

# **Materials Issues in Inertial Fusion Energy (IFE) First-wall Experiments - Response of Wall Materials to Repeated Pulses of High-energy Ions\***

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# Presentation Outline

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- **IFE Threat Spectra and Relation to MFE Conditions: RHEPP-1 can simulate ion threat to first wall**
- **Description of RHEPP-1 and Sample Exposure Setup**
- **Tungsten and Tungsten alloys: Brittleness leads to thermomechanical distress**
- **Graphite/Carbon Composites:**  
    **Sublimation loss may be main problem – physical sputtering?**
- **‘Engineered’ Materials as an alternative to flat wall**
- **Conclusions and Follow-on Work**

## In an IFE Reactor, the First-wall is subjected to a Programmed High Fluence of Energetic Ion pulses ( $3 \times 10^8/\text{year}$ )

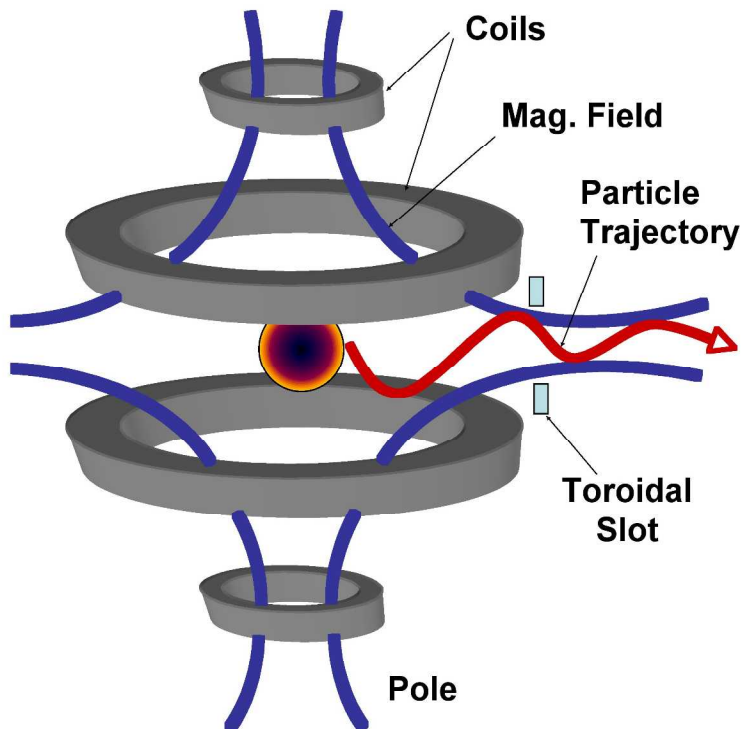
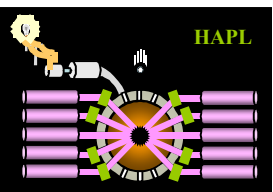


Image provided by R. Raffray,  
UC San Diego

- Unlike MFE, no Steady State phase, only 'transient' events.

Level per pulse is known, ~ few MeV ions normal to surface. At 10 Hz operation,  $3e8$  pulses per year

- At 10 nm erosion/pulse, 3 METER thickness lost per year. So NOTHING can be lost per pulse. Melting should be avoided as well.
- Biggest threat below Melting is Thermomechanical stress
- Leading Geometry: Spherical w/ or w/o gas fill
- Backup Geometry (LEFT): Cusp with 'beam dump' on axis
- Leading materials: tungsten and W alloys, SiC



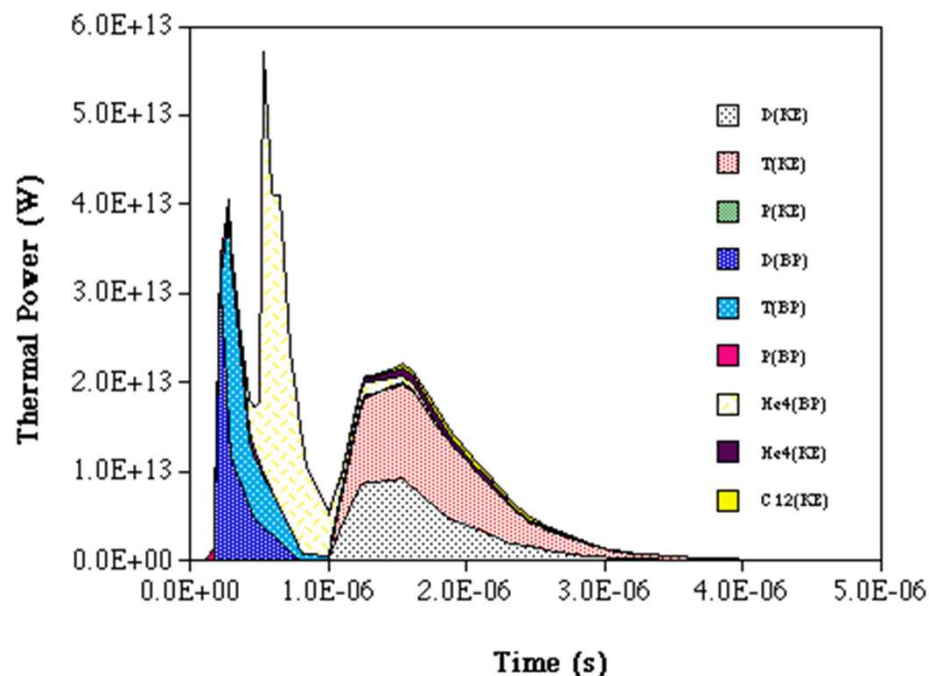
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# Laser IFE Direct Drive Threat Spectra

Time-of-Flight Ion Power Spread



Simulation: Thermal Power to Wall in Ions  
from 154 MJ Yield. Wall Radius: 6.5 m

- For Direct-drive Laser IFE:  
~70% neutrons, 1-2% x-rays  
**30% ions (50-50 fusion and 'debris')**
- Ions: several MeV, ~ few  $\mu\text{sec}$  each,  
**8-20 J/cm<sup>2</sup>** fluence, judged  
Significant Threat
- X-rays: ~ 1 J/cm<sup>2</sup>, up to 10 keV energies,  
judged less significant threat
- RHEPP-1: 700 keV N, higher for N<sup>+2</sup>,  
100-150 ns pulsewidth, 75-95 GW/m<sup>2</sup>
- RHEPP-1 energy delivery too short, but  
otherwise good fidelity with reactor ion threat

$F = P \cdot \sqrt{t}$ : High Heat Flux conditions with Heat Diffusion  
effect included. *Comparison:*

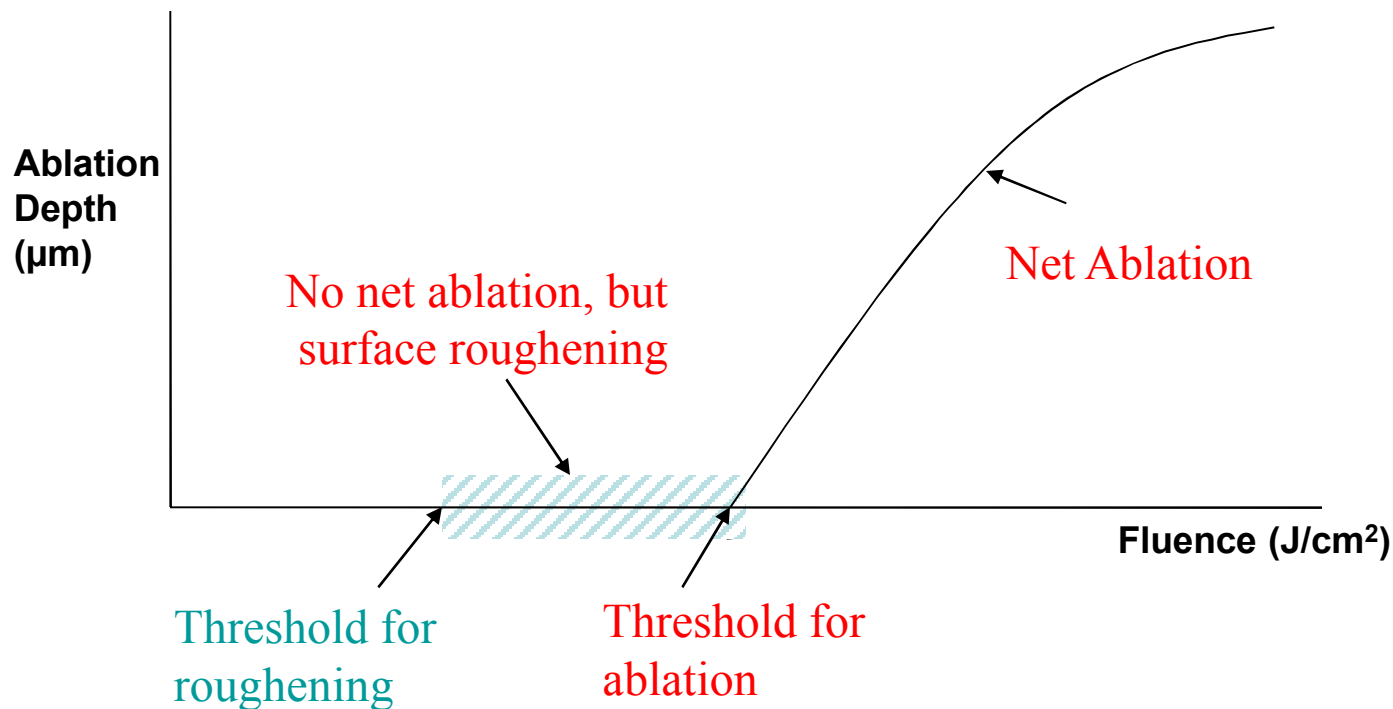
ITER ELMs (est):

**22.4 - 67.1 MW m<sup>-2</sup> s<sup>1/2</sup>**

RHEPP-1:

**33 - 112 MW m<sup>-2</sup> s<sup>1/2</sup>**

## Regimes of IFE Materials Response to Ions



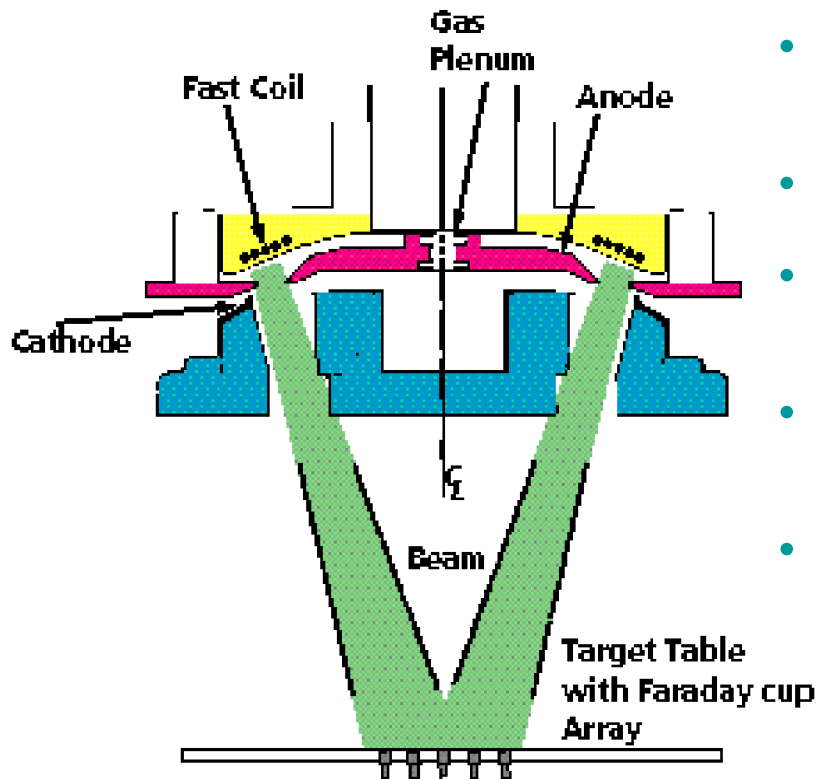
Goals (for each material): examine net ablation to validate codes  
find threshold for ablation

**DONE**

**Area of Interest**

Understand roughening. Is there mass loss?  
Find threshold for roughening  
(Fluence/pulse, No. of pulses)

## Overall View of RHEPP-1 vacuum chamber and treatment area



- 600-800 kV. Pulse Width  $\sim 100\text{-}200\text{ ns}$
- Up to  $250\text{ A/cm}^2$
- Beams from  $\text{N}_2$ , Ar, He used here
- Overall treatment area  $\sim 150\text{ cm}^2$
- Diode vacuum  $\sim 10^{-5}\text{ Torr}$



Tray shown here replaced by 'scallop' holder that avoids Beam center

## Not all ions are created equal: Surface morphology changes on SS303 samples depending upon treatment ion(s)

Proton Beam 25 pulses



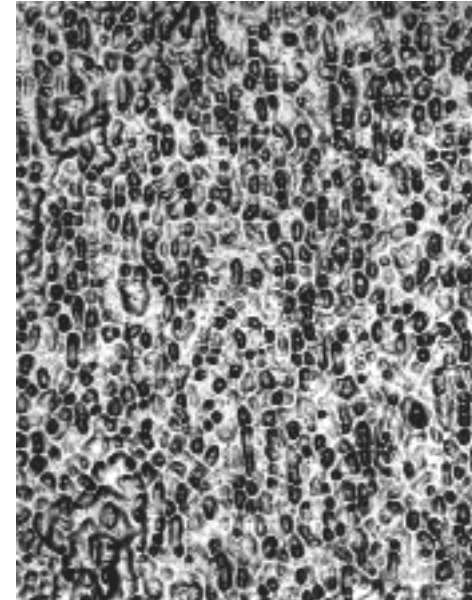
Modeling (Fe):  
Max Temp: 1658K  
'No' melting

Nitrogen Beam 25 pulses



Modeling (Fe):  
Max Temp: 2593K  
Melt depth: 0.7 μm

Argon Beam 25 pulses

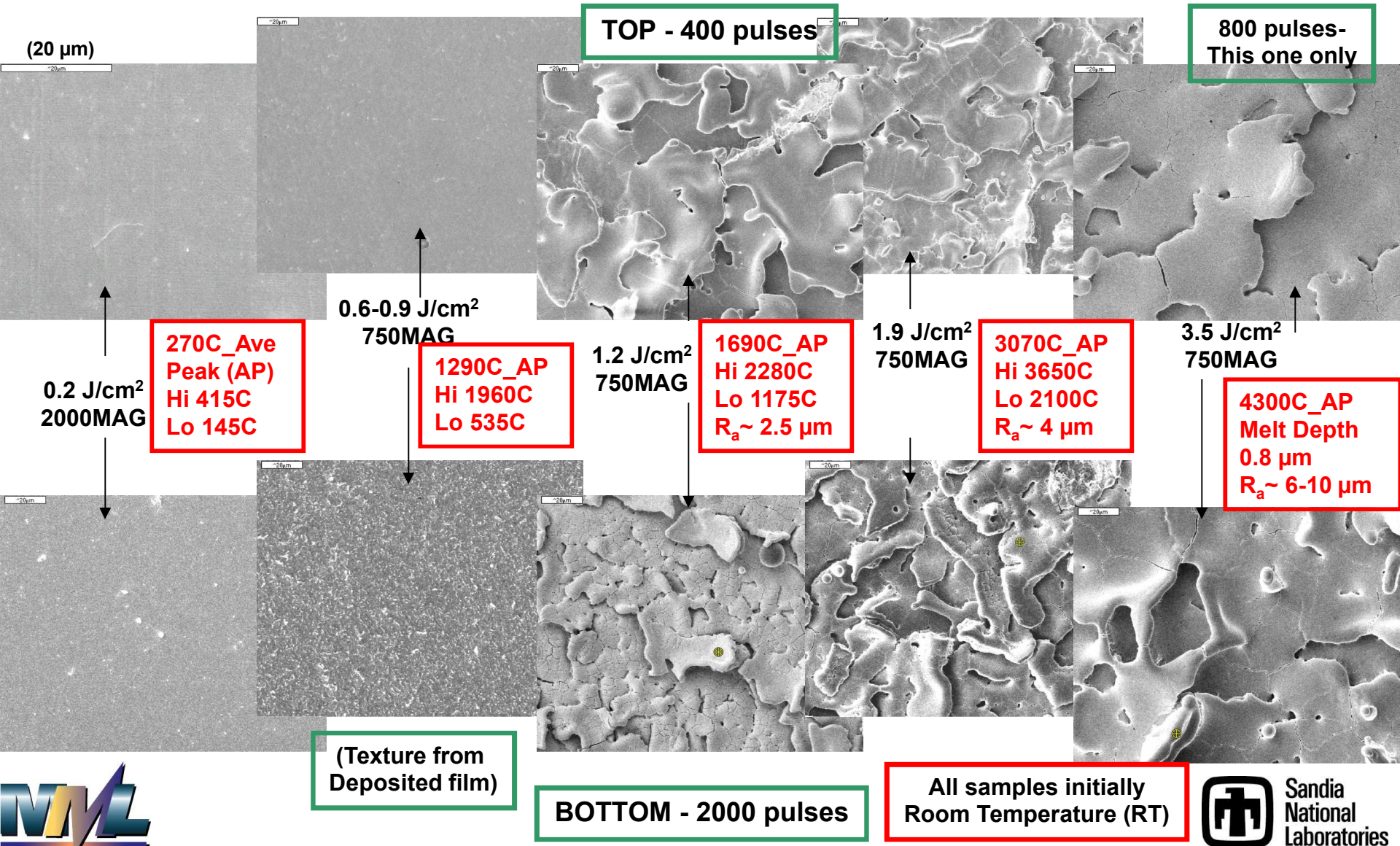


Modeling (Fe):  
Max Temp: 2072K  
Melt Depth: 0.45 μm

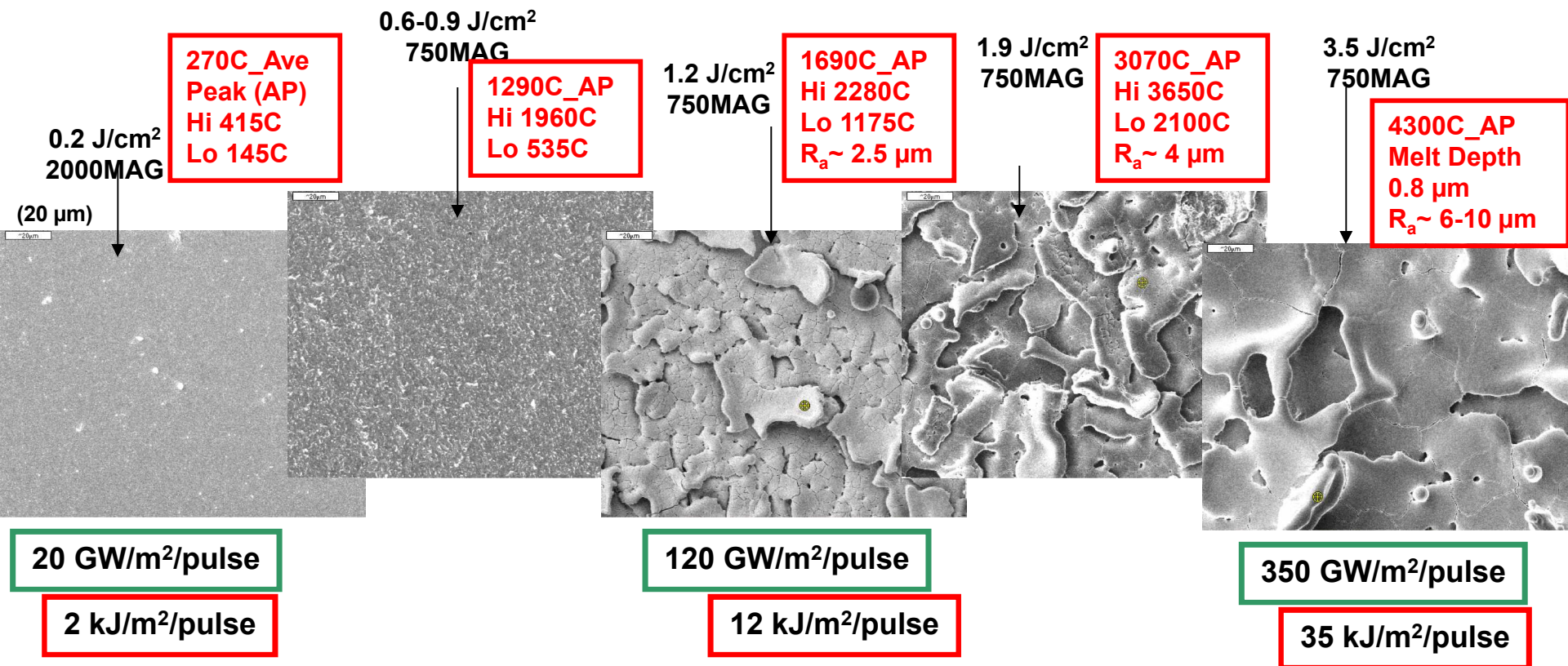
- SS303 exposed to 25 shots three different ion beams @ 2 J/cm<sup>2</sup>
- 'Finer' surface features with Nitrogen, Argon in micrographs
- Periodicity ~ 50 μm for N, Ar surface, ~ 70 μm for proton-treated surface. Main contribution to morphology - sulfur

# Tungsten Materials

# SEMs of Polycrystalline (PM) Tungsten Roughening: Threshold at $\sim 1 \text{ J/cm}^2$ , roughening saturates after $\sim 400$ pulses



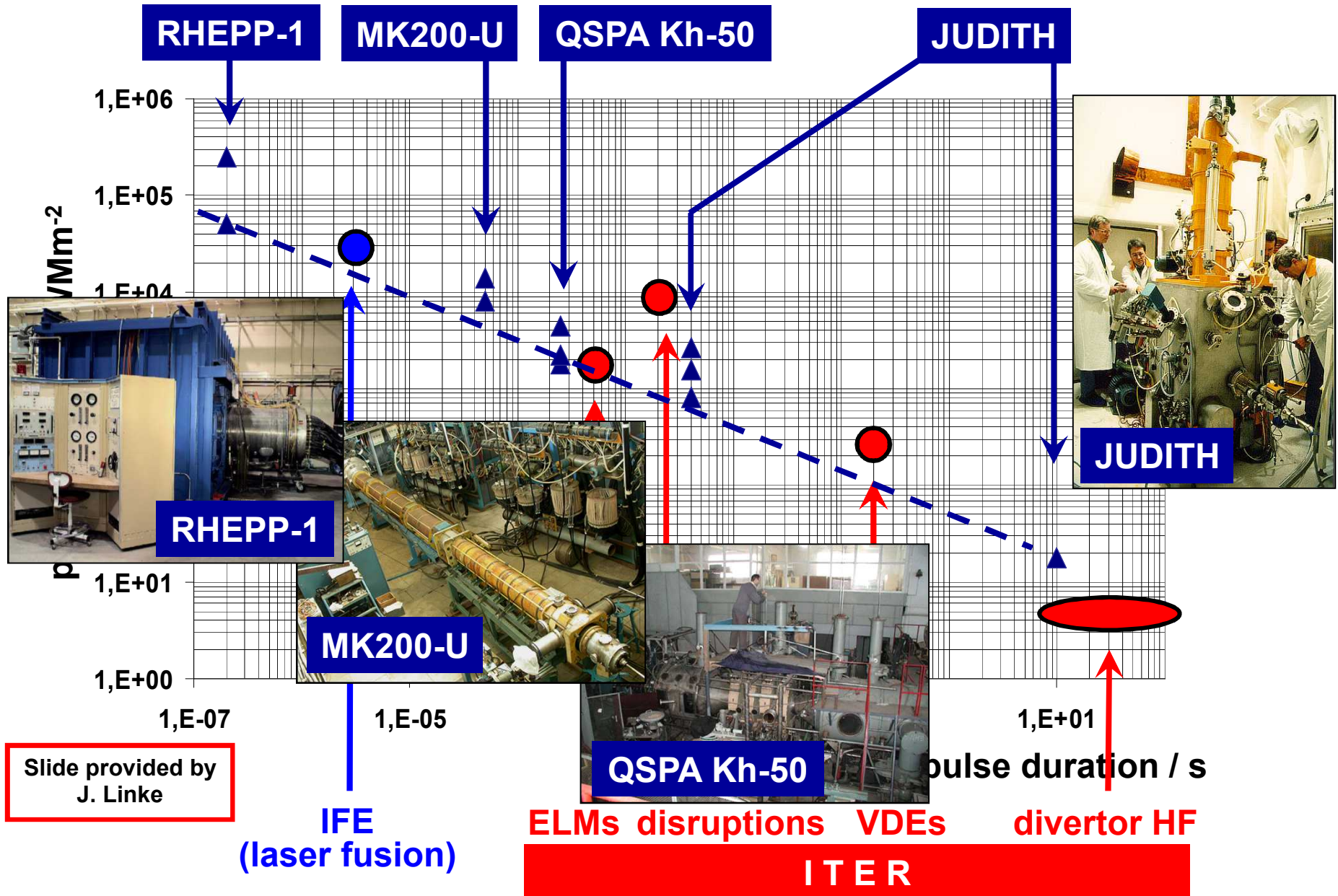
The 2000-exposure surfaces were exposed to as much as 350 GW/m<sup>2</sup> average power over 100 ns



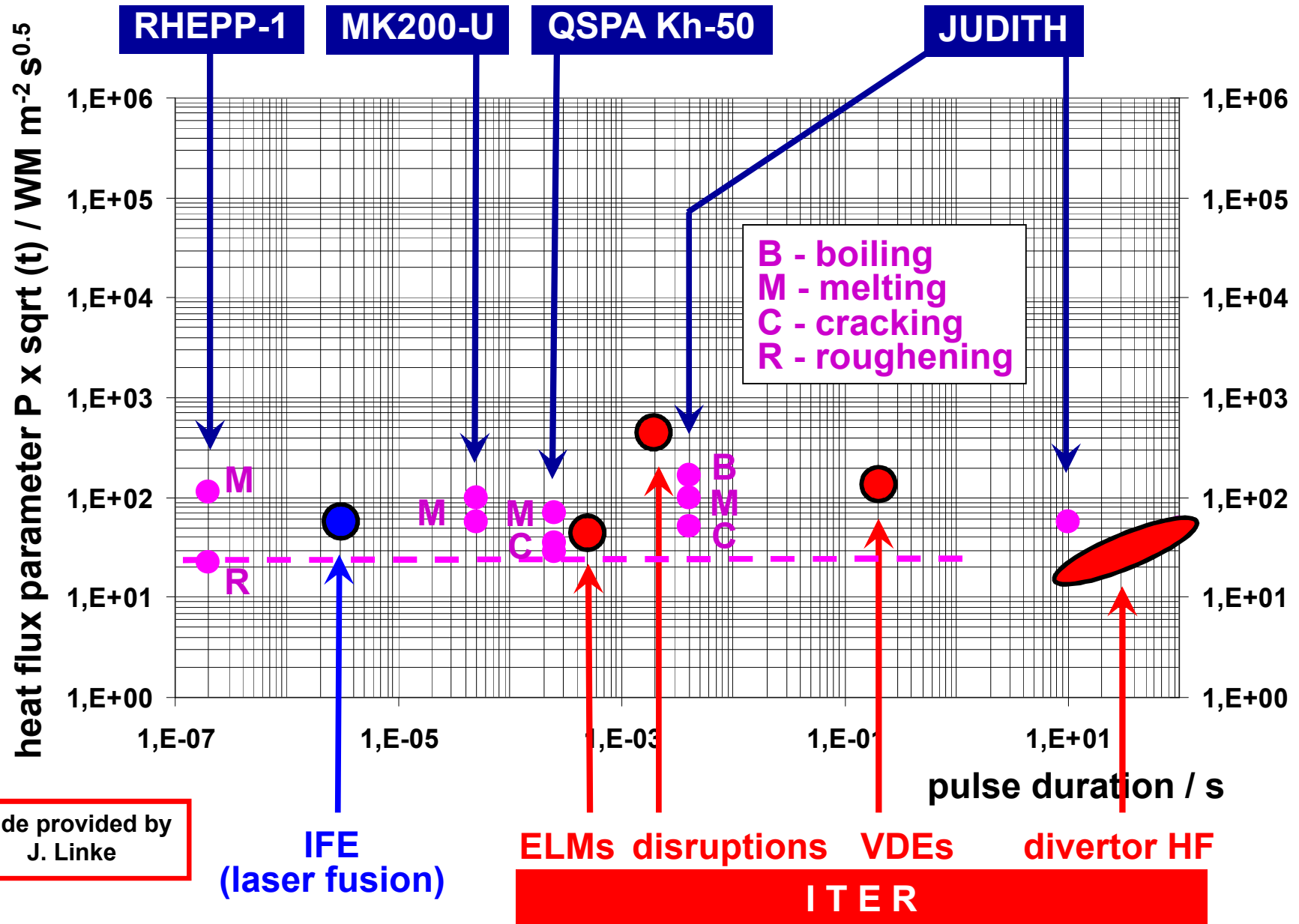
- 700 kV for 100 ns @ 15A/cm<sup>2</sup> = 1 J/cm<sup>2</sup>
- Only the rightmost surface above melted on each pulse
- Maximum cumulative dose 70 MJ/m<sup>2</sup>

All samples initially  
Room Temperature (RT)

# Simulation of short transient thermal loads



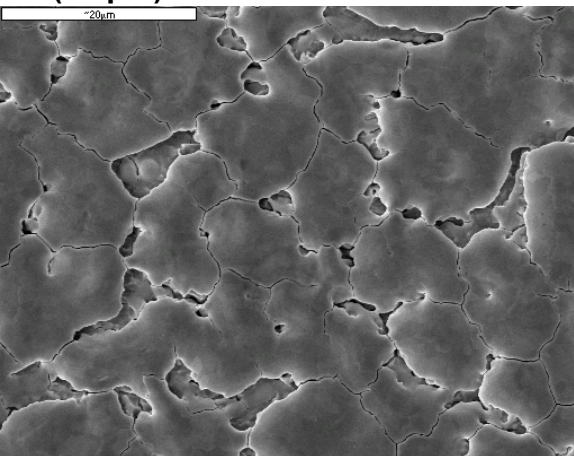
# Performance of W under short transient thermal loads



Slide provided by  
J. Linke

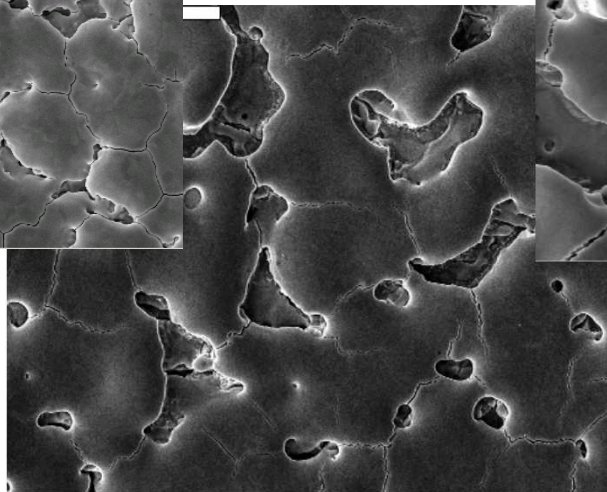
## SEMs of lightly deformed PM Tungsten (non-melt): appears stress cracking starts, then exfoliation, forming 'valleys'

(20  $\mu\text{m}$ )

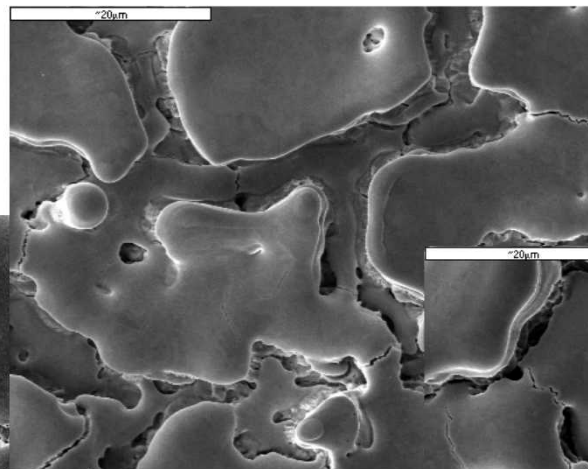


400 pulses

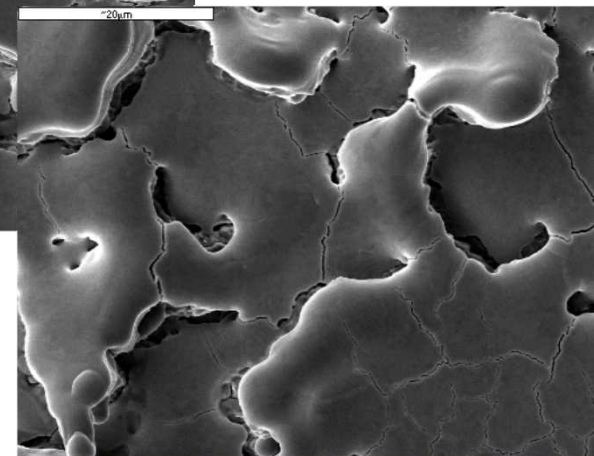
800 pulses



1200 pulses



1600 pulses

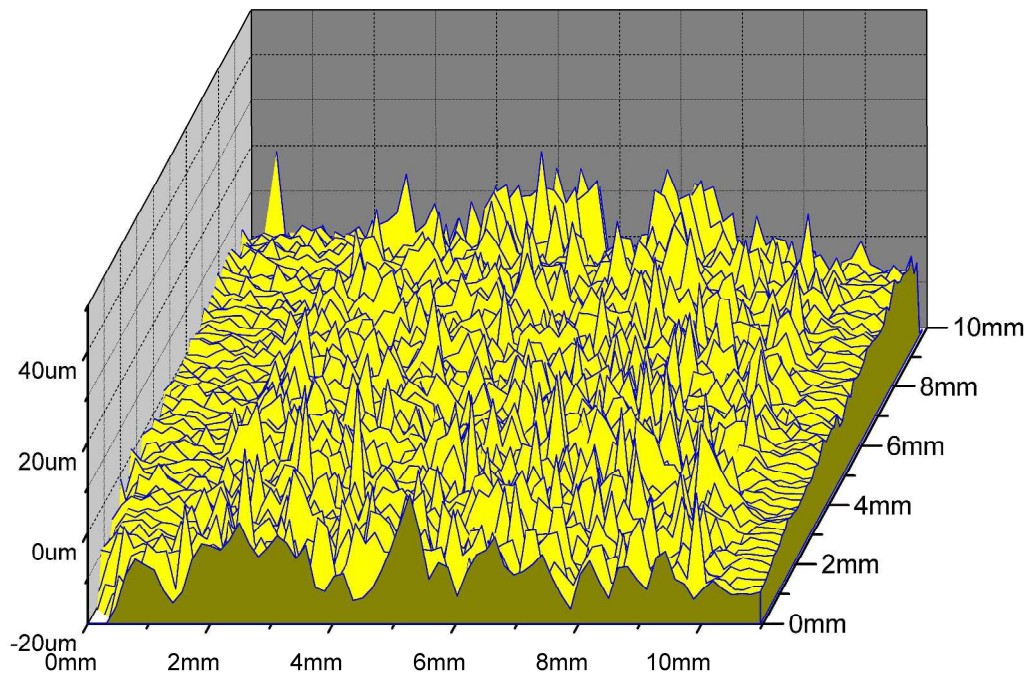


All images  
2000X

- Heated PM Tungsten (600C) exposed to Nitrogen beam at  $\sim 1.5 \text{ J/cm}^2$  - peak temp  $\sim 3300\text{K}$
- Rounded 'knobs' are actually high points.  
**Surface rises during treatment.**

## PM Tungsten after 1600 pulses (non-melting): Mostly mountains up to $\sim 30 \mu\text{m}$ height

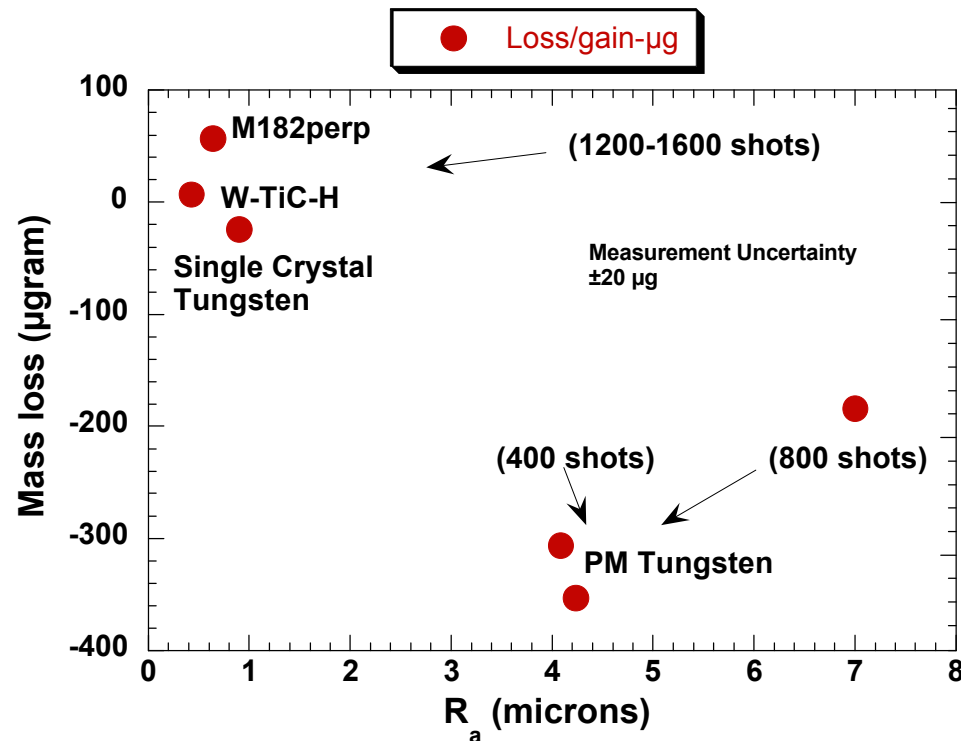
Tungsten 1600 Pulses



- Heated/treated PM Tungsten examined with NEXIV laser interferometry
- Comprehensive line-out scan: max height  $30 \mu\text{m}$ , min height  $< 10 \mu\text{m}$  compared to untreated
- Very deep microcracking not visible here
- Hypothesis: mountains are due to CTE expansion that does not recover

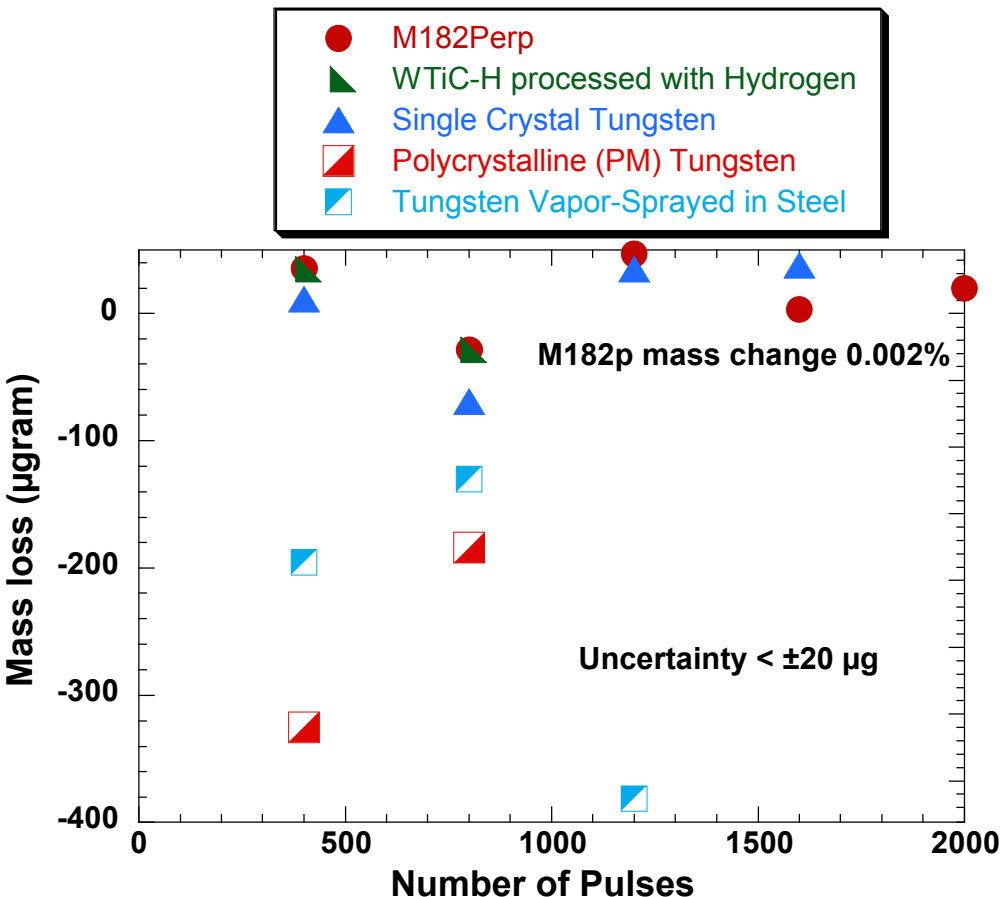
Cannot confirm mass loss by height study. Must weigh before/after exposure

## Sandia Metrology weight measurements support connection between Roughening and Mass Loss



- Samples of each listed material exposed for multiple 400-shot series, and weighed pre-and post-shot
- Exposure level/pulse: 1.2 - 1.7 J/cm<sup>2</sup>  
Measurement Uncertainty <  $\pm 20 \mu g$
- Two samples of polycrystalline (PM) Tungsten lost ~350 µg in 400 pulses, with  $R_a \sim 4 \mu m$ ; another 400 pulses produced even more roughening, -184 µm more mass loss
- M182Perp, W-TiC-H, and Single Crystal Tungsten remained < 1 µm  $R_a$ , and suffered little mass loss.

## Mass Loss with Shot Number: M182Perp and Single Crystal W show almost no loss



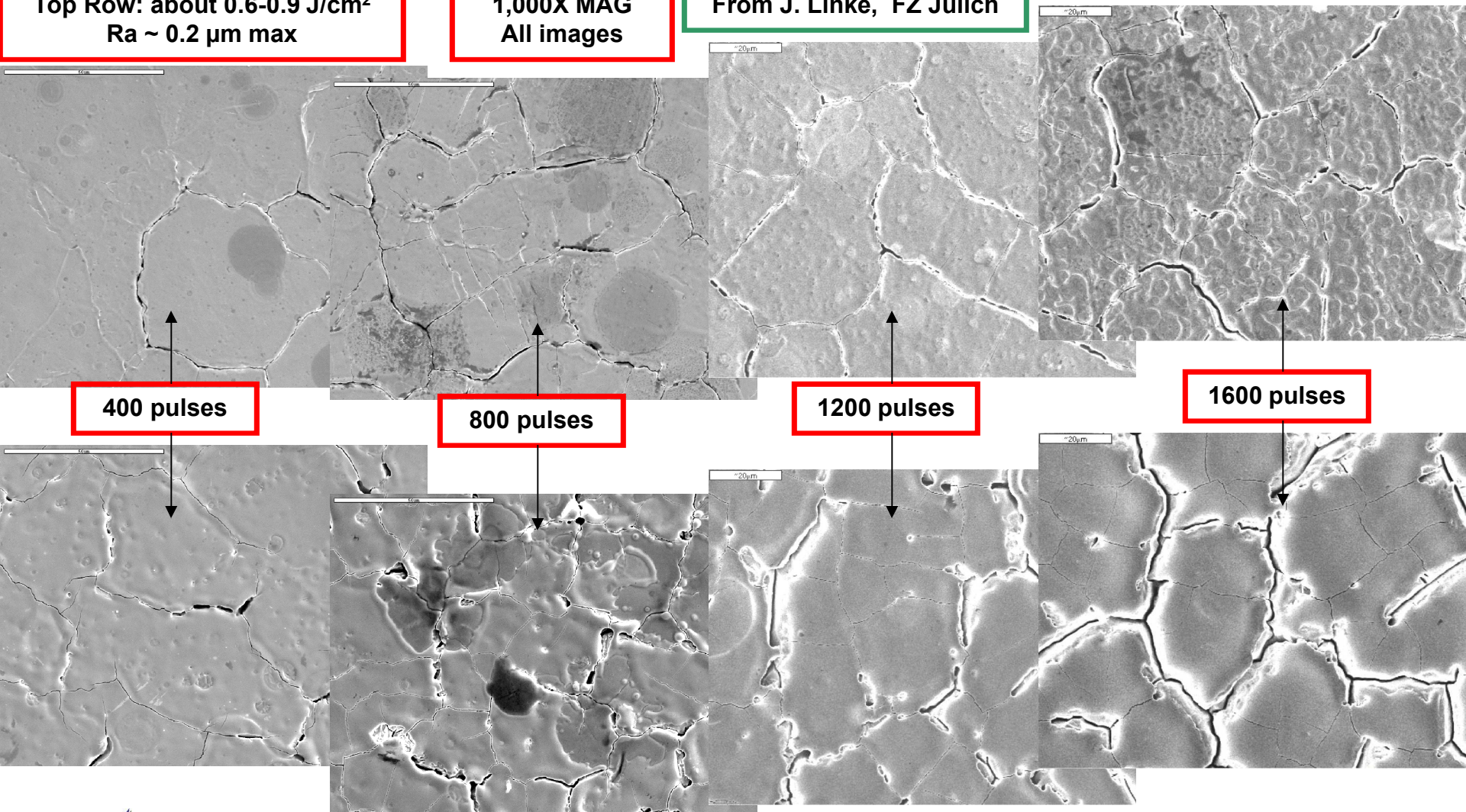
- Mass loss for PM Tungsten terminated after 800 pulses - 350 and 184  $\mu\text{g}$  loss on two 400 shot sets
- Vapor-Sprayed Tungsten (on Ferritic Steel) losses up to 400  $\mu\text{g}$  per 400 pulses
- Mass Gain due to entrained material (Cu) from diode region

# SEMS of Tungsten M182 perp after 1600 pulses: Little topology change below 1 J/cm<sup>2</sup>, some roughening w/ pulse number at ~ 1-1.5 J/cm<sup>2</sup>

Top Row: about 0.6-0.9 J/cm<sup>2</sup>  
Ra ~ 0.2 μm max

1,000X MAG  
All images

From J. Linke, FZ Julich



400 pulses

800 pulses

1200 pulses

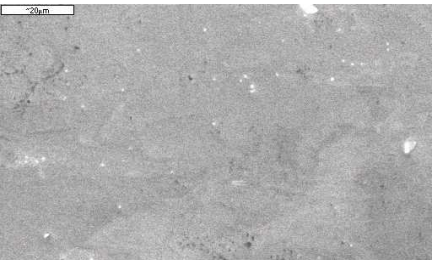
1600 pulses

Bottom Row: ~ 1 - 1.5 J/cm<sup>2</sup>  
Ra ~ 0.35-0.45 μm

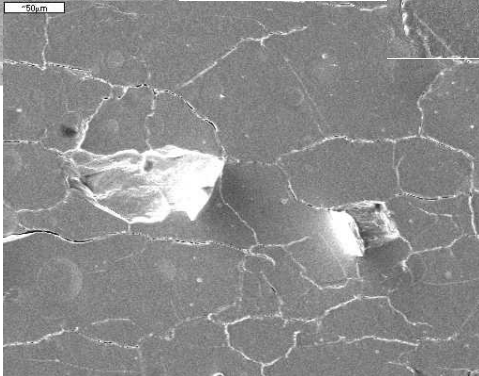
Little or no mass loss to 1600 pulses

## M182 Plansee Tungsten, cut with grains parallel to surface (SEMs): surface-lying grains become unzipped with increasing fluence

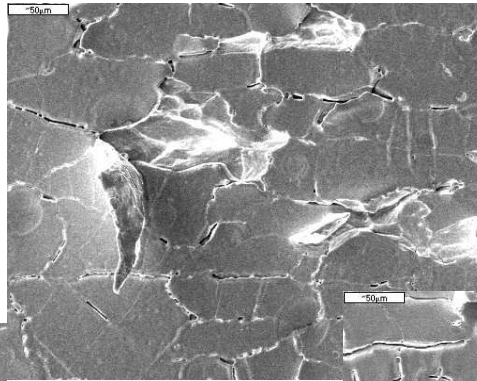
No Treat



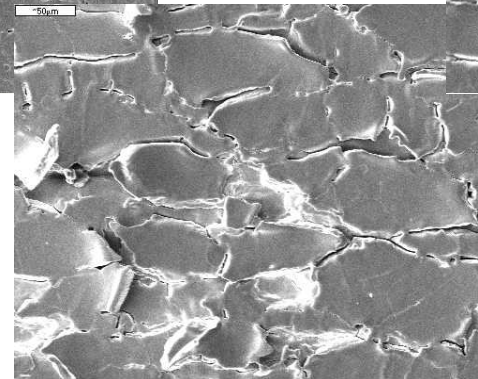
1,000X MAG



~ 0.7  
J/cm<sup>2</sup>



About 1.3  
J/cm<sup>2</sup>



All treated images 300X MAG.

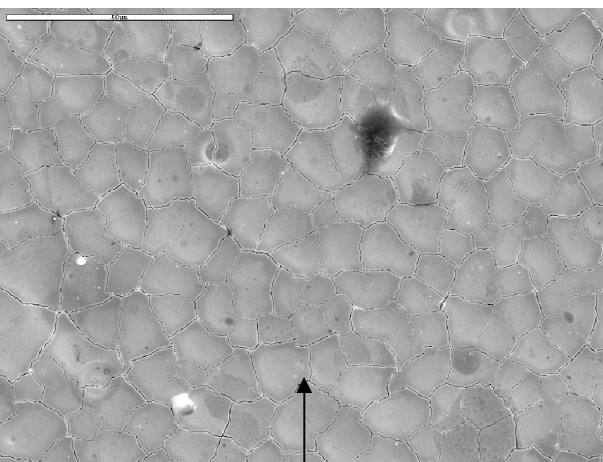
$R_a$  : reaches 4 - 4.5  $\mu\text{m}$  at 1.3 J/cm<sup>2</sup> (same as PM Tungsten)  
Only apparent AFTER 400 pulses (these images)



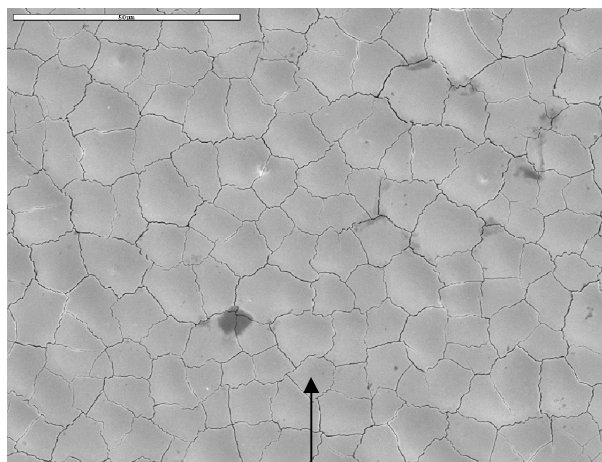
# W-TiC-A 'pre-stressed\*' 2.5 cm-wide sample (SEMs): (presumed) stress-relief seems to restrict grain corner exfoliation

Top Row ~ 1.0 J/cm Ra ~ 0.16  $\mu$ m

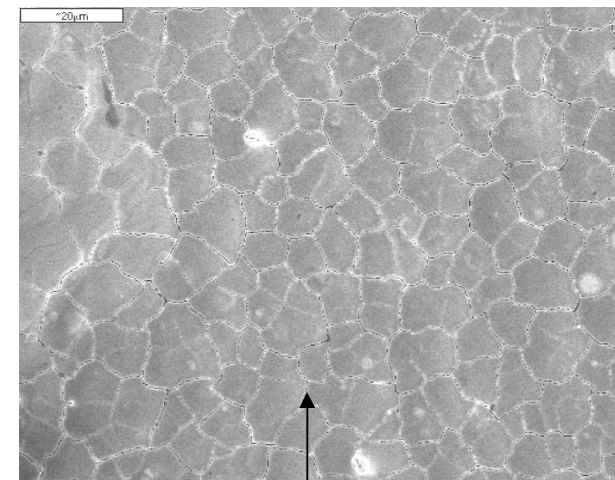
From H. Kurishita (Tohoku U.)



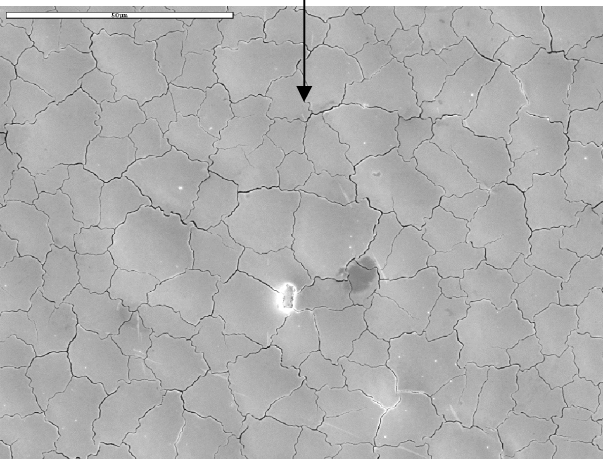
400 pulses



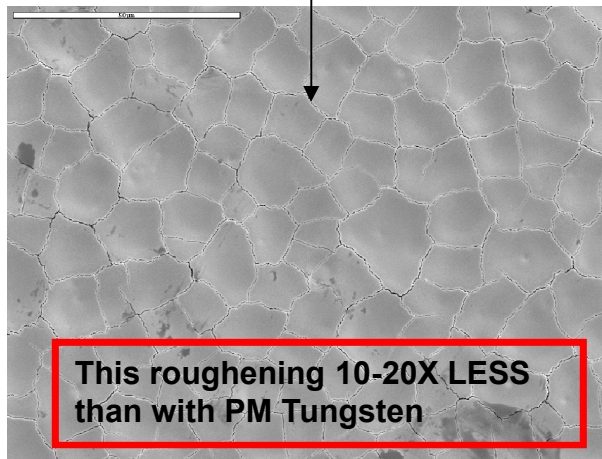
800 pulses



1200 pulses

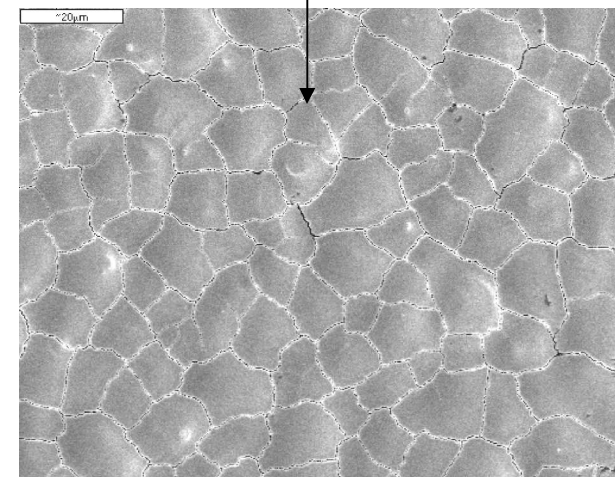


~ 1.4 J/cm<sup>2</sup>



This roughening 10-20X LESS  
than with PM Tungsten

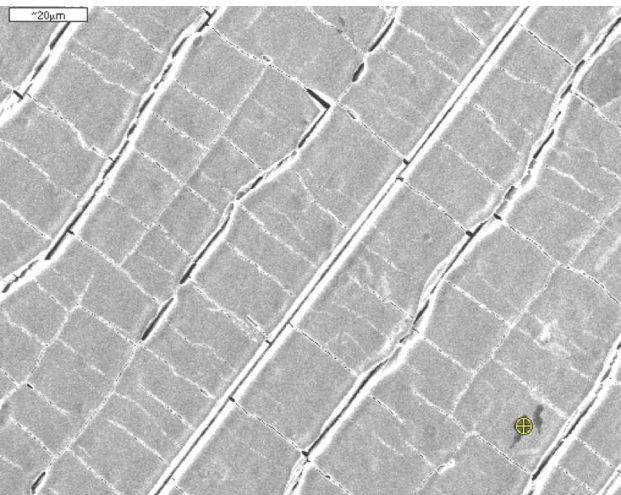
All images -  
1,000X MAG



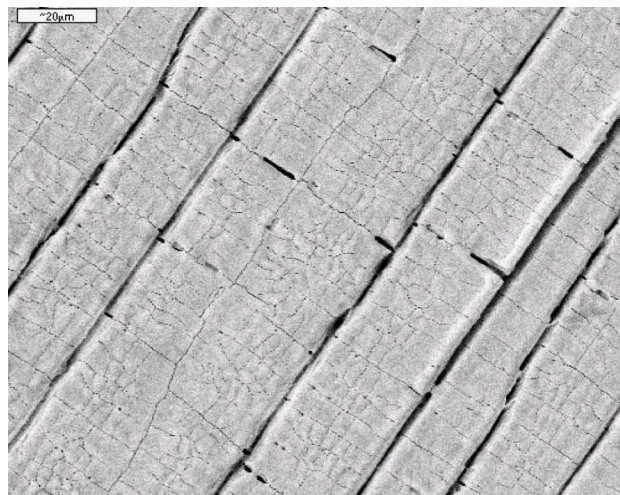
Right Two ~ 1.7 J/cm<sup>2</sup>  
Ra ~ 0.28  $\mu$ m

## RHEPP-1 Surface Roughening

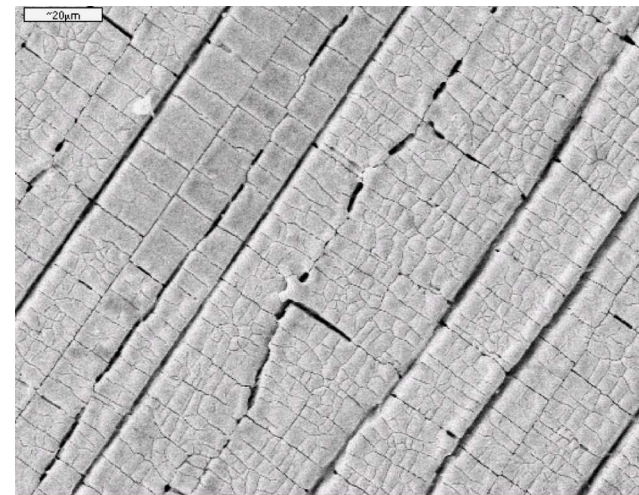
**SEM, SingXtal RT,  $\sim 1.1 \text{ J/cm}^2$  : longitudinal cracks  
form, width stays constant, settling in between**



**400 pulses**

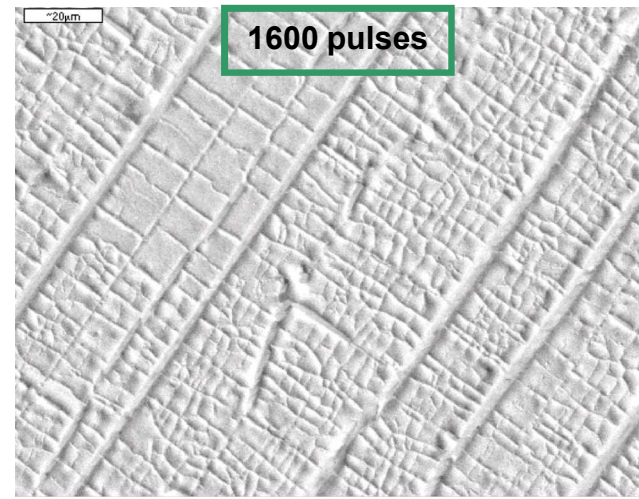
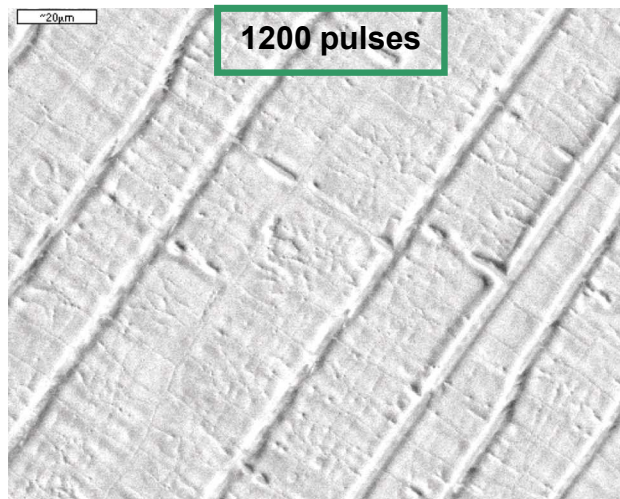


**1200 pulses**



**1600 pulses**

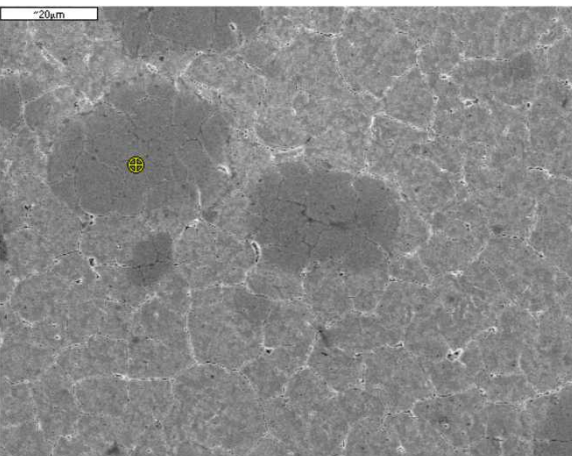
**BSE Images**



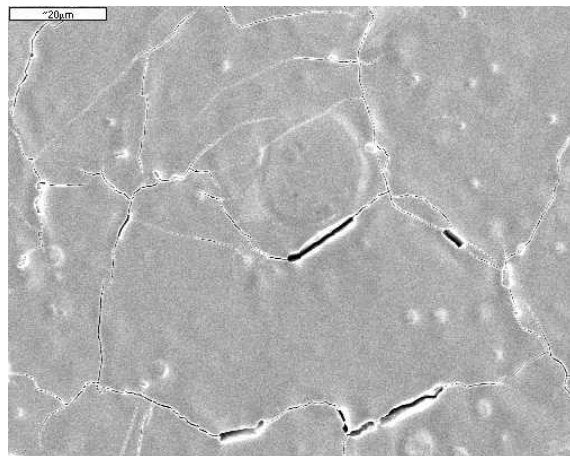
Three forms of Tungsten, treated at ~ same fluence (400 pulses):  
Grain-refinement/strengthening, or below-surface burial  
seem to restrict roughening/mass-loss.

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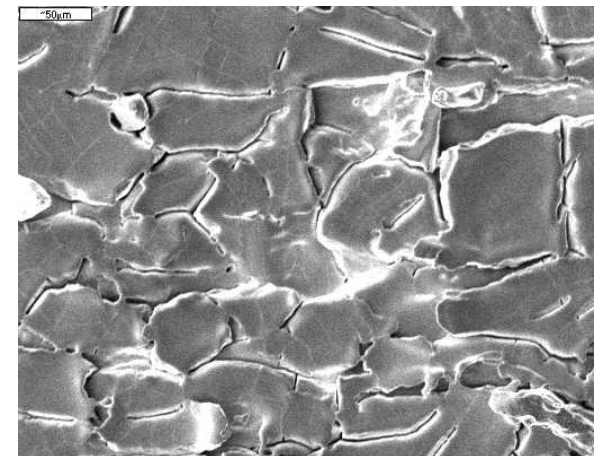
1,000X MAG



1,000X MAG



300X MAG



W-0.5%TiC 1.5 J/cm².  
Ra = 0.04 µm

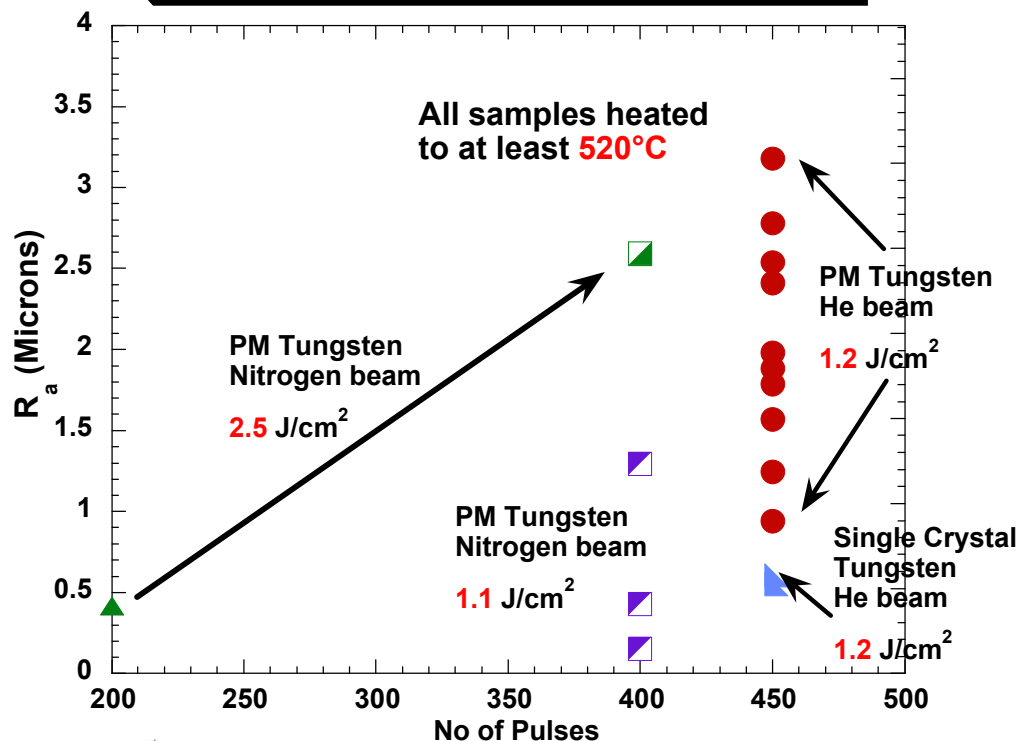
M182Perp ~ 1.25 - 1.5 J/cm²  
Ra ~ 0.15 µm

M182Parallel ~ 1.3 J/cm²  
Ra ~ 4.5 µm

Two on right are SAME material

## Comparison of $R_a$ Roughness, PMW: He beam produces more roughening with the same fluence ( $\sim 1.2 \text{ J/cm}^2$ )

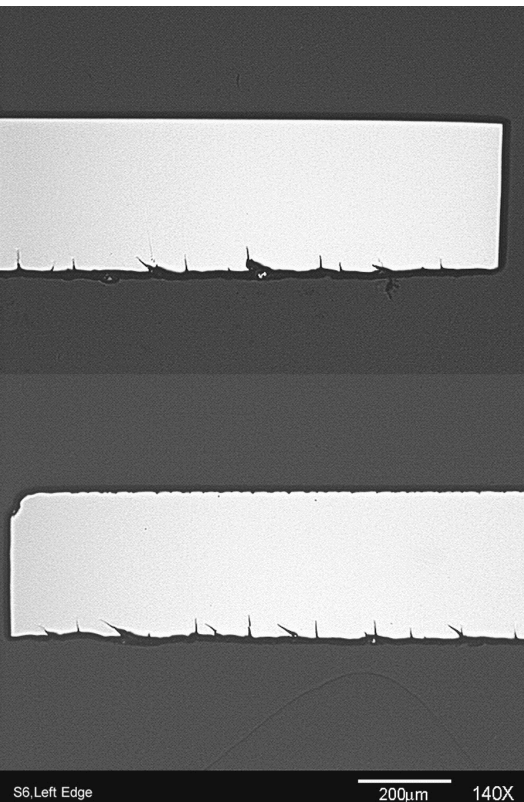
- PM W He beam 450 pulses  $1.2 \text{ J/cm}^2$
- ▲ Single Crystal W He beam 450 pulses  $1.2 \text{ J/cm}^2$
- ▲ PM W N beam 200 pulses  $2.5 \text{ J/cm}^2$
- PM W N beam 400 pulses  $2.5 \text{ J/cm}^2$
- PM W N beam 400 pulses  $1.1 \text{ J/cm}^2$



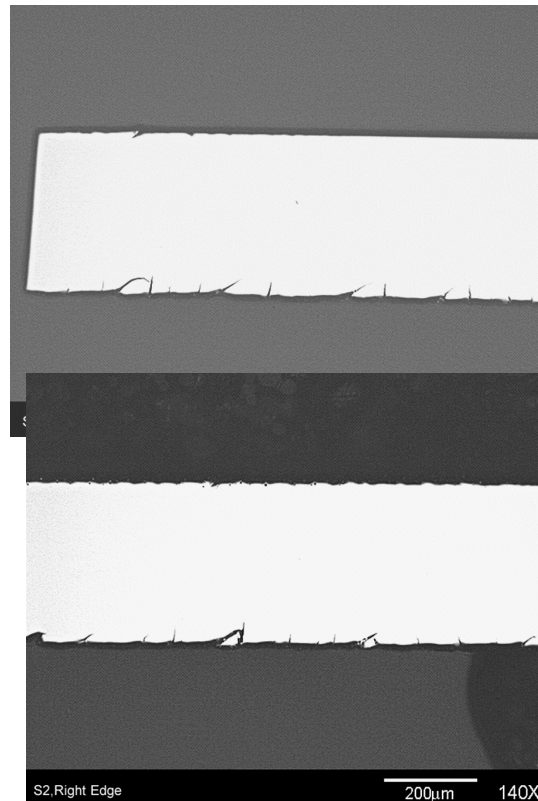
- Red Dots - PM W exposed to He beam,  $1.2 \text{ J/cm}^2$ , 450 pulses
- Purple Squares - PM W exposed to N beam, 400 pulses,  $1.1 \text{ J/cm}^2$
- He beam roughens PM W worse than N beam (note data scatter). Roughness similar to N beam at  $2.5 \text{ J/cm}^2$  (above melt threshold)
- SingXtal W (pink triangles) shows much less roughening at 400 pulses

# Single-Crystal Tungsten, 400 and 1600 pulses: No fatigue-cracking in-depth

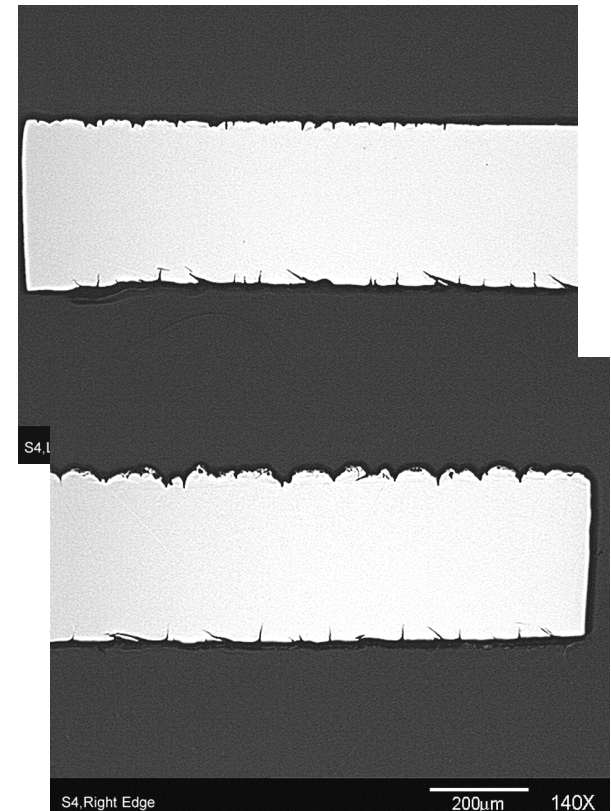
1.2 J/cm<sup>2</sup> 400X Upper 1600X lower



1.9 J/cm<sup>2</sup> 400 pulses



3.5 J/cm<sup>2</sup> 400 pulses



All 140X MAG

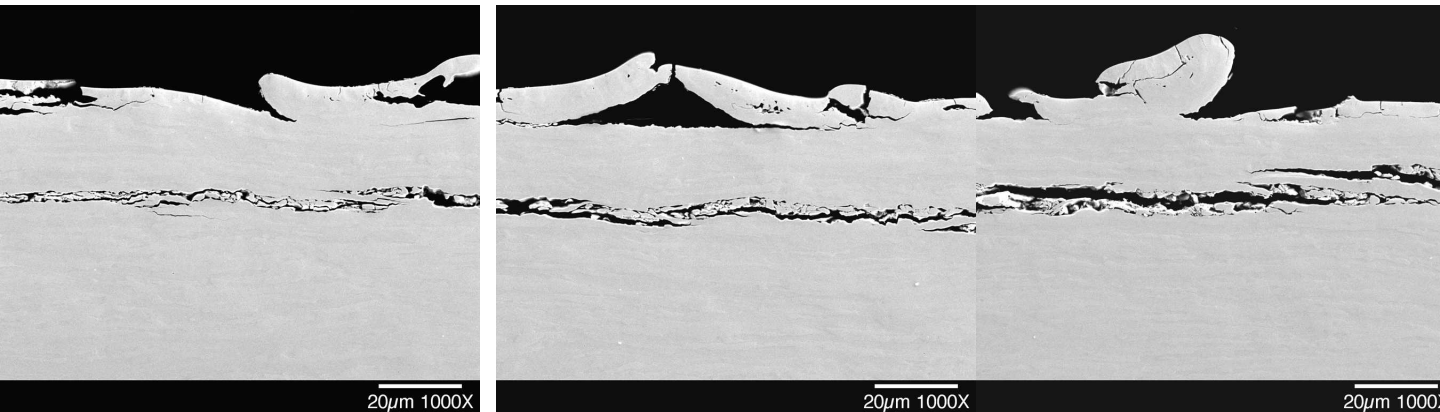
1600 pulses

1600 pulses

Top surface is treated in all cases  
Cuts on bottom surface look like sample prep

## RHEPP-1 Surface Roughening

### PM Tungsten, 1.9 J/cm<sup>2</sup> RT, sectioned SEMs (near Melt): Large distortions in near-surface zone

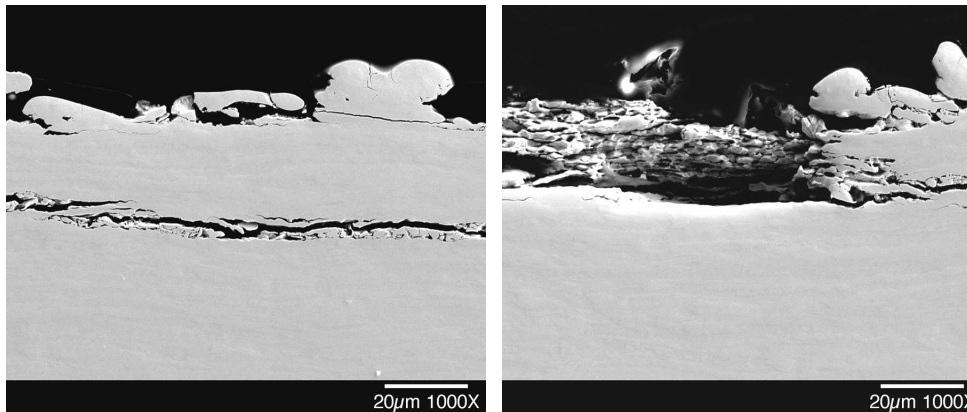


400 pulses

Increasing pulse number



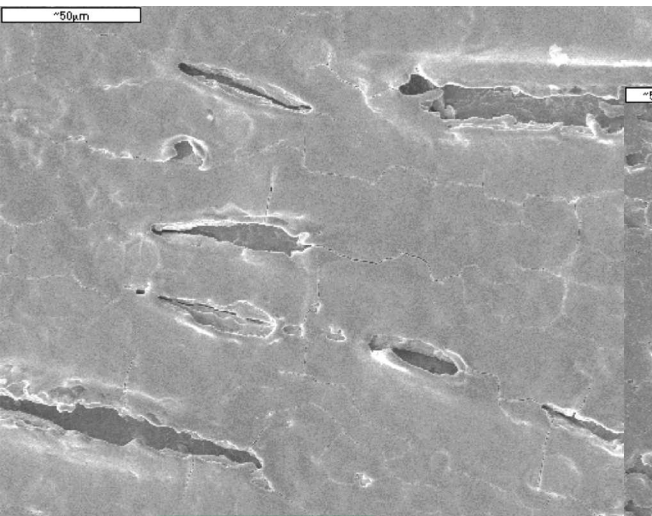
- Wholesale failure down to 20 µm level in last image



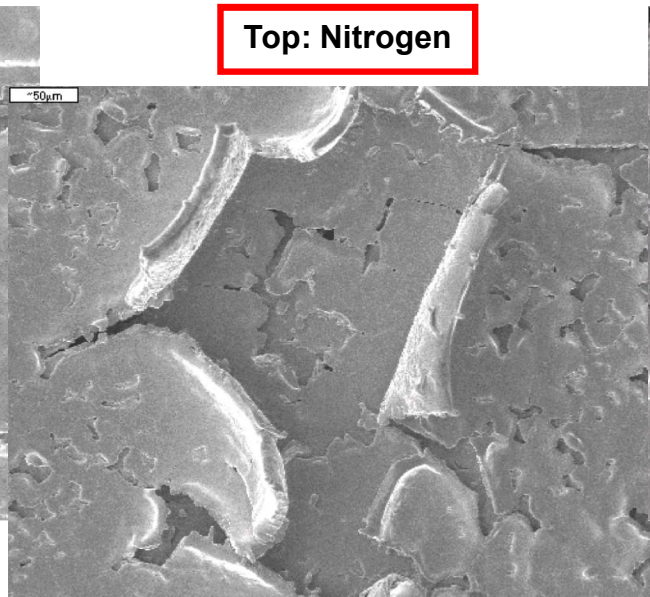
All images BEI 1000X MAG

1600 pulses

# M Tungsten exposed to Nitrogen (top) and Neon (bottom): Surface remains intact with Ne, no mass loss

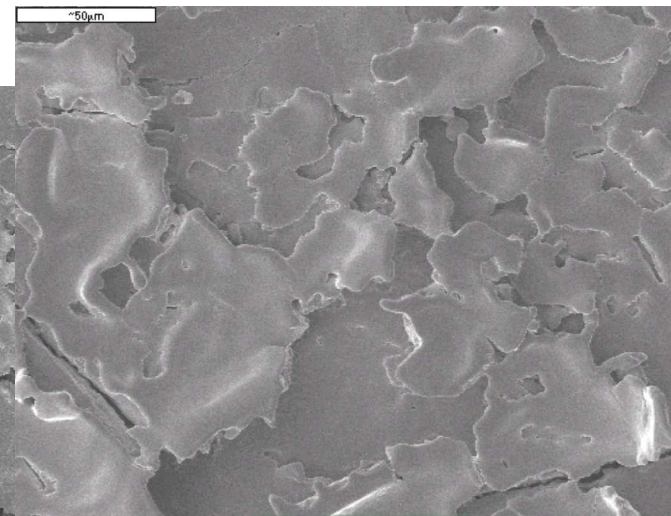


< 1.2 J/cm<sup>2</sup>

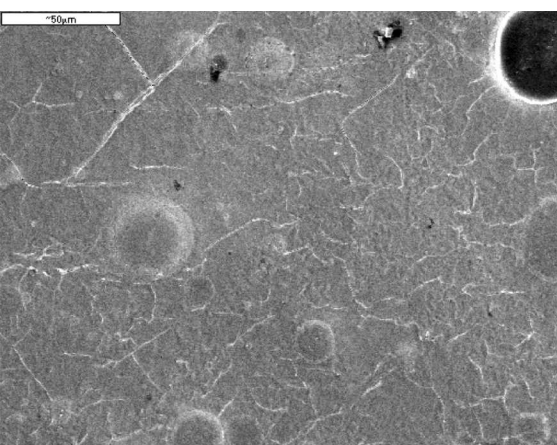


Top: Nitrogen

~ 1.2 J/cm<sup>2</sup>



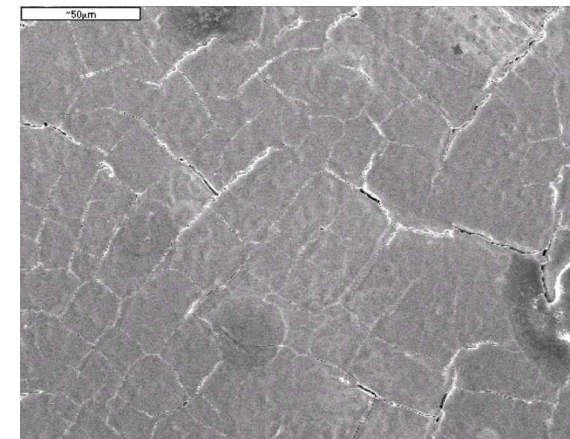
> 1.2 J/cm<sup>2</sup>



All images 500X Mag except above (250X)

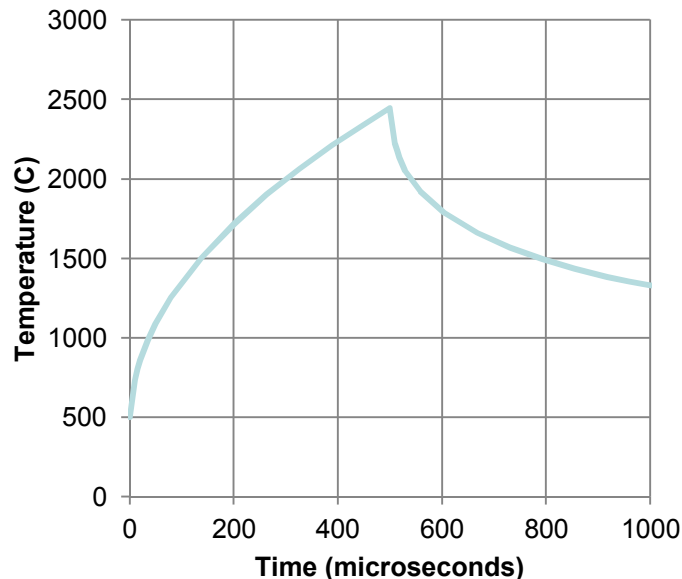
Bottom: Neon

Mass loss: N -306.1 µg, Ne +167.4 µg  
(gain due to entrained Cu)



500X Mag

## Fracture Modeling: Comparison of Tungsten exposed to IFE and MFE ELM Conditions

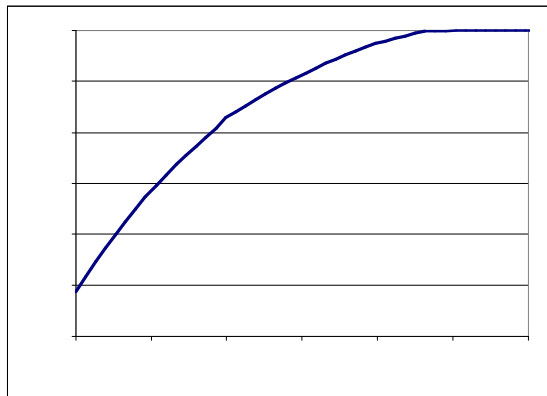


- 3 mm Tungsten on ferritic steel exposed to single heat pulse. Fluence: 0.7 MJ/m<sup>2</sup> over 500  $\mu$ sec.  $T_{\text{initial}} = 500\text{C}$ , Tungsten properties from ITER Material Properties Handbook.

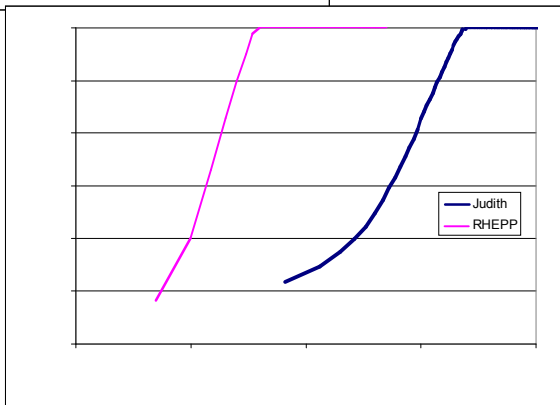
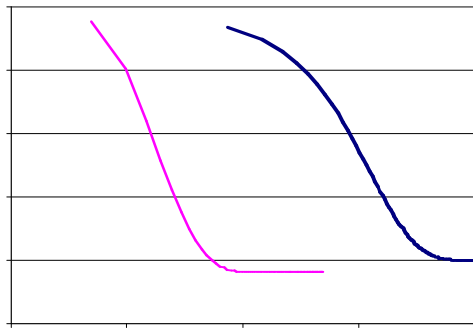
- Heat deposited at surface

- (Top Left): Surface temp reaches ~2500C

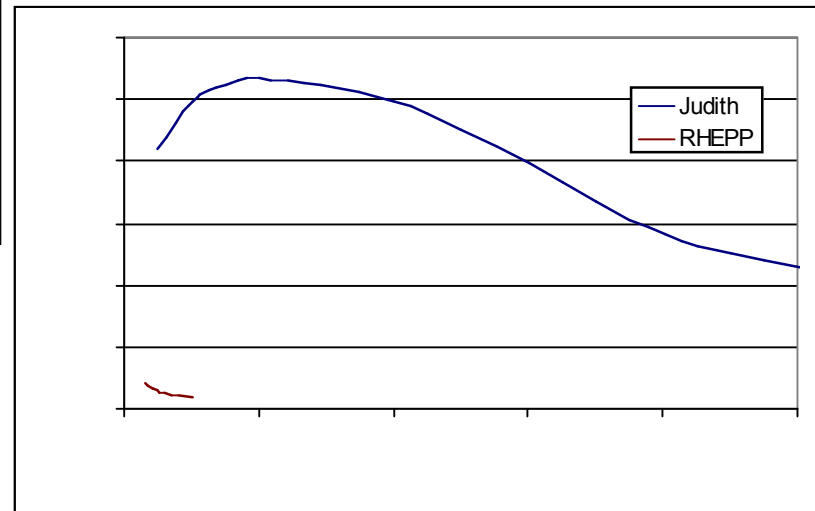
- (Bottom Left): Plastic Strain reaches 1%, gradient to >250  $\mu$ m depth



## Unlike RHEPP heating, the ELM-like pulse produces fracture stresses at the fatigue crack threshold after one pulse



- (Top Left) RHEPP pulses with fluence chosen to reach same surface maximum temperature - 2500C
- (Mid Left) Plastic Strain Curves: Both effects MUCH deeper for the ELM case
- Bottom: Stress Intensity for the 'ELM' case at 25  $\text{MPa}\cdot\text{m}^{0.5}$  - at fatigue cracking threshold for tungsten (20-40  $\text{MPa}\cdot\text{m}^{0.5}$ ). RHEPP at  $\sim 2 \text{ MPa}\cdot\text{m}^{0.5}$
- This could explain why RHEPP thermomechanical effects take hundreds of pulses to develop.



## What if we allow the surface to melt? Will this smooth it out? Higher Fluence Test

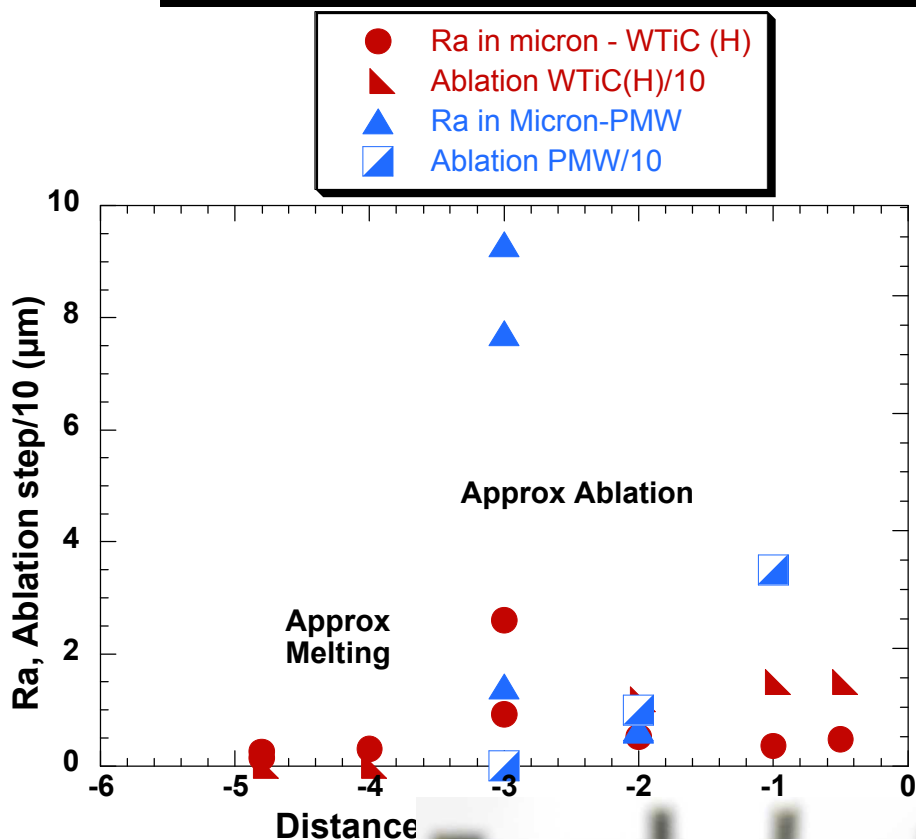
Samples mounted before Start  
Beam Center off to Right



- Samples mounted at Left:
  - M182Perp
  - W-0.5%TiC-Argon (Kurishita)
  - W-0.5%TiC-Hydrogen (Kurishita)
  - W25%Re
  - PM Tungsten
  - Single Crystal W
  - Mo
  - Nb
  - Cu 3 9s
  - Cu 5 9s
- 'Normal' exposure towards Left. Beam center at right ( up to 10 J/cm<sup>2</sup>)
- PM Tungsten strips used to mask samples
- Vacuum ~ 1e-5 Torr

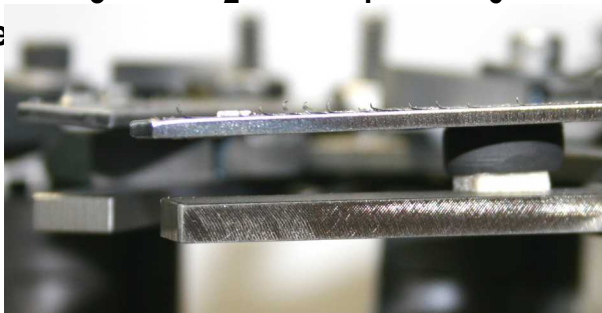
Nitrogen beam

## Roughening behavior of W-0.5%TiC(H), PM W at high fluence: $R_a$ highest at ablation onset. Melting leads to increased roughness

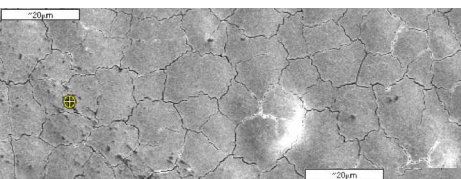
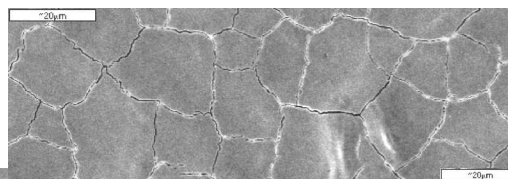
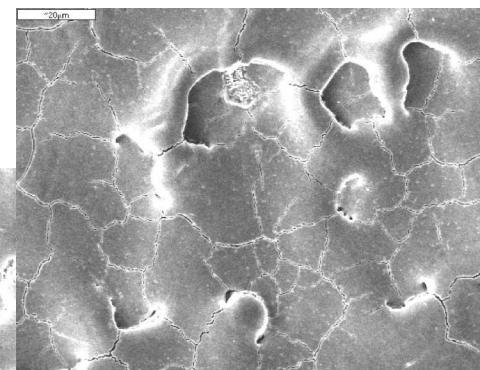
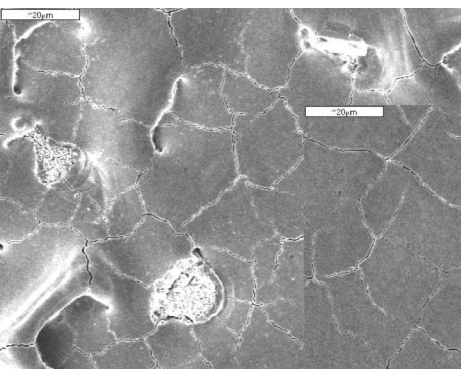
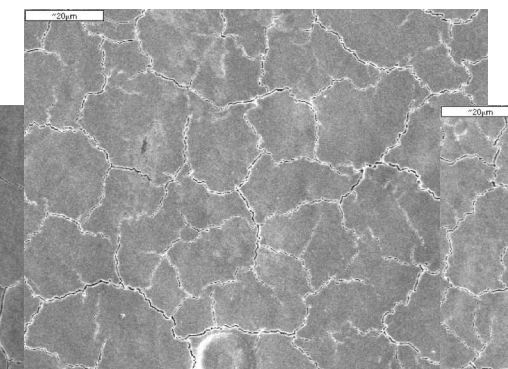


- Two each W-0.5%TiC (H) and PM Tungsten (Snead) samples exposed in fluence range from melting to above ablation threshold (per pulse).
- Sample melt leads to much higher roughness ( $R_a$  reaches 2.5  $\mu\text{m}$  for WTiC, 9.5  $\mu\text{m}$  for PM W)
- Roughness reduced beyond ablation threshold, but 15  $\mu\text{m}$  (WTiC) to > 35  $\mu\text{m}$  (PMW) material removed after 400 pulses
- Similar behavior for Mo, Cu, Nb. Ablation steps exceeding 35  $\mu\text{m}$  observed (> 900Å/pulse removed)
- BIG surprise: W25Re. Hardly ANY roughening.
- (LEFT): 'fingers' protruding from W shield

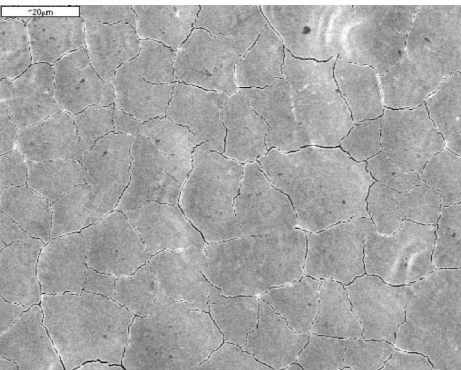
Nitrogen Beam



**SEMs, W0.5%TiC (H), S4.8 to BEAM CENTER:  
Same roughness at both extremes, 15  $\mu\text{m}$  Step at R = 1 cm (400 pulses)**

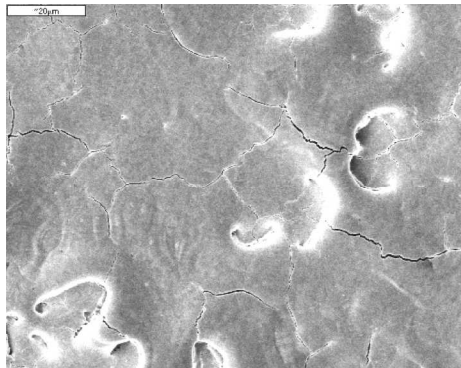
**R = 4.8 cm****R = 4.0****R = 3.5****R = 3.25****R = 3.75****R = 2.75****R = 1****>15  $\mu\text{m}$  Step****R = 3****R = 2****12  $\mu\text{m}$  Step****15  $\mu\text{m}$  Step****R = 0**

## SEMs, W0.5%TiC - Argon: More brittle destruction evident compared to WTiC (H)

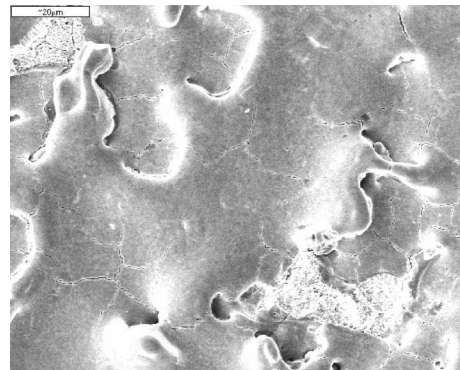


R = 5 cm

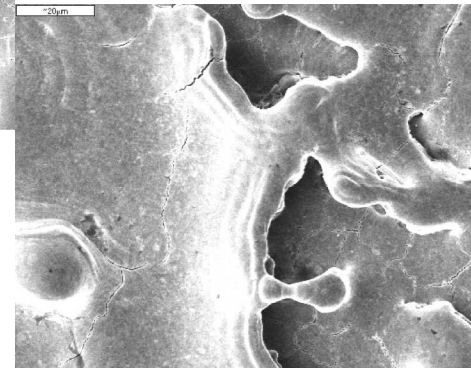
R = 4.5



R = 4.25



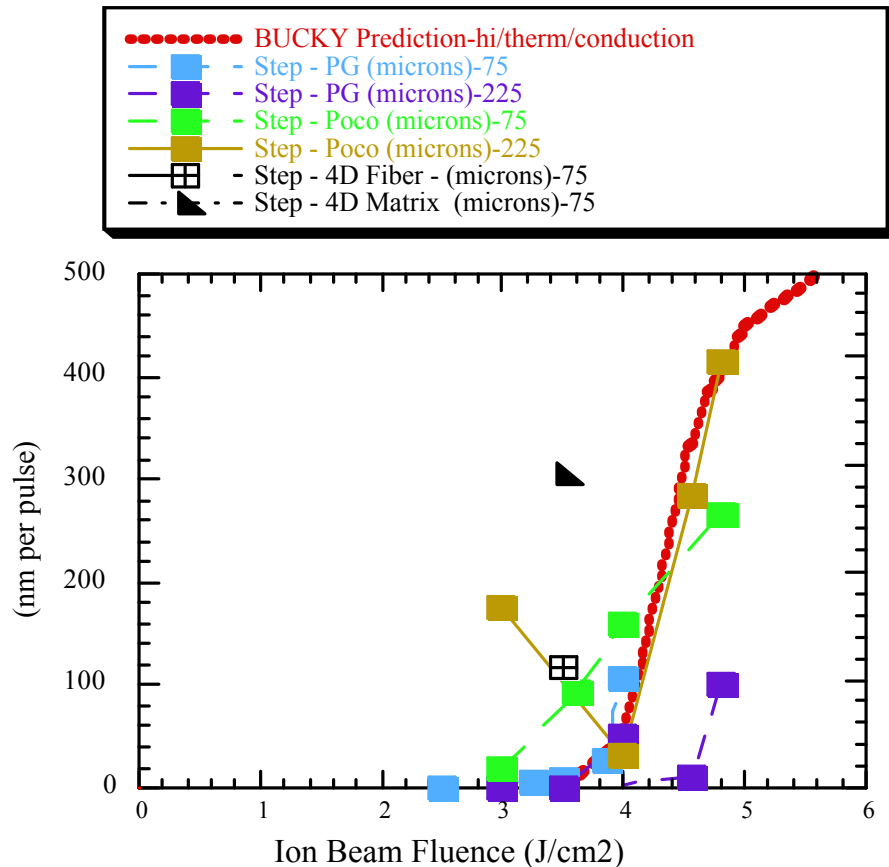
R = 3.5



~ 2 µm Step

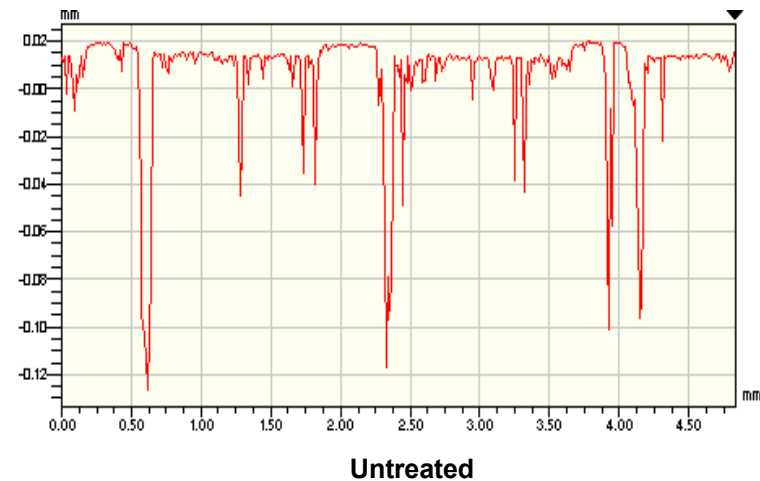
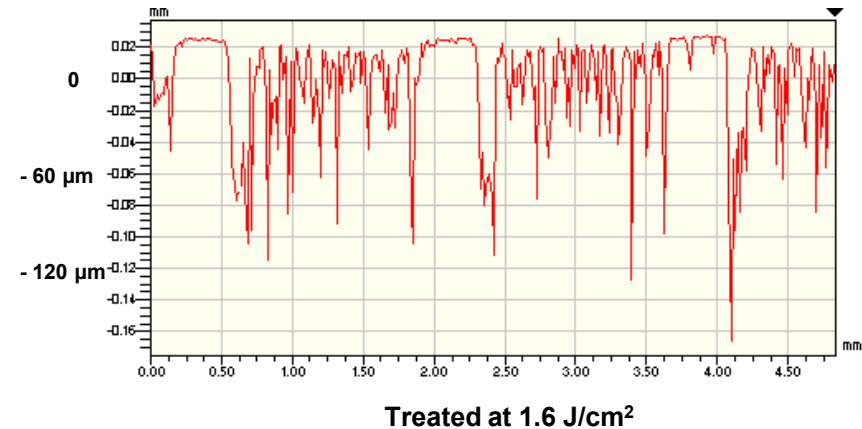
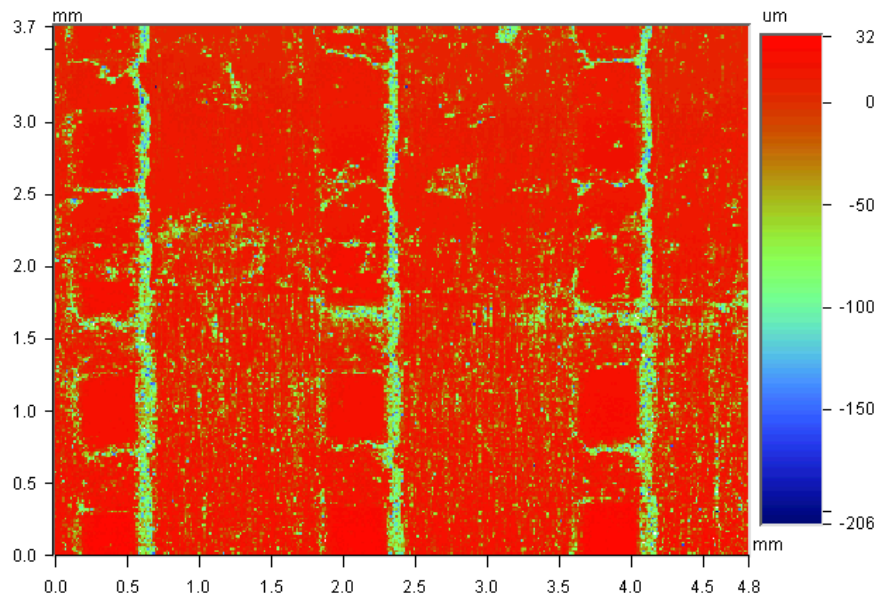
# Carbon Materials

## Early exposures with H - C beam qualitatively confirms BUCKY predictions of 3.5 J/cm<sup>2</sup> ablation onset



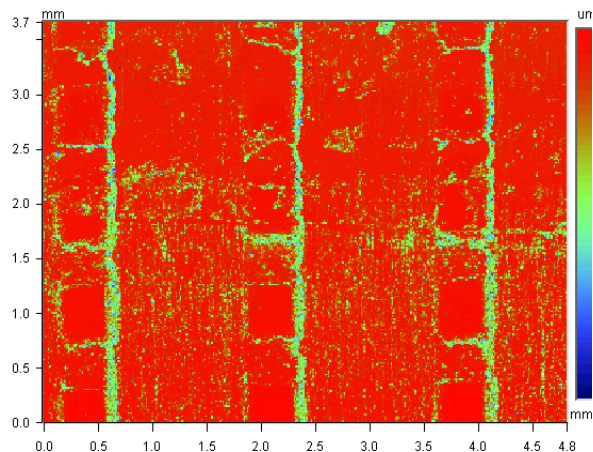
- Mechanically polished pyrolytic graphite (PG), POCO, and 4D carbon composite weave exposed to 75 pulses/225 pulses of 70% C /30% H beam at doses of 1.9 to 5 J/cm<sup>2</sup>
- PG ablation threshold ~ 4 J/cm<sup>2</sup>
- Poco ablation threshold ~ 3 J/cm<sup>2</sup>
- Above threshold, rapid increase in ablated material per pulse with dose. Data scatter reflects uncertainty in dose
- Composite matrix ablates more than PG/Poco, fibers comparable (sample rough)

# FMI-222 unheated CFC exposed to MAP N for 1000 pulses at 1.6 J/cm<sup>2</sup>: Significant erosion of matrix

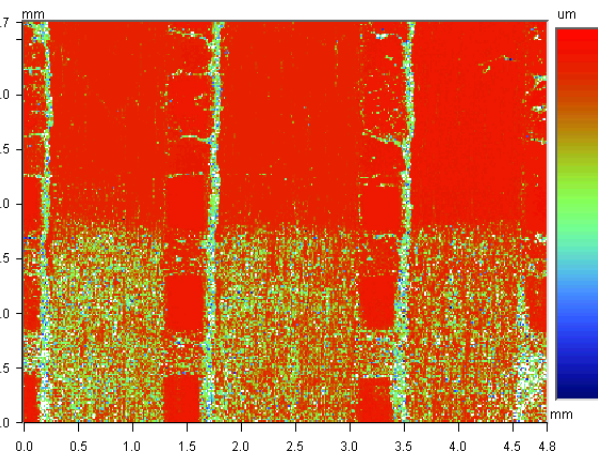


# FMI-222 Fiber ends appear ablation-resistant; Matrix loss $\sim 0.3 \mu\text{m}/\text{pulse}$ at $4.0 \text{ J}/\text{cm}^2$ :

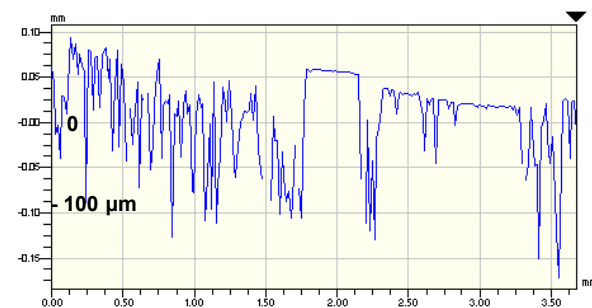
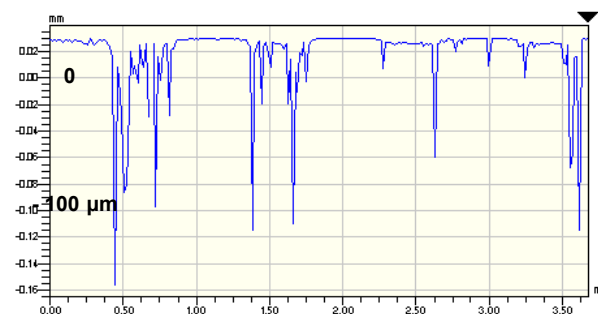
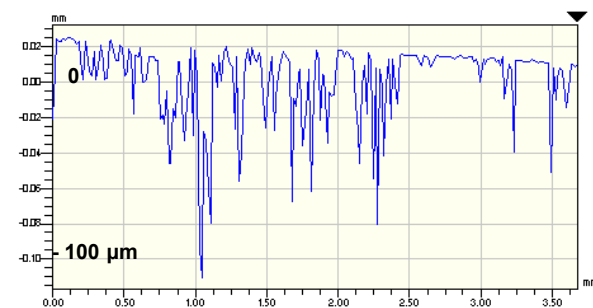
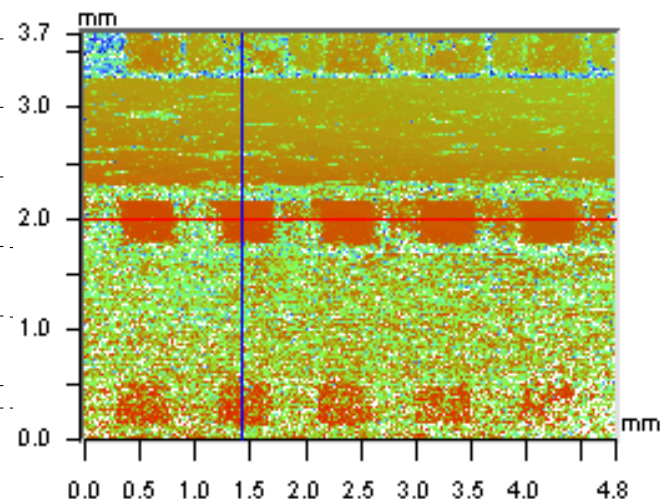
Treated at  $1.6 \text{ J}/\text{cm}^2$  1000 pulses



Treated at  $2.6 \text{ J}/\text{cm}^2$  600 pulses



Treated at  $4.0 \text{ J}/\text{cm}^2$  600 pulses

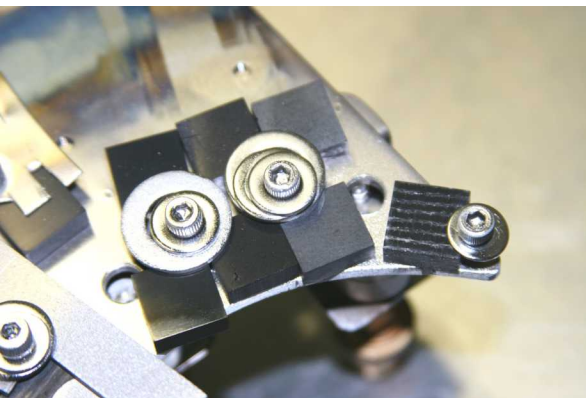


## Several carbon samples from Juelich exposed to 400 pulses @ 1-1.25 J/cm<sup>2</sup>: All lose less mass than PM tungsten

Samples mounted (3, HI and LO)  
before Start, Beam Center off to Right

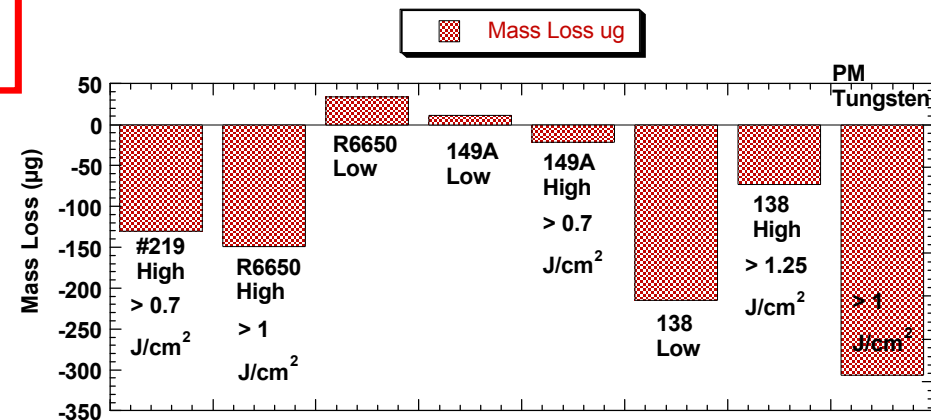


(Above) REFERENCE  
PM Tungsten between  
screws



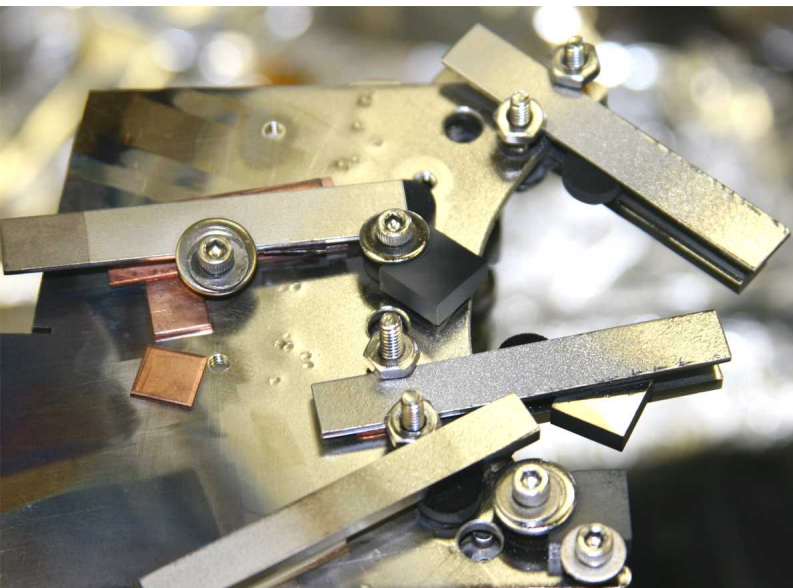
(Above) After 400 shots

- Several C samples exposed at HI (above) PM W 1 J/cm<sup>2</sup> level, and LO (below), as per picture at left.
- Samples:
  - 219 - CFC, PITCH/PAN fibers, HI only
  - R6650 - isotopic fine grain graphite
  - 149A - Ti-doped RGTi from Russia
  - 138 - unidirectional perp CFC (MFC1) from Japan
- All these lost LESS mass at LO and HI than PM W after 400 pulses (below)
- Mass GAIN below due to Cu contamination due to Beam. Not known why 138 HI lost less mass than 138 Lo.



MAP Nitrogen

## Higher Fluence exposure of Juelich Carbons: Beyond 1.3 J/cm<sup>2</sup> fluence/pulse leads to significant mass loss



Samples mounted for higher fluence compared to C400 (Below). Tungsten shields extend towards beam center (right)

- **Samples:**
  - R6650 Repeat at 1.3 - 1.5 J/cm<sup>2</sup>
  - Pyrolytic Graphite cut perpendicular to C-Planes: 2 - 4 J/cm<sup>2</sup>
  - 149A - Repeat at 1.5 - 2 J/cm<sup>2</sup>
- **Results:**
  - R6650 roughened from 0.18 to 3  $\mu\text{m}$ , 2  $\mu\text{m}$  step even at 1.3 J/cm<sup>2</sup> (50Å ablative loss/pulse)
  - Pyrolytic: almost 4  $\mu\text{m}$  step at 2 J/cm<sup>2</sup>, beyond measurement ability at 4 J/cm<sup>2</sup>
  - 149A: roughened from 0.15 to 0.6  $\mu\text{m}$ , mass loss likely but not confirmed



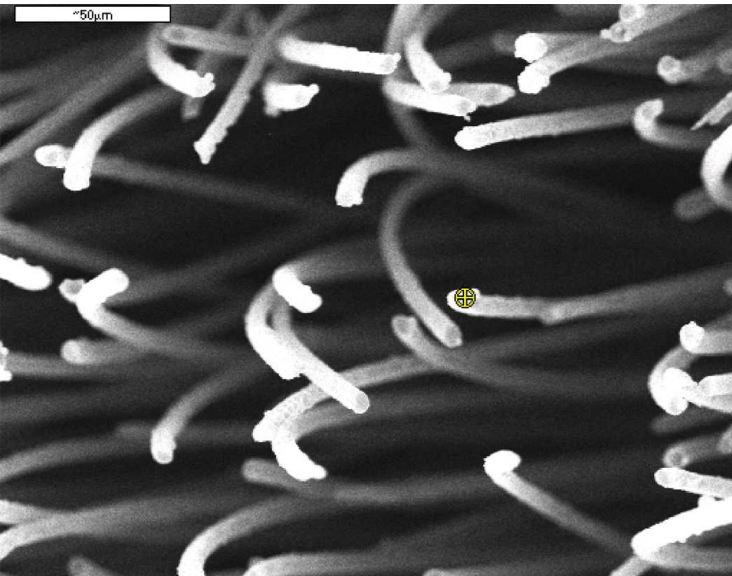
Earlier C400 Series  
(from Previous Slide)

## RHEPP-1 Surface Roughening

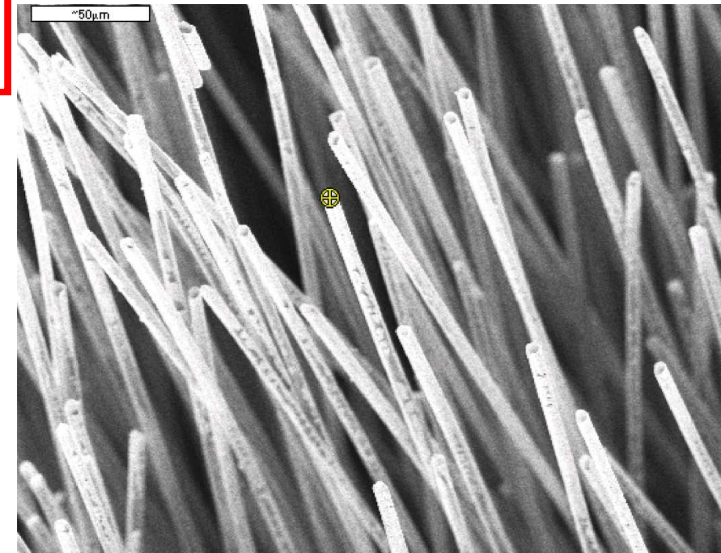
# Tungsten-coated Carbon Velvet survives 1600 pulses amazingly well

From T. Knowles, ESLI

Carbon PAN fibers w/ 1.6  $\mu\text{m}$  W coating,  
2% areal coverage



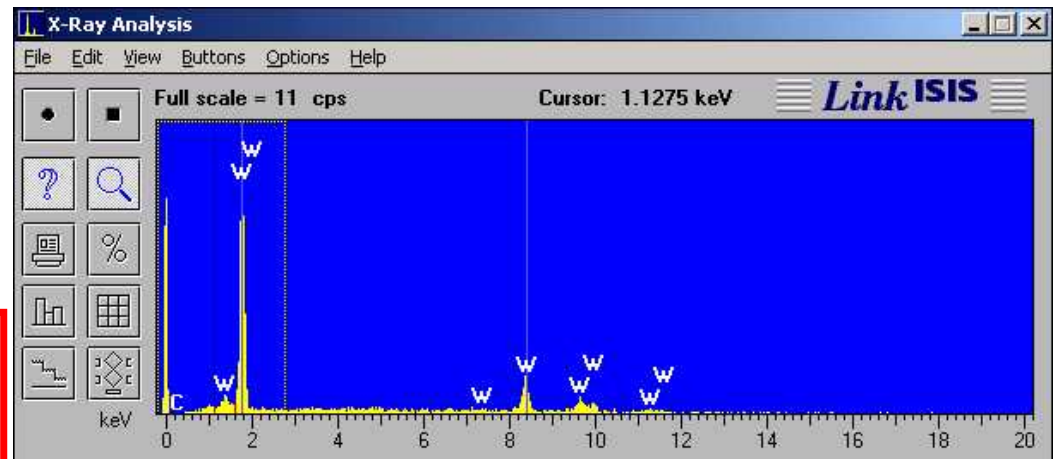
(RIGHT)  
520C (nominal), 1600  
pulses, 1.5 J/cm<sup>2</sup>/pulse  
  
NOTE: W remaining  
on tips (see below)  
and sides



(ABOVE)  
RT @ ~2.8 J/cm<sup>2</sup>, 1600 pulses

NOTE: bent tips, flat ends have W  
removed, rounded ends still have W

This reinforces recent JUDITH result:  
Mechanical strength of PAN fibers may  
more than make up for their lower Thermal  
Conductivity compared to PITCH



EDS scan of tip (cross): W rich

## Exposure Results Summary\_1

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- **Tungsten:**
  - Surface roughens due to thermomechanical stress, occurs **BELOW** the melt point. Relief develops over hundreds of pulses. Heating to 500C, i.e. above the DBTT, delays but does not stop this process.
  - Roughening Threshold for multi-pulse exposure  $\sim 1 - 1.2 \text{ J/cm}^2$  - corresponds to  $\sim 1400 - 1700\text{C}$  maximum surface temperature.
  - Grain size and orientation very important. Worst form is polycrystalline lightly-deformed, - develops high relief ( $5+ \mu\text{m } R_a$ , up to  $100 \mu\text{m P-V}$ ). This relief is linked to confirmed mass loss.
  - Deformed tungsten with grains perpendicular to surface (M182) suffers much less roughening, as does W-25Re alloy (not shown).
  - W-0.5%TiC (Kurishita) - fine grain Tungsten shows robust survivability, low roughening. Processing with Hydrogen seems to improve grain boundary strength compared to Argon, as predicted.

## Exposure Results Summary\_2

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- **Graphite:**
  - Some forms of graphite, e.g. R6650, MFC-1, 149A, show little/no mass loss at  $1 \text{ J/cm}^2$ , i.e. at higher than roughening threshold for tungsten. This corresponds to surface temperature of 1800K, according to 1-D BUCKY modeling of high-thermal conductivity graphite.
  - Above this point, however, ablation/sublimation result in significant roughening and rapid mass loss.
  - These values are WELL below BUCKY modeling of sublimation threshold for high-conductivity graphite, -  $3.5 \text{ J/cm}^2$ .
- ‘Engineered’ surfaces like ‘Velvet’ may represent the best solution for surface survivability. Robust PAN fiber survivability consistent with recent JUDITH findings that use of PAN limits brittle destruction see in PITCH fibers.
- Fracture Modeling indicates 1) single RHEPP pulses are well below threshold for Tungsten surface cracking, and 2) single ELM-like pulses produce much deeper strain and are at or near the Tungsten fracture threshold

# Future Work

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- **No Dry-wall solution yet for full-exposure condition, although some promising materials (M182p, W-coated Velvet, W25%Re). Cusp - configuration could allow SiC for Main Wall, replaceable materials at ion dump (equator)**
- **Although PM Tungsten roughening reaches saturation, no other form of tungsten has shown similar behavior. So we may not be able to show ultimate survivability in RHEPP experiments.**
- **Need to include He entrainment, possibly neutron effects, in same sample set**
- **Need to study diffusion of contaminants into wall materials. Auger measurements show C diffusion into 520° C heated tungsten deep into near-surface region (tens of microns)**