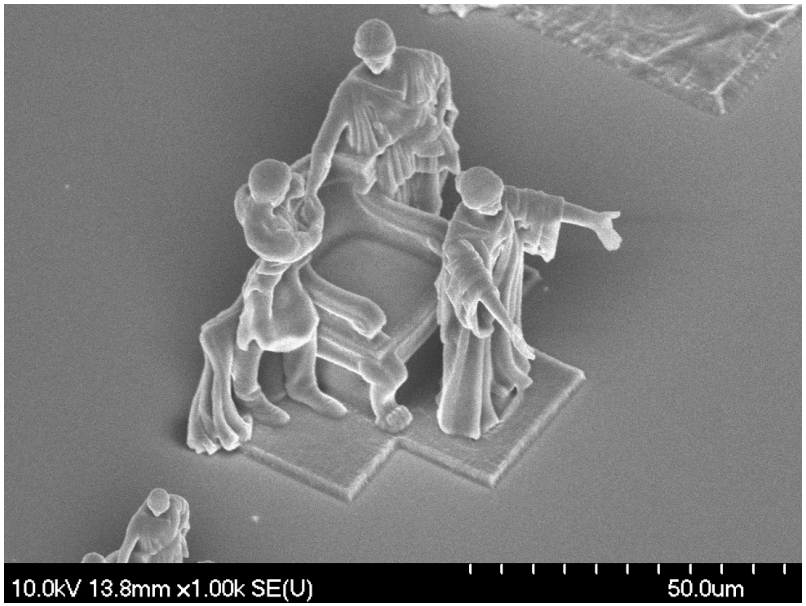
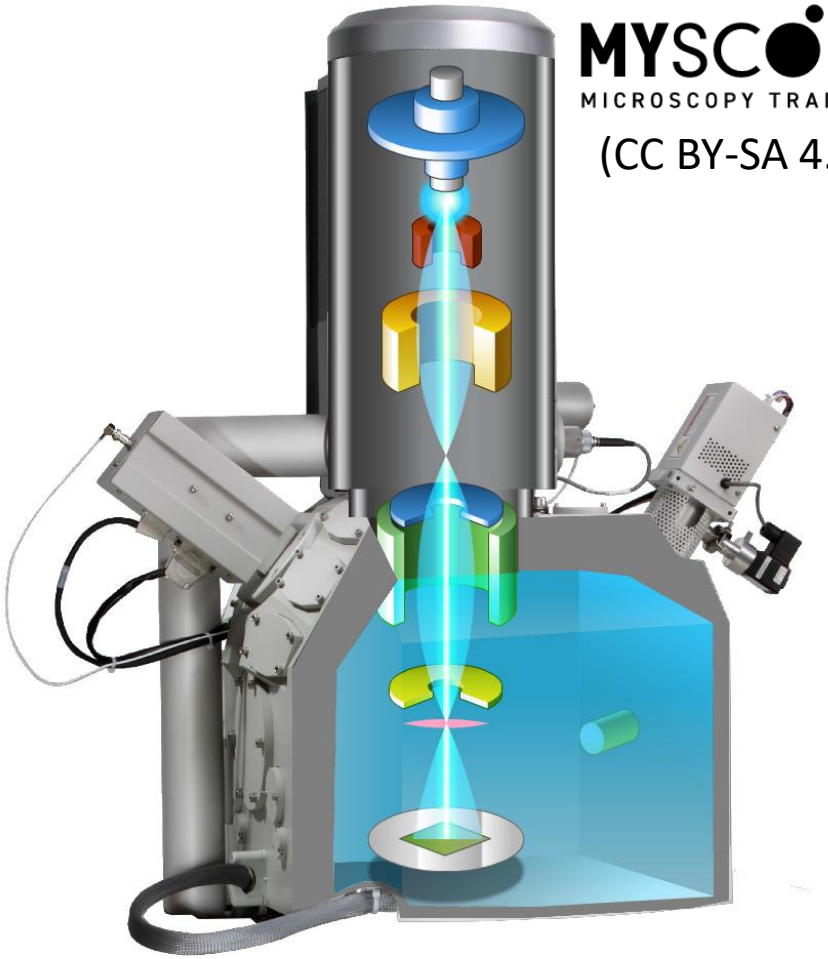
Scanning Electron Microscopy

Scan a beam of electrons back and forth across a sample surface





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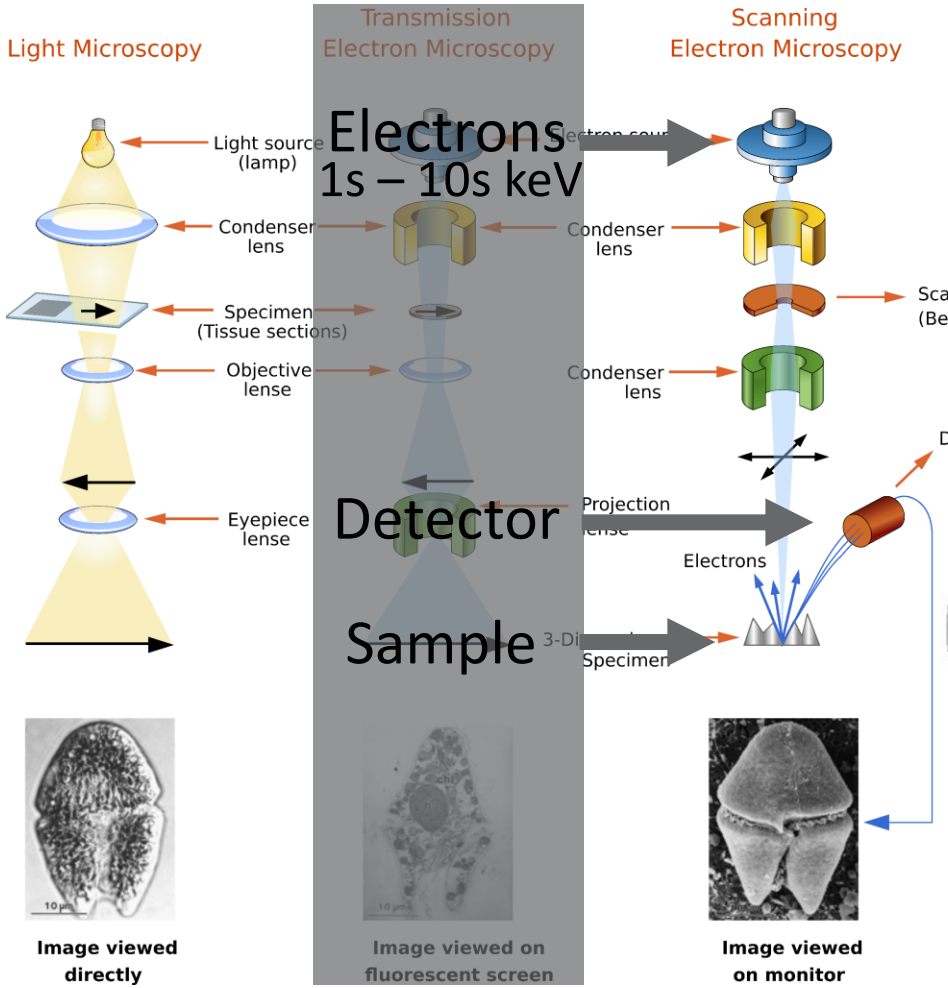
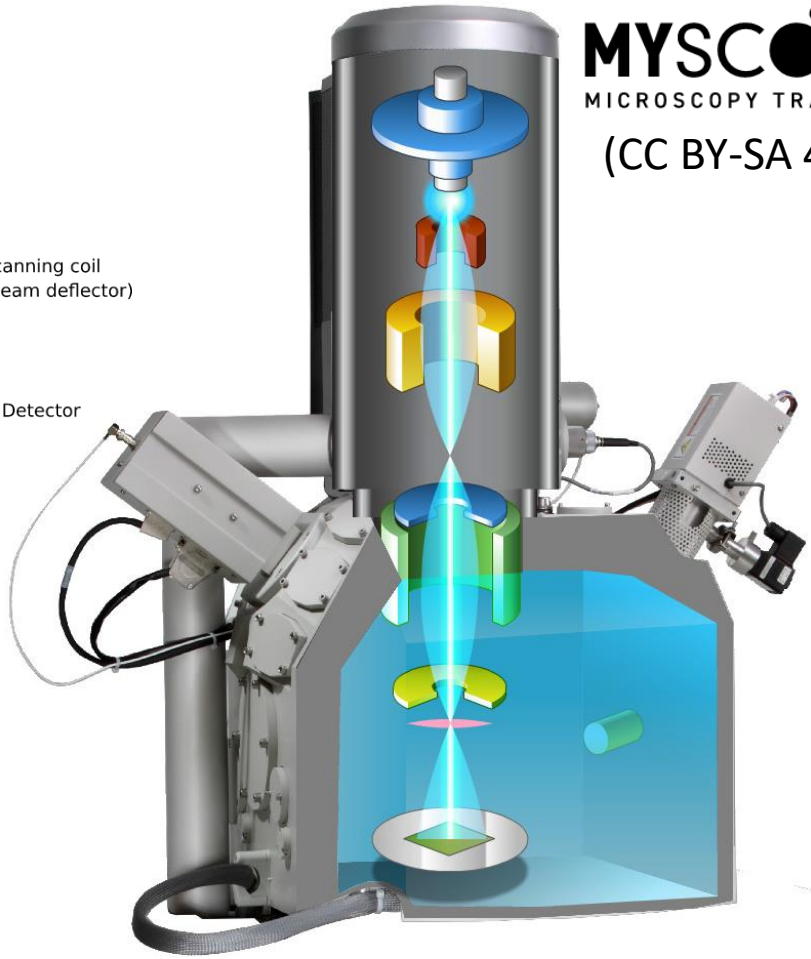
https://myscope.training/#/SEMlevel_3_1

Scanning Electron Microscopy





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https://myscope.training/#/SEMlevel_3_1

Scanning Electron Microscopy

More details next Monday:

Introduction to
Scanning Electron Microscopy

Monday, **April 27th, 11am** central

Dr. Honghui Zhou

MRL Senior Research Scientist

go.illinois.edu/MRLbig4webinars

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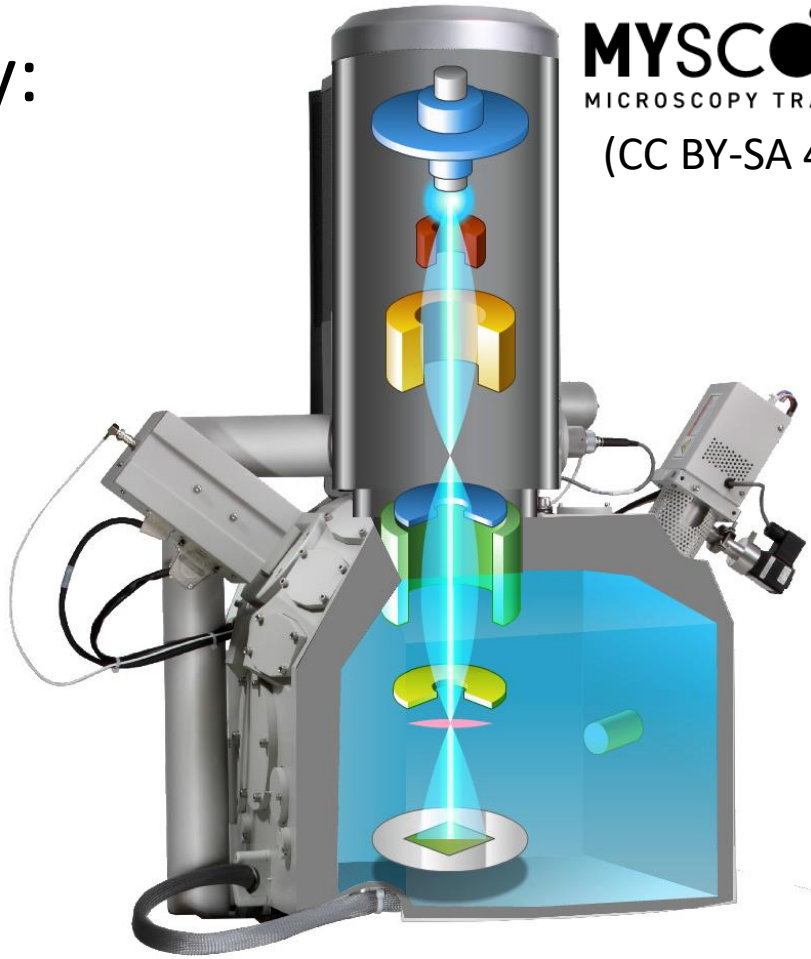
WISCONSIN
UNIVERSITY OF WISCONSIN-MADISON

PennState Materials Research Institute

UNIVERSITY OF MINNESOTA



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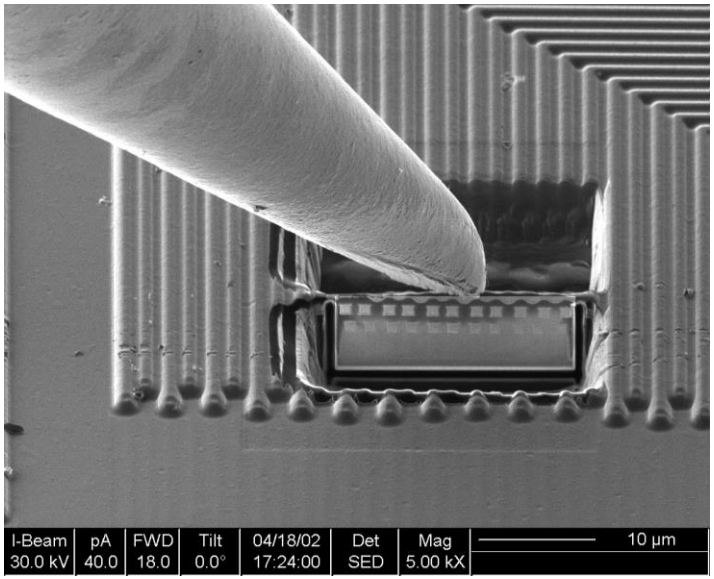
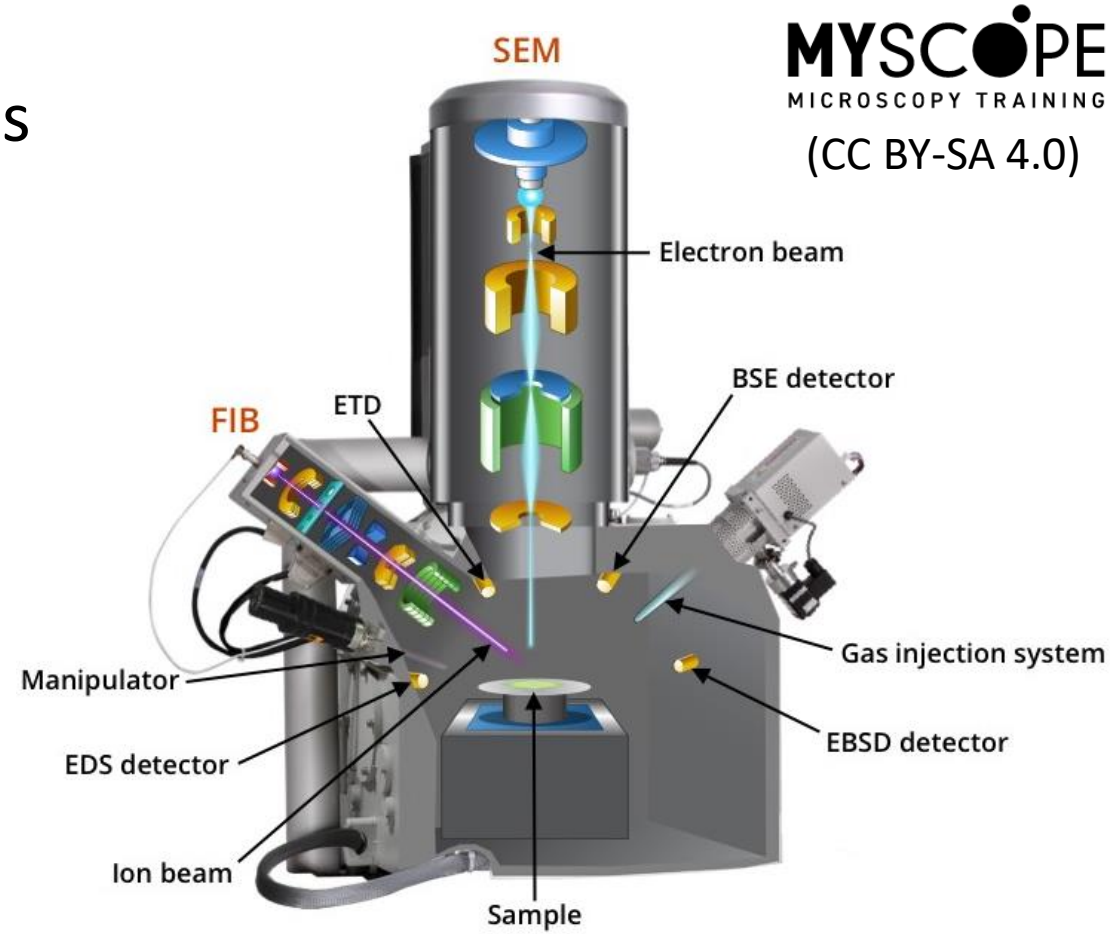
Focused Ion Beam

- Add a column of ions
- Lay down material
- Cut or mill sample



TEM Sample Preparation of Computer Chip Using Liftout Technique
Michael Marshall, CMM



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Transmission Electron Microscopy

https://www.jeol.co.jp/en/science/em.html

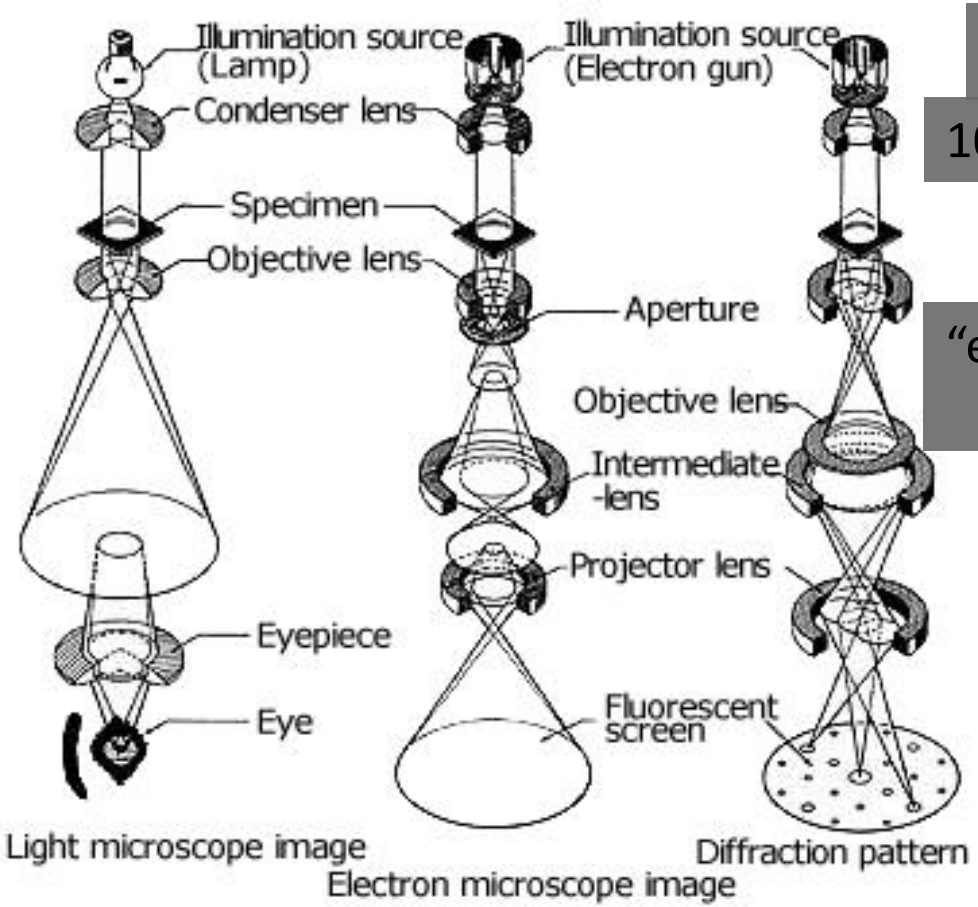


diagram from JEOL
(electron microscope manufacturer)

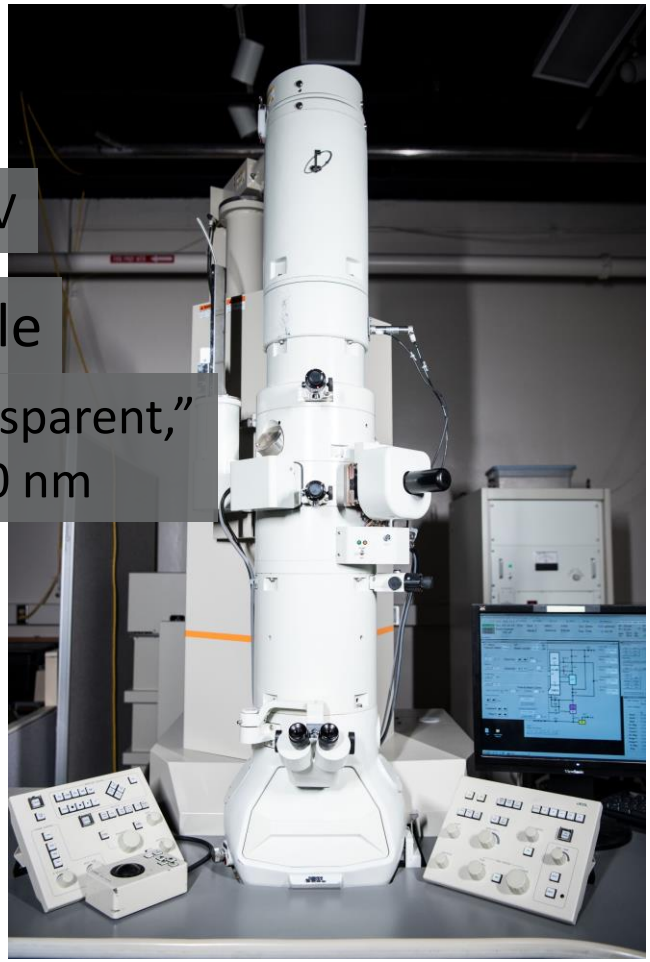
Electrons

10s – 100s keV

Sample

"electron transparent,"
i.e., < 100 nm

Detector

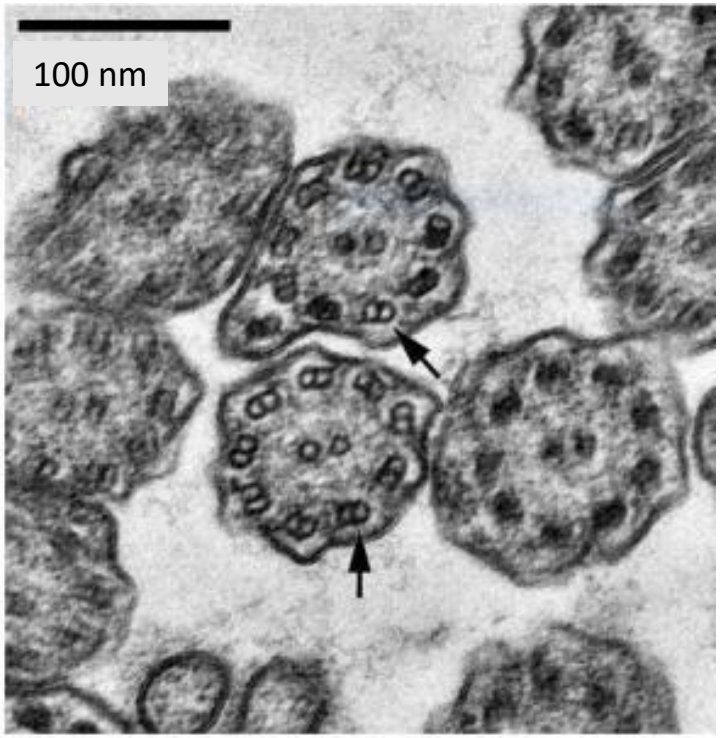


MRL's JEOL 2100 Cryo TEM

Transmission Electron Microscopy

Ultrathin sections of a canine nasal biopsy imaged under standard conditions (200kV). Arrows highlight microtubules inside the cilia.

–Kristen Flatt, MRL staff



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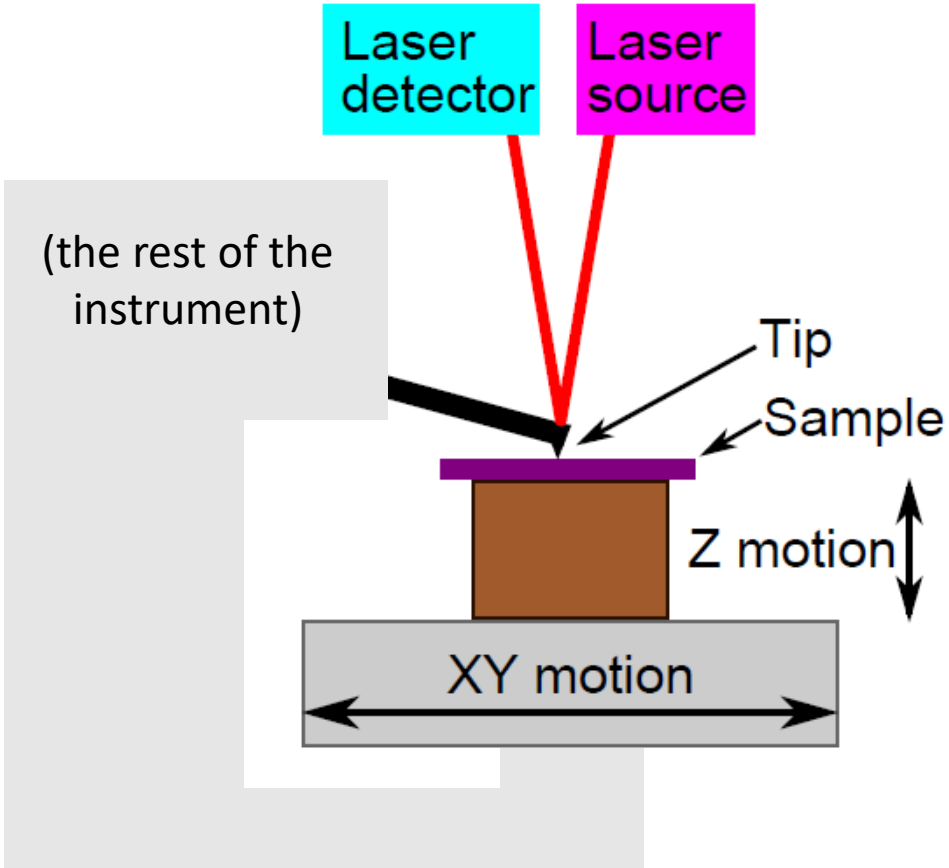
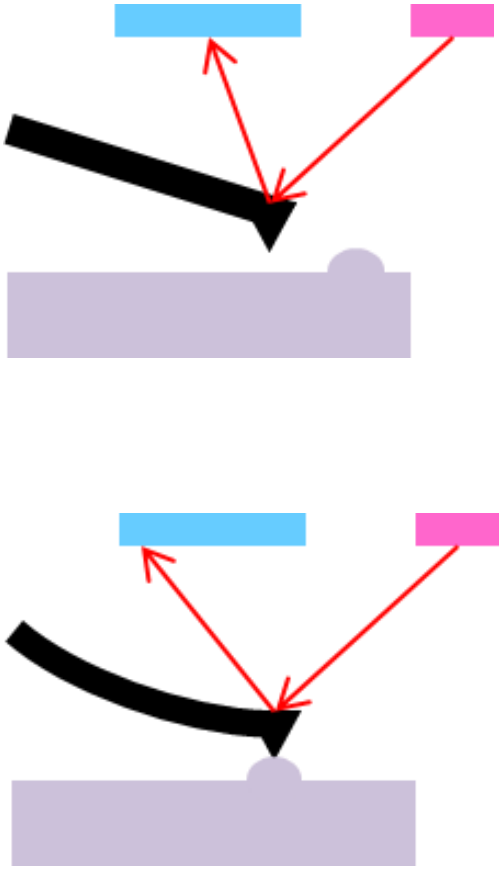
Already have: JEOL 2100 Cryo TEM
Coming soon: Thermo Glacios Cryo-TEM

Atomic Force Microscopy

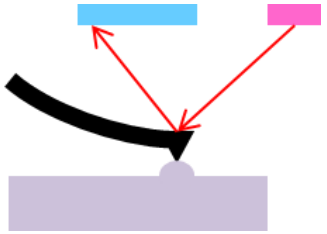


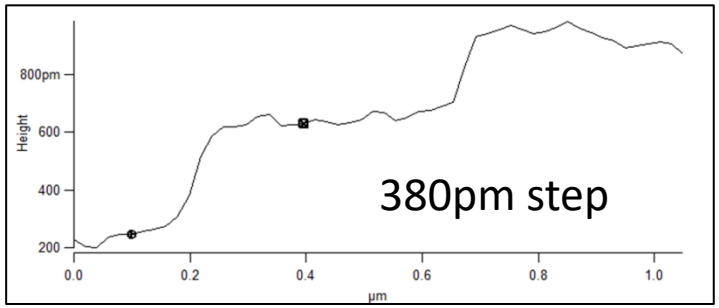


Atomic Force Microscopy

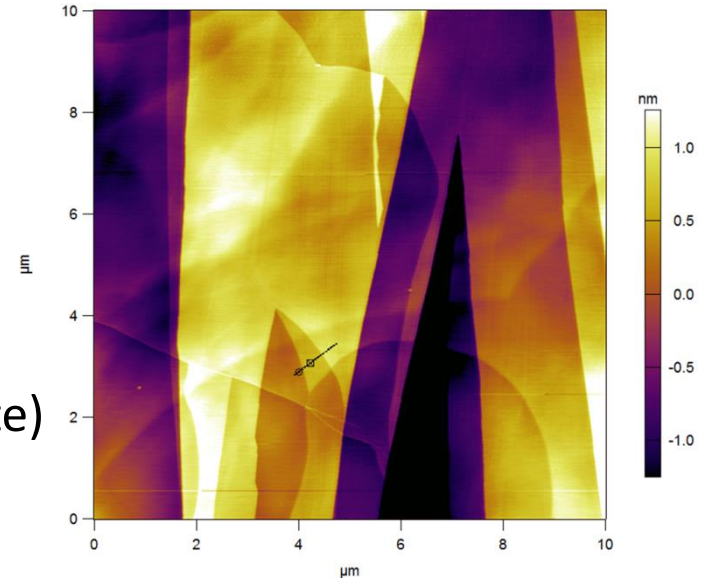
"Basics of Atomic Force Microscopy" webinar

mediaspace.illinois.edu/channel/MRL_Webinars





HOPG (highly oriented pyrolytic graphite)



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What Is It Made Of?

- Surface Analysis
  - X-ray photoelectron spectroscopy
  - Secondary ion mass spectroscopy
  - Atom probe tomography
- Raman spectroscopy
- X-ray analysis
  - Energy dispersive spectroscopy/X-ray fluorescence
  - X-ray diffraction
  - X-ray reflectivity

Compositional Analysis

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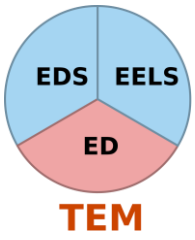
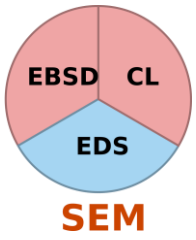
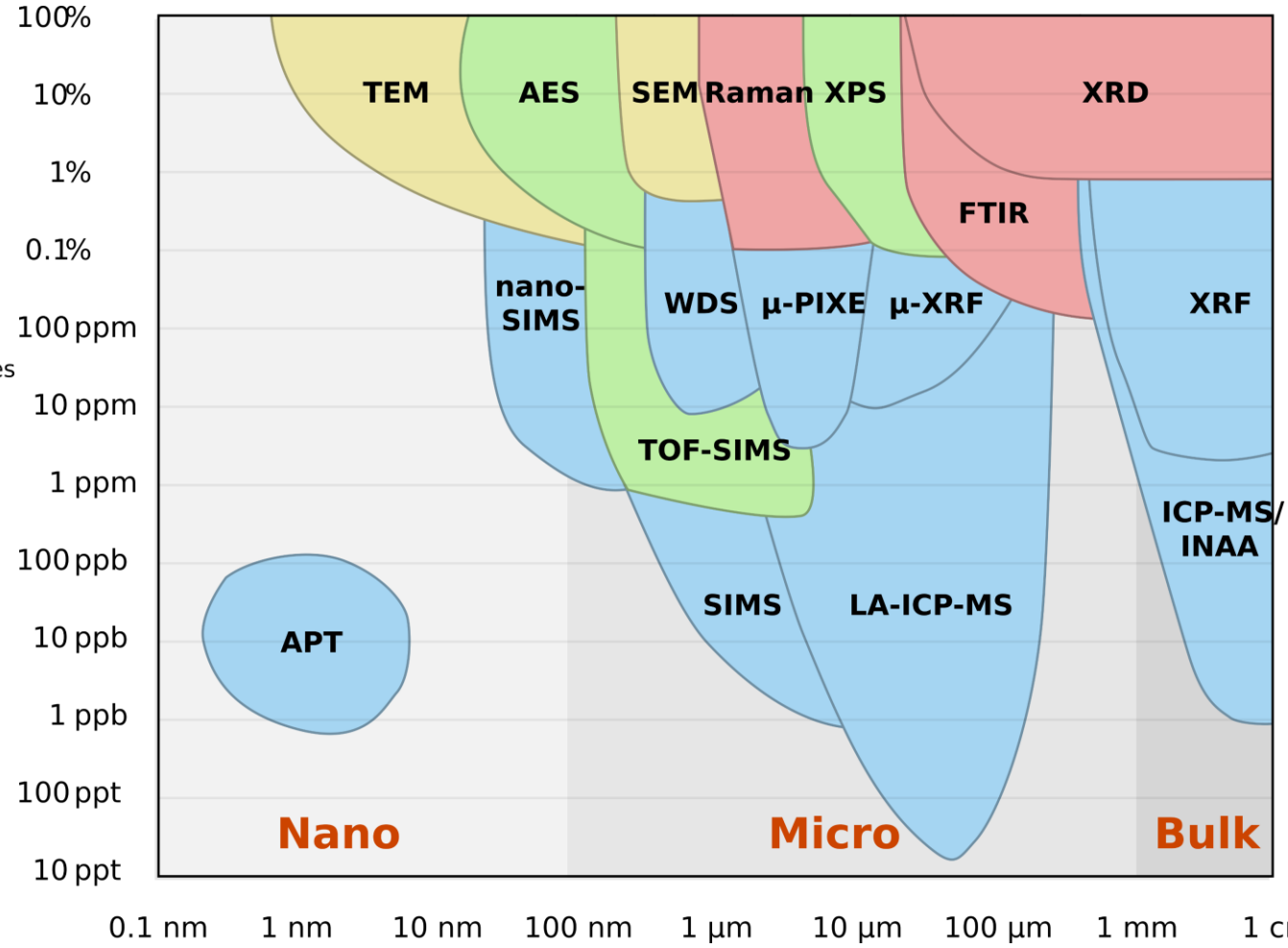
- Elemental composition
- Structural information
- Surface and thin film analysis
- SEM and TEM host multiple techniques







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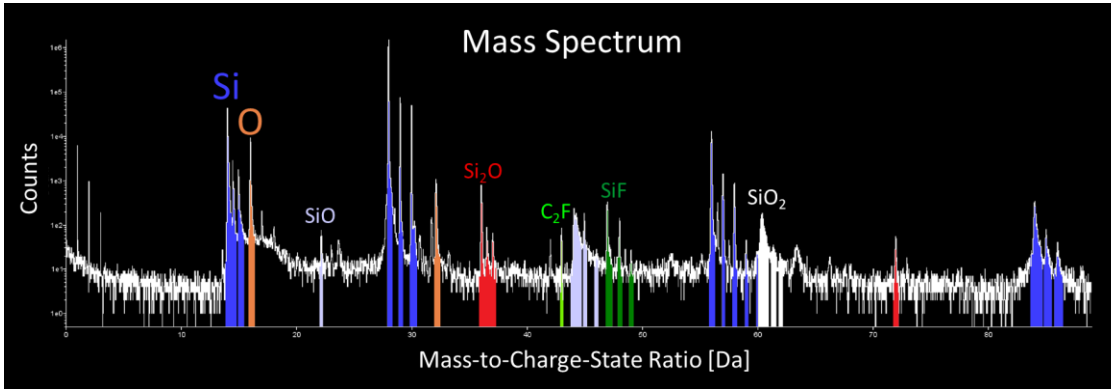
https://myscope.training/#/EDSlevel_2_2

Surface Analysis

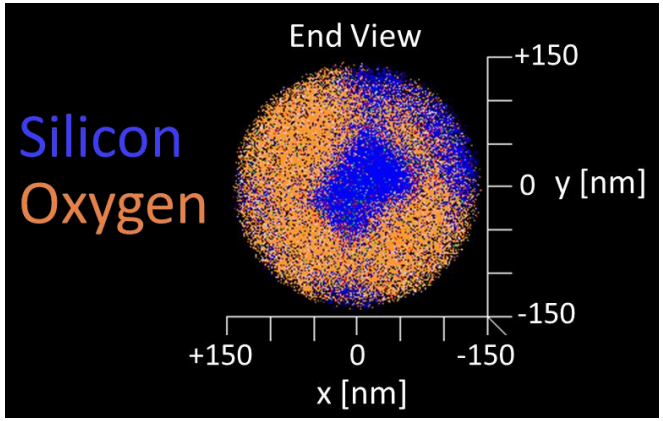
- XPS (X-ray photoelectron spectroscopy)
  - **April 23rd**, noon, **go.illinois.edu/MRLwebinars**
  - X-rays hit sample, knock electrons out
  - Top 10 nm of sample, ~ 1 mm analysis area
  - Chemical information and binding states
- SIMS (secondary ion mass spectrometry)
  - **April 30th**, noon, **go.illinois.edu/MRLwebinars**
  - Ions sputtered from sample, mass spec identifies atoms and molecules

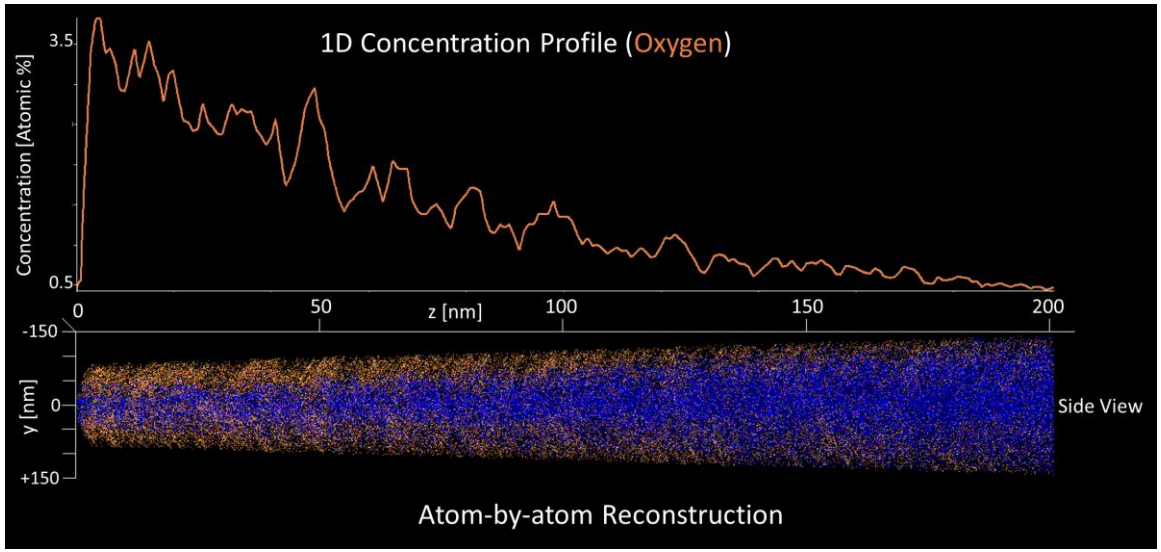
Atom Probe Tomography

go.illinois.edu/AtomProbe



silicon microtip with oxide layer





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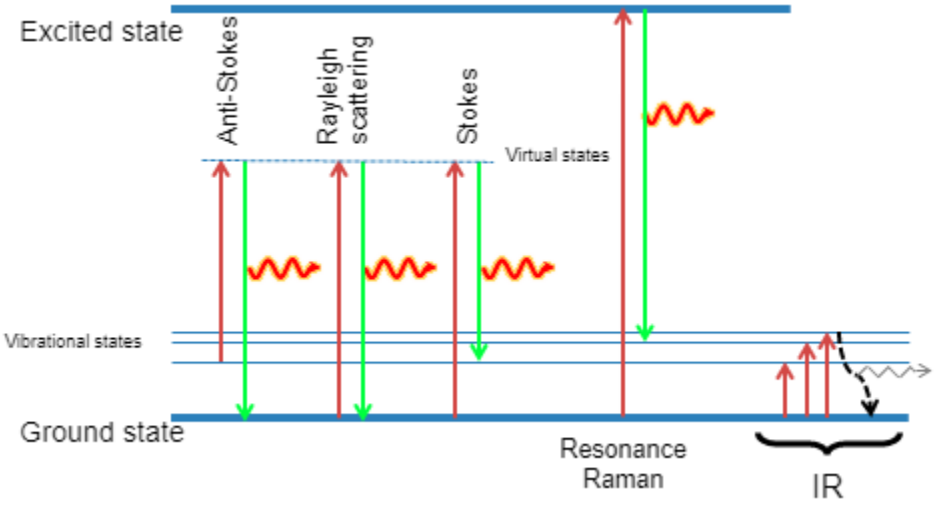
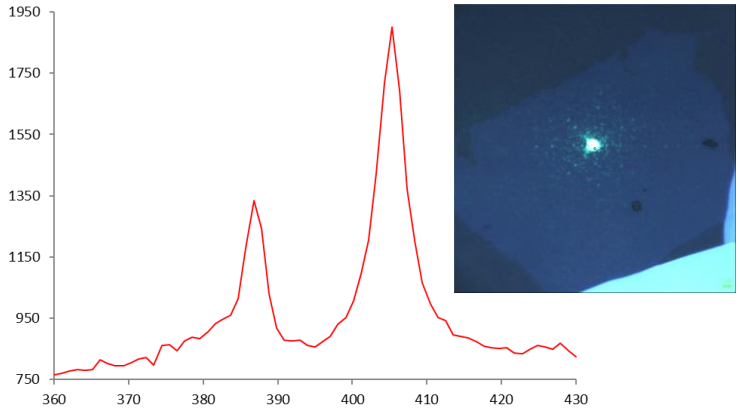
Cameca LEAP 5000XS

Raman Spectroscopy

- Inelastic light scattering
- Laser excites phonons, molecular vibrations



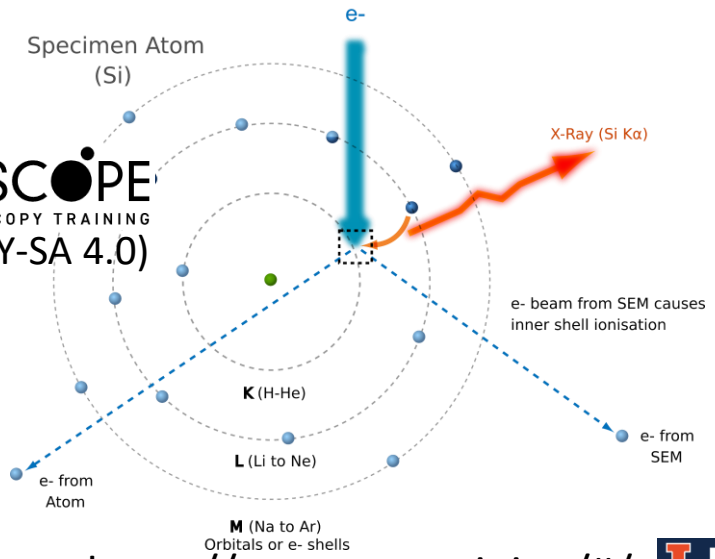


Confocal Raman on MoS_2
data courtesy of Mike Wang (formerly Nam Group, now professor at University of South Florida)

X-Ray Compositional Analysis

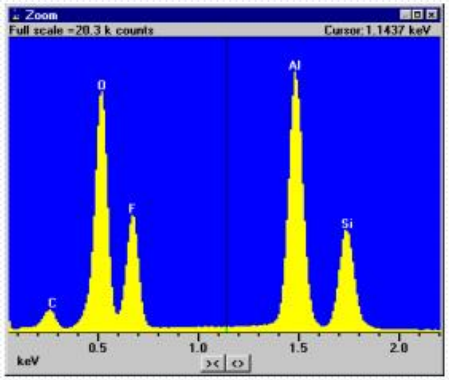
- Characteristic X-rays
- Analytical SEM (EDS)
- X-ray fluorescence

Characteristic X-Ray Production

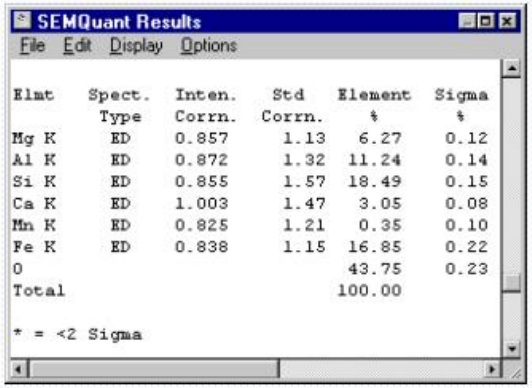


https://myscope.training/#/EDSlevel_2_9

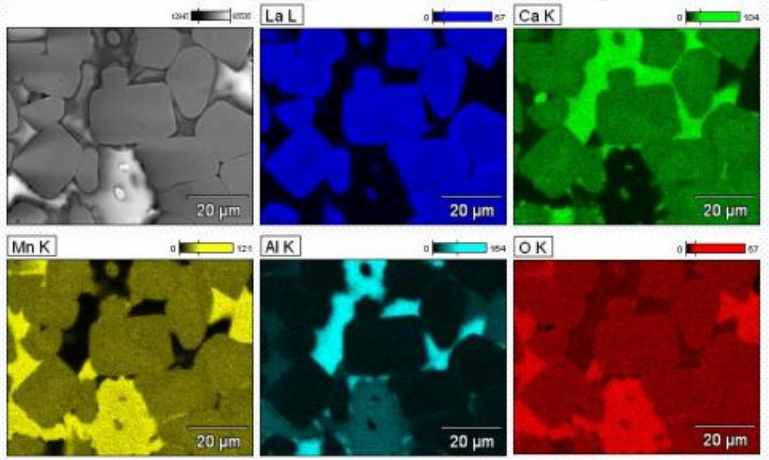
Point (area) analysis



Quantification

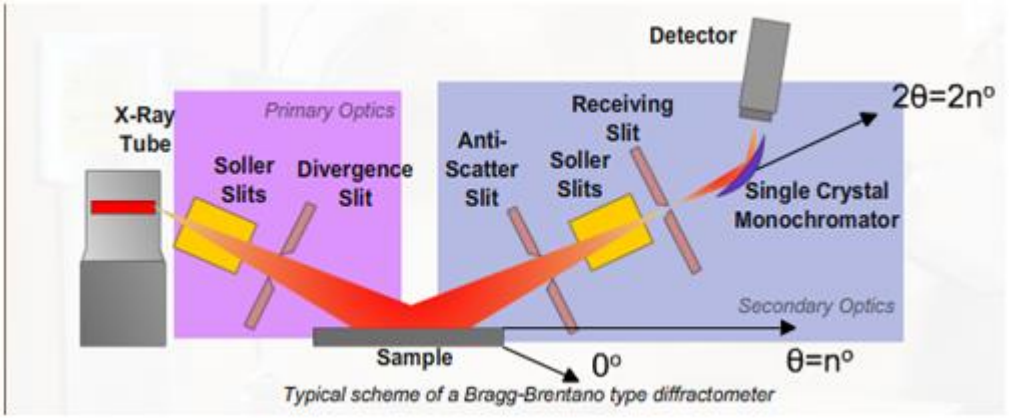


Spectrum Imaging – Area Mapping, Line Scan



X-Ray Characterization

- X-ray diffraction
  - Crystallinity
  - Composition
- X-ray reflectivity
  - Film thickness
  - Roughness of buried interfaces



https://myscope.training/legacy/xrd/background/whatisxrd/

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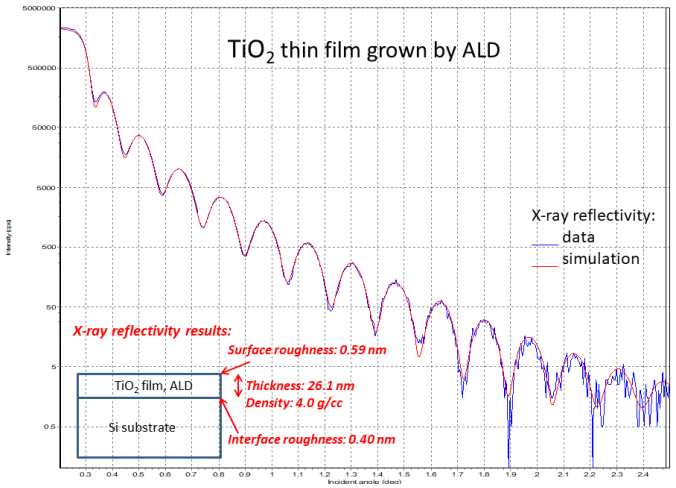

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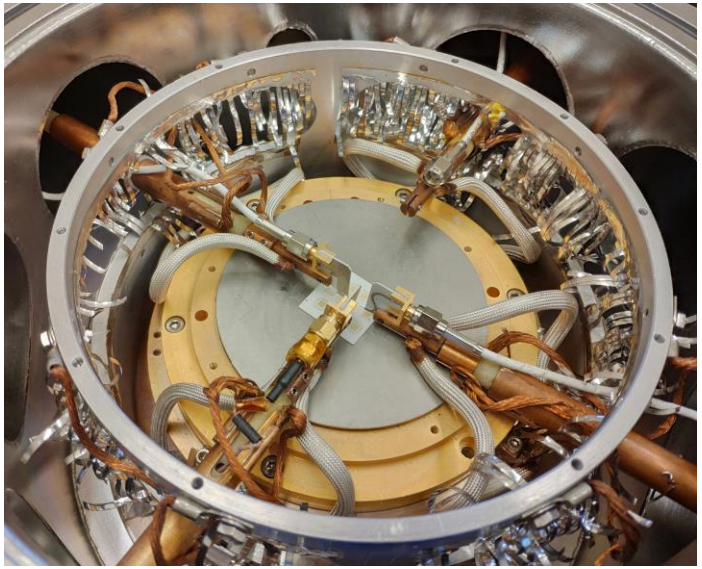
How Does It Behave?

- Mechanical characterization
  - Dynamic mechanical analyzer (bending, pulling)
  - Rheometer (twisting)
  - Nanomechanical analysis (nanoscale compression)
- PPMS/MPMS
- Electrical characterization
  - 4-point probe/sheet resistance
  - Cryogenic probe station
  - Conductive AFM



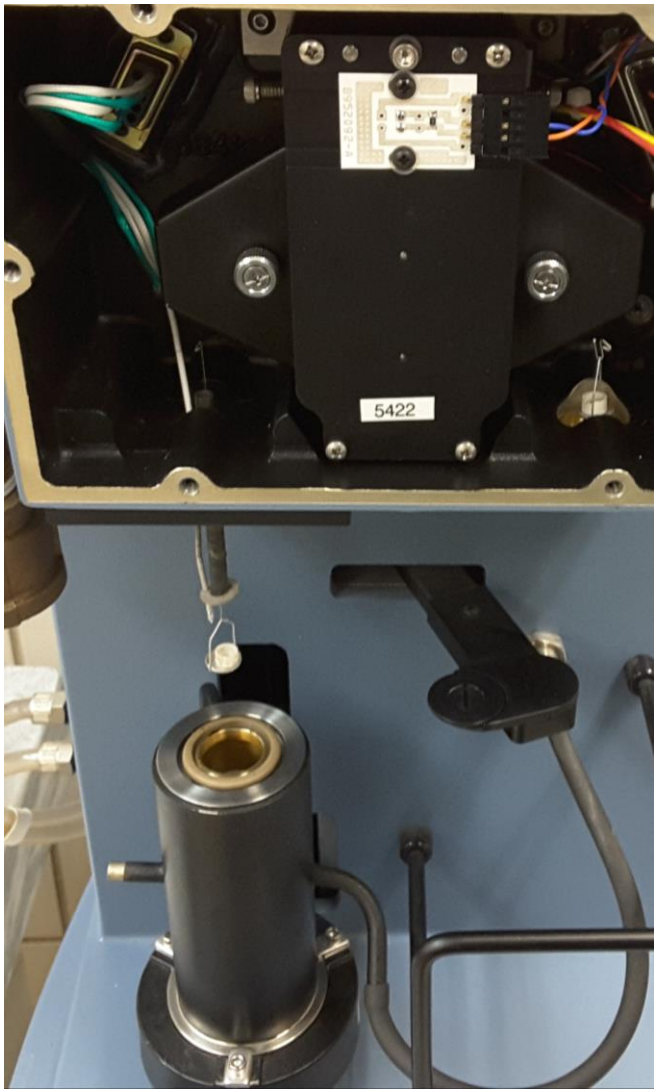
Thermal Analysis

"Soft Materials Characterization: An Instrument Overview"

- mediaspace.illinois.edu/channel/MRL_Webinars
- "basics of chromatography, thermal and mechanical analyses, as well as a sprinkling of elastic light scattering, as they relate to soft materials research"

Thermogravimetric Analysis

- Glass transition temperature and other phase transitions
- Oxidation, decomposition, thermal stability



MRL Webinar Series

go.illinois.edu/MRLwebinars Thursdays at noon central time

Date	Title
April 2 (recording available)	Basics of atomic force microscopy
April 9 (recording available)	Soft materials characterization: An instrument overview
April 16 (recording available)	Advanced scanning electron microscopy – What is available at MRL?
April 23	Introduction to X-ray photoelectron spectroscopy (XPS)
April 30	Introduction to secondary ion mass spectrometry (SIMS)
May 7	3D optical profilometry
May 14	Practical microanalysis based on scanning electron microscopy

mrl.illinois.edu/facilities

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Resources

- **go.illinois.edu/MRLwebinars**
  - Introduction to XPS 4/23, Introduction to SIMS 4/30 (noon)
  - mediaspace.illinois.edu/channel/MRL_Webinars
- **go.illinois.edu/MRLbig4webinars**
  - Introduction to Scanning Electron Microscopy 4/27 (11am)
- https://myscope.training

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submit proposal → be trained → do science

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