

Overview of BNC Research

Collaboration, Discovery and Delivery

J.P. Allain De La Torre for:
Monica Cortez Allain
Managing Director
Birck Nanotechnology Center



Discovery Park – a hub for Interdisciplinary Innovation and Commercialization



The BNC concept

- ◆ Engaging interdisciplinary, societal-scale challenges and opportunities in healthcare, information access, energy, and the environment with approaches enabled by nanoscale science and engineering
- ◆ Community encompassing a very broad span of disciplines
- ◆ In the same space to the degree possible
- ◆ The best in facilities and tools
- ◆ Everything shared
- ◆ Fluid...no “ownership” of space
- ◆ Sustainable financial model



The BNC facility – A unique instrument for nanoscale research



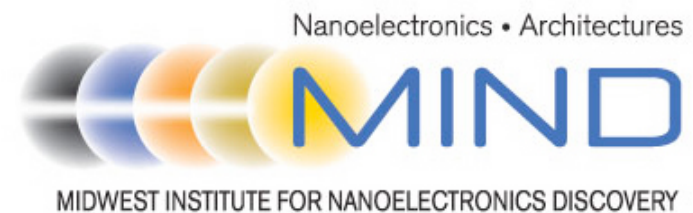
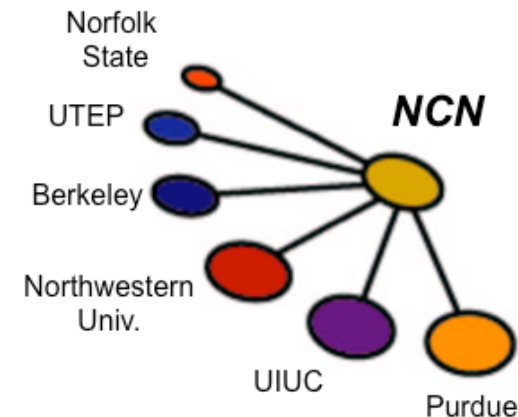
- 187,000 ft², \$58M + ~\$35M in equipment
- First integration of bio-pharma and semiconductor cleanroom
- Exceptional quality cleanroom - 25,000 ft²; 45 % Class 1 (ISO Class 3)



- Low Vibration (NIST A in cleanroom and A-1 in Hall lab)
- “Nanotechnology Grade” DI water plant (<15 ppt boron)
- 1, 0.1, and 0.01°C control
- Nanotech research space available for start-up companies

Research strengths

- ◆ **Nanometrology**
- ◆ **Nanoelectronics**
- ◆ **Computational nanotechnology**
- ◆ **Nanophotonics/plasmonics**
- ◆ **Energy conversion**
- ◆ **MEMs/NEMs and microfluidics**
- ◆ **Nano-bio**
 - ◆ Nanobiotechnology
 - ◆ Nanomedicine
 - ◆ Implantable microdevices

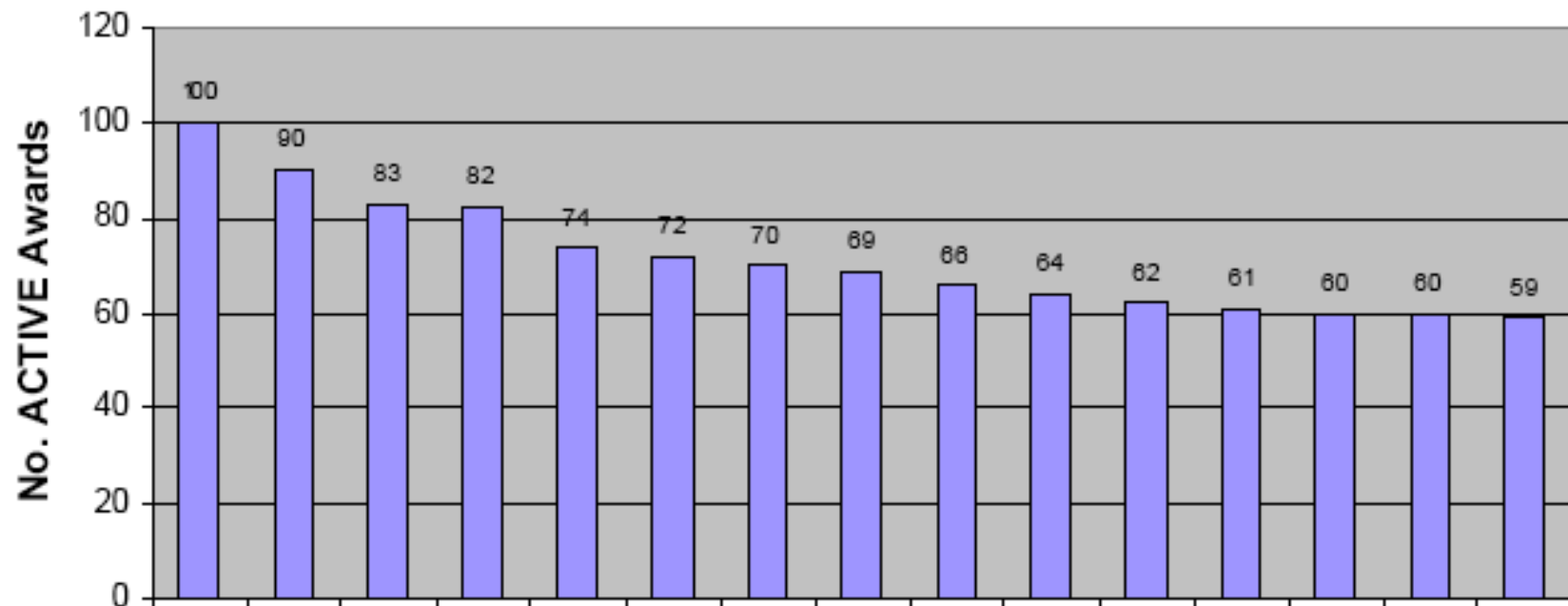


A unique instrument for nanoscale research

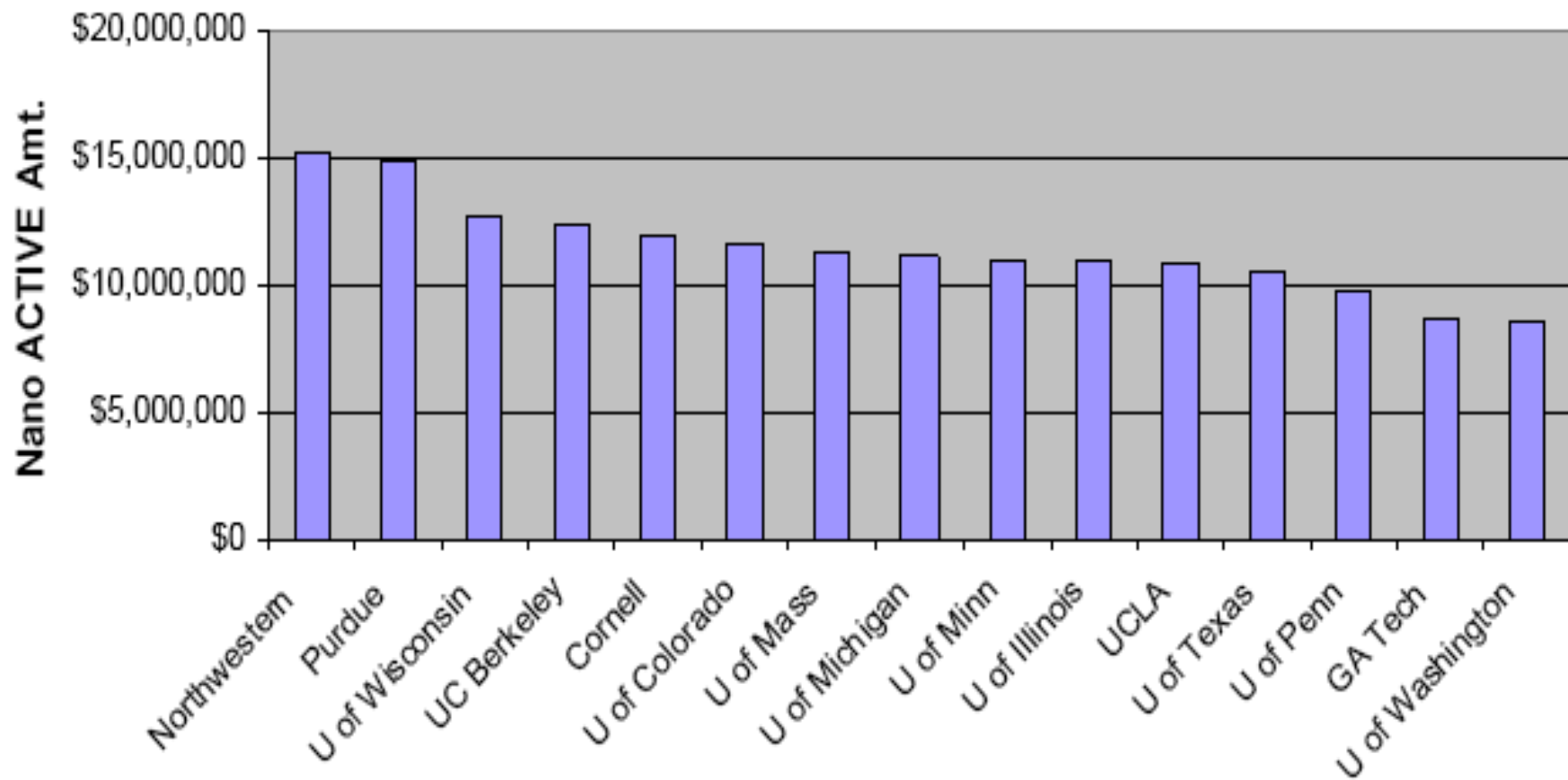


- Scanning Tunneling Microscopy (STM)
 - NIST A-1 (Vibration control)
 - Temperature control (0.01°C)
 - Electromagnetic Shielding
 - Scanning Probe Microscopy (SPM)
 - Transmission Electron Microscopy (TEM)
 - Atomic Force Microscopy (AFM)
 - X-ray Photoemission Spectroscopy (XPS)
 - Scanning Electron Microscopy (SEM)
 - X-ray Diffraction
 - Auger Electron Spectroscopy (AES)
- MUCH-MUCH More

Top 15 Institutions by Number of Active NSE Awards in 2009

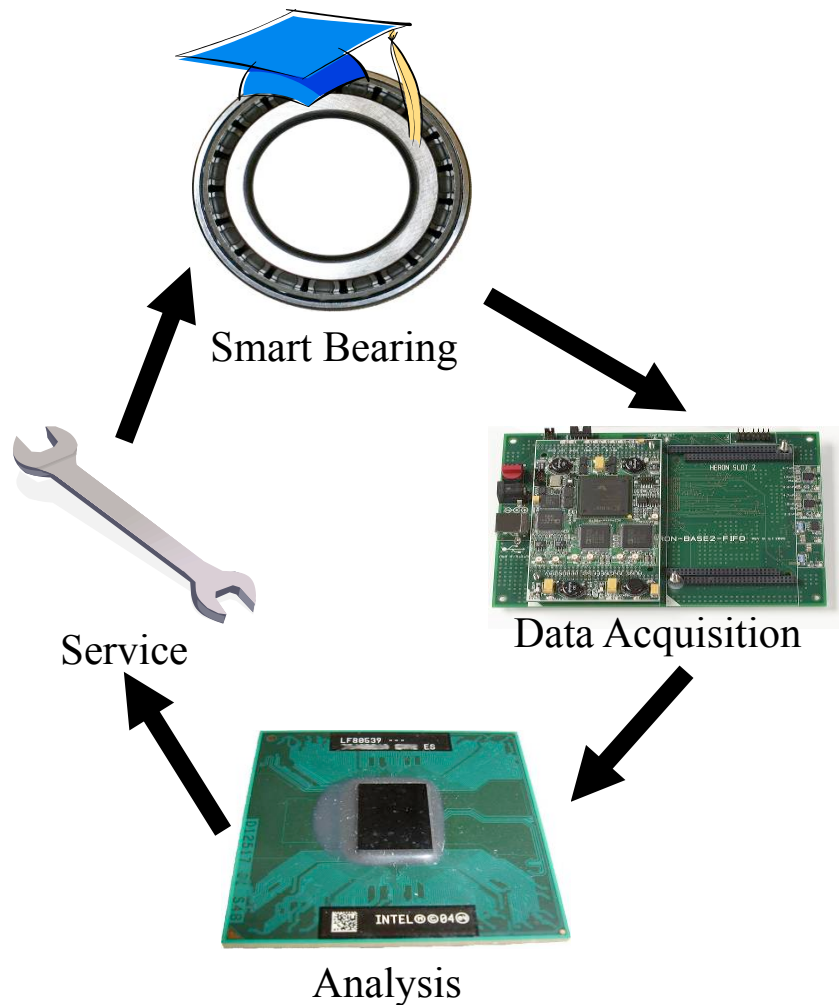


Top 15 Institutions by NSE Active Amount in 2009



M.C. Roco, NSF Nanoscale Science and Engineering Grantee's Conference, Dec.7, 2009.

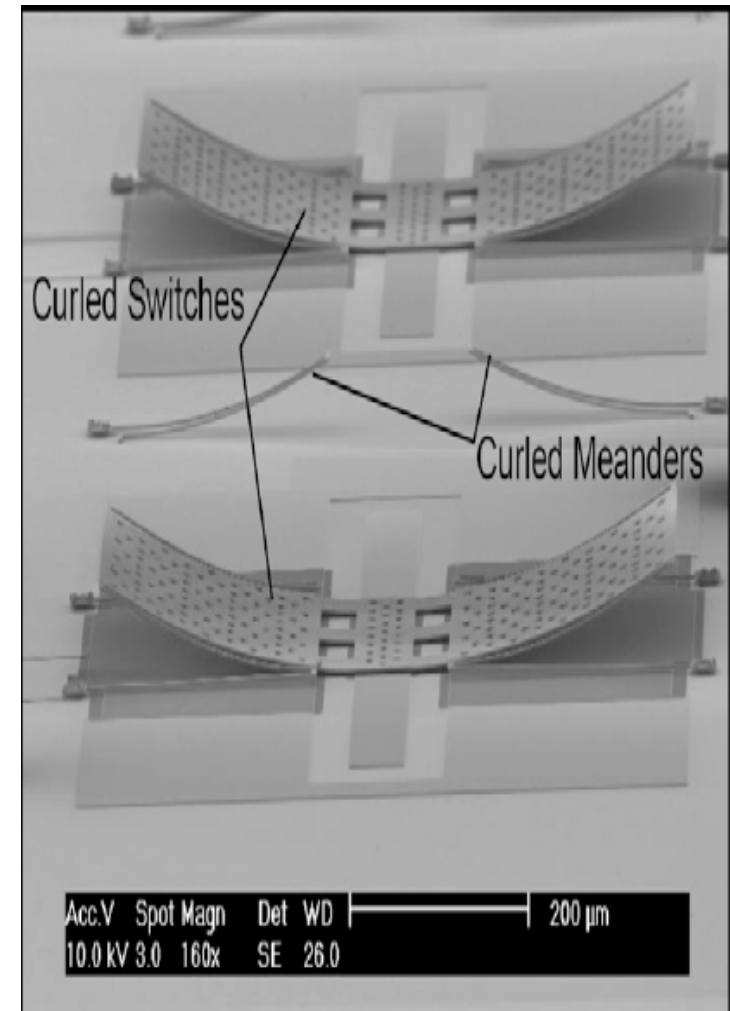
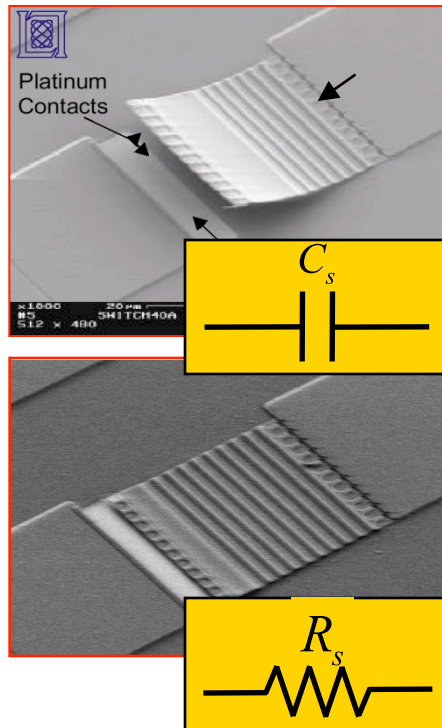
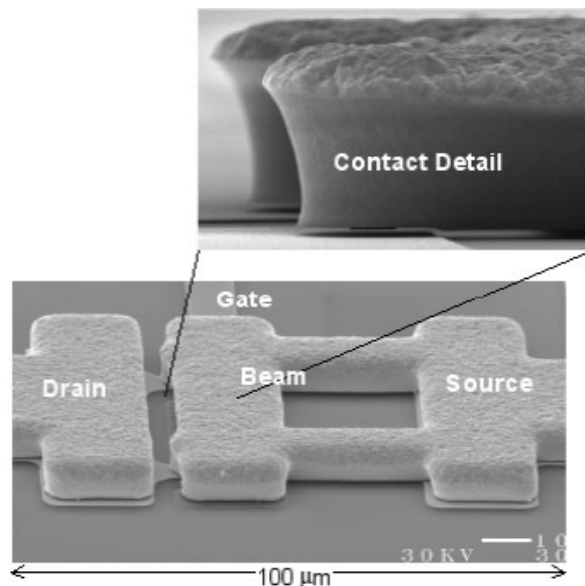
Wireless Harsh-Environment MEMS Sensors



- Maintenance of military aircraft consumes a large amount of time and resources
 - *CH-53E requires > 44 hours of maintenance / hour of flight time*
- Limited ability for condition monitoring

MEMS . . .

Micro-Electro-Mechanical Systems

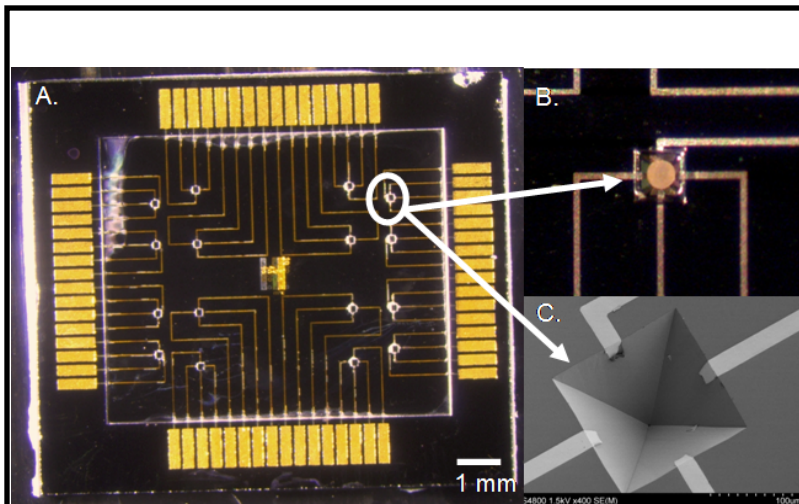


Applications . . .

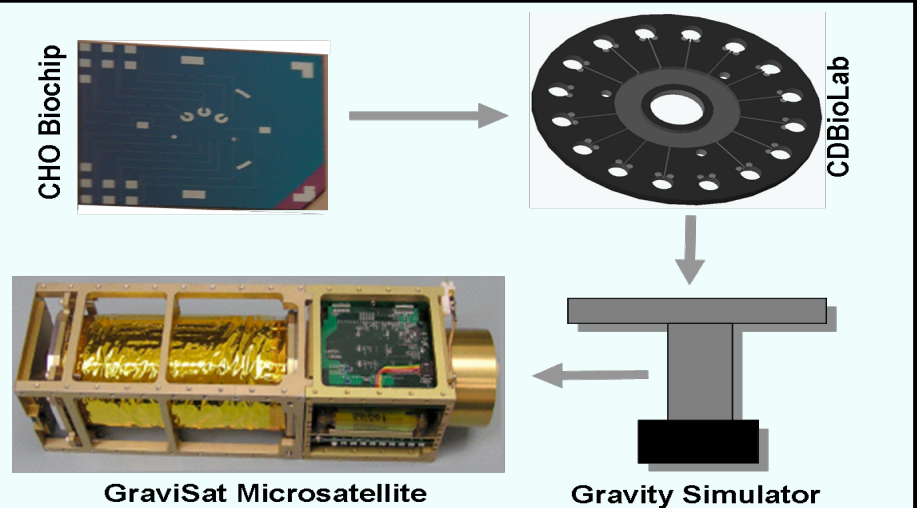
- Bearing Life Monitoring
- Switches and Capacitors in RF Circuits

Lab-on-a-chip based Biosensors

- Lab-on-a-chips fabricated using silicon microfabrication techniques
- Wet and dry etching, metal evaporation and SU-8 photolithography
- Sensor selectivity imparted through specialized chemical formulations coated on electrodes
- Voltage or current based sensing
- Applications
 - Fundamental Biological/Biomedical Research
 - Biomedical Diagnostics
 - Environmental Monitoring
 - Agriculture



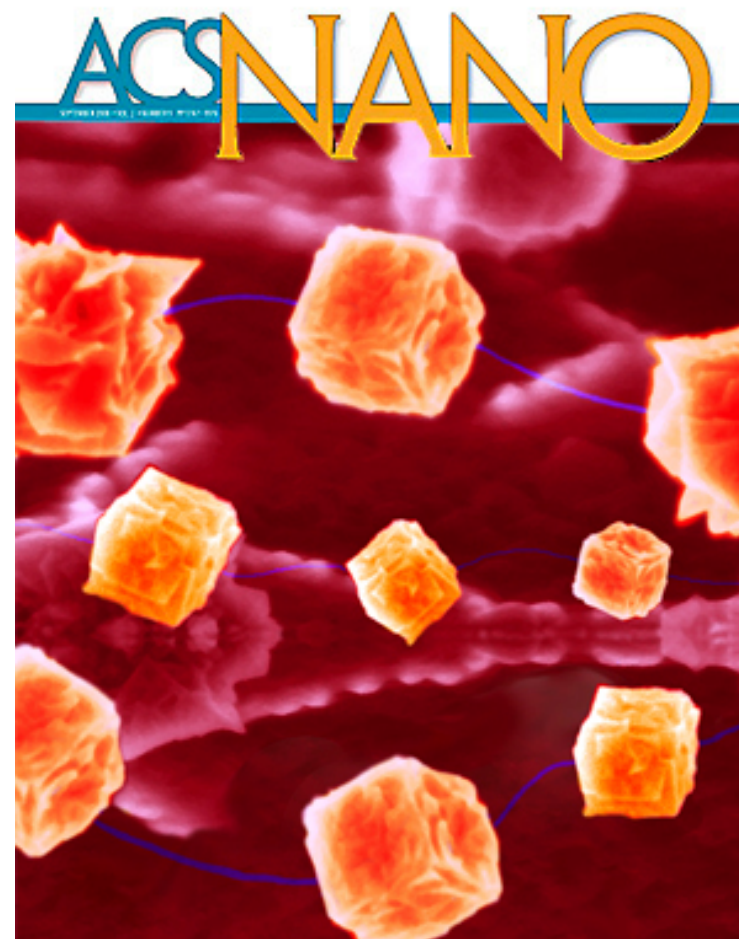
CEL-C Biochip – Sensors for Measuring calcium concentrations around single cells



CHO Biochip – Sensors for measuring pH, carbonate and oxygen around cultured bacteria in space

Nano-tetherball biosensor

- Precisely detects glucose
- Nanotubes have a diameter (2nm)~25,000x thinner than human hair
- Sensor requires 5x less glucose to generate a signal
- Sensor has wider range of glucose concentration
- SWNT serve as electronic sensor



In the press:

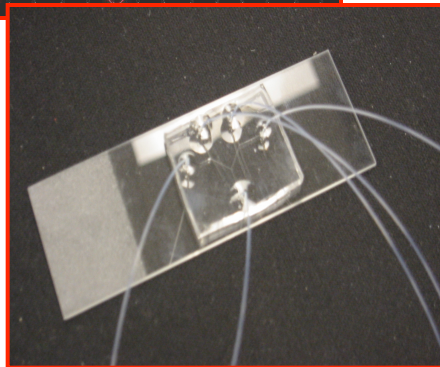
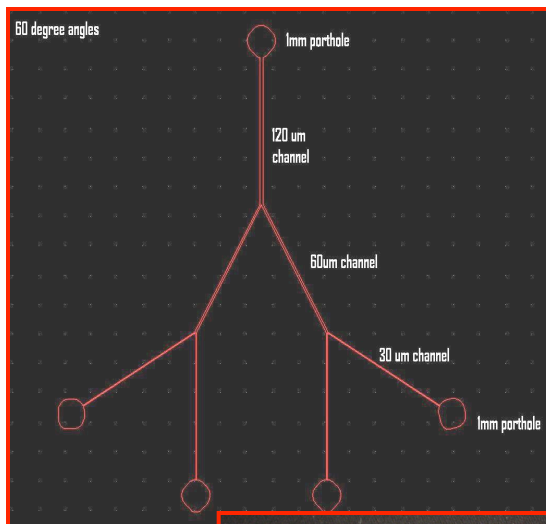
Magnetic Ferro-Paper (Prof. Babek Ziaie, ECE)

WEST LAFAYETTE, Ind. - Researchers at Purdue University have created a magnetic "ferropaper" that could be used to make low-cost "micro-motors" for surgical instruments, tiny tweezers to study cells, and miniature speakers. The research is based at the Birck Nanotechnology Center in Purdue's Discovery Park.



Example - Government-Industry-University partnership: A Portable BioMEMS Blood Analyzer for NASA

Meggie Grafton¹⁻³, Lisa M. Reece²⁻⁴, Paul W. Todd⁵, James F. Leary¹⁻⁴



¹Weldon School of Biomedical Engineering, ²Birck Nanotechnology Center, ³Bindley Biosciences Center, ⁴Basic Medical Sciences, Purdue University
⁵Techshot, Inc. (Greenville, IN).

This NASA-SBIR funded academic-industry partnership between Purdue and Techshot, Inc, is addressing an urgent NASA need – rapid blood analyses to assess the health of astronauts in space. The portable BioMEMS device, being designed and fabricated in the Birck Nanotechnology Center, analyzes absolute red and white blood cell numbers as well as assessing the altered immune cell status of the astronauts in micro-gravity environments.

BNC Collaborations Provide..

- Collaboration with top researchers in various core areas of nanotechnology.
- Access to state-of-the-art equipment
- Resources to enhance Funding Opportunities
 - OVPR Large Proposal Development Services (>1M)
 - Proposal Preparation Resources
 - Seed Grant Programs (Internal to Purdue and in collaboration with IU and/or other institutions)
 - Possible Cost sharing opportunities
- Research Space and Office of Technology Commercialization expertise

Bindley Bioscience Center



29,000 ft² of laboratory space for
interdisciplinary life sciences &
bioengineering

Shared faculty offices

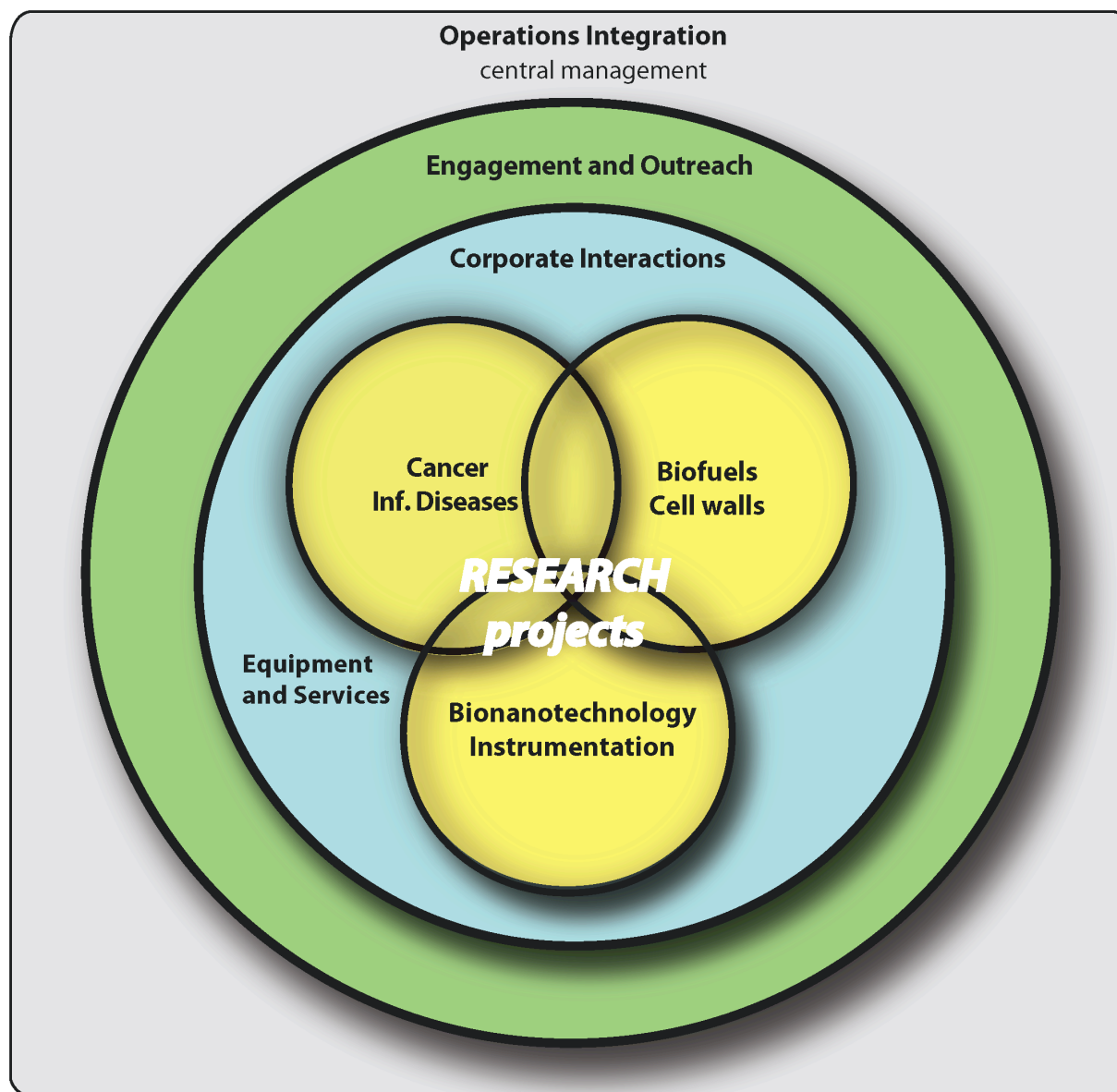
\$15 million 2-story structure

Skywalk to Birck Nanotechnology Center

Flexible Research Space

- Creates an open environment for interactive communication.
- Allows for dynamic lab spaces rather than disciplinary-defined labs for:
 - Biomolecular technologies (esp. metabolomics and proteomics)
 - Advanced imaging. cytometry, high speed cell sorting
 - Biophysical analysis. physiological biosensor development
 - Integrated screening (high content, chemical and biological screening, protein expression)
 - Center for Global Research and Intervention in Infectious Diseases (cGRIID)
 - Center for Analytical Instrumentation Development (CAID)
 - Nanochemistry, biopolymer synthesis
 - Computational biology, bioinformatics and systems biology

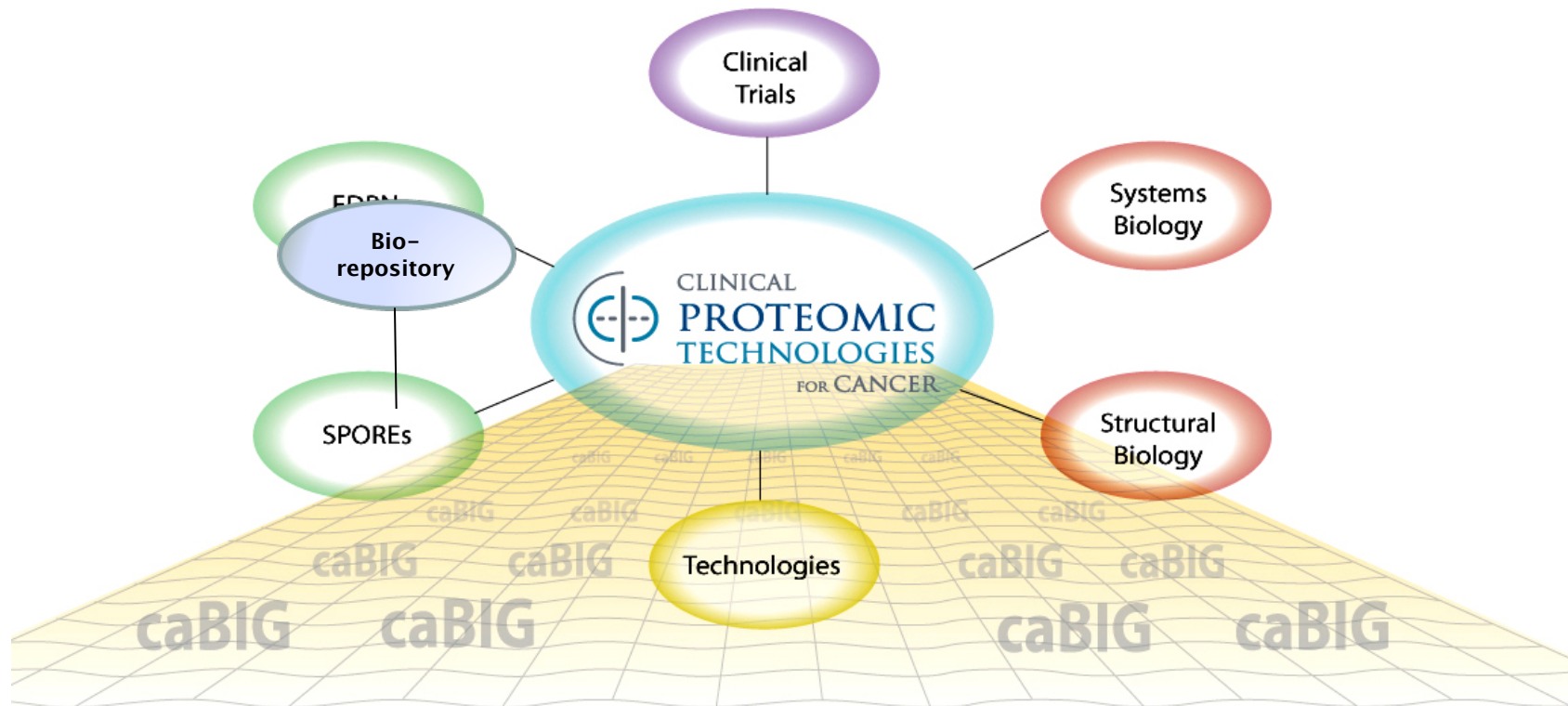
The Bindley 2010



Bindley Activities

Research projects	Cancer research and biomarkers Infectious diseases Biofuels Bionanotechnology Instrument technology
Corporate interactions	Expertise for corporate projects Facilitate start-ups Access to capabilities
Equipment & research capabilities	'Omics technologies Screening, detection Sensors, MEMS Flow, sorting, imaging Data integration
Engagement & outreach	Regional corporate development Global partnerships

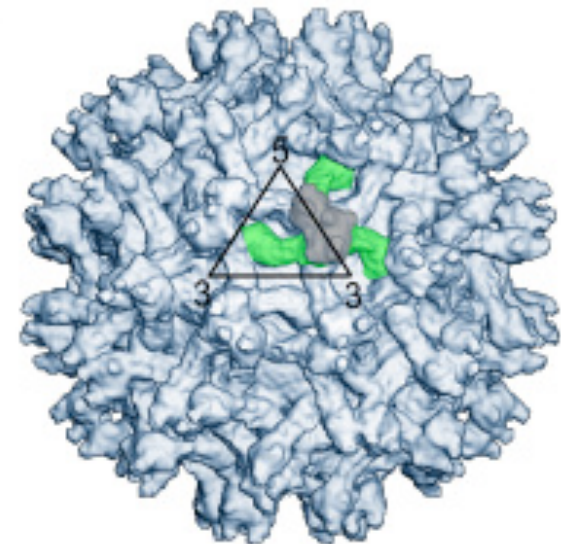
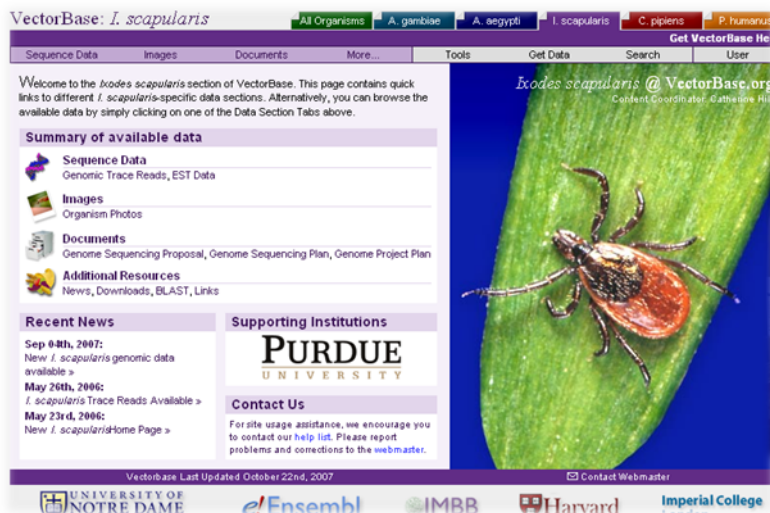
Research areas - cancer



- 'omics analyses – CPTAC (National Cancer Institute), Cancer Care Engineering, Multiple Myeloma Research foundation funding
- methods development
- biorepository
- flow/sorting
- screening & drug discovery

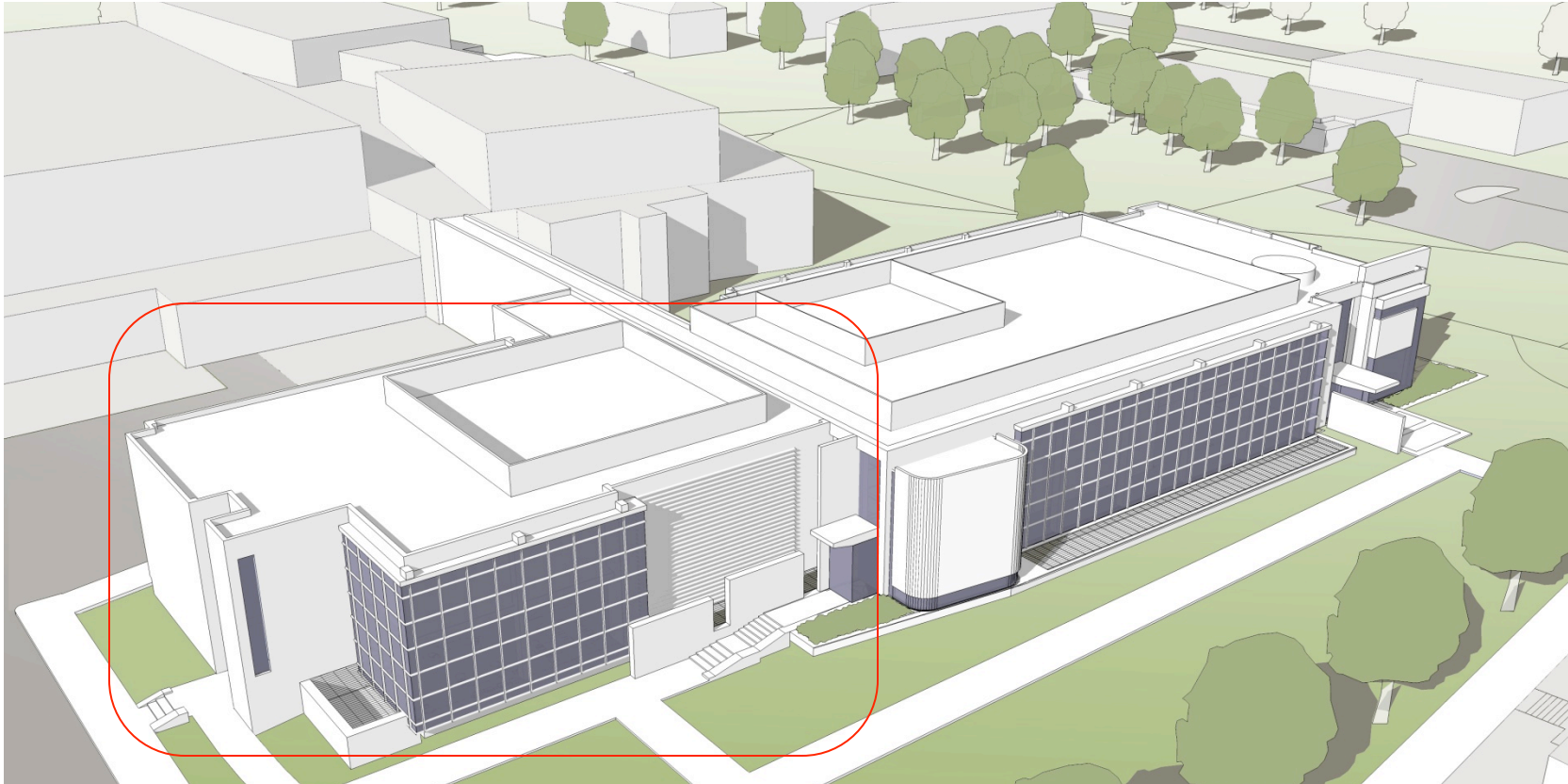
Research – infectious disease

- Center for global intervention in infectious disease, cGRIID
 - viruses, vectors, structural biology, insecticide discovery with novel targets
- Method development for molecular detection
- Vaccine development
- Instruments for research and diagnostics/therapeutics



- Biological mass spectrometry
 - small molecules, proteins: profiling and targeted analyses
- Biophysics
 - molecular interactions, SPR, Isothermal calorimetry
- Flow cytometry, cell sorting and selection
 - surface attached and suspension cell purification (BL2)
- Imaging (bioscience imaging facility)
 - new and state-of-the-art: microscopic and whole animal
- Integrated screening technologies
 - automation: high throughput screening and expression cloning
- In vivo pharmacology (swine)
 - stress-free sampling and dosing: unique capability
- Informatics and data management
 - custom approaches for handling and mining data

Coming soon ...



Bindley II addition – NIH C06 award \$14.9M,
April 2010 – occupancy expected Fall of 2013

A multidisciplinary cancer and disease research facility

Directed Irradiation Synthesis: manipulating matter in nanoscale self-organized systems

J.P. Allain^{1,2,3}

O. El-Atwani^{1,2}, S. Suslov, S. Ortoleva³, B. Heim¹, D.L. Rokusek,
E. Yang, C.N. Taylor, A. Cimaroli, A. Suslova, N. Samuelson,
E.K. Walker¹, D. Zigon¹

¹School of Nuclear Engineering

²School of Materials Science and Engineering

School of Biomedical Engineering

³Birck Nanotechnology Center

Seminario Nanociencia 2010

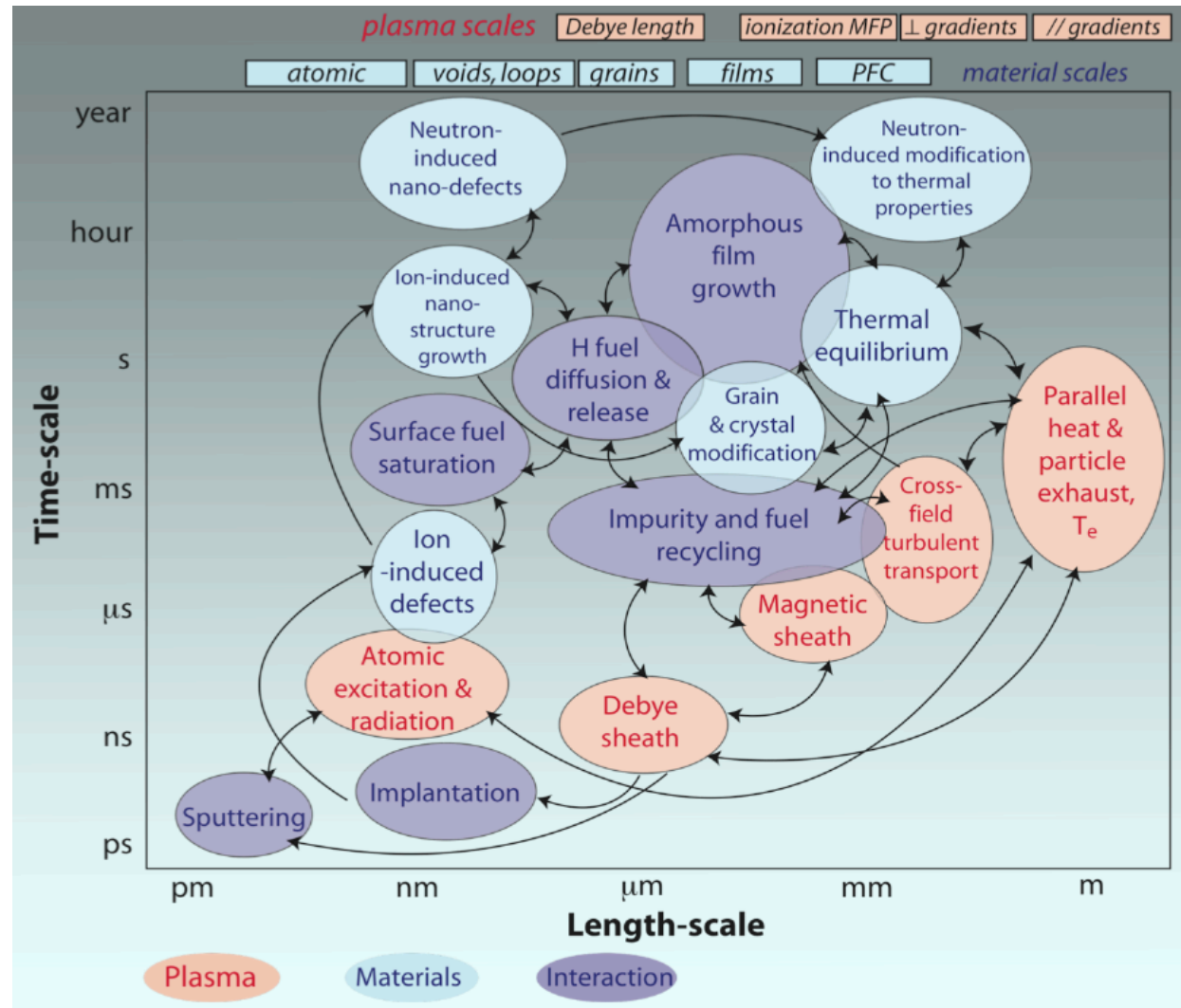
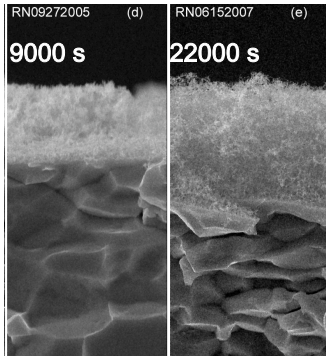
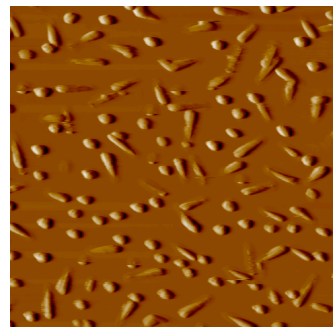
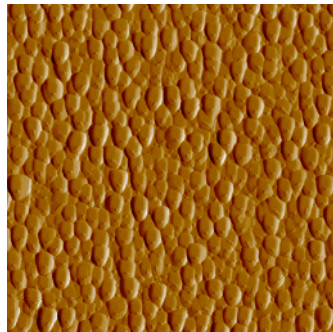
Barranquilla, Colombia

21-23 October, 2010

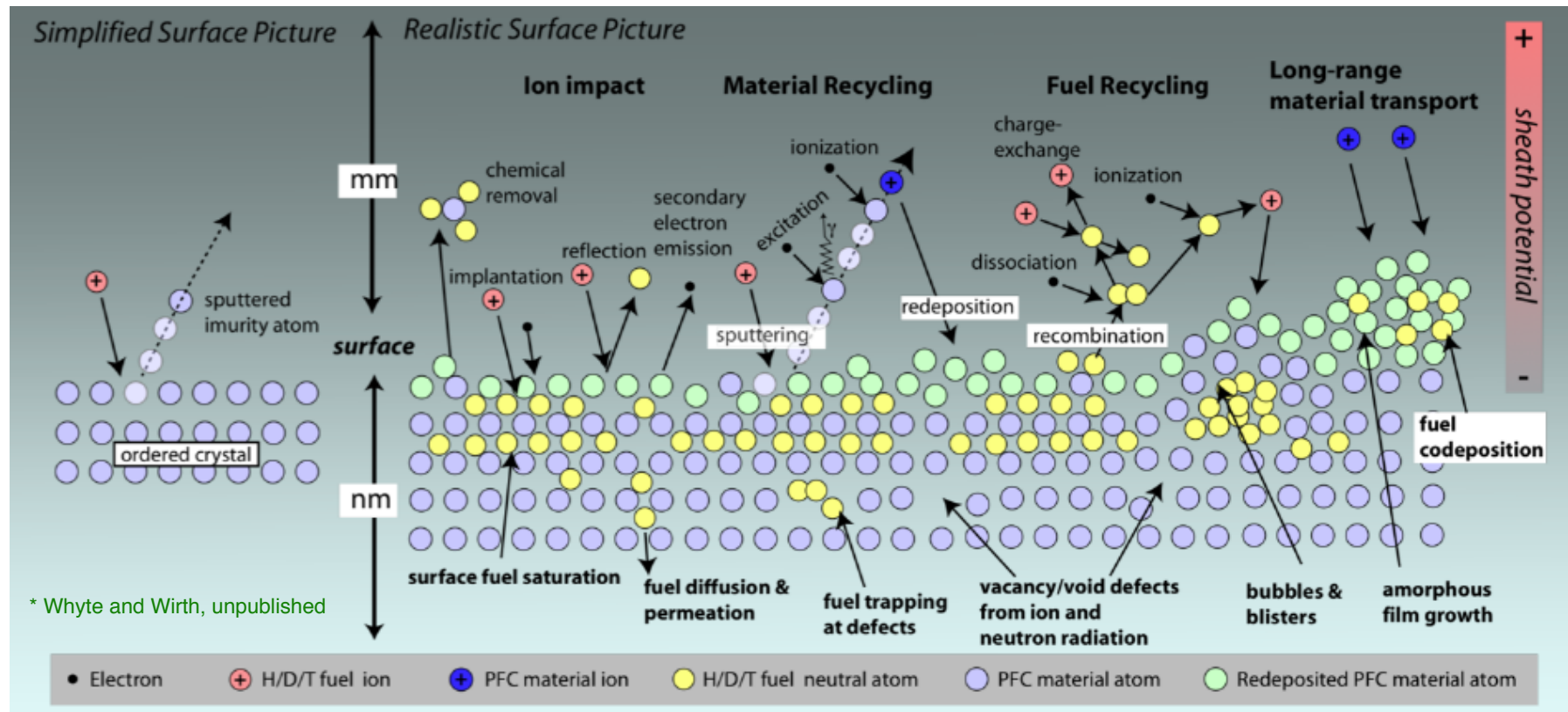
Outline

- Motivation for *in-situ* irradiated surface characterization
 - *self-organized vs disordered systems*
- *In-situ* observation and manipulation tool set
- Case studies of irradiation-driven systems:
 - *nanolithography with 13.5-nm EUV radiation*
 - *lithium-doped graphitic surfaces in fusion devices*
 - *compound semiconductor nano-patterning*
- Summary

Irradiation-driven vs thermal-activated systems: instabilities and self-organization at the plasma-surface interface

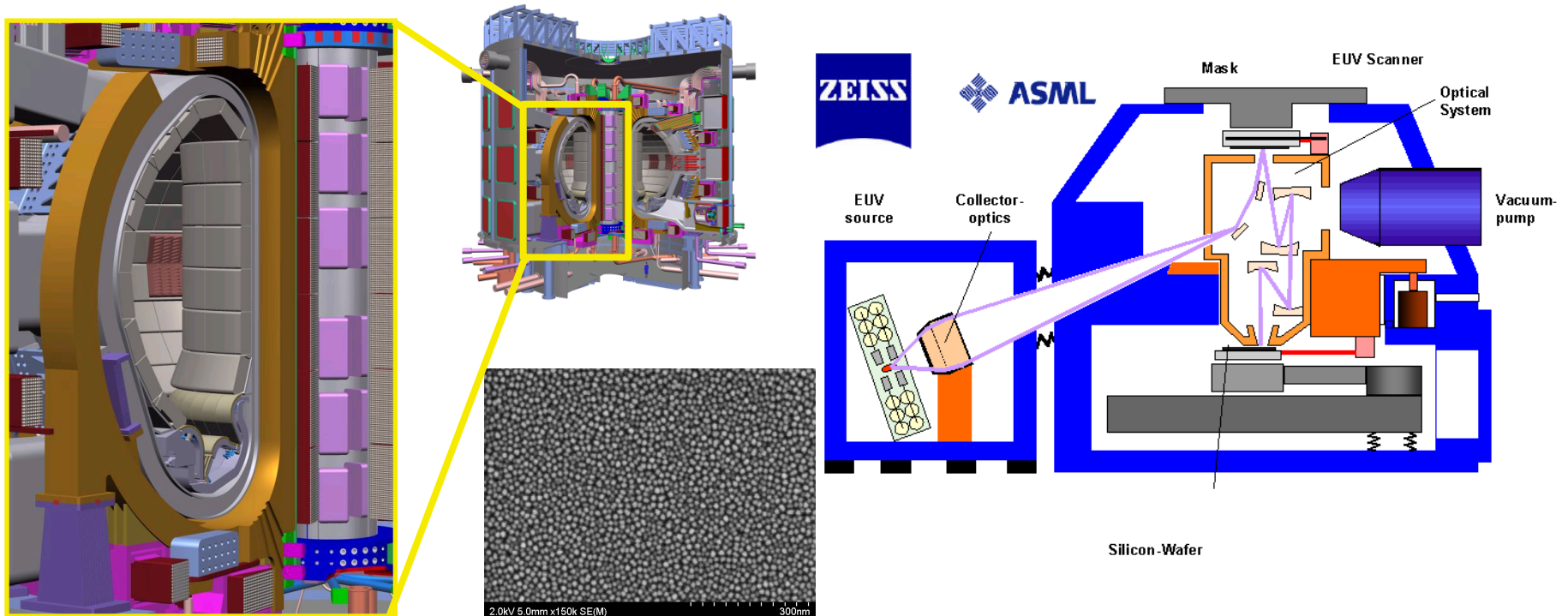


Moving from a simplified view of the plasma-surface interface to a more rich and complex



The complexity of the extreme PSI environment requires a more complex set of characterization tools that must probe *dynamically* ultra-shallow regions

Irradiation environments at near-sputter threshold energies

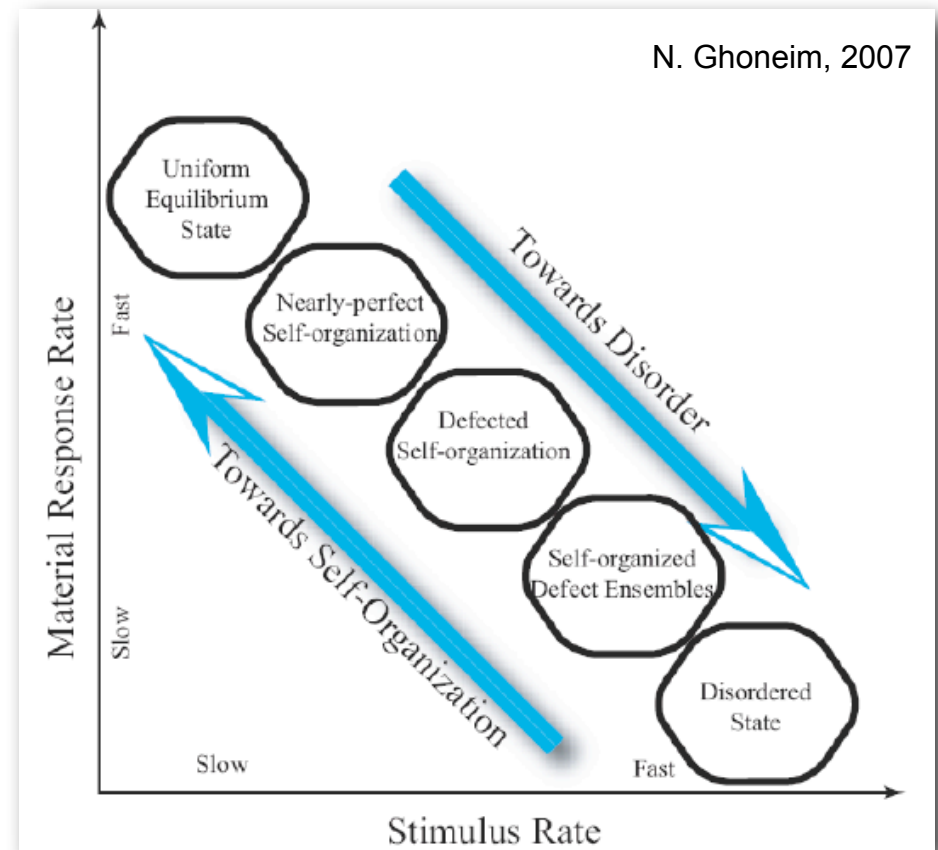


- In the plasma edge of a nuclear fusion reactor, the plasma boundary with the wall surface and sub-structure controls the inventory of hydrogen fuel
- Collector mirror surface of 13.5-nm light from high-density plasma sources
- Irradiation-driven nano-structures are primarily dictated by the sputter depth and surface relaxation mechanisms

Driving matter between disorder and self-organization

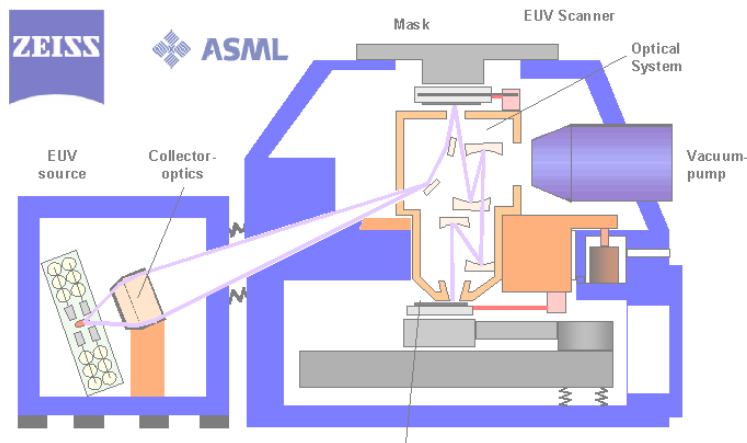
simulating extreme environments:
ion-induced damage
(e.g. fusion PMI*)

exploiting irradiation-
matter interactions
to *synthesize* new
structures

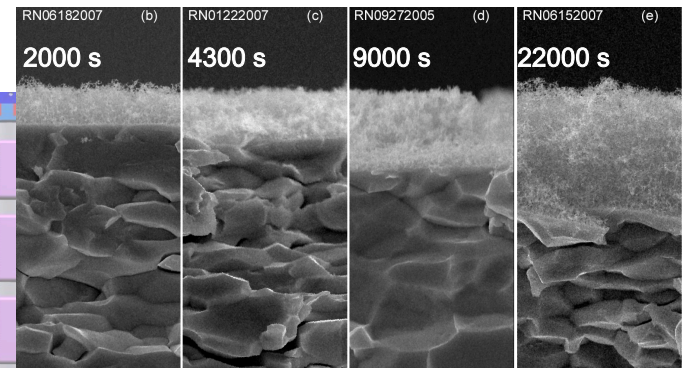
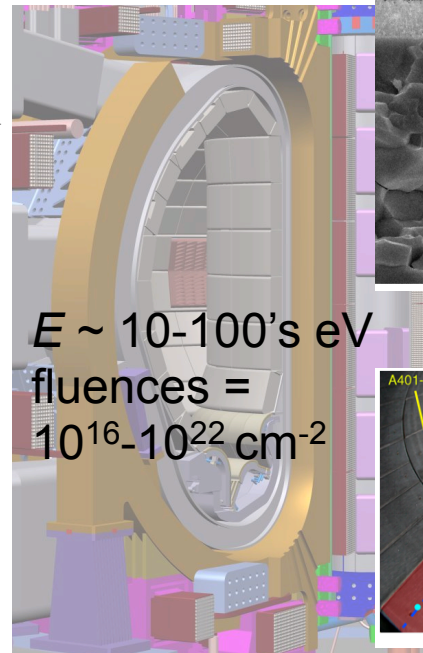


- Irradiation of matter (stimulus) leads to modification of matter inducing self-organized structures or disordered state (i.e. deterioration of properties)
- Understand how to manipulate matter (*directed irradiation synthesis*) to modify structure at nanoscale for new properties

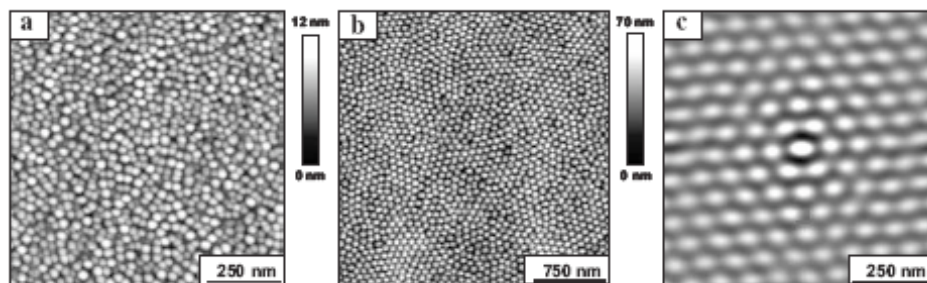
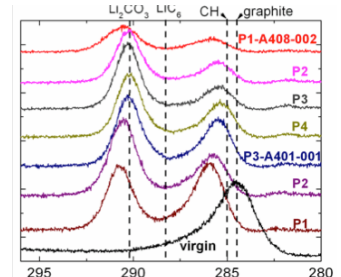
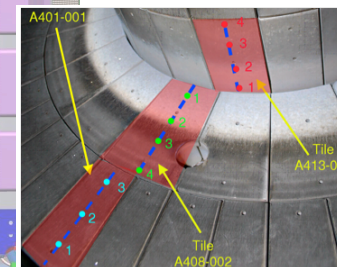
Spatial and energy scales of irradiation-driven systems under near-threshold sputter regime



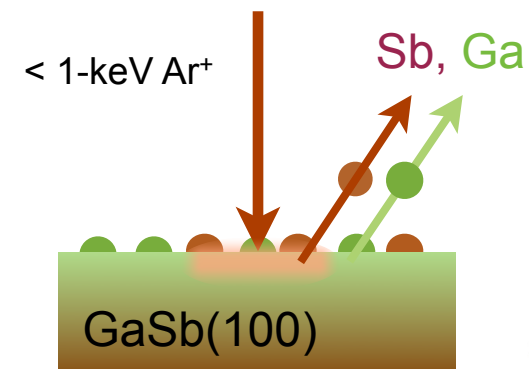
$E \sim 100\text{-}1000\text{'s eV}$
 fluences =
 $10^{16}\text{-}10^{19}\text{ cm}^{-2}$



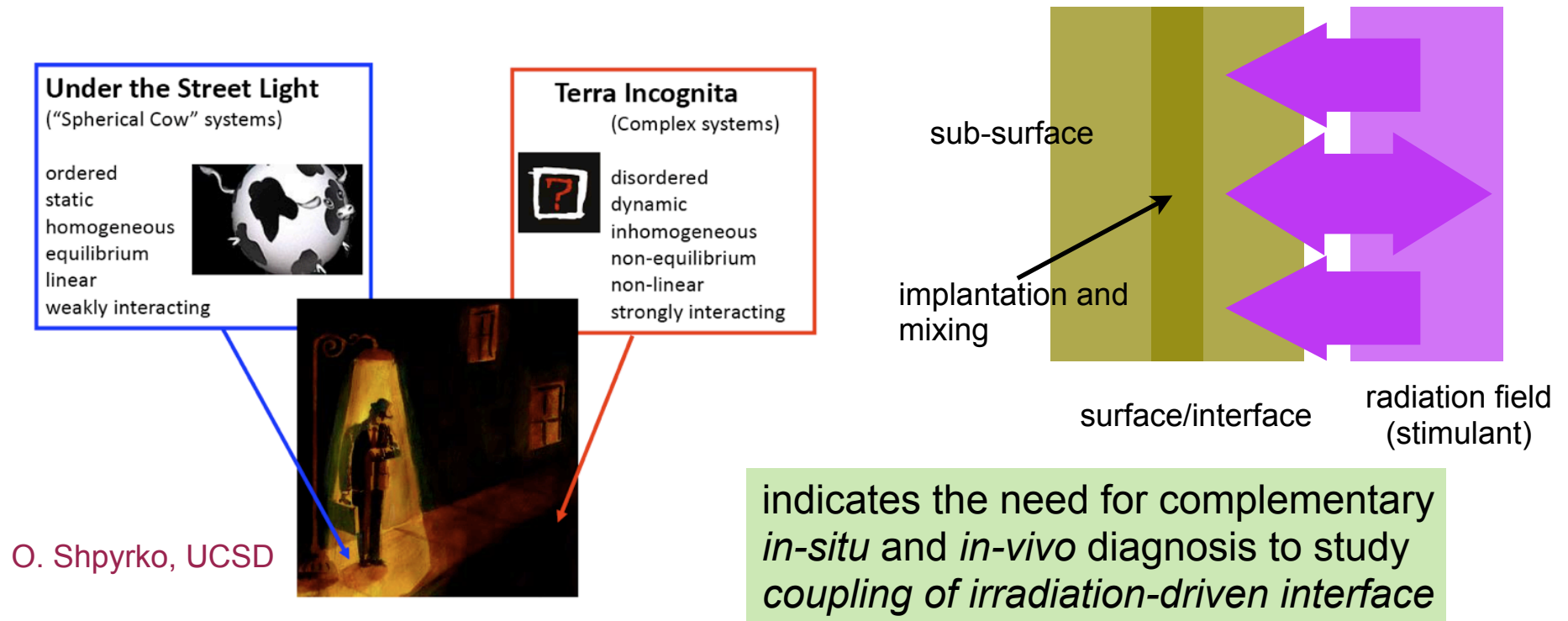
Baldwin and Doerner, 2008



$E \sim 10\text{-}100\text{'s eV}$, fluences = $10^{14}\text{-}10^{18}\text{ cm}^{-2}$

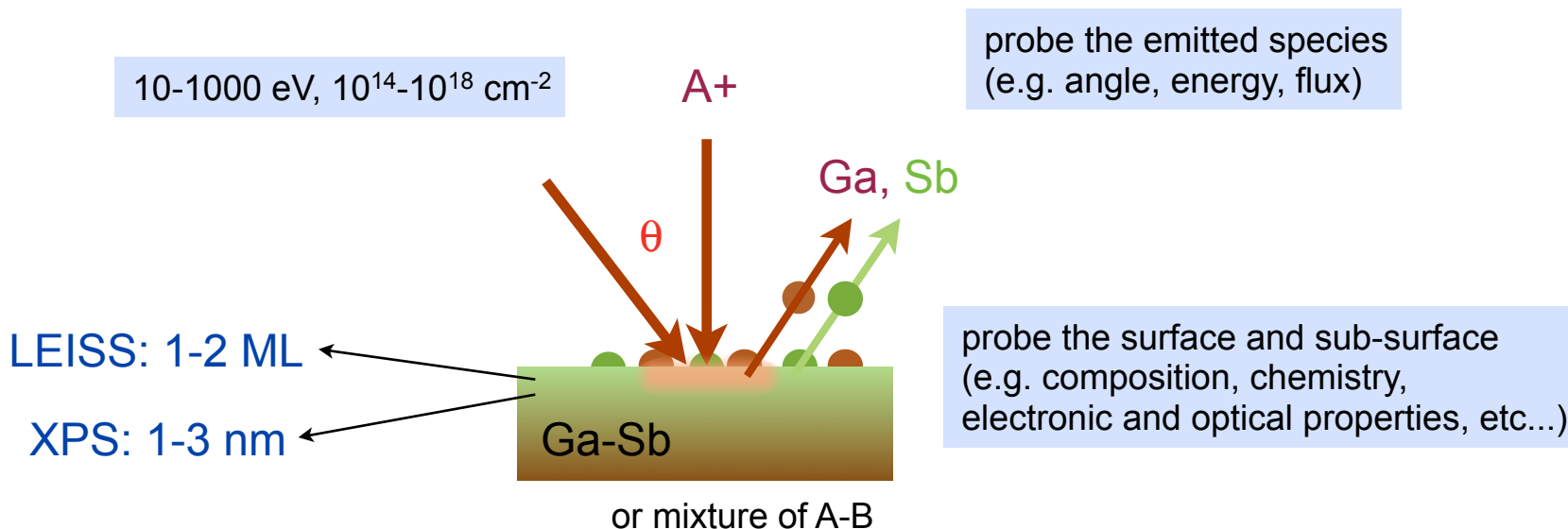


Critical knowledge gap between “real” or “complex” systems and “isolated” systems in condensed matter

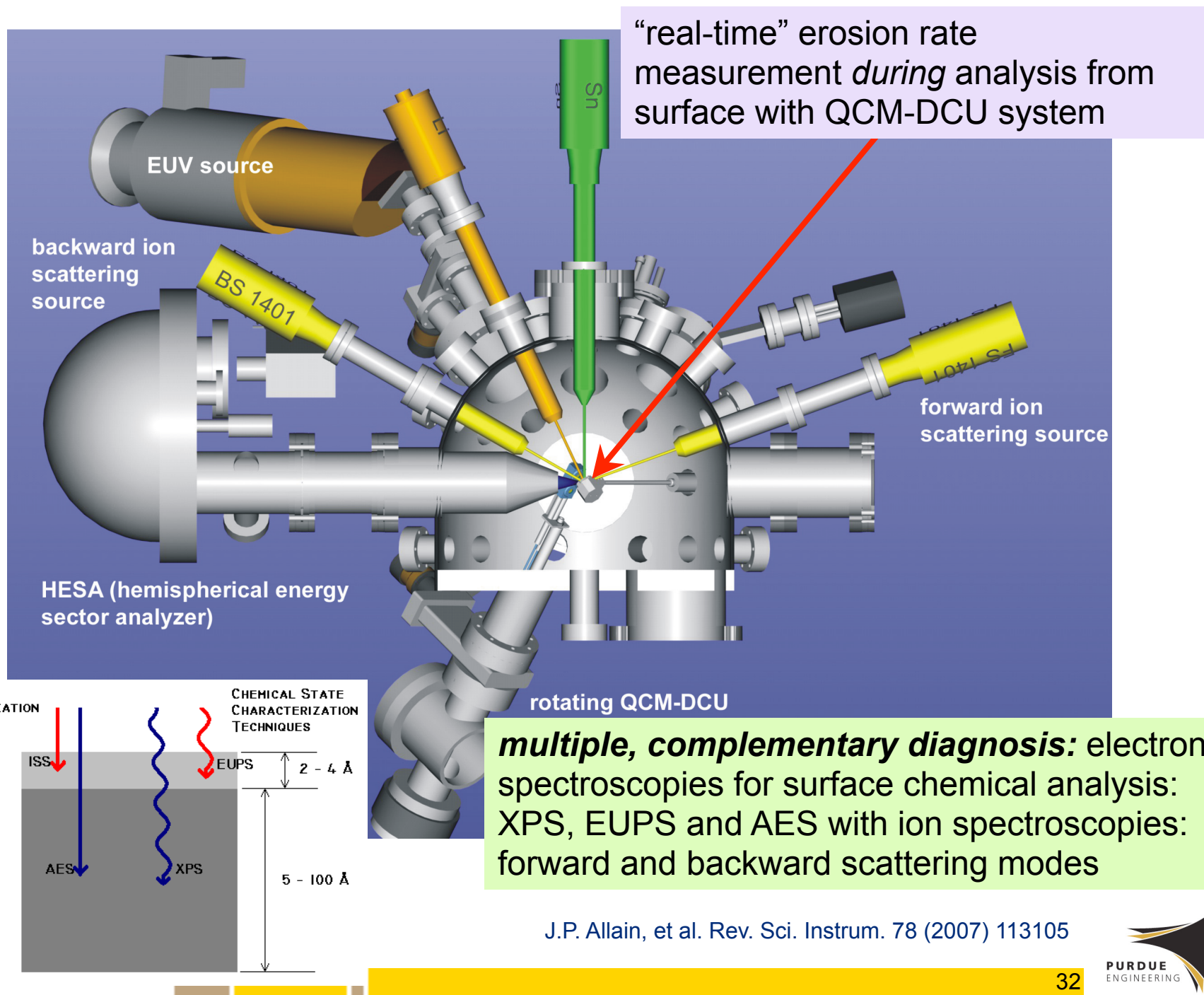


- Understanding of complex matter in low-dimensional state systems limited by “probing” and “manipulating” approaches
- Need for dynamic measurement of surfaces under controlled irradiation fields
- Indicates the need for complementary *in-situ* and *in-vivo* diagnosis to study coupling of irradiation-driven interface

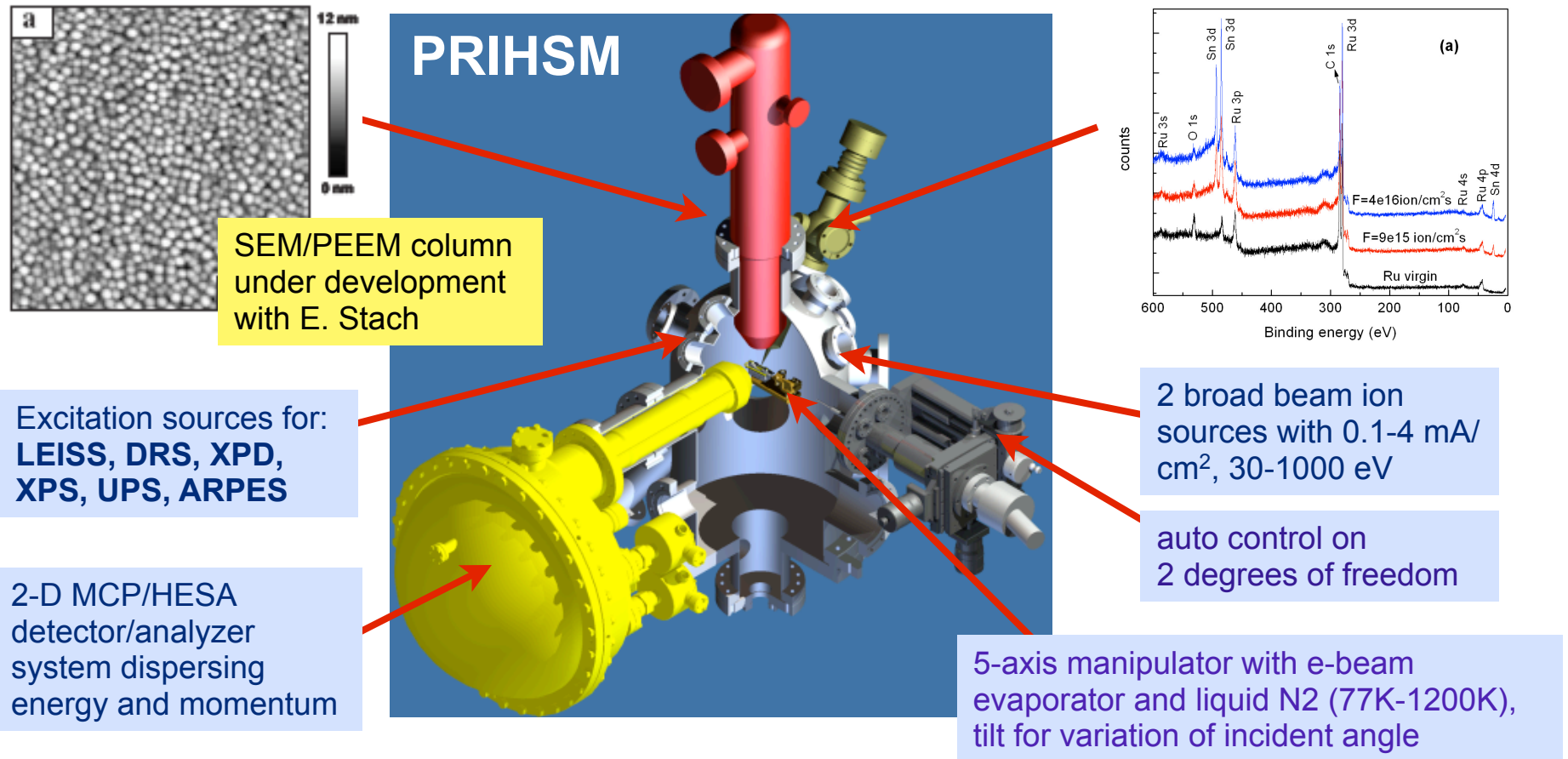
The Tool Set: *In-situ* characterization of an irradiation-driven surface



- amorphous layer: low-energy ion scattering spectroscopy (**LEISS**) in backscattering mode with 1.5-keV He ions
- sub-surface layer (1-5 nm) with **XPS**
- We combine the two techniques *in-situ* during irradiation with various modification sources

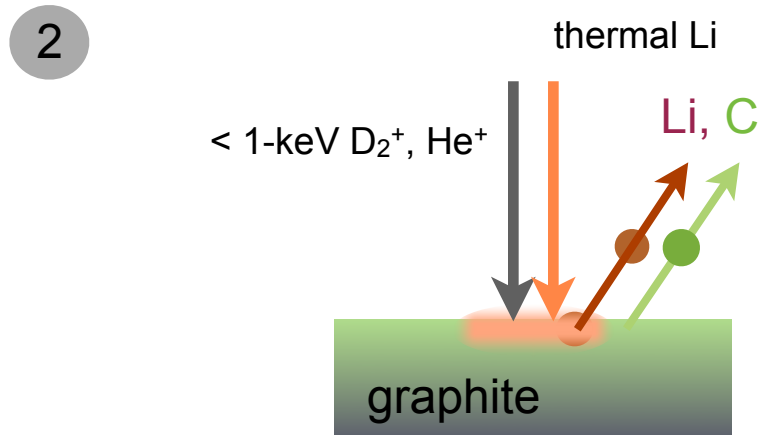
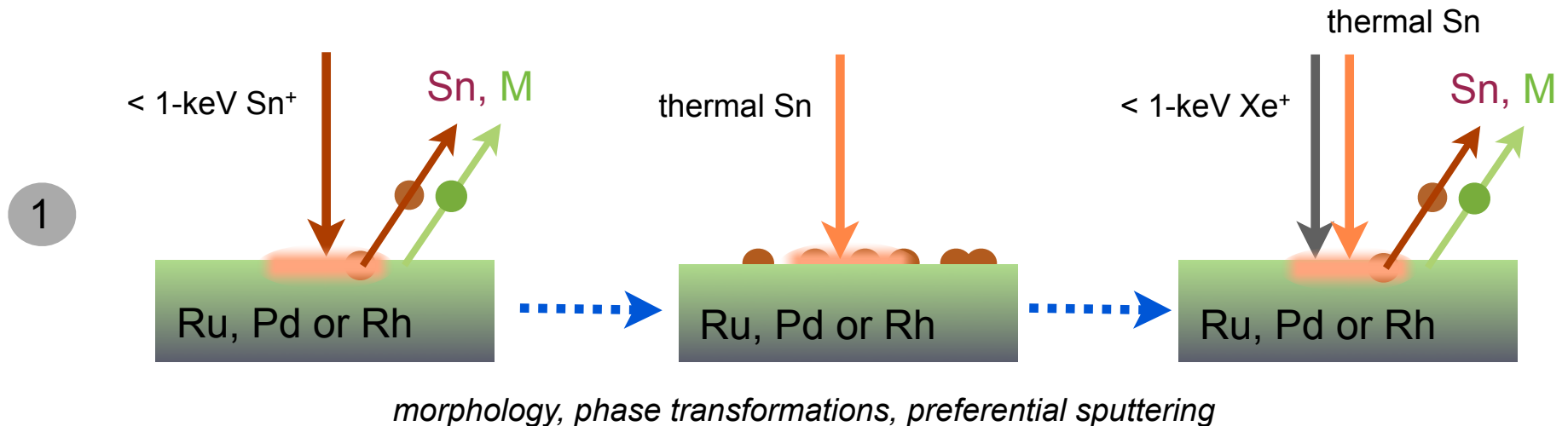


Modification ion sources with in-situ tool set

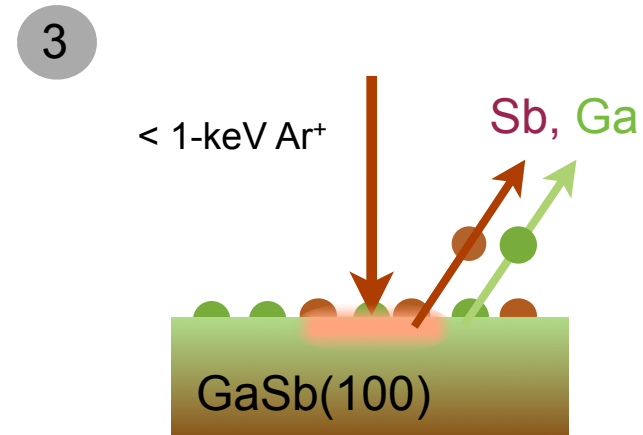


- **Conditions:** GaSb(100) samples irradiated by Ar⁺ at 50, 100 and 200 eV, normal incidence with 40-50 μAcm^{-2} for fluences up to 10^{18} cm^{-2}
 - *We operate in the sputter threshold regime and study early stage growth*
- PURDUE** – e.g. $\sim 10^{15}\text{-}10^{17} \text{ cm}^{-2}$

Case Studies: in-situ surface characterization of irradiation-driven surfaces

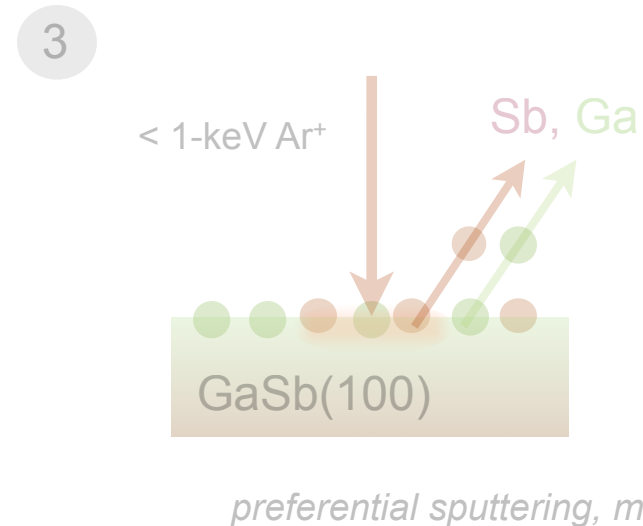
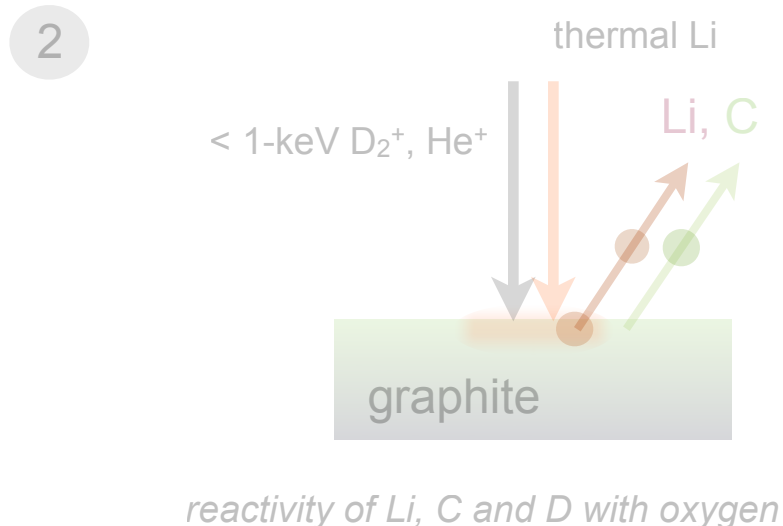
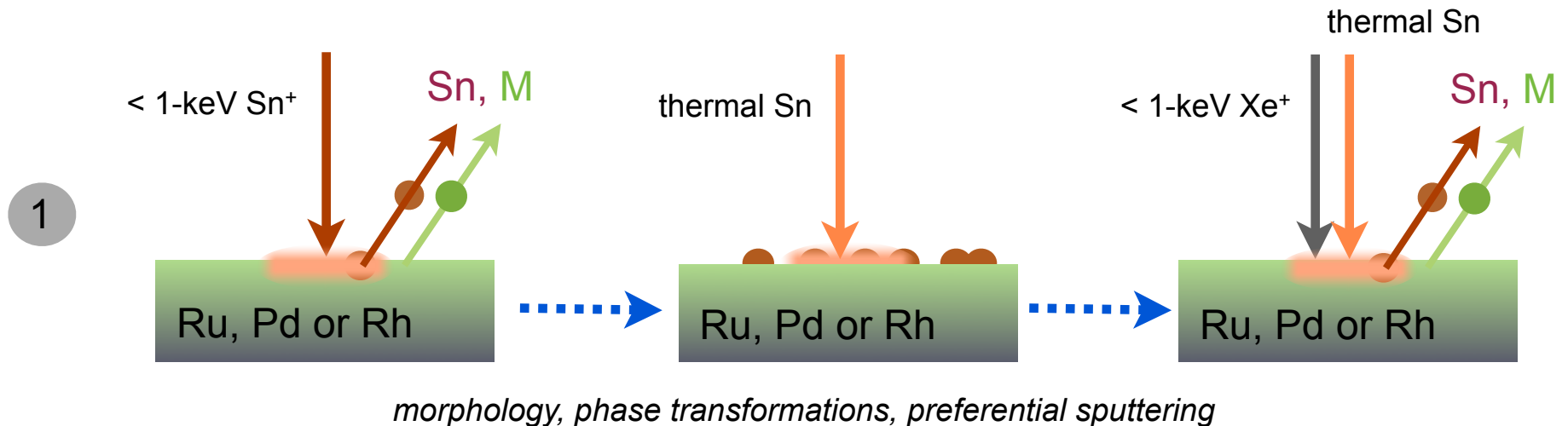


reactivity of Li, C and D with oxygen

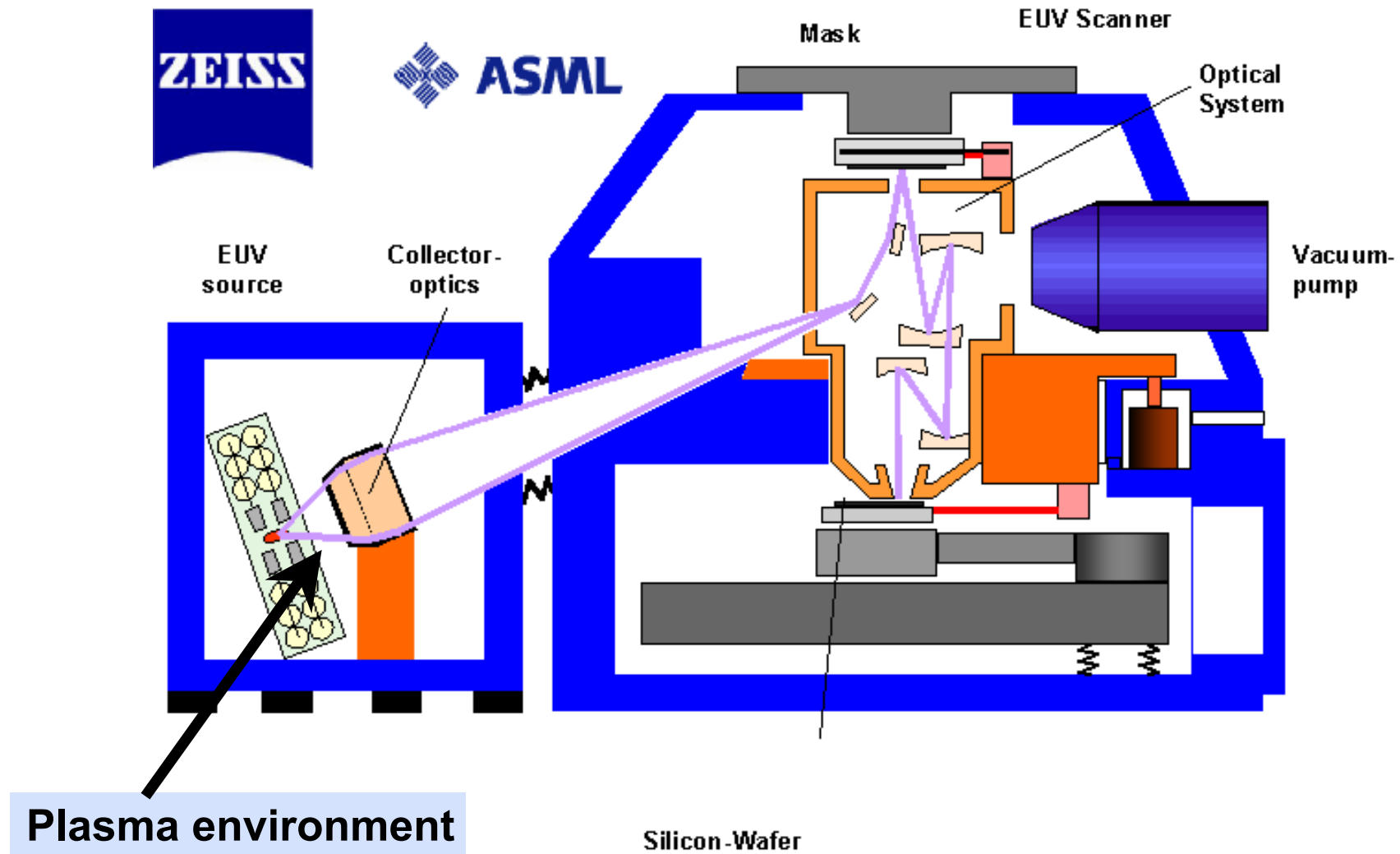


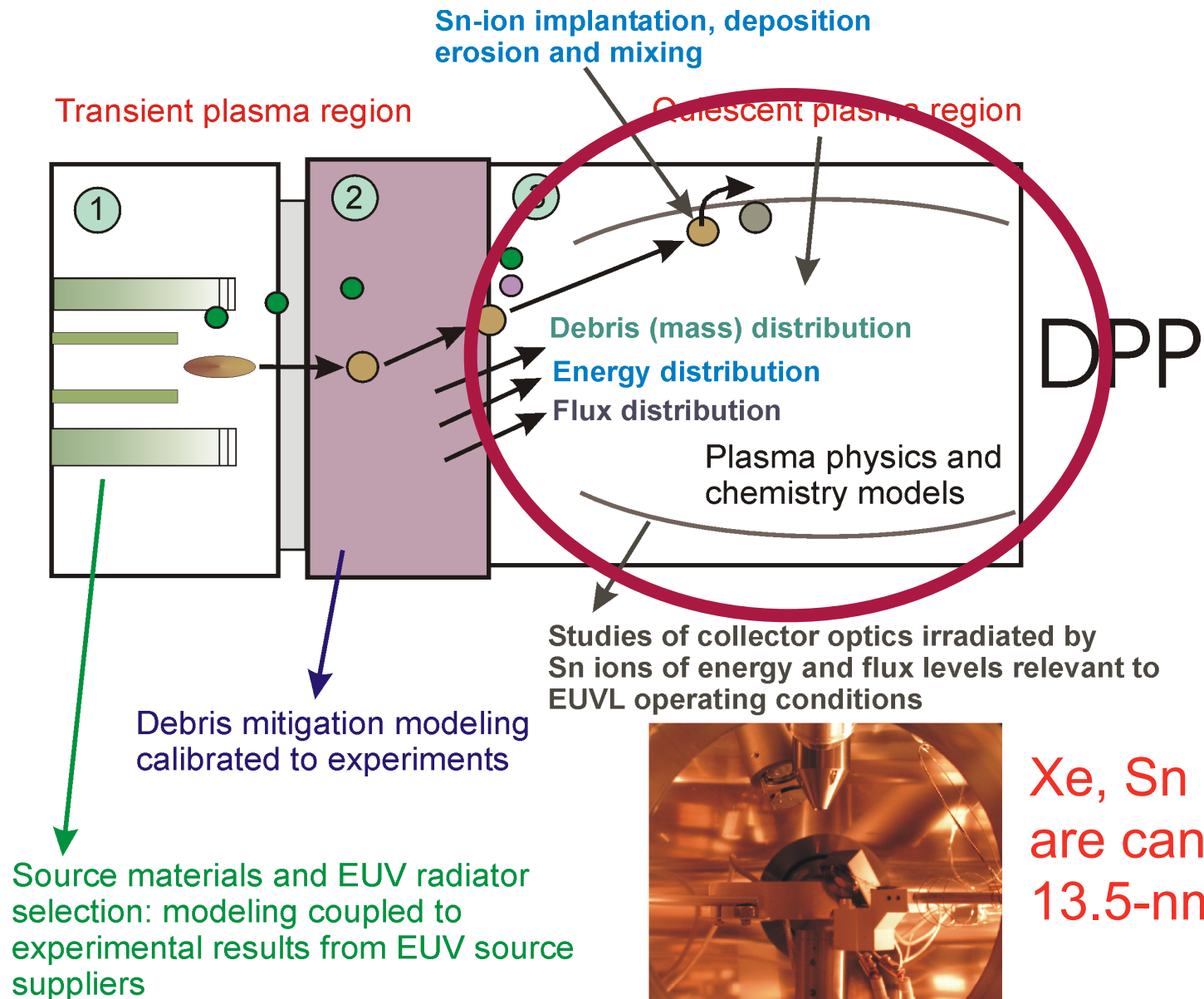
preferential sputtering, morphology

Case Studies: in-situ surface characterization of irradiation-driven surfaces

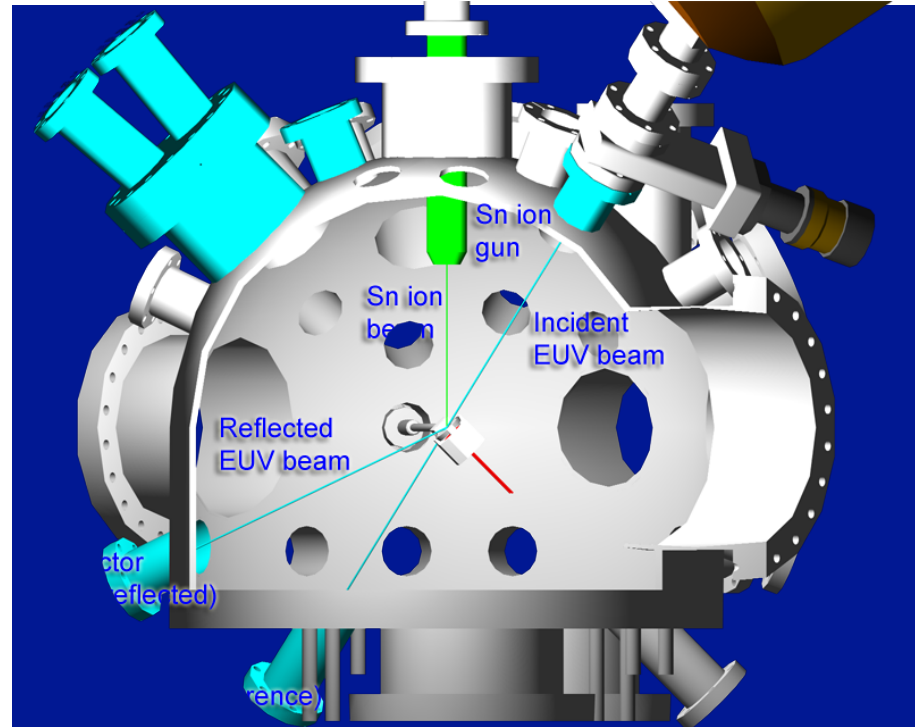
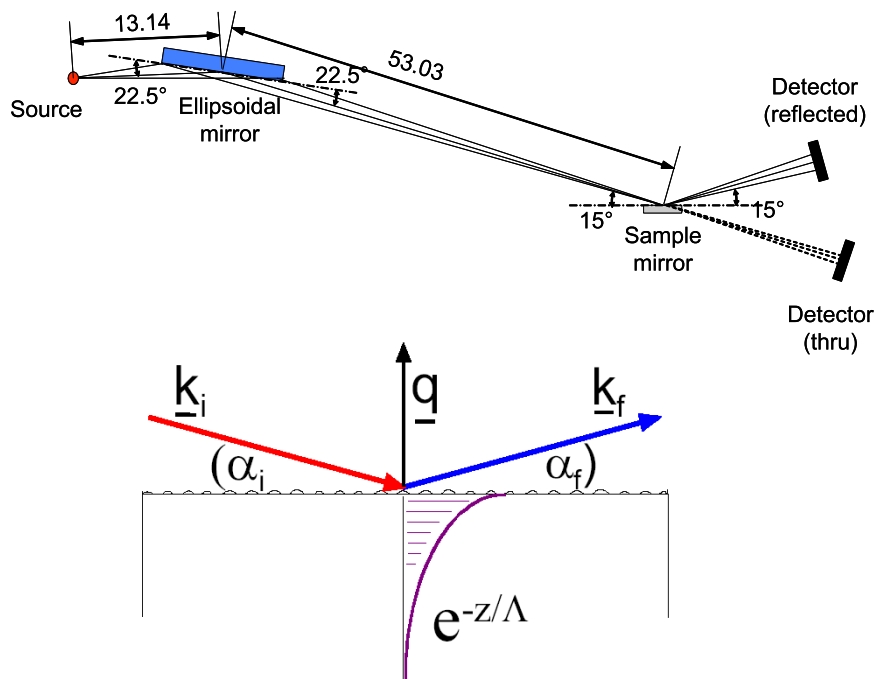


EUVL-Scanner: schematic



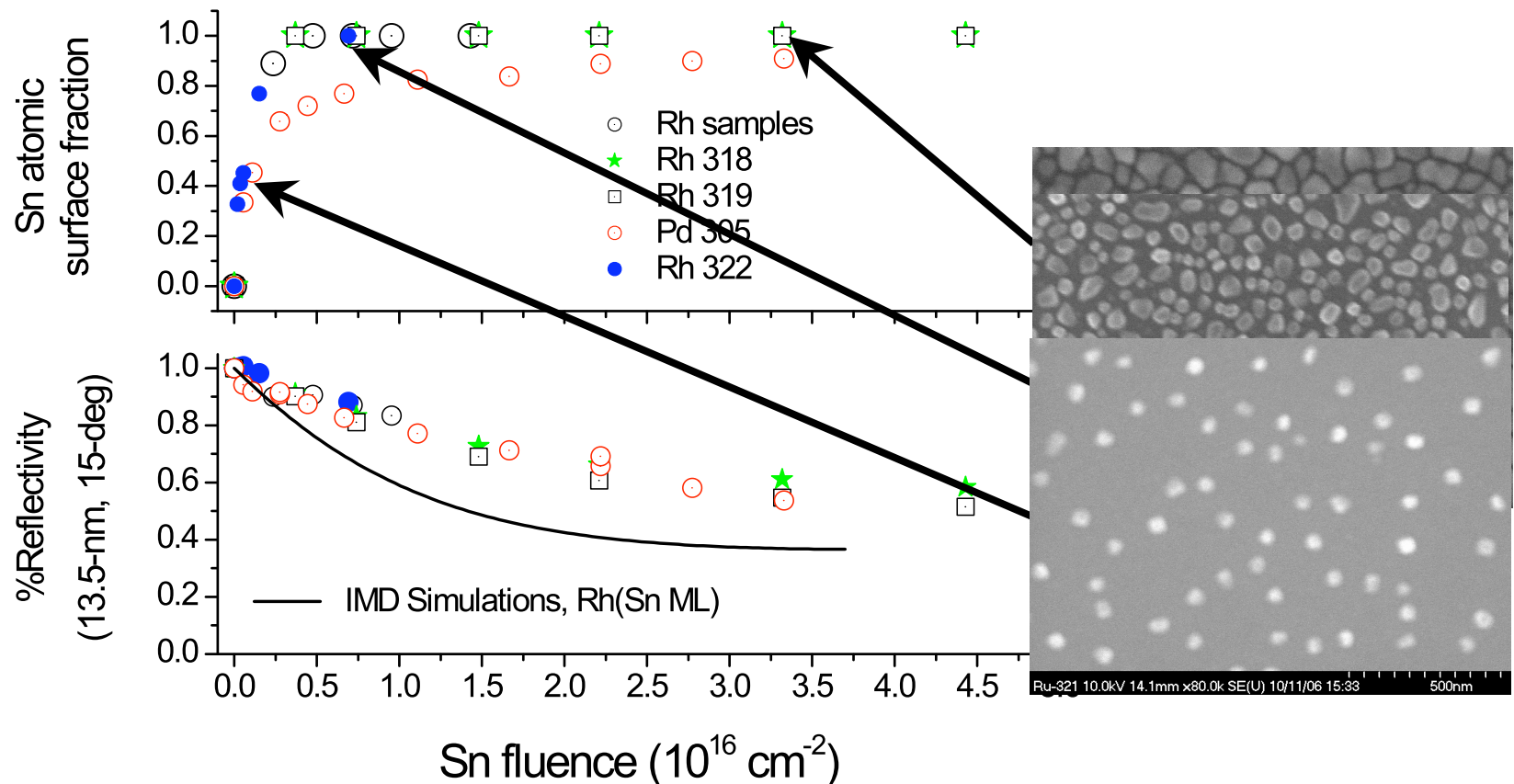


EUV reflectometry for grazing incidence mirror studies



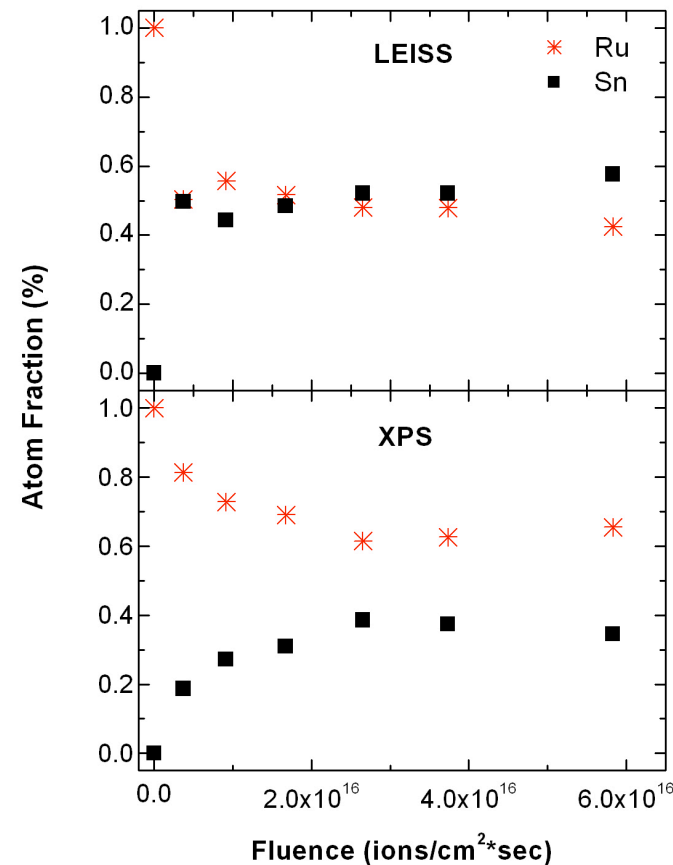
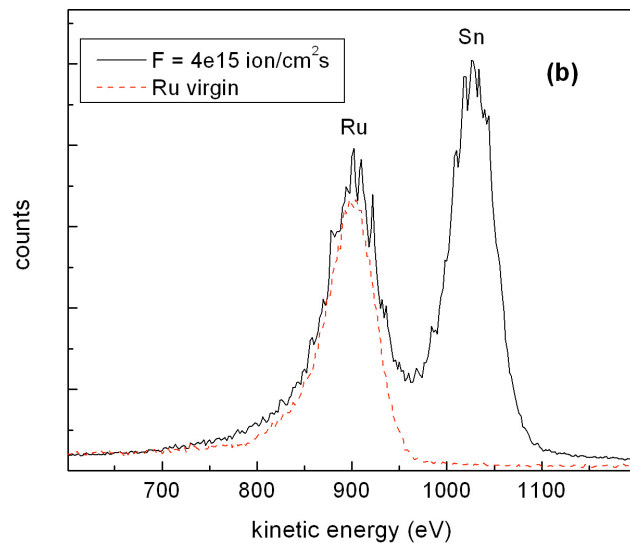
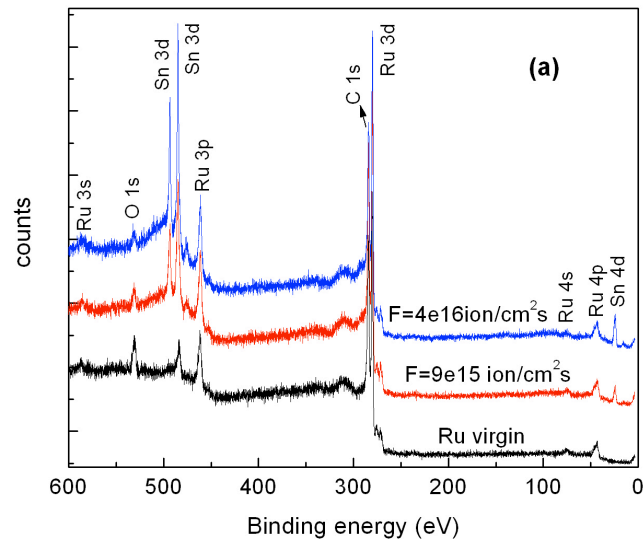
- The In situ EUV reflectometry system in IMPACT diagnoses the reflectivity response of candidate mirrors **during** irradiation of Sn ions or thermal atoms **and** measurement of the chemical and elemental state of the mirror surface

*In-situ surface elemental evolution and EUV reflectivity during exposure to **thermal** Sn*



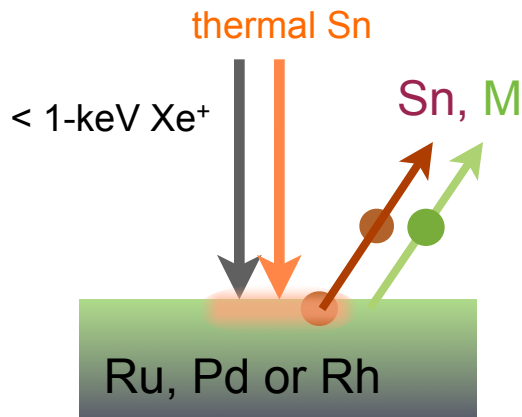
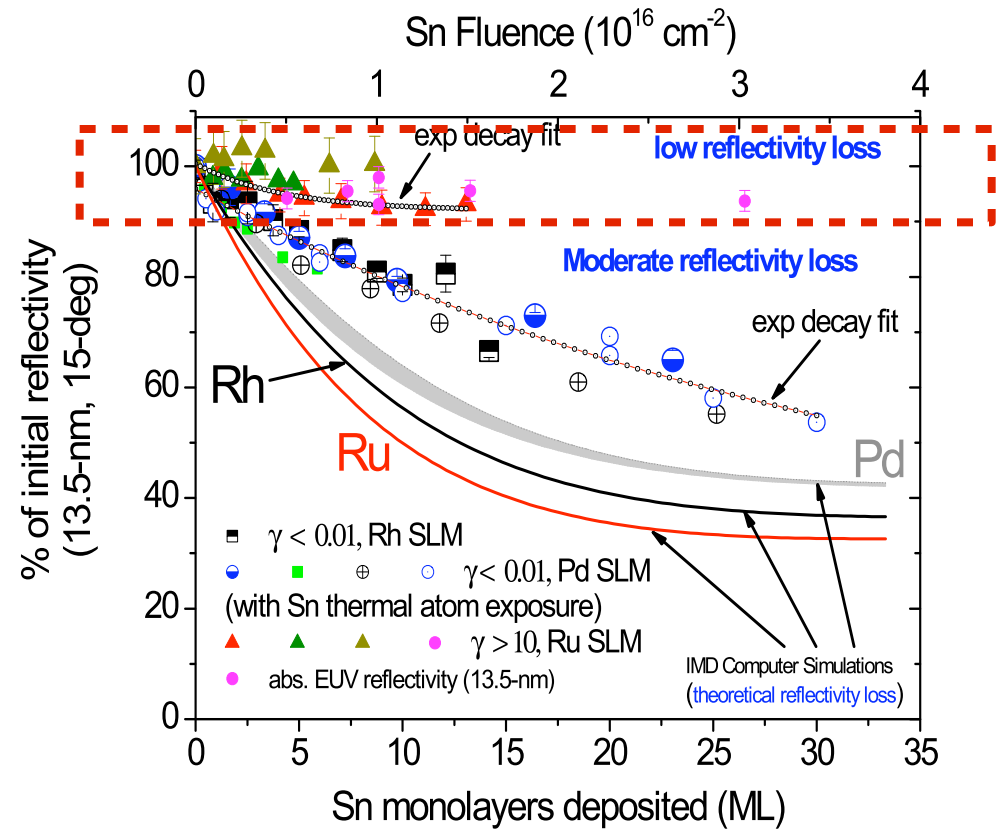
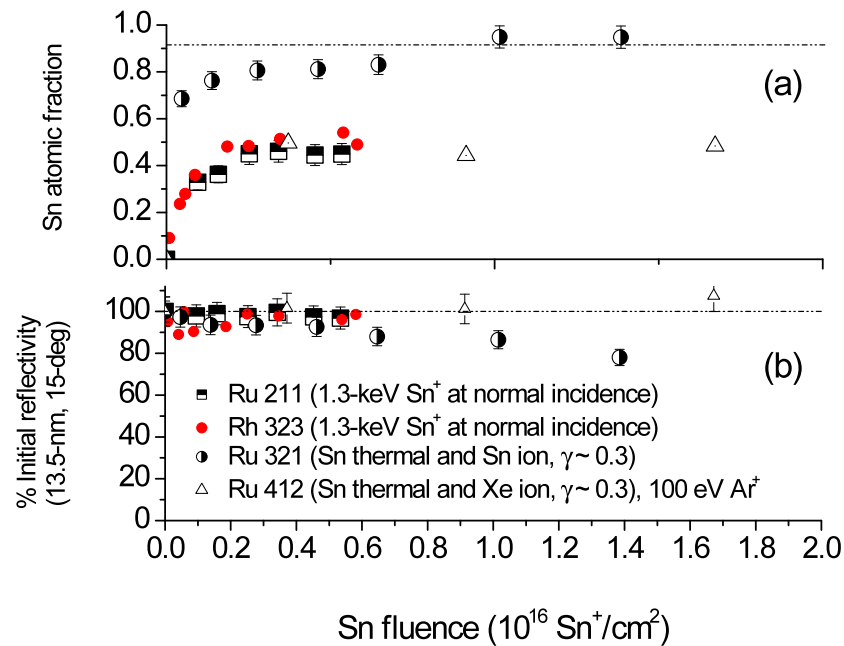
- Relative reflectivity at EUV decay is slower than theoretically predicted
- In-situ measurements indicate that this is due to partial coverage of Sn deposition on the mirror surface due to the coalescence of Sn islands during exposure

Probing spatial scales with complementary surface-sensitive techniques



- 1.3-keV Sn^+ on Ru mirror surfaces implanted at normal incidence
- Note the distinct probing regions of XPS vs LEISS

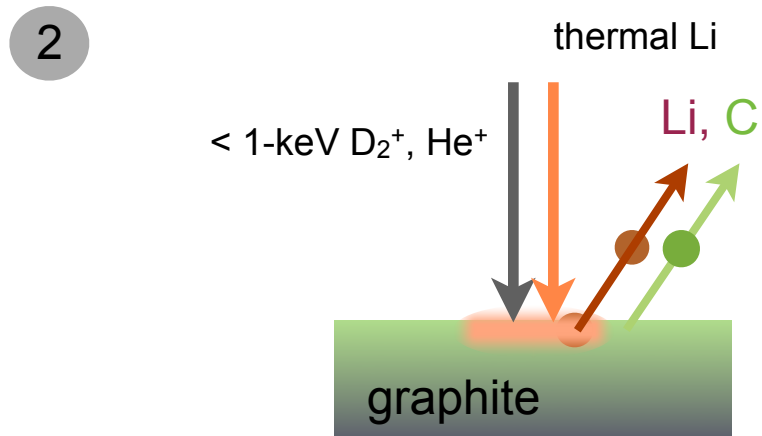
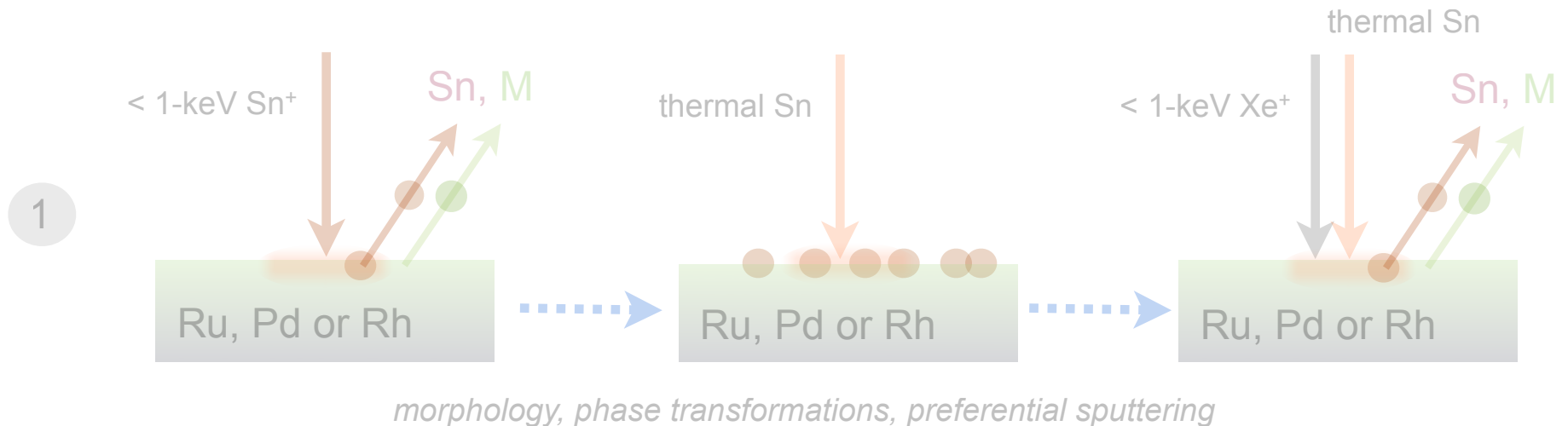
Ion and atom balance induce complex nanostructures



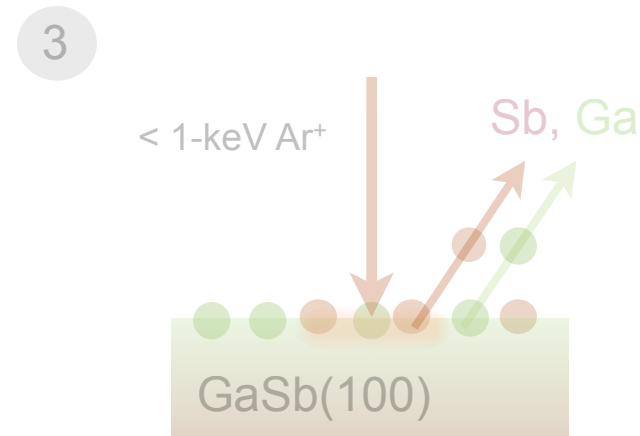
- Balance of ion implantation with erosion against the thermal deposition of Sn atoms can aid in "tuning" Sn concentration on surface and EUV reflectivity loss

Absolute 13.5-nm reflectivity ex-situ

Case Studies: in-situ surface characterization of irradiation-driven surfaces

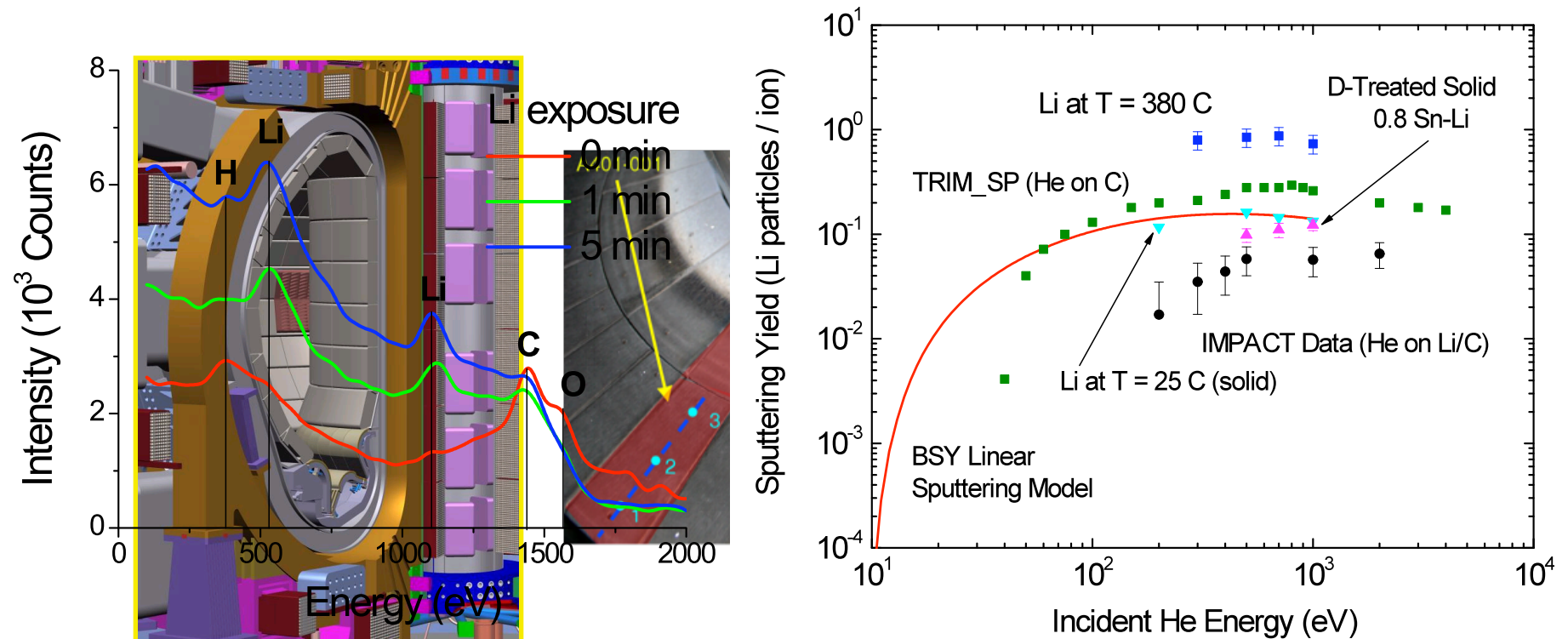


reactivity of Li, C and D with oxygen



preferential sputtering, morphology

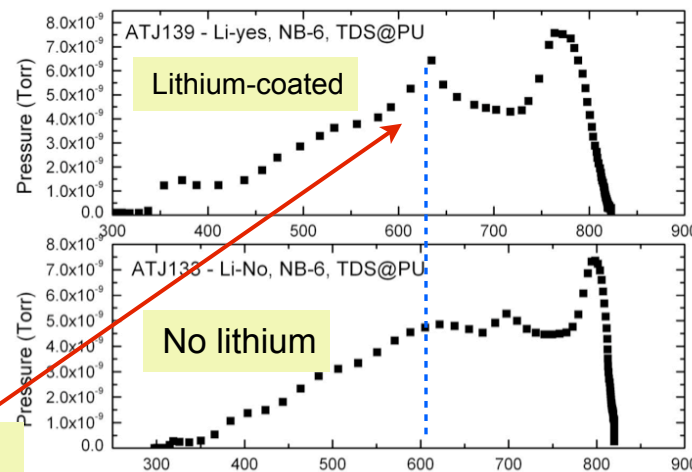
Lithium coatings on graphite: surface effects on erosion, particle retention



- Lithium intercalates to the basal planes of graphite. Difficult to maintain 100% lithium layers on top few ML. Oxygen typically bound with lithium
- Substantial reduction of both *physical* and *chemical* sputtering by *D* or *He* bombardment when comparing lithiated graphite surfaces to either pure Li or C

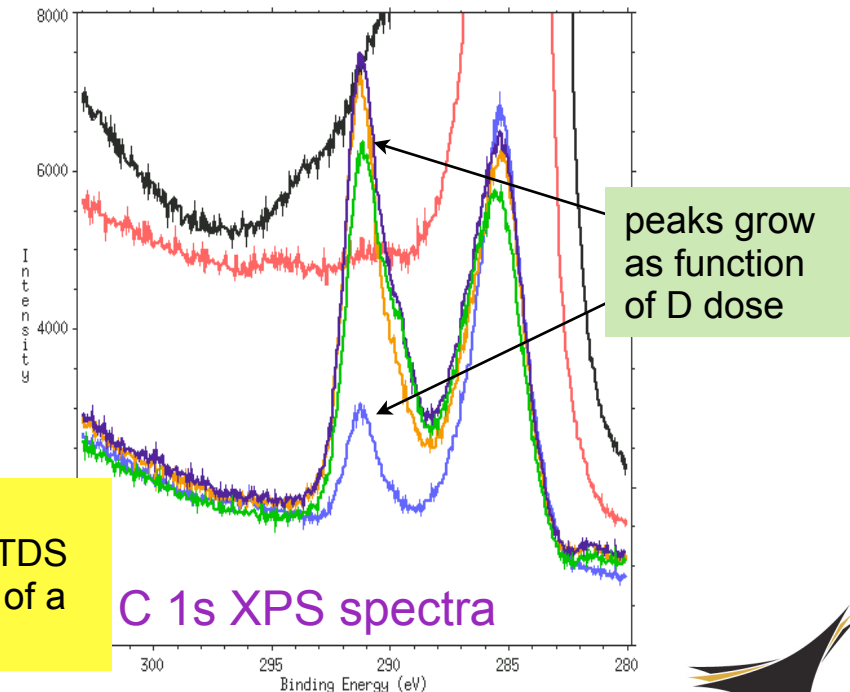
In fusion reactors, manipulation of hydrogen isotopes in a region of a few 10's of nm to order of μm 's

- Critical to the recycling of D when using lithium as a PFS (plasma-facing surface) is the top layer of atoms
- Sputtered particles emanate from the first 2-3 ML of a metal surface (although the damage zone can be 10-100's nm below)
- Recombination of implanted D occurs in the first few layers at the surface-vacuum interface dictating *recycling to the plasma edge*



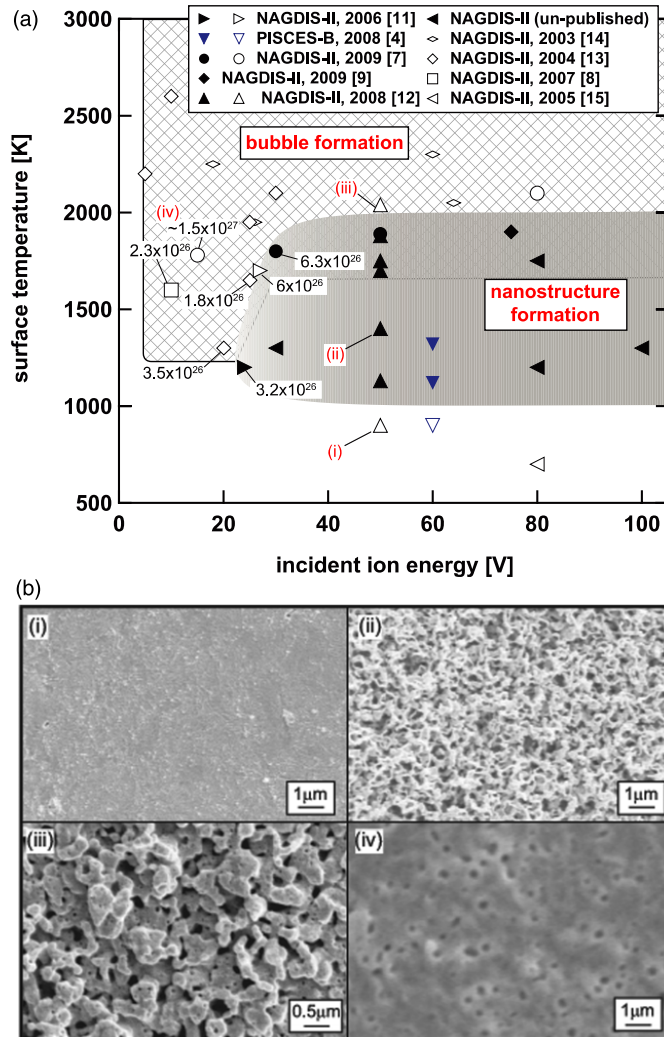
“weakly-bonded”
deuterium peak

Critical Li-C-D and Li-O-D chemical state elucidated by in-situ XPS **together with** TDS analysis provided corroborated evidence of a weakly-bonded state of D in Li-C-O



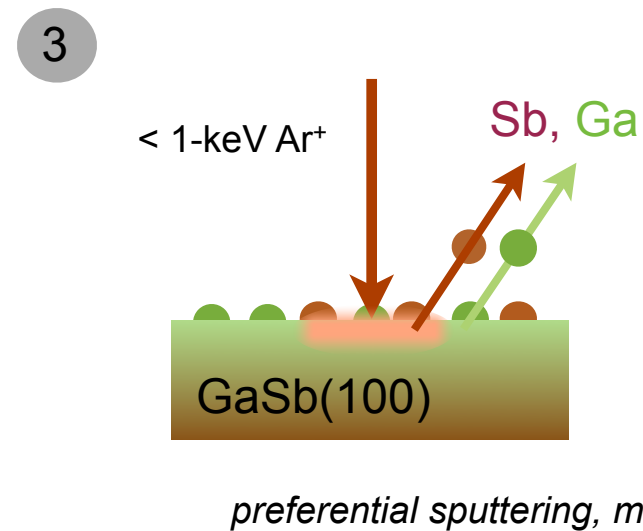
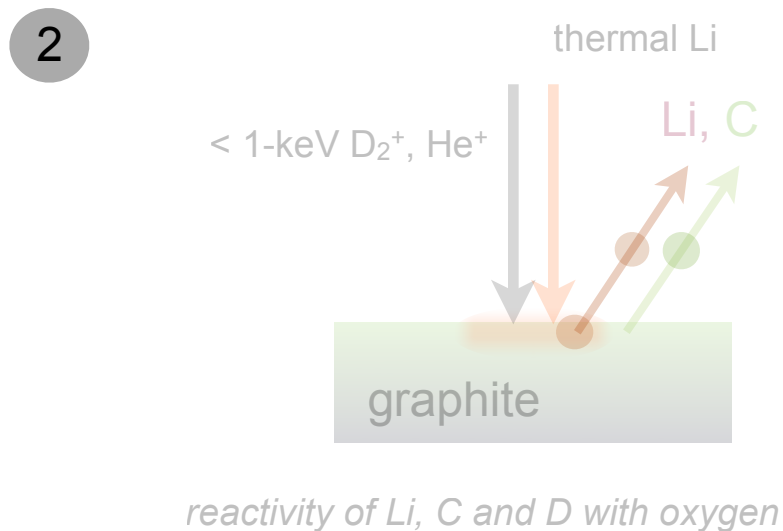
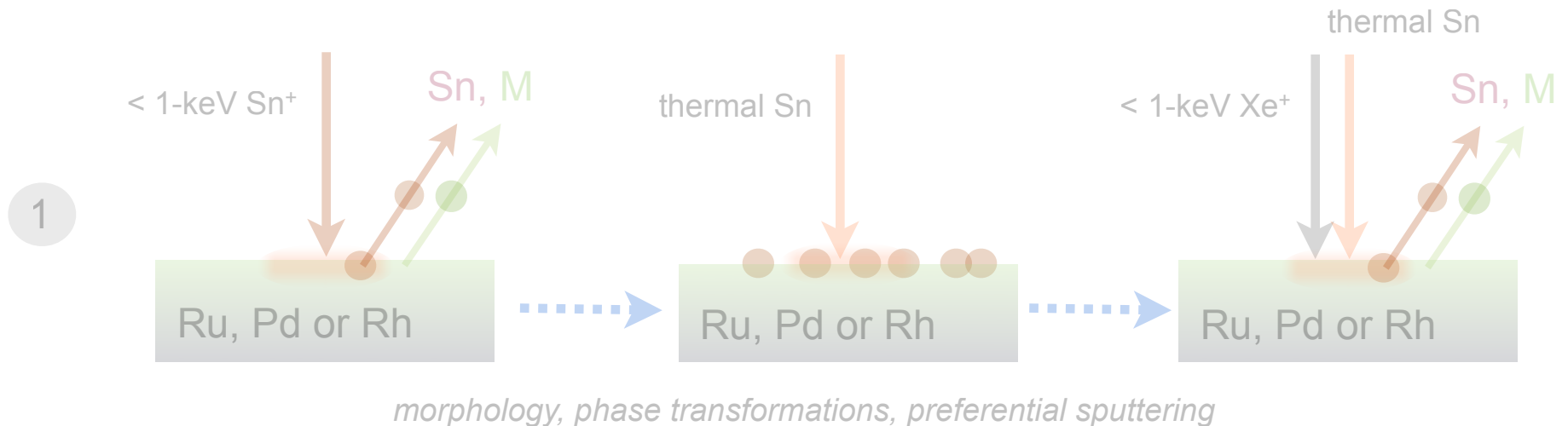
C 1s XPS spectra

Self-organization under nuclear fusion reactor conditions for He irradiation of tungsten

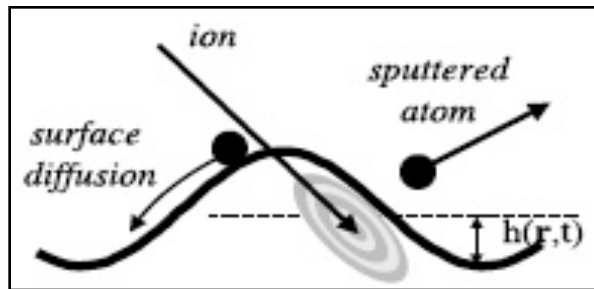


- Self-organized nanostructures under near-threshold energies (damage/sputtering)
- What are the consequences of such structures at the plasma-surface interface?
 - structures are in the spatial scale of implantation
 - effect on retention/recycling of hydrogen/helium
 - effect on erosion, recombination, surface diffusion
- New *radiation resistant* materials

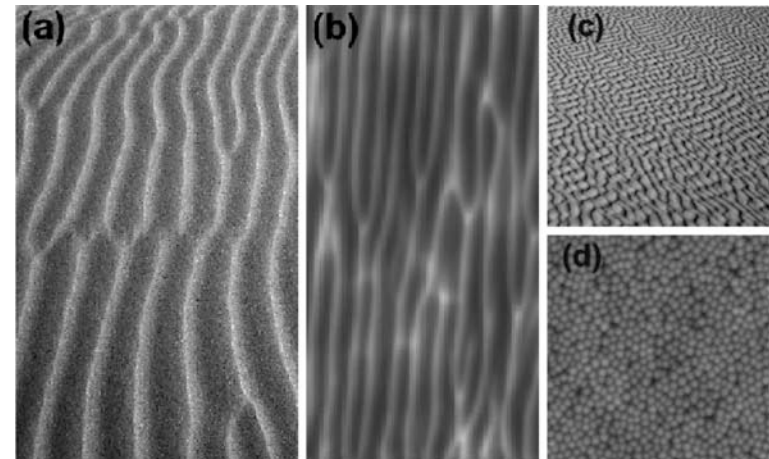
Case Studies: in-situ surface characterization of irradiation-driven surfaces



Self-organized structures and directed irradiation synthesis



E. Chason *et al.* 2001. *Nucl. Instrum. Methods B* **178**: 55-61.



(a) Ripples on sand dunes in Morocco, (b) 3.7x6.7 μm AFM image of Si surface immersed in argon plasma, (c) "dots" on a sand dune, (d) 1x1 μm² AFM image of GaSb irradiated by 0.7-keV Ar⁺ ions under normal incidence.*

Kuramoto-Sivashinsky (KS) equation*:

$$\frac{\partial h(x,y)}{\partial t} = v_o + \nu \nabla^2 h - D \nabla^2 (\nabla^2 h) + \frac{\lambda}{2} (\nabla h)^2 + \eta(x,y,t)$$

temporal evolution of surface height

avg erosion velocity

ion-induced eff. surface tension

surface relaxation due to diffusion (both thermally activated and ion-induced)

λ : tilt-dependent erosion rate, governs long-term order effects

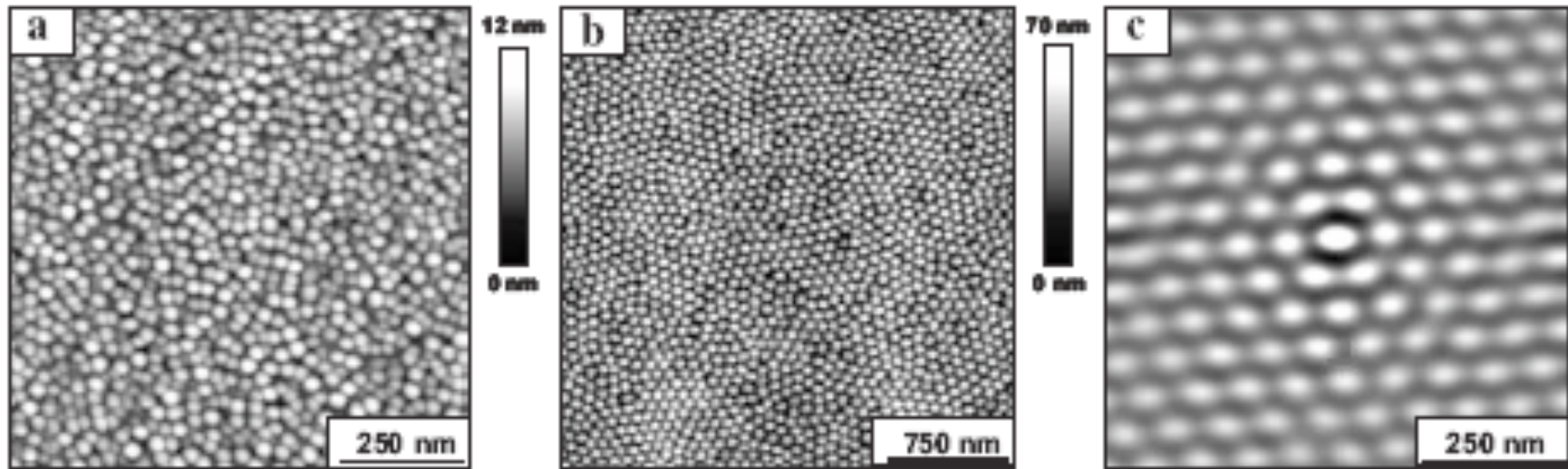
white noise parameter: stochastic nature of ion sputtering

- Forces and mechanisms driving self-organization in nano-structures.
- Pathway for ion-beam nanopatterning *and* functionalization of surfaces

Motivation

- Models are elucidating on more complex mechanisms driving nanopatterning at low energies
- Erosion vs diffusive vs re-distributive mechanisms need to be better understood
- Correlation of surface nanopatterning to concentration evolution of multi-component surfaces is poorly understood, in particular at energies approaching the sputter threshold (<100 eV)
- In-situ techniques may aid when coupled with computational modeling in deciphering the mechanisms that drive nanopatterning at energies approaching the sputter threshold
- What are the mechanisms driving nanopatterning of multi-component surfaces at energies at or below the sputter threshold?
 - Ar^+ on GaSb at normal incidence¹:
 - $Y(E=50\text{eV}) = 0.05$; $Y(E=100\text{ eV}) = 0.25$

Nanopatterning III-V semiconductors



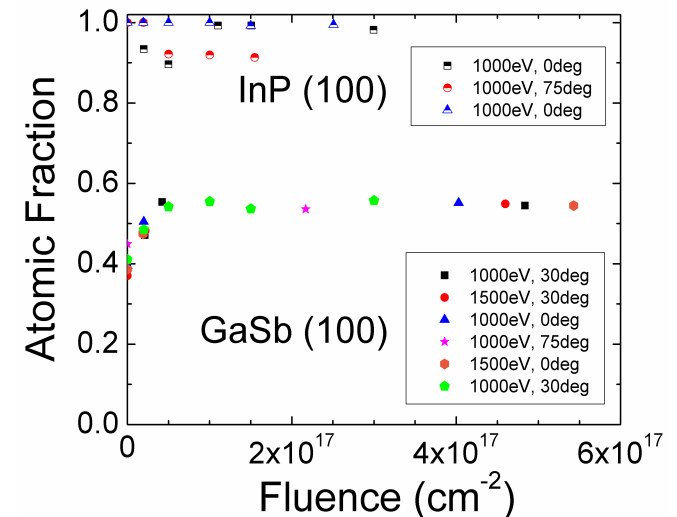
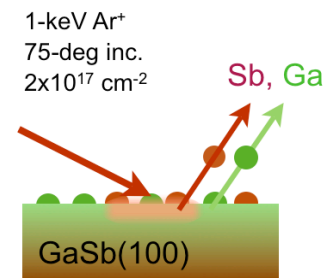
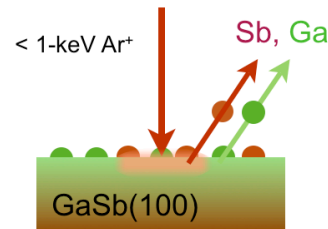
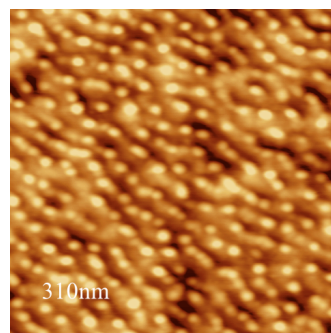
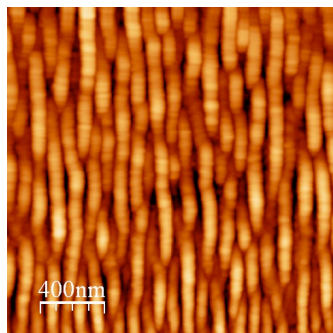
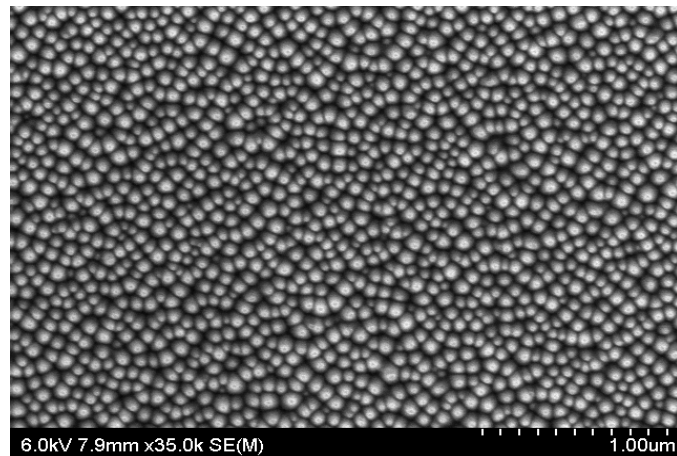
Fluence = $5 \times 10^{16} \text{ cm}^{-2}$

Fluence = $5 \times 10^{17} \text{ cm}^{-2}$

- Ion-induced morphology on semiconductor surfaces evolves and depends on irradiation fluence (dose)
- 500 eV Ar sputtering with $400 \mu\text{A}/\text{cm}^2$ ion incidence at 40 degree-incidence on InP
- Ion sculpting can be used for surface templating or quantum dot fabrication

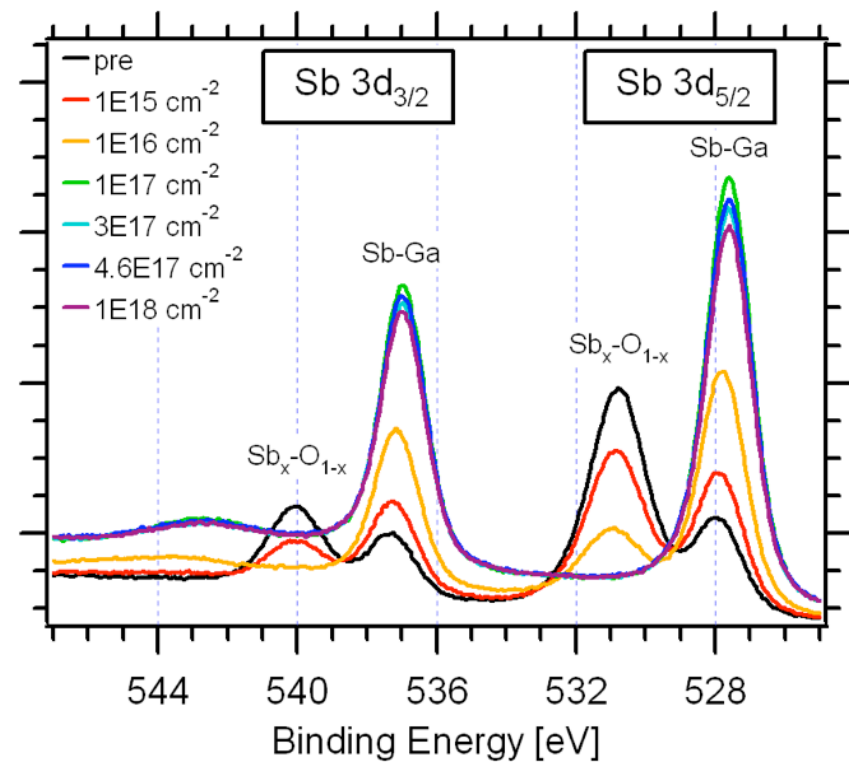
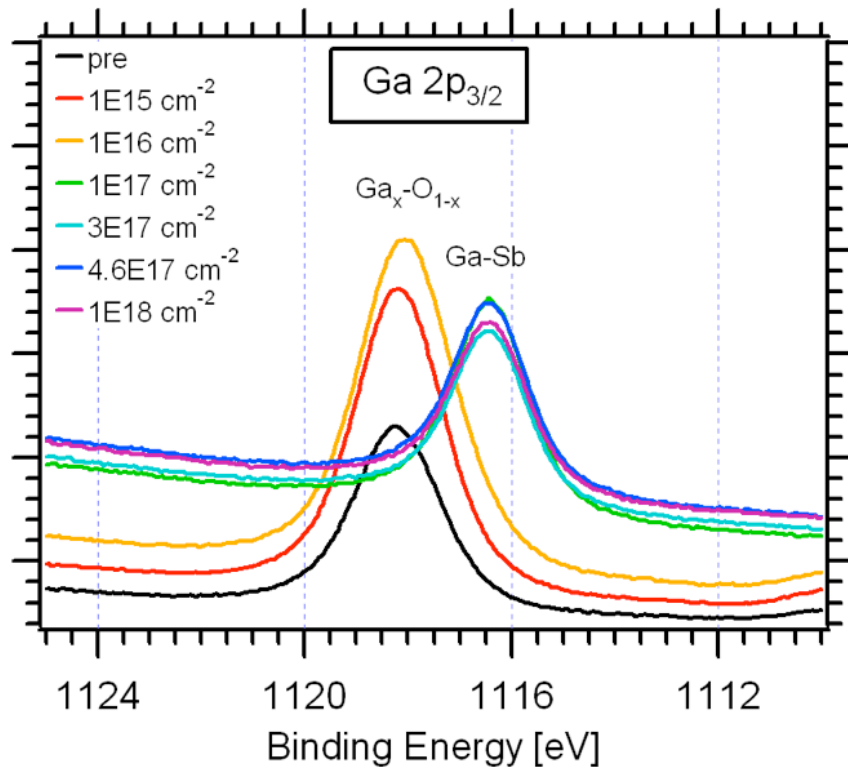
F. Frost and B. Rauschenbach, Appl Phys A 77, 1-9 (2003)

Evidence of threshold energy-induced morphology is complex and likely not explained by BH models



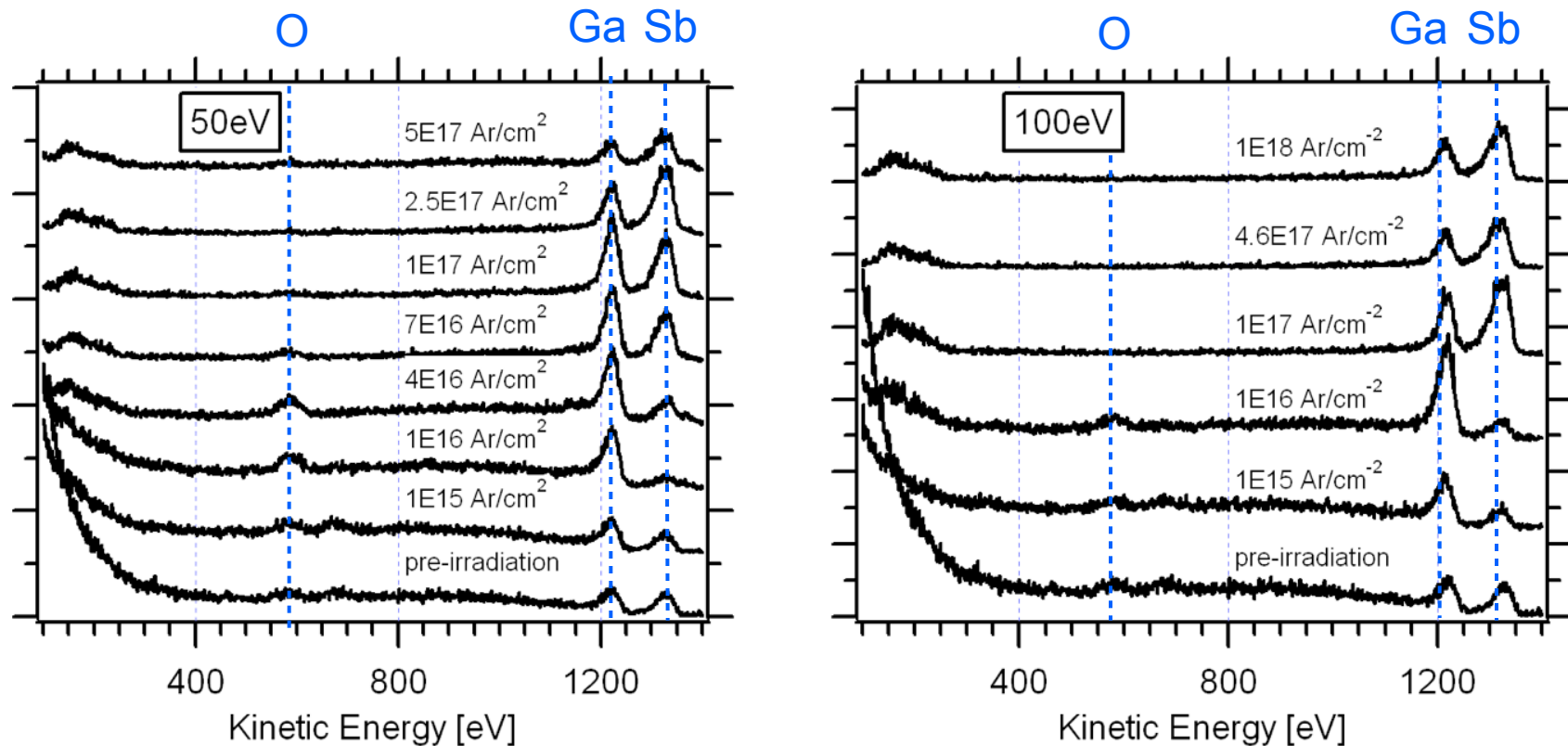
- Defect kinetics in the damaged zone could be critical to driving of the nanoscale structure on surface
- For energies studied 50-200 eV, this region in GaSb is only 0.6-1.6 nm

In-situ XPS results for irradiated GaSb(100)



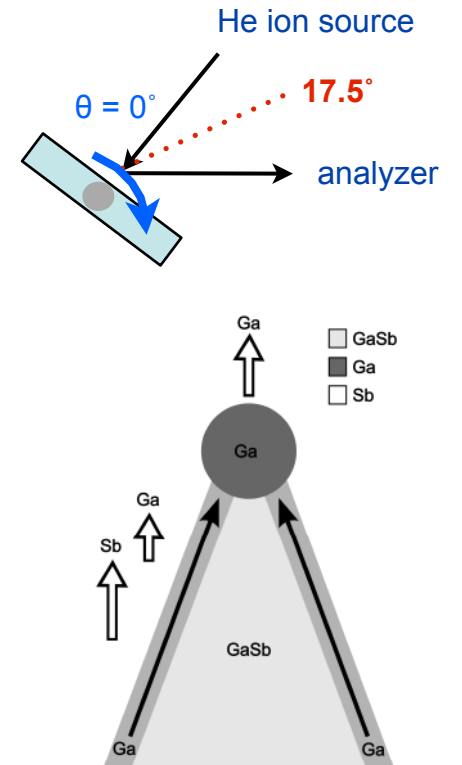
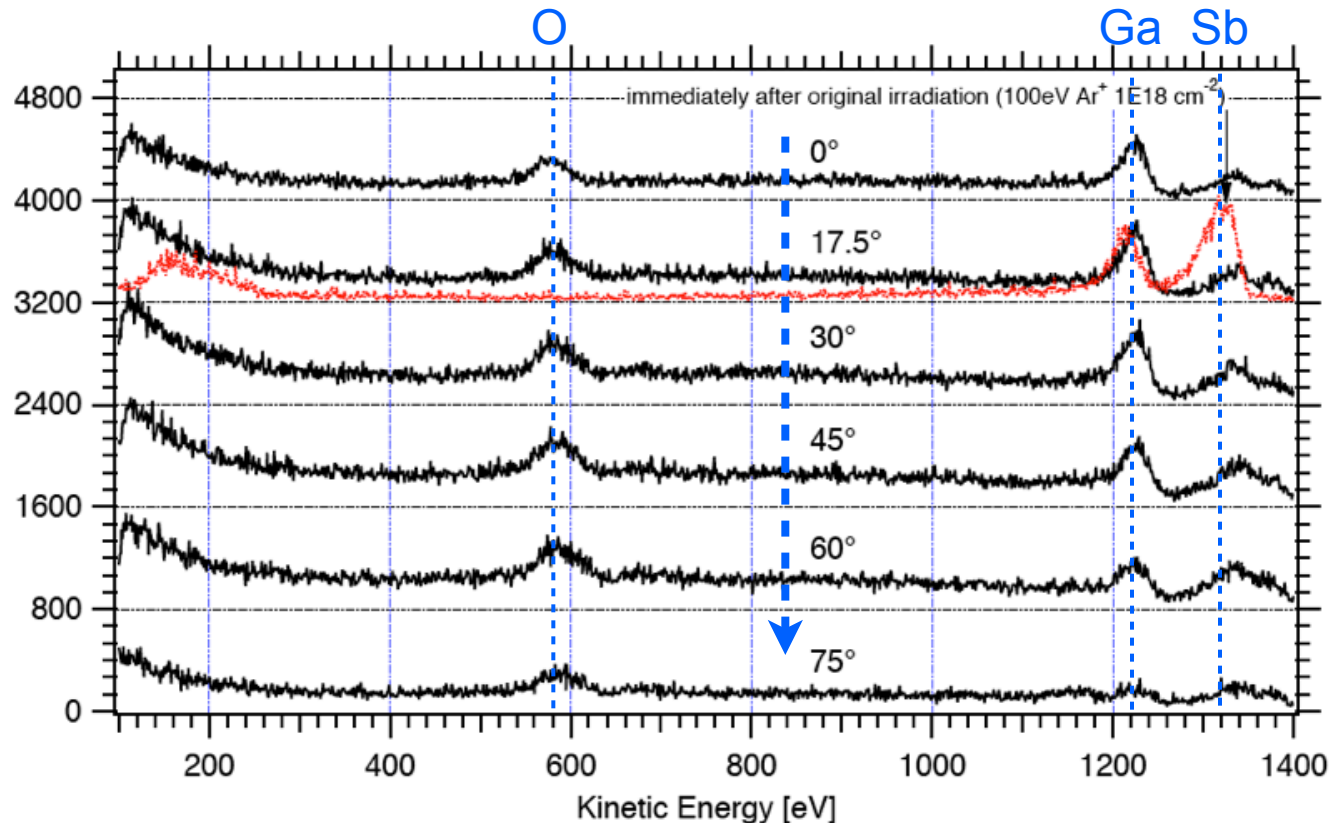
- XPS gives clear indication of Ga oxide as well as Sb oxide on subsurface region.
- between 10¹⁶ and 10¹⁷ cm⁻² clear indication that surface oxides are no longer dominant
- sub-surface shows only Ga-Sb bonding indicative of crystalline structure

In-situ LEISS results for irradiated GaSb(100)



- Early stages evidence of Ga oxide predominantly on the first few ML
- From 10^{16} to 10^{17} cm⁻² a dramatic increase in Sb is evidenced
- Sb elastic lab cross section = $0.0065 \text{ \AA}^2/\text{sr}$; Ga = $0.0102 \text{ \AA}^2/\text{sr}$

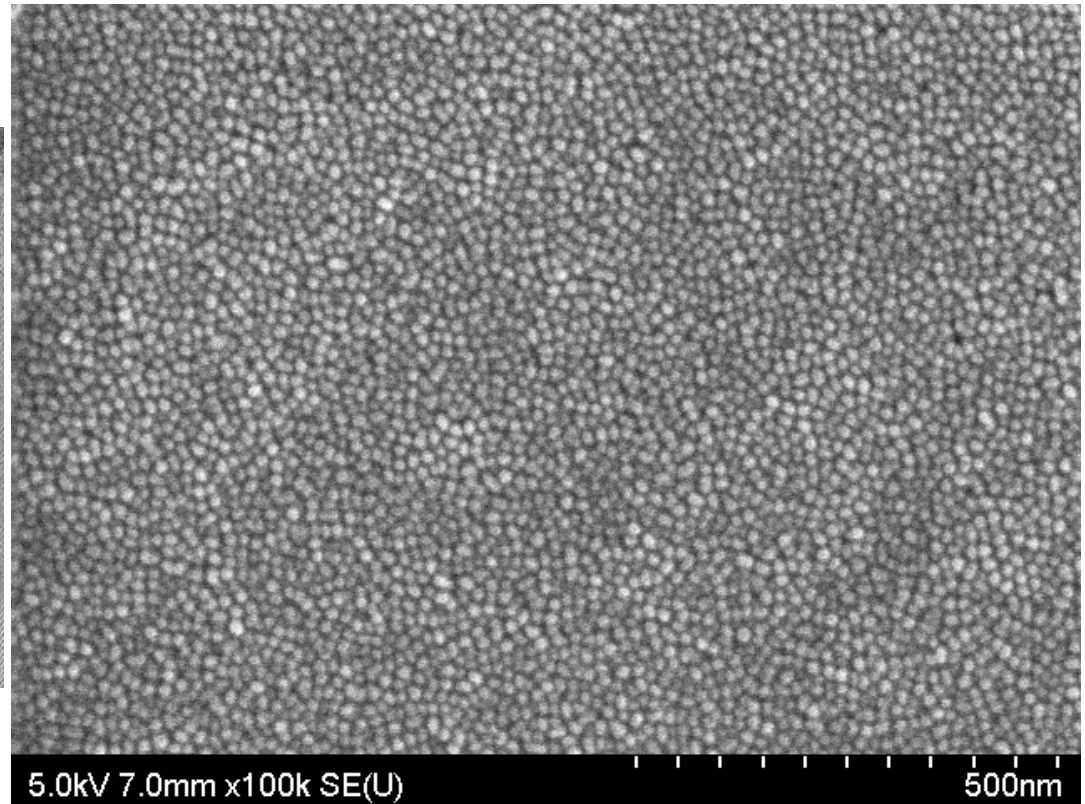
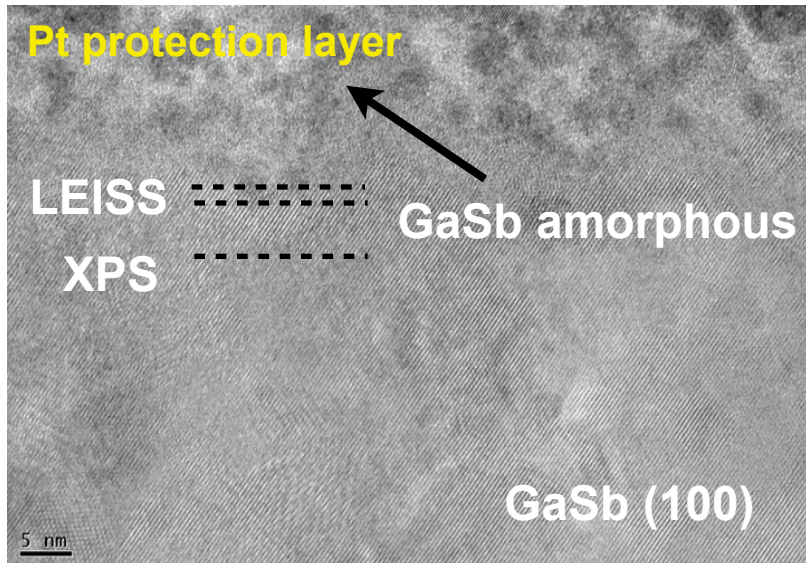
What happens with the irradiated samples exposed to atmosphere for analysis?



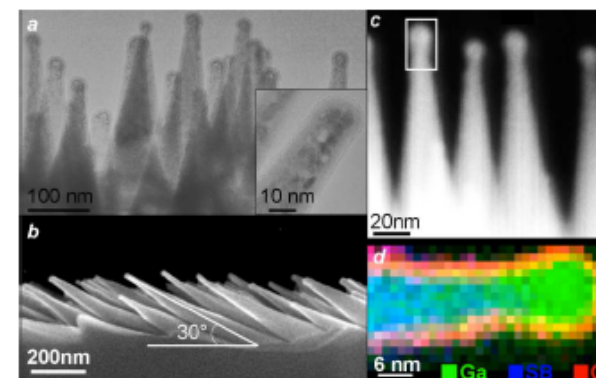
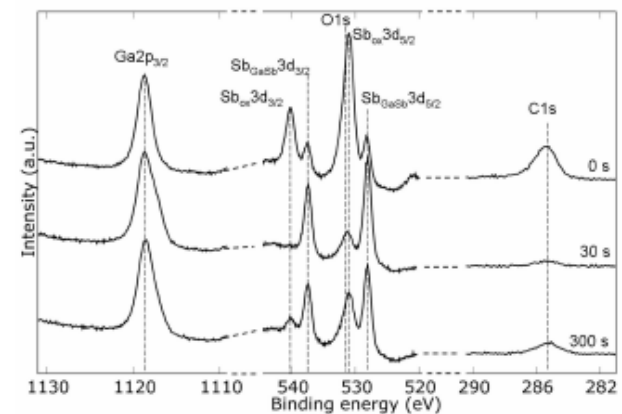
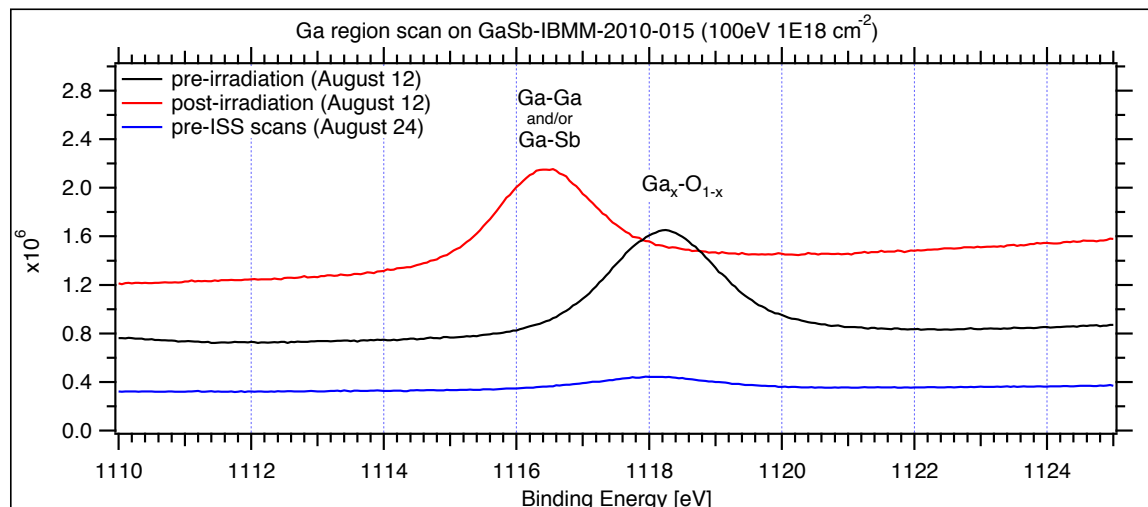
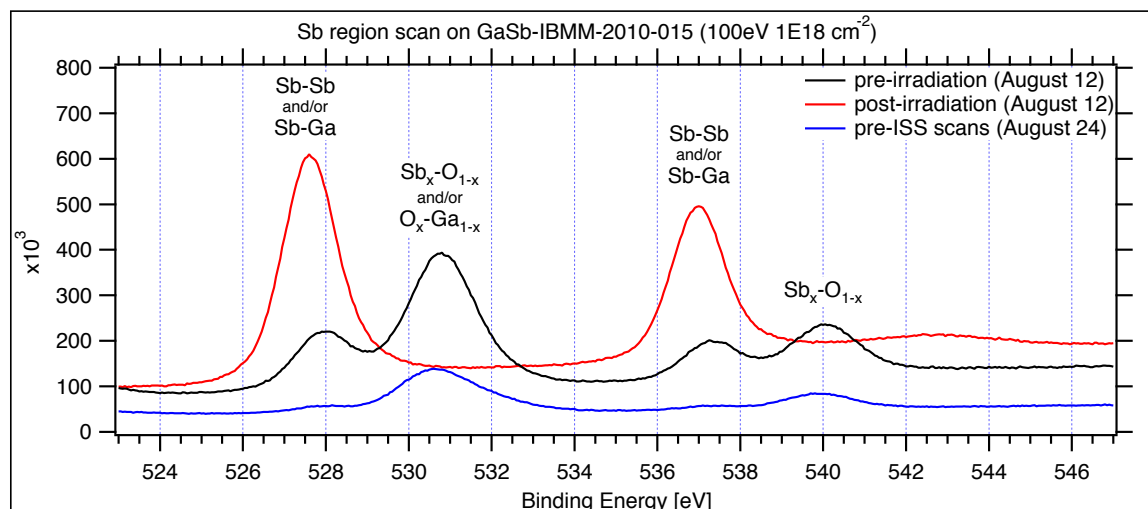
Le Roy et al. PRB 2010

- Patterned sample exposed to air and returned for characterization after several days
- LEISS spectra for various tilts “probing” from the tip of the dot/pyramid to its sides
- When compared to non-contaminated data clear segregation of Ga to surface upon reduction of oxide (here the surface free energy is critical)

TEM and HR-SEM work on GaSb



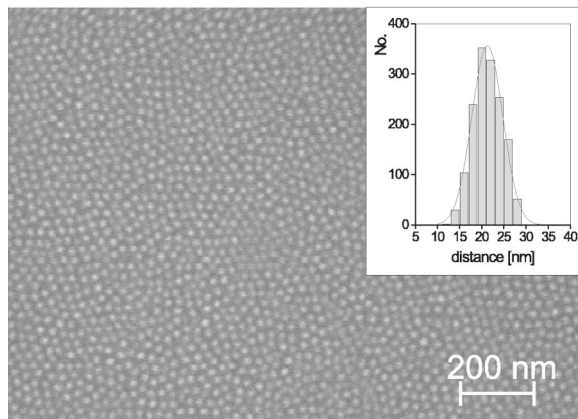
- 13-15 nm nanostructures synthesized by 50 eV Ar⁺ at normal incidence
- Top 1-1.2 nm is an amorphous layer and dominated by Sb atoms (LEISS)
- Subsurface is crystalline structure with stoichiometric 1:1 composition (XPS)



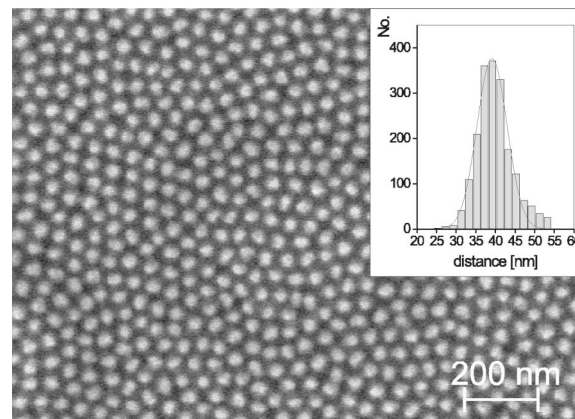
Le Roy et al. J. Appl. Phys 2009

In-situ characterization elucidated on mechanism for nanostructure formation and the role of an ultra-thin amorphous top layer

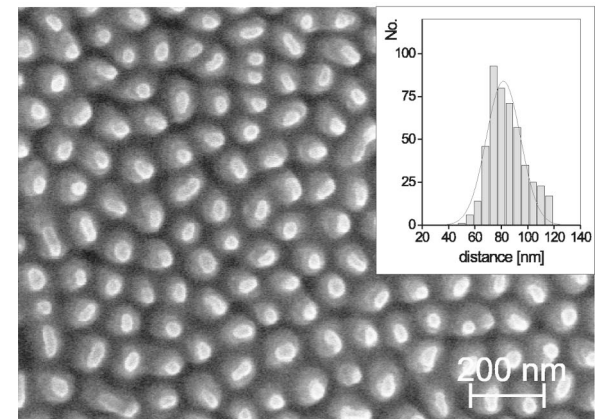
Ar⁺ on GaSb and role of incident particle energy on nanopattern



500 eV



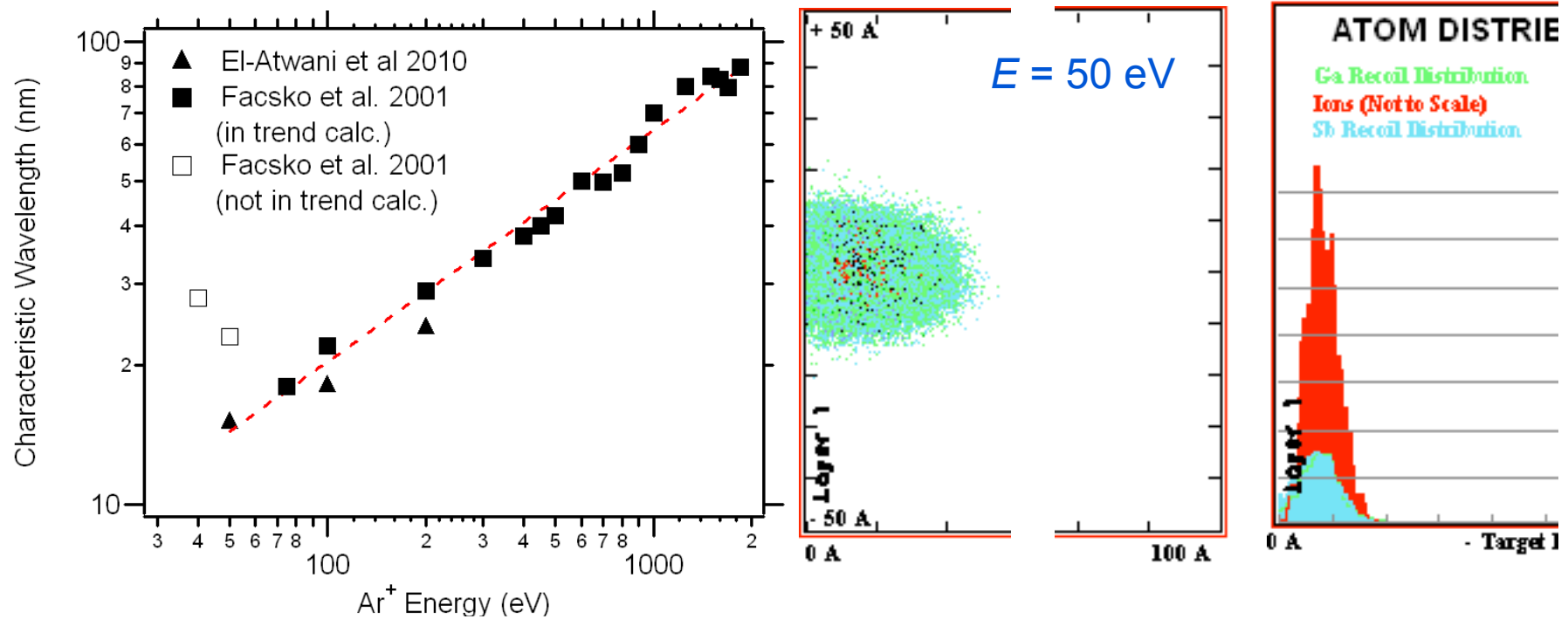
1000 eV



1500 eV

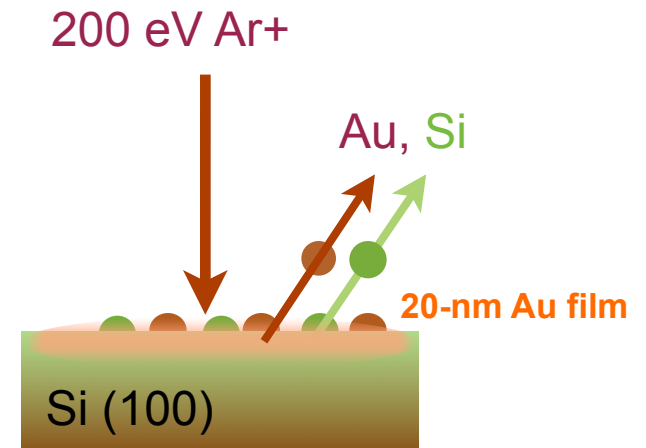
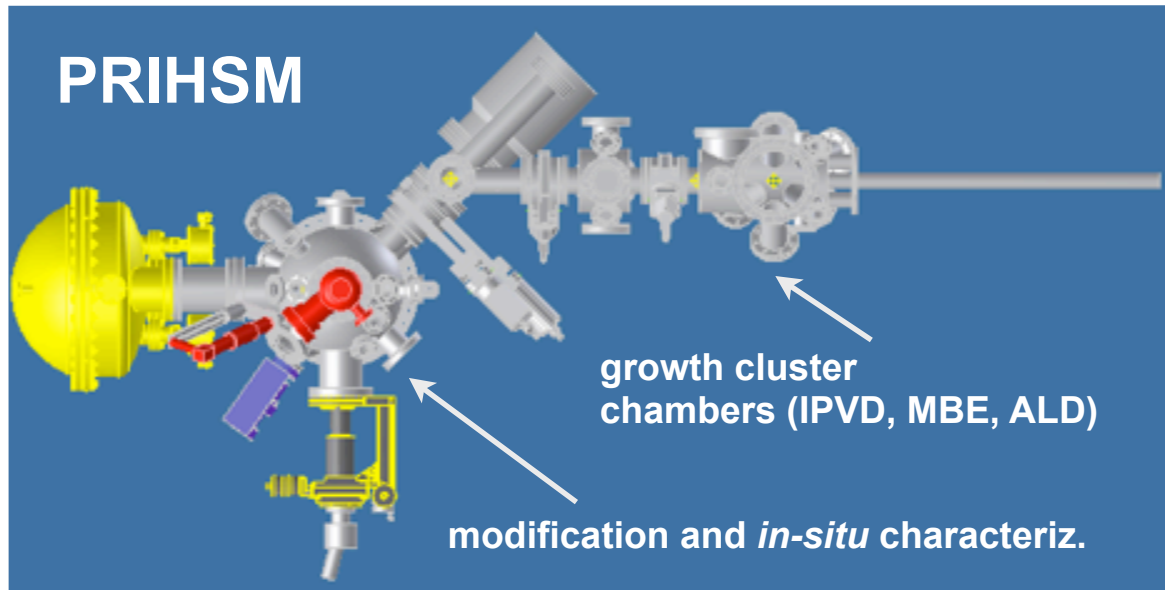
S. Facsko and H Kurz. Phys Rev B, 63 (2001) 165329.

Energy dependence of dot formation on GaSb



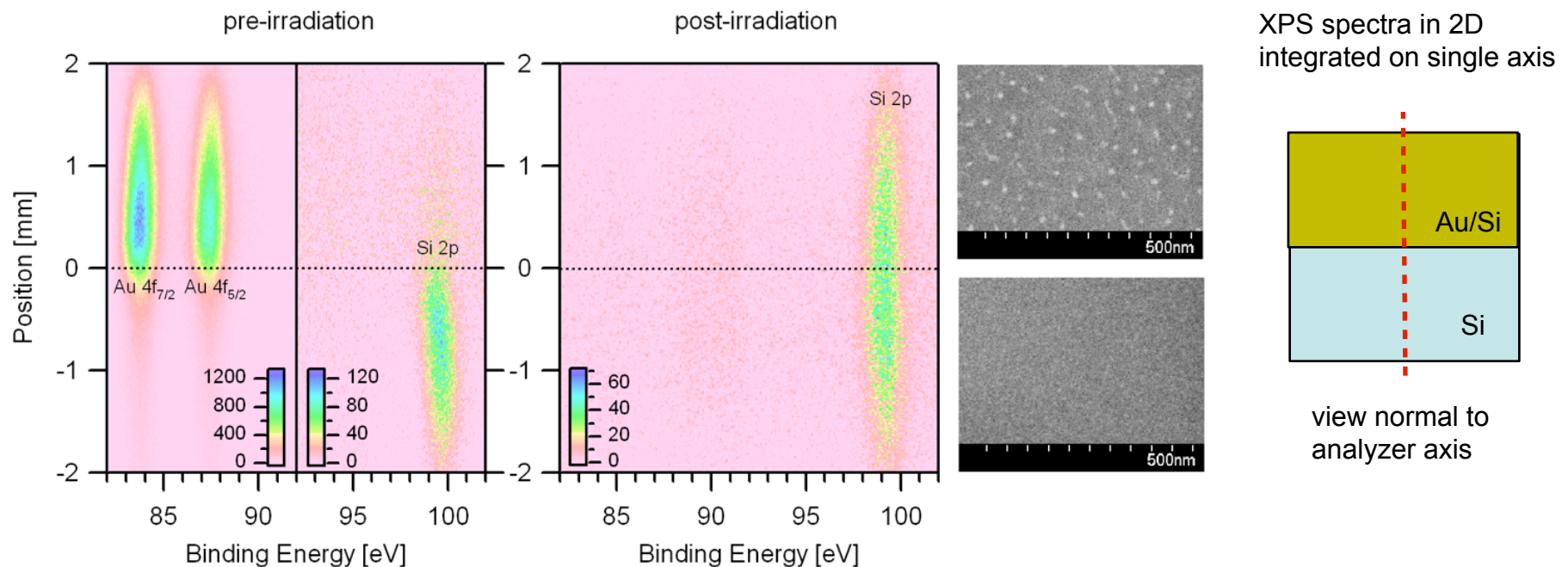
- Correlation between incident particle energy and characteristic “size” of nanostructures
- In this case “size” is measured as distance from center-to-center of the dots
- BCA based calculations (SRIM 2008) show damage region in first 1-2 nm,

Si(100) surface nanopatterning



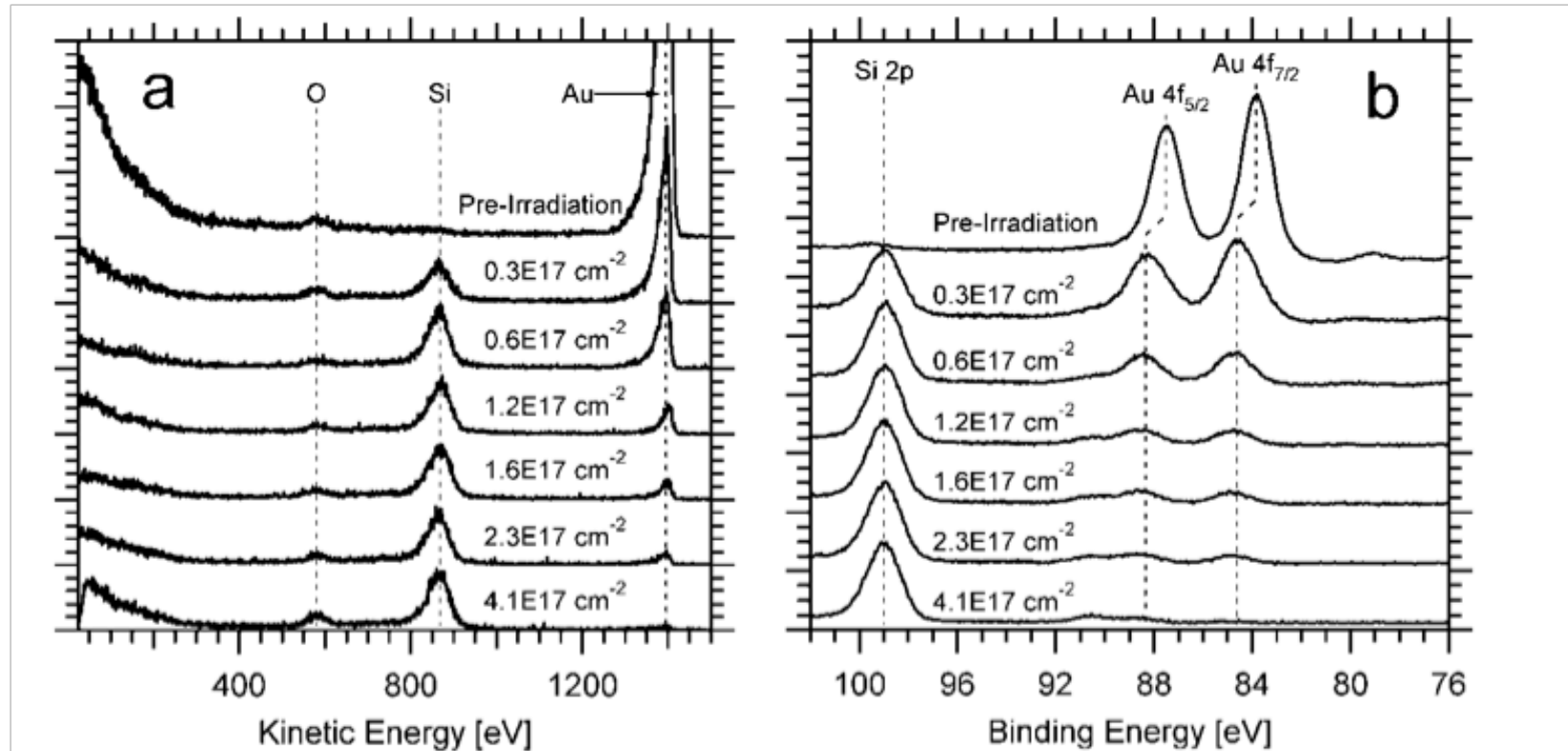
- Thin layers of Au deposited on Si(100) using MBE with various thicknesses from 3-30 nm.
- Au used as an immiscible component to study nanopattern formation on Si at normal incidence and near sputter-threshold energies (~50-200 eV)
- Irradiation up to 10^{18} cm^{-2} and study with *in-situ* XPS, *ex-situ* SEM, AFM

Nanopatterning of Si(100) with ultra-thin Au films irradiated with 200 eV Ar⁺

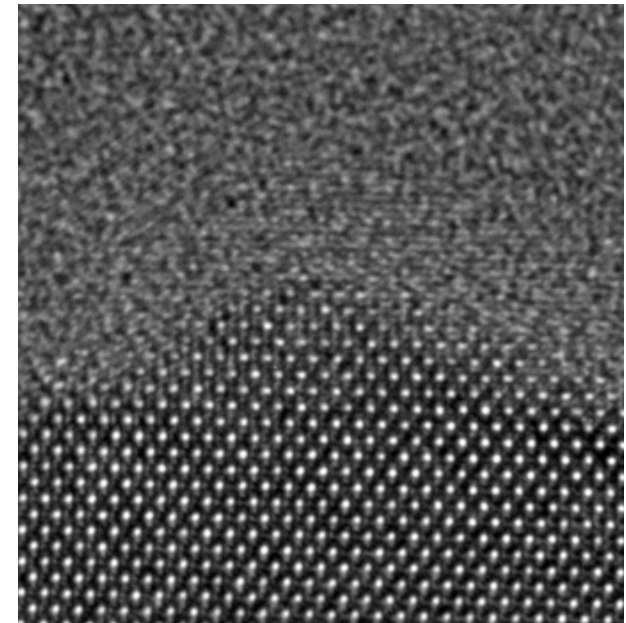
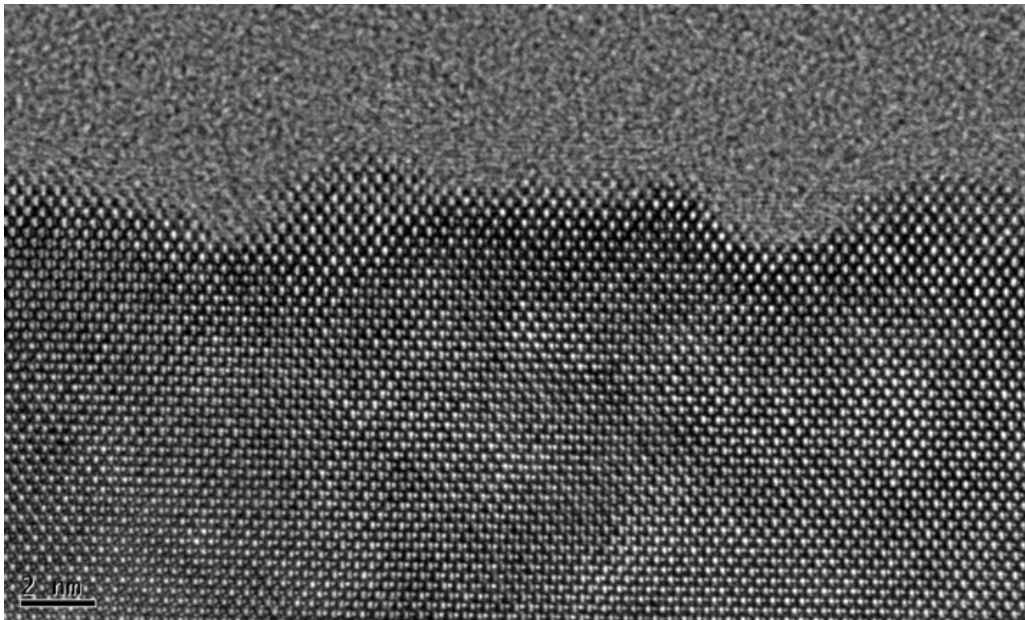


- 2D detector allows us to observe a macroscale-resolved scan. Half of Si(100) sampled masked and other half coated with thin Au film
- Irradiation at 200 eV Ar⁺ for 10¹⁸ fluence and normal incidence yields nanostructures only on region coated with gold
- In-situ XPS allows us to confirm that structures are not gold

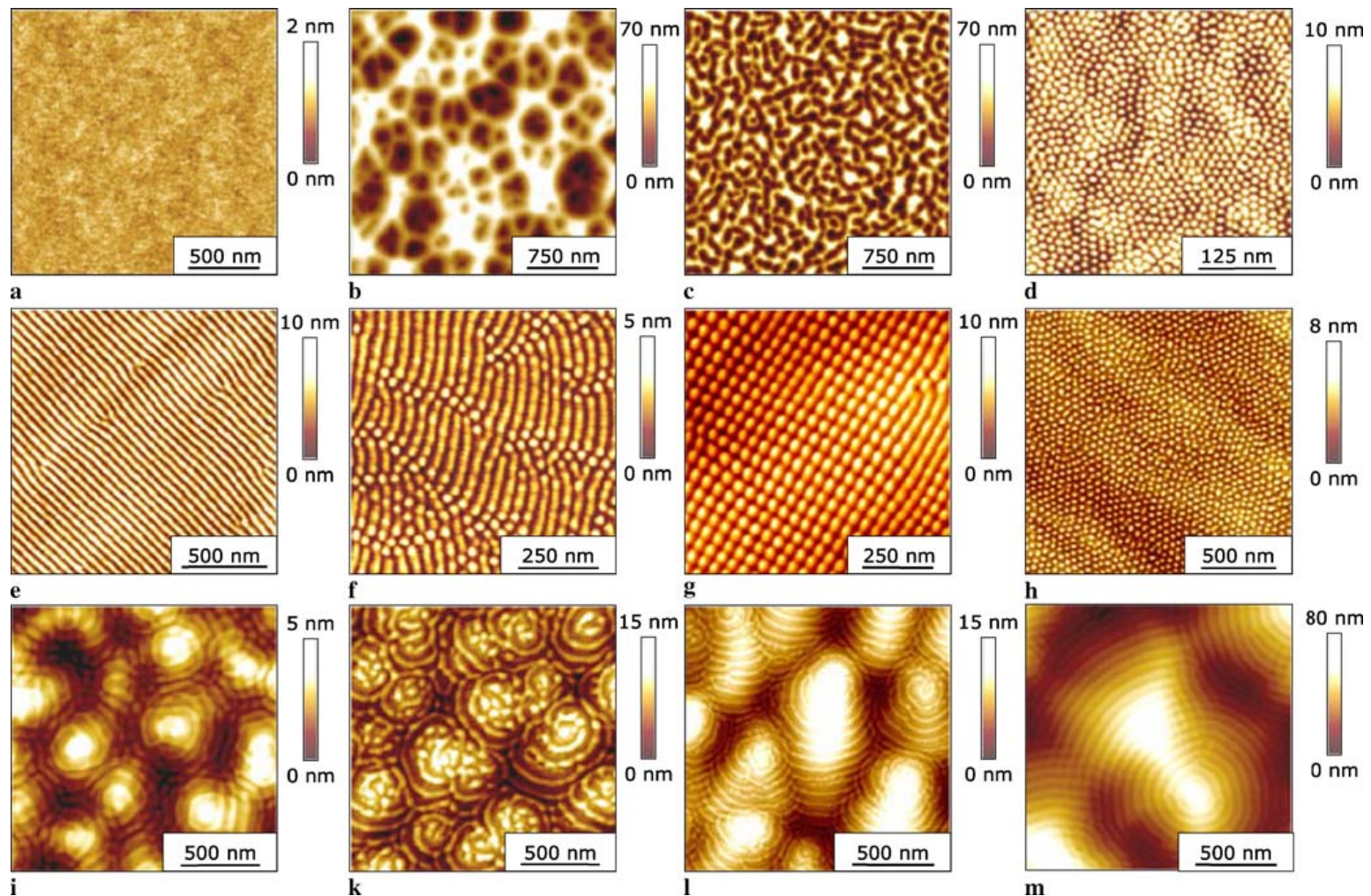
Formation of silicide induced nanopatterning of silicon elucidated by complementary surface-sensitive techniques



Si ordered nanostructures self-organized under low-energy irradiation



- 200 eV Ar⁺ ions are used at normal incident to the sample surface yielding at a threshold fluence of about 10^{17} cm⁻² (i.e. in only a few seconds...)
- One could scale up technique to combine nanopatterning and multi-layer coating deposition with ultra-thin films



By controlling near-threshold damage ion irradiation of surfaces one can attain a variety of nano-patterned surfaces. Simply varying incident angle, energy, species in a scalable and parallel nano-manufacturing approach

Summary

- *In-situ* surface characterization *during* particle irradiation can elucidate on relevant mechanisms of irradiation-driven self-organization
- Limiting factors of experimental approach lies on spatial and temporal scales probed
 - Use of complementary surface diagnostics
 - Complement with computational simulations
- Case studies demonstrate importance of ion-induced surface modification and implications on surface functionality (i.e. the case for 13.5-nm reflectivity)
- Correlating ion-induced nano-patterning to surface chemistry and elemental evolution can elucidate underlying mechanisms at particle energies near sputter threshold

Extra Slides

Thank You for your Attention