

We have previously reported on the long-term exposure of flat candidate first-wall armor materials (up to 2400 pulses of energetic helium ion fluences) on the RHEPP-1 ion beam facility at Sandia National Laboratories. Initially mirror-finish forms of tungsten, exposed either at room temperature or preheated up to 600°C, have evolved various forms of surface relief and cracking (up to 100 μm peak-valley) when exposed below their melting temperature. The powder-metallurgy (PM) form of tungsten has evolved the prominent surface relief, evidently from the thermomechanical instability of the (large) surface grains. Some three-dimensional 'engineered' materials such as 'needle' arrays of both carbon and drawn tungsten have demonstrated promising robustness and resistance to surface cracking and mass loss. The long dimension of these materials is aligned with the direction of energy flow. Since the needle widths are on the order of few to tens of microns, we suspect that the improved robustness is due to either/both of 1) reduced effective heat load on the surface, or 2) improved mechanical properties of the micro-engineered materials (e.g. reduced grain size, aligned grains, etc).

We have recently exposed various forms of textured materials¹ manufactured using chemical vapor deposition, and containing W, Re, and Mo in the form of cone or rounded nodule shapes in various combinations. Samples of 1 cm² size with feature sizes 3-30 μm have shown robust survival at fluence levels that have shown severe roughening on flat PM tungsten. Examination of the surface appearance (SEM) of the various materials suggests that they do not tend to develop the cracking, pitting, and other features seen in flat large-grained samples. Structures less than 1 μm in size tend to melt readily. Perhaps surprisingly, samples with $\sim 3\mu\text{m}$ feature sizes, and those with thin tungsten coating on Re substrate have shown promising performance. We report here on experiments in which nano-crystalline tungsten thin films are applied to flat substrates with various thermal conductivities (Cu, W, Re, Ti). Goals are a) survivability evaluation of fine-grain W compared to PM tungsten, and 2) study of effect of substrate on sample survivability. Treated samples are examined by scanning electron microscopy (SEM) and sectioning. Latest results will be presented.

¹Supplied by Utramet, Pacoima, CA 91331.

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Presentation Outline

- IFE Direct-drive Threat Spectra: why 3-D wall may be necessary.
- RHEPP-1 can replicate thermal loading to first-wall (154 MJ yield as example)
- Flat-wall material performance*
- Surface tilt as threat mitigation - needles as a geometry*. Mo-coated needles: very robust performance
- 3-D dendritic materials from CVD manufacture. SEM surface views after 400 - 800 helium pulses at various per-shot fluences show good survivability
- Flat substrates coated with Mo and W thin films. First look.

Publications:

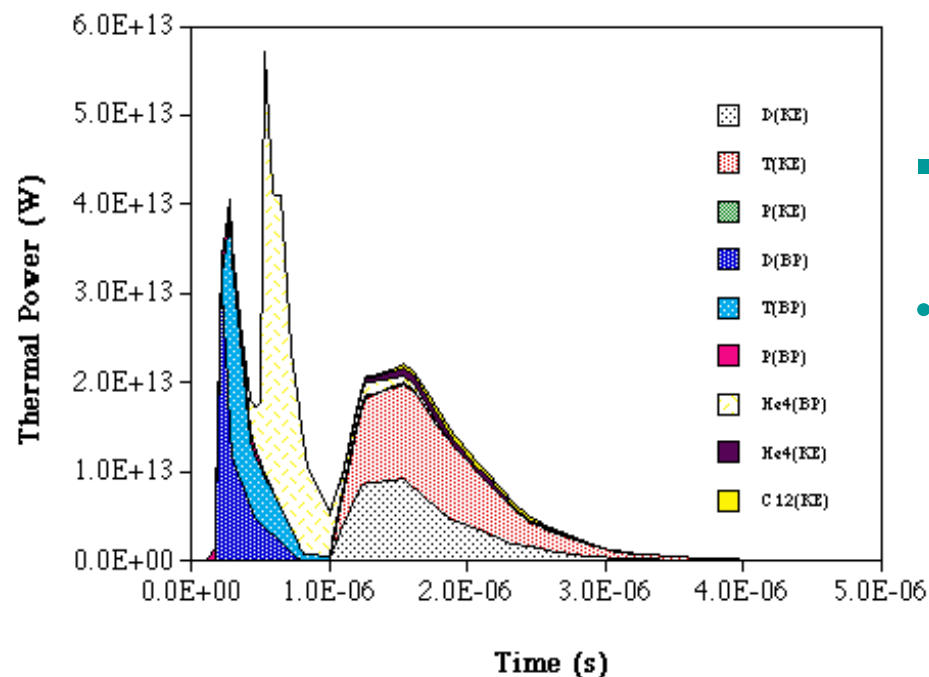
- 1) T. J. Renk *et al*, Survivability of first-wall materials in fusion devices: an experimental study of material exposure to pulsed energetic ions, publication 1/12 in Fusion Science & Technology.
- 2) T. J. Renk and B. Williams, Three-dimensional 'textured' coatings as first-wall materials: exposure to energetic ions on RHEPP-1, publication 10/11 in Fusion Science & Technology

*This work performed within the High Average Power Laser Program, funded by NRL (DOE NNSA DP).

Laser IFE Direct Drive Threat Spectra: Ions, neutrons, He retention

Data courtesy Rene Raffray

Time-of-Flight Ion Power Spread



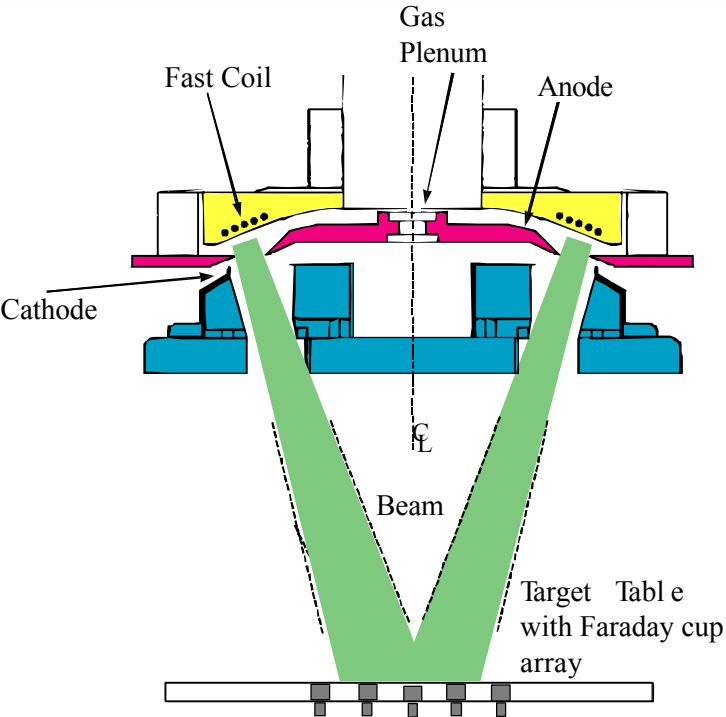
Simulation: Thermal Power to Wall in Ions
from 154 MJ Yield. Wall Radius: 6.5 m

- For **Direct-Drive Laser IFE**:
 - ~70% neutrons - not discussed here
 - 1-2% x-rays - not considered a threat
 - **30% ions (50-50 fusion and 'debris')**
- Ions: several MeV, ~ few μ sec each,
8-20 J/cm² fluence - significant threat
- **Dry wall Ion Issues (@ 10 Hz, 3e8 pulses/yr):**
 - **Thermomechanical stress** - roughening below melt, mass loss. Addressed in prior RHEPP experiments.
 - **Helium retention.** ~1e18/cm² pore formation threshold for pitting formation seen in prior experiments. This reached well before 1 yr.
 - **Tritium co-deposition:** mitigated by elevated wall temperature, reduces operating headroom.
 - **Neutron embrittlement:** Not addressed here. Partly simulated by ion exposure (surface only). 2000 RHEPP shots ~ 1dpa

@ 10 Hz reactor operation, wall mass loss tolerances must be low

- **10 Hz = 3×10^8 pulses/yr**
- **Assume 6.5 m radius spherical chamber, tungsten armor**
- **Limit linear wall loss to < 2 cm/yr**
- **Then mass loss /pulse must be held to $2/3$ Å or less**
- **@ 2 cm/yr loss, total tungsten mass loss is 200 metric tons**

Overall view of RHEPP-1 vacuum chamber and treatment area

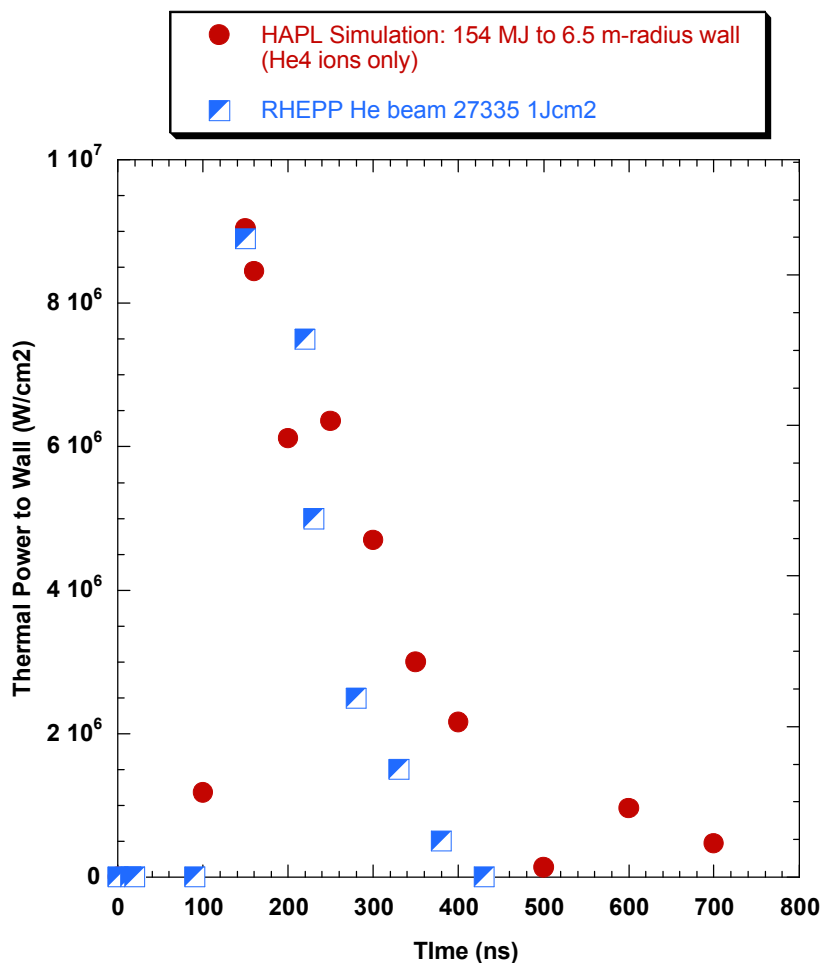


- 600-800 kV. Pulse Width ~ 100 -200 ns
- Up to 250 A/cm²
- Beams from N and He used here – away from center
- Overall treatment area ~ 150 cm²
- Diode vacuum $\sim 10^{-5}$ Torr



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

The RHEPP helium beam well simulates IFE threat from both He ion and total ion current



- RHEPP-1: 700-800 keV, 100-150 ns pulsewidth, 75-95 GW/m²
- **RED (LEFT):** Simulation, 154 MJ IFE yield, at 6.5 meter wall radius, He₄ ions only. **BLUE (LEFT):** RHEPP pulse 27335, He beam, 1 J/cm² fluence, 9 MW/cm² power density.
- He component is ~ 1/3 of total ion pulse (fusion plus debris ions). Total reactor pulsewidth ~ 1.5 μsec FWHM, vs ~ 80 ns Thermal Power FWHM for RHEPP.
- Using Heat Flow Parameter **$F=P*\sqrt{t}$** (takes heat conduction into account), J/cm² needs to be 3 J/cm² to include all ion effects. But shorter delivery increases *effective* RHEPP dose by 4x.
- RHEPP He beam matches pulse heating time for reactor pulse, and 1-2 J/cm² matches overall power delivery to first wall expected for 154 MJ pulse.
- Each RHEPP He pulse @ 1 J/cm² delivers ~ 1.25×10^{13} He/cm², and (from heat flow modeling) produces ~ 1100°C maximum surface temperature

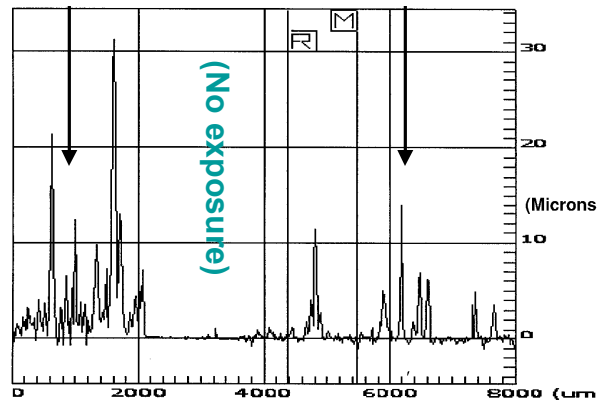
Thermomechanical stress

Reference: polycrystalline (PM) tungsten roughens with number of pulses with both nitrogen and helium ion exposure. Diagnostic: R_a as measured by 1-D profilometry

I-D 8 mm Profilometer Scans, 450 shots He (Left) and Nitrogen (Right) of each plot

He Beam 1.3 J/cm²

N Beam 4J/cm²

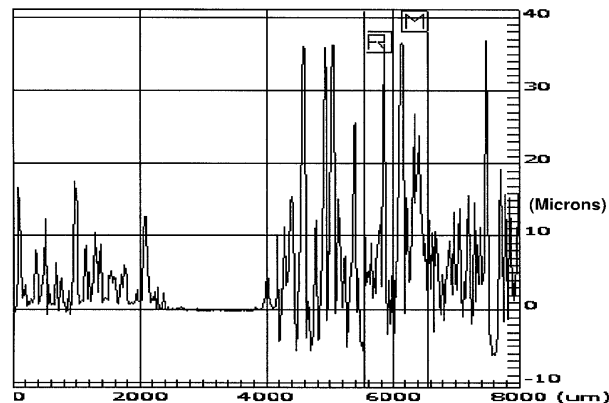


(He-450 pulses)

(N-200 pulses)

$R_a \sim 1-3 \mu\text{m}$
 $P-V \sim 10-30 \mu\text{m}$

$R_a \sim 1-3 \mu\text{m}$
 $P-V \sim 5-15 \mu\text{m}$

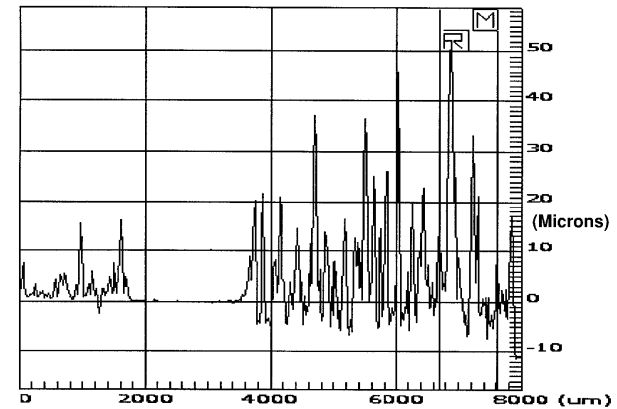


(He-450 pulses)

(N-400 pulses)

$R_a \sim 1-3 \mu\text{m}$
 $P-V \sim 10-30 \mu\text{m}$

$R_a \sim 4-9 \mu\text{m}$
 $P-V \sim 20-35 \mu\text{m}$



(He-450 pulses)

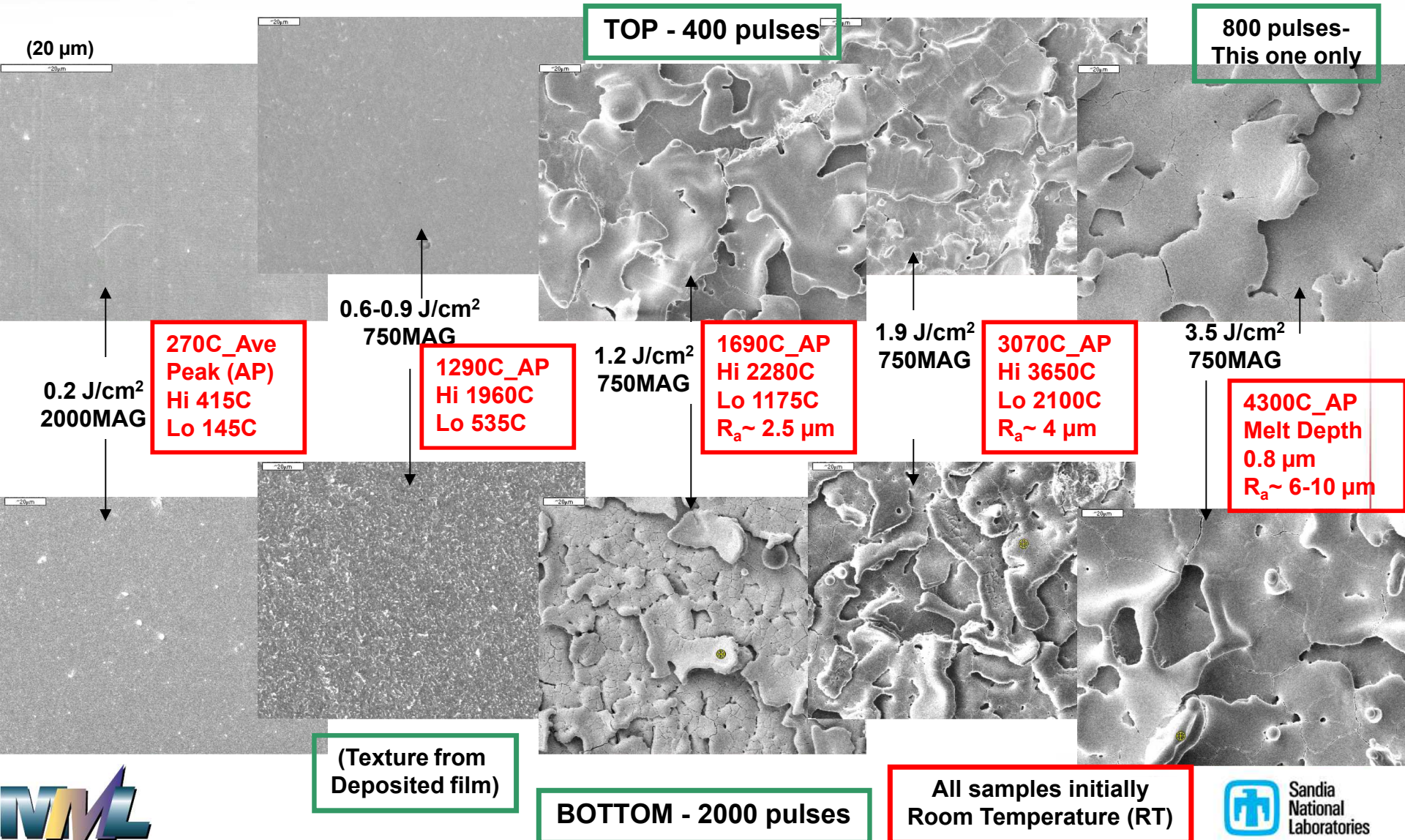
(N-600 pulses)

$R_a \sim 1-3 \mu\text{m}$
 $P-V \sim 10-30 \mu\text{m}$

$R_a \sim 7-10 \mu\text{m}$
 $P-V \sim 50-70 \mu\text{m}$

- Roughening of PM Tungsten: 450 pulses He @ 1.3 J/cm² roughens more than 200 pulses N @ 4 J/cm² (Melt)
- N-beam roughening catches up and passes at 400 pulses

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



Other materials roughen less. SEMs: M182 oriented grain W (top), W-TiC-A 2.5 cm-wide sample. Hypothesis: burying grains (M-182) and strengthening grain boundaries (W-TiC-A) helps restrict roughening.

1 - 1.5 J/cm²
R_a 0.35 to ~ 1 μm

From J. Linke, FZ Julich

Little or no mass loss to 2400 pulses

400pulses

1200 pulses

2000 pulses

800 pulses

1600 pulses

2400 pulses

50μm Electron Image 1

400pulses

800 pulses

1200 pulses

From H. Kurishita (Tohoku U.)

This roughening 10-20X LESS
than with PM Tungsten

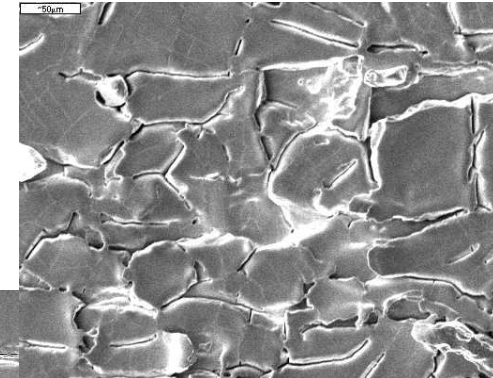
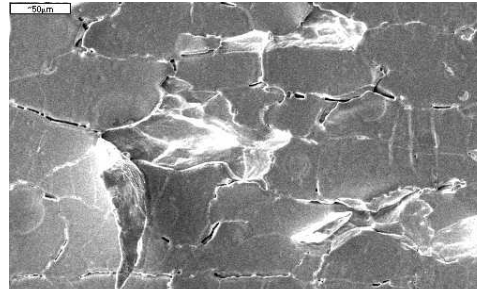
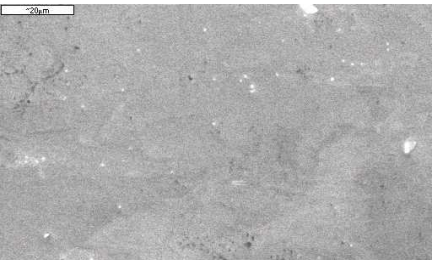
~ 1.4 J/cm²

All images -
1,000X MAG

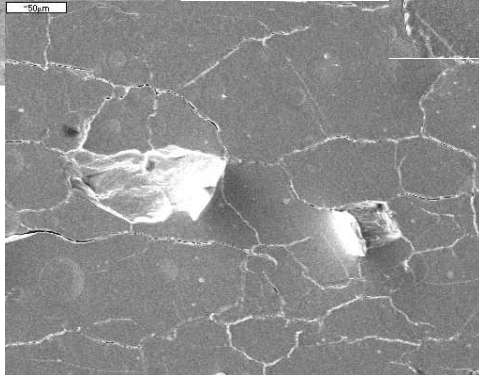
Right Two ~ 1.7 J/cm²
Ra ~ 0.28 μm

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

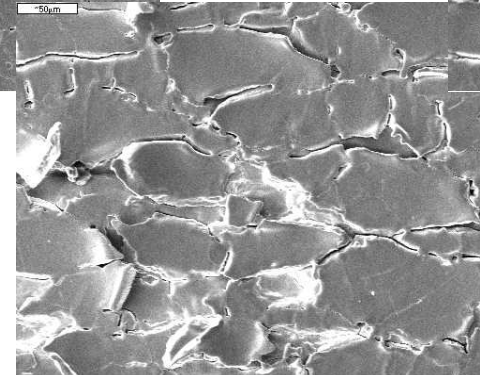
No Treat



1,000X MAG



~ 0.7
J/cm²



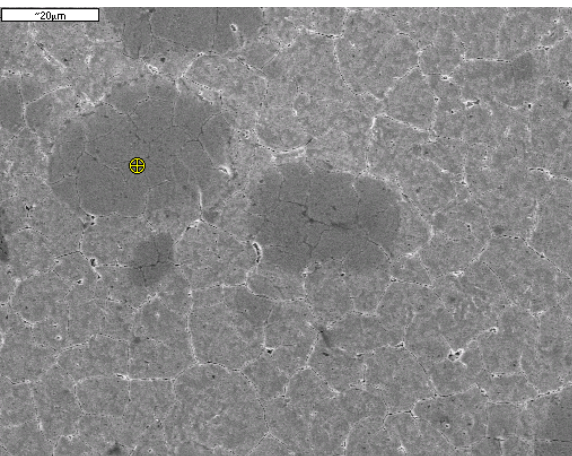
About 1.3
J/cm²

All treated images 300X MAG.

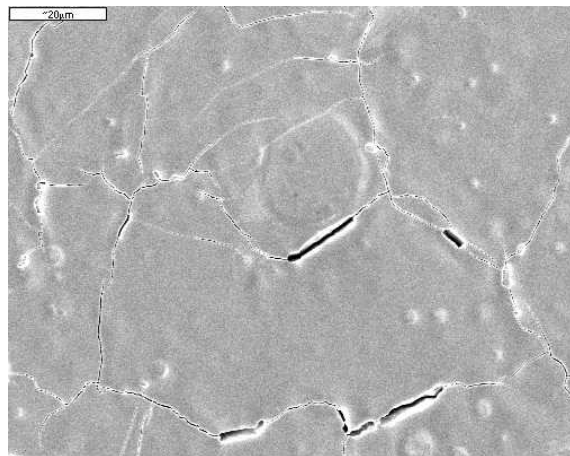
R_a : reaches 4 - 4.5 μm at 1.3 J/cm² (same as PM Tungsten)
Only apparent AFTER 400 pulses (these images)

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

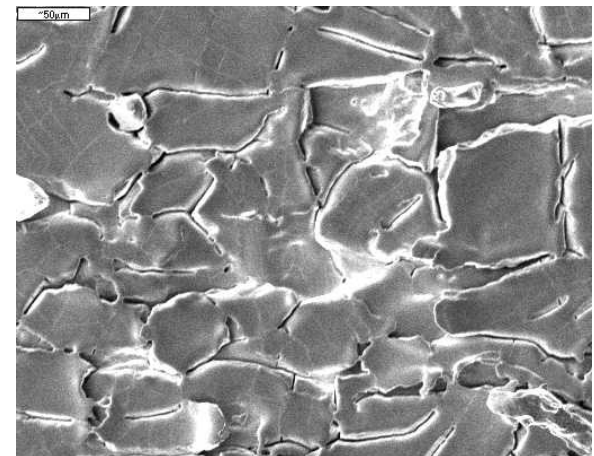
1,000X MAG



1,000X MAG



300X MAG



W-0.5%TiC 1.5 J/cm².
Ra = 0.04 μm

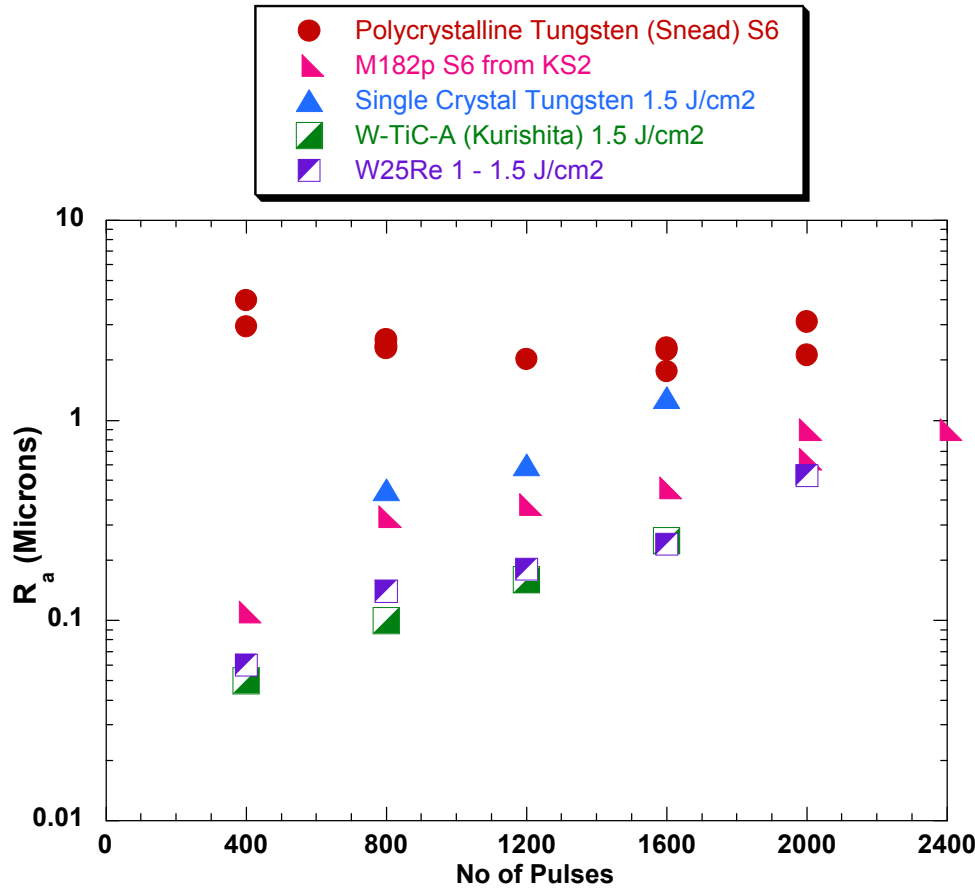
M182Perp ~ 1.25 - 1.5 J/cm²
Ra ~ 0.15 μm

M182Parallel ~ 1.3 J/cm²
Ra ~ 4.5 μm

Two on right are SAME material

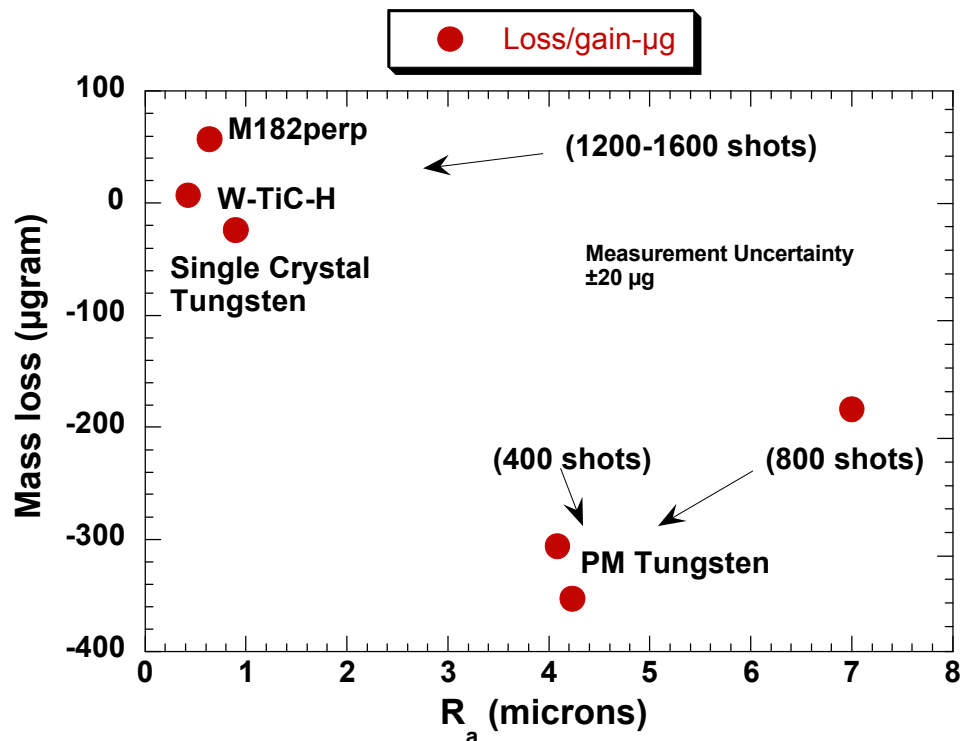
RHEPP-1 nitrogen exposure

Even though PM tungsten roughens most, other W materials do not show saturation w/pulses. Expect that eventually they will catch up to PM tungsten.



- Red Dots - Powder Met tungsten, about 1.2 J/cm²/pulse. Data saturates above 400 pulses
- Pink - M182p to 2400 pulses. Reaches 1 μ Ra.
- Single-Crystal W, W-TiC-A, and W25Re exposed at higher fluence

Weight measurements pre- and post-exposure support connection between Roughening and Mass Loss

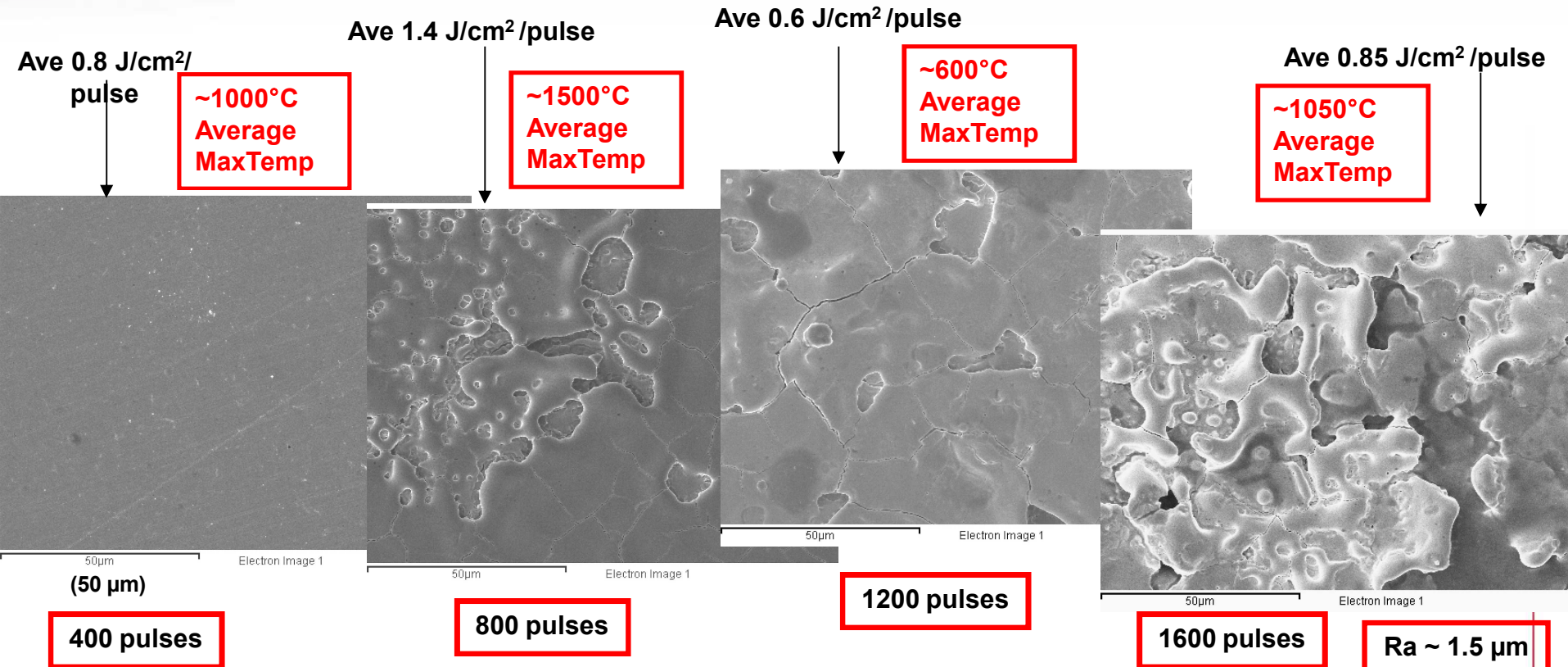


- Samples of each listed material exposed for multiple 400-shot series, and weighed pre-and post-shot
- Exposure level/pulse: 1.2 - 1.7 J/cm²
Measurement Uncertainty $< \pm 20 \mu g$
- Two samples of polycrystalline (PM) Tungsten lost ~350 µg in 400 pulses, with $R_a \sim 4 \mu m$; another 400 pulses produced even more roughening, -184 µm more mass loss
- M182Perp, W-TiC-H, and Single Crystal Tungsten remained $< 1 \mu m$ R_a , and suffered little mass loss.

Reference: flat polycrystalline tungsten exposed to 1600 total He pulses

All samples initially Room Temperature (RT). Fluence varies each 400 shots

All images 1000X magnification



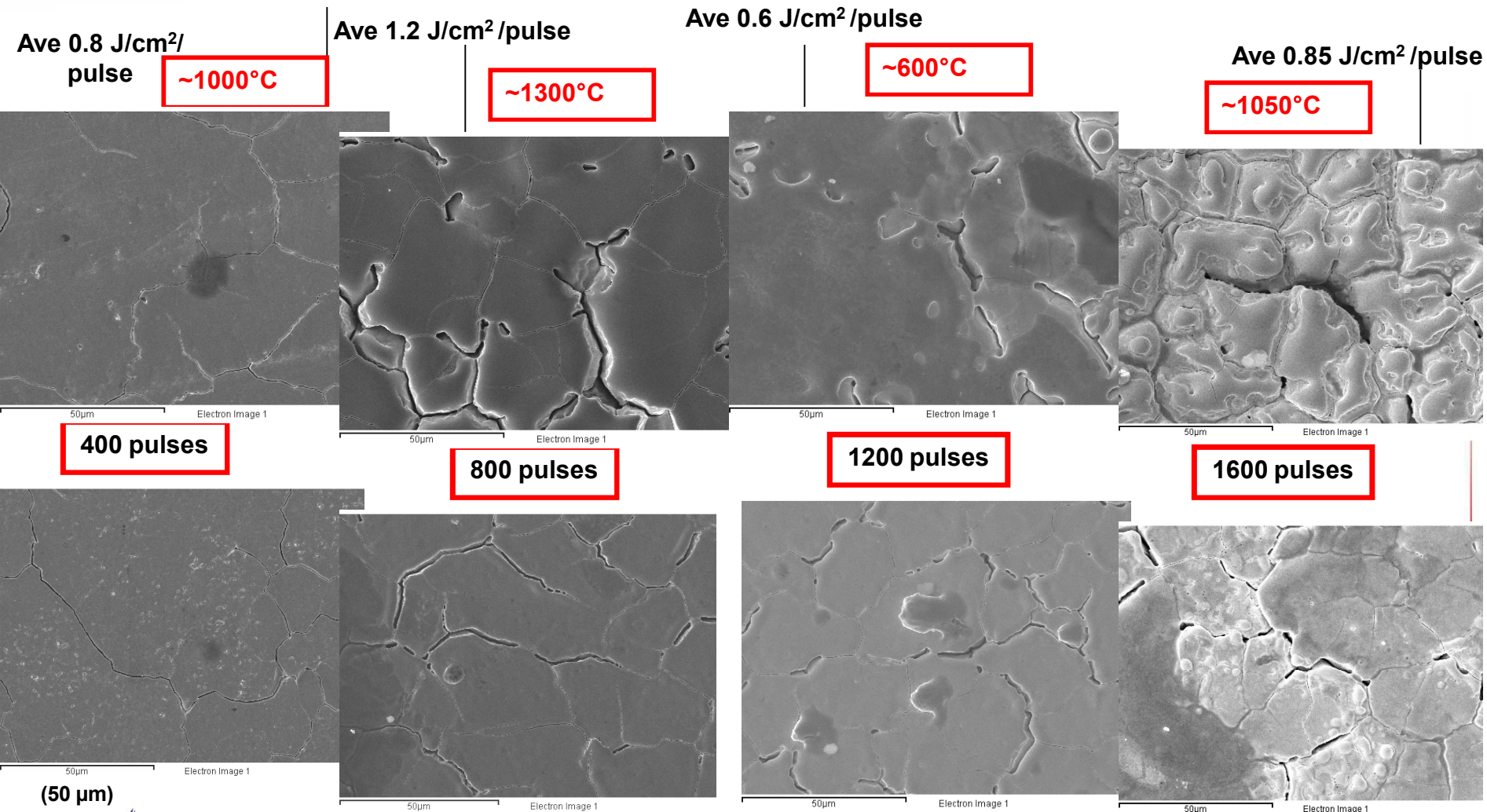
- Average estimated maximum surface temperature < 1500°C
- No effect 1st 400 pulses: below roughening threshold
- Using \sqrt{t} scaling: 0.8 J/cm² equivalent of 0.4 MJ/m². Consistent with QSPA plasma exposure of tungsten PFCs (ref: A. Zhitukhin et al, JNM 363-365 (2007) 301-307).
- Final 400 pulses @ 0.85 J/cm²: probable cumulative mass loss. Total He implantation well below 1e18/cm² reported as pore threshold.

(Est) total He implantation
~ 1.8e16 /cm²

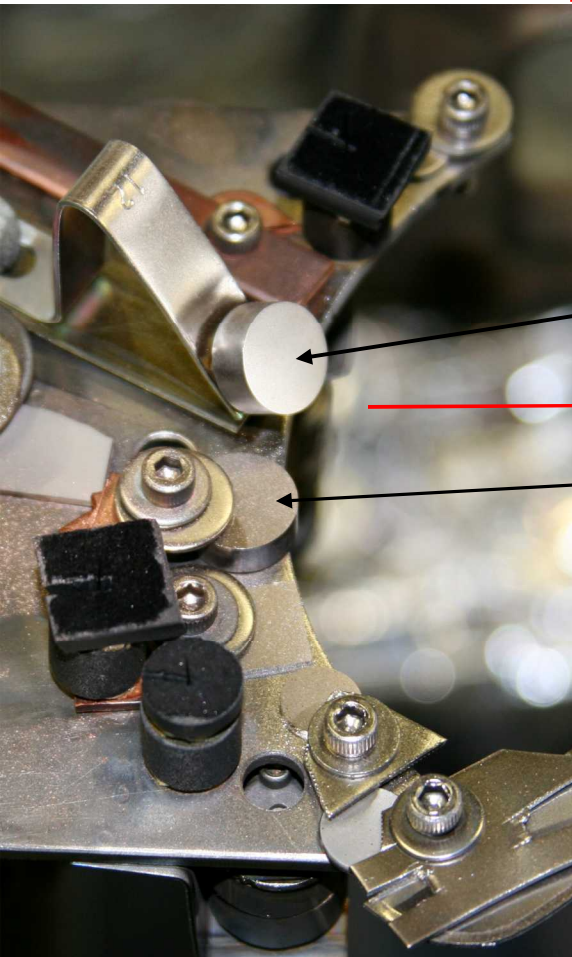
M184(P) oriented grain tungsten to 1600 pulses. Pore formation, bubbles

All samples initially Room Temperature (RT)

All images 1000X magnification

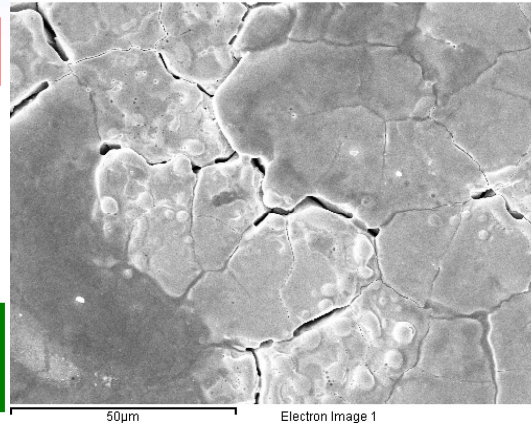


Bottom row is TILTED. Reduces roughening over flat sample



$R_a \sim 0.26 \mu\text{m}$

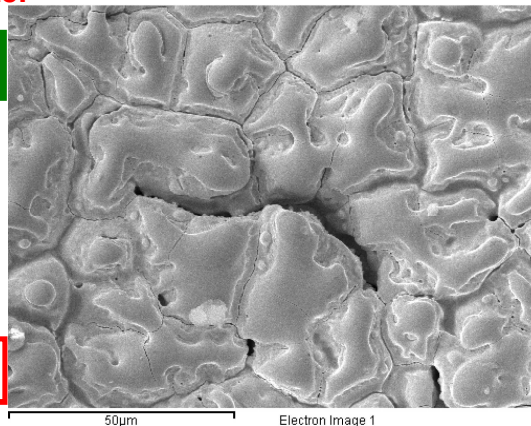
M184 55°
'Tilt'



M184 Tilt – 1600 He pulses

M184

$R_a \sim 1.5 \mu\text{m}$



Flat M184 – 1600 He pulses

- Tungsten M184 oriented grains parallel to energy delivery (ITER spec) exposed to 1600 He pulses, flat and 55° tilted to incoming ions.
- Tilted sample shows much less roughening
- Shows that heart conduction, not direct energy deposition, is cause of morphology change

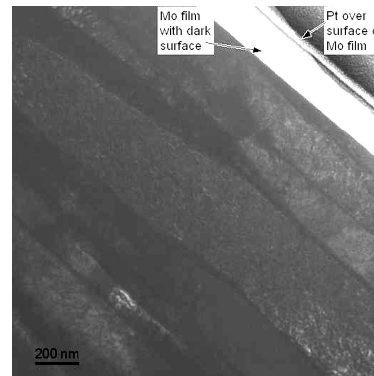
3-D tilted geometry: 'Needles' survive multi-shot RHEPP exposures well. No pores/bubbles seen in near-surface region after 1600 pulses.

Incoming Beam

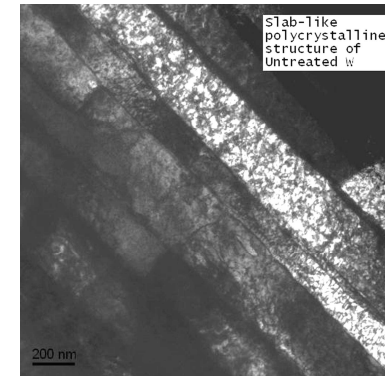
Tested W needle dimensions:
Tip Length 3000 μm
Radius 125 μm
Rtip 0.25 μm
Shaft 1.5 cm X 250 μm diam
Mounted for exposure (below)



- Single needles and arrays exposed 1600 He pulses.
- (below) TEM of W needle w/ 200 nm Mo coating, compared to virgin uncoated (right). Both cuts made 2 mm from tip. Exposed μ -structure identical to virgin (no voids, bubbles), Mo coating intact.

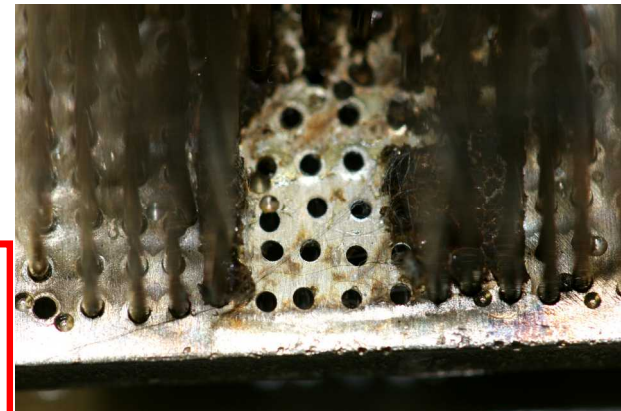


Mo coated - 1600 pulses



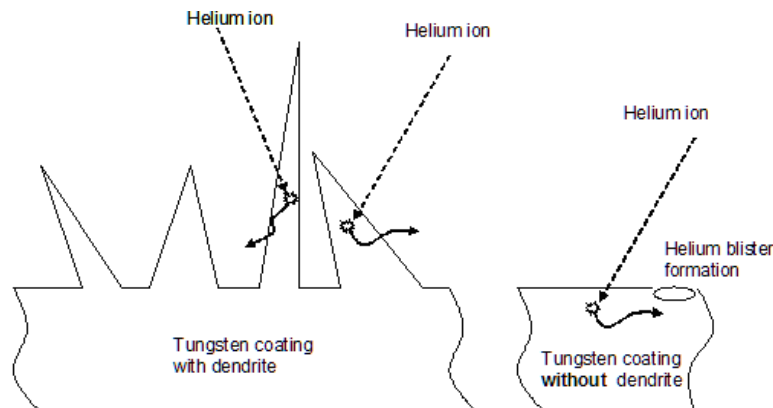
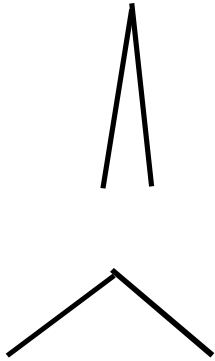
W needle uncoated virgin

RIGHT: needle 'forest' after 400 He pulses:
Al edge heavily melted, in needle 'forest',
no Al substrate damage (needles are
removed for photo)



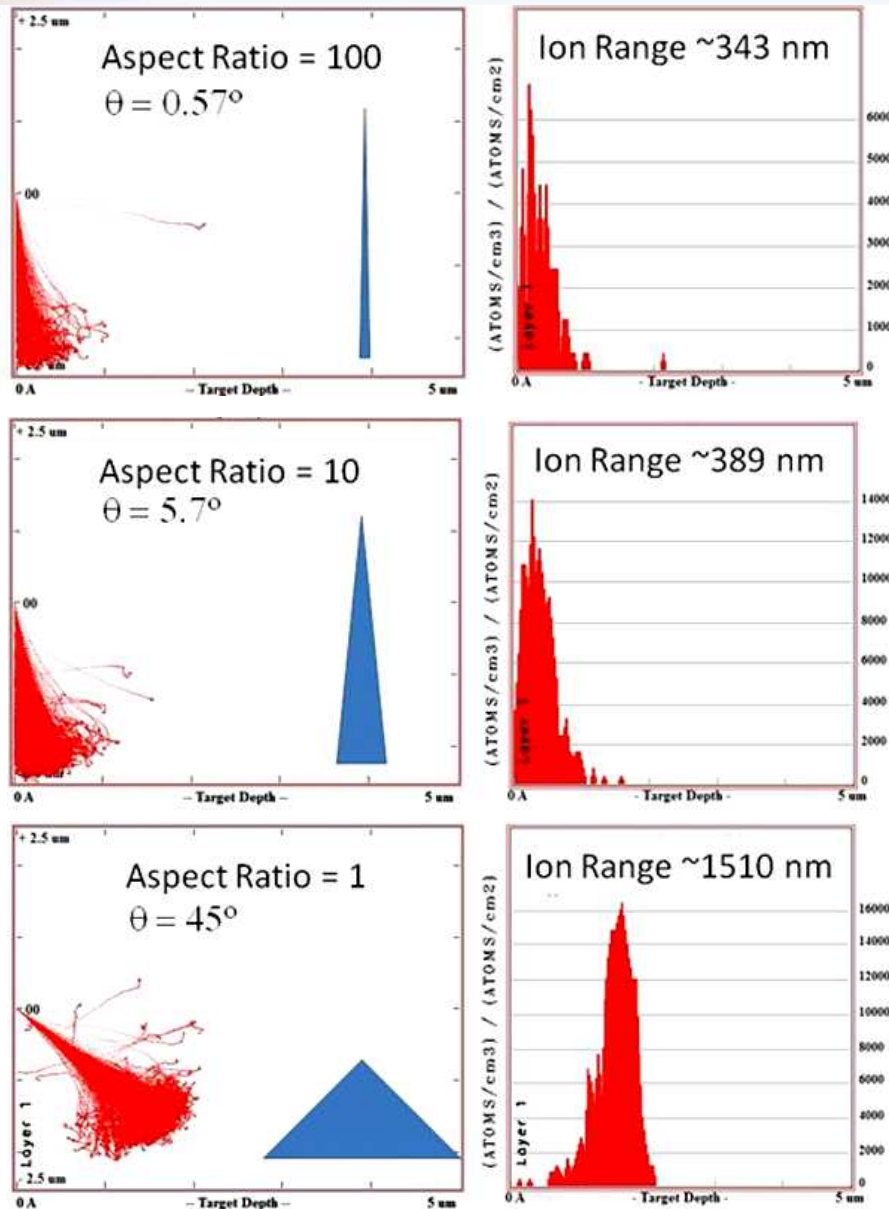
Design Issues for 3-D First-Wall Materials

- Overall feature size, aspect ratio, tip geometry (sharpness, etc), assembly into groups. 'Tips' should erode at same rate as flat geometry. Behind tip, erosion rate \sim total cross-sectional feature area/ total wall surface. Single-element materials vs complex structures (nano-composites, coatings)
- Tall aspect ratio: much reduced fluence on sides, shallow He penetration should allow for easier escape (below). Disadvantage: many structures needed to cover surface, tip erosion increases (heat 'meets in middle'), too tall may affect (cryogenic) pellet temperature. For both cases, material removed from sides is likely to re-plate on same or other tips.
- Short aspect ratio: less fluence reduction on sides, deeper He penetration. Advantage: fewer structures needed.
- Considerable evidence for enhanced durability of metallic structures as feature size decreases $< \sim 50 \mu\text{m}$. Question: can such features survive (considerable) thermomechanical shock?
- Reference: S. Sharafat *et al*, J. Nucl. Mat. **347** (2005) 217-243



Helium penetration varies with aspect ratio

Surface
 θ
Ion

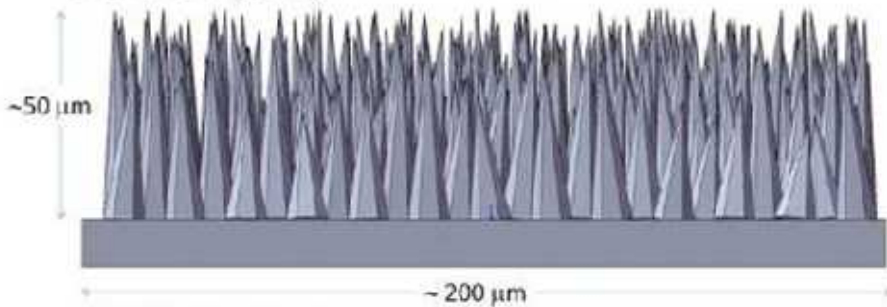


- Helium penetration increases in depth greatly for < 10 Aspect Ratio.
- Reference: S. Sharafat, UCLA-Mech. & Aerospace Engr. Dept., private communication, April 2010.

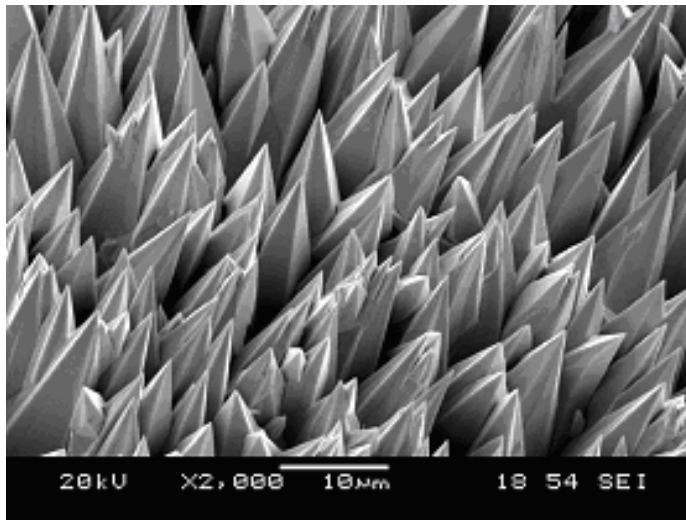
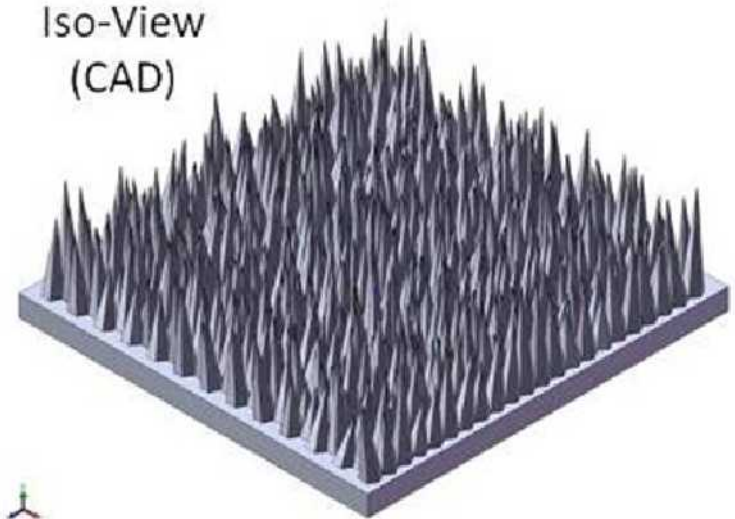
Ultramet dendritic materials are made by chemical vapor deposition using tungsten, molybdenum, and/or rhenium raw materials on similar-material substrates

Conceptual drawings

Side View (CAD)



Iso-View
(CAD)



SEM, example, as-manufactured

10 μm

A first experiment: pure Re dendrites on Re substrate

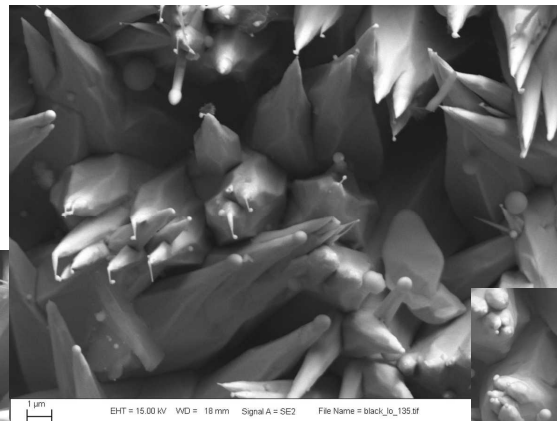
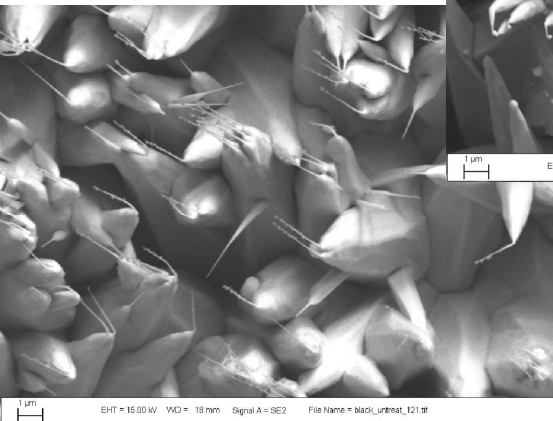
Slight melting at lowest fluence, failure at $0.55 \text{ J/cm}^2/\text{pulse}$

400 helium shots
All fluences are $\pm 25\%$

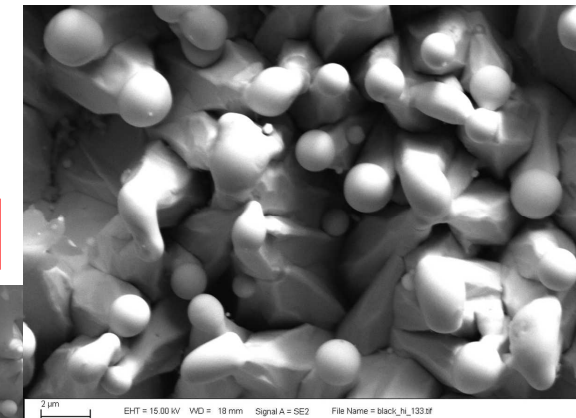
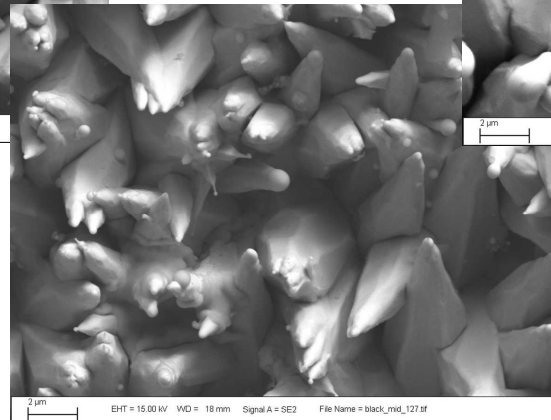
$0.25 \text{ J/cm}^2/\text{pulse}$

$\sim 0.55 \text{ J/cm}^2/\text{pulse}$

Untreated



$\sim 0.35 \text{ J/cm}^2/\text{pulse}$

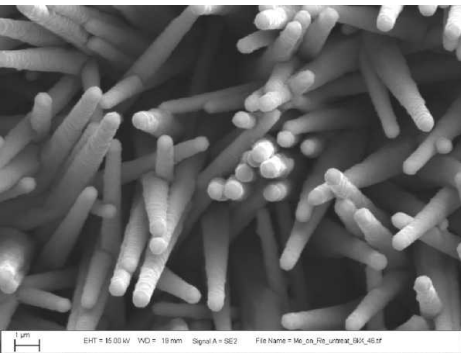


Suspect a) too fine a structure, and b) low Re thermal conductivity 48 W/m-K (W is 170)

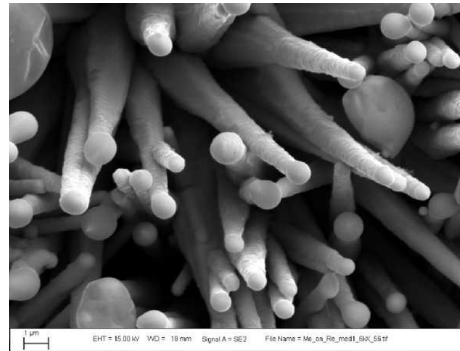
400 He shots

Case 2: Mo coating on Re dendrites. Too fine a structure

Untreated

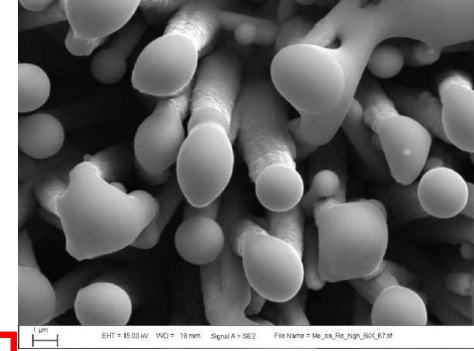


0.45 J/cm²

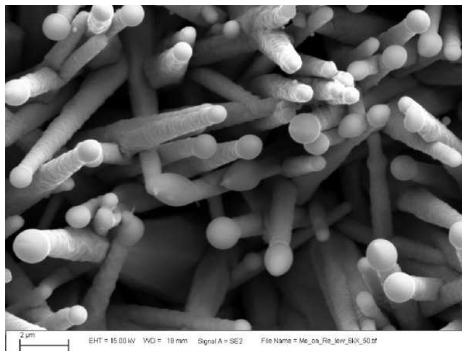


6,000x

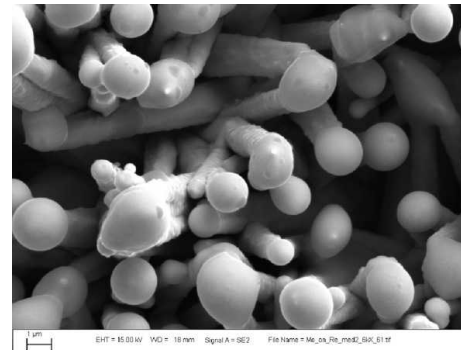
1.7 J/cm²



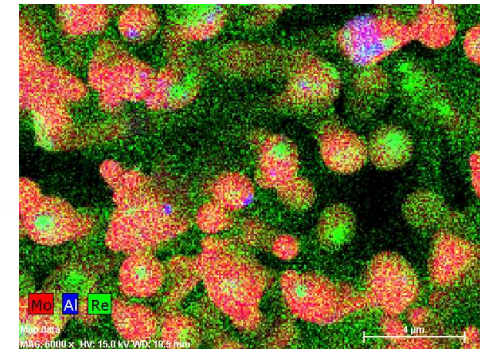
0.2+ J/cm²



1 J/cm²



Note: fine structure still present on side walls below melted tip



- Untreated rods ~0.7 μm wide at tip. Tip melting evident even at 0.2 J/cm²
- Mo melts back, revealing Re substrate (green tiny tips at right).
- Mo Thickness important to heat flow. 1-D Heat Flow for 0.5 μm Mo on Re drops melting fluence from 2.8 to 1.8 J/cm². 2 μm thickness gets it back to 2.5 J/cm².

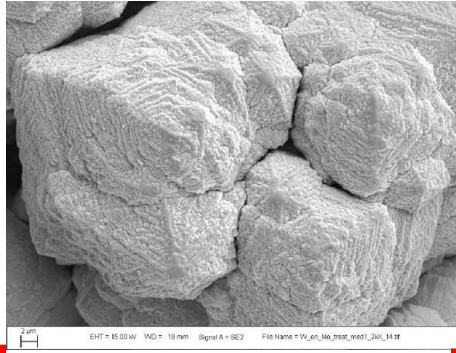
Case 3: larger scale pure W on Mo substrate - more robust

400 He shots

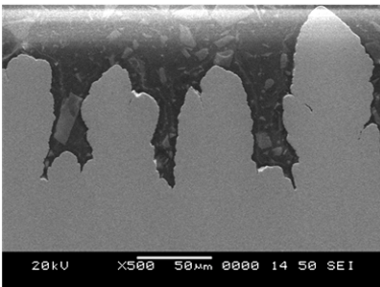
2,000x

0.9 J/cm²

2 J/cm²



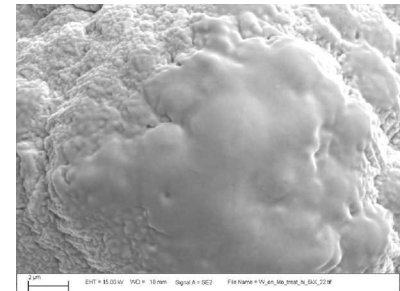
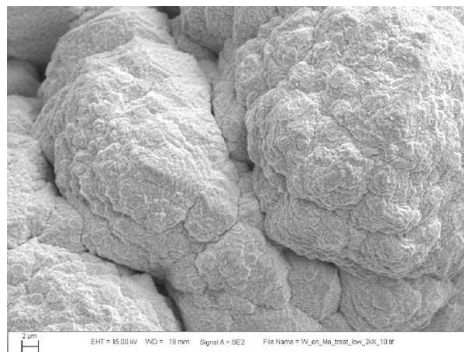
Untreated. (below) x-cut, 50 µm scale



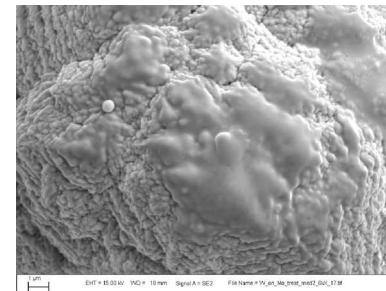
0.55 J/cm²

2 µm

1.45 J/cm²



6,000x



6,000x

- Untreated: larger-scale structures with much fine structure
- Very slight morphology change, with melting at ridge tops
- Downslope, fine structure even at 2 J/cm²

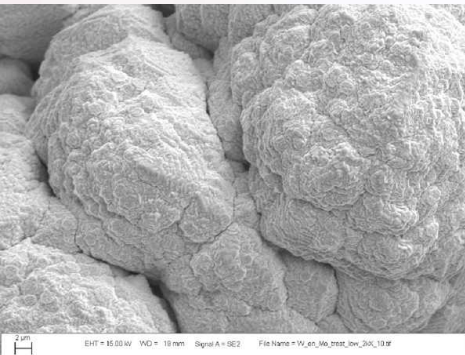
Different locations each fluence

Case 3: small change with 400 more pulses, stress cracks start

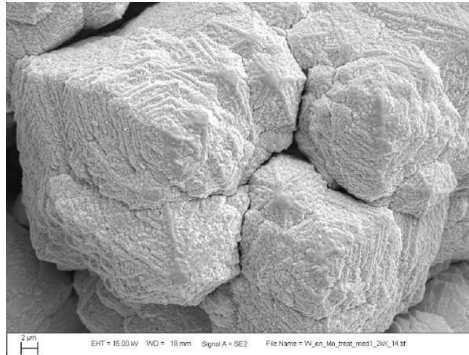
— 2 μm

2,000x

TOP - 400 He pulses



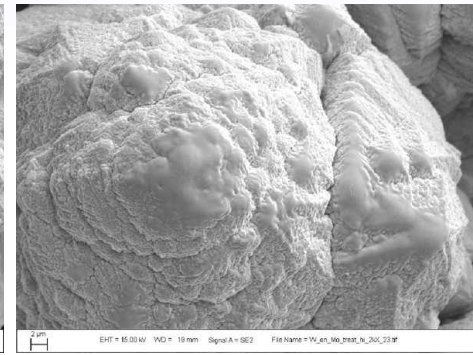
0.55 J/cm²



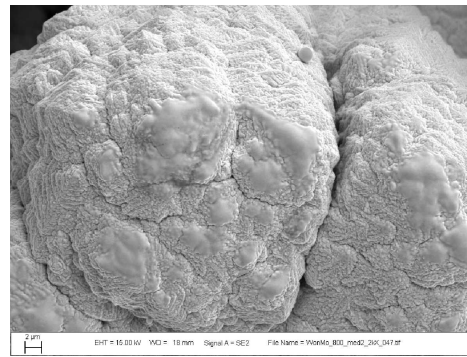
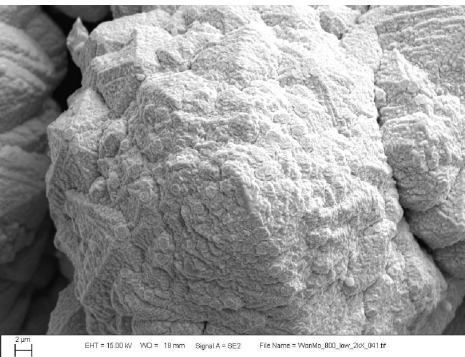
0.9 J/cm²



1.45 J/cm²

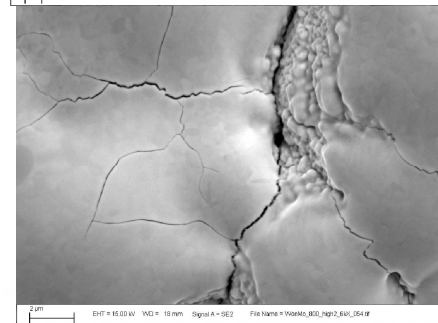


2 J/cm²



BOTTOM - 800 He pulses

- Only very tops of ridges show melt at 0.55 J/cm²
- Small melt changes 400-800 pulses, even at 2 J/cm²
- Observable melt/reflow in places, with beginning of crack networks, at 2 J/cm²

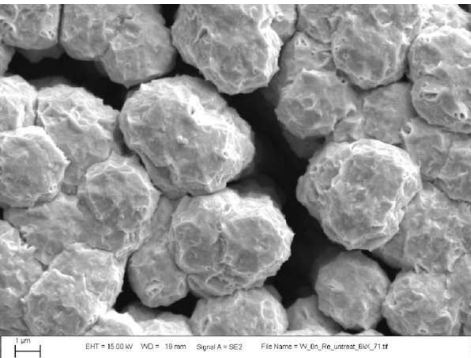


6,000x

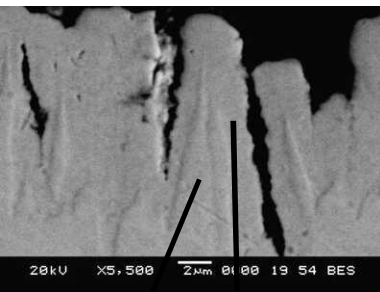
Case 4: fine-scale W on Re dendrites performs best

400 He shots

6,000x



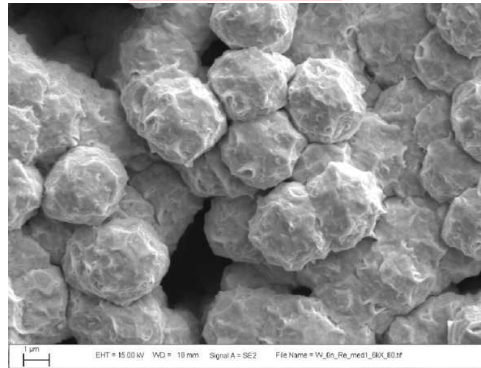
Untreated. (below) x-cut, 2 μm scale



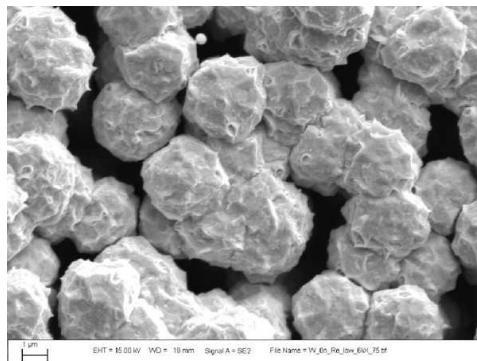
Re core

1-2 μm W

0.55 J/cm²

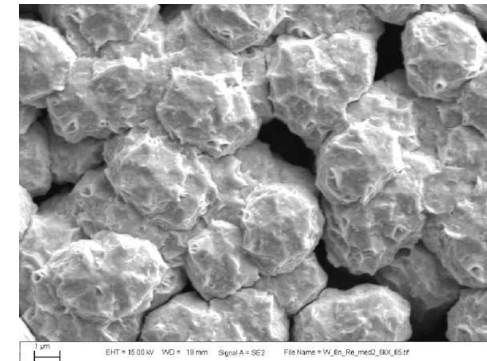


0.35 J/cm²

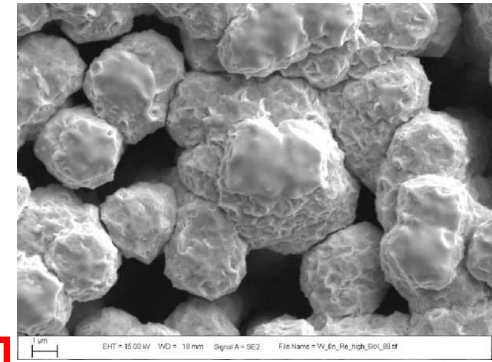


— 1 μm

0.9 J/cm²



1.55 J/cm²

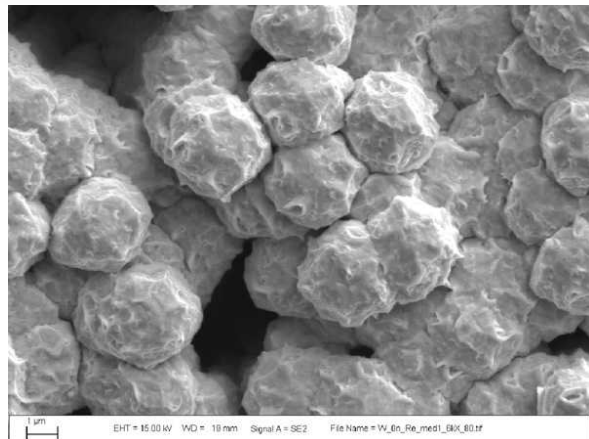


- ~ 5 μm scale composite W/Re shows no effect at 0.9 J/cm², with melting at ridge tops at 2 J/cm².
- EDS indicates no Re signal - W coating is intact.
- 1-D SIM of a) pure W - 3.2 J/cm² melt threshold; b) 0.5 μm W on Re - drops to 2.5 J/cm²; c) 2 μm W on Re - back to 3.1 J/cm². Suggests thicker W coating may perform better.

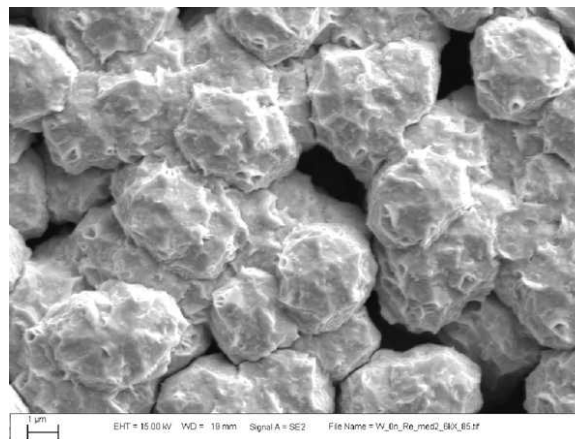
Case 4: little change/no cracks with 400 more pulses

6,000x

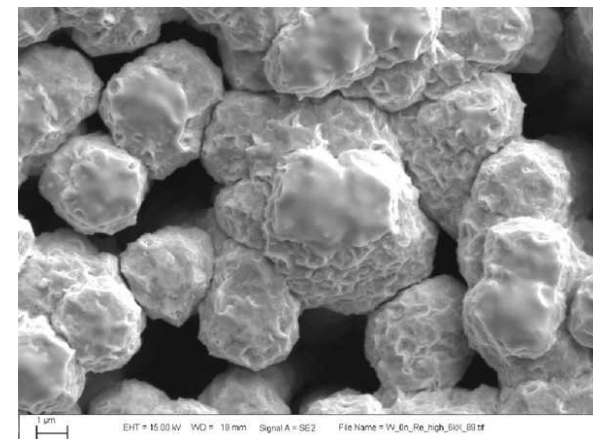
TOP - 400 He shots



0.55 J/cm²

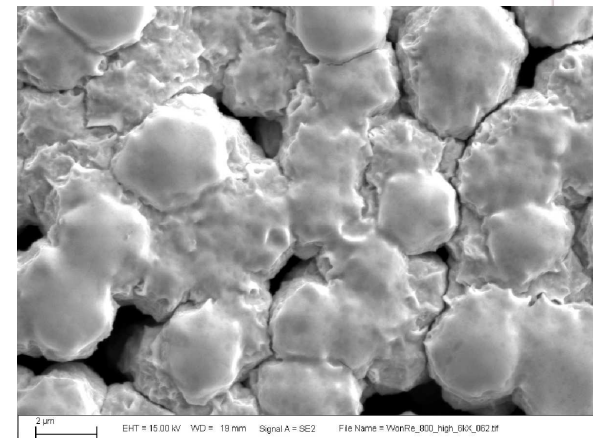
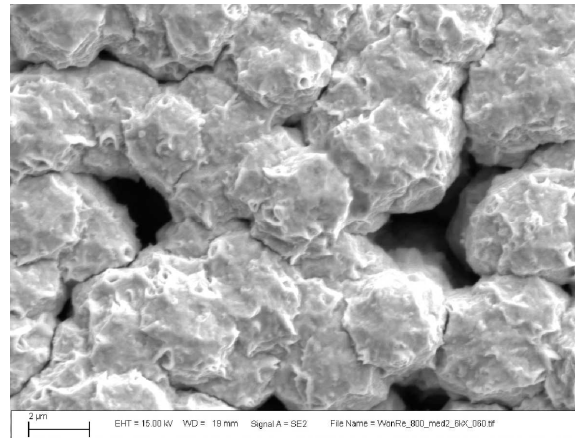
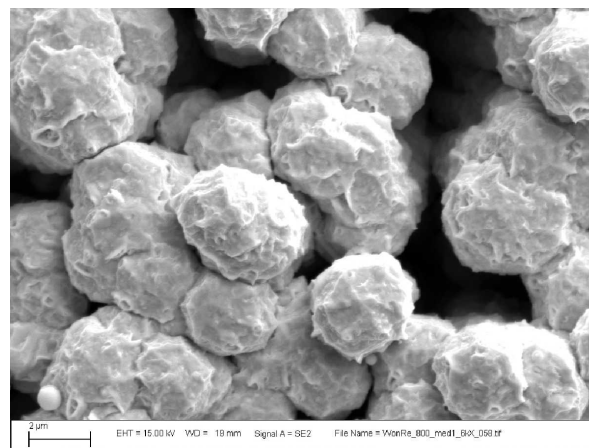


0.9 J/cm²



1.55 J/cm²

— 1 µm



BOTTOM - 800 He shots

2 µm

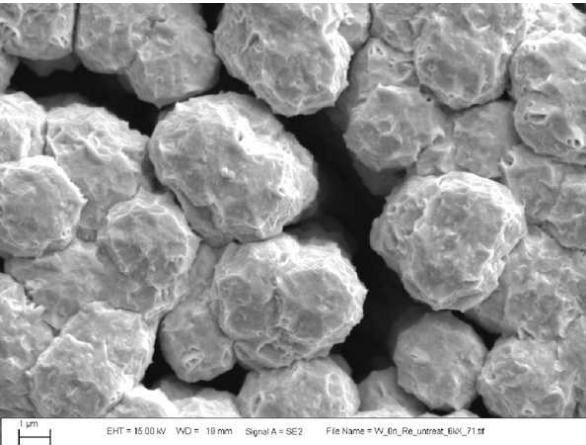
- More extensive ridgetop melt, but no cracks yet, at 2 J/cm².
- No effects evident through 800 pulses at 0.9 J/cm² and below.

Contrast: fine-scale pure W - more melting

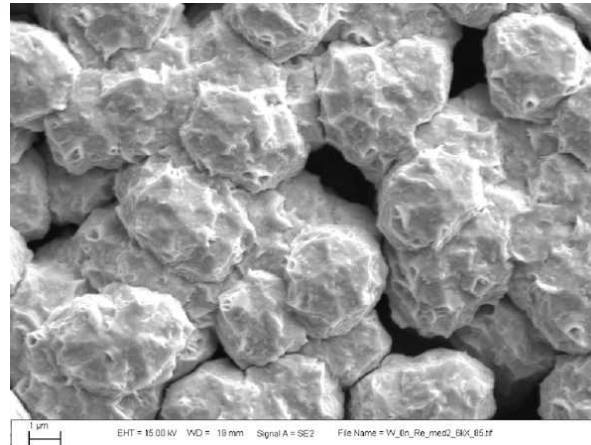
400 He shots

6,000x

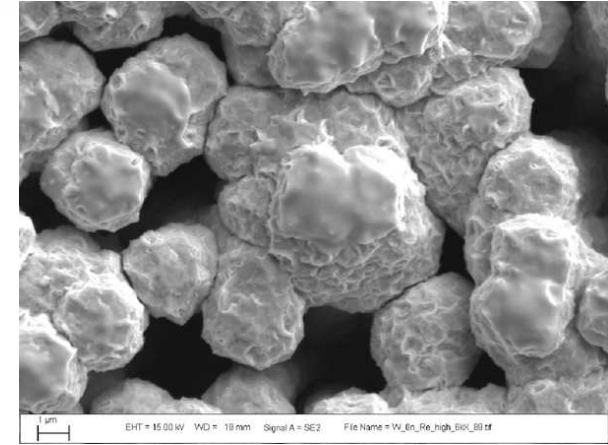
— 1 μm



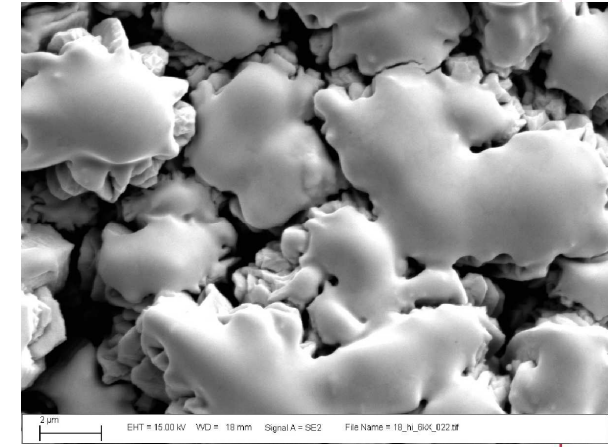
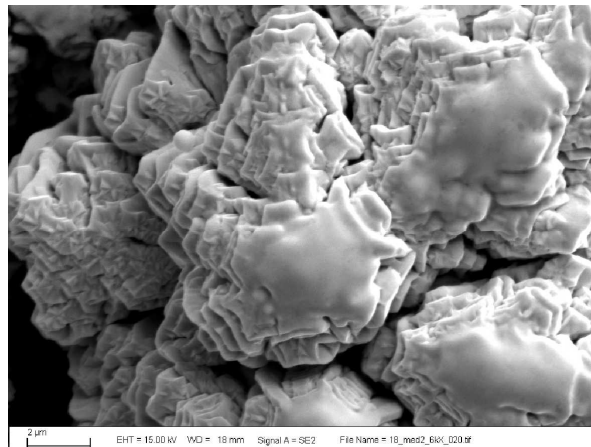
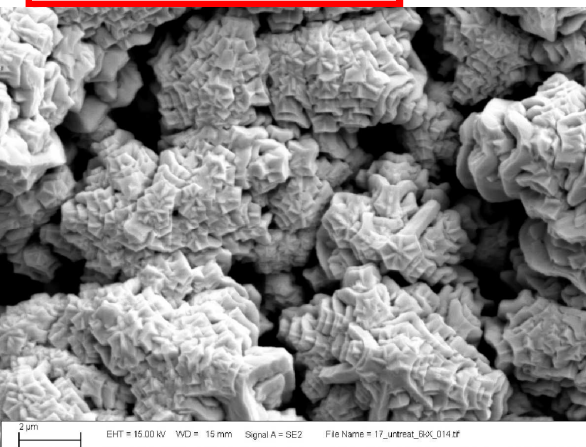
Untreated



0.9 J/cm²



1.55 J/cm²

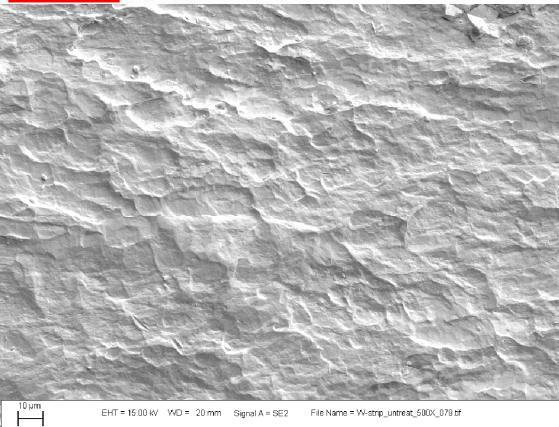


- TOP: W on Re already shown. BOTTOM: fine-scale pure W
- Appears that delicate structure W (bottom) not as robust as W/Re.

Contrast 2: polycrystalline flat tungsten (Schwartzkopf, unpolished) becomes unzipped at 1.4 J/cm^2

400 He shots

500x



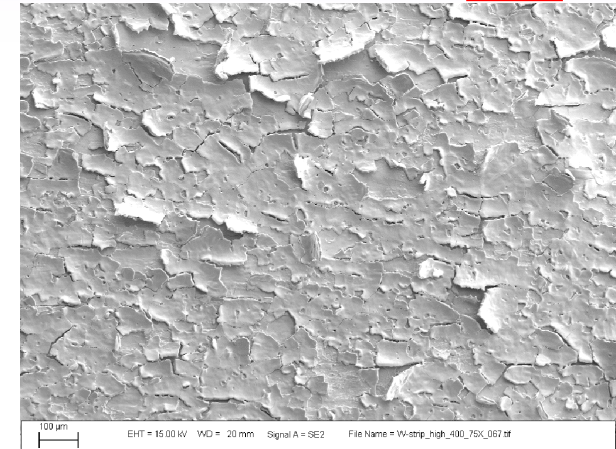
Untreated

100x



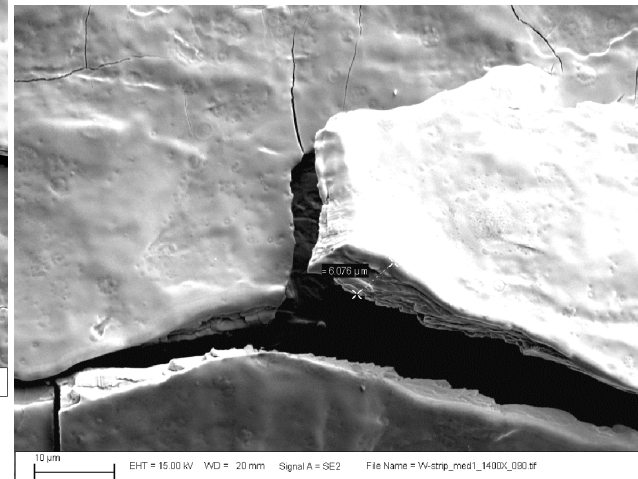
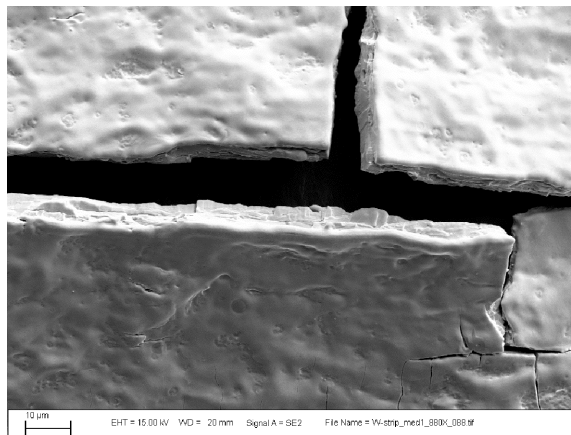
0.5 J/cm²

75x



1.4 J/cm² (also below)

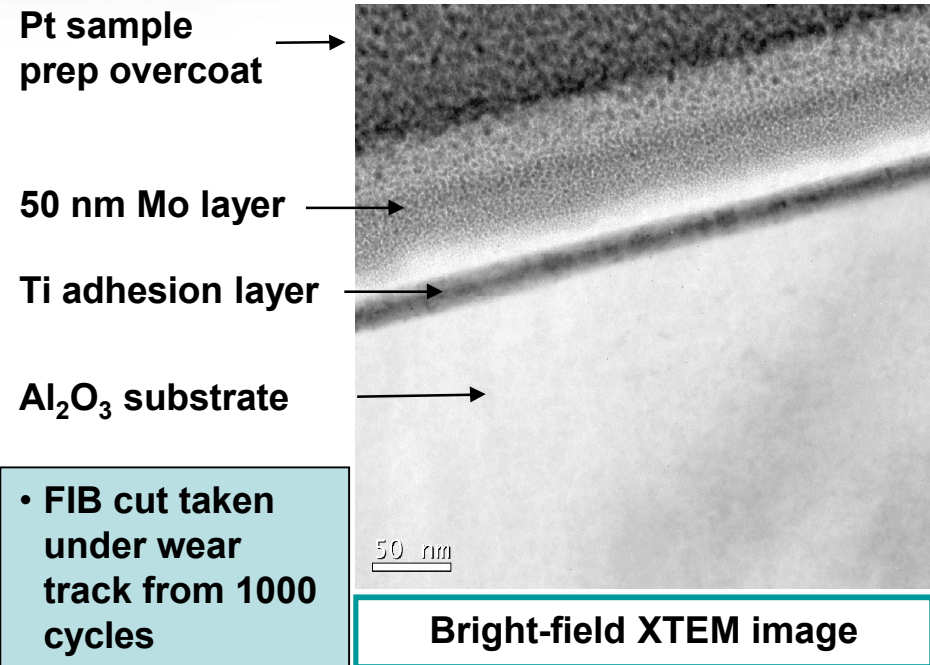
880x



1400x

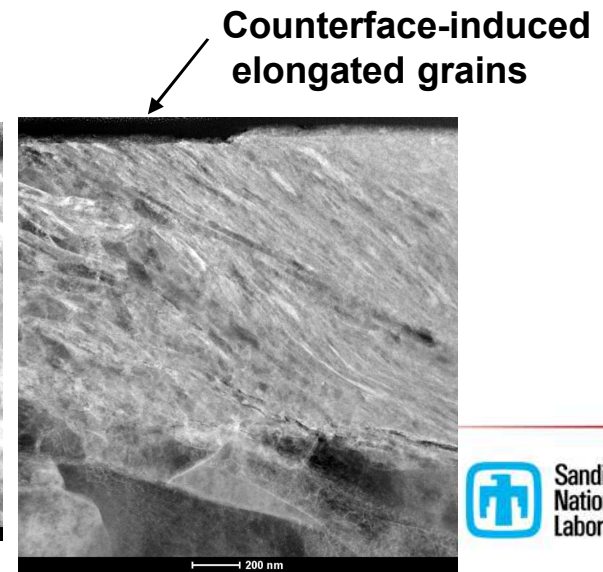
- Melt/reflow evident $> 0.5 \text{ J/cm}^2$
- 5 µm-thick surface slabs detach from substrate.

Motivation for coating study: Microstructure of BCC nano-grain Mo drastically affects linear tribology performance

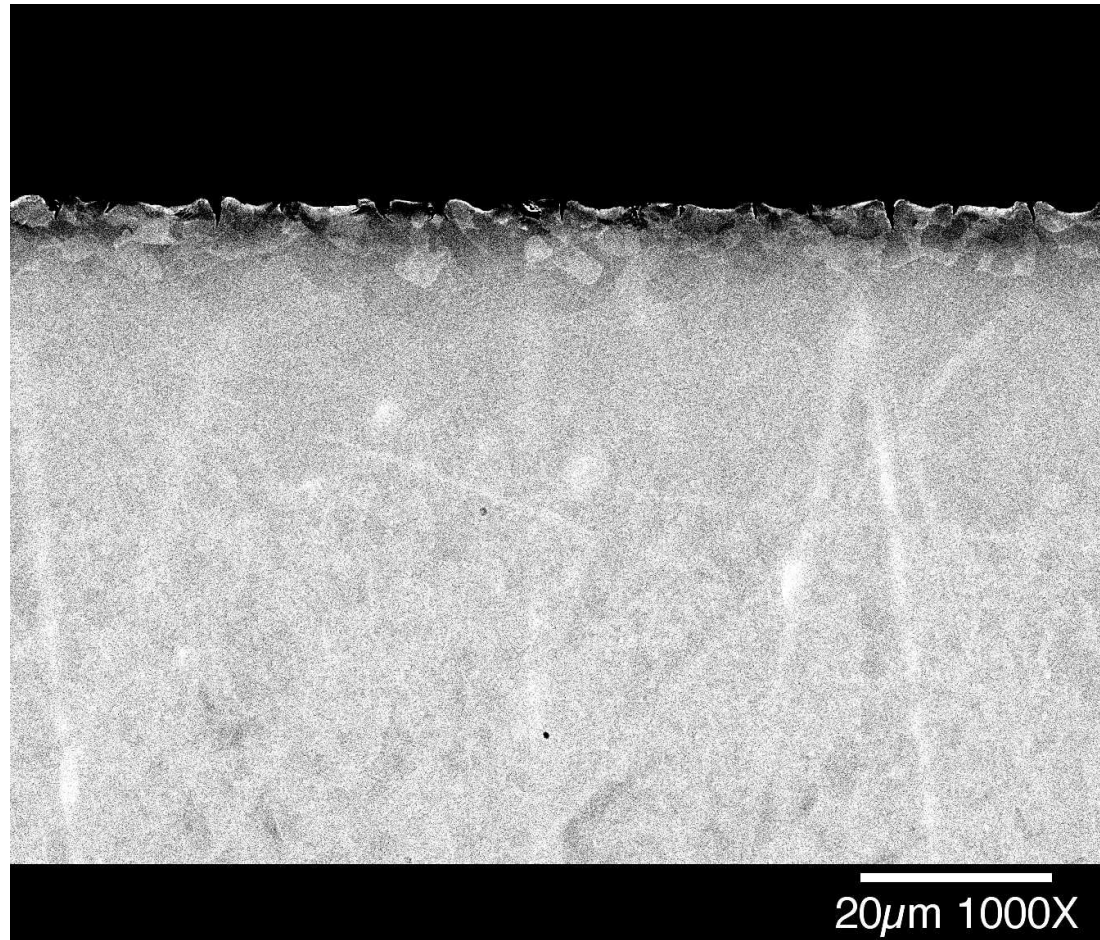


- 50 nm-thick ablated Mo layer w/ 5-10 nm grain size (left) subjected to 1000 linear wear cycles w/ Si₃N₄ counterface. FIB cut taken under sliding track shows no wear.
- (Below) same test done on bulk Mo ablation target shows wholesale mechanical failure with elongated grains in 1 μm-thick layer.

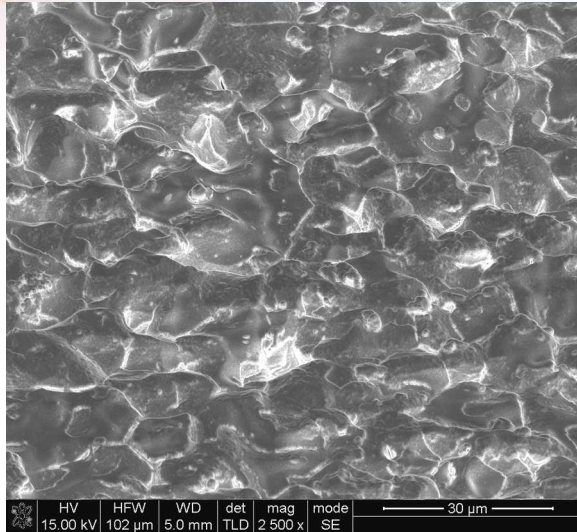
- Mechanical shear failure layer ~ 1 μm thick on bulk Mo after 1000 cycles



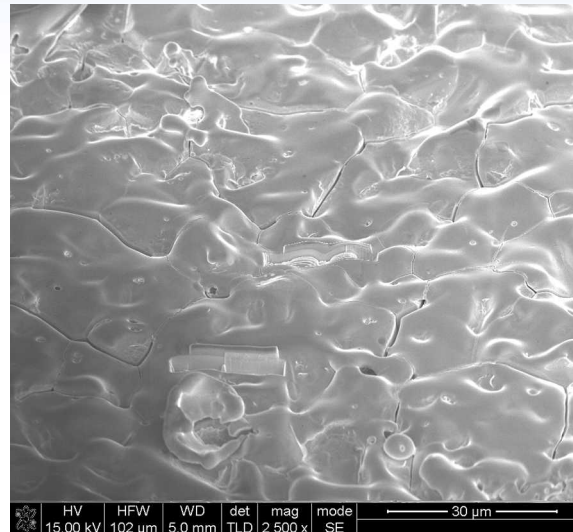
**Argument against fine-grain stability under thermomechanical loading: SEM, Single-Crystal Tungsten 2000 pulses at 520°C:
Clear evidence of recrystallization**



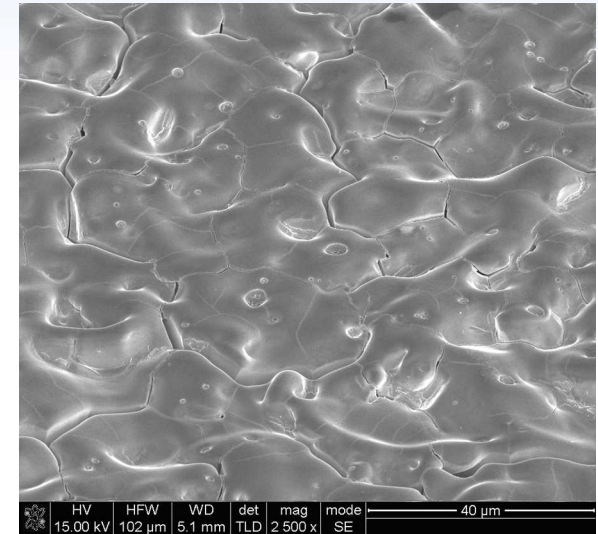
BCC coating 1: 1 micron sputtered tungsten on tungsten



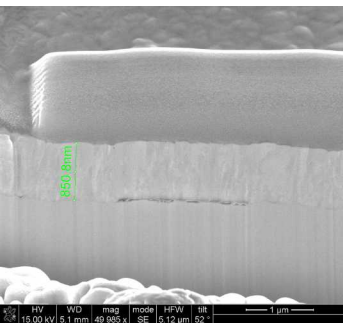
Surface as-deposited



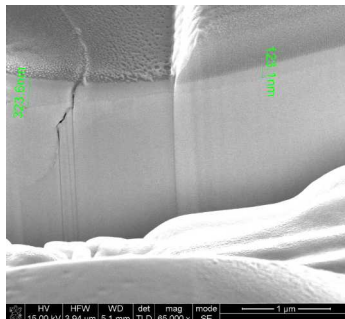
Surface after 1200 He pulses



Uncoated surface after 1200 He pulses



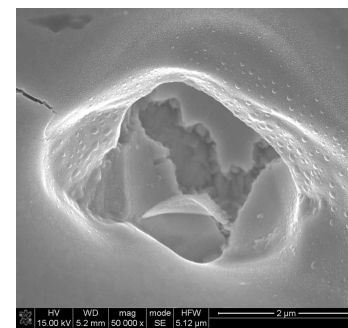
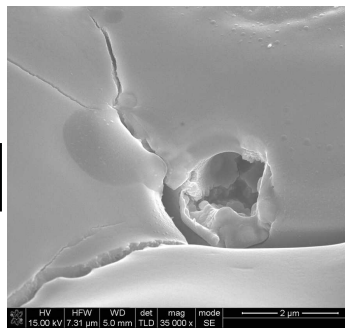
Before



After

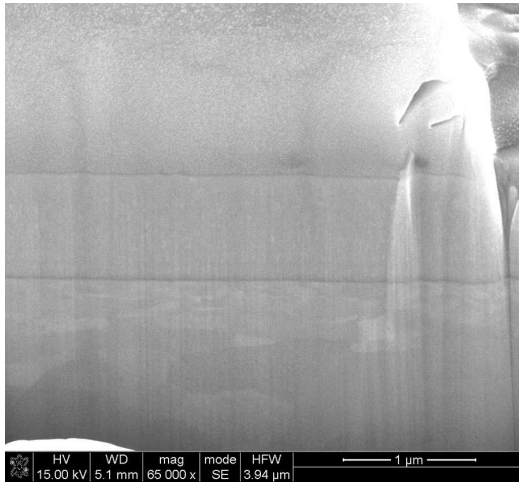
Coated

- Substrate is rough. Slight improvement in treated surface appearance with coating
- Original 850 nm thickness (left): columnar structure either modified by exposure, or eroded. ~ 200-300 nm left.
- Both surfaces show bubble formation

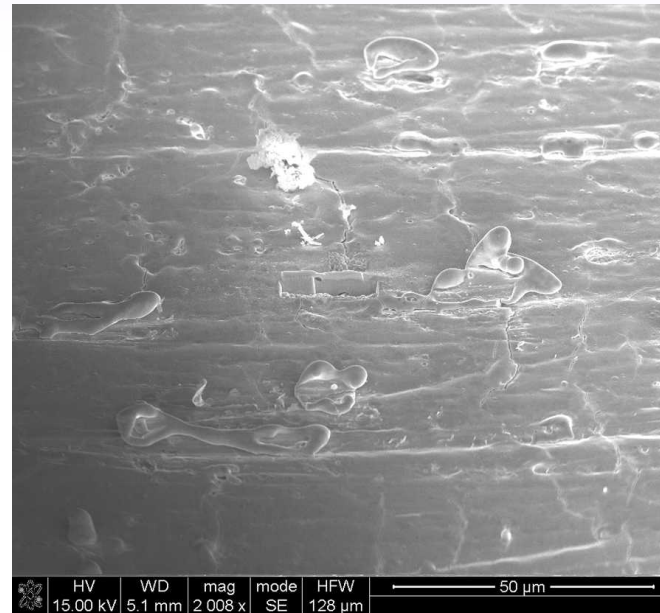


Uncoated

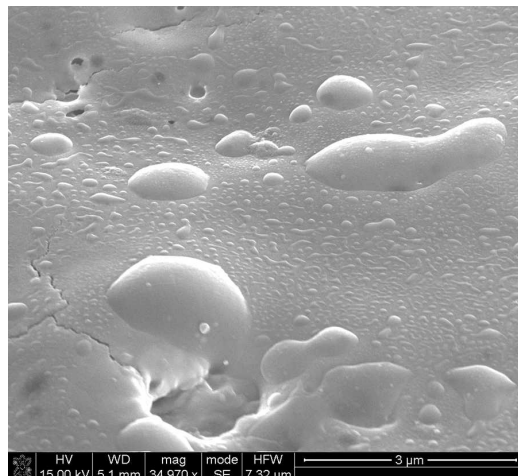
BCC coating 2: 1 micron ablated Mo layer (from RHEPP) - same process as coated the Mo needle



FIB cut: 900 nm fine-grain Mo



Surface after 1200 He pulses



Close-up shows extensive pore formation

- FIB shows fine-grain layer.
- (LEFT) surface after exposure shows balled-up Mo. Evidently Mo layer delaminated, balls form from surface tension
- (unprotected) surface shows extensive pore formation
- Possible that 3-D layer increases mechanical adhesion
- Mo on Re, Cu substrates not tested yet. Some coating appears to remain after 1200 He pulses

Summary

- RHEPP-1 helium pulses can replicate thermal wall loading from IFE reactor pulses (154 MJ yield shown), and deliver $\sim 1.25 \times 10^{13}$ He/cm² He implantation/pulse @ 1 J/cm².
- Extensive prior database: every flat dry wall material exposed appears to suffer unacceptable mass loss after 2400 pulses (8 reactor minutes @ 10 Hz).
- Tilting flat surface mitigates roughening - 3D wall surface takes advantage of this (needles, needle arrays, and dendritic structures from CVD).
- Ultramet-manufactured textured dendritic surfaces, composed of either pure W, Mo, Re on Mo substrates, or composite e.g. W on Re, exposed to 400 - 800 RHEPP-1 He pulses.
- ~ 5 μ m-scale composite structure (e.g. W on Re) shows little/no effect from 800 He pulses at 0.9 J/cm² and below. Pure W requires larger-scale to achieve similar performance.
- Flat W and Mo coatings - mixed results. W was not fine-grain. Layer is reduced in thickness either by erosion or by microstructure change. Mo appears to have delaminated, unlike needle coatings. Could be that 3-D shape (dendrites or needles) improves coating adhesion. No sign of pores on needles or dendrites.