Research and plans on irradiated tungsten at the University of Illinois, Center for Plasma-Material Interactions

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1st IAEA Research Coordination Meeting on Plasma-wall Interactions with Irradiated Tungsten and Tungsten Alloys in Fusion Devices
Vienna, Austria, 25-27 November, 2013

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CPMI Center for Plasma-Material Interactions

Outline

- PMI/PFC research in irradiated W at Illinois
- Motivation for nanostructured W studies
- Processing of extreme refined-grain W
- *In-situ* TEM results of dynamic defect behavior in extreme refined-grain W
- High-flux irradiations in Pilot-PSI and Magnum-PSI at DIFFER
- Challenges to characterization and computational modeling of hydrogen isotope interactions in irradiated W
- Summary

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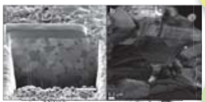
Fusion work in irradiated W at Illinois

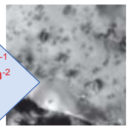
Processing:
Spark plasma sintering
severe plastic deformation



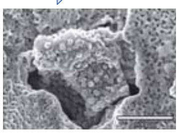
Multi-scale irradiation on extreme-refined grained W

Irradiation behavior of hot lithium coatings on refractory and graphitic substrates

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



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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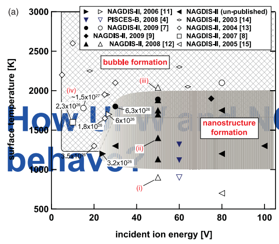
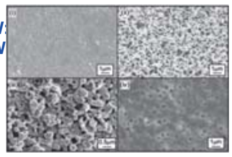
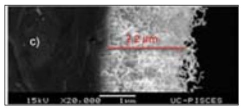
Nanostructuring of W by He irradiation

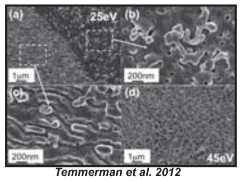




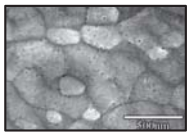
S. Kajita et al. Nuclear Fusion, 49 (2009) 095005
M.J. Baldwin et al. Nuclear Fusion 48 (2008) 035001



Doerner et al. 2011



Temmerman et al. 2012



O El-Atwani et al. JNM 2012

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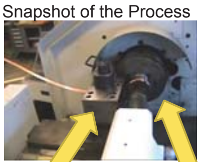
Severe plastic deformation leads to extreme refined grained tungsten

- Irradiations were performed on UFG (ultrafine-grained) and NC (nanocrystalline) samples prepared by large strain extrusion machining[1,3]
- Coexisting of ultrafine (≤ 500nm)[1] and nano-crystalline (≤ 100 nm)[2] grains adjacent to each other permitted the observation of the behavior of both types of grains and their irradiation tolerance
- Current research focused on processing of full nanocrystalline W grain materials in collaboration with S. Chandrasekar (IE, Purdue)
- Samples size varied depending on characterization and irradiation experiments
  - 3-mm UFG/NC W samples prepared to 300-nm thickness with FIB-SEM
  - 5-mm samples prepared for high-flux irradiation plasmas (DIFFER collab with deTemmerman)

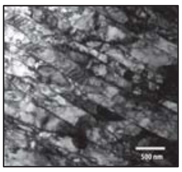
1. T.L. Brown, Acta Mater. 2009
2. Q. Wei, et al. Materials Science and Engineering A 493 (2008) 58–64
3. M. Ravi Shankar, et al. Acta Mater. 54 (2006) 3691

Snapshot of the Process



Extrusion Machining tooling

Commercially available lathe



UFG Tungsten

M. Efe et al, Scripta Materialia, 70 (2014) 31-34

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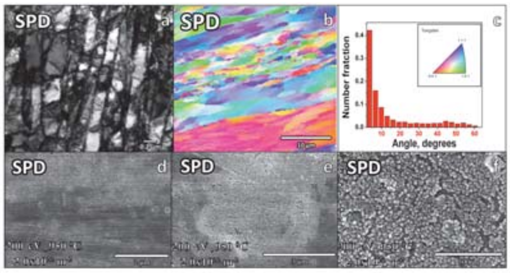
Irradiation of SPC NC-W with 200 eV He$^+$ with moderate fluxes

Irradiation with helium ($2X10^{18}$ ion.cm^{-2} or $2x10^{22}$ ion.m^{-2})
Helium energy = 200eV (no displacement damage)
Temperature = 950 C (both thermal vacancy and interstitial migration are possible)



Nanostructuring of the shear band regions

O. El-Atwani et al, J. Nucl. Mater. 434 (2013) 170

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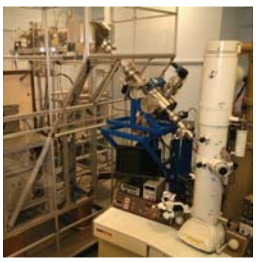
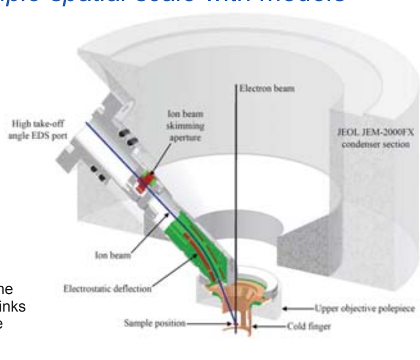
Observing defect dynamics with in-situ TEM can elucidate effects: couple spatial scale with models





- 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 $4x10^{18}$ m^{-2} with 2-keV He+ ions at 950 ^0C

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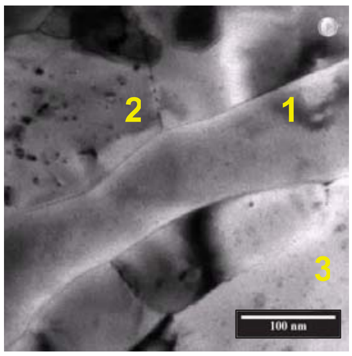
University of HUDDERSFIELD

In-situ TEM observation during 2-keV He$^+$ irradiation at 950 C

Fluence from 2-2.4x10^{19} m^{-2}

- Movement of loops occurs between pinning defects
- Irradiation enhanced diffusion (high defect concentration and enhanced mobility) needed for these defects to shuttle between pinning defects



During irradiation

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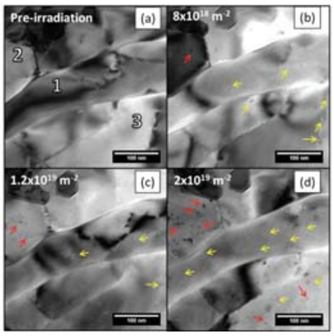
Early and latter stages of damage observed with in-situ TEM

2 keV He+ ion irradiation of tungsten at 950°C

- **(a)** nanocrystalline (1) and ultrafine (2 and 3) grains before irradiation
- **(b)** at a fluence of 8×10^{18} ions.m^{-2} and greater He bubble nucleation (bubbles indicated by yellow arrows)
- **(c)** after irradiation to a fluence of 2.4×10^{19} ions.m^{-2} showing point defect cluster formation (indicated by red arrows) occurred preferentially in grains 2 and 3
- **(d)** after irradiation to a fluence of 3.2×10^{19} ions m^{-2}, a higher areal density of point defect clusters and small dislocation loops evident in grains 2 and 3 while grain 1 demonstrates a uniform distribution of bubbles and a significantly lower areal density of defect clusters and dislocation loops.



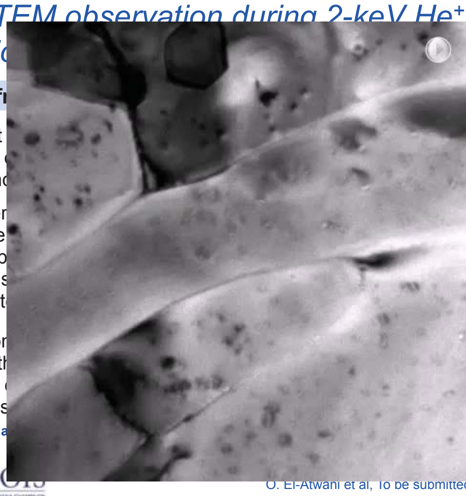
O. El-Atwani et al, To be submitted 2013

In-situ TEM observation during 2-keV He+ irradiatio

Fluence fr ... **adiation**

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

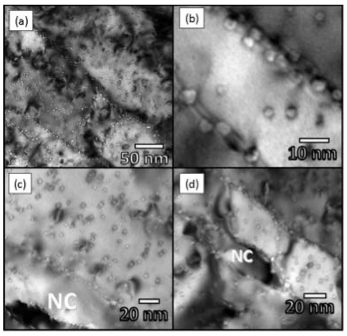
(H. Iwakiri et a



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

Summary of the in-situ TEM studies

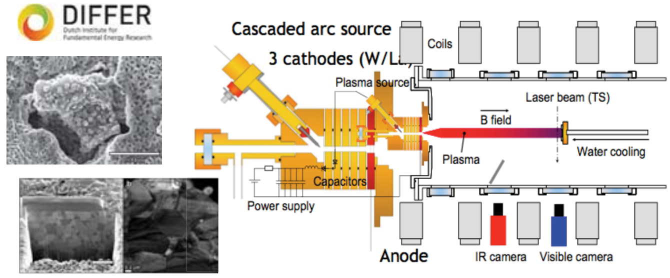
- Grain boundaries are He sinks (large bubbles on the grain boundaries).
- Intra-granular bubble and defect formation in relatively large grains (e.g. > 60-nm)
- Grains of less than ~ 60-nm in size* yielded a 50% lower areal bubble density compared to larger grains (60-100 nm) and ultrafine grains (100-300 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

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

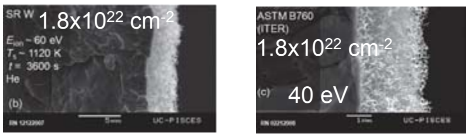
Collaboration with FOM-DIFFER (Dutch Institute for Fundamental Energy Research)



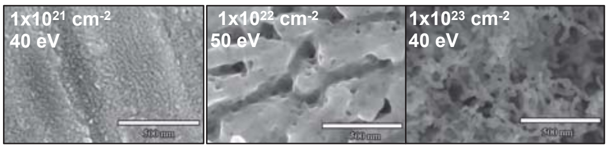
- Three lines of research: **1) high-flux irradiation (quiescent + transient) of nanostructured W and Mo, 2) Surface morphology and chemistry of nanostructured Low Z coatings on W and Mo, 3) temperature-dependent grain erosion studies of nano-structured W and Mo**

Collaboration with G. De Temmerman, T. Morgan et al.

Comparison with literature (fuzz formation)



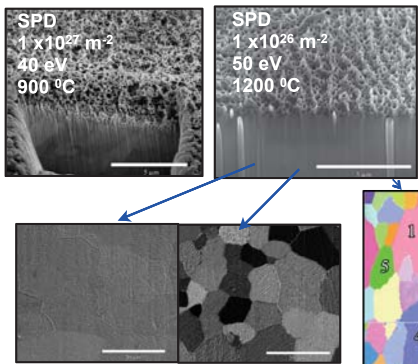
Baldwin et al, JNM, 2010



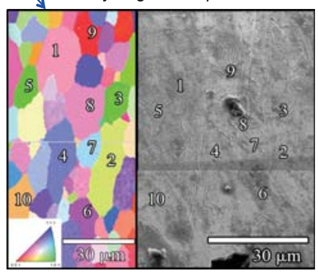
SPD samples (UFG) have higher fuzz formation fluence thresholds, however fuzz thickness growth rate is faster

O. El-Atwani et al, in preparation 2013

Nanostructure and morphology evolution on SPD-W

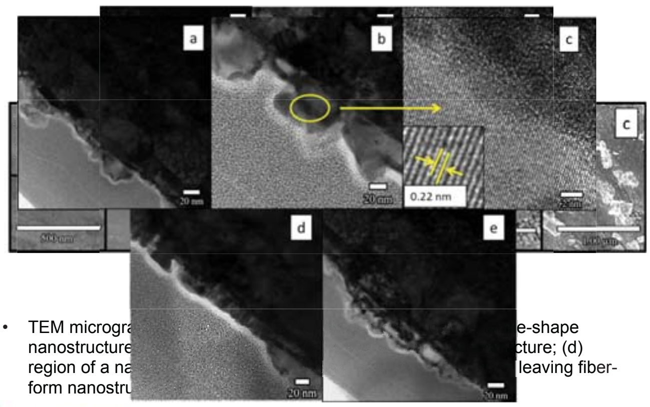


- Surface nanostructuring in multiple scales; evidence of He bubble emission at surface
- Crystallographic dependence of irradiation-driven surface patterning: *implications for hydrogen isotope interactions*



Collaboration with G. deTemmerman (DIFFER)

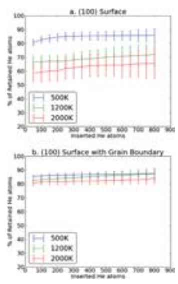
SEM and TEM micrographs of the ultrafine grained tungsten sample irradiated with 30 eV He particles to a fluence of 1x10^{19} cm^{-2} at 900 °C

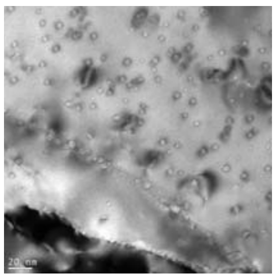


- TEM microgra... nanostructure... region of a na... form nanostru... e-shape ... cture; (d) ... leaving fiber-

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

He clusters at grain boundaries of SPD tungsten: comparisons to atomistic modeling

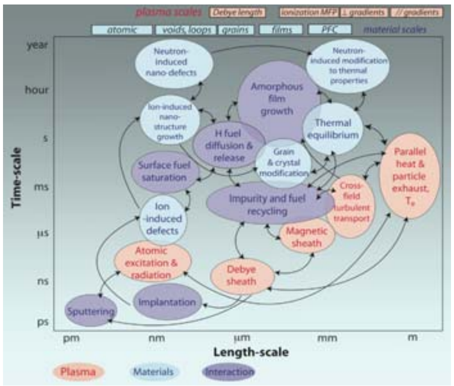




- Work by F. Sefta and B. Wirth et al. open up understanding of self-driven mechanisms for He cluster formation and role of grain boundaries
- How do we couple impurity-driven surface structuring and defect evolution in irradiated materials? How do we close the gap in space and time between models and diagnosis?

F. Sefta et al., Nuclear Fusion 53 (2013) 073015

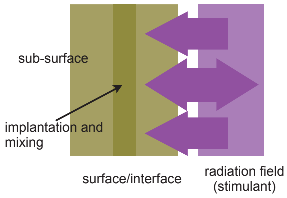
Irradiation-driven vs thermal-activated systems: instabilities and self-organization at the plasma-surface interface



B. Wirth et al. MRS Bulletin (2011), 36: 216-222

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

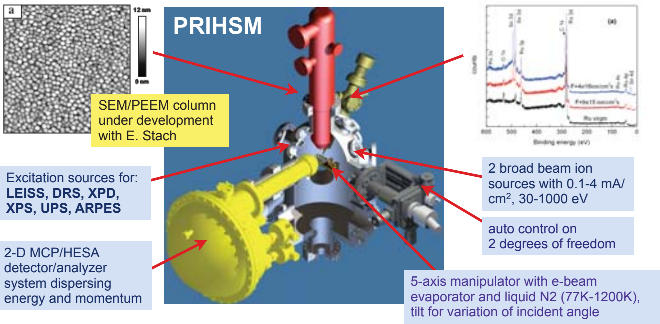
O. Shpyrko, UCSD



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

- 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*

Modification ion sources with in-situ tool set

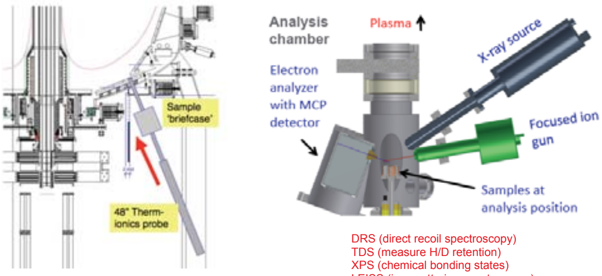


- Conditions: GaSb(100) samples irradiated by Ar+ at 50, 100 and 200 eV, normal incidence with 40-50 µAcm^{-2} for fluences up to 10^{18} cm^{-2}
- ***We operate in the sputter threshold regime and study early stage growth***
  - ***e.g. ~ 10^{15}-10^{17} cm^{-2}***

J.P. Allain et al. To be submitted 2013

Tokamak in-situ diagnosis of plasma-material interface: measurement of dynamic response

post-irradiation testing *will not* elucidate on *dynamic* effects



DRS (direct recoil spectroscopy)
TDS (measure H/D retention)
XPS (chemical bonding states)
LEISS (ion scattering spectroscopy)

NSTX Materials Analysis and Particle Probe (MAPP) with in-vacuo surface analysis: surface chemistry; To be installed Fall 2013 for integration with diagnostic tests in early 2014 and NSTX-U plasma runs beginning Sept 2014

C.N. Taylor et al. Rev. Sci. Instrum. 83 (2012) 10D703.

Summary

- Grain orientation and size have correlated effects on defect formation and surface nano-patterning and morphology evolution
- Defects dominated by high-density clusters within large grains (> 100-200 nm) and faceted He bubbles at grain boundaries in extremely refined-grained W
- In-situ TEM on commercial tungsten samples with micron-level grains and bubble density comparison reveal the efficiency in trapping He atoms at grain boundaries in ultrafine-W and nano-crystalline W (NCW).
- Future work with dual-beam *in-situ* TEM experiments (He and W) on UFG W (also doped) to investigate hydrogen isotope retention and migration in damaged W
- Computational modeling of He-induced defects in W face serious challenges: multi-scale spatio-temporal defect dynamics that are intimately connected to their nano/microstructure and driven far-from equilibrium in burning plasma fusion device environments
- Role of *in-situ* diagnosis of irradiated materials and how to close the spatio-temporal gap between measurements and modeling