COLUMBIA | NANO INITIATIVE

COLUMBIA UNIVERSITY

Characterization and Fabrication Techniques for Nanoscience and Nanotechnology Research

Electron Microscopy at Columbia Nano Initiative

The Webinar will Begin at 1 PM eastern time

Dr. Nava Ariel-Sternberg
Director, Columbia Nano Initiative Labs

Dr. Amir Zangiabadi
Director, Electron Microscopy Labs

PennState College of Engineering

NSF

This Webinar Is Hosted By

ATE Central acts as an information Hub for the National Science Foundation ATE Grantee Community

atecentral.net/webinars

Dr. Nava Ariel-Sternberg
Director,
Columbia Nano Initiative Labs

Dr. Amir Zangiabadi
Director,
Electron Microscopy Labs

Robert Ehrmann
Managing Director,
NACK Network

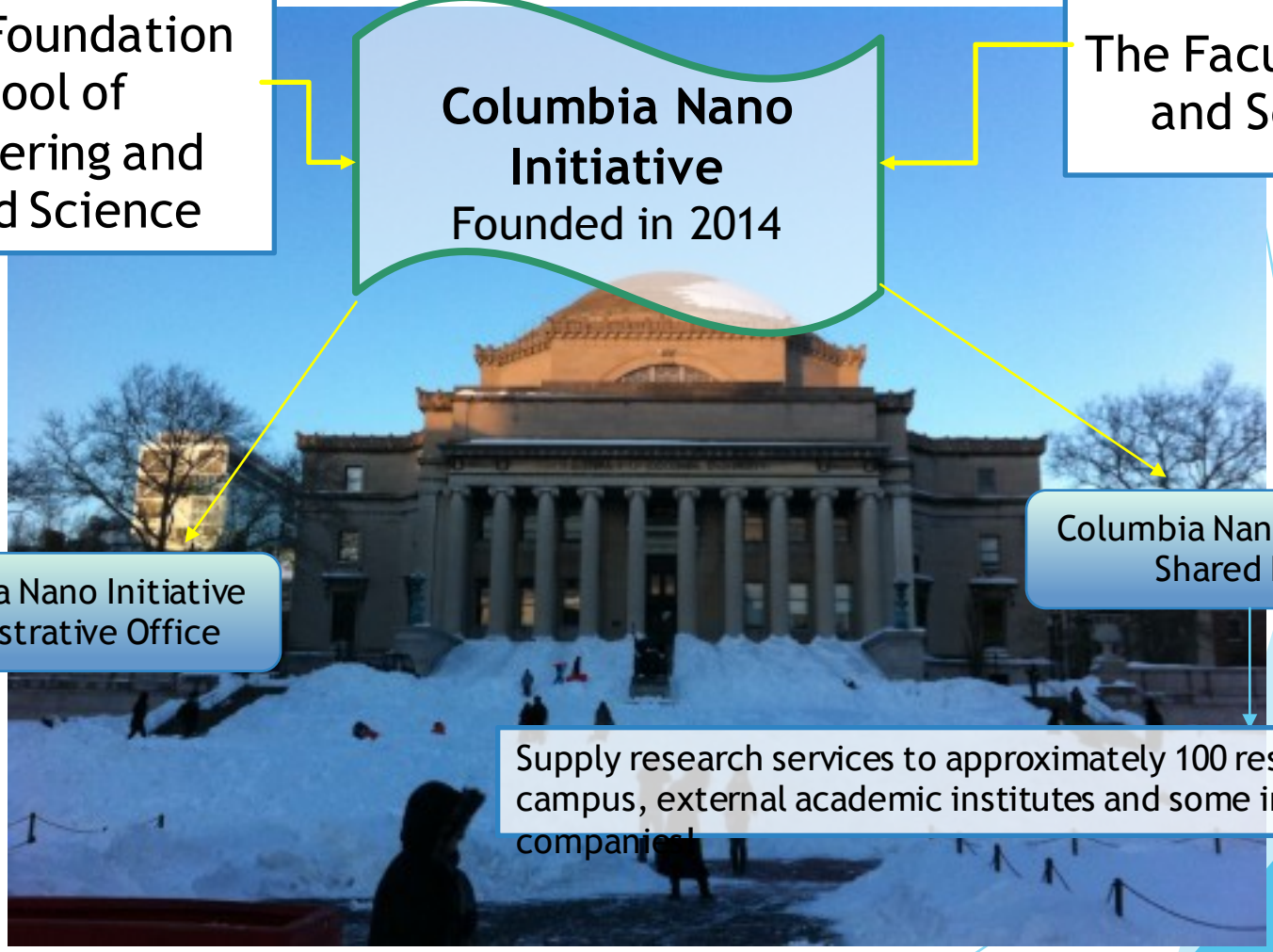
Webinar Objectives

- Overview of CNI Shared Labs, research capabilities, and fields of research
- Overview of Electron Microscopy and sample preparation
- Examples from recent research projects at CNI using Electron Microscopy

The Fu Foundation School of Engineering and Applied Science

Columbia Nano Initiative
Founded in 2014

The Faculty of Arts and Sciences



Columbia Nano Initiative Administrative Office

Columbia Nano Initiative Shared Labs

Supply research services to approximately 100 research groups on campus, external academic institutes and some industrial companies

Research at CNI

2D Materials and Devices

Silicon Photonics

Bioelectronics

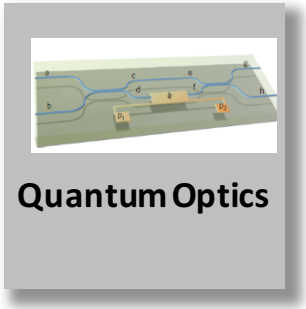
Photonics Design and Architecture

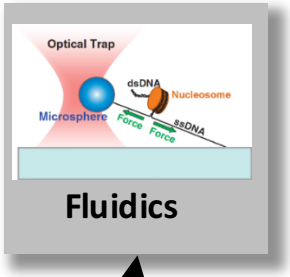
Non conventional and flexible electronics

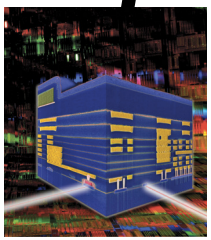
Superatoms

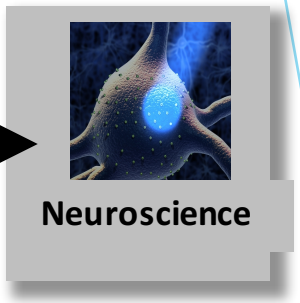
NOVEL RESEARCH AREAS ENABLED BY SILICON PHOTONICS

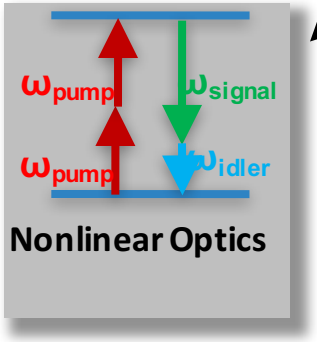
Prof. Michal Lipson

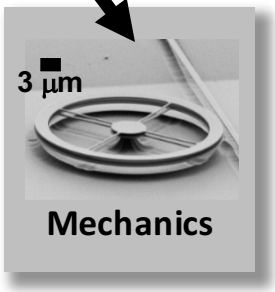










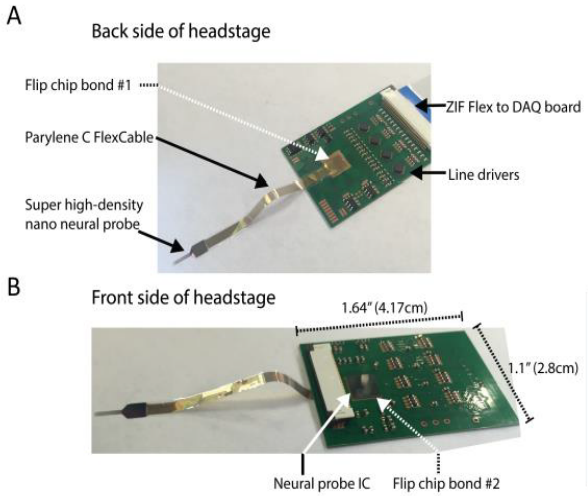


http://lipson.ee.columbia.edu/

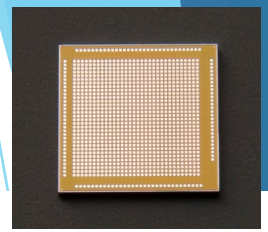
Bioelectronics for Neuroscience applications: 1024-channel prototype

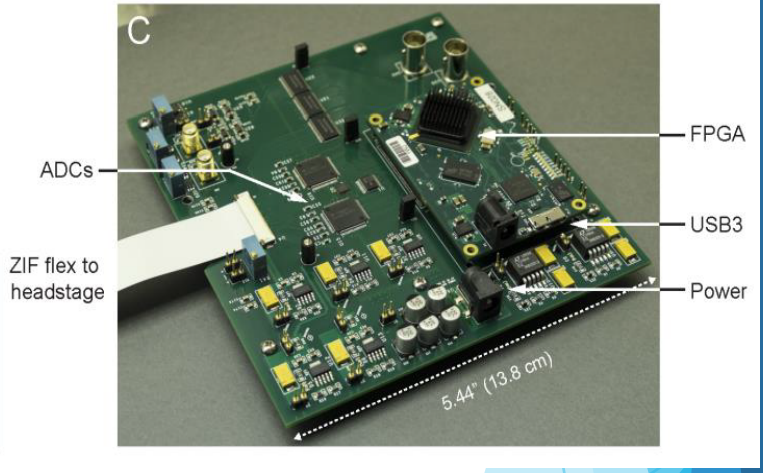
Prof. Kenneth Shepard

1,024 channels per layer. 10 layers.



In vivo 1k-channel NeuroProbe





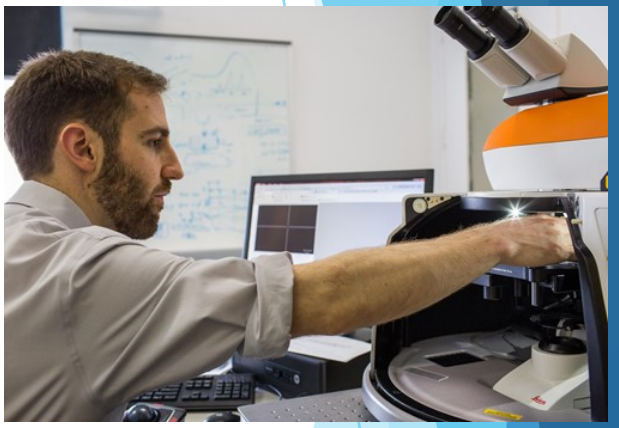
https://bioeeweb.ee.columbia.edu/wordpress/research/

CNI Shared Facilities

Electron Microscopy

Clean Room

Materials Characterization



Over 400 users from approximately 100 research groups.
External users are welcome!

Meet the staff – CNI Shared Facilities

Dr. Nava Ariel-Sternberg

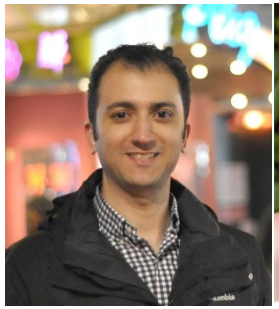
Director of Shared Facilities

James Vichiconti

Director of Clean Room

Dr. Dan Paley

Director of SMCL



Dr. Amir Zangiabadi

Director of EM lab

Nirit Porecki-Shamay

Senior Clean room Engineer

Dr. Jaeeun (Jen) Yu

Clean room Engineer

Melody Gonzalez

Research Operation Assistant

CNI Clean Room

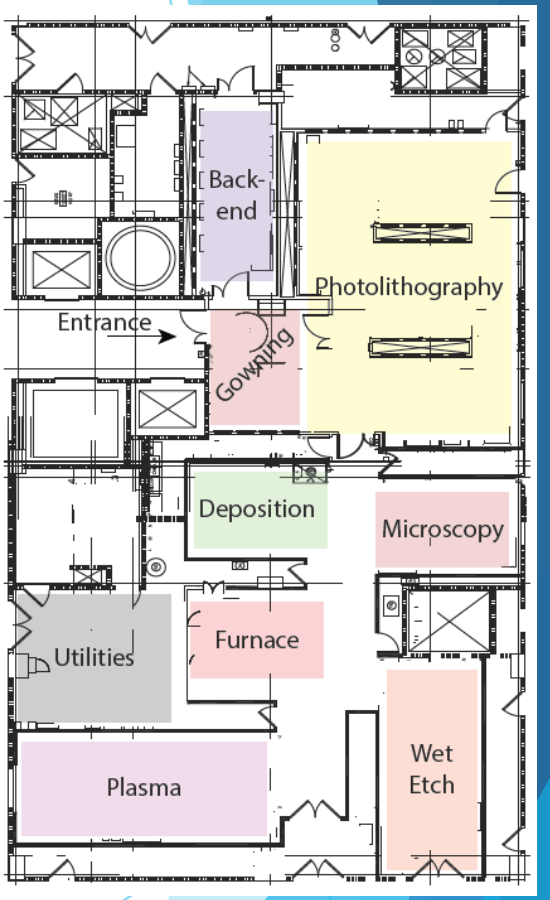
- An environmentally controlled lab – tight limits for temperature and humidity, air exchange
- Particle filtering, class 1000 to 10,000
- Clean room apparel

Airborne Particulate Cleanliness Classes (by cubic meter):						
CLASS	**Number of Particles per Cubic Meter by Micrometer Size**					
	0,1 micron	0,2 micron	0,3 micron	0,5 micron	1 micron	5 microns
ISO1	10	2				
ISO2	100	24	10	4		
ISO3	1,000	237	102	35	8	
ISO4	10,000	2,370	1,020	352	83	
ISO5	100,000	23,700	10,200	3,520	832	29
ISO6	1,000,000	237,000	102,000	35,200	8,320	293
ISO7				352,000	83,200	2,930
ISO8				3,520,000	832,000	29,300
ISO9				35,200,000	8,320,000	293,000

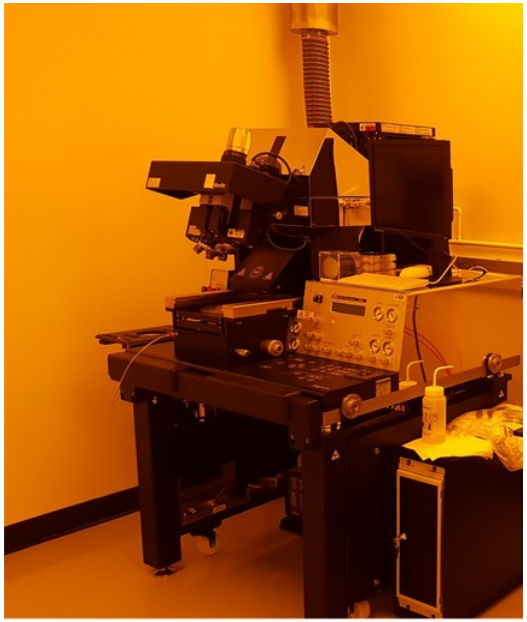
*Terra Universal Inc.

Clean Room Utilities and Supporting system

- Non Contact Cooling Water system to cool equipment
- DI water supply for processing
- N2 and compressed dry supply
- AHUs, dehumidifier and Clean Steam Generator for tight temperature and humidity control
- Exhaust for chemicals fumes and air exchanges
- Lab monitoring system
- Safety systems - Toxic Gas monitoring System (TGMS)

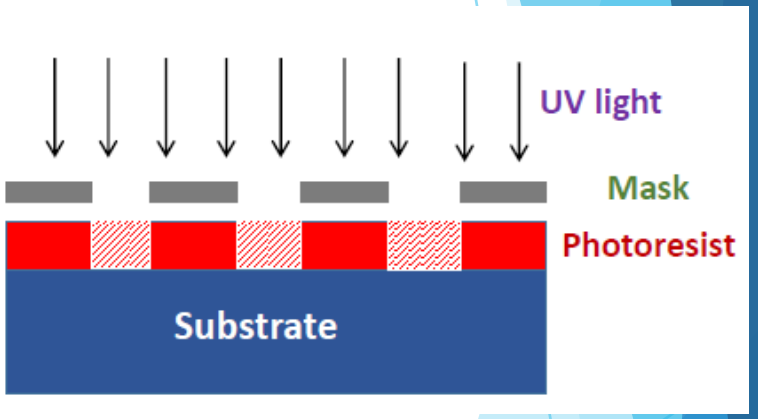


Patterning: Photolithography and Etch



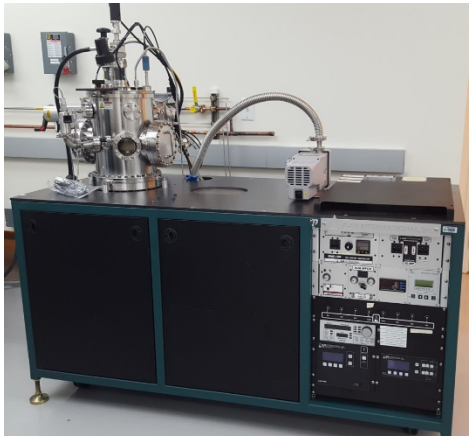
- SUSS Mask aligner for UV exposure down to 248nm (sub-micron resolution)

- Oxford Reactive Ion Etching



Deposition and thin film growth

Expertech LPCVD furnace



AJA magnetron sputtering

Angstrom e-beam evaporation

Back End: Connecting the device to the outside world

Westbond Wirebonders

Disco DAD 3220 Dicing saw

CMP: Poli-400L

Materials Characterization Lab

Surface Analysis and surface area

Crystallographic structure and orientation

Particle size, Molecular weight distribution, and Z-potential

Optical and Magnetic Properties

Molecular structure and bonding information

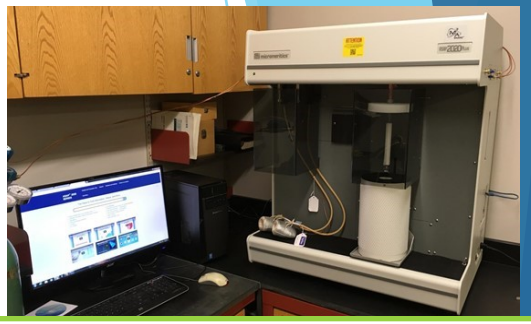
Thermal Properties

2D materials device fabrication and characterization

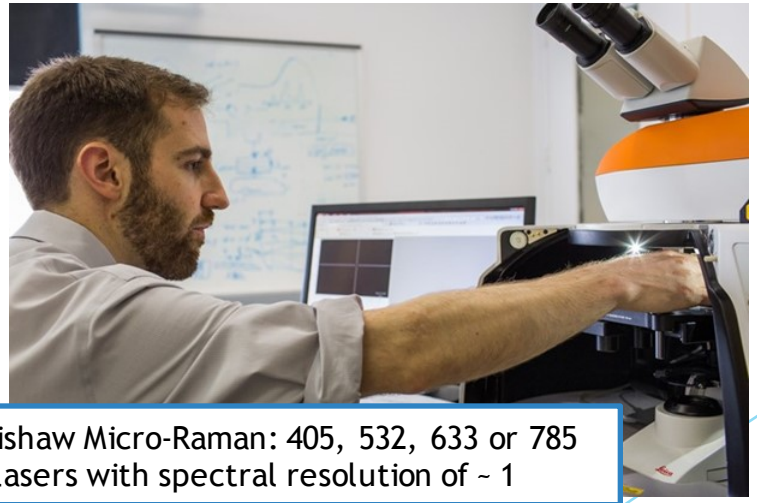
Surface Analysis and bonding

XPS: For surface elements survey and depth profiles

Bruker AFM

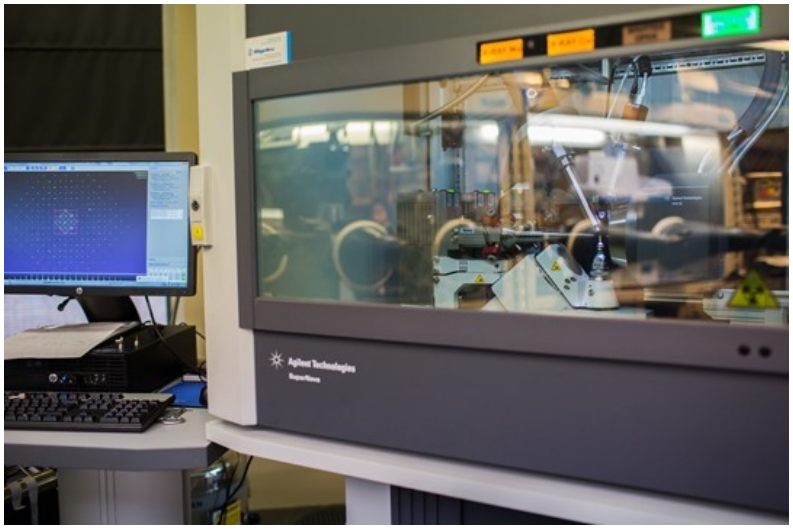


BET analyzer: Surface area by measuring nitrogen adsorption isotherms for porous materials at 77 K.



Renishaw Micro-Raman: 405, 532, 633 or 785 nm lasers with spectral resolution of ~ 1

Crystallographic structure, phase, orientation Information



Agilent SuperNova SCXRD: Mo/Cu dual micro-focus source of 50W.

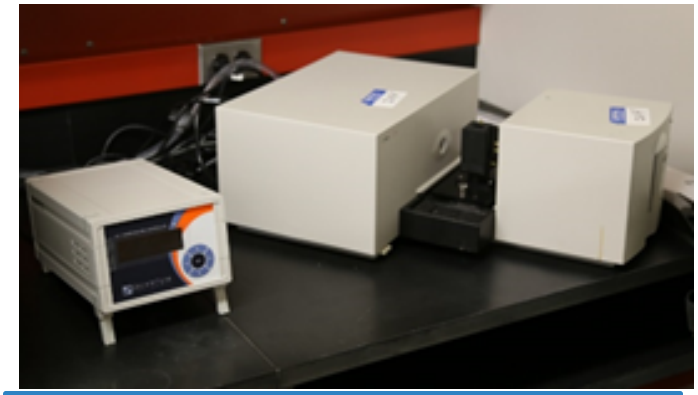
PANalytical PXRD: For powder crystallographic analysis. Temp. measurements in the -173-400°C range

Particle size, Molecular size distribution, and Z-Potential

GPC: gel permeation chromatography analysis of polymers at different solvents and temperatures.

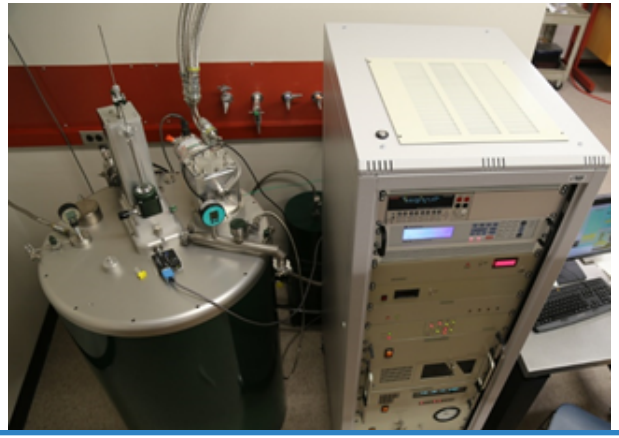
Dynamic Light Scattering (DLS) and Z-potential to measure: particle size, molecular weight, and zeta potential for organic and aqueous colloids, nanoparticles, and proteins.

Optical and Magnetic Characterization



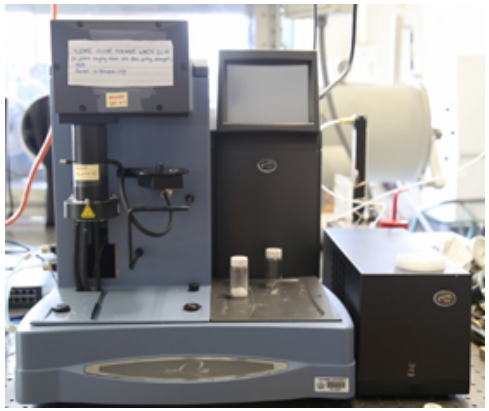
Spectrophotometer: measuring absorbance in the 190-1100 nm range. Temp measurements in the -20-110°C.

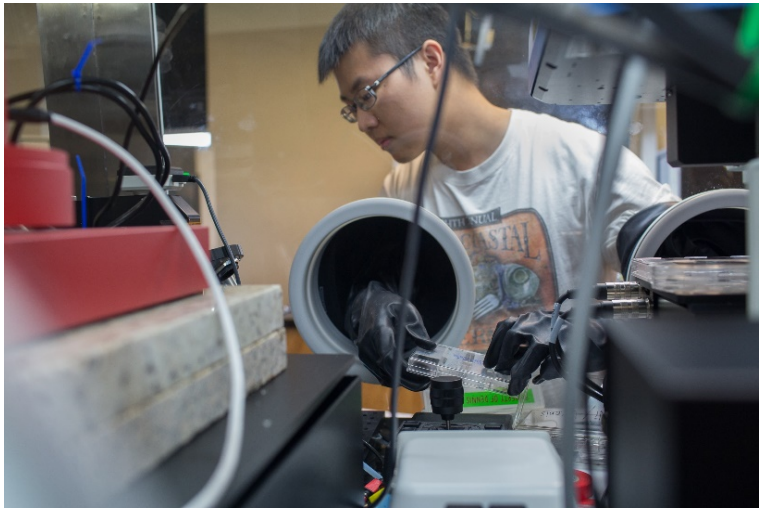
Woollam Ellipsometer: thin film thickness and refractive index measurements

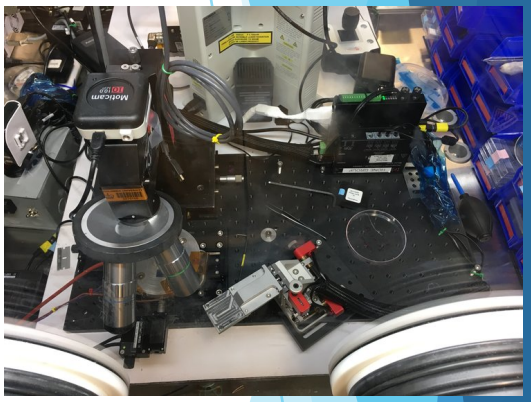


SQUID: DC and AC measurements of magnetic susceptibility. Sample temperature between 1.8 and 300 K.

Thermal Properties and 2D materials device fabrication and Characterization





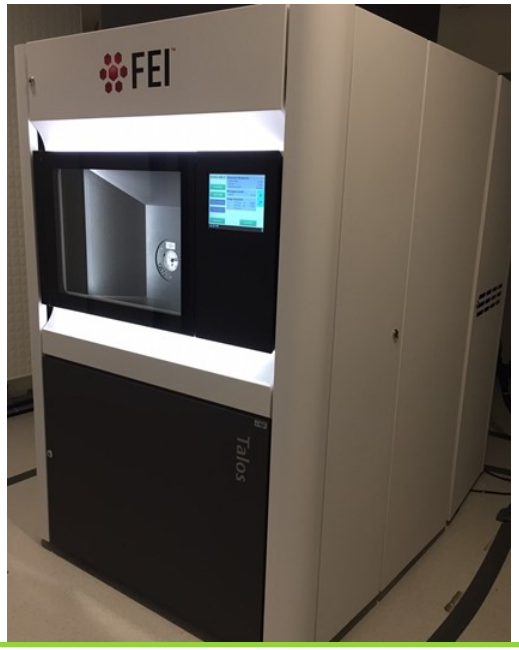


TGA: Thermal analysis temperatures between ambient and 1000 °C.

2D Material processing in protective environment: Autofinder (microscope with computer-controlled xyz axes and a remote-controlled micromanipulator, 0.5μ precision)

Electron Microscopy and Imaging

FEI Nova NanoSEM 450:
- FEG with Through lense SED, Everhart Thormley SED, Low Vacuum SED, Through lens BSED detectors.
- NPGS – Nabity system for e-beam writing



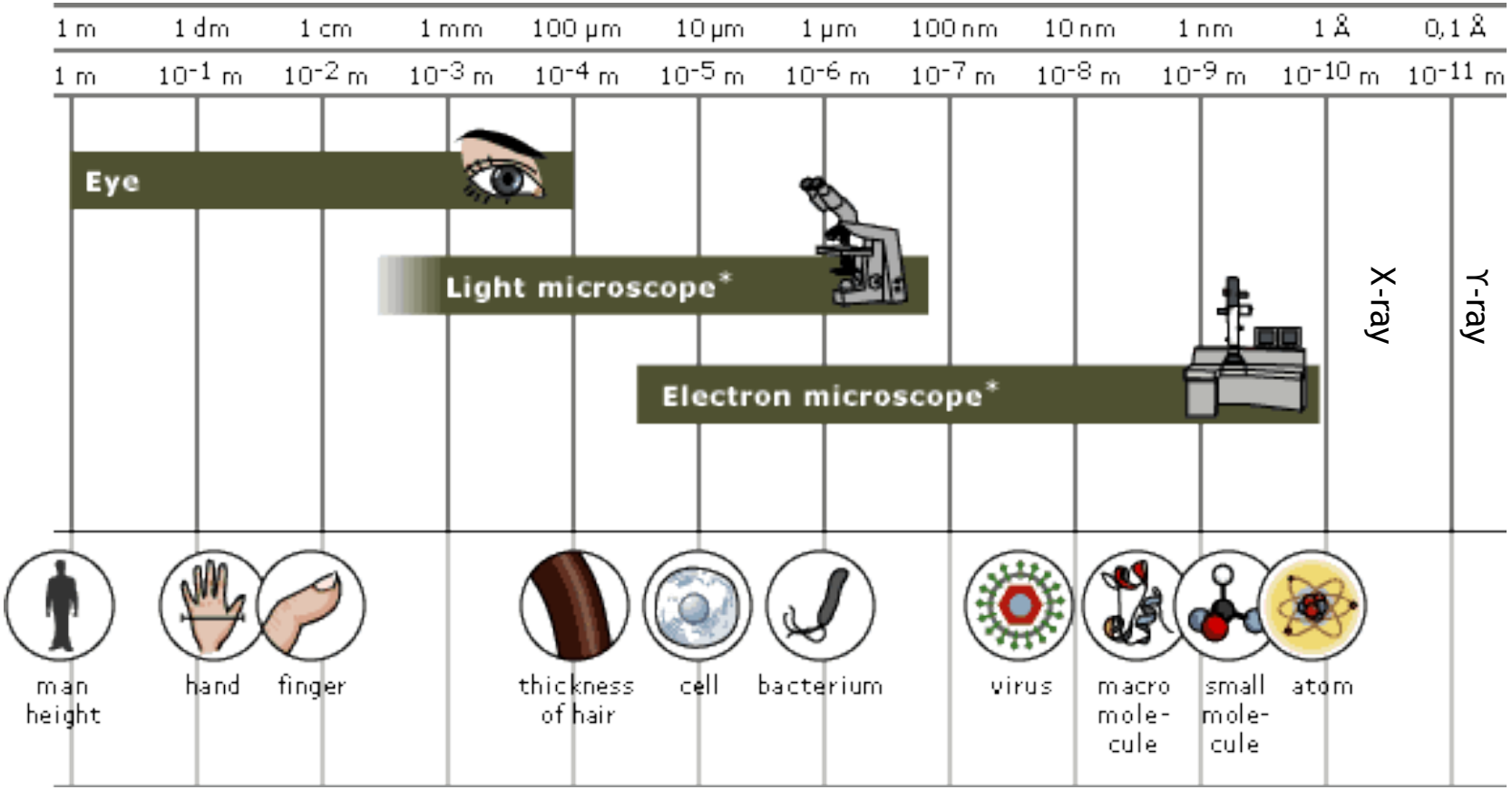
FEI Talos F200X TEM/STEM:
- Max acc voltage of 200 kV, configured for 80kV as well
- Super X-EDS system; 4 Silicon drift detectors (SDD)
- TEM point resolution (nm) 0.25
- Sample preparation suite

Zigma VP Zeiss SEM:
- FEG with Inlens, SE, BSED and VPSE detectors
- Bruker EDS system

Types of Microscope

Using electrons to "see" objects to atomic level

- Similar to optical microscopy except with electrons rather than photons
- Used to image samples with a resolution of 10 Å
- Can image many different structural geometries
- Mostly limited by radiation damage from the electron beam



http://nobelprize.org/educational_games/physics/microscopes/powerline/index.html

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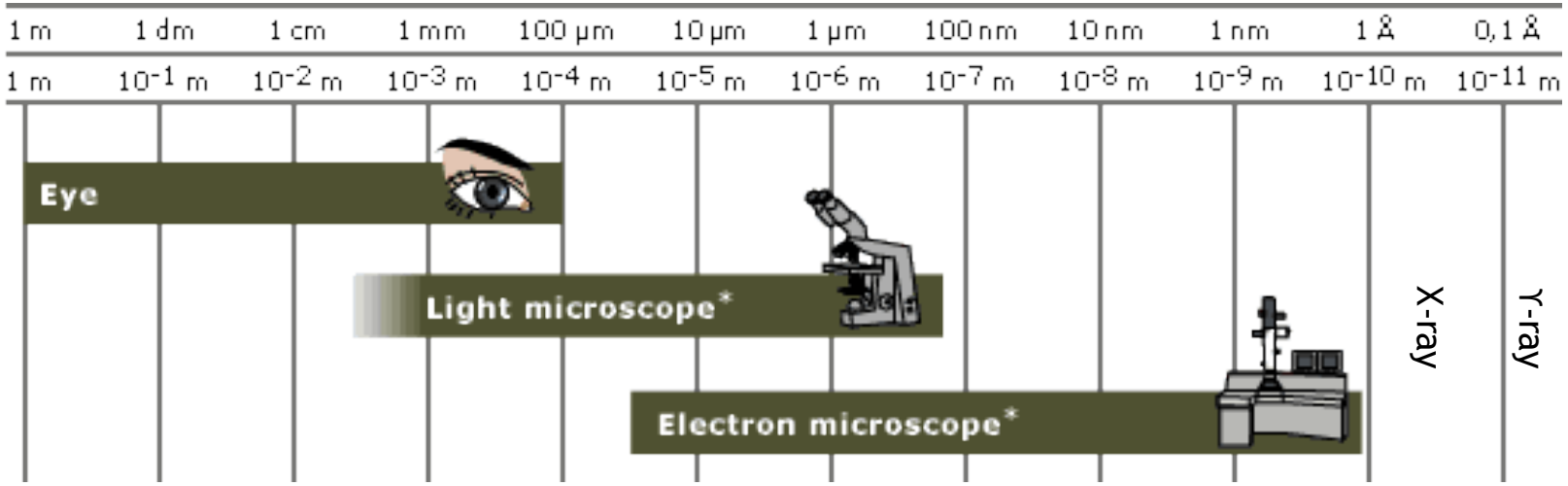
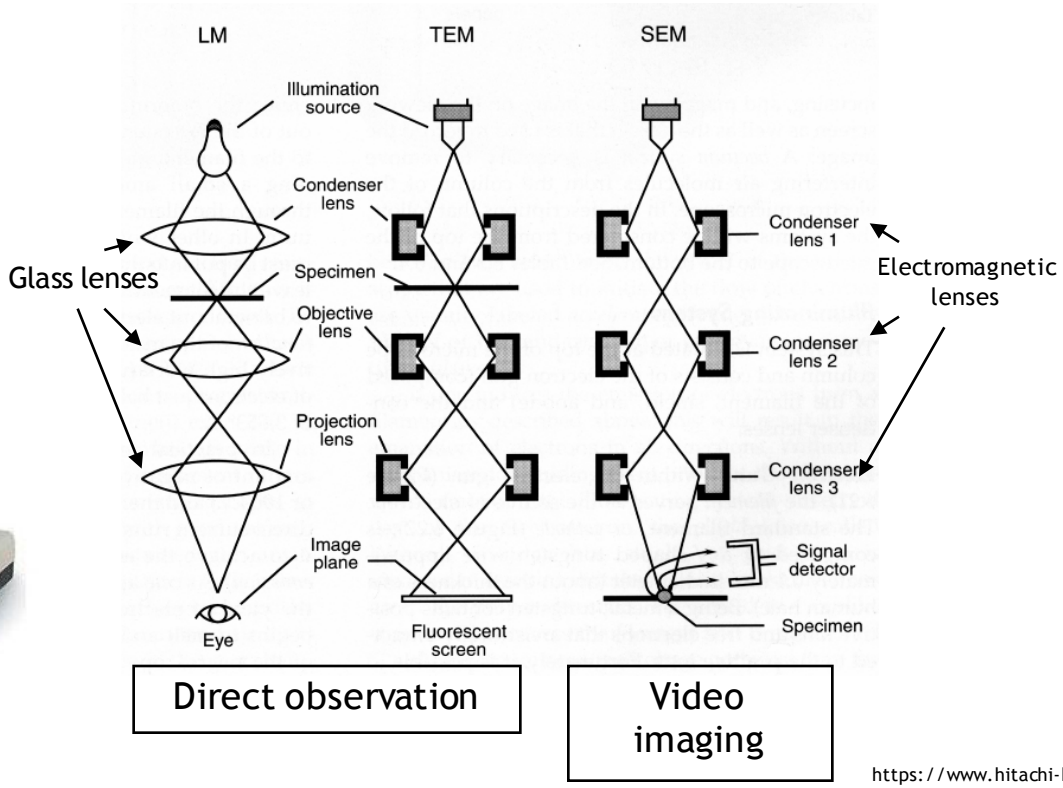


Electron Microscope

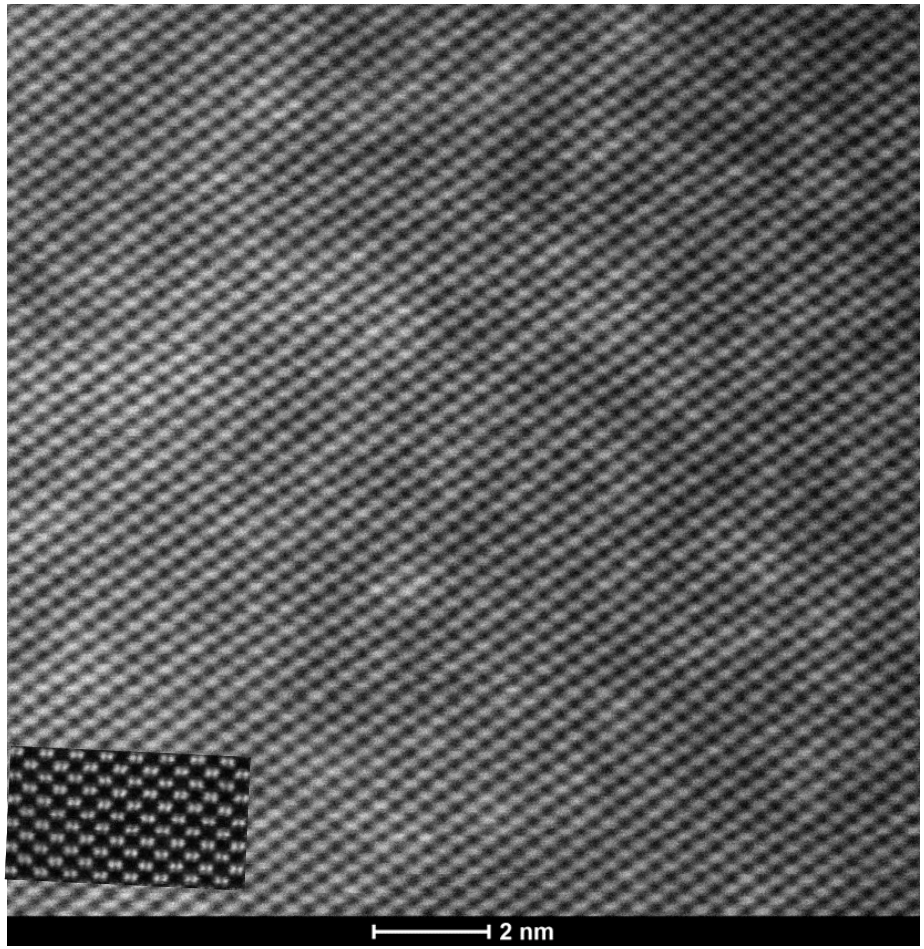
Same principle, but in very different shapes



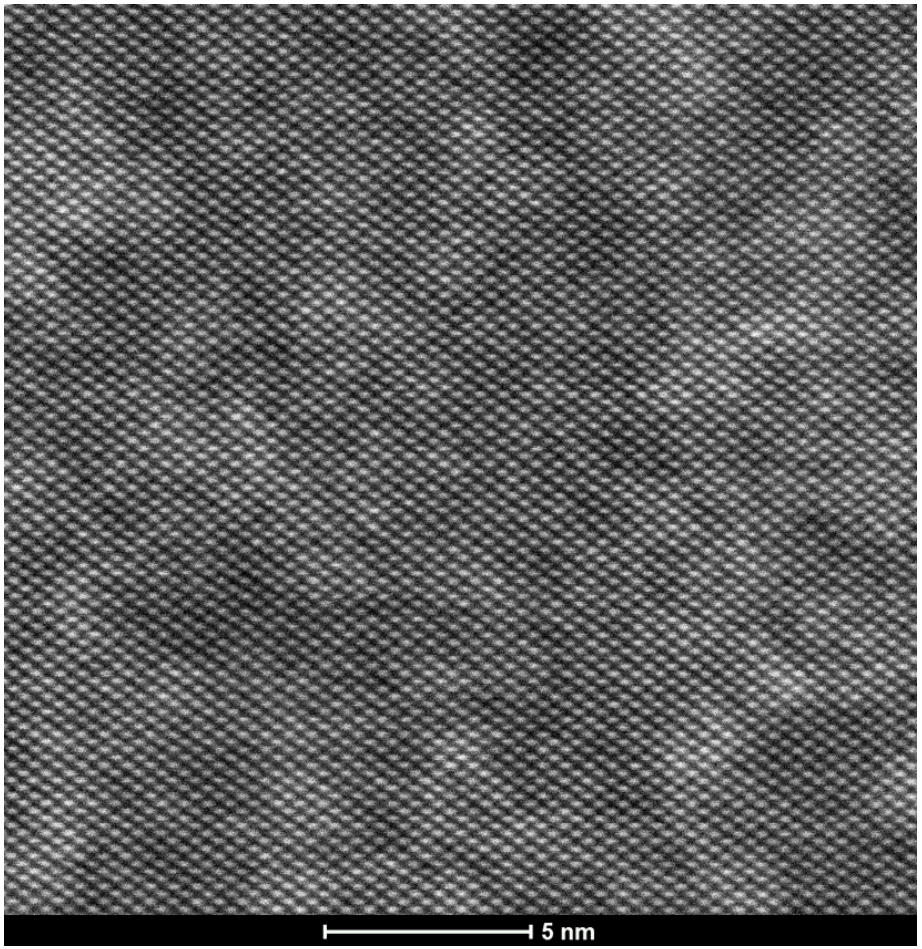
https://www.hitachi-hightech.com/global/sinews/si_report/07046/

Testing the Resolution in STEM

May 2017



March 2015 (approved in Czech Republic)



Specimen Preparation

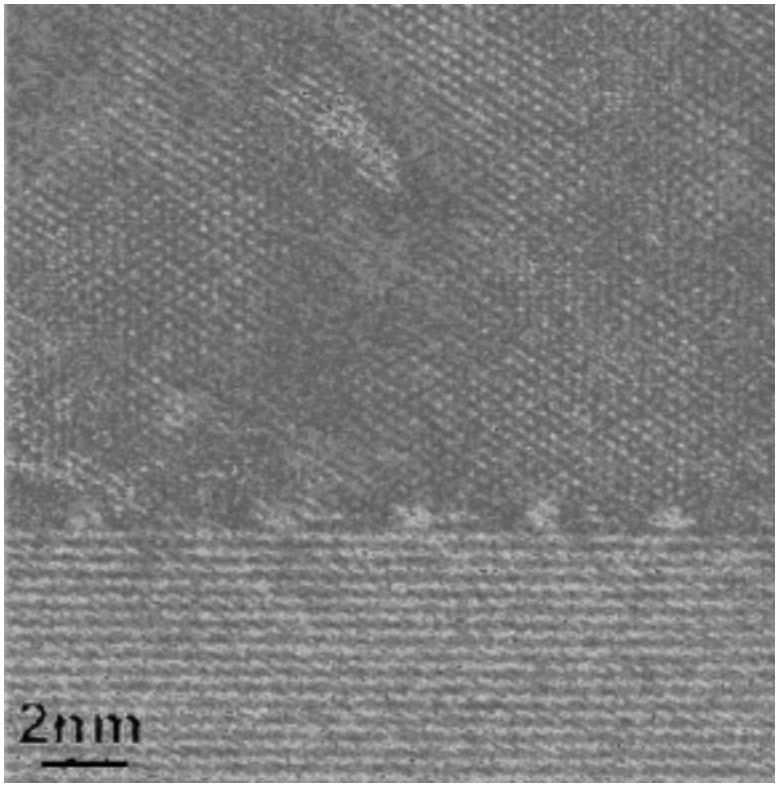
- general techniques for materials sciences

Direct lattice resolution in polydiacetylene single crystal showing (010)lattice planes spaced at 1.2 nm.

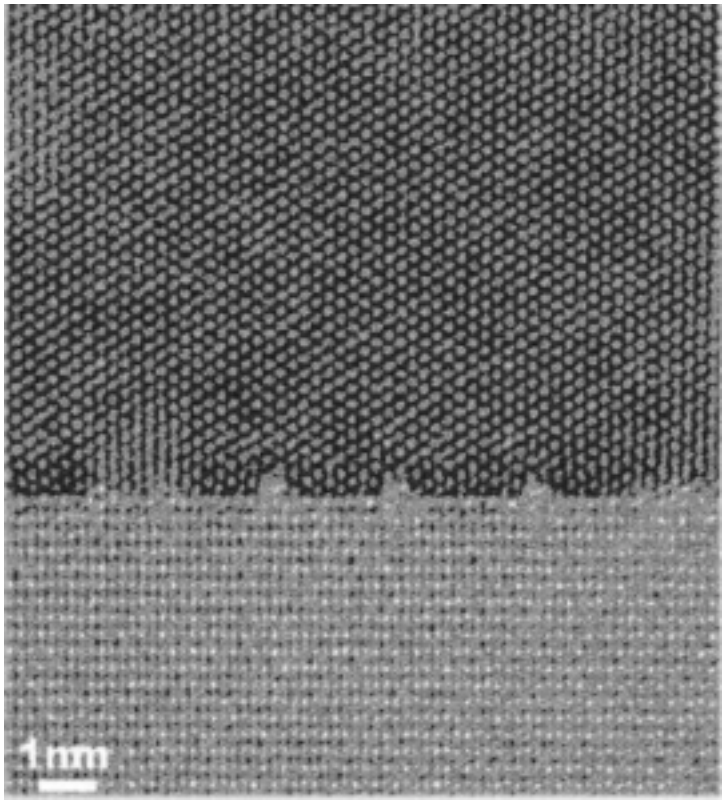
http://www.ph.qmw.ac.uk/images/molwires.jpg

Why Sample Preparation is so Important?

- Bad sample prep, unclear observation, wrong analysis!

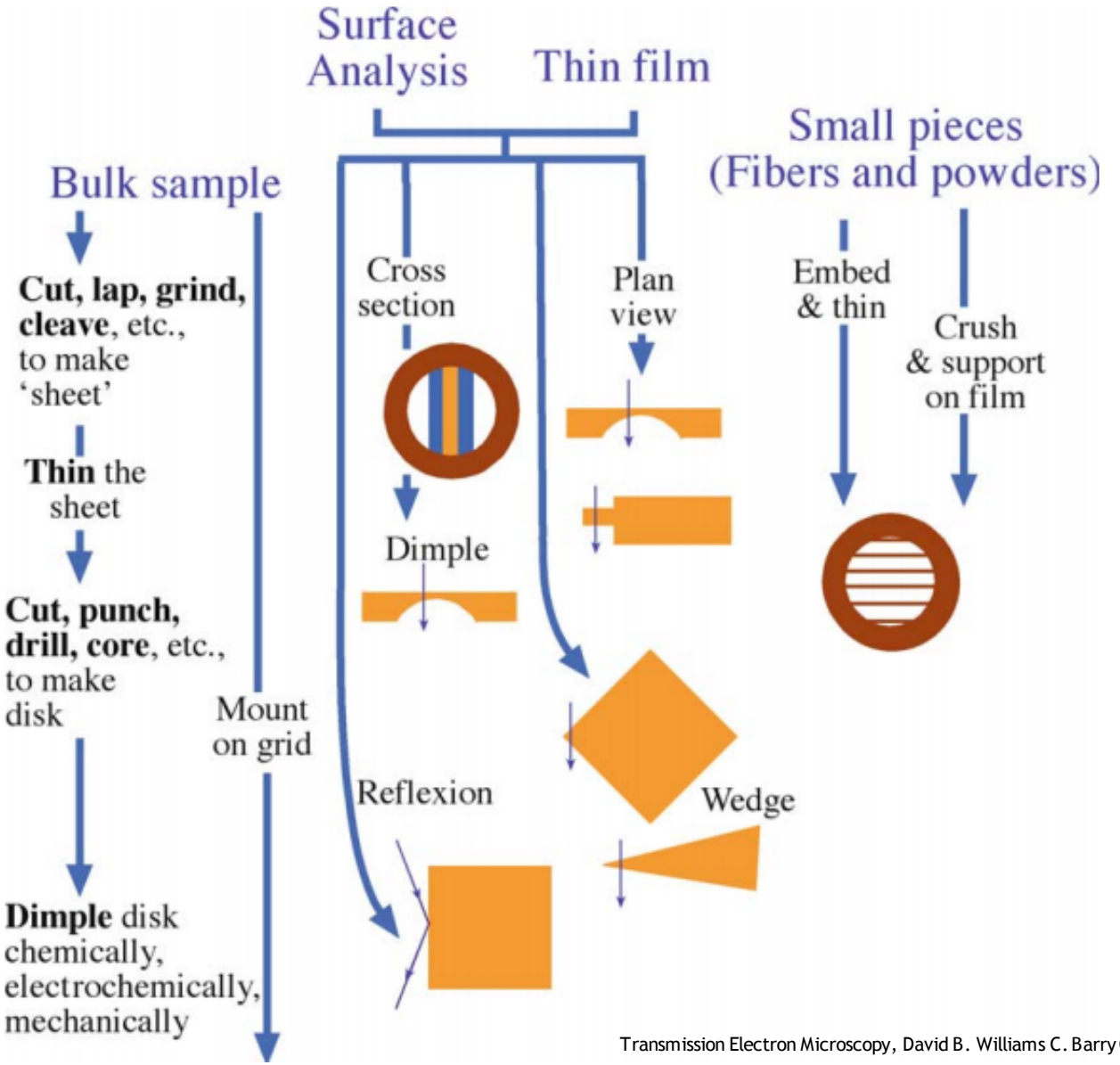


Cross section, Ion Milling (>2kV)



Cross section, Ion Milling (down to 100V)

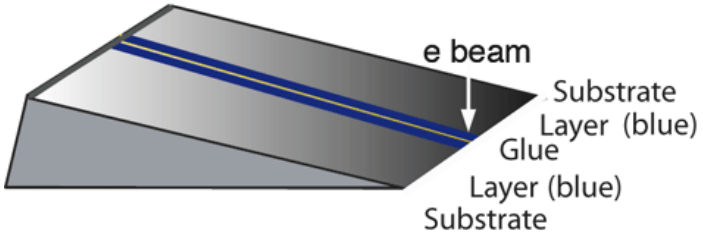
Sample Preparation Overview

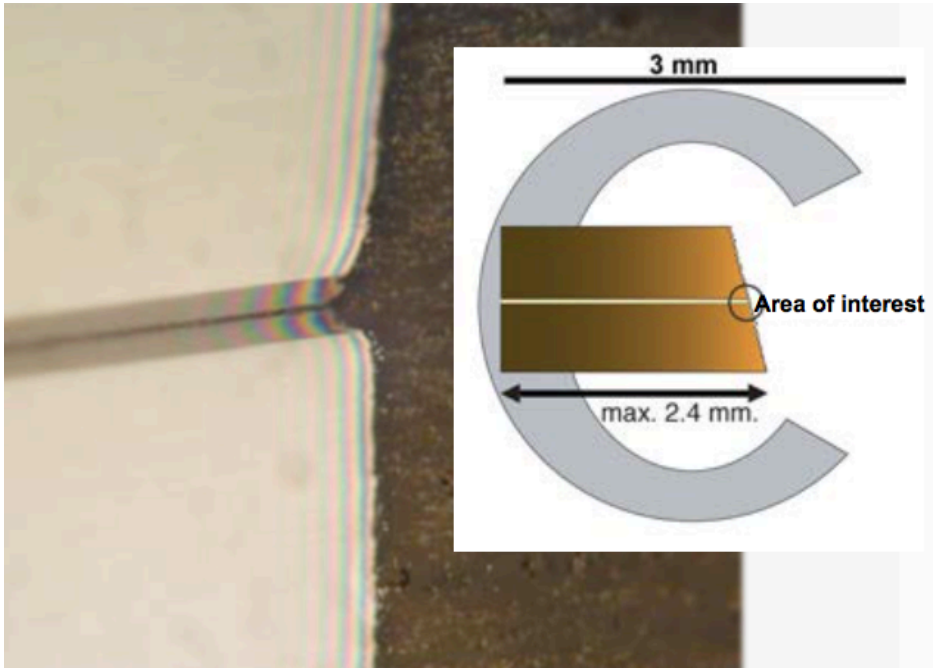


Transmission Electron Microscopy, David B. Williams C. Barry Carter 2009

Mechanical → Tripod Method

- Mechanical thinning, in a wedge configuration, down to electron transparency or to a thickness that requires very short ion milling time.
- Polishing with diamond-impregnated lapping films; Finish with colloidal silica

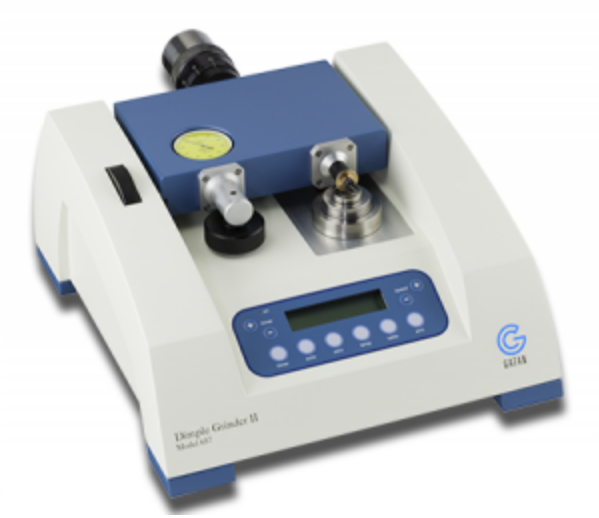


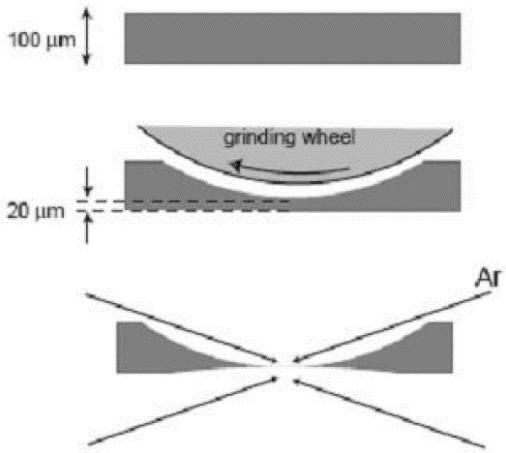


TiO_2 / Silicon,
Optical microscope, reflected light

Dimple Grinding

Thinning the central part of the sample to less than 20 μm before ion milling.





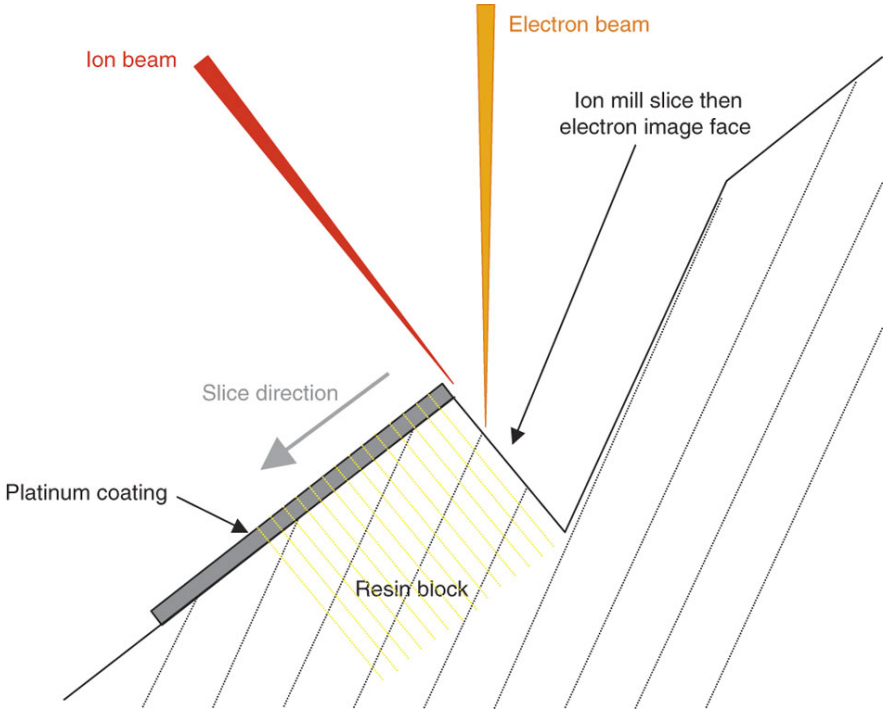
Mechanical grinding
+
Polishing

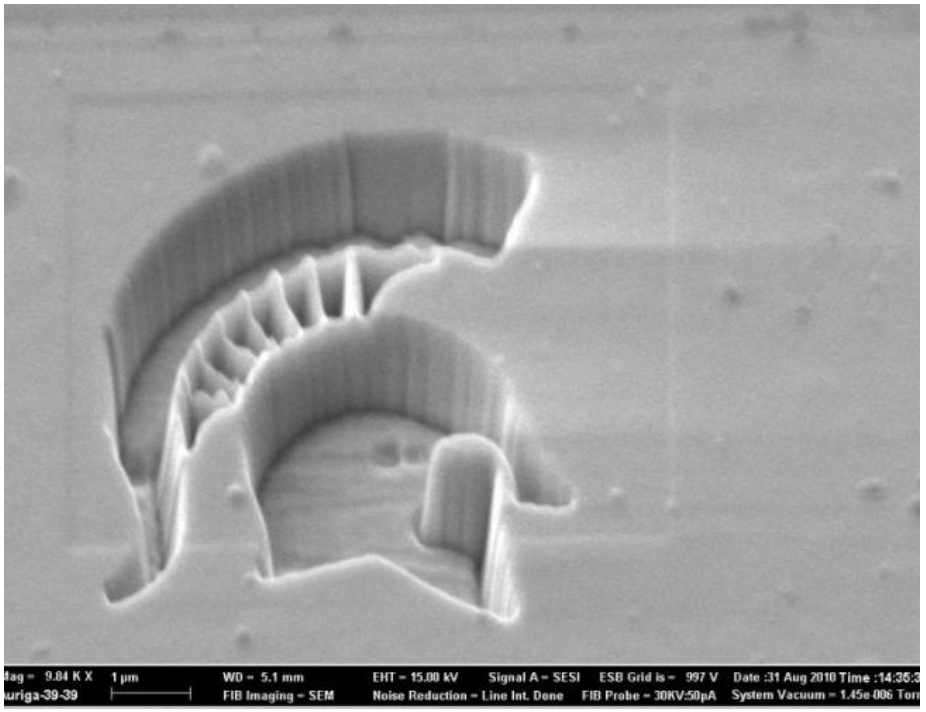
Dimple Grinding

Ion milling

Focused Ion-Beam (FIB) – Collaboration with CUNY

Using precise focused ion beam to select the sample. Then using manipulator to pickup the sample



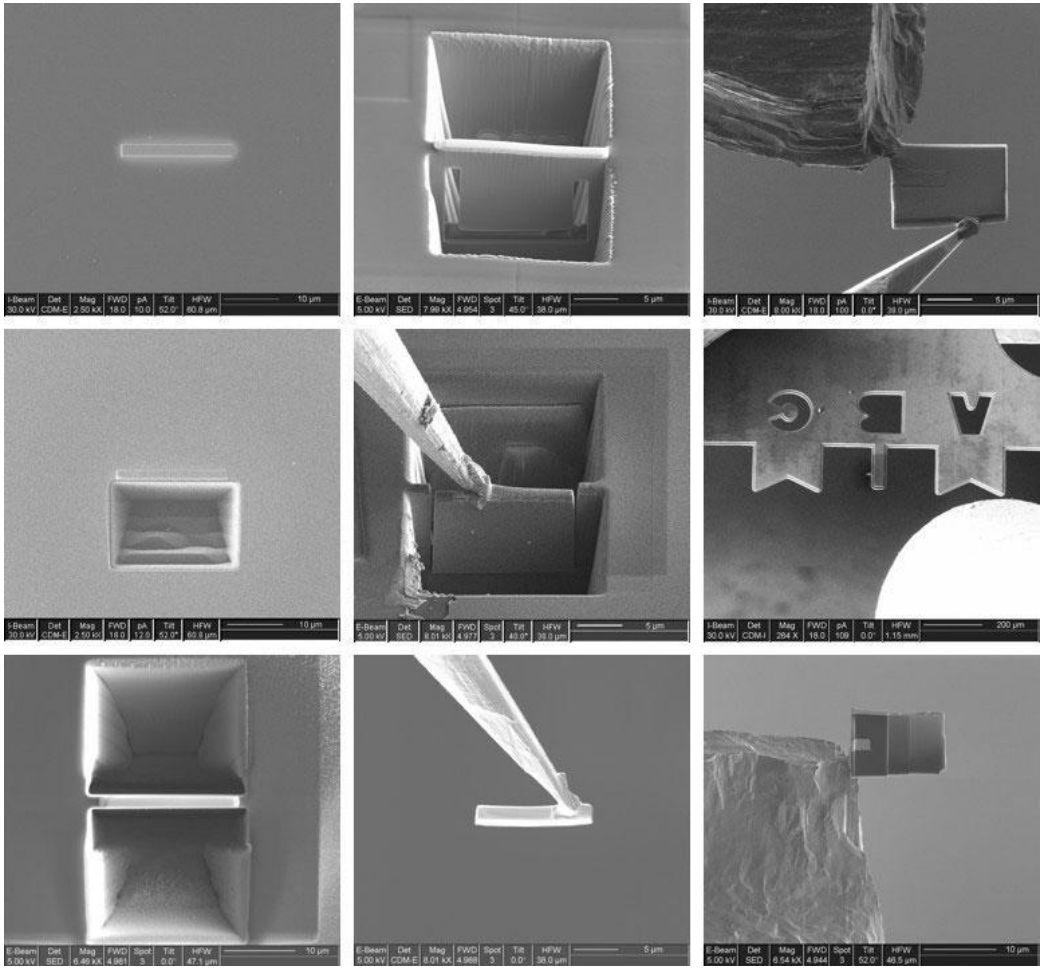


http://www.nature.com/nprot/journal/v6/n6/abs/nprot.2011.332.html

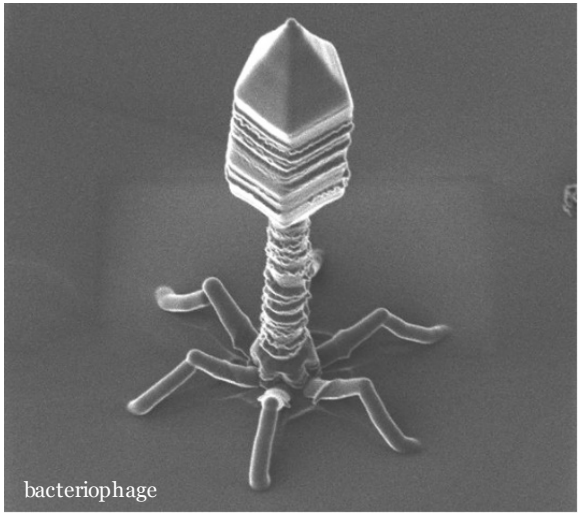
Focused Ion Beam (FIB) Sample Preparation

https://www.youtube.com/watch?v=vNOpzDViAhE

Focused Ion-Beam (FIB)



Biological Specimen Preparation



http://pressblog.uchicago.edu/2011/05/20/traffic-carl-zimmer-and-timothy-lu.html

Overview of Biological Specimen Preparation – Focusing on Sectioning

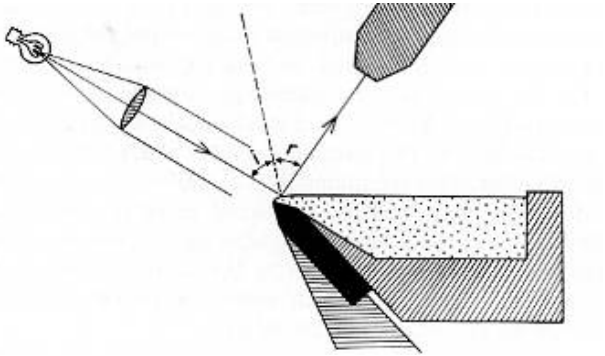
Killing & Fixation
- Death; Molecular stabilization

Dehydration
- Chemical removal of H_2O

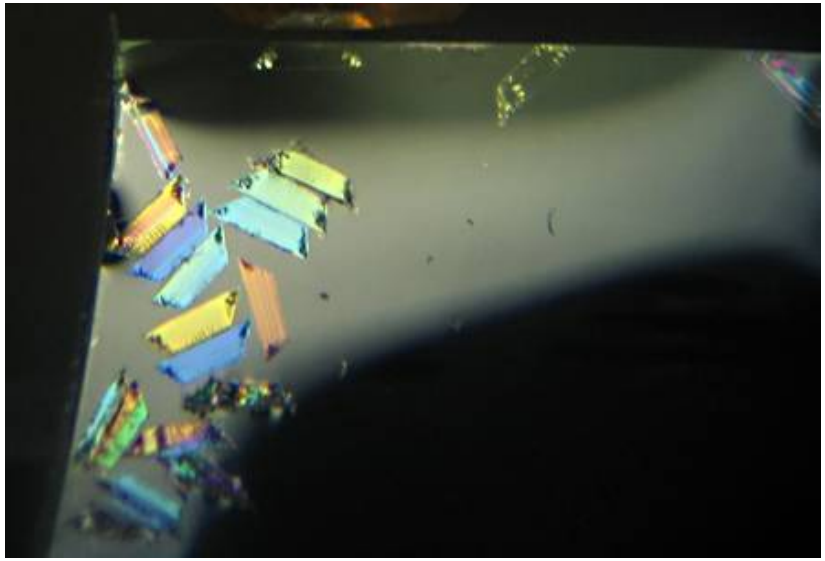
Infiltration
- Replace liquid phase with resin

Embedding & Polymerization
- Make solid, sectionable block

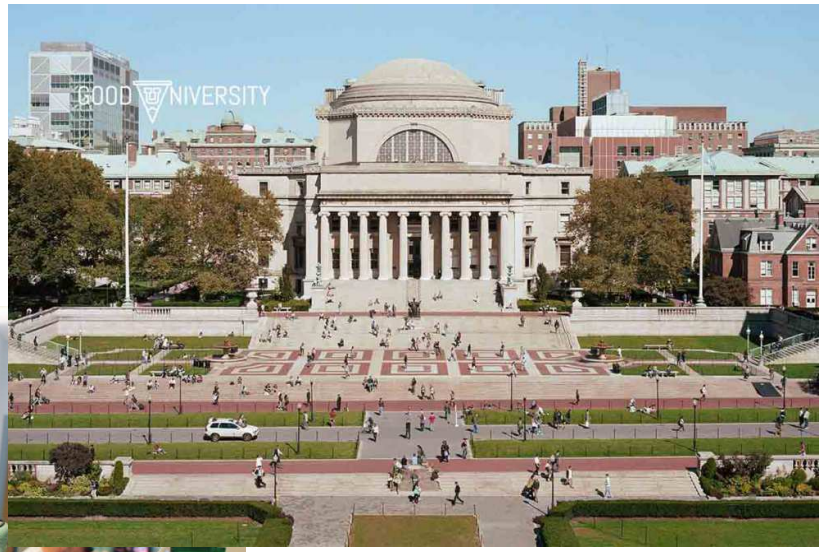
Sectioning
- Ultramicrotome, mount, stain

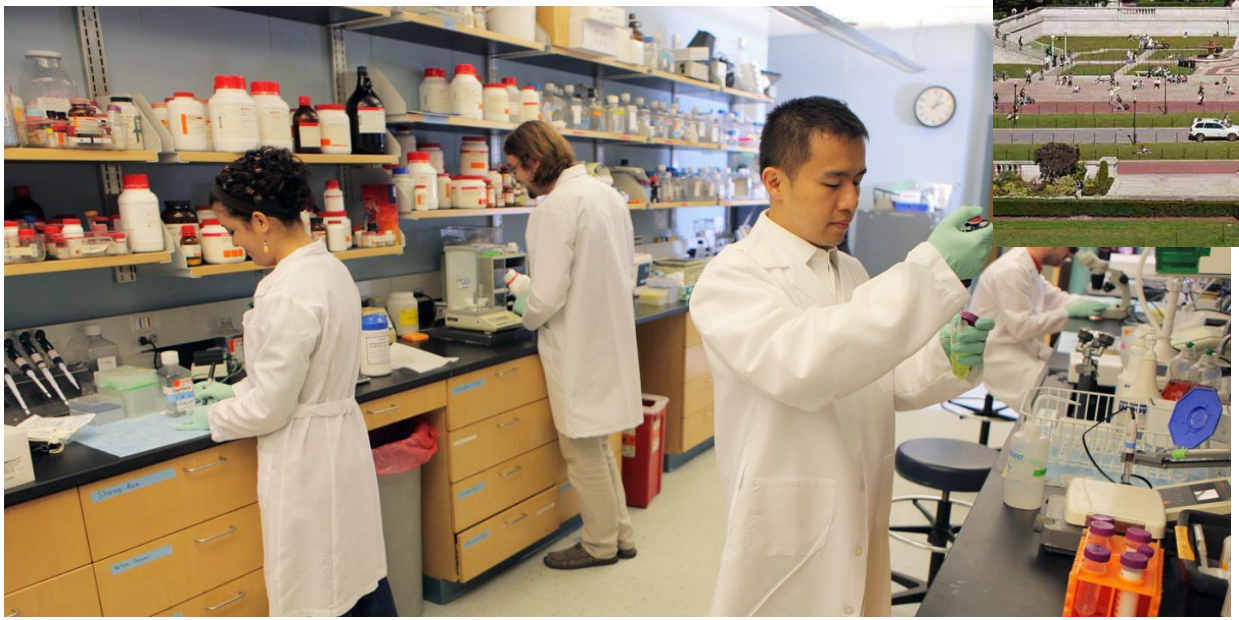


Interference reflection angle from Sjöstrand (1967)

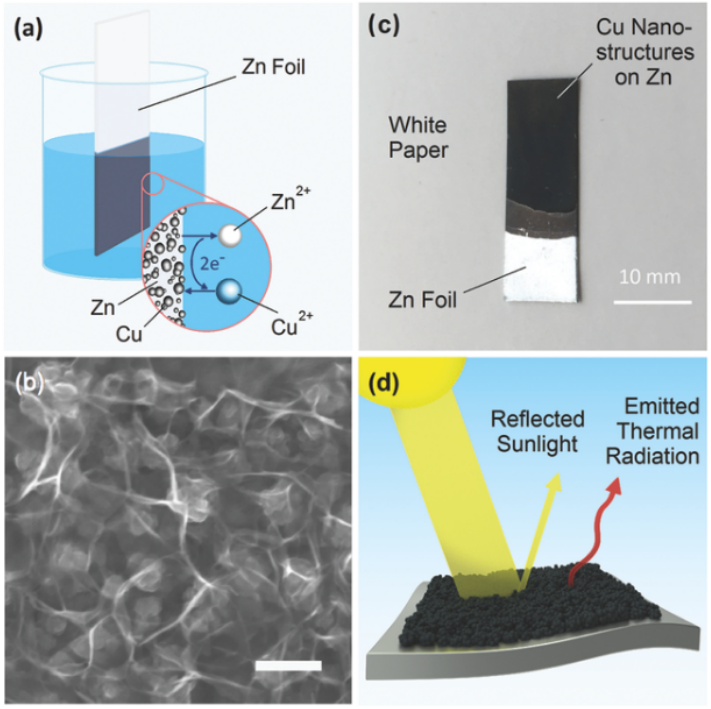


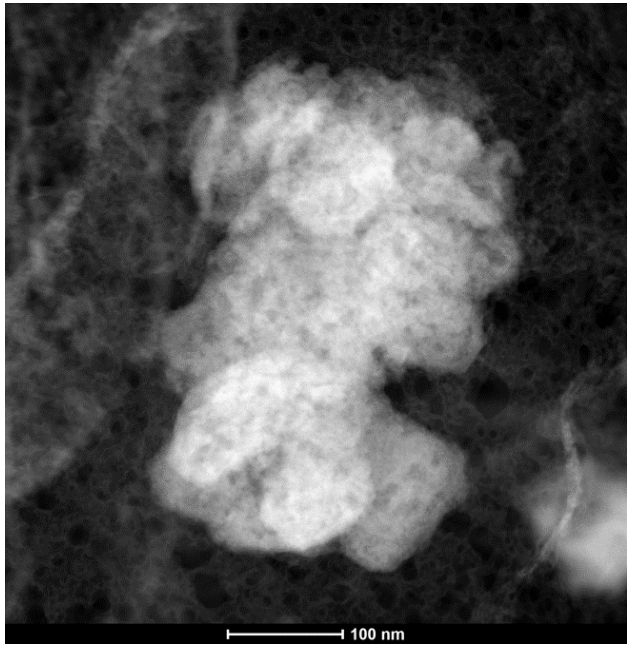
Examples from Research Studies at Columbia Nano Initiative Columbia University

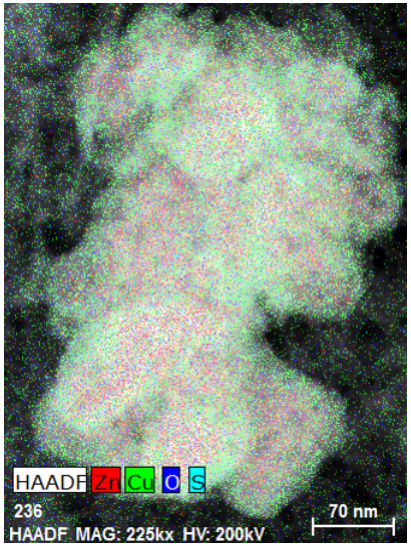




Solar-Thermal Energy Absorber





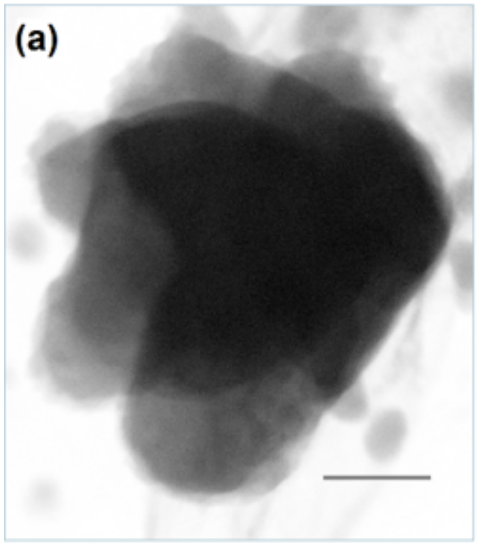


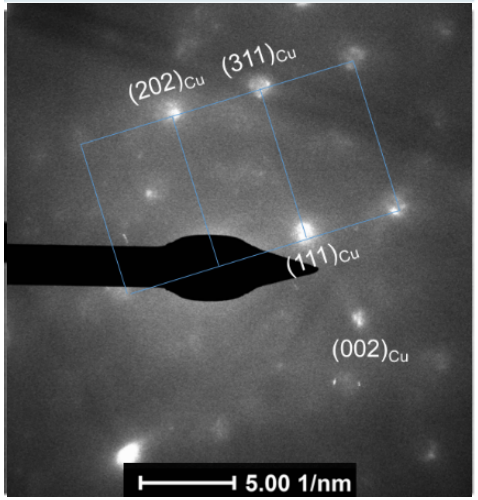
At 200 C, copper tends to diffuse outside of the particles

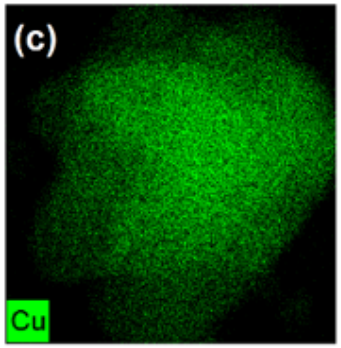
J. Mandal et al, Adv. Mater. 2017, 29, 1702156

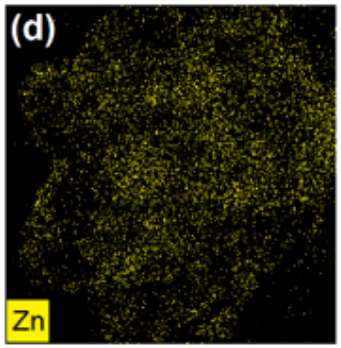
Courtesy of Prof. Y. Yang

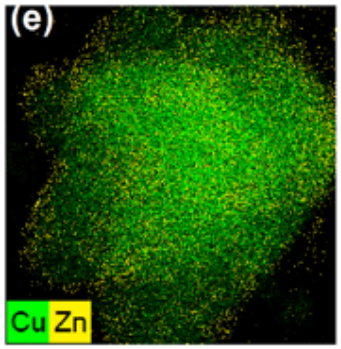
Solar-Thermal Energy Absorber

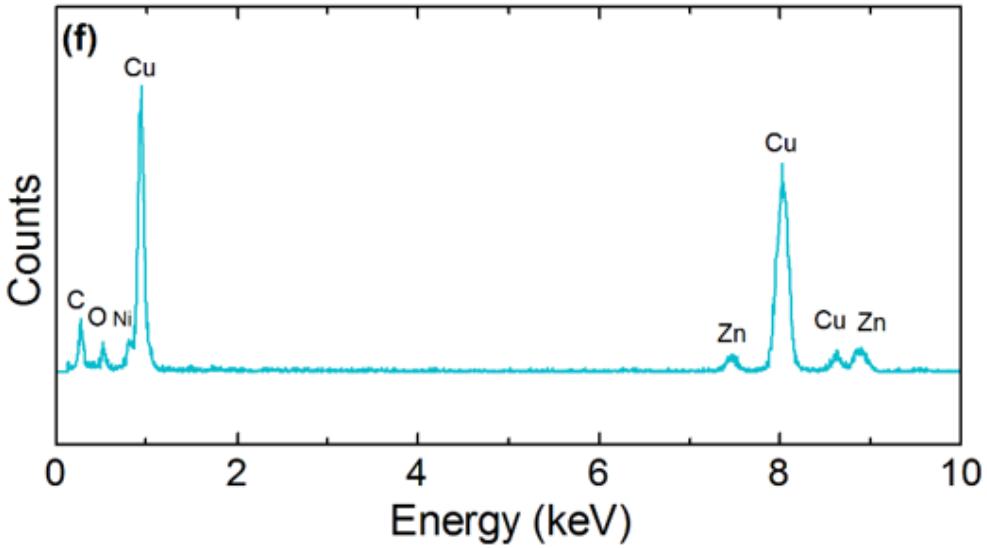


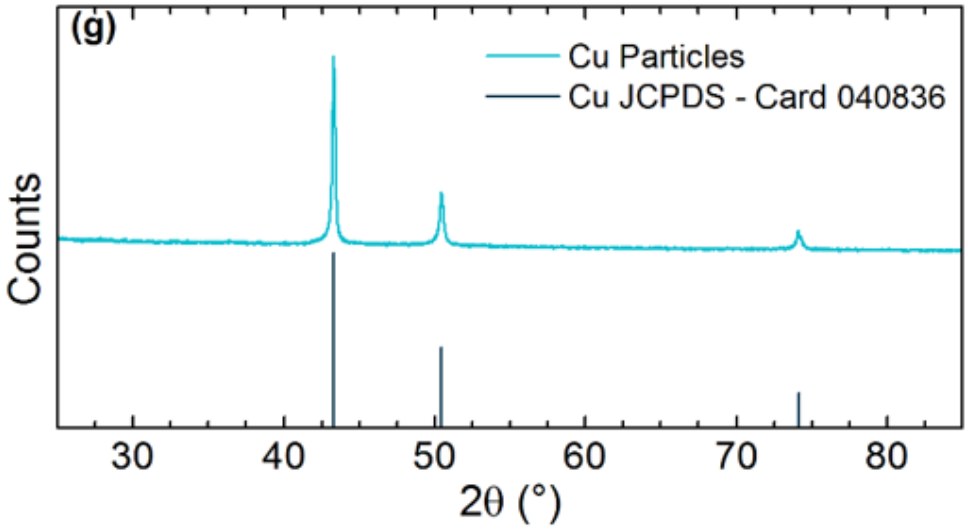








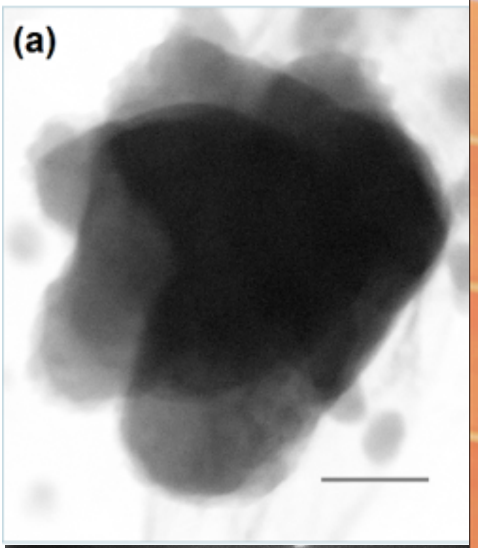


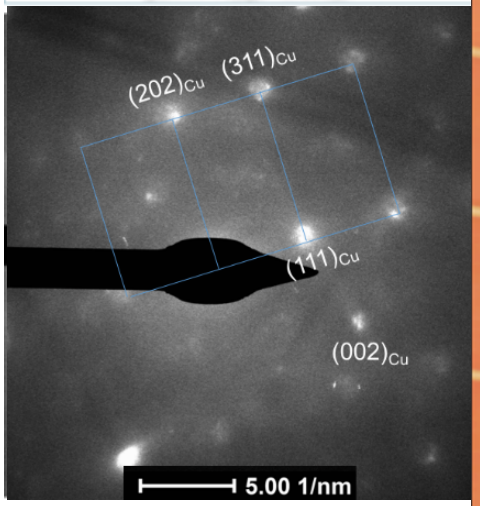


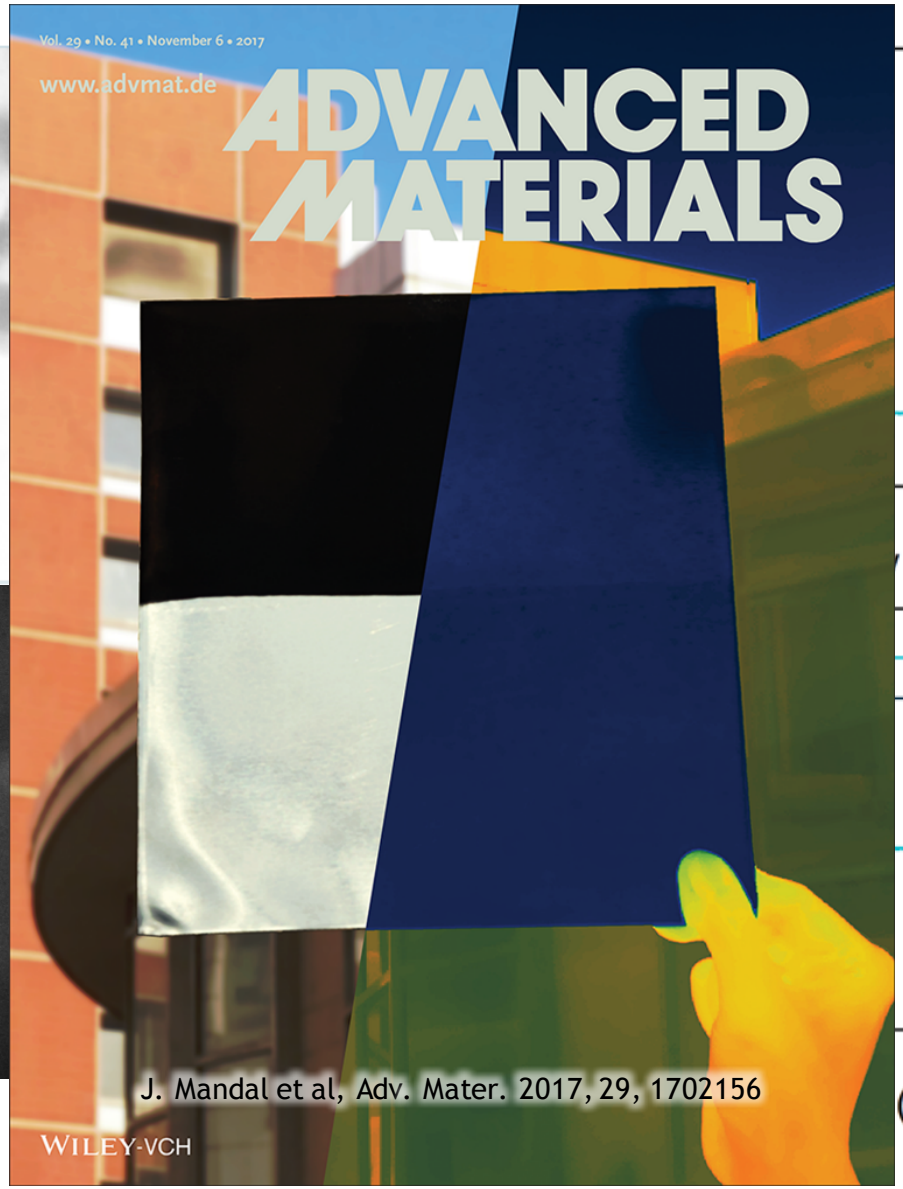
J. Mandal et al, Adv. Mater. 2017, 29, 1702156

Courtesy of Prof. Y. Yang

Solar-Thermal Energy Absorber







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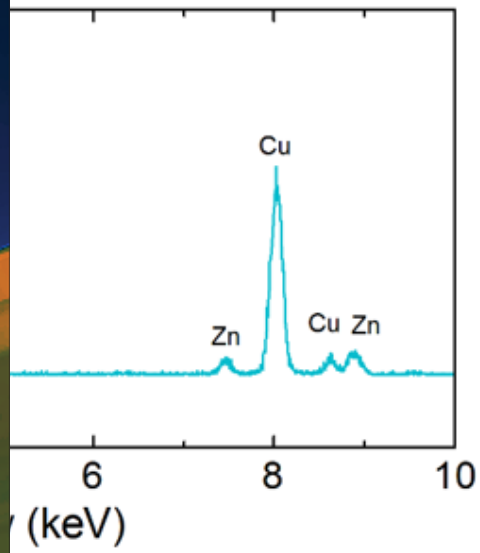
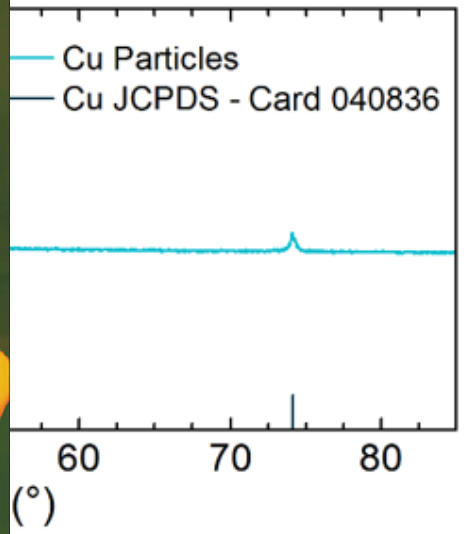




Courtesy of Prof. Y. Yang

Light Emitting Graphene

Applying voltage between two graphene layers leads to a short emission of light in specific spectrum (green, blue,…)

Cr/Au 5nm/300nm

SiO2 ~285nm

Graphene 5nm (red color)

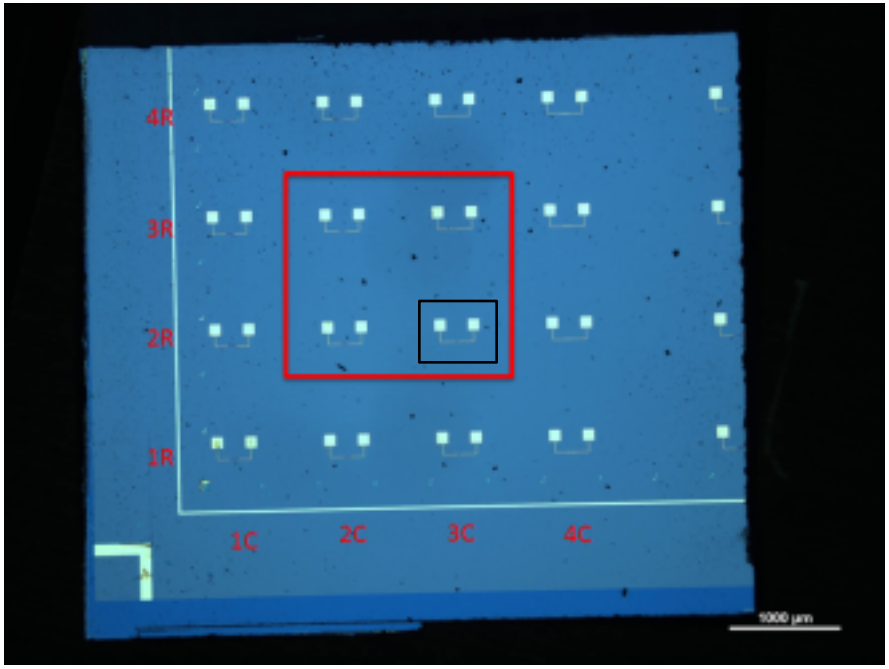
graphene oxide ~20nm (green color)

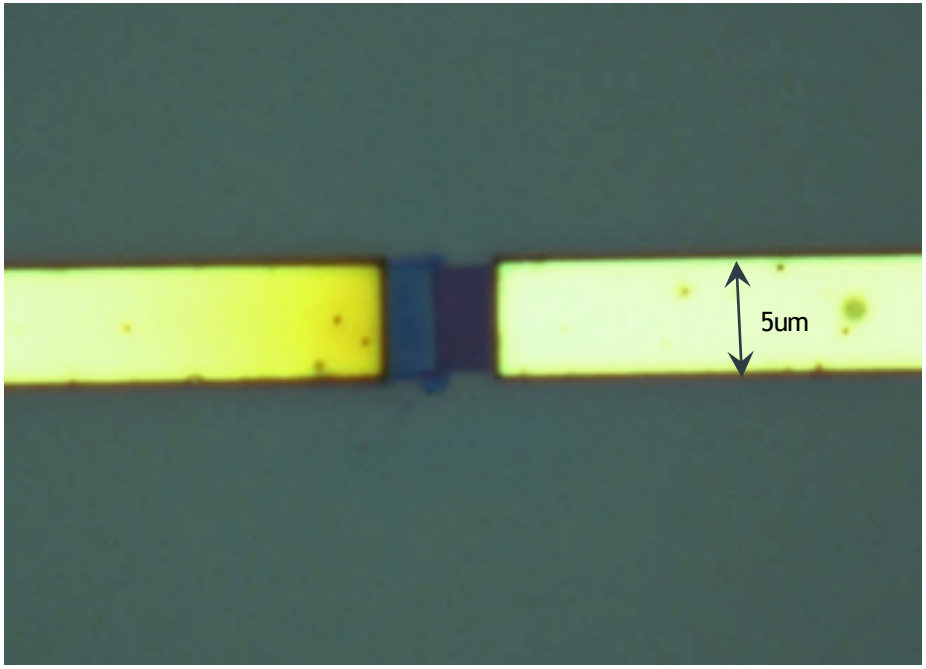
Si substrate 500um

Courtesy of Profs. K. Shepard, J. Hone, and K. Barmak

Light Emitting Graphene

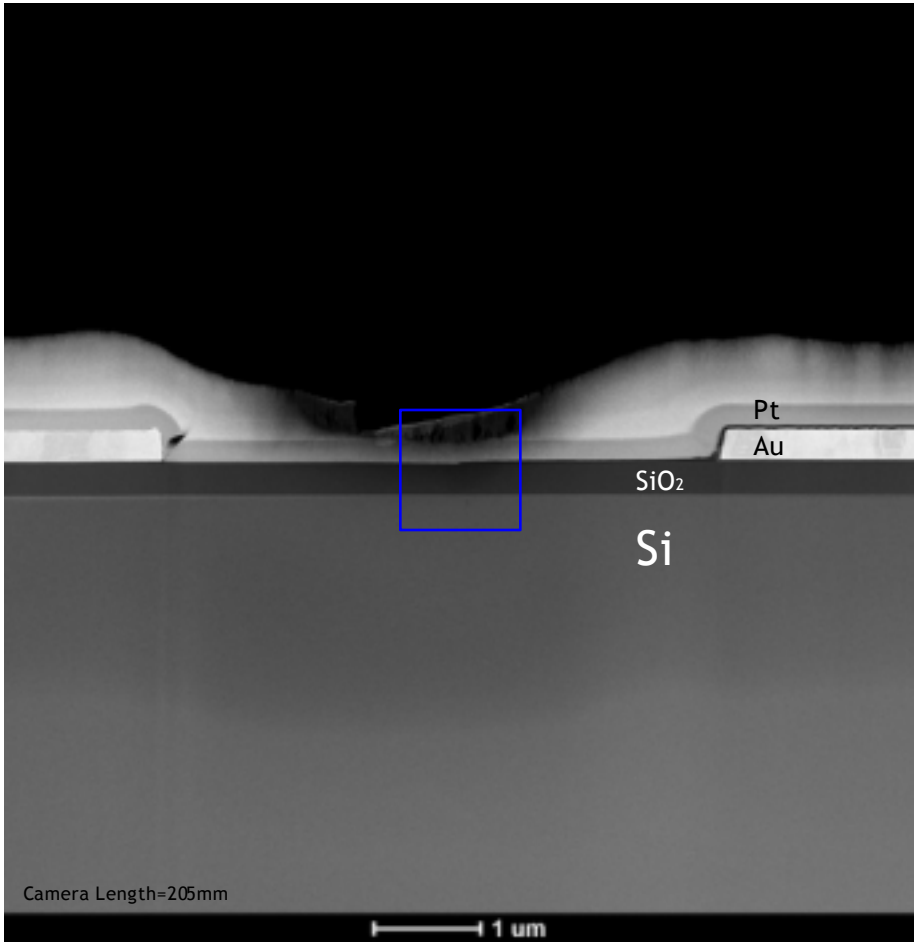
Making cross sectional TEM sample to study the chemistry and structure of the intersection

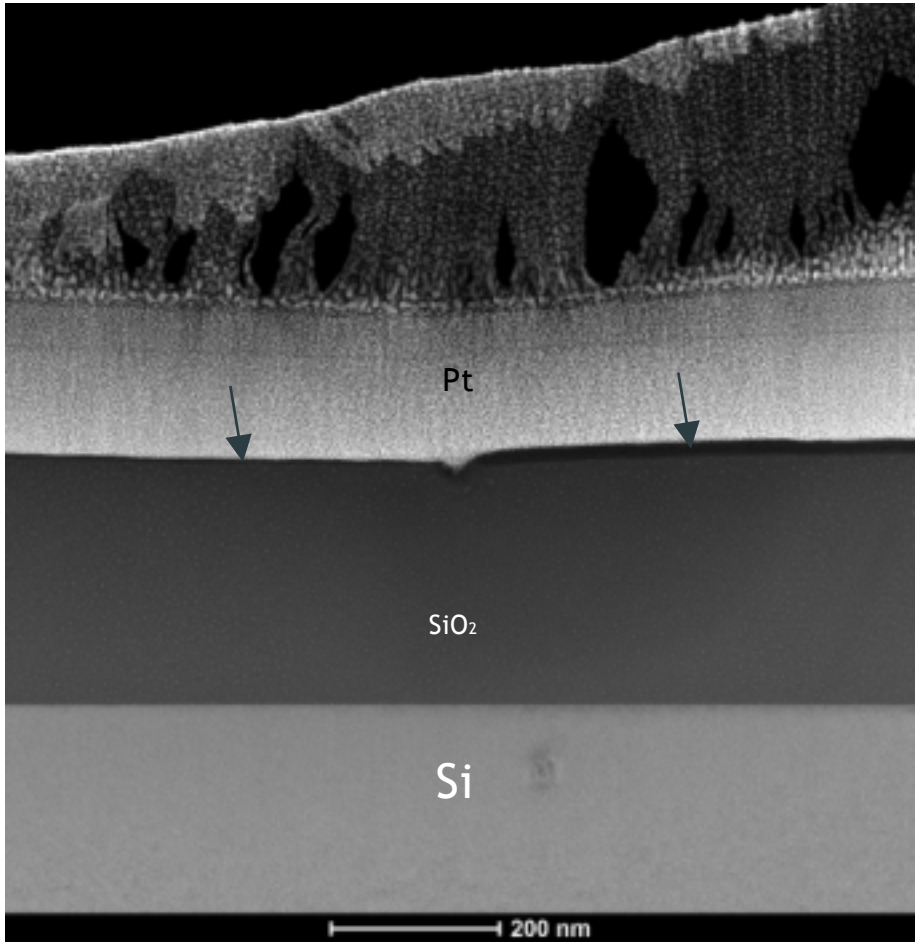




Courtesy of Profs. K. Shepard, J. Hone, and K. Barmak

STEM imaging

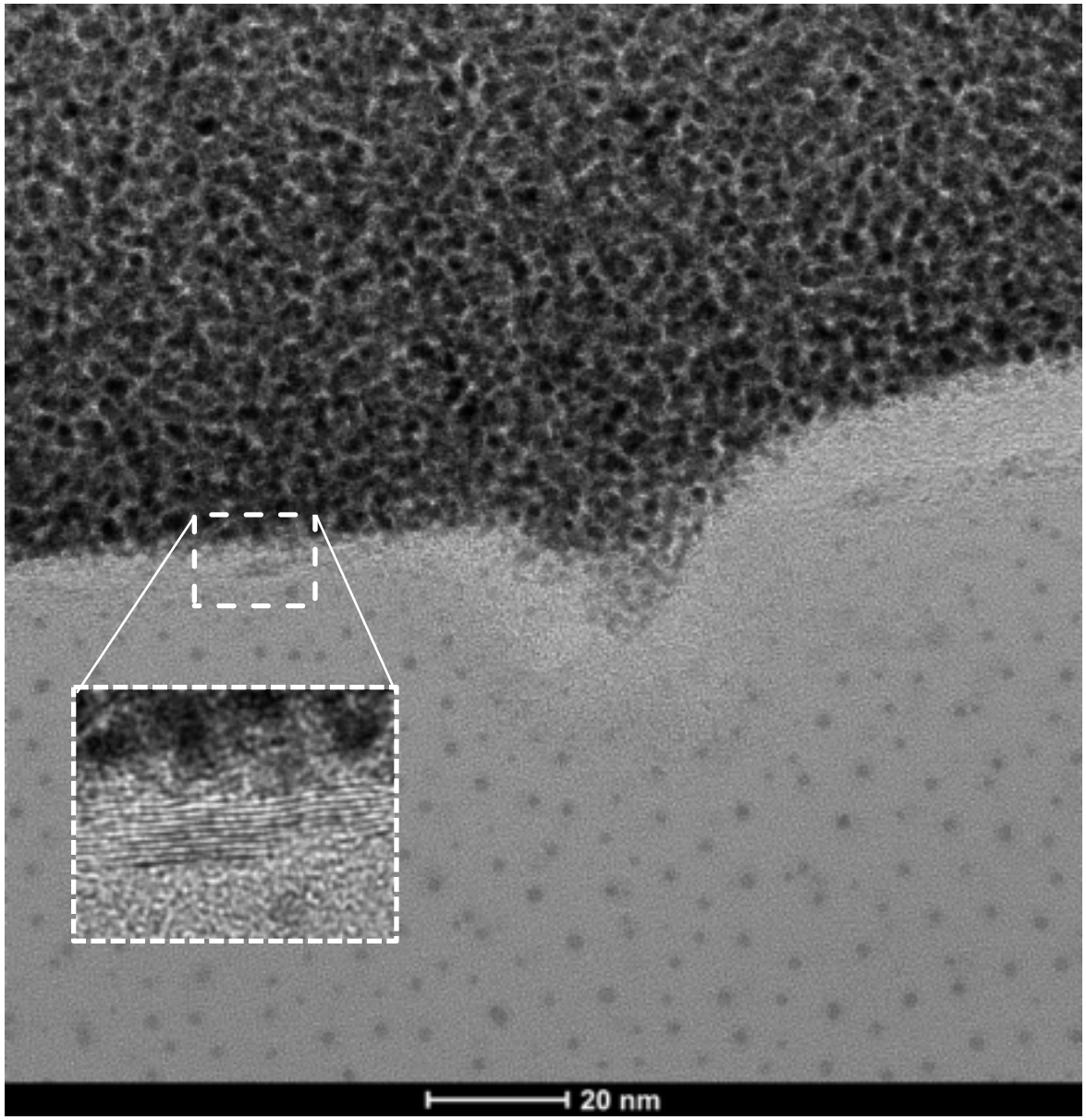




Courtesy of Profs. K. Shepard, J. Hone, and K. Barmak

Light Emitting Graphene

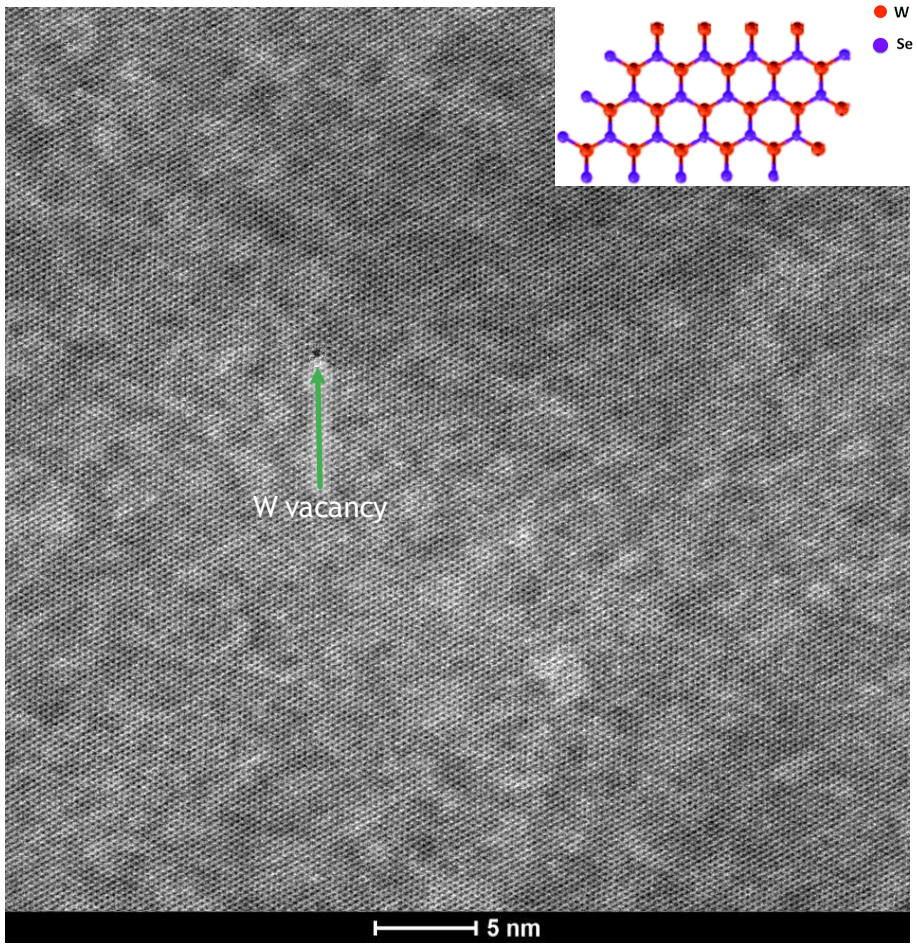
TEM-BF-imaging

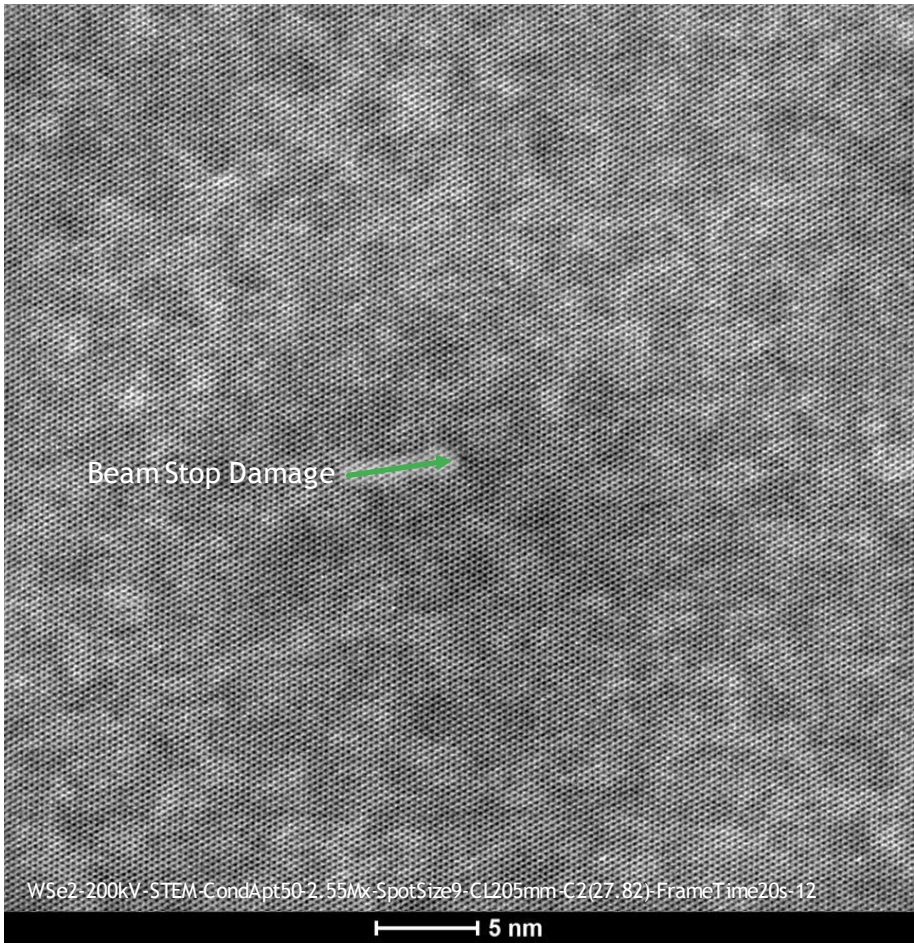


Courtesy of Profs. K. Shepard, J. Hone, and K. Barmak

Single Layer WSe_2 – Tungsten vacancy

- At this magnification, W vacancies are visible (not necessarily the Se vacancies)
- The defect density (W vacancy) estimated in the order of $10^{10}\mathrm{cm}^{-1}$, which is equivalent to finding 1 vacancy among ~100,000 W atoms. Each one of these pictures contain 20,000 W atoms.

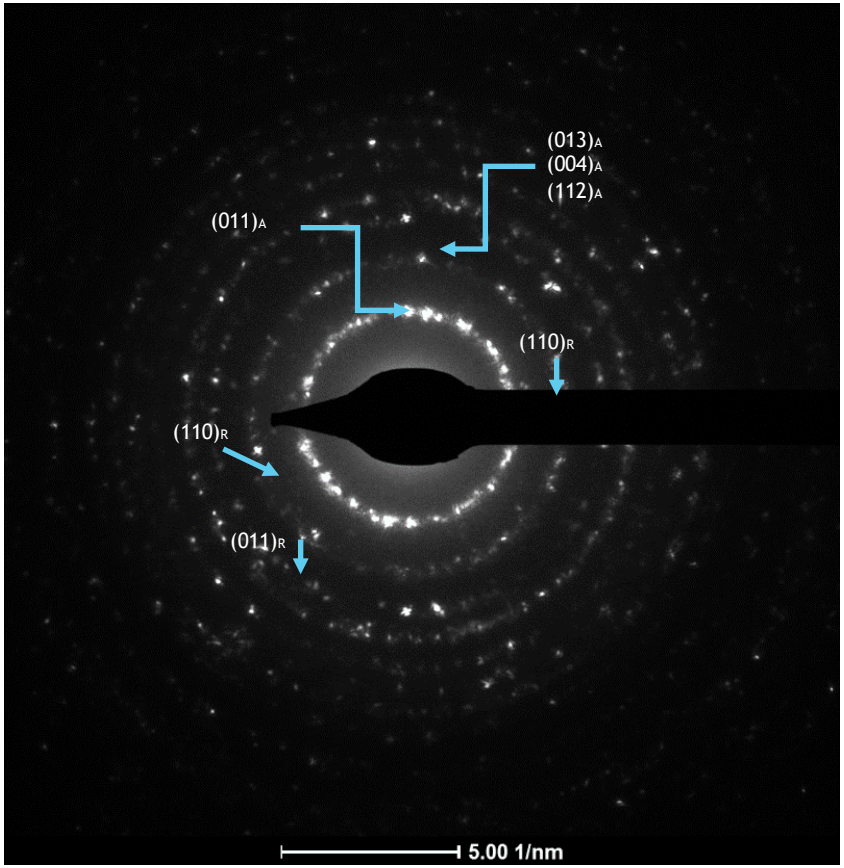


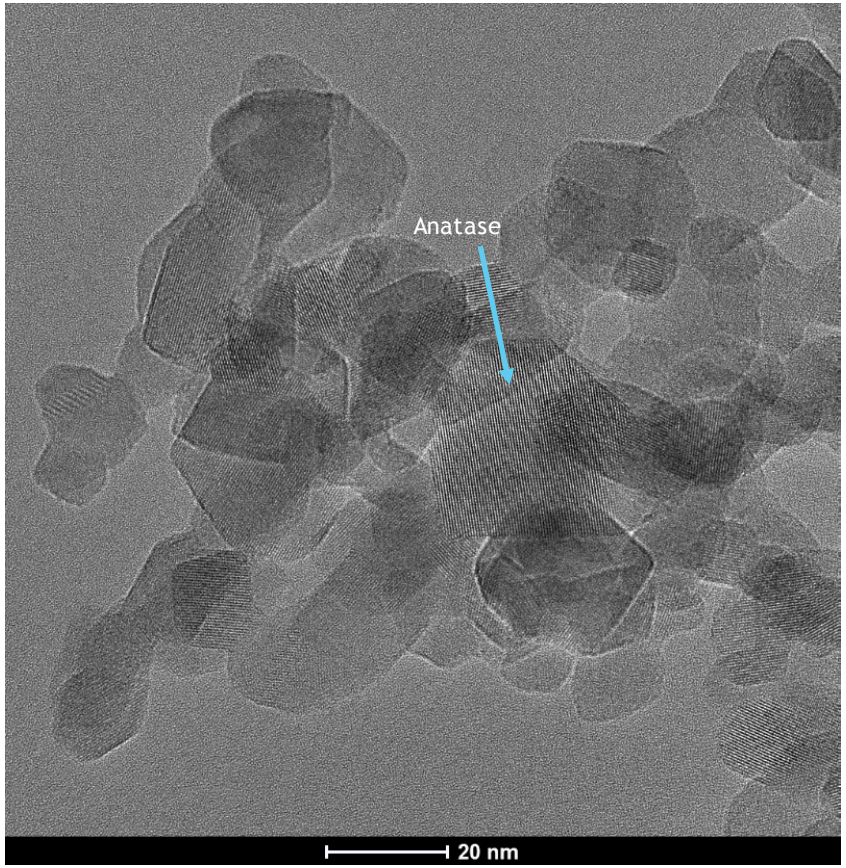


Courtesy of Profs. J. Hone and K. Barmak

Ruthenium on Titanium dioxide (Ru/TiO2) Catalyst – Collaboration with Barnard College

- The TEM images mainly show the anatase grains (with ~30 nm grain size)
- Some grains are oriented in a way the their atom columns are observable. This is being used to detect rutile grains.
- Faceting in some grains can be seen.

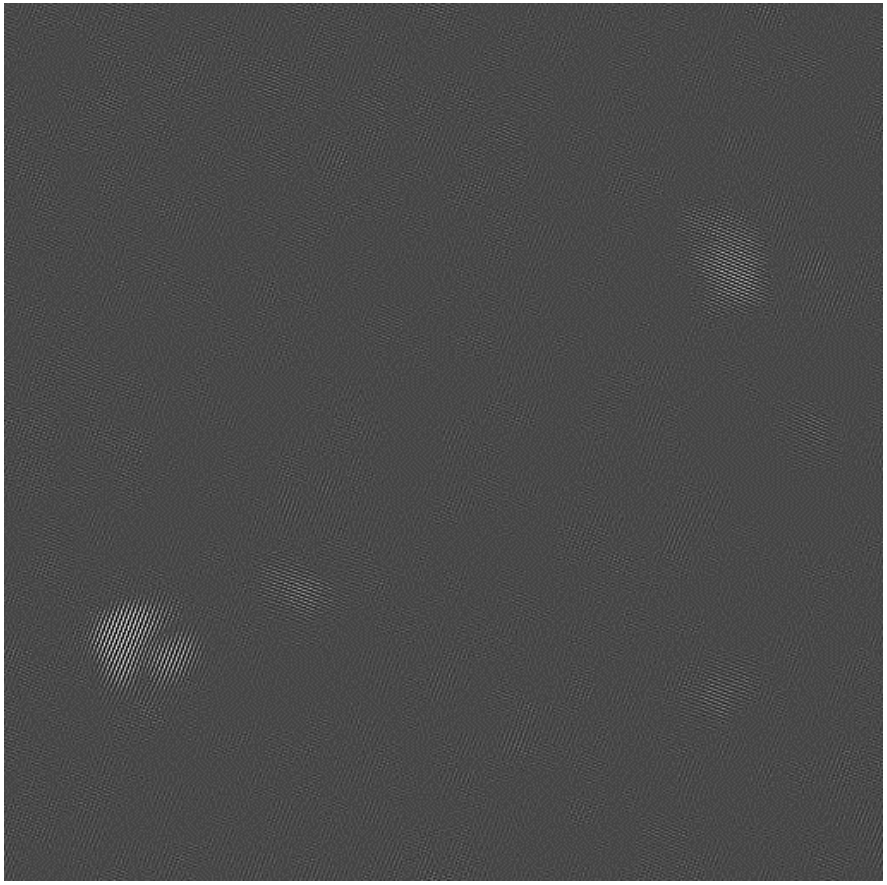


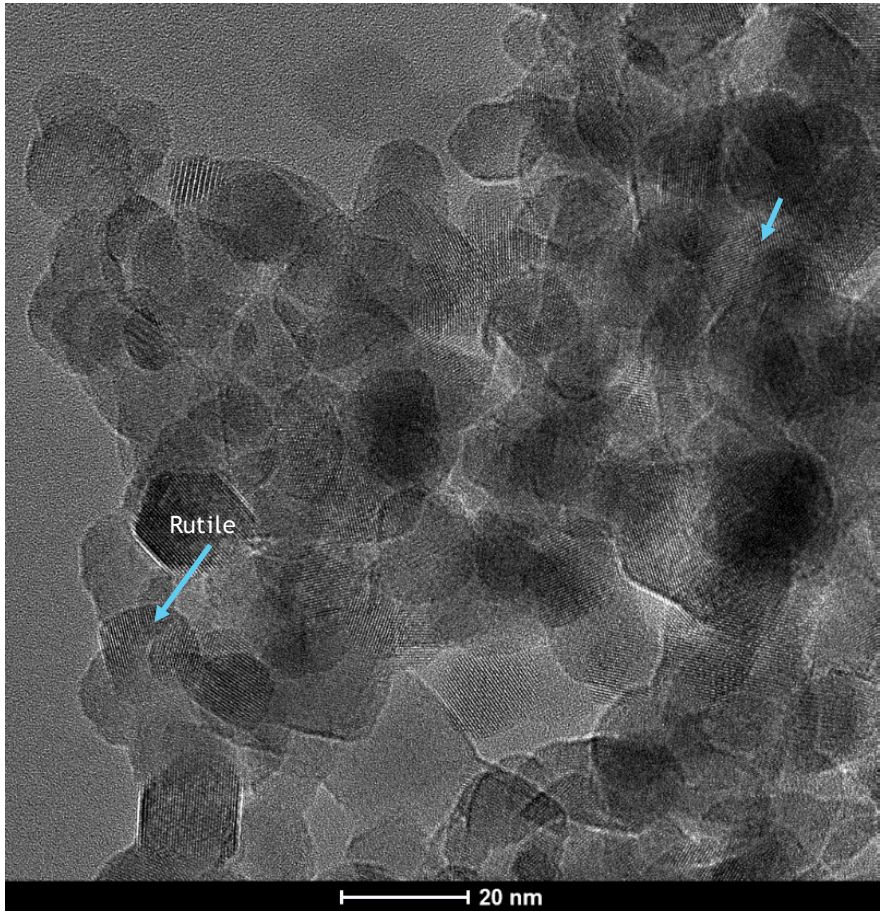


Courtesy of Profs. K. Barmak and R. Austin

HRTEM and Fast Fourier Transform (FFT) analysis

- After examining several HRTEM images, one rutile grain was found (which is fulfilling the Bragg angle). $(110)_R$ plane is masked.
- This grain is smaller/have similar size compared to the anatase grains.

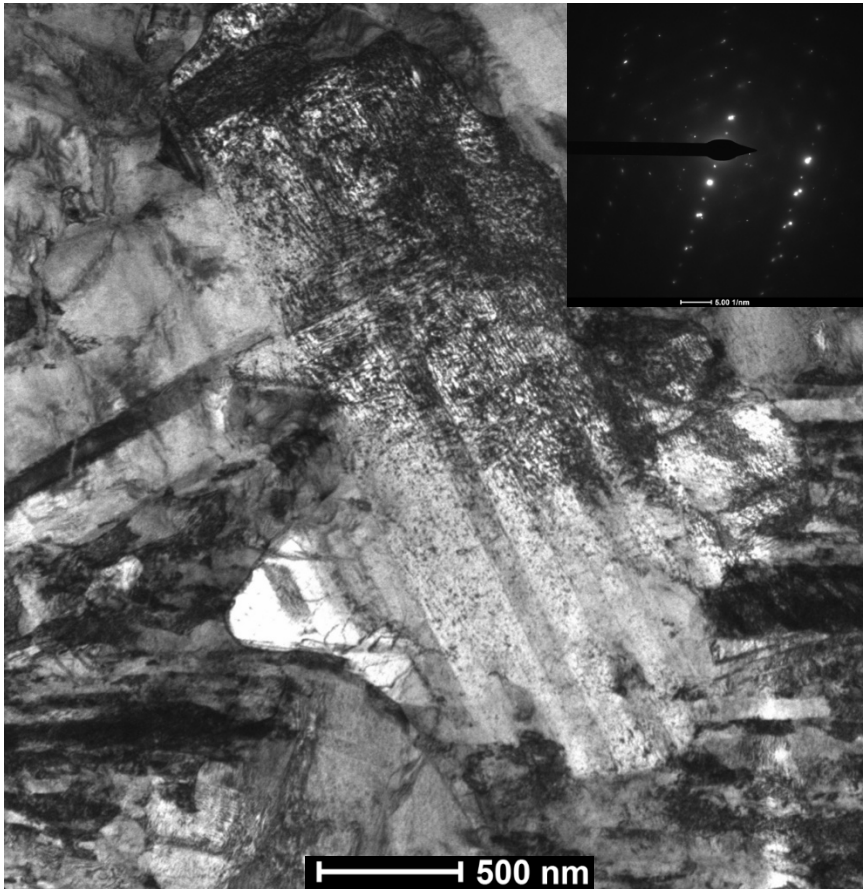


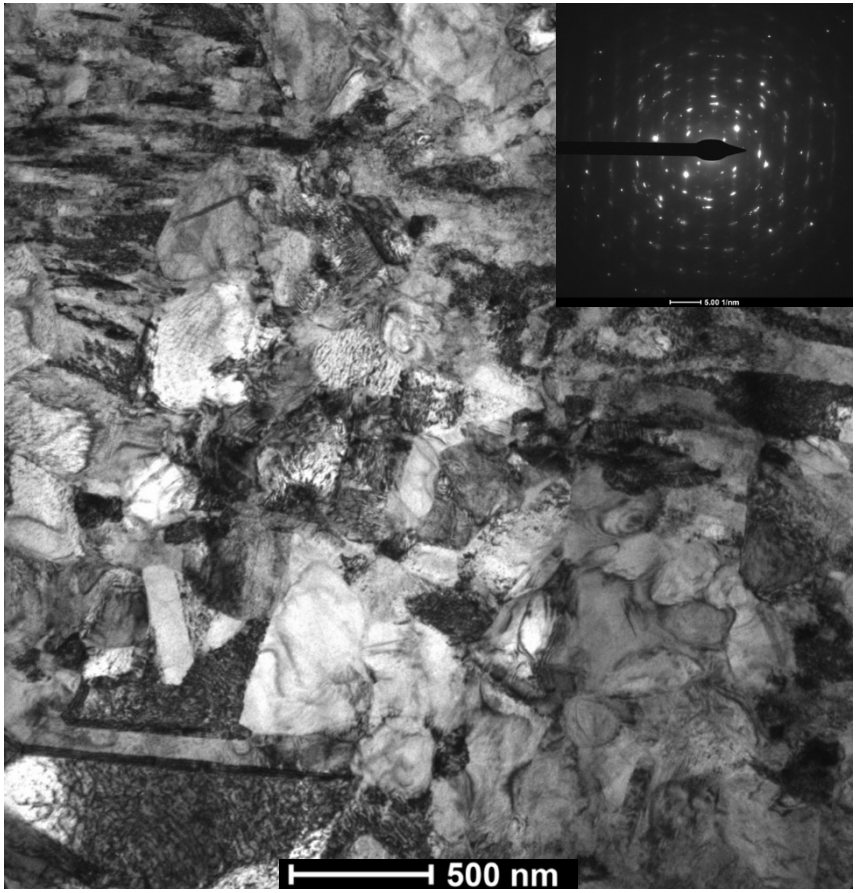


Courtesy of Profs. K. Barmak and R. Austin

FePd Highly Magnetic Material - Collaboration with Northeastern Univ.

20150831 Deformed 505˚C (TD-ND) – (TD-RD is under preparation)

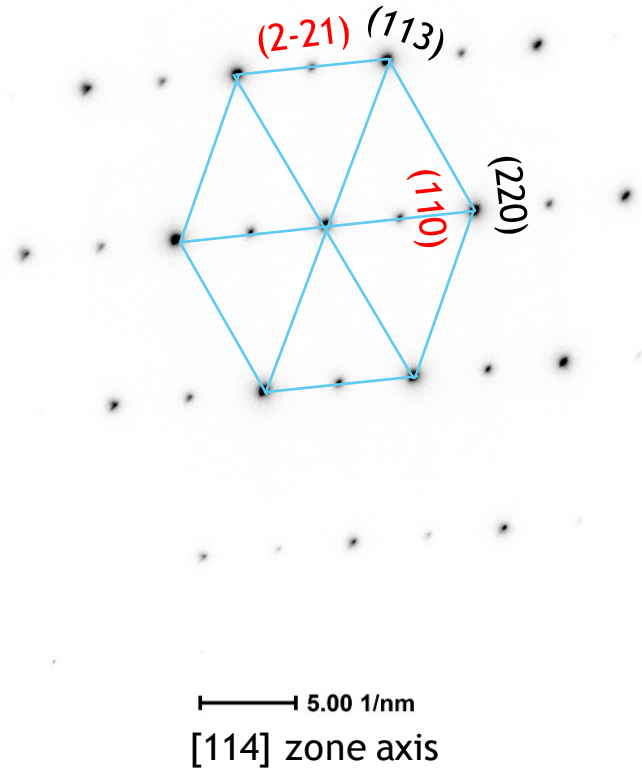




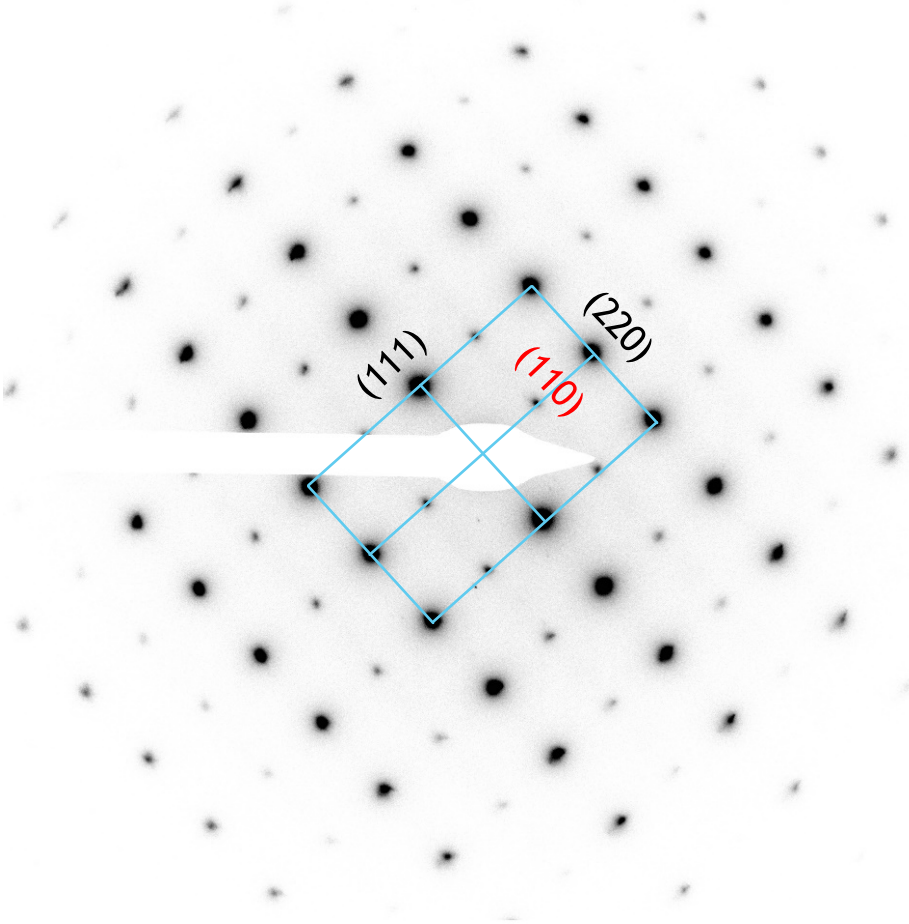
Courtesy of Profs. K. Barmak and L. Lewis

Collaboration with Northeastern Univ.

20150831 FePd Ames ISO 505-undeformed



[114] zone axis

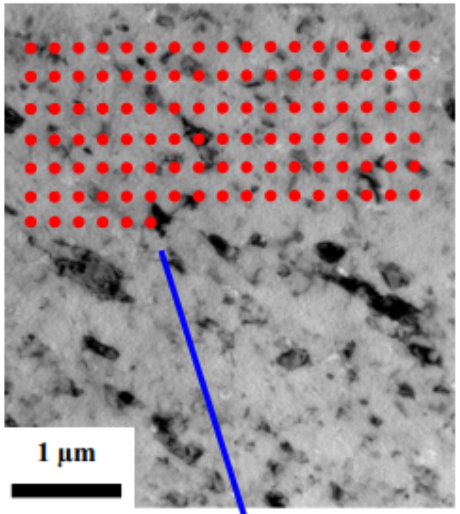


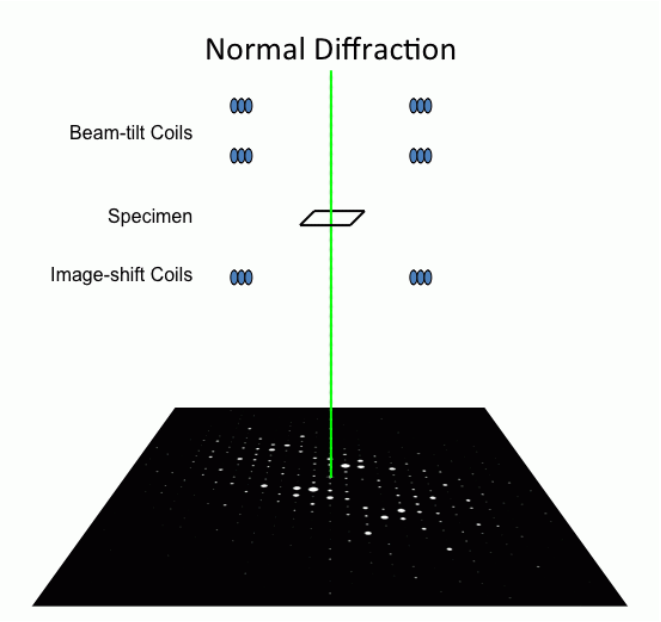
[110] zone axis

Courtesy of Profs. K. Barmak and L. Lewis

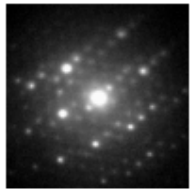
ASTAR – Crystal Orientation Mapping

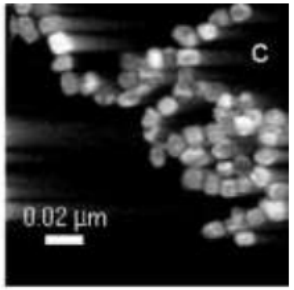
- A sample area is scanned by a nanometer electron beam
- Spot diffraction patterns are collected from scanned sample area
- Cross-correlation comparison of all acquired patterns with all simulated template
- Crystal orientation identification

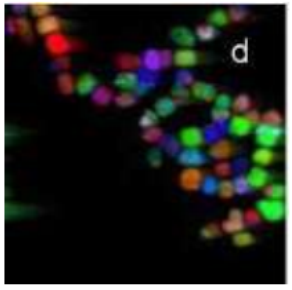




Example :Severely deformed 7075 Aluminium Alloy

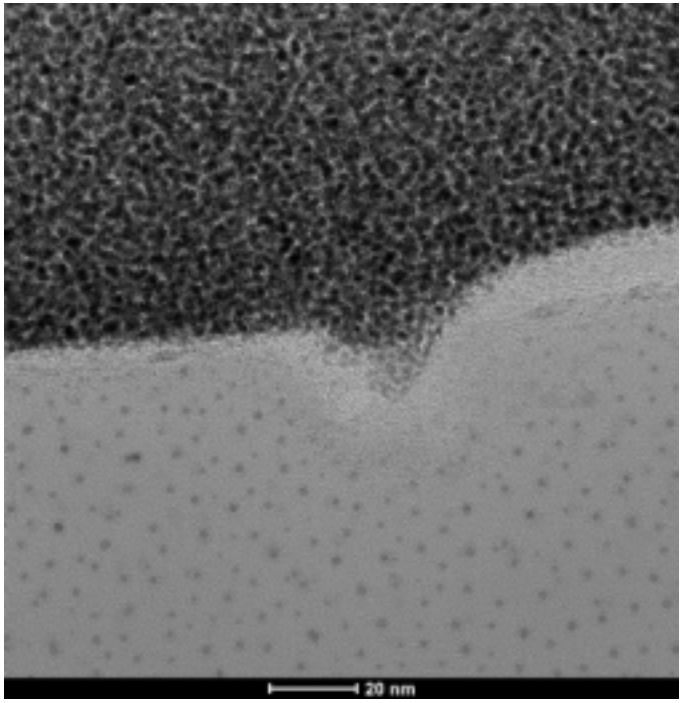


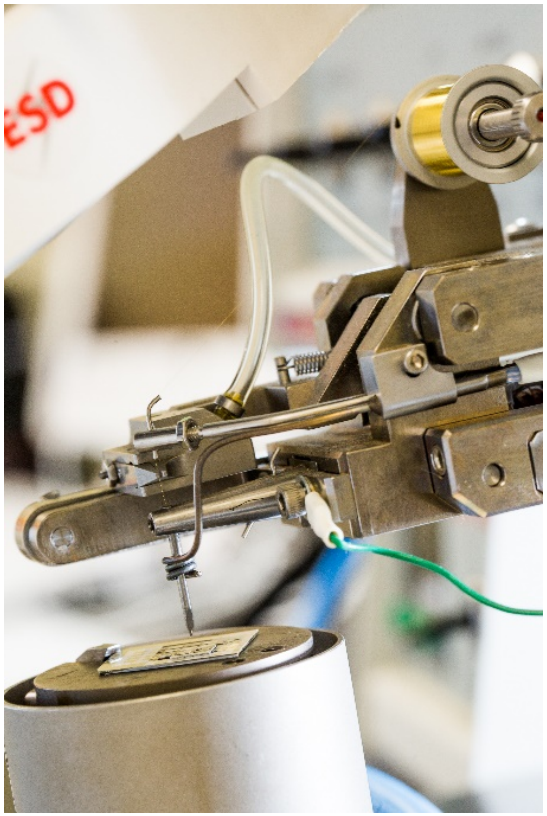




Question?

For more information go to:
http://cni.columbia.edu/shared-labs/ or contact:
na2661@columbia.edu
az2476@columbia.edu, or
cnilabs@columbia.edu

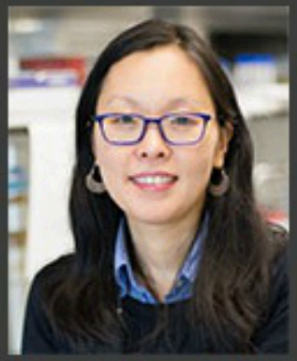




Our Next Webinar In The Series

Protein Engineered Nano Materials

Friday, March 23, 2018 at 1 PM Eastern Time



Presenter:
Jin K. Montclare, PhD
Associate Professor
Director of the Convergence for Innovation and Entrepreneurship
(CIE) Institute
New York University

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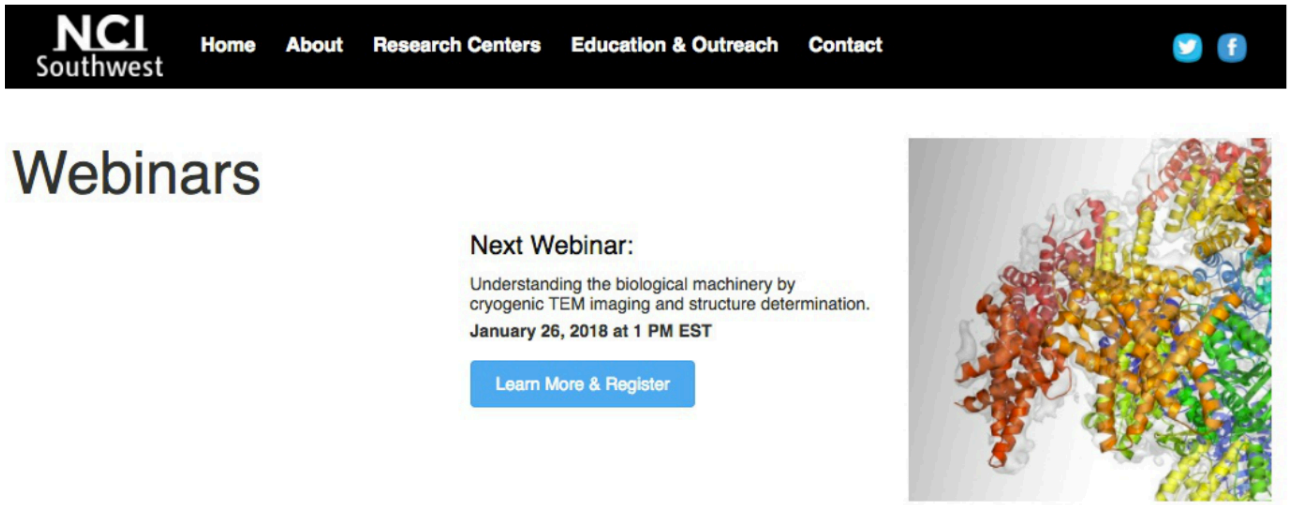

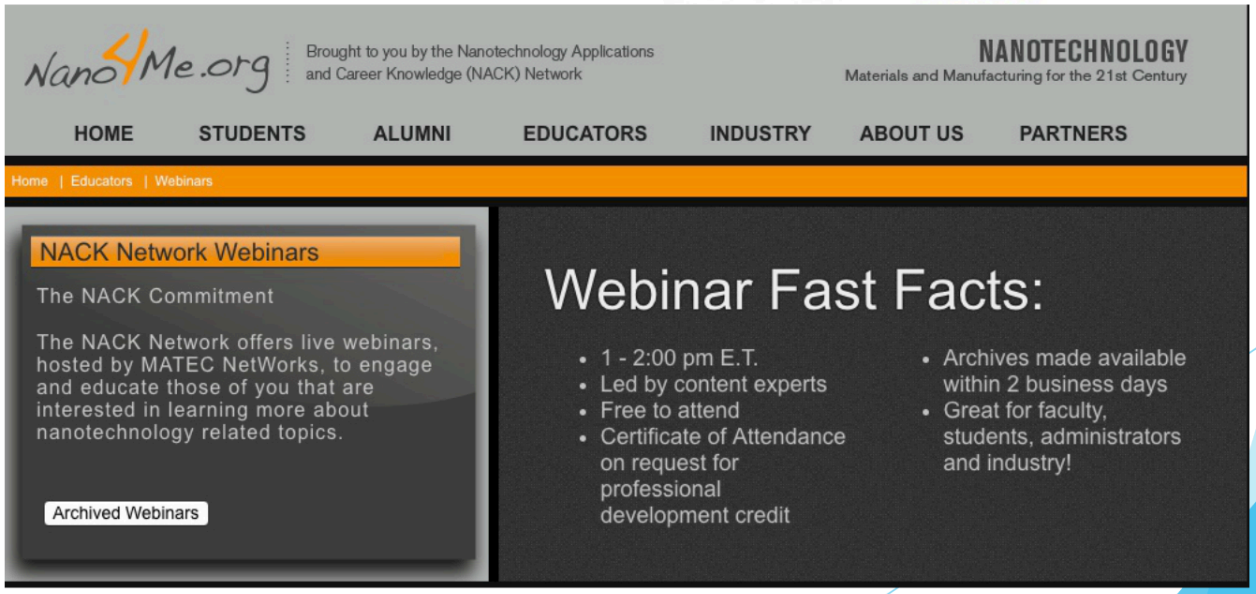

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