Advanced adaptive nanomaterials under extreme conditions: current progress and challenges

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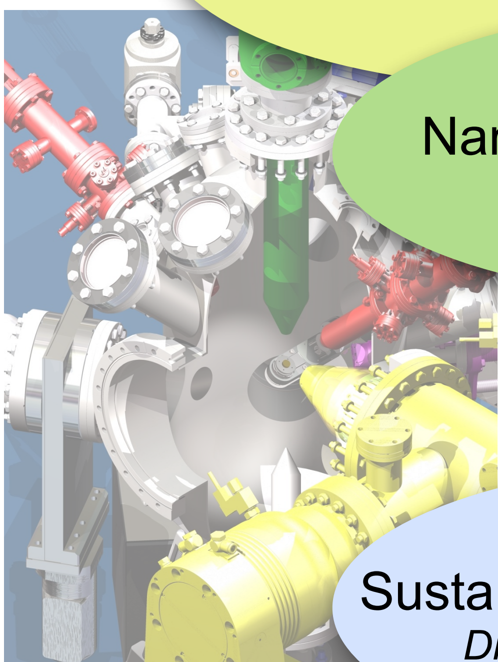
Fermilab Colloquium
Batavia, Illinois
22 October, 2014

*We design self-organized nanostructures with **directed irradiation synthesis** and **directed plasma nanosynthesis** to enable multi-functional and multi-scale properties at surface and interfaces of dissimilar material systems (e.g. polymer and metals, ceramics and biomaterials).*



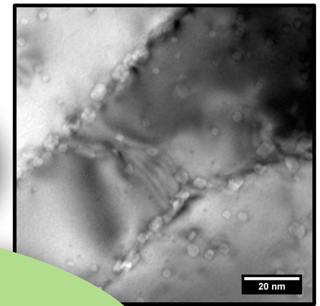
Advanced Fusion Interfaces

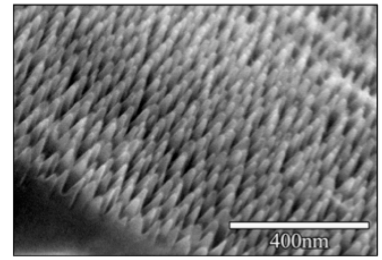
Nanostructured Functional Materials

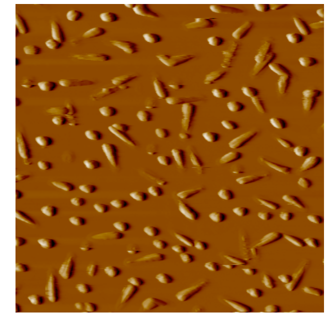
Multi-scale computational irradiation surface science

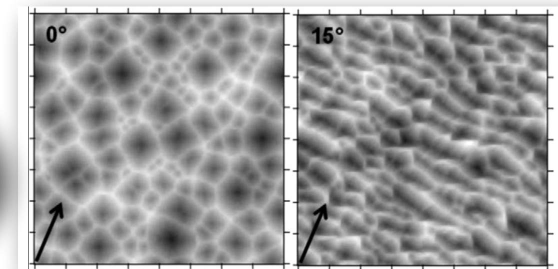
Sustainable nanomanufacturing
Directed Irradiation Synthesis

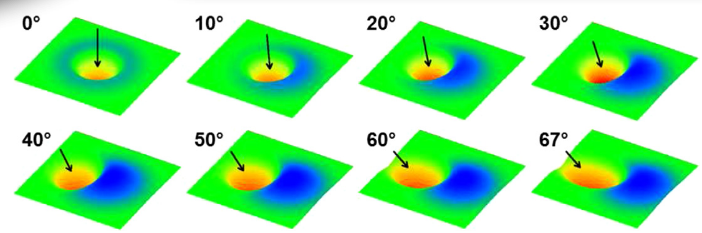
Advanced Functional Biointerfaces

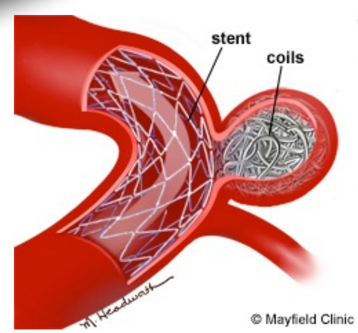


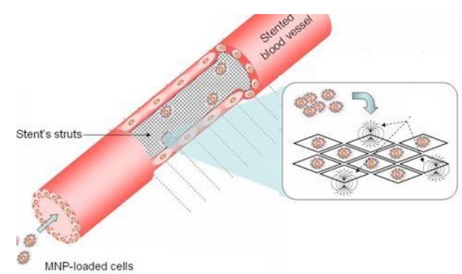












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The Radiation Surface Science and Engineering Laboratory (RSSEL): Allain Research Group

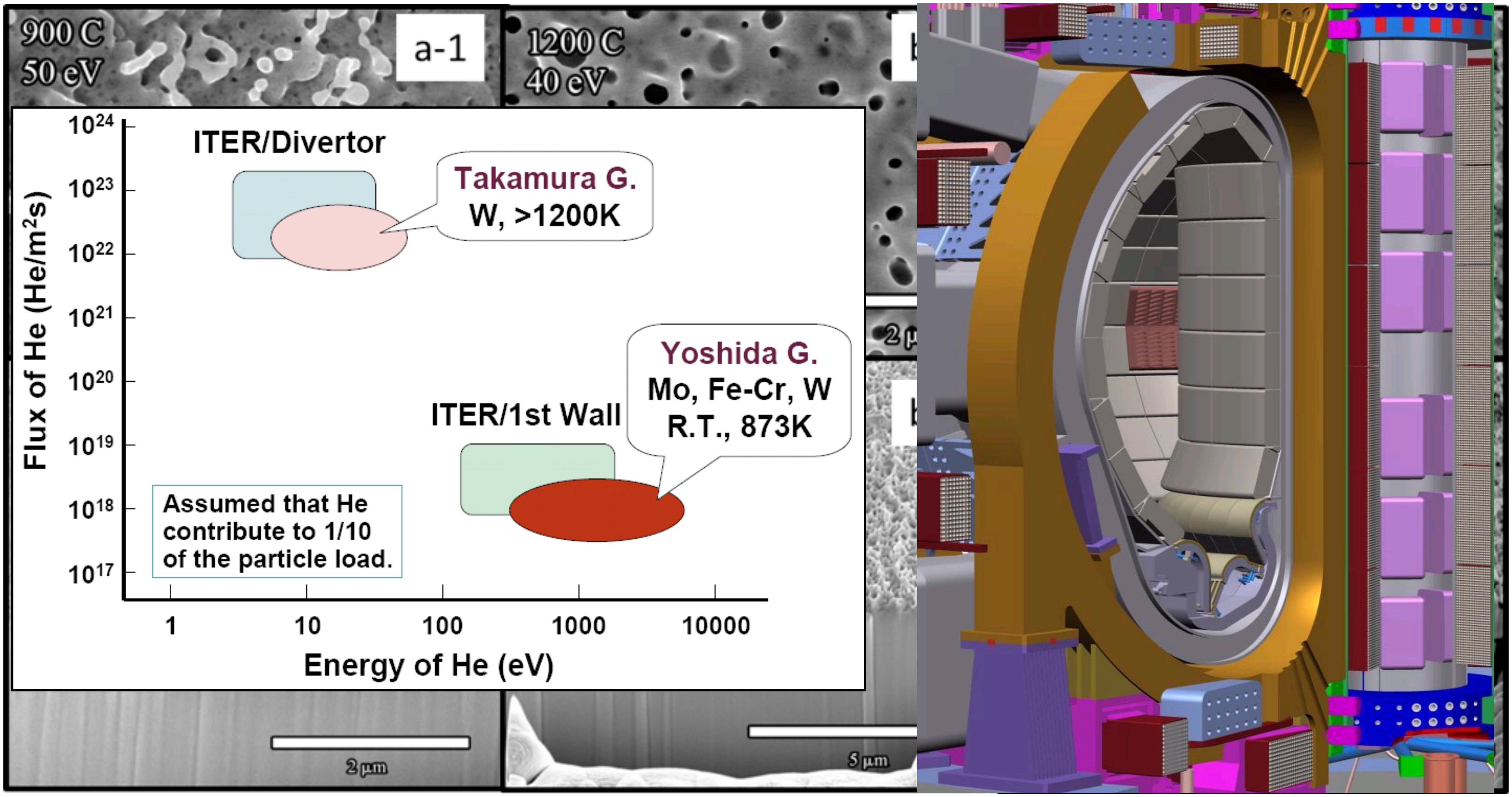
- *Graduate Students*
- *NPRE*
  - Brandon Holybee
  - Zachary Koyn
  - Felipe Bedoya
  - Anton Neff
  - Michael Lively
  - Alethia Barnwell
  - Ming Kit Cheng
- *Bioengineering*
  - Sandra Arias
- *Materials Science and Engr.*
  - Zhejun Zhang
- *Undergraduate Students*
  - Emily Lindgren
  - Kenny Nash (NE)
  - Joe Strehlow (Physics)
  - Mike Spinuzza (ME)
  - Ryan Gonsalves (Math)
  - Paula-Angela Mariano (EE)
  - Natalia Migdal (NPRE)
- *Postdocs*
  - Akshath Shetty
- *Visiting Professors*
  - Juan Jose Pavón
- *Visiting Students*
  - Viviana Posada, M.S.
  - Monica Echeverry-Rendón, M.S.

Outline of Talk

- Deciphering self-organization in extreme environments
- Searching for radiation-resistant materials
  - The case of extreme-refined W for nuclear fusion
  - *In-situ* TEM results of dynamic defect behavior in extreme refined-grain W
  - High-flux irradiations in Pilot-PSI and Magnum-PSI at DIFFER
- Multi-phase, hierarchical composite materials
- Summary and Outlook

Acknowledgements:
Osman El-Atwani (PhD 2012, postdoc 2012-2013)
Brandon Holybee, Zak Koyn (PhD students UIUC)
Felipe Bedoya (PhD student UIUC)

Irradiation-driven surface structures in fusion



- Candidate materials such as refractory metals (e.g. W, Mo) face severe irradiation-driven damage under expected conditions of future plasma burning experimental reactors such as ITER

[1]DEMO fluence at FW: ~10^{21}-10^{22} m^{-2}, E ~ 0.2-5 keV
divertor: ~10^{27}-10^{28} m^{-2}, E ~ 10-80 eV

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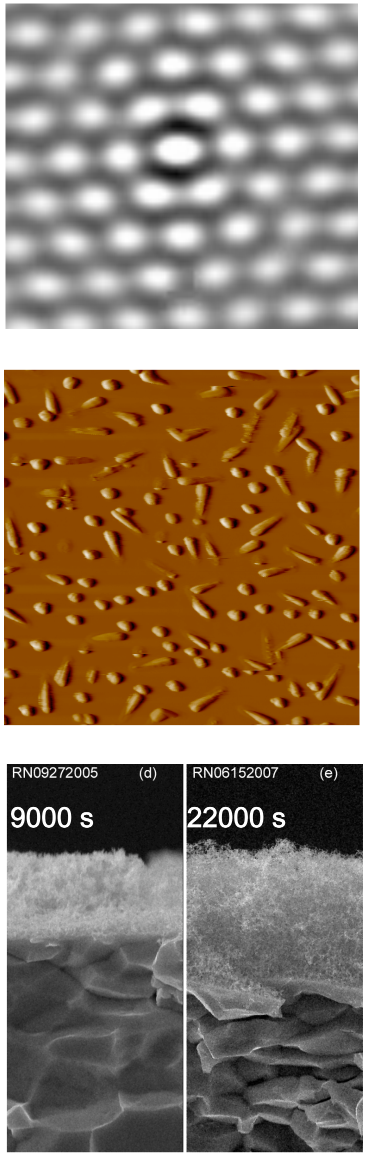
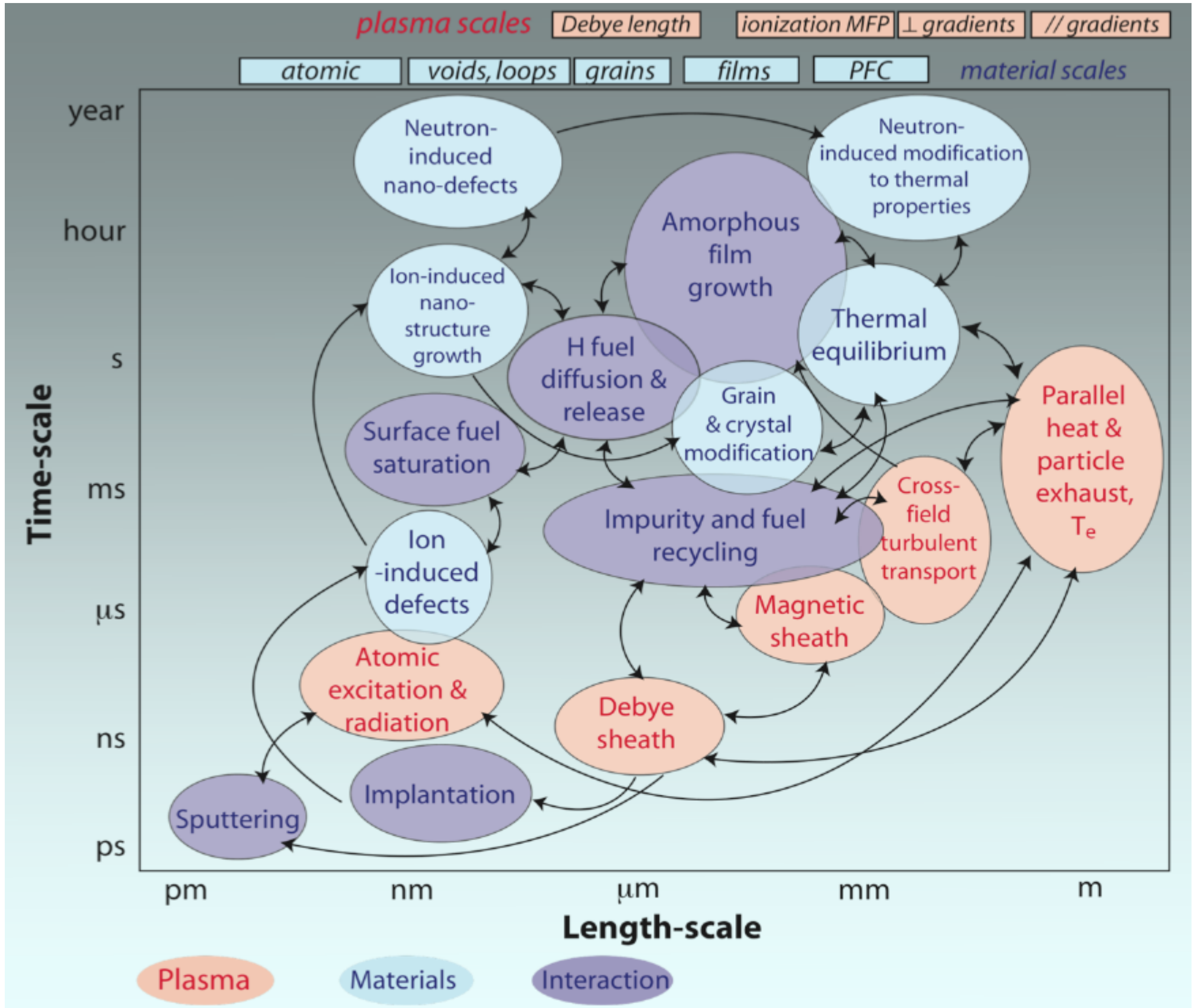
[1] C. Wong, PSI-18

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

Complexity and disorder



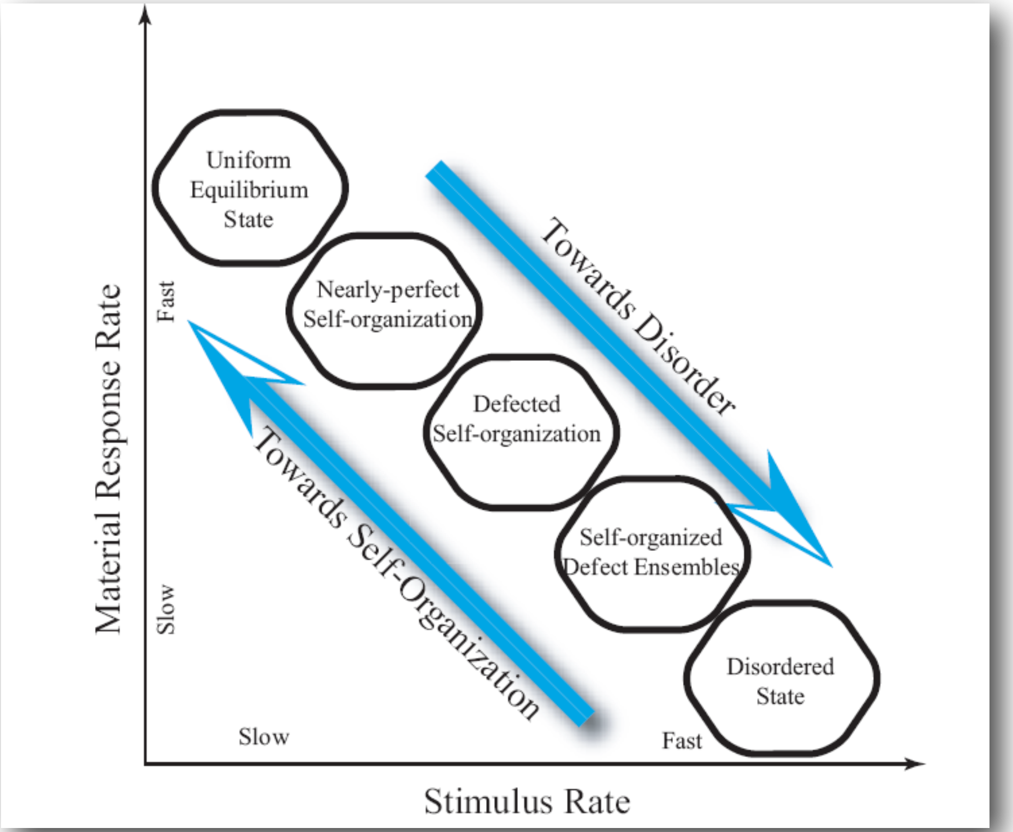


B. Wirth et al. MRS Bulletin 2011

Driving matter between disorder and self-organization by energetic ions

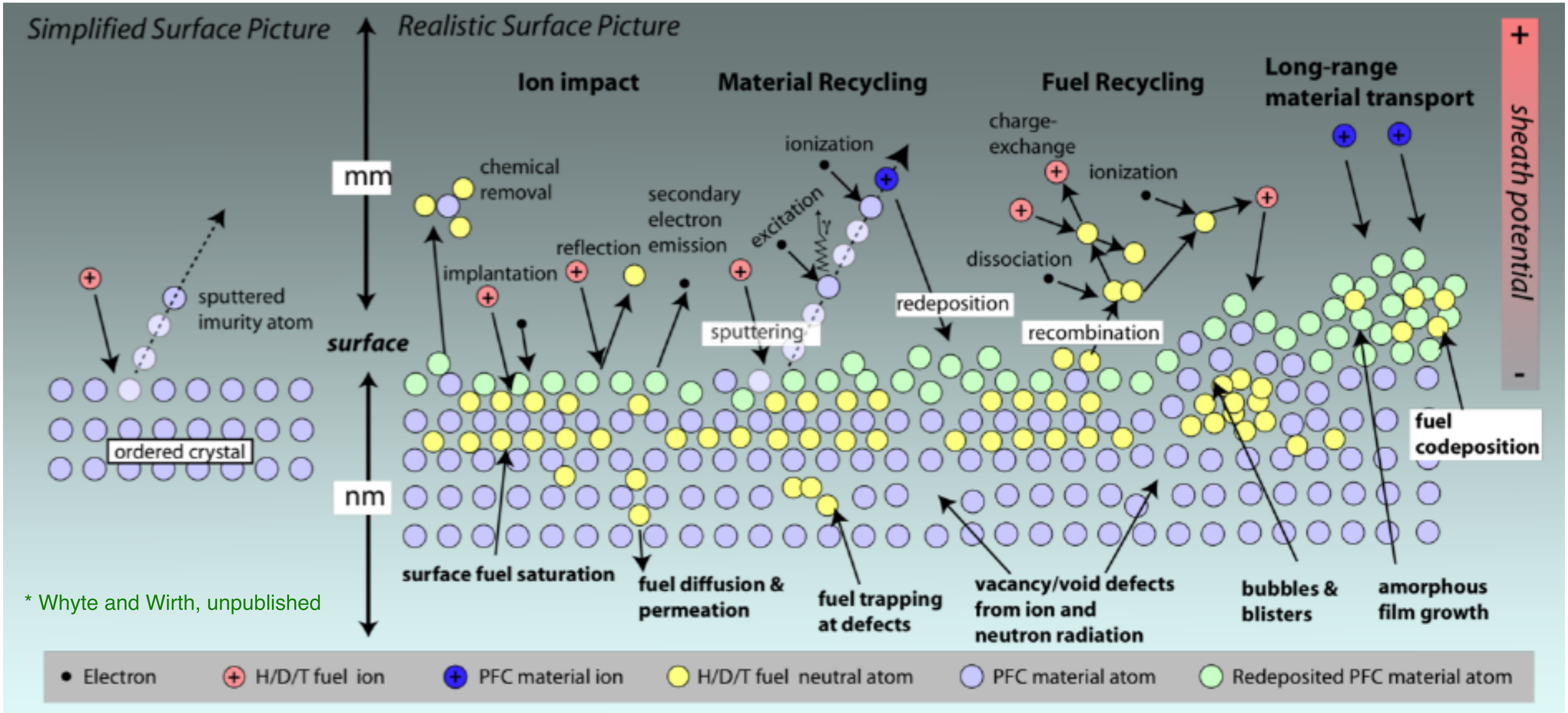
extreme environments: ion-induced damage

ion-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 (metastable phases)

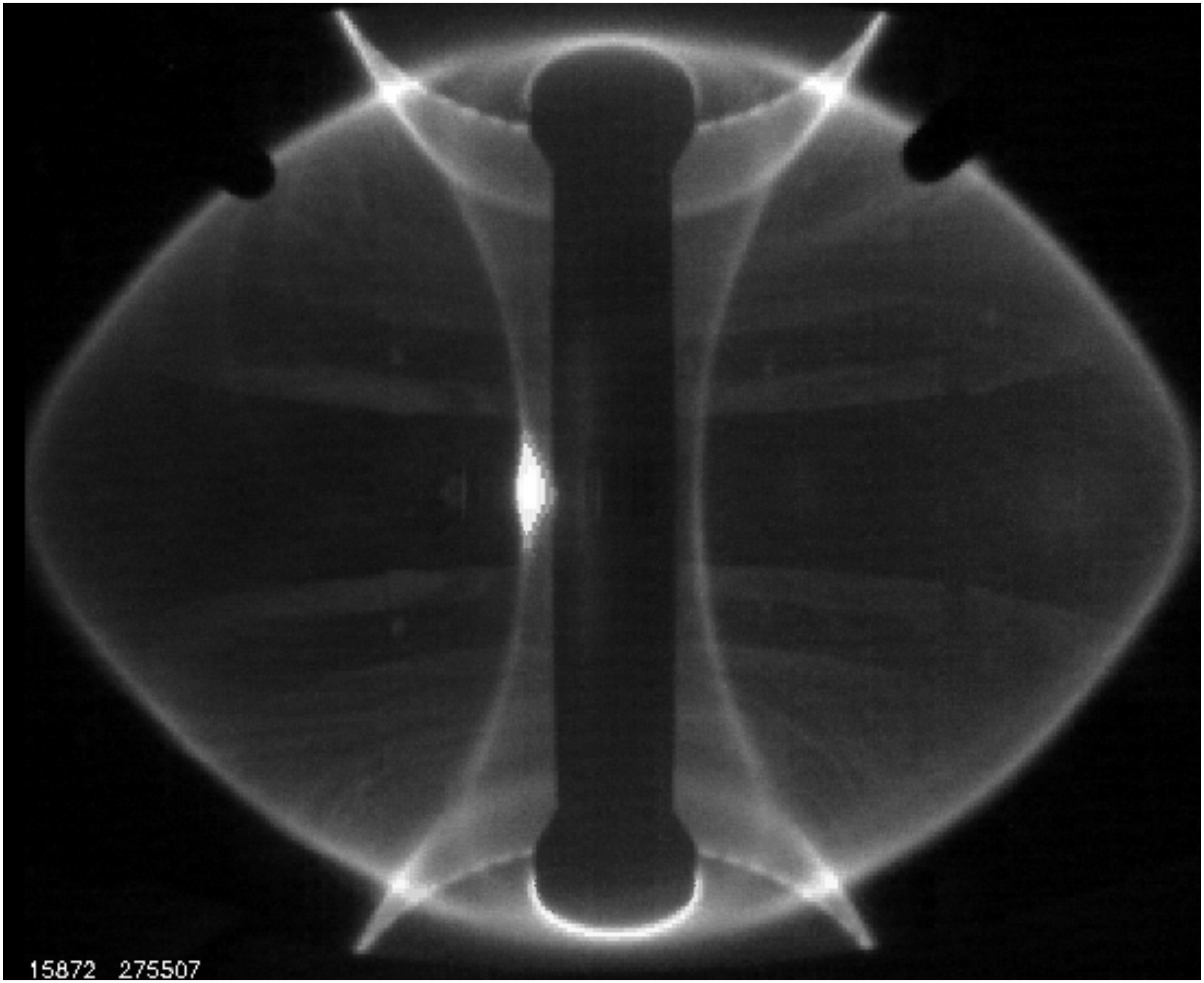
Moving from a simplified view of the plasma-surface interface (PSI) to a more rich and complex PSI



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

Courtesy of: B. Wirth and D.G. Whyte

Extreme and quiescent plasma events in tokamak fusion devices



Courtesy: Alan Sykes, EURATOM/UKAEA, Culham Science Centre

UKAEA Fusion Working in Europe

What will conditions inside a demonstration fusion power plant reactor (DEMO)?

Issue / Parameter	Present Tokamaks	ITER	DEMO	Consequences
Quiescent energy exhaust *GJ / day*	~ 10	3,000	60,000	- active cooling - max. tile thickness ~ 10 mm
Transient energy exhaust from plasma instabilities $\Delta T \sim MJ / A_{wall}(m^2) / (1\ ms)^{1/2}$	~ 2	15	60	- require high $T_{melt/ablate}$ - limit? ~ 60 for C and W - surface distortion
Yearly neutron damage in plasma-facing materials *displacements per atom*	~ 0	~ 0.5	20	- evolving material properties: thermal conductivity & swelling
Max. gross material removal rate with 1% erosion yield *(mm / operational-year)*	< 1	300	3000	- must redeposit locally - limits lifetime - produces films
Tritium consumption *(g / day)*	< 0.02	20	1000	- Tritium retention in materials and recovery

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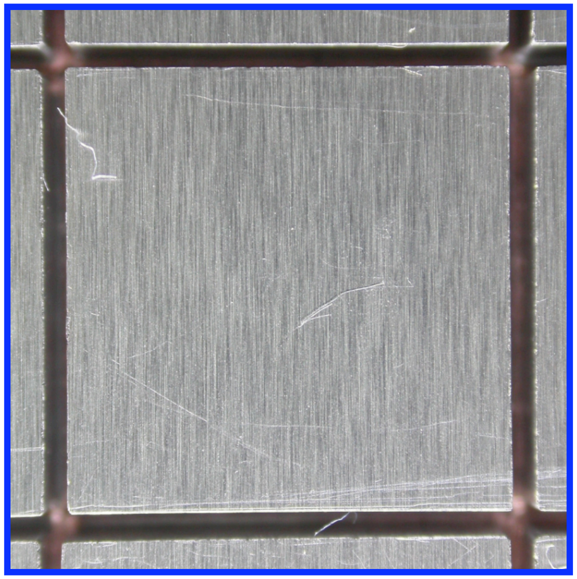
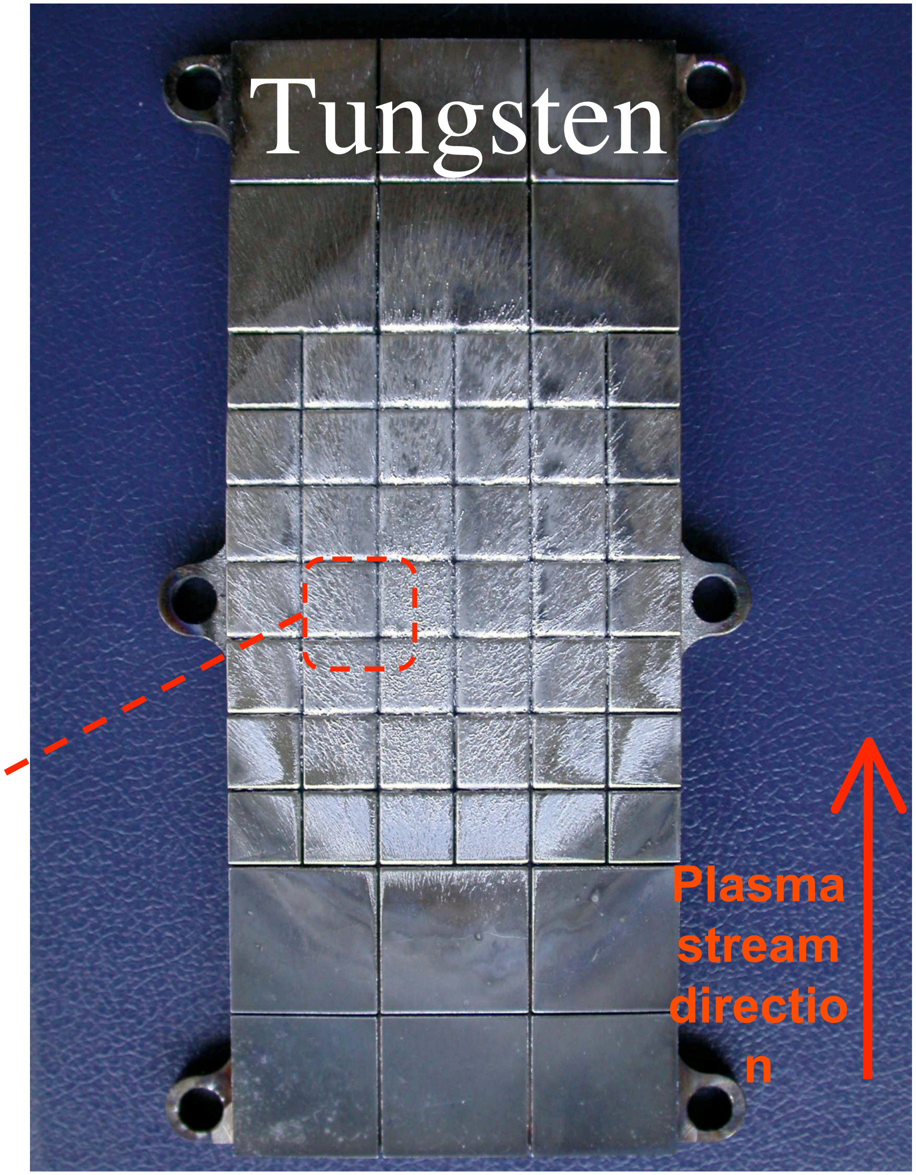
[1] D.G. Whyte, HHFC Meeting, UCSD, December 2008

Before exposure



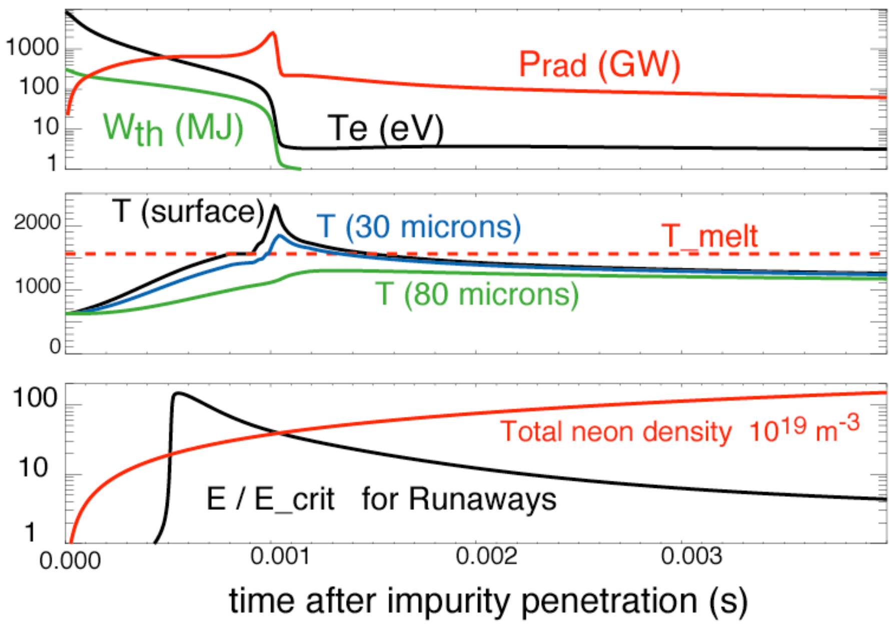
After 5 “large” ELMs (~2 MJ / m^2)



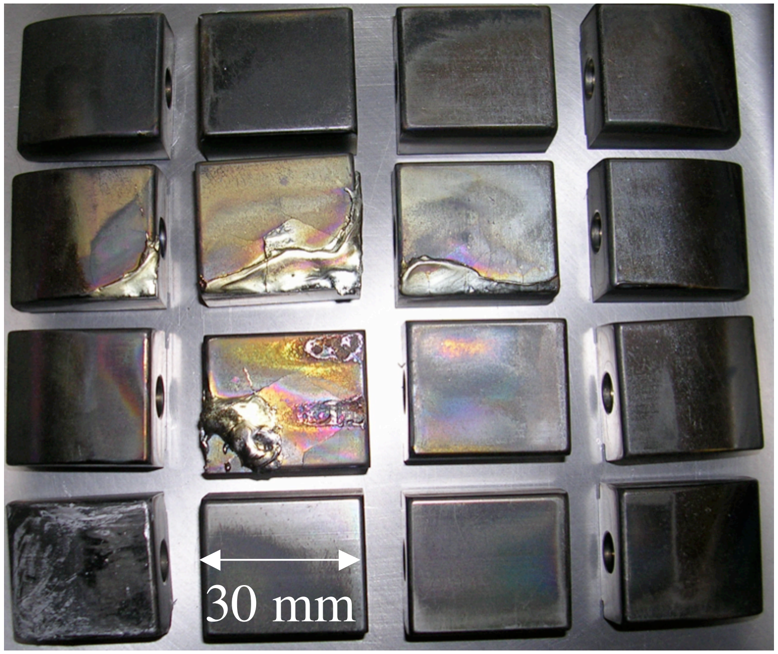
Similar arguments apply to even “minor” transient heating in reactor-class devices. e.g. material heating limits lead to very restrictive ELM size in ITER

[1]D.G. Whyte, HHFC Meeting, UCSD, December 2008

Thermal energy dissipation (order of msec) can be easily triggered by PSI failure



C-Mod Molybdenum (T_{melt}=2900 K) limiter melted during disruptions



- Dilute MFE plasma (n~10^{20} m^{-3}) extinguished by small particulate
  - 2 mm "drop" of W == $N_{e,ITER}$

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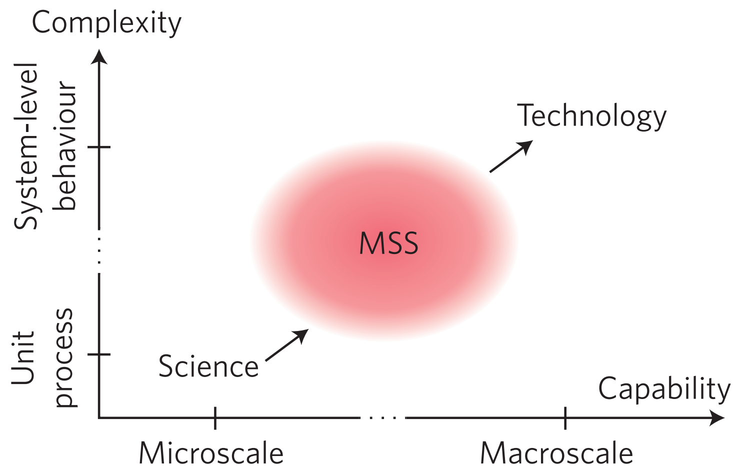
[1]D.G. Whyte, HHFC Meeting, UCSD, December 2008

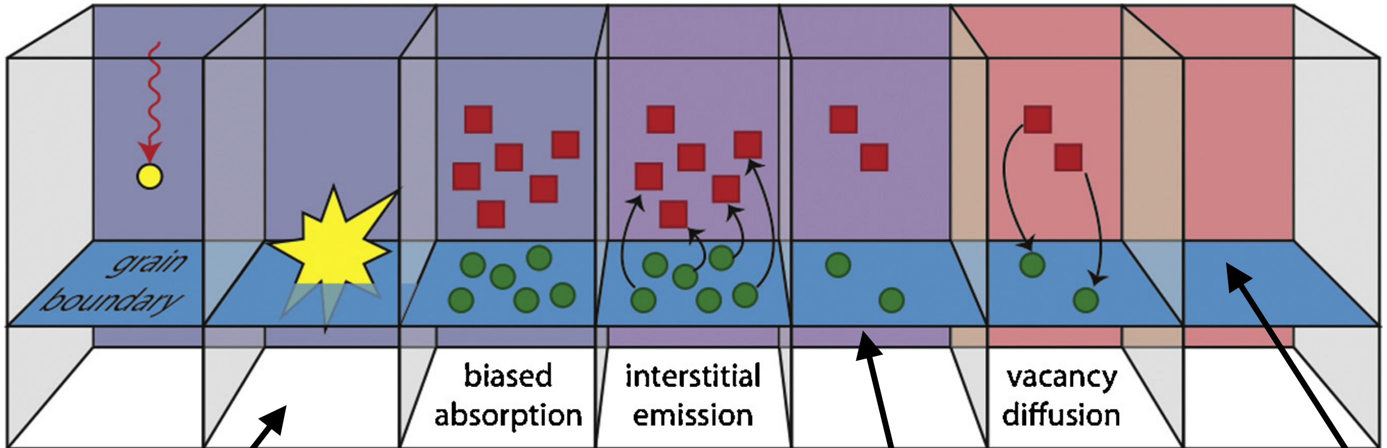
Tailoring properties at the mesoscale



S.Yip and M. Short, Nature 2013

- Dissipative structures opens a new approach to materials synthesis
- Self-healing and autonomous materials
  - Materials that freely can repair themselves after damage without any external influence
- Adaptive materials in harsh/extreme environments
  - Intrinsic properties that during exposure to a *defined* extreme environment the material performance is maintained or improved
- Multi-scale materials design from atomic to macro connected via the meso-scale

Grain-boundary mediated self-healing in nano crystalline metal systems



An energetic particle, such as a neutron, hits an atom in the material, giving it large kinetic energy

After the cascade settles in a few psec, point-defects (interstitials and vacancies) remain. The interstitials quickly diffuse to the GB

The trapped interstitials can re-emit from the GB into the bulk, annihilating the vacancies on time scales much faster than vacancy diffusion

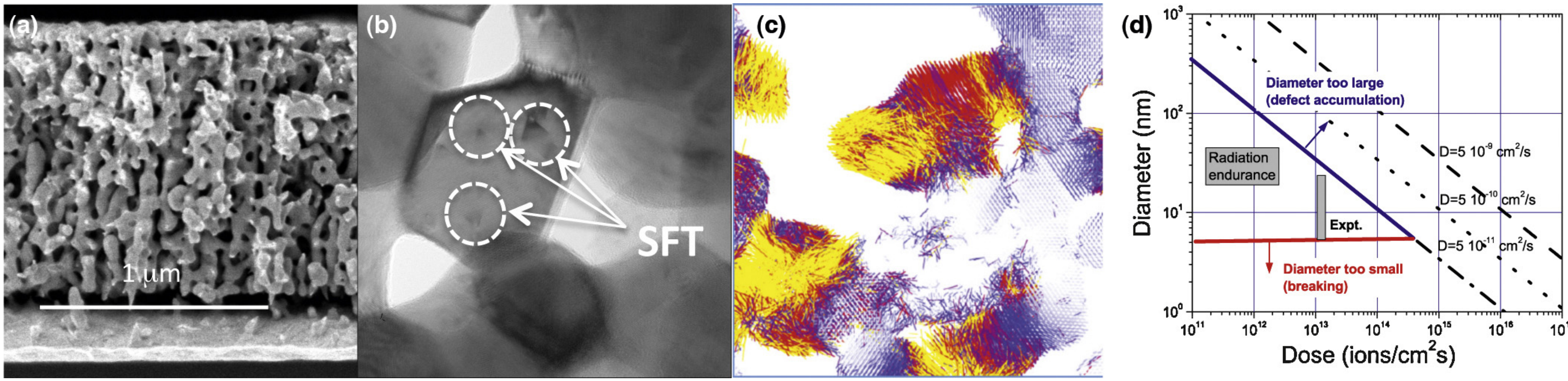
At longer time scales, the remaining vacancies diffuse to GB recombining with trapped interstitials completing the healing of the material.

The atom displaces many other atoms in its path, creating a collision cascade, which overlaps with the grain boundary

After the interstitial emission events have occurred, some vacancies out of reach persist and system reaches a relatively static situation

I.J. Beyerlein et al. Materials Today, 16 (2013) 443

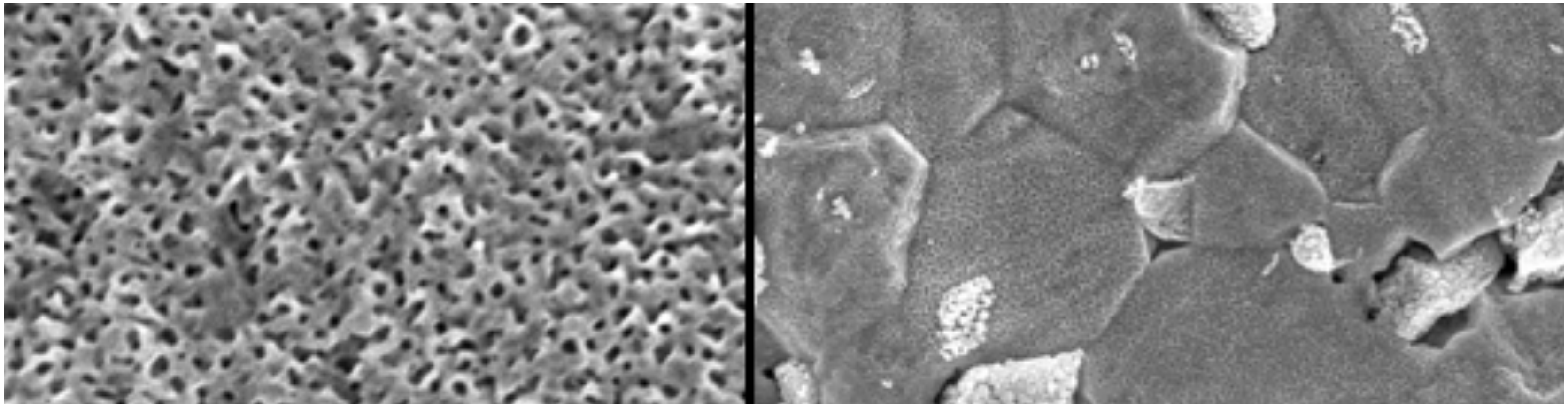
Designing radiation tolerant nanomaterials



- Manipulating the interface density and morphology can open up a materials design pathway for radiation-tolerant materials
- Programming for self-healing at the atomic scale by tailoring grain-boundary density and defect engineering for optimal function in the defined extreme environment
- How to tailor these properties? What fundamental understanding is necessary? Can you scale from the laboratory bench to industrial levels?

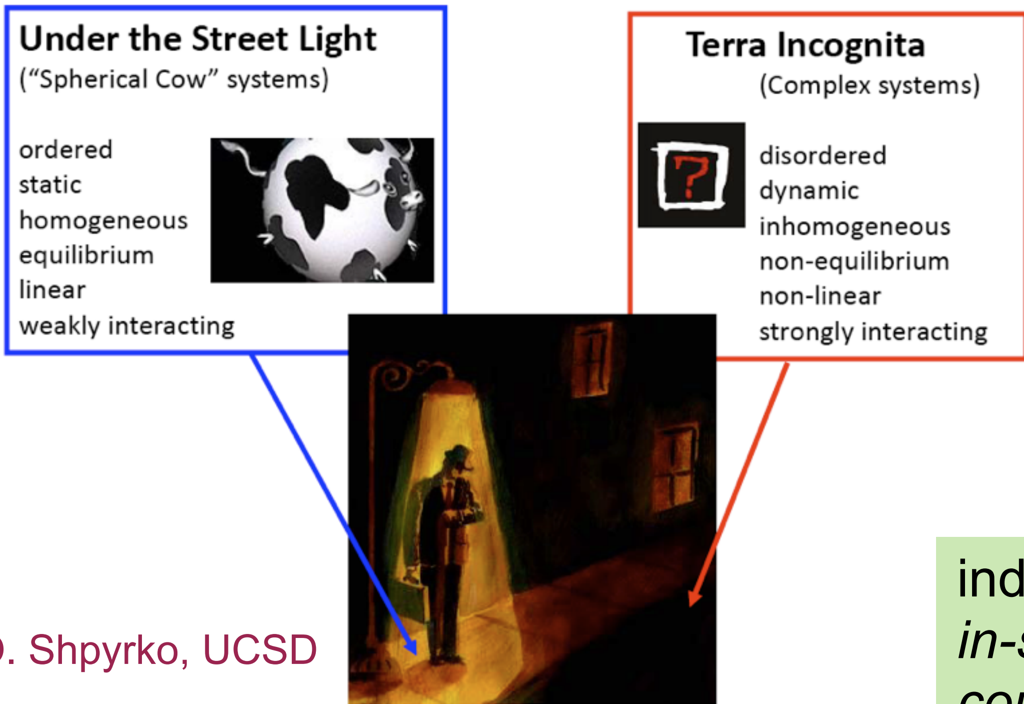
I.J. Beyerlein et al. Materials Today, 16 (2013) 443

Material interfaces in “real environments” in tokamak fusion devices

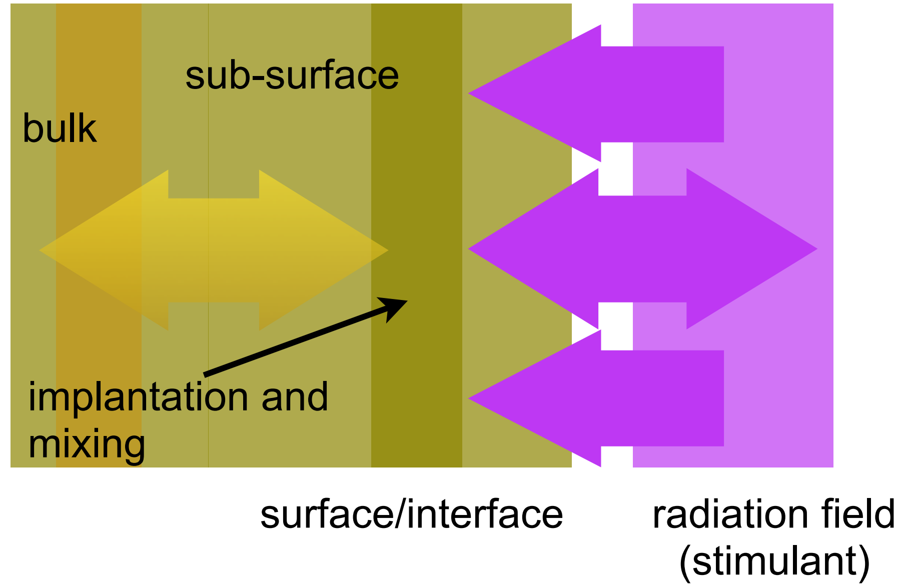


- Our knowledge of the *dynamic coupling* of fusion plasma and its interface with the solid wall surface is primitive
- The *spatio-temporal evolution* of that surface is even less understood
- Traditionally limited to post-irradiation diagnosis of events that are inherently dynamic and synergistic
- In addition, today there is <u>no computational model</u> that can predict the morphological and topographical evolution of the plasma-material interface under synergistic conditions in the fusion edge

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



O. Shpyrko, UCSD



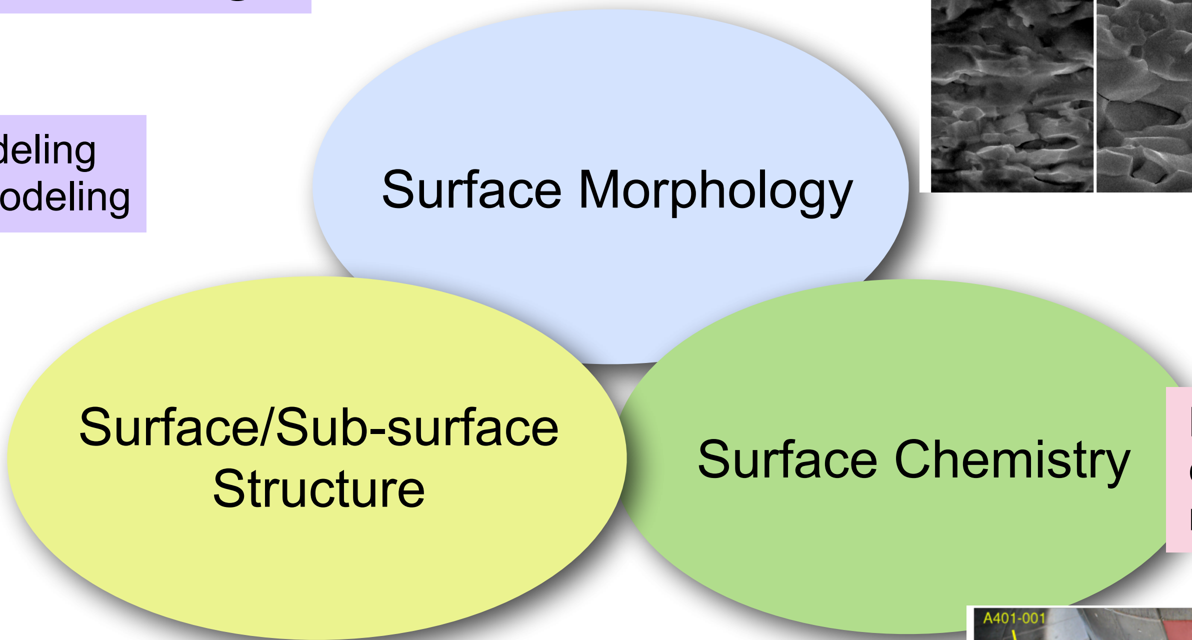
indicates the need for complementary *in-situ* and *in-vivo* diagnosis to study *coupling of irradiation-driven interface*

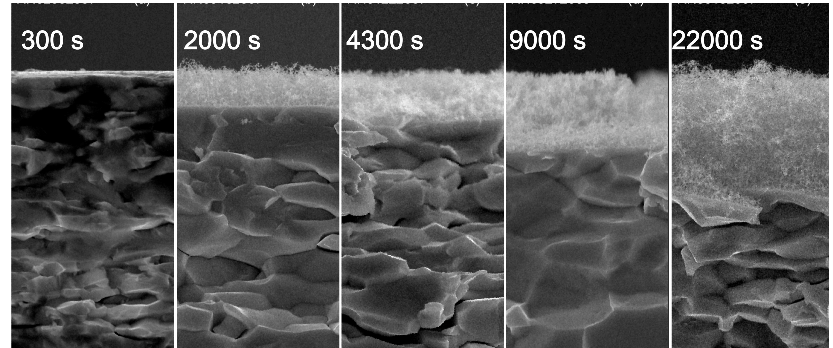
- Need for dynamic measurement of surfaces under controlled irradiation fields
- Indicates the need for complementary in-situ and in-vivo diagnosis to study *coupling of irradiation-driven interface*
- Materials design: processing, properties, function *and dynamic performance*

Plasma-material coupling is a multi-dimensional problem: need for comprehensive and coordinated modeling and experiments

neutron damage

atomistic modeling
multi-scale modeling

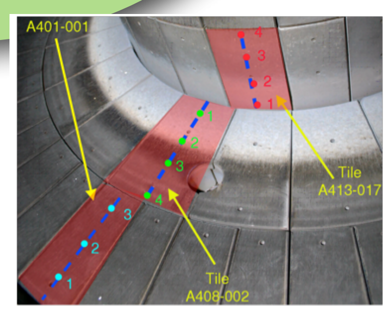


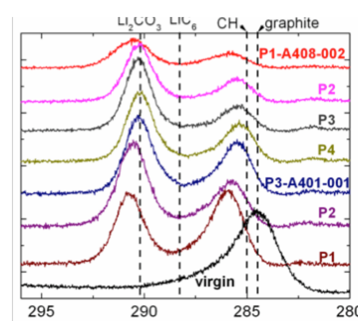


Baldwin and Doerner, 2008

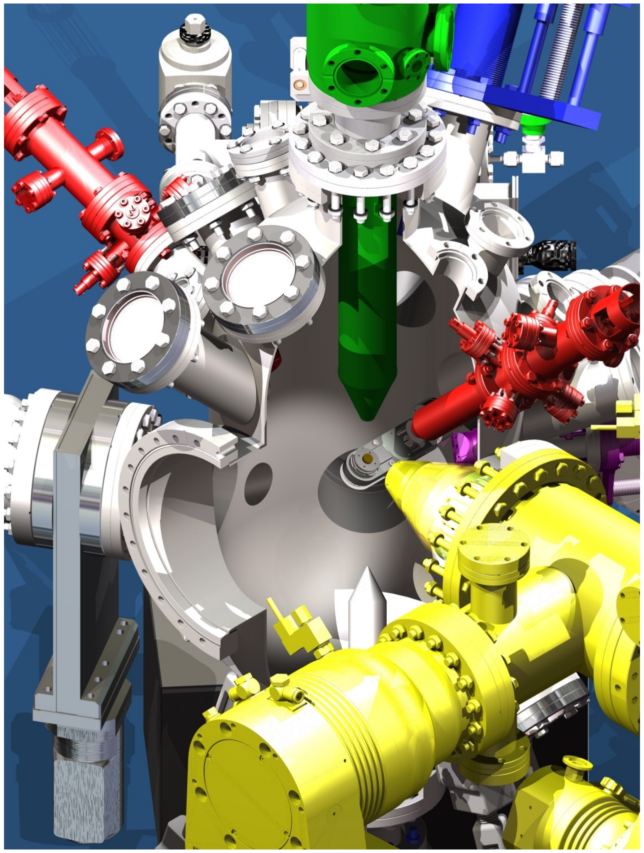
PMI models are limited and cannot predict complete material behavior

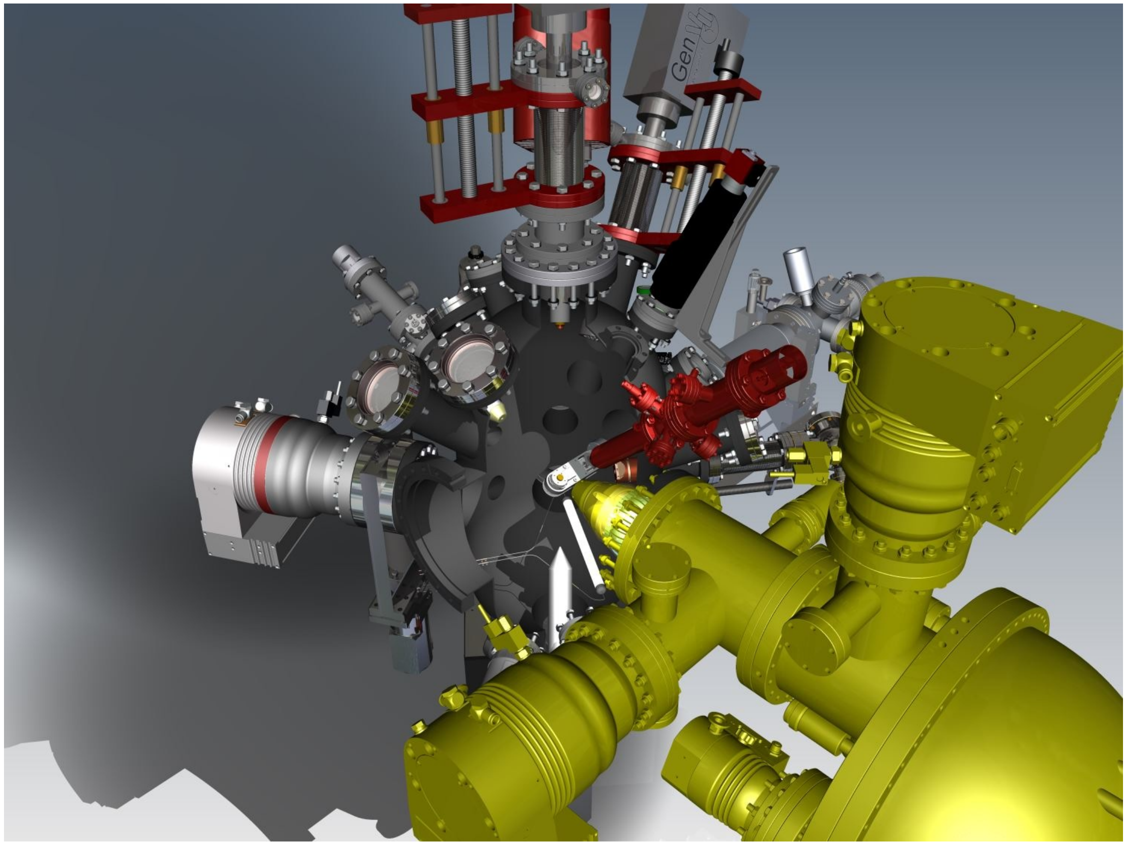
Combination of theory/modeling with well-diagnosed experiments is *key* to understanding and predicting behavior in nuclear fusion plasma edge





Multiple-beams and plasma-induced nanostructuring of a variety of materials with in-situ characterization





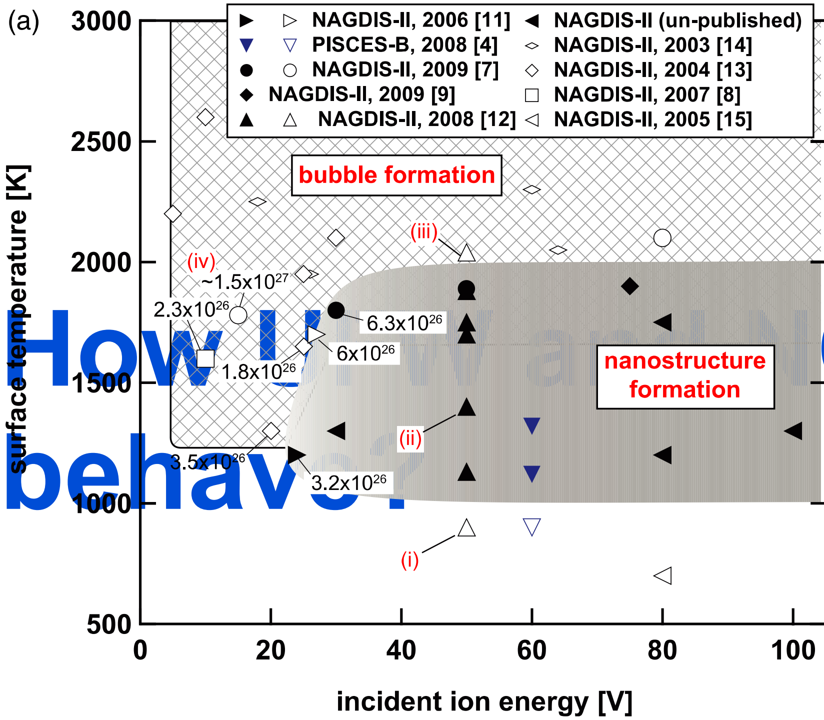
- IGNIS will be a state-of-the art facility enabling the diagnosis of surfaces *during* plasma irradiation at high pressures (from UHV to mTorr and up to 10 Torr)
- Correlating plasma-induced nanostructuring and surface composition on gas-covered surfaces probed *during* plasma exposure and connecting parameters of the modifier source (e.g. plasma, ions, etc..) to the surface nanostructures synthesized

Allain et al. University of Illinois

Nanostructuring of W by He irradiation

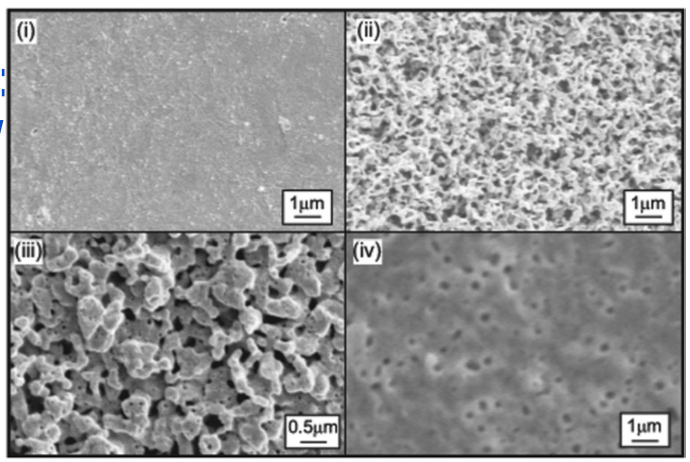
How U… CW

behav…?



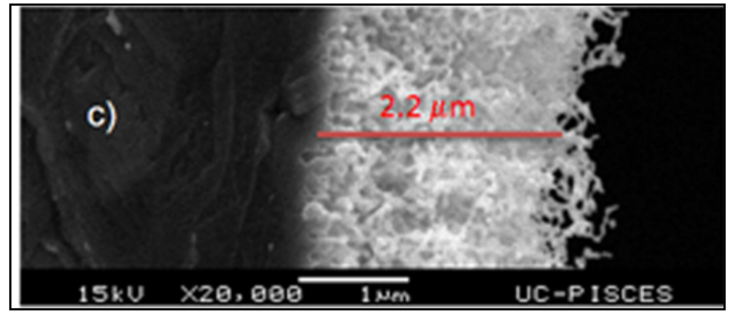
UFW:

NCW

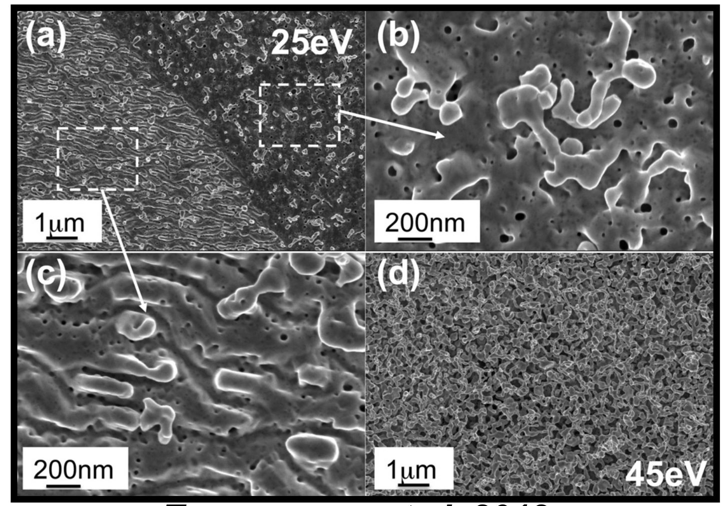


S. Kajita et al. Nuclear Fusion, 49 (2009) 095005

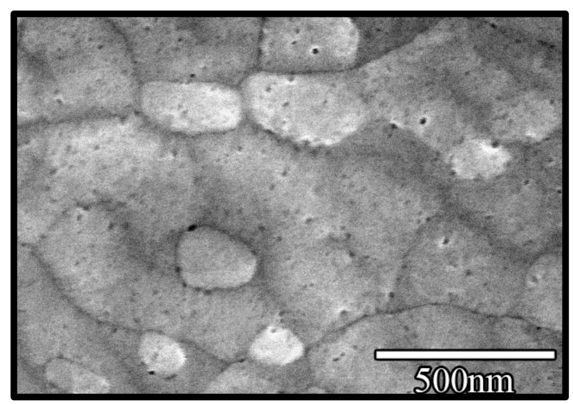
M.J. Baldwin et al. Nuclear Fusion 48 (2008) 035001



Doerner et al. 2011

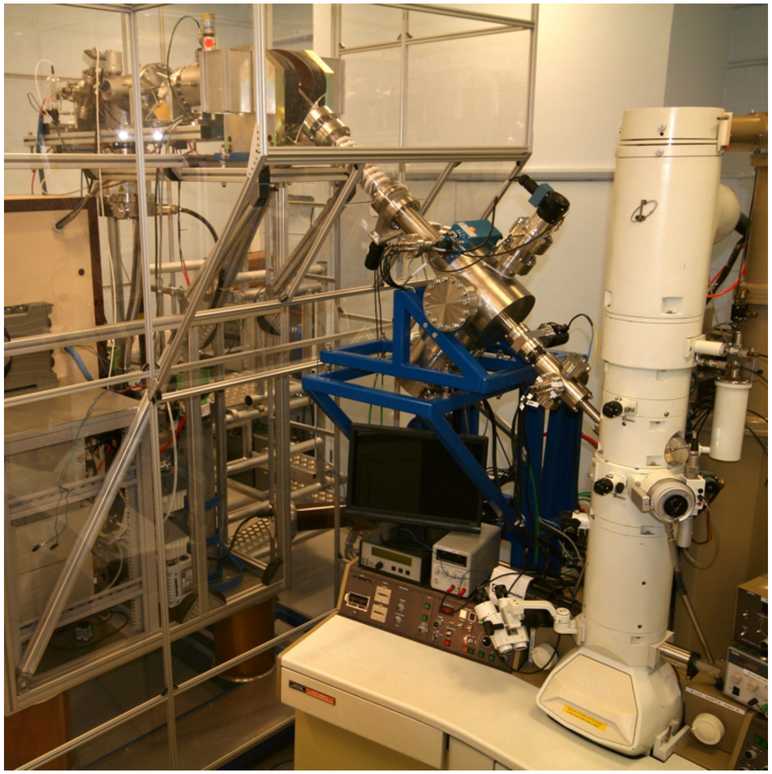


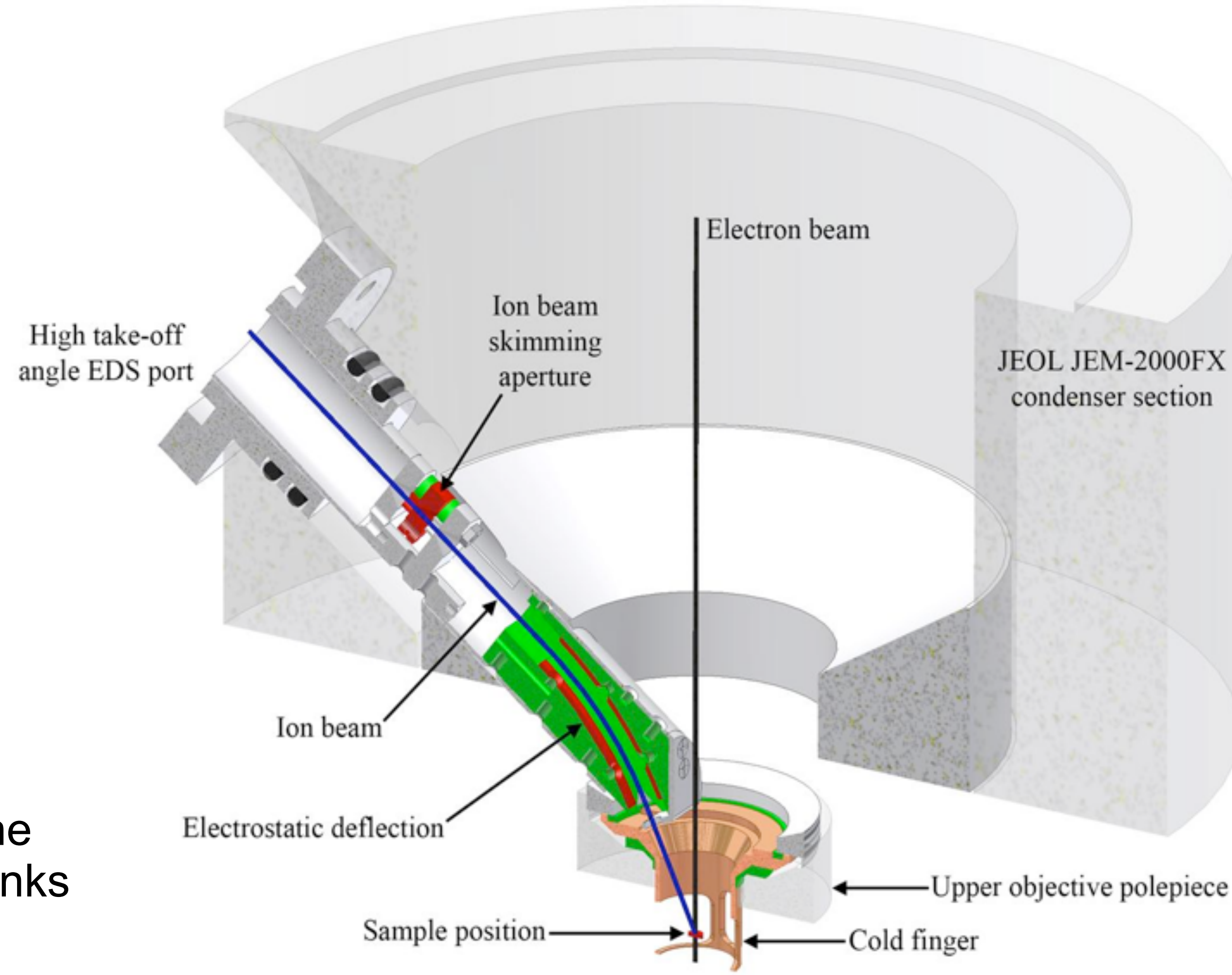
Temmerman et al. 2012



O El-Atwani et al. JNM 2012

Observing defect dynamics with in-situ TEM can elucidate effects: couple spatial scale with models





- Irradiation was performed using the MIAMI facility with Dr Jonathan Hinks and Prof. Stephen Donnelly at the University of Huddersfield.
- Fluences used were multiples of 4×10^{18} m^{-2} with 2-keV He+ ions at 950 ^{0}C

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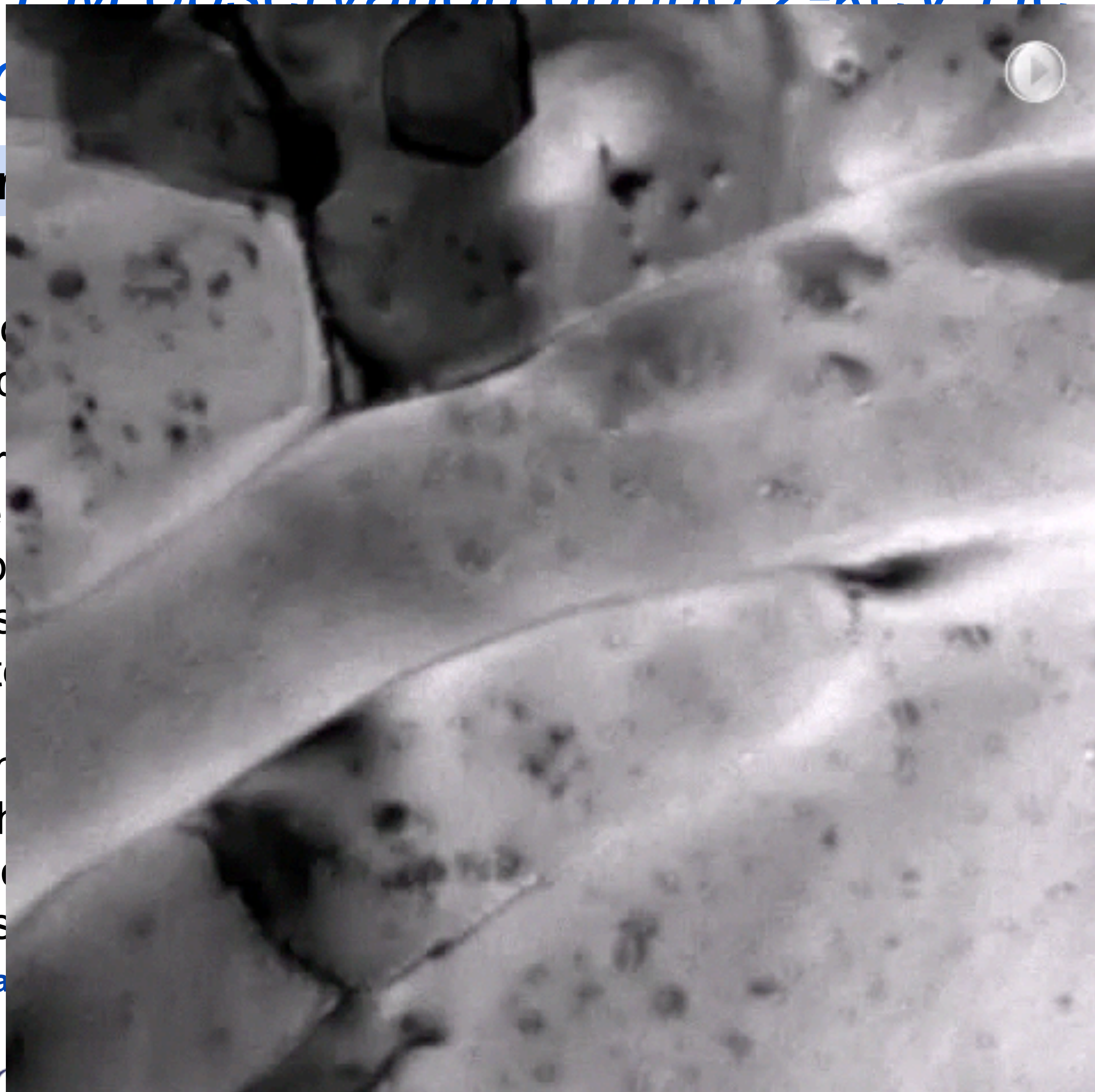
low fluence regime

University of HUDDERSFIELD

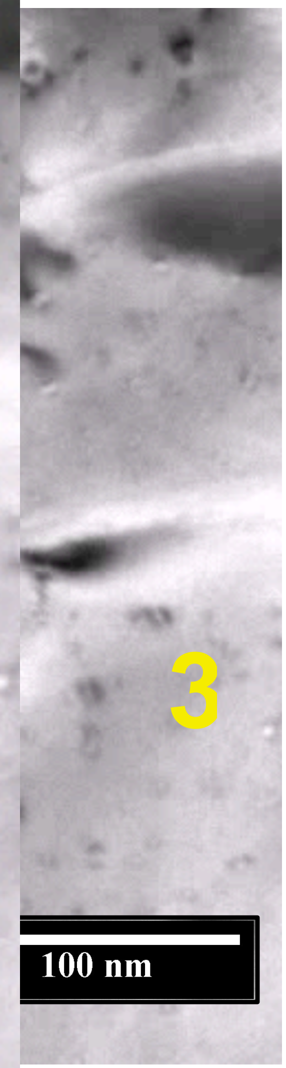
In-situ TEM observation during 2-keV He^+ irradiati



Fluence fr **adiation**

- No defect
  observed
  Only found
- The larger
  higher the
  density ob
  interstitials
  NC tungst
- Dislocation
  and growth
  indication
  complexes

(H. Iwakiri et a



O. El-Atwani et al, To be submitted 2013

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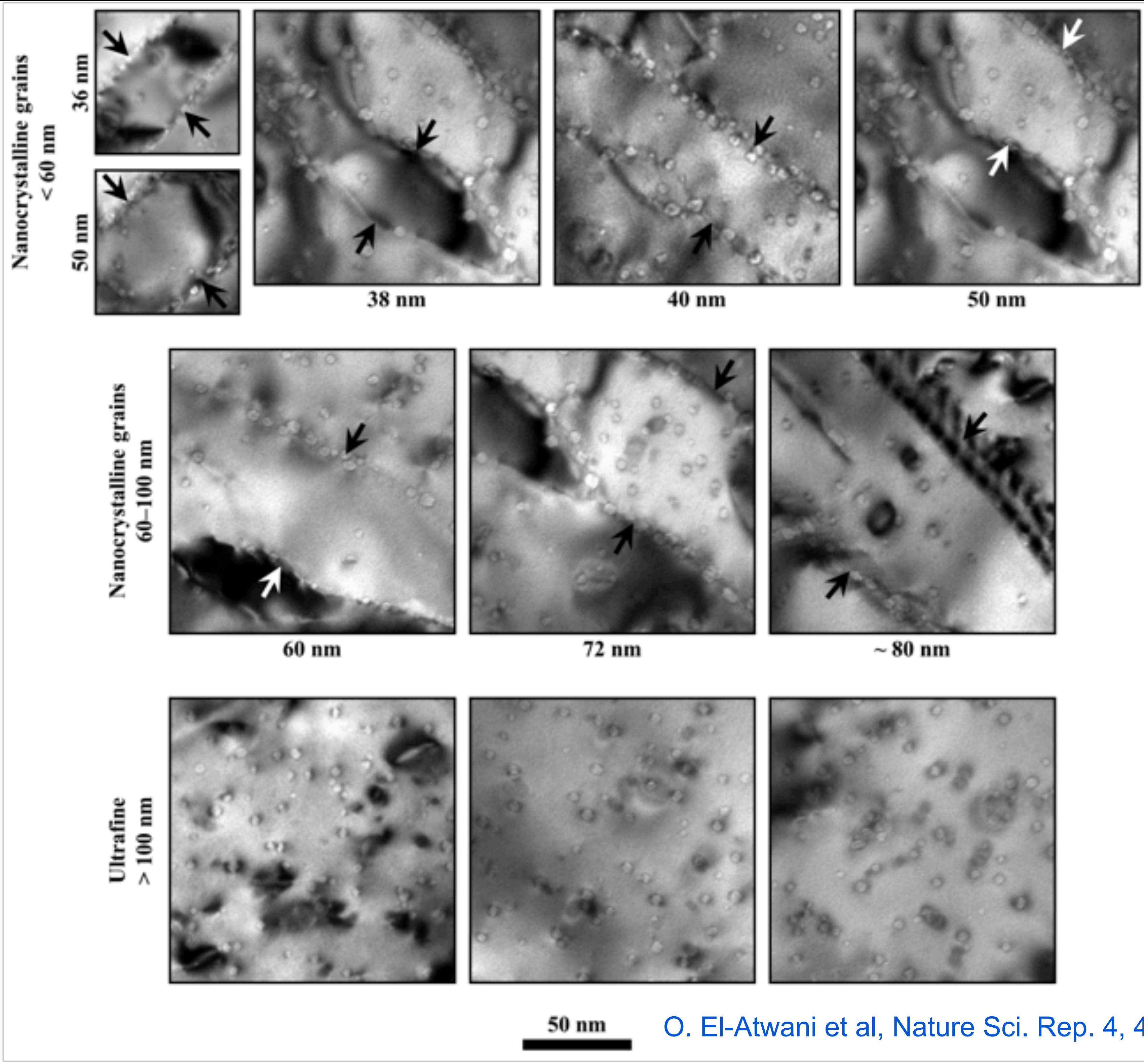
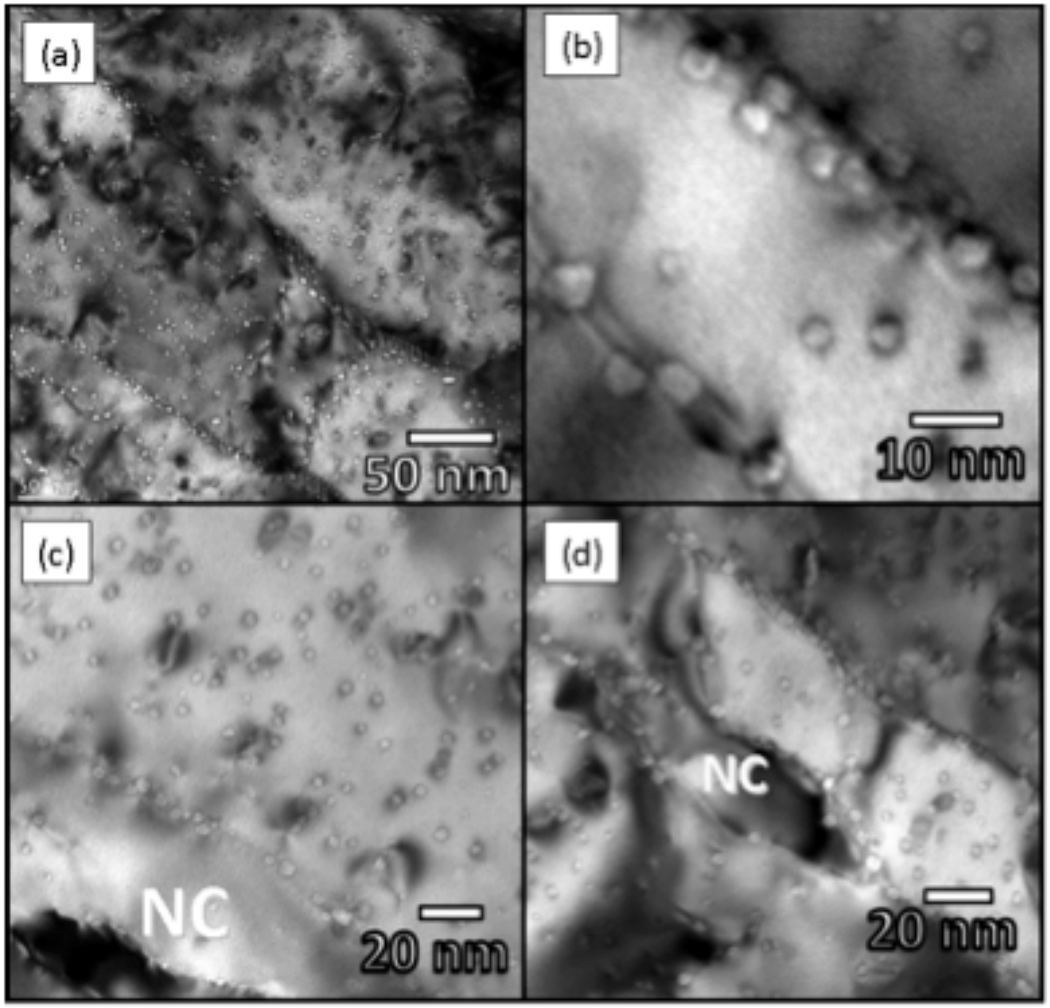


O. El-Atwani et al, Nature Sci. Rep. 4, 4716 (2014)

Summary of the in-situ TEM studies

- Grain boundaries are He sinks (large bubbles on the grain boundaries).
- Intra-granular bubble and defect formation in relatively large grains (e.g. > 60-nm)
- Grains of less than ~ 60-nm in size* yielded a 50% lower areal bubble density compared to larger grains (60-100 nm) and ultrafine grains (100-300 nm). Defect clusters were not observed on those grains.
- Bubbles on grain boundaries were faceted (high He concentration)



O. El-Atwani et al, To be submitted 2013

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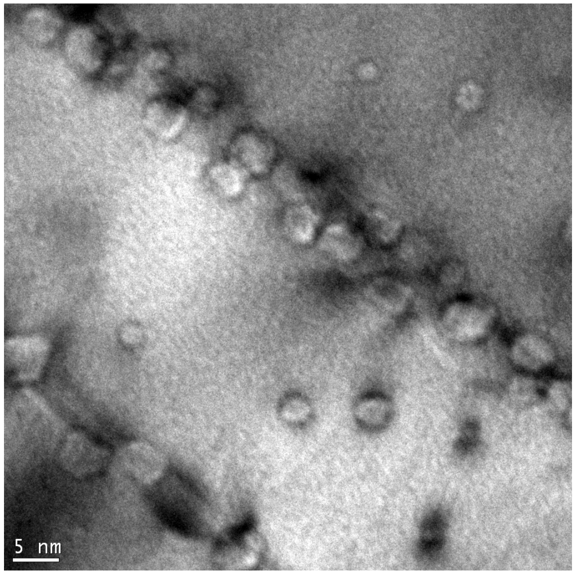
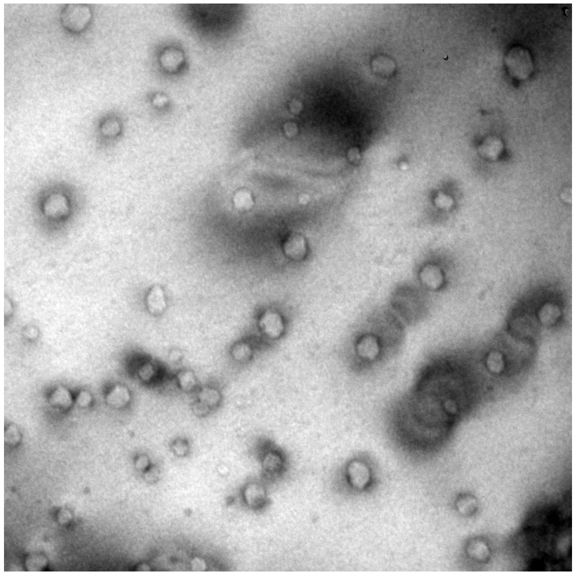
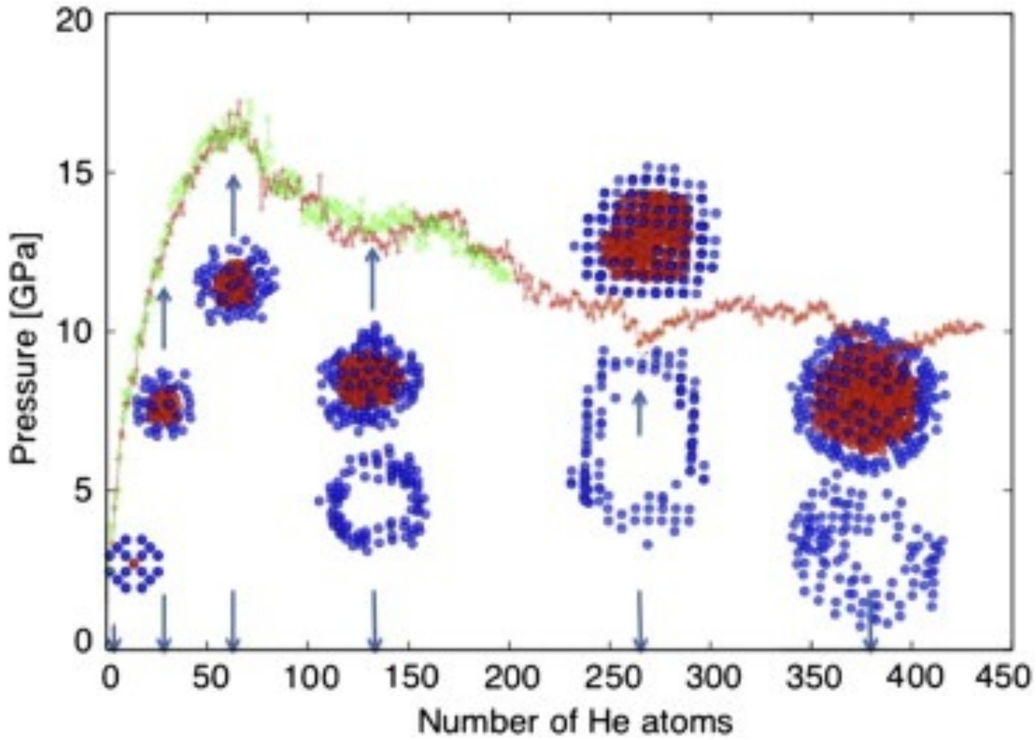
*size is a characteristic length defined by shortest distance between grain boundaries

He bubble faceting and interstitial dislocation loops in irradiated tungsten





Allain and El-Atwani et al. 2014 unpublished

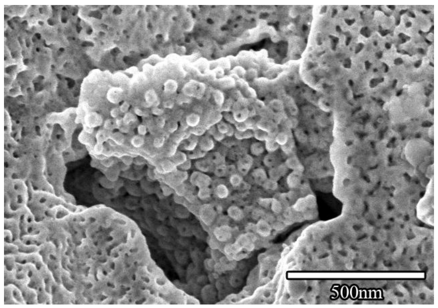


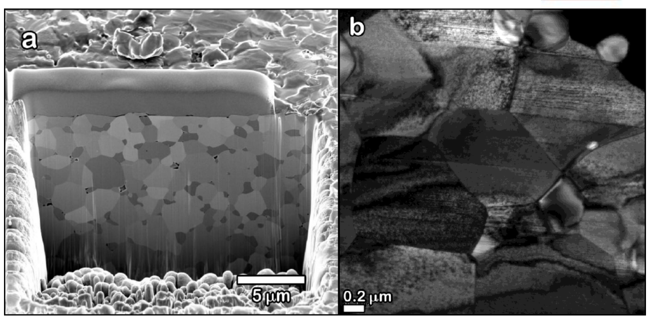
Pressure versus number of He atoms in a bubble seeded with an initial cavity of 27 vacancies in Fe (red + curve) and in Fe-10 at% Cr (green × curve). Snapshots at 0, 27, and 60 He atoms show He atoms in red and distorted Fe atoms in blue. Snapshots at 140, 260, and 375 He atoms show the moment of emission of interstitial dislocation loops. At 260 He atoms, faceting is clearly seen.

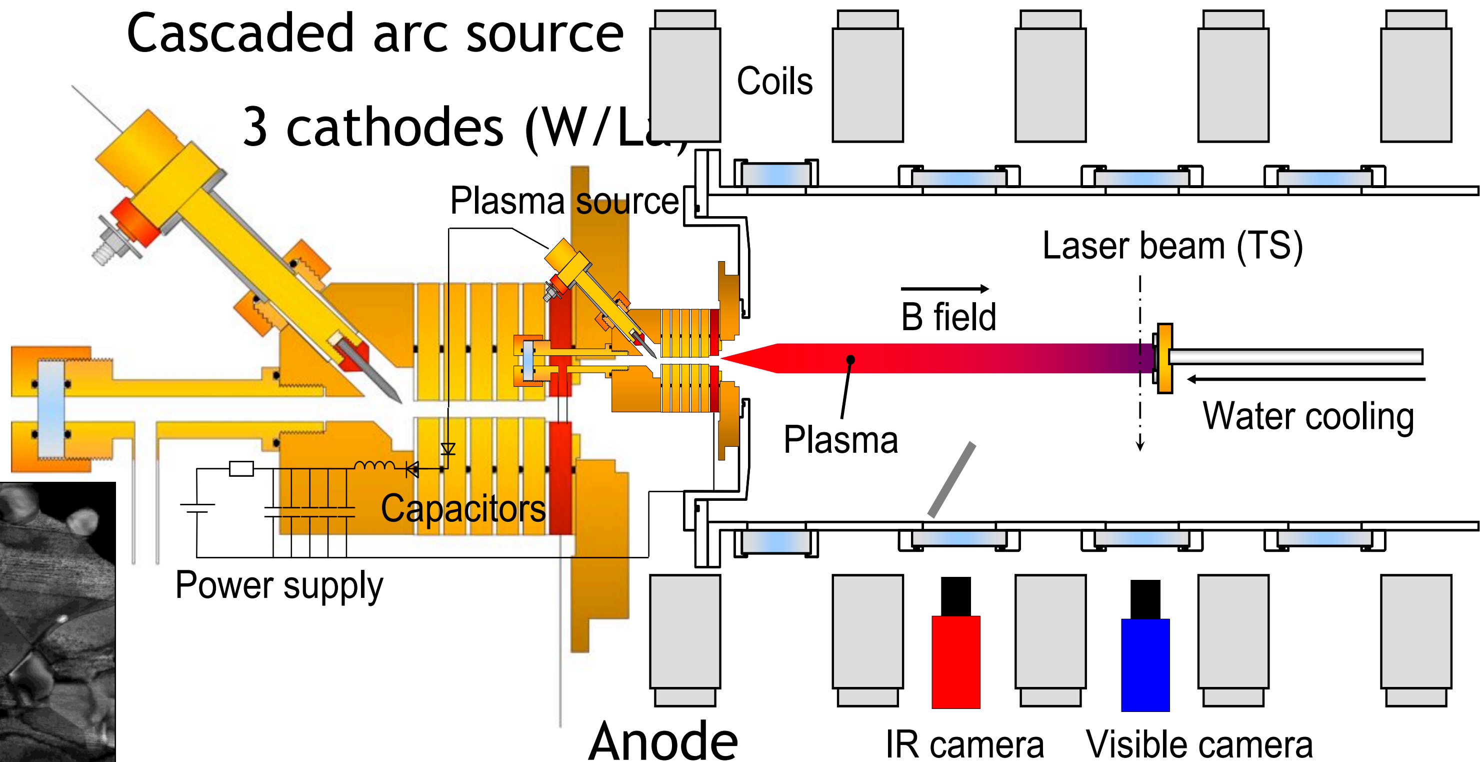
A. Caro et al, J. Nucl. Mater. 418 (2011) 261

Collaboration with FOM-DIFFER (Dutch Institute for Fundamental Energy Research)

DIFFER
Dutch Institute for Fundamental Energy Research



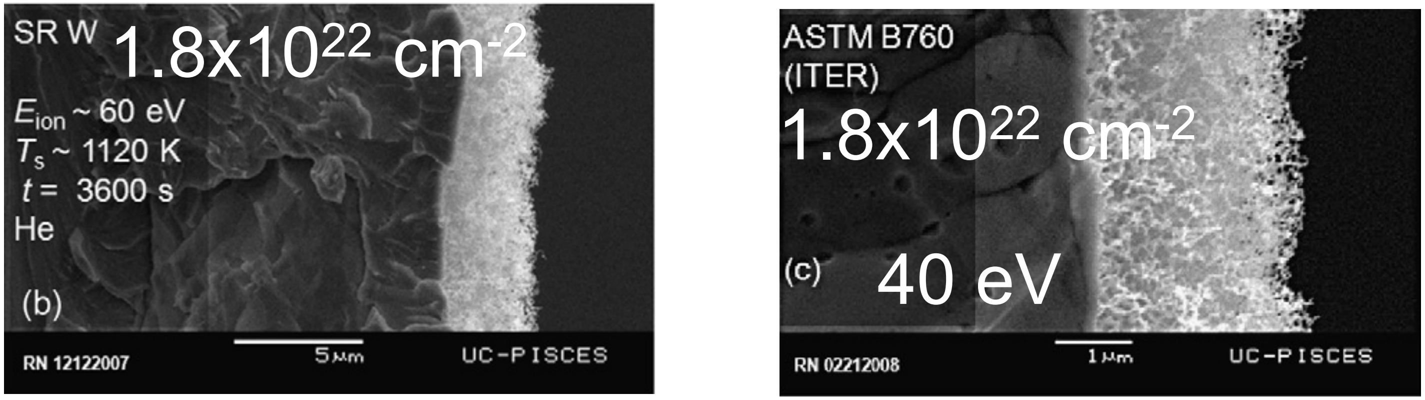




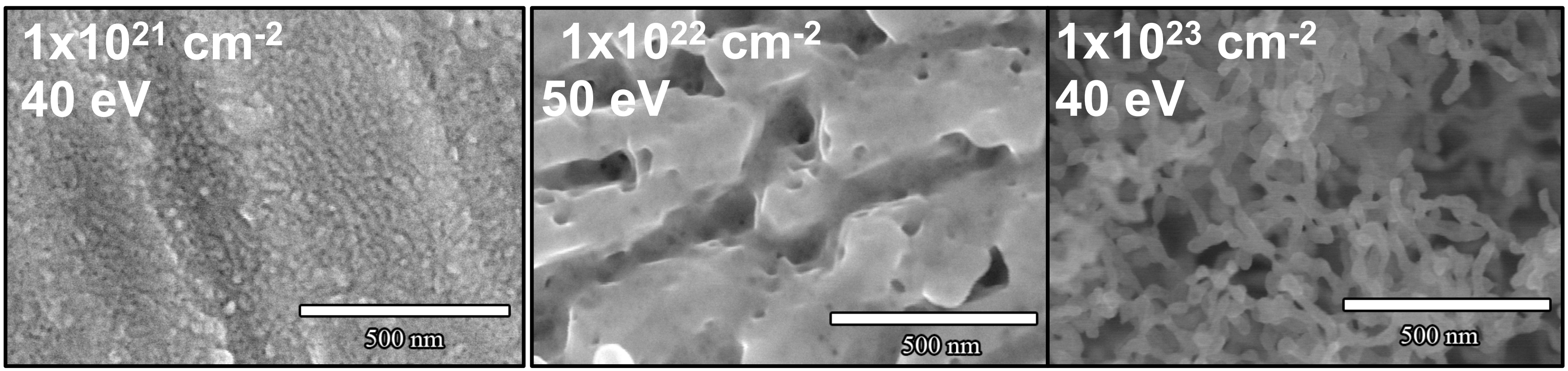
- Three lines of research: **1) high-flux irradiation (quiescent + transient) of nano-structured W and Mo, 2) Surface morphology and chemistry of nano-structured Low Z coatings on W and Mo, 3) temperature-dependent erosion studies of nanostructured W and Mo**

Collaboration with G. De Temmerman, DIFFER

Comparison with literature (fuzz formation)



Baldwin et al, JNM, 2010



SPD samples (UFG) have higher fuzz formation fluence thresholds, however fuzz thickness growth rate is faster

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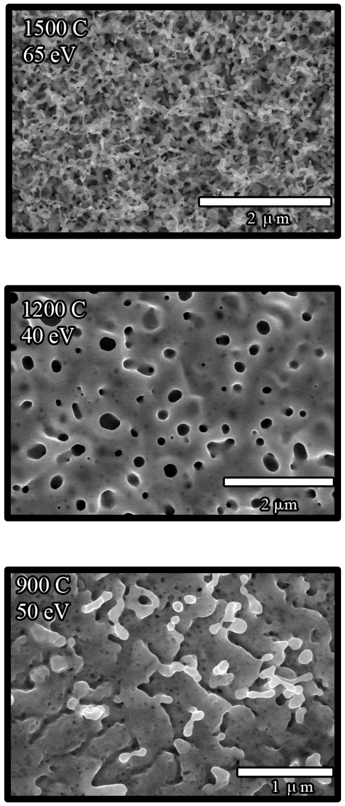
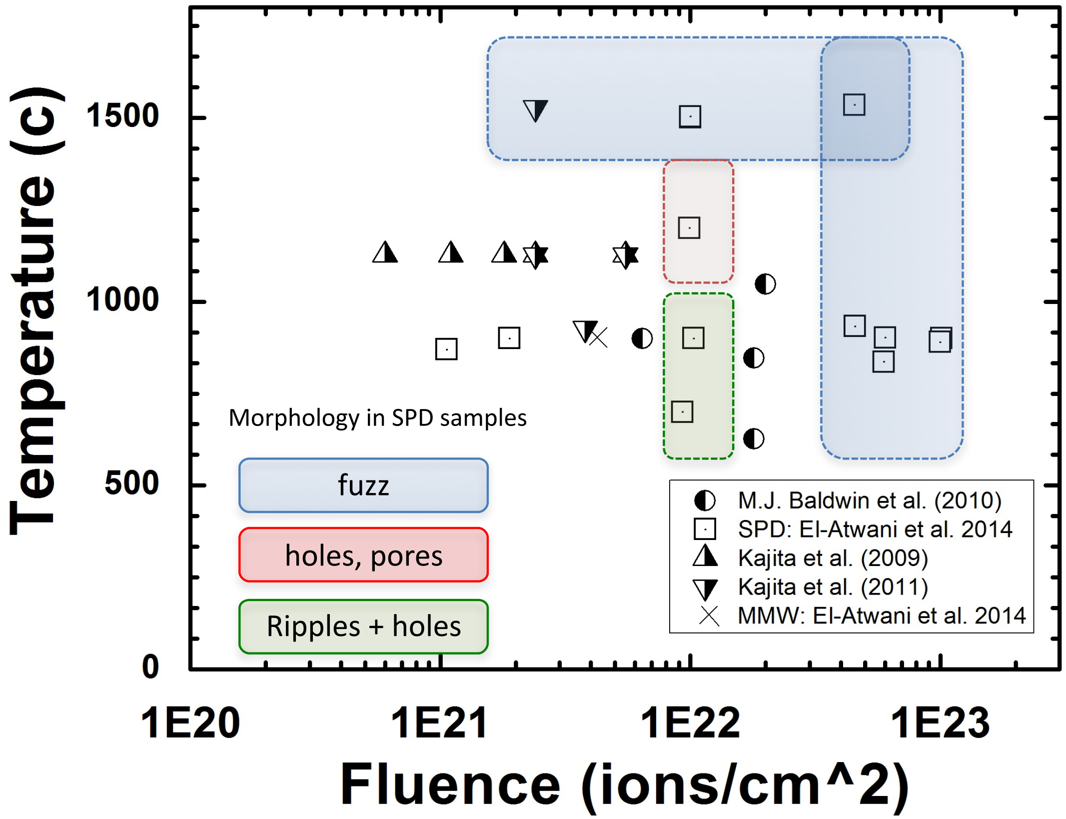
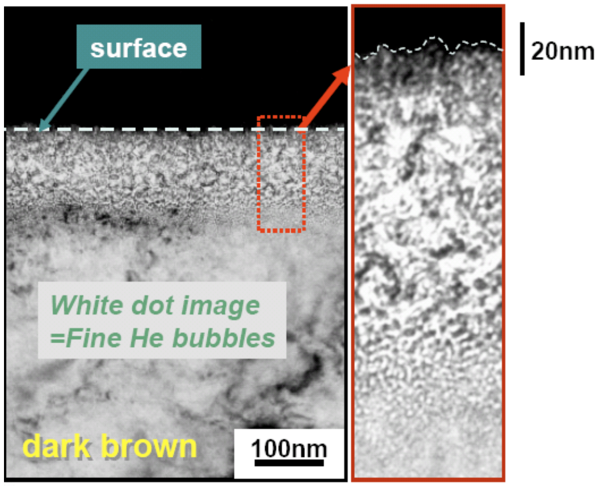
O. El-Atwani et al, Nucl. Fusion 54 (2014) 083013

Rich surface morphology driven by irradiation-induced instabilities at the plasma-material interface



Fluence = 10^{22} cm^{-2}



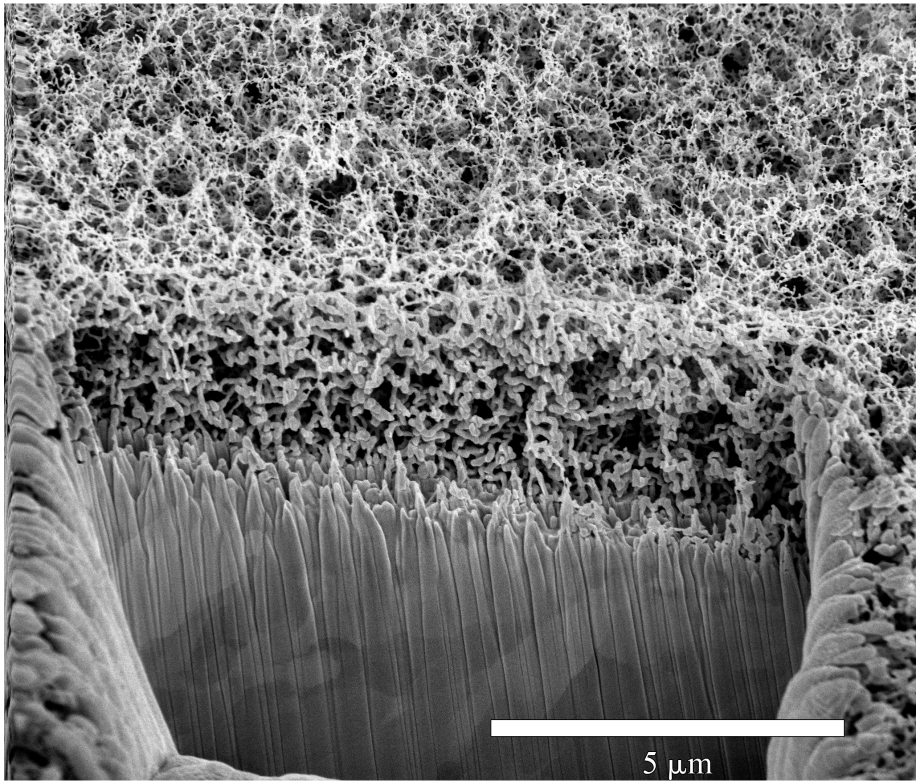


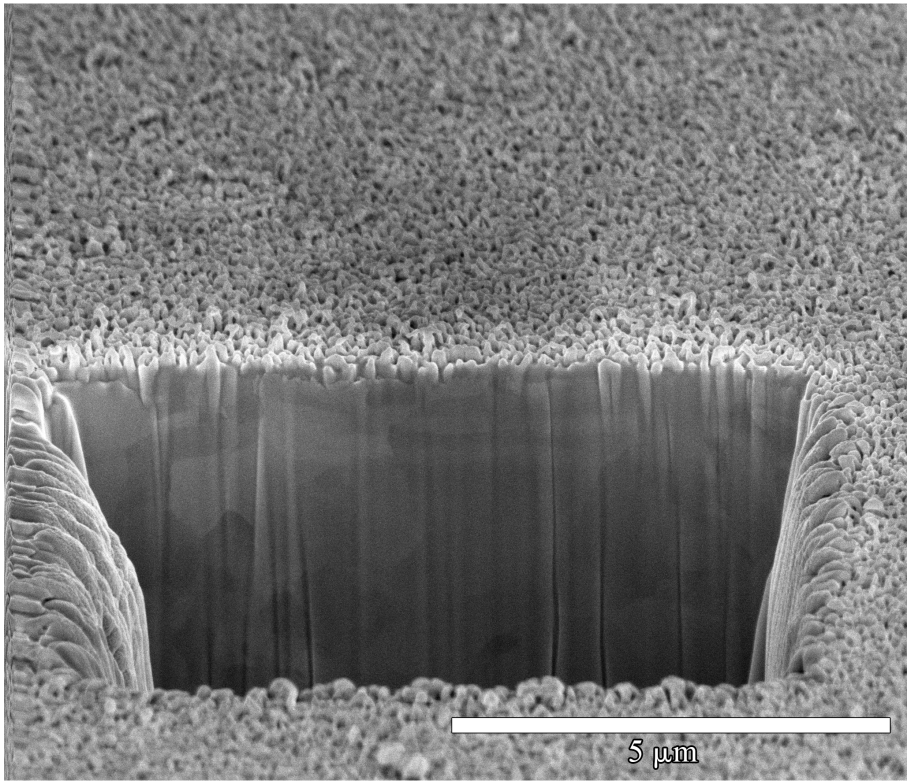
Yoshida et al. 2005

O. El-Atwani et al, Nucl. Fusion 54 (2014) 083013

- Both system temperature of W and irradiation fluence of He particles can generate a complex morphology on the surface that is limited to penetrating depth of about 100-nm. This interface in the plasma can influence H retention and erosion

Understanding the role of grain-boundary density on the mitigation of He-induced damage in UFG tungsten

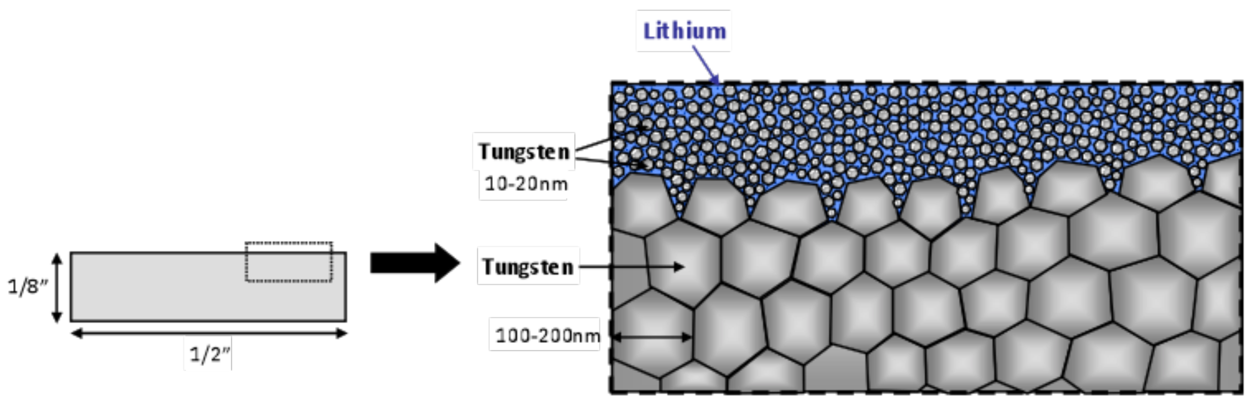


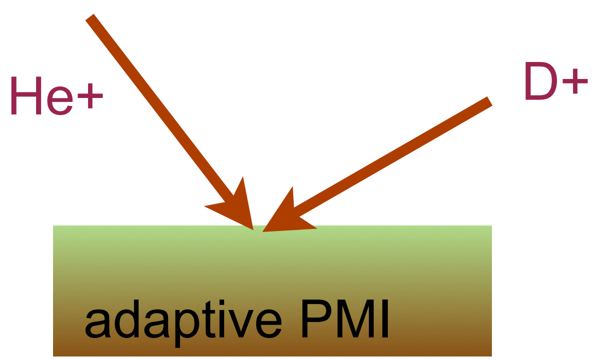


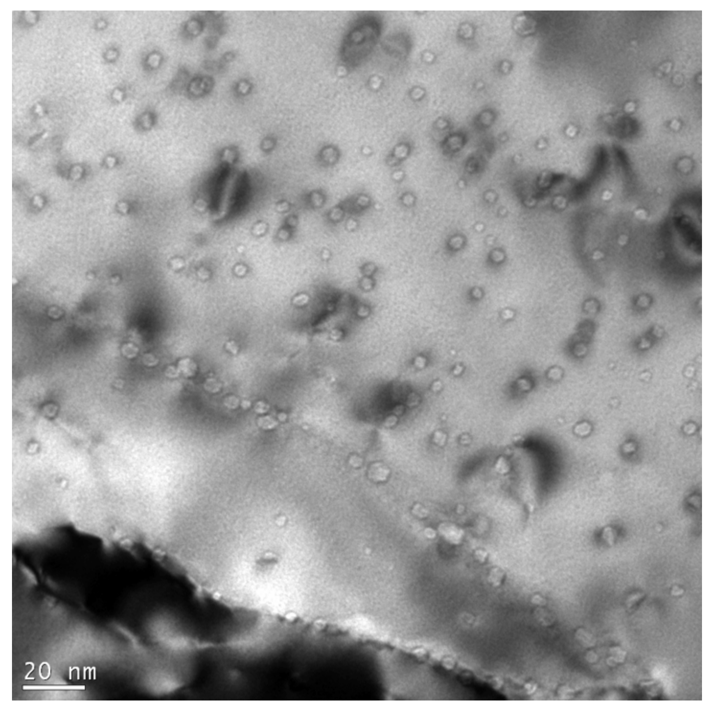
- Significant reduction in sponge-like structure (tungsten fuzz: from He-bubble growth)
- Reduction correlated to large grain-boundary density in UFG and NC-W
- However, there remains structural damage to surface. Can we design a robust self-healing system responsive to the aggressive nuclear fusion environment?

Outlook: adaptive nanocrystalline and nano-composite refractory-metal PFCs

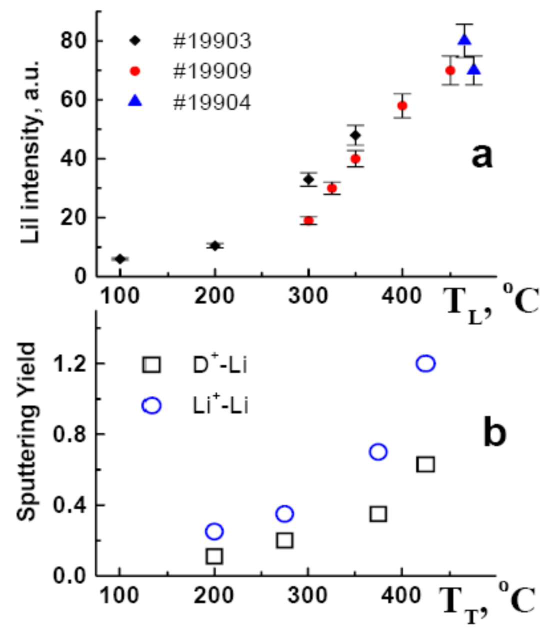
- We are deciphering process, properties, function and dynamic performance of nanocrystalline W coupled to liquid-metals for adaptive and self-healing functions
- In-situ irradiation-driven mechanisms on complex materials (liquid/solid interfaces)
- Extreme environment testing at collaborator facilities (e.g. DIFFER, etc...)
- Linking low-Z to high-Z solutions for PMI performance: D/He irradiation, recycling, permeation (e.g. T with INL), links to computational MS (Wirth)

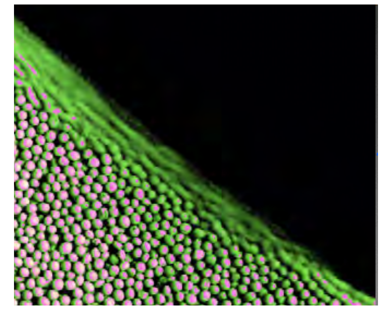






Coupling liquid thin-films and porous refractory metals: Multi-phase, multi-scale prompt response





- Free surface liquids and their response to low-energy irradiation, still remain less understood in the "thermal sputtering" regime*
- More critical is the interface between the free surface liquid and its material substrate under irradiation. Behavior at nano/micro scale of liquid percolation and its effect on meso/macro scale

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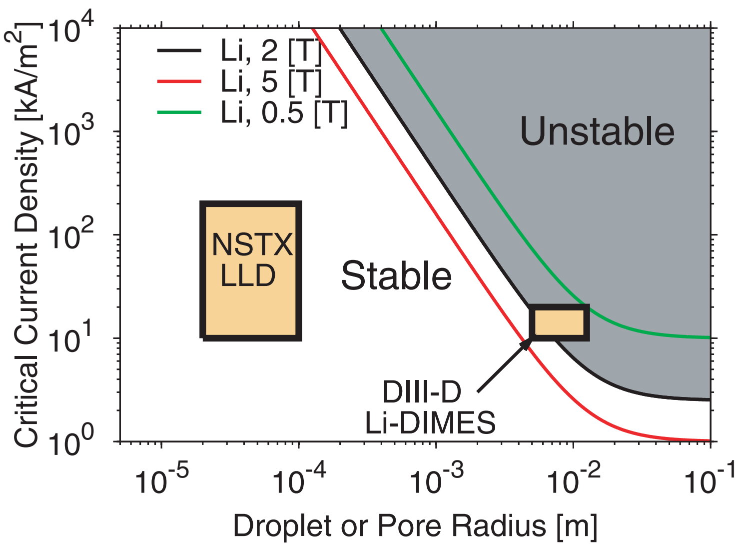
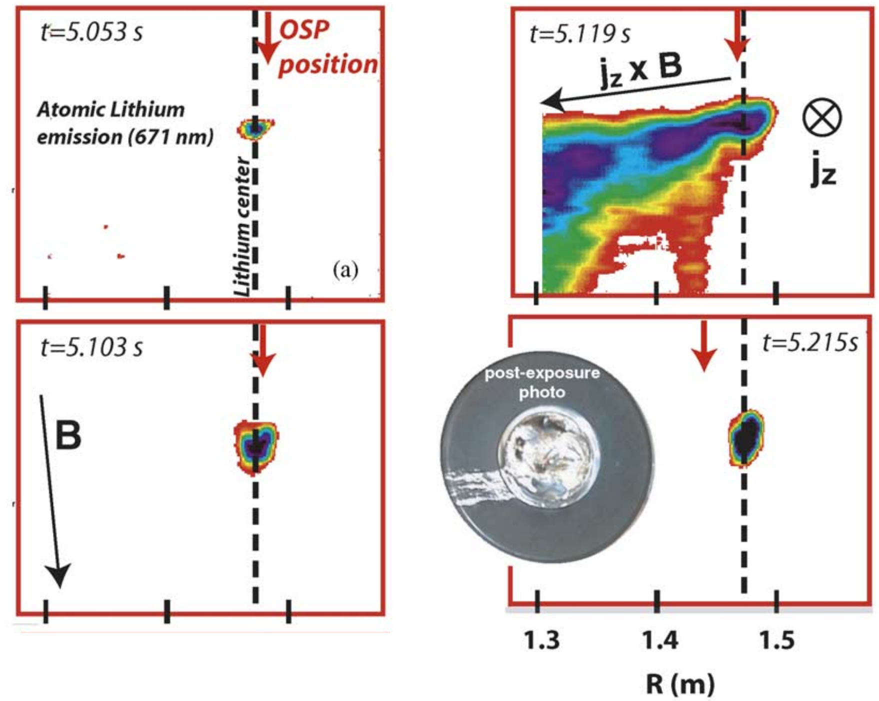
*thermal sputtering: enhanced non-linear erosion beyond melting point of liquid under particle irradiation

Heim and Allain, 2012, NIMB

Stabilizing free-surface liquid layers



M.A. Jaworski et al. Nucl. Fusion 53 (2013) 083032

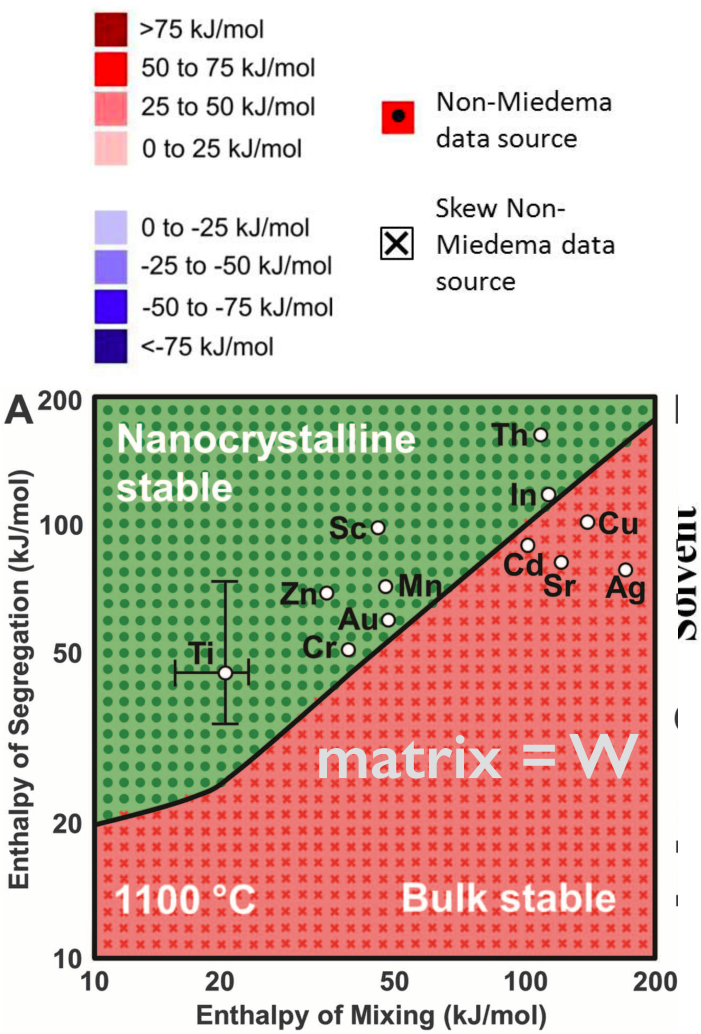


D.G. Whyte et al. Fusion Engr Design 72 (2004) 133

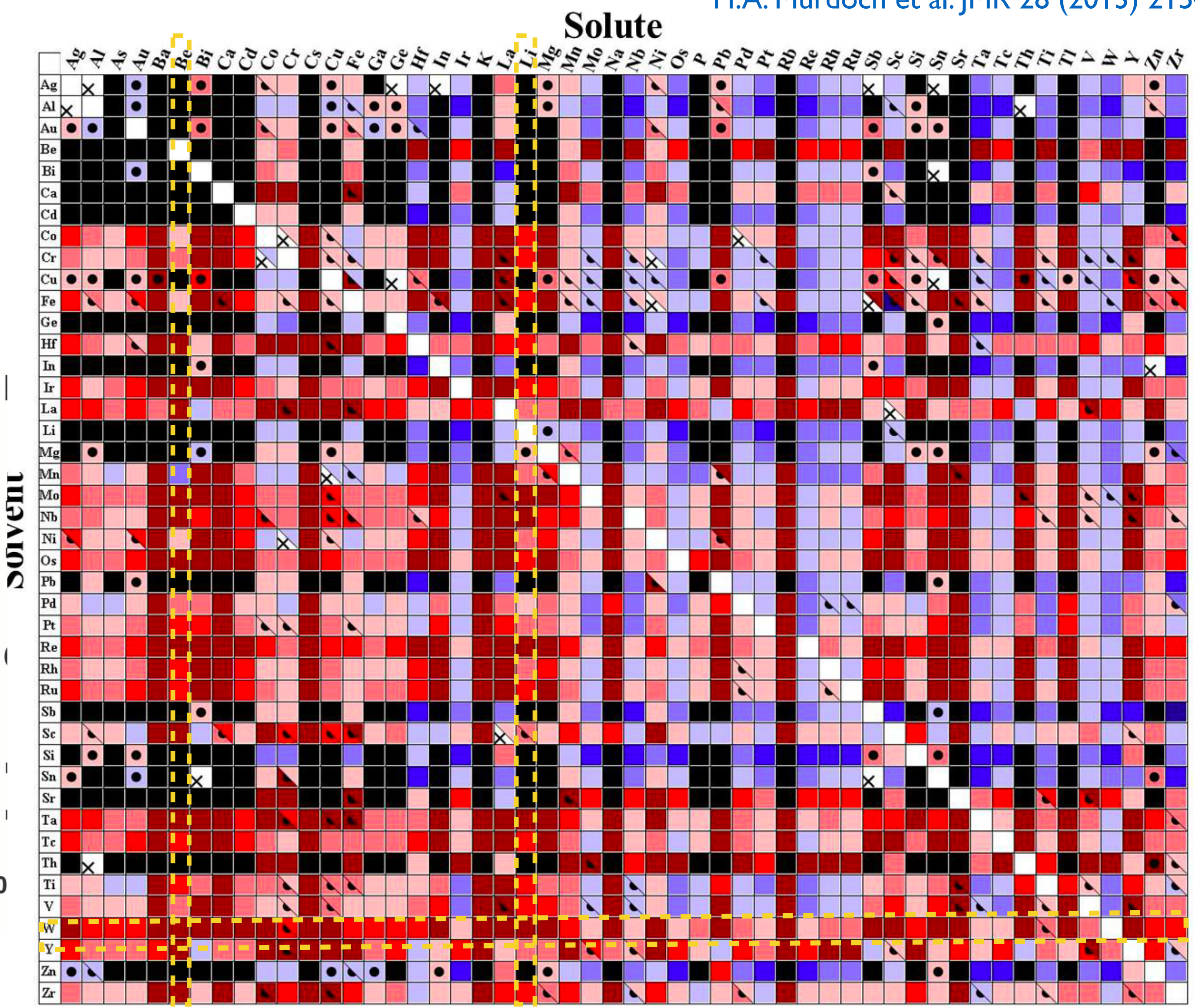
- LLD demonstrated two important results in the context of plasma-liquid interactions:
  - Mo influx disappears once lithium melting pt is reached during MHD events
  - No *macroscopic* amounts of lithium were injected from the LLD campaign
- As indicated by Jaworski et al. *"This usage of a porous substrate for stabilization of a free-surface liquid metal has been demonstrated in a diverted tokamak"*

Designing advanced nano crystalline alloys

H.A. Murdoch et al. JMR 28 (2013) 2154

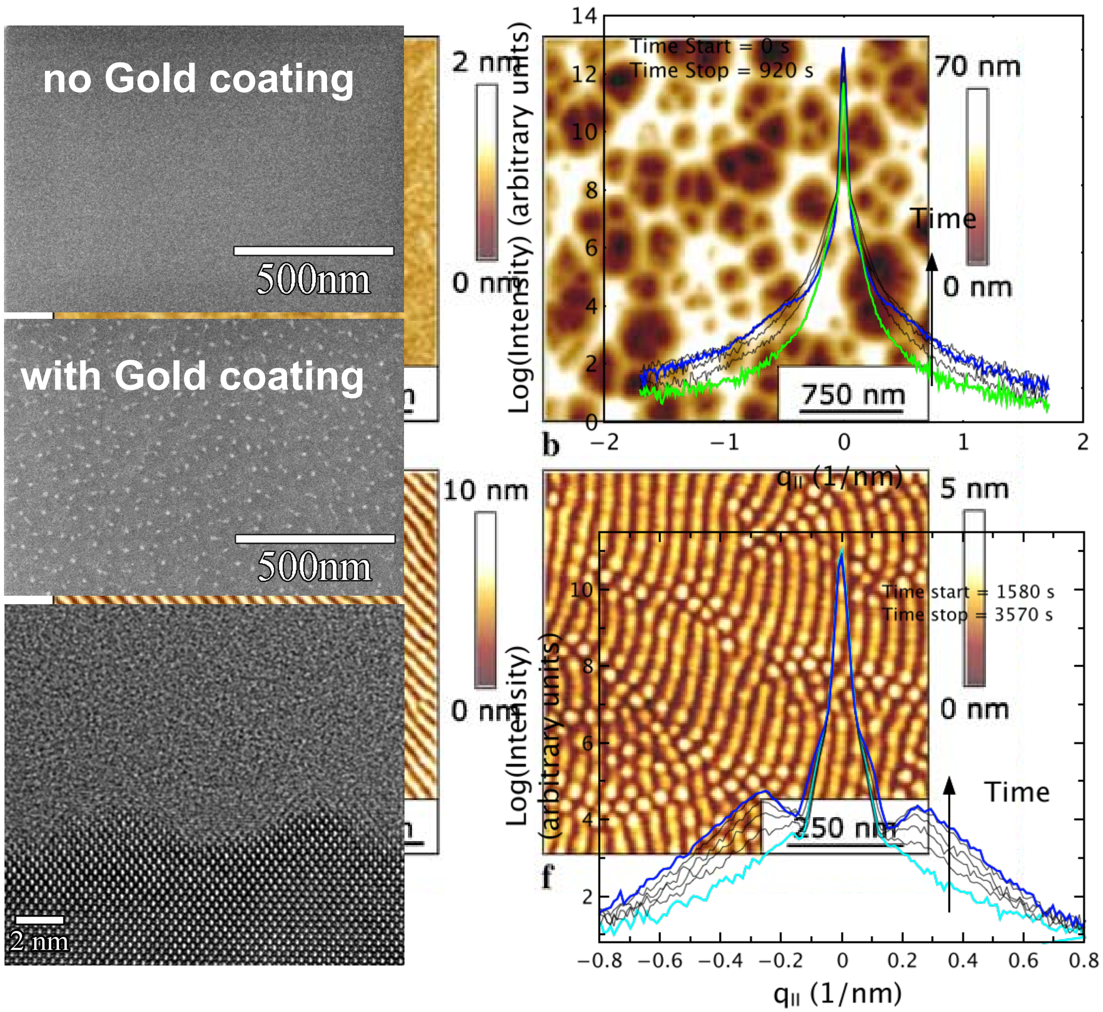


Stability map for nano crystalline W alloys

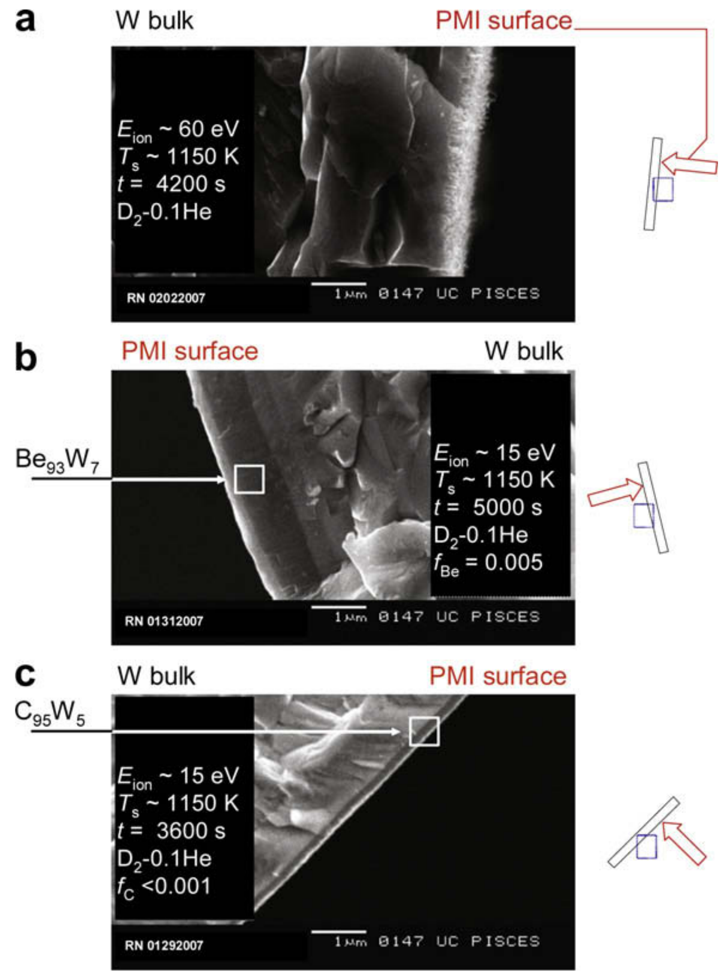


T. Chookajorn et al. Science 337 (2012) 951

Triggering instabilities and multi-scale patterning on irradiated surfaces: composition-driven mechanisms



El-Atwani and Allain JAP 2013



Baldwin et al, JNM, 2009

low concentrations of metal impurities triggering completely different structuring (patterning) conditions on Si vs refractory metals: role of bonding and defect dynamics

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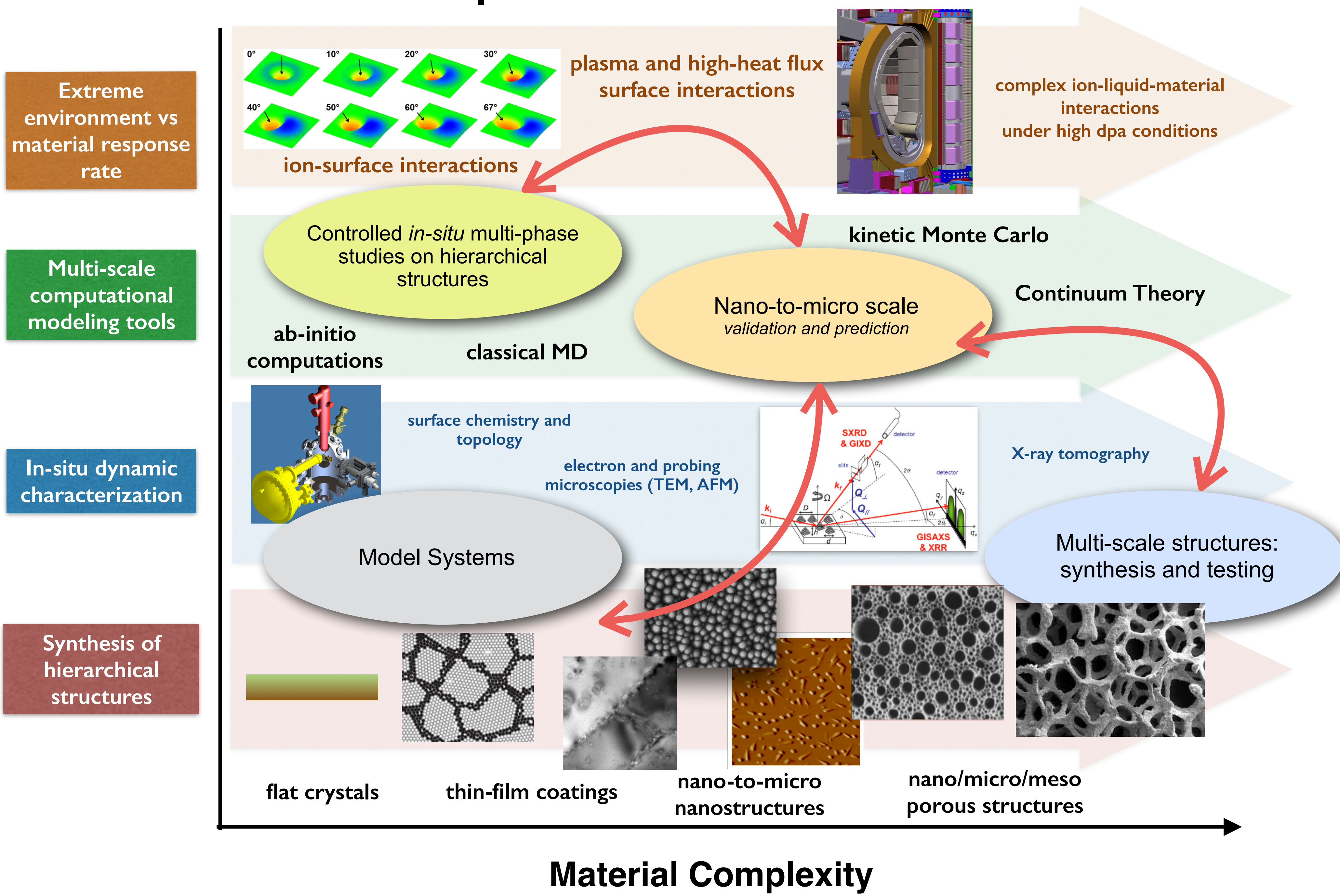
Summary: Strategy to develop multi-phase and multi-scale materials adaptive to extreme environments



Summary and Outlook

- Today we have a versatile toolkit of materials synthesis approaches, in particular, directed irradiation synthesis (DIS) and directed plasma nano synthesis (DPNS) leading to the discovery of multi-functional nano materials for extreme environments
- The demanding conditions of radiation-based environments to materials is also motivating *dynamic* and *in-situ* characterization to more ably tailor these materials to be adaptive
- Nuclear fusion environments were presented as an example of where adaptive multi-phase materials are currently being designed and programmed to be self-healing and adaptive
- Similar materials design paradigm could be used in other extreme environments that require multi-functional properties
- Novel materials must also meet requirements for scalability in the context of industrial processing

Thanks for your attention!

Extra Slides

Scientific Reports paper

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SUBJECT AREAS:
MATERIALS SCIENCE
TRANSMISSION ELECTRON MICROSCOPY

Received 20 December 2013
Accepted 12 March 2014
Published 17 April 2014

In-situ TEM observation of the response of ultrafine- and nanocrystalline-grained tungsten to extreme irradiation environments

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The accumulation of defects, and in particular He bubbles, can have significant implications for the performance of materials exposed to the plasma in magnetic-confinement nuclear fusion reactors. Some of the most promising candidates for deployment into such environments are nanocrystalline materials as the engineering of grain boundary density offers the possibility of tailoring their radiation resistance properties. In order to investigate the microstructural evolution of ultrafine- and nanocrystalline-grained tungsten under conditions similar to those in a reactor, a transmission electron microscopy study with *in situ* 2 keV He^+ ion irradiation at 950°C has been completed. A dynamic and complex evolution in the microstructure was observed including the formation of defect clusters, dislocations and bubbles. Nanocrystalline grains with dimensions less than around 60 nm demonstrated lower bubble density and greater bubble size than larger nanocrystalline (60–100 nm) and ultrafine (100–500 nm) grains. In grains over 100 nm, uniform distributions of bubbles and defects were formed. At higher fluences, large faceted bubbles were observed on the grain boundaries, especially on those of nanocrystalline grains, indicating the important role grain boundaries can play in trapping He and thus in giving rise to the enhanced radiation tolerance of nanocrystalline materials.

The performance of materials in extreme environments poses important fundamental questions about the behaviour of condensed matter under far-from-equilibrium conditions. These conditions create challenges in materials design and synthesis as highlighted in a recent report[1]. Extensive research has focused on ion irradiation of metals[2,3], semiconductors[4] and soft materials[5] to elucidate the response of these materials to extreme conditions. For example, the conditions found in nuclear fusion reactors have triggered recent research into the radiation tolerance of plasma facing components[6], resistance to morphological changes[7–9] and degradation of their mechanical properties[10–12]. Athough extensive work[6,13] has focused on identifying candidate materials such as refractory metals (for example, tungsten and molybdenum), fundamental understanding of the atomistic processes which give rise to their radiation resistance has been limited by the small number of studies[14–16], in which the dynamic response of these materials is observed directly.

In the context of plasmas in nuclear fusion reactors, tungsten is considered one of the best options as a plasma facing component (PFC) material[17,18]. However, it is known that irradiation of tungsten with He (one of the main products of nuclear fusion reactions) can lead to microstructural changes such as bubbles[19], pores[20], nanostructures[9] and fuzz formation[21] in addition to radiation induced hardening[7] and embrittlement[13]. In the quest for so-called "radiation resistant" materials, efforts have included investigations into novel ultrafine- and nanocrystalline-grained materials[22,23] (such as tungsten in the current study) due to the conjecture that radiation resistance can be improved by increasing the grain-boundary area[24,25]. In the context of the current study, we define a radiation resistant material as one which will demonstrate reduced surface nanostructure formation under plasma facing conditions. Grain boundaries are known to be good He and defect sinks[26–28], both of which can drive the aforementioned microstructural changes[29]. Research studies on nanocrystalline grained metals (such as copper[30], gold[31] and nickel[30]) and ceramics (such as ZrO_2)[32] have demonstrated higher radiation

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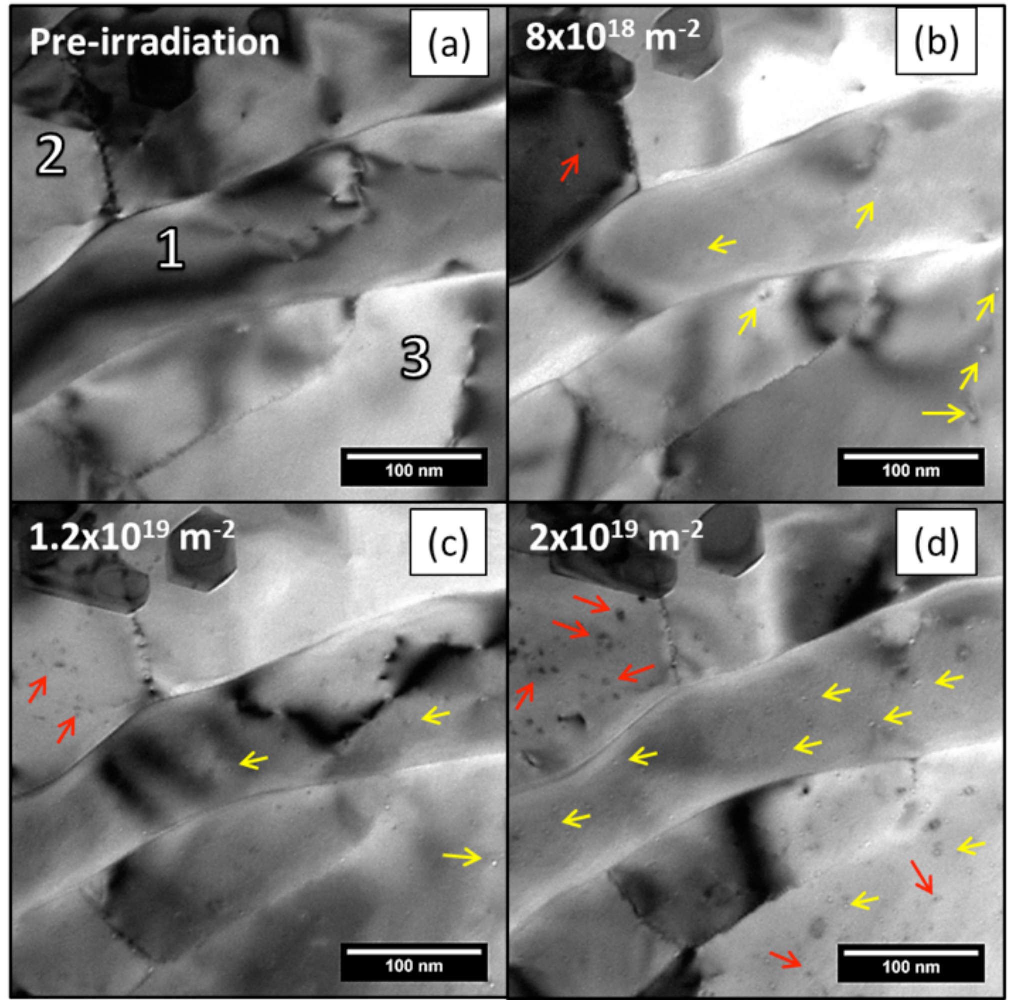
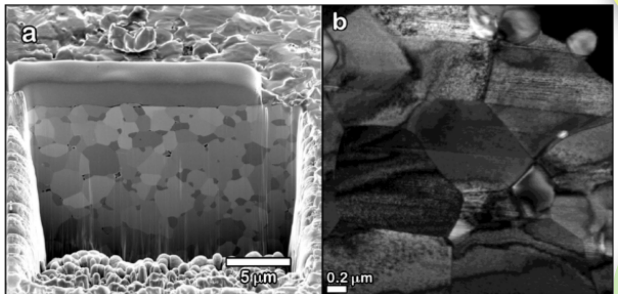


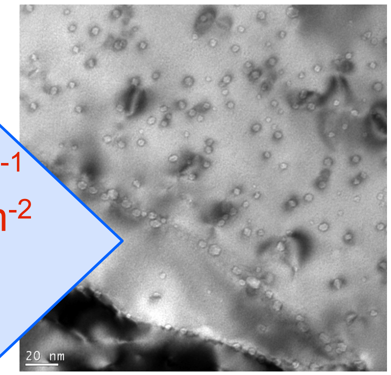
PMI work at Illinois in Prof. Allain's group

Processing:
Spark plasma sintering
severe plastic deformation

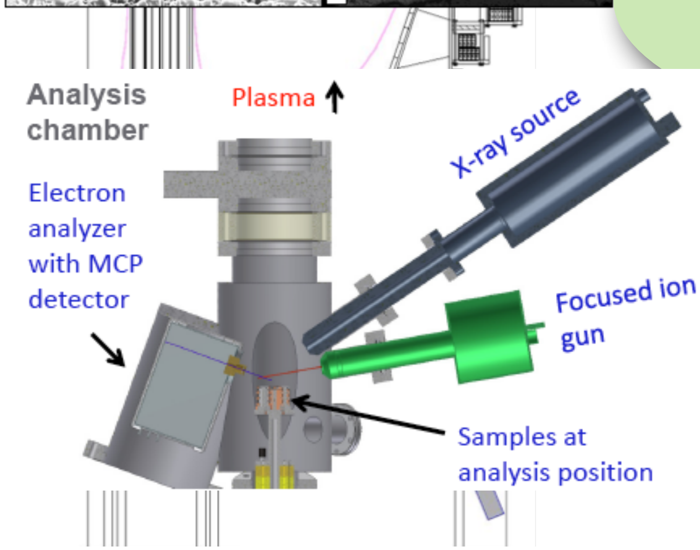


Multi-scale irradiation on extreme-refined grained W

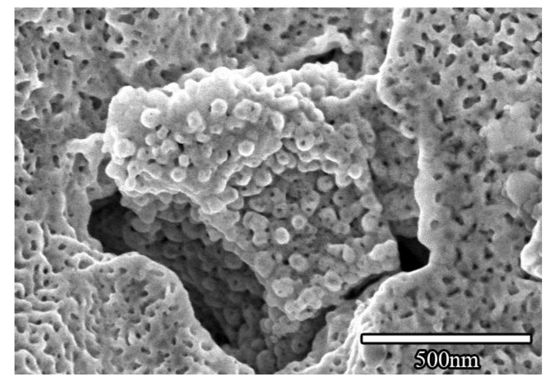
Fluxes: 10^{18}-10^{24} $m^{-2}s^{-1}$
Fluence ~ 10^{18}-10^{26} m^{-2}
T ~ RT up to 1200K
E ~ 10 eV to 2-keV



Irradiation behavior of hot lithium coatings on refractory and graphitic porous substrates



In-situ PMI diagnostics: MAPP in NSTX-U



- Process-property-performance relationships studied in well-diagnosed *in-situ* experiments at Illinois and collaborators worldwide. Emphasis on nanoscale materials design and *in-situ* testing coupled to computational models

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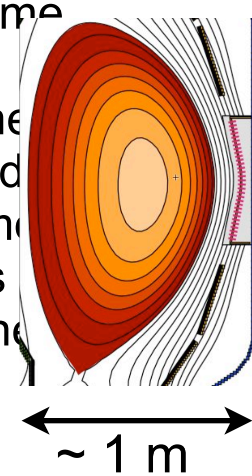
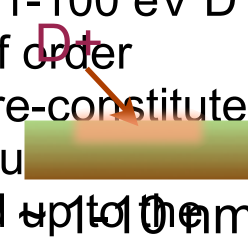
Unraveling the "black box" of the plasma-material interface in tokamak devices

- Fundamental interactions between the energetic particles from the plasma and its interface with the wall have for a long time been known to be important
- However, systematic understanding of this coupling has been challenged by the aggressive environment at the fusion plasma edge
- Wall material options were also driven in part by their influence on plasma performance (e.g. impurity erosion, particle recycling, etc...)
- In-vessel coatings deposition (e.g. boronization) led numerous efforts in manipulating plasma behavior with conditioning of the wall[1]
- however most of these efforts relied mostly on "trial and error" as the PMI (plasma-material interaction) empirical parameter space was developed over tim
- spatial scales became evidently important: sputter depth, pene h of 1-100 eV D ions ranging from 1-10nm in most fusion PFC materials, and d es of order 100-1000 nm, surface chemistry interactions at the first few m and re-constituted material films that vary from a few nm to 100-1000 nm results to elu plasma-surface interactions from an atomistic level through the and up to the macro-scale behavior of the plasma edge to the core.





[1]B. Lipschultz et al. Phys. Plasmas 13 (2006) 056117
[2]G. Federici et al. Nucl. Fusion 2001
[3]G.F. Councell et al. J. Nucl. Mater. 290 (2001) 255

Transport creates and moves impurities

Ions:

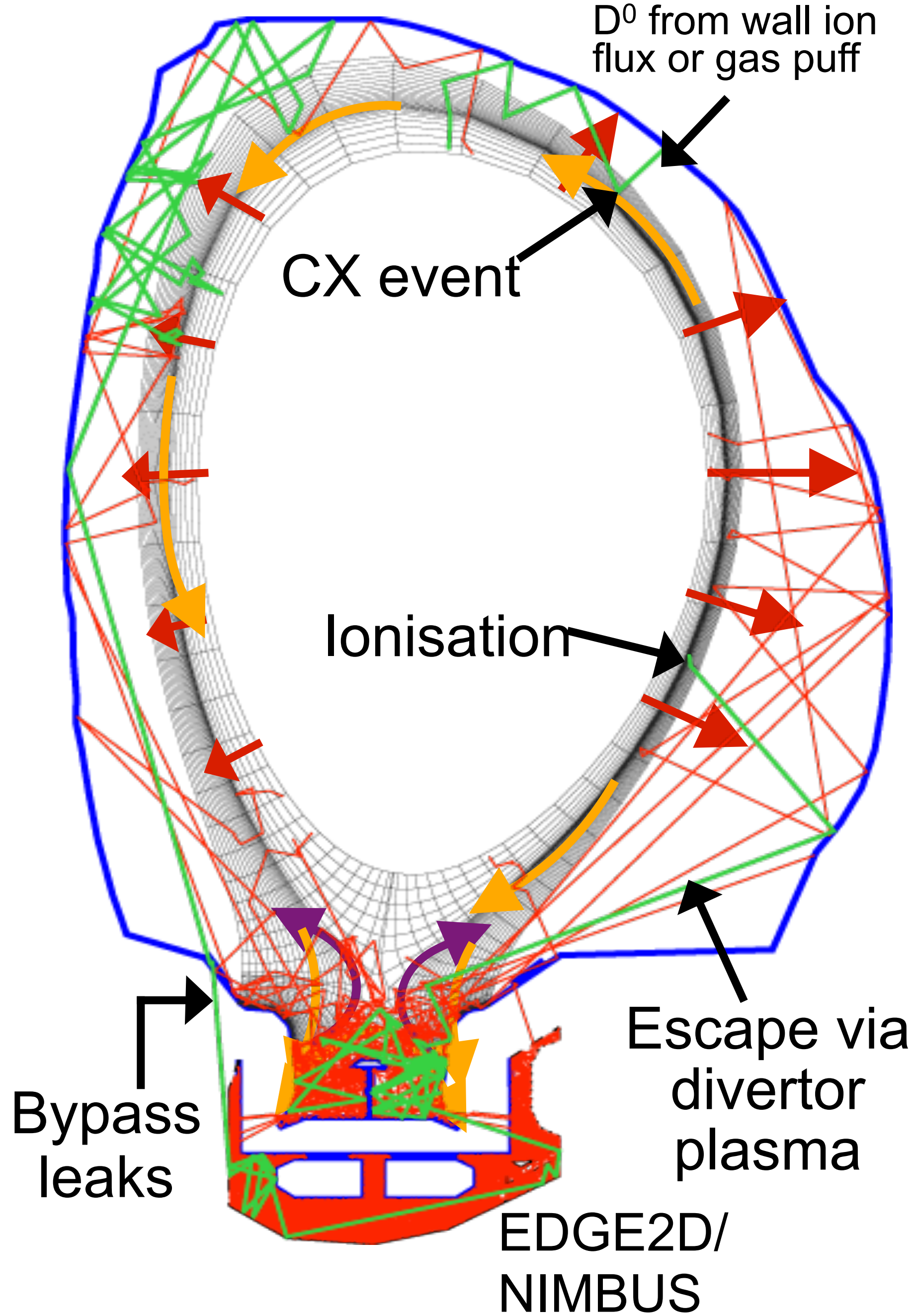
Cross-field transport – turbulent driven ion fluxes can extend into far SOL
→ recycled neutrals
→ direct impurity release
ELMs can also reach first walls

Eroded Impurity ions "leak" out of the divertor (∇T_i forces)

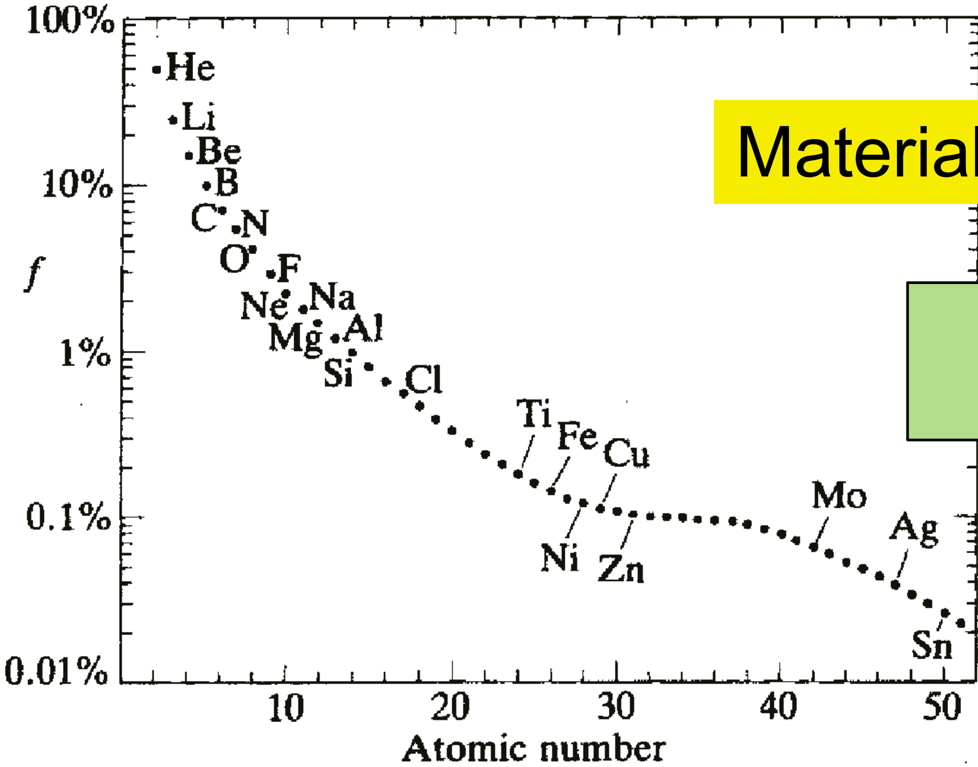
SOL and divertor ion fluid flows can entrain impurities

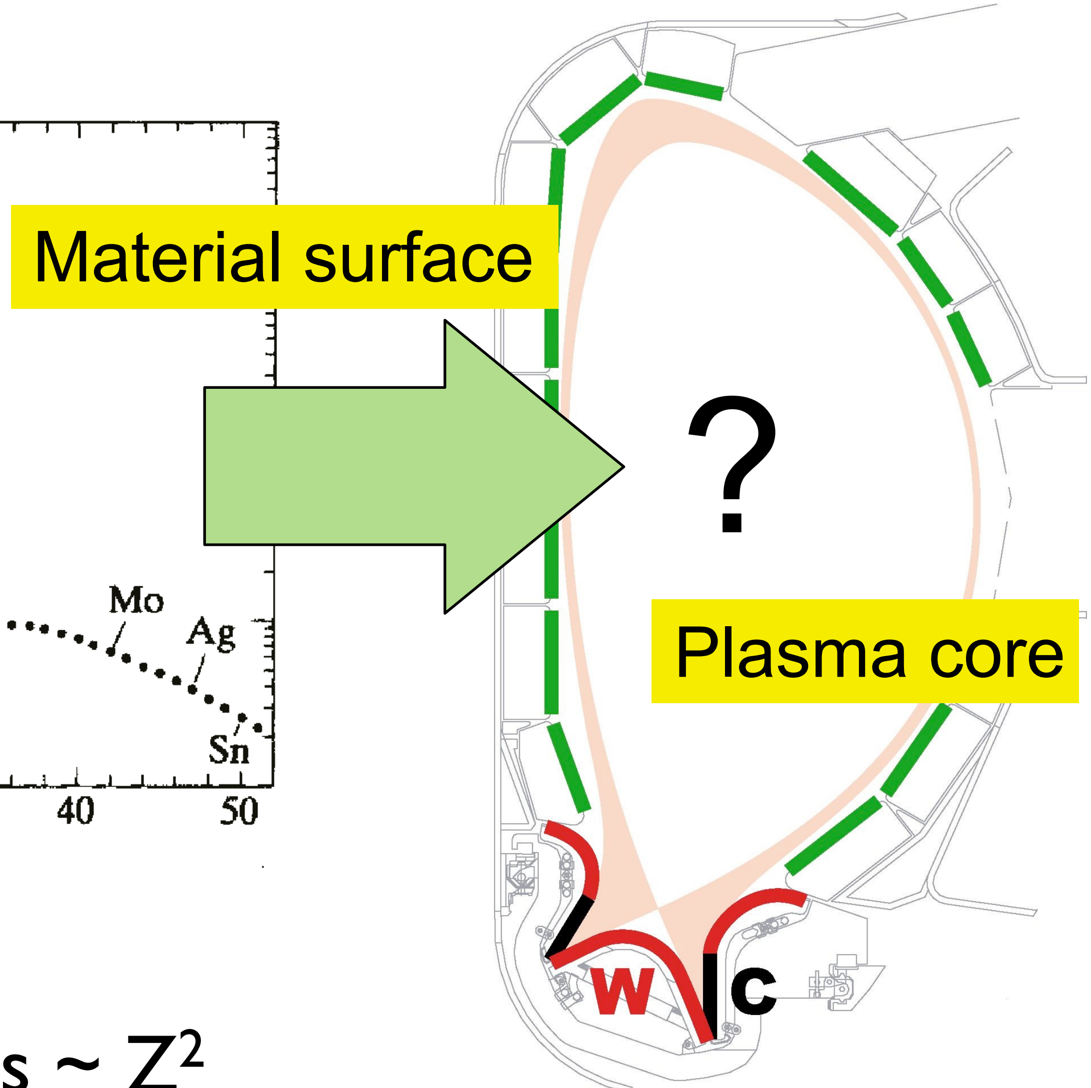
Neutrals:

- From divertor plasma leakage, gas puffs, bypass leaks → low energy CX fluxes → wall sputtering
- Lower fluxes of energetic D^0 from deeper in the core plasma
- A problem for first mirrors in ITER



Managing materials losses in a multi-component plasma-material interface



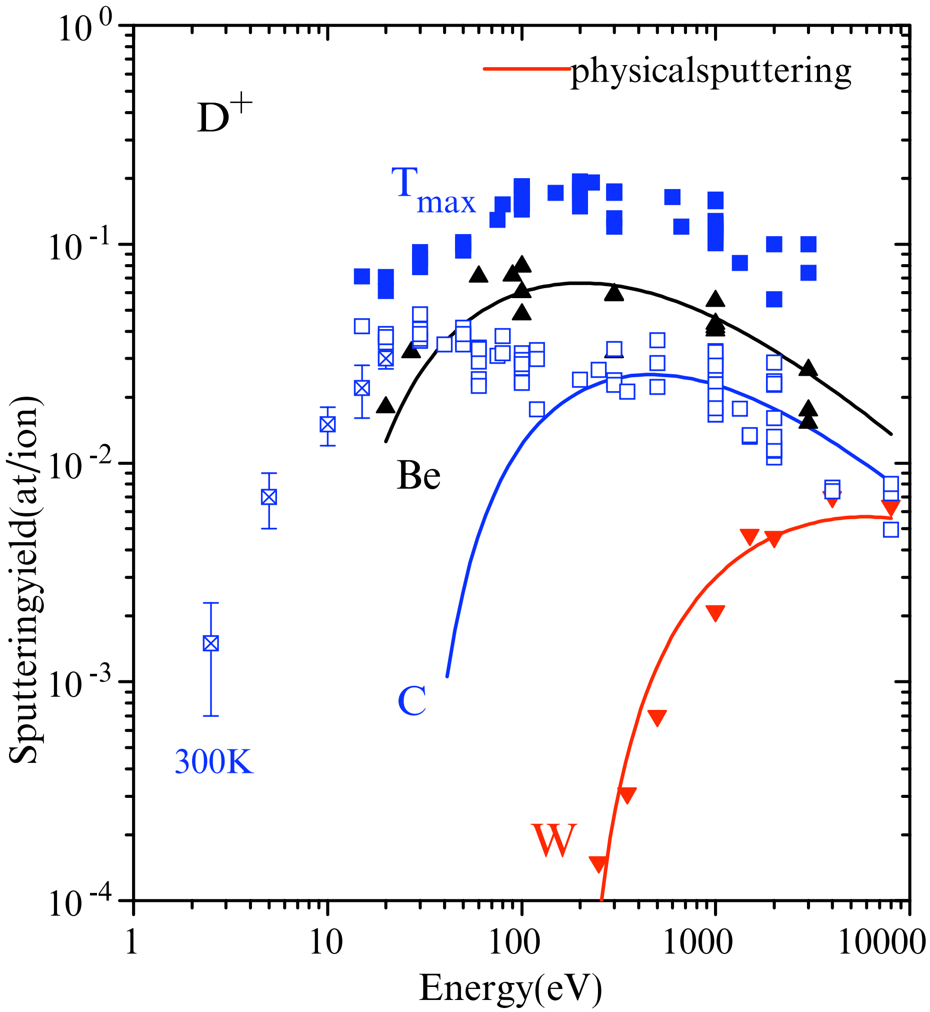


radiation losses ~ Z^2

ITER cross section

What is the problem? We lack the basic understanding and diagnosis of PSI processes in fusion devices[1]

- Intermediate steps uncertain between erosion and core plasma
  - Intense power flux density: *materials placed near thermal limits*
- Surface layers of PFM are rapidly and continually being reconstituted by plasma erosion and re-deposition
- Plasma transports ensures large gradients in plasma conditions across magnetic flux surfaces



[1]D.G. Whyte, HHFC Meeting, UCSD, December 2008