

# **A Review of RHEPP Results in First-Wall Materials Treatment**

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# Executive Summary

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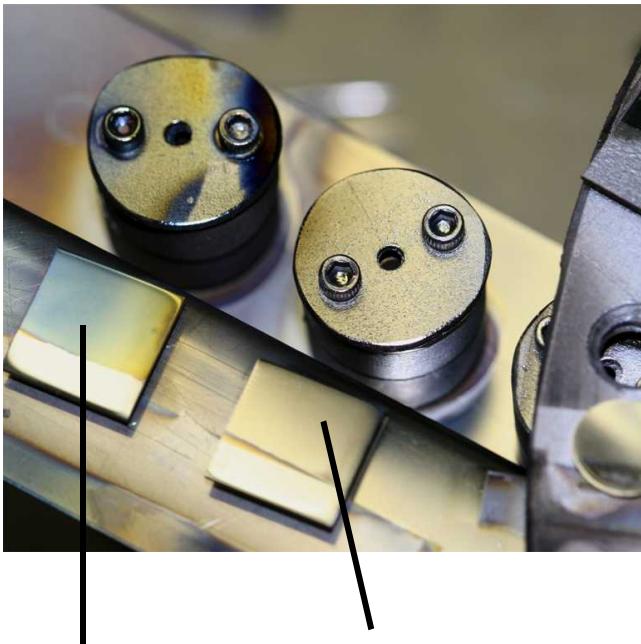
- **Tungsten Exposures - presently at KS2\_2400 shots and counting**
  - Polycrystalline Tungsten roughens severely, loses mass
  - M182perp Plansee Tungsten (commercially available): little or no mass loss to 2400 pulses, controlled but unsaturated roughness growth. Single-crystal W, W-TiC (Kurishita) also robust.
  - Vapor-sprayed W over Steel appears to lose mass steadily
  - Lack of roughness saturation requires other facility for ultimate validation. JULICH-II coming up with multi-shot 30 keV e-beam capability
- **Graphite exposures - C400, C2\_400**
  - ~ 1 J/cm<sup>2</sup> may be viable, large mass loss at higher fluences
  - Knowles CCW, CCW/W hold up well
  - CFCs should be oriented so that fibers go into material surface. PAN may be more attractive than PITCH due to mechanical robustness
- **‘Melt’ Campaign\_400: Melting does not look viable**
- **SiC: no successful material yet. Diamond-coated SiC evolves with shot number into graphite-rich surface, protection is lost.**



# Tungsten Materials

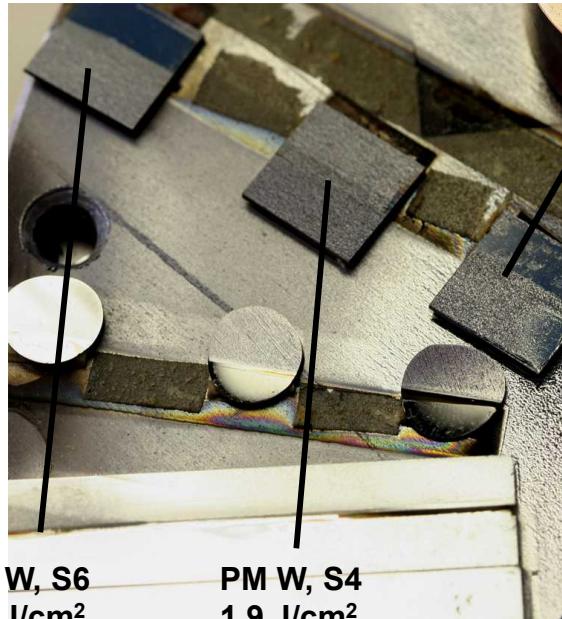
## RHEPP-1 Roughening Threshold, PM W, Multi-pulse

Images and data from KS2000 series: S10 and S8 are sub-threshold for roughening, S6 above (Snead PM W). Note temperature excursions from modeling, based on fluence scatter from FCup data.



PM W, S10  
0.2 J/cm<sup>2</sup>  
270C\_AP  
Hi 415C  
Lo 145C

PM W, S8  
0.6-0.9 J/cm<sup>2</sup>  
1290C\_AP  
Hi 1960C  
Lo 535C



PM W, S6  
1.2 J/cm<sup>2</sup>  
1690C\_AP  
Hi 2278C (5%)  
Lo 1175C (5%)  
 $R_a \sim 2.5 \mu\text{m}$

PM W, S4  
1.9 J/cm<sup>2</sup>  
3070C\_AP  
Hi 3650C  
Lo 2100C  
 $R_a \sim 4 \mu\text{m}$

PM W, S2  
3.5 J/cm<sup>2</sup>  
4300C\_AP  
Melt Duration  
159 ns  
Melt Depth 0.8  
 $\mu\text{m}$   
 $R_a \sim 6-10 \mu\text{m}$

Beam Center

S6 = 6 cm from  
Beam center

Recent series:  
Center gone,  
S10 < Fluence < S5

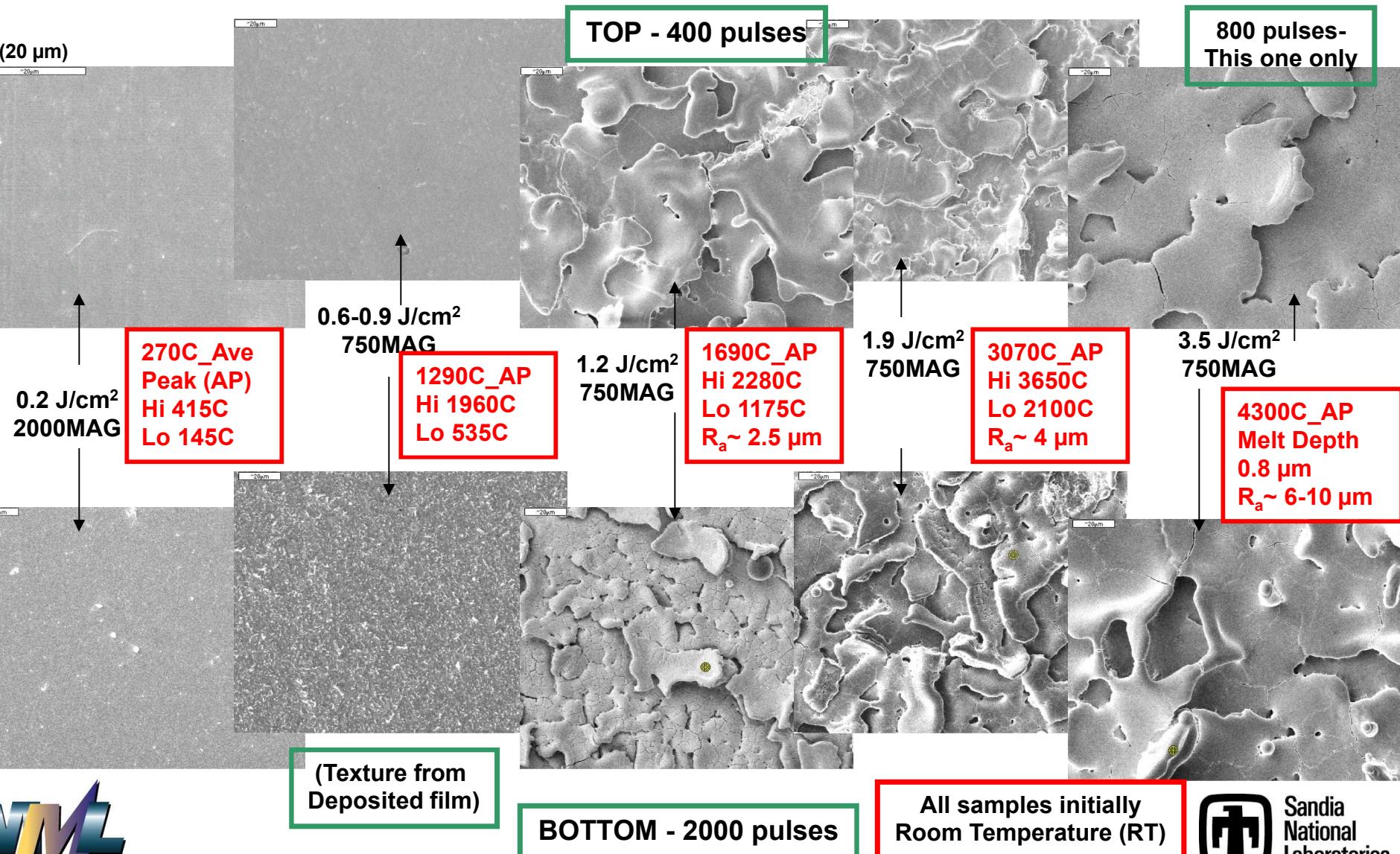
These PM W appear unaffected

These PM W are very rough

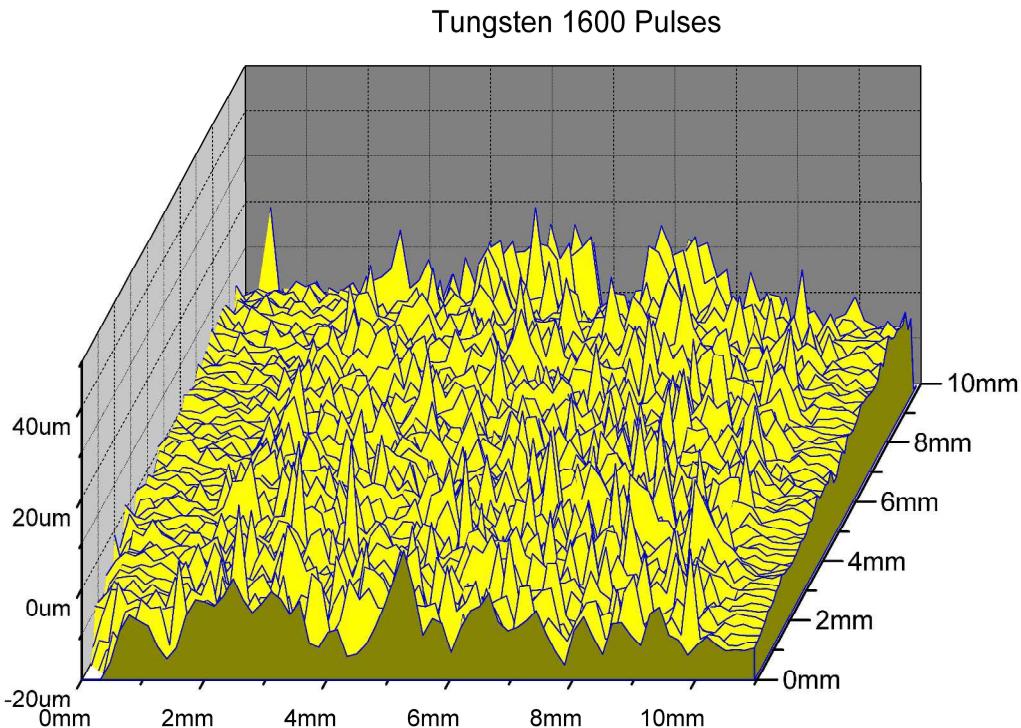
AP = Average High Surface Temp  
Hi and Lo occur on 5% of pulses

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

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



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



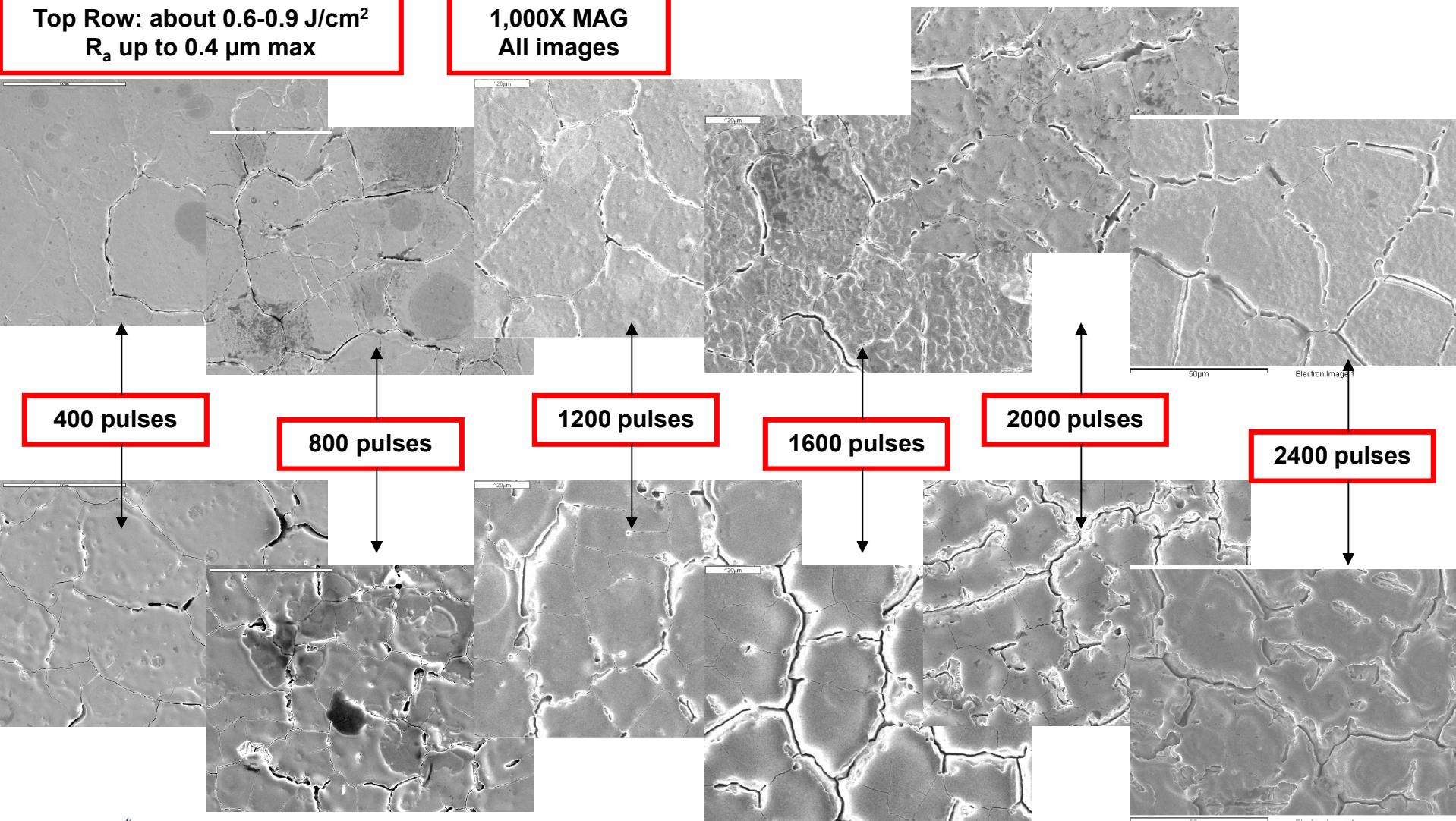
Cannot confirm mass loss by height study. Must weigh before/after exposure

- Heated/treated Powder-Met (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

## SEMS of Tungsten M182 perp after 2400 pulses: Little topology change below 1 J/cm<sup>2</sup>, some roughening w/ pulse number at ~ 1.5 J/cm<sup>2</sup>

Top Row: about 0.6-0.9 J/cm<sup>2</sup>  
R<sub>a</sub> up to 0.4 µm max

1,000X MAG  
All images

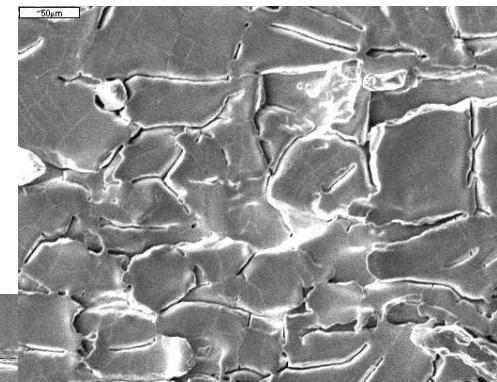
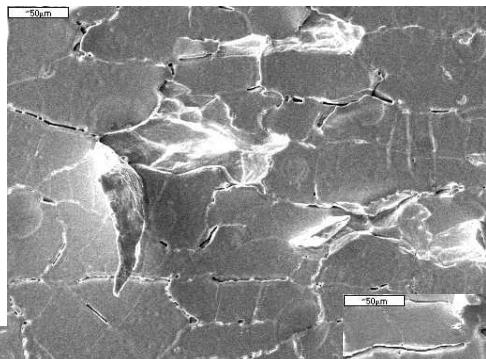


Bottom Row: ~ 1 - 1.5 J/cm<sup>2</sup>  
R<sub>a</sub> 0.35 to ~ 1 µm

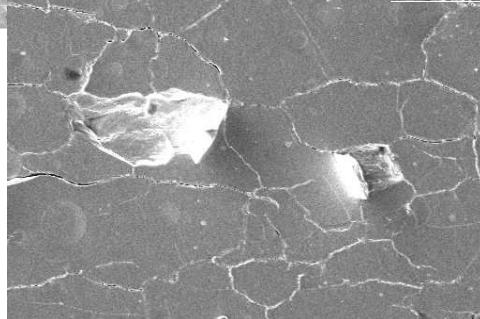
Little or no mass loss to 2400 pulses

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

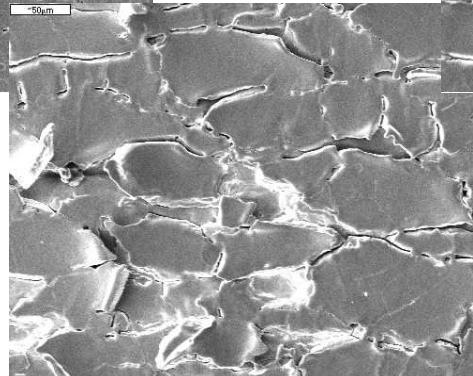
No Treat



1,000X MAG



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

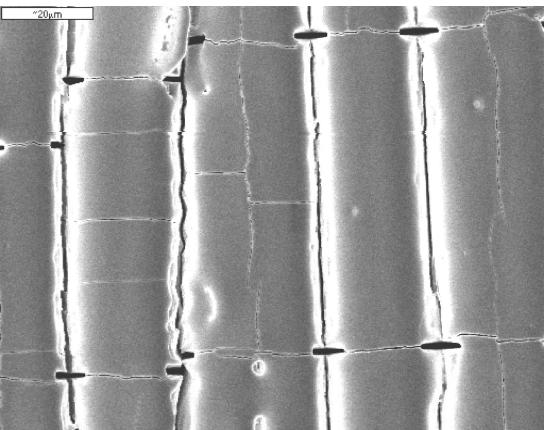


About 1.3  
J/cm<sup>2</sup>

All treated images 300X MAG.

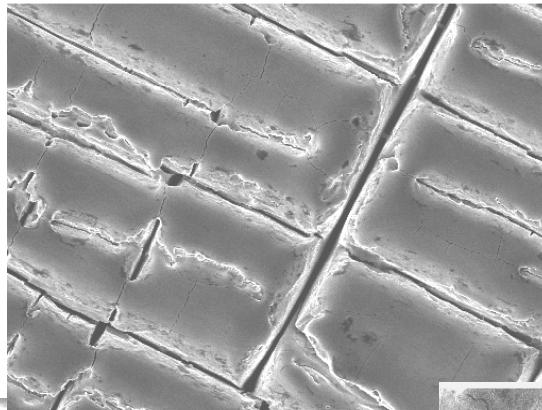
$R_a$  : reaches 4 - 4.5  $\mu$ m at 1.3 J/cm<sup>2</sup> (same as PM Tungsten)  
Only apparent AFTER 400 pulses (these images)

## SEM, Single Crystal Tungsten at 300K, $\sim 1.5 \text{ J/cm}^2$ : Evolution 0.4 to 1.3 $\mu\text{m}$ $R_a$ , erosion around crack edges indicated

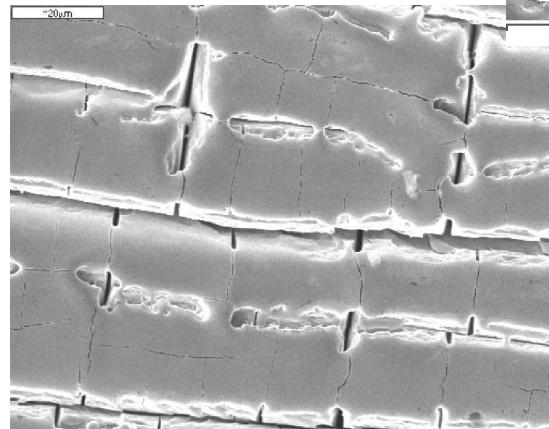


400 pulses

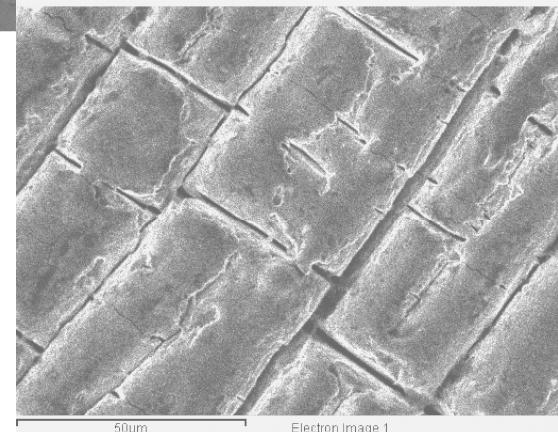
800 pulses



1600 pulses



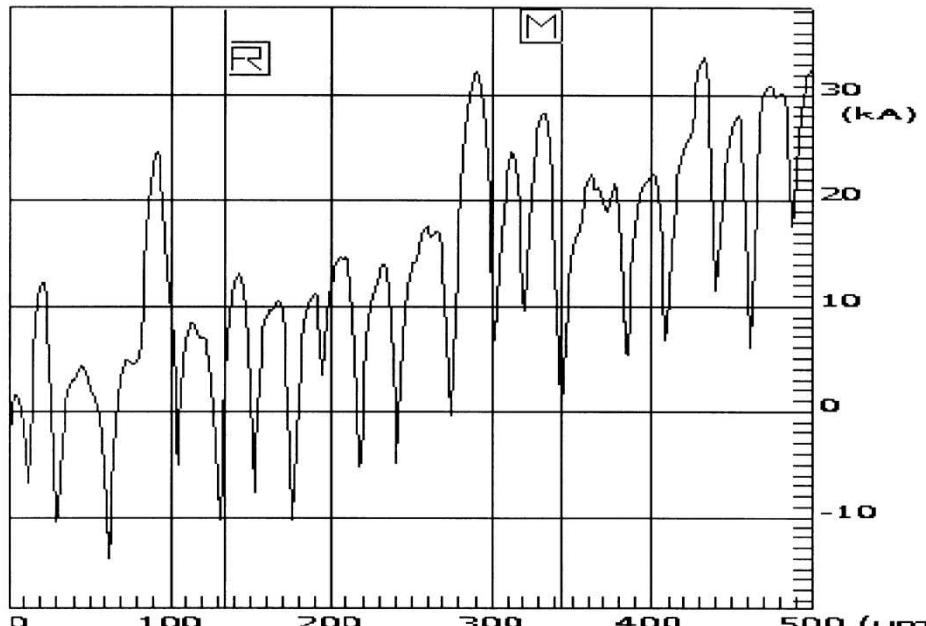
1200 pulses



This Retest of KS2000 at Request of MWG, with accompanying Mass Loss data. Morphology evolution looks the same as KS Series

KS2 Shot series 8/06 - 7/07

## Single Crystal Tungsten at 300K, Weight 'Loss' Estimate: Implied mass loss from morphology profile not seen in measurements



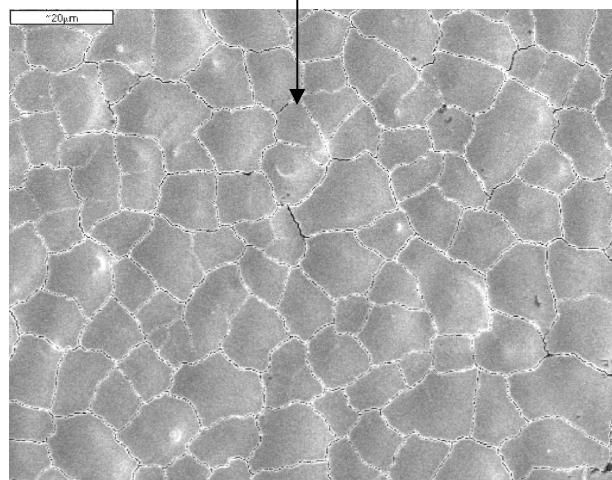
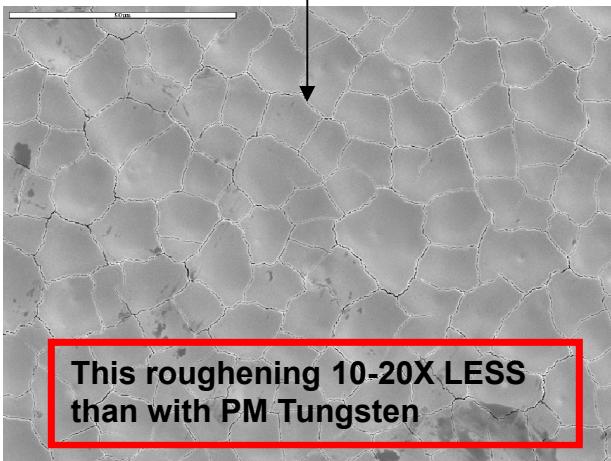
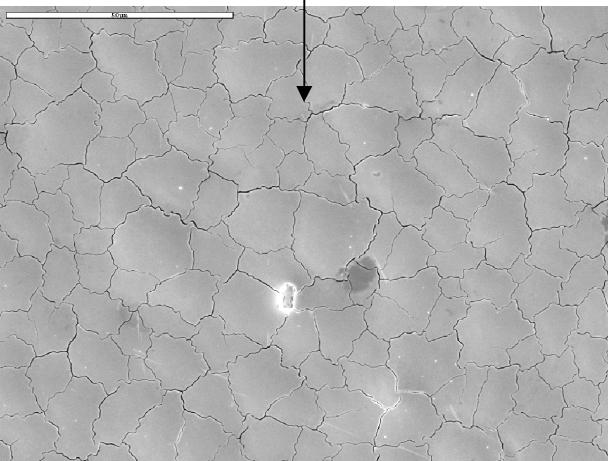
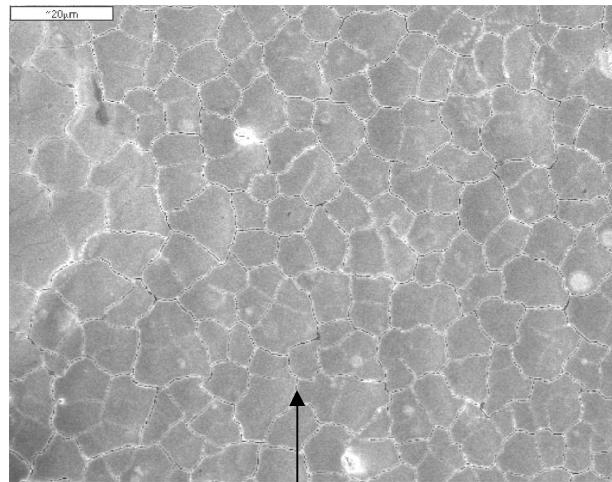
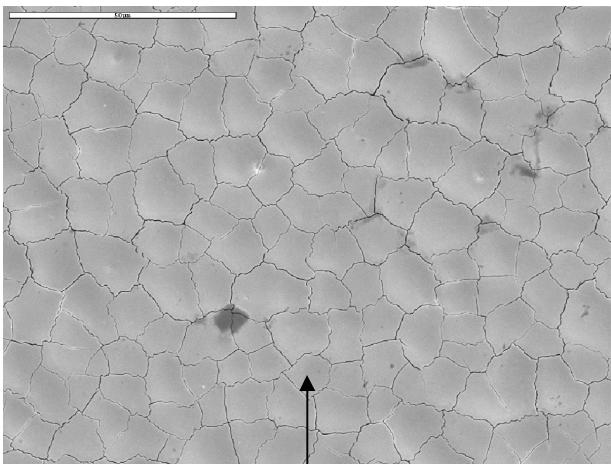
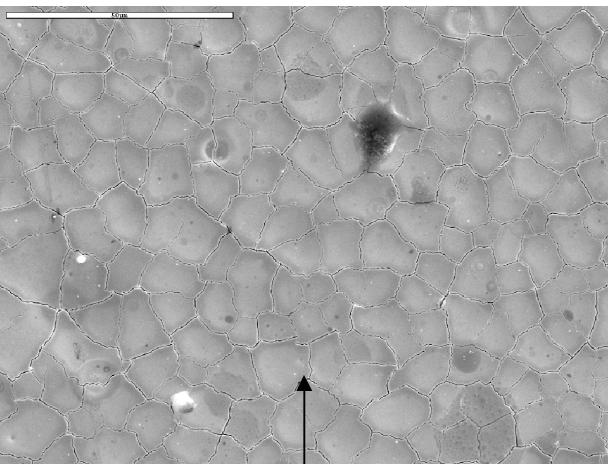
Dektak Scan - 800 pulses

- [Left] 1-D Dektak of Single-crystal W surface shows periodic bumps at 800 pulses. Similar scans at 400 and 1200 pulses show same qualitative features -  $0.5 - 1 \mu\text{m} R_a$ , and Peak-Valley heights up to 70  $\mu\text{m}$ .
- Assume profile of 'flattened' triangular cross-section - height 25  $\mu\text{m}$ , spacing 20  $\mu\text{m}$  in rows, 5  $\mu\text{m}$  flat-top in-between. Calculate volume implied by 'missing' triangles.
- Sample width 0.85 cm. Get 425 grooves, each 600  $\mu\text{m}$  long. Total 'missing' volume  $4.78\text{e-}5 \text{ cm}^3$ , and @ 19.3 g/cc this is  $\sim 920 \mu\text{gr}$ .
- There are transverse grooves, so this is an underestimate.
- Totals for Single Crystal sample tested (1600 pulses total): +11.5, -69.4, +34.9, +38  $\mu\text{gr}$  = 15  $\mu\text{gr}$  net GAIN. Error bar in this number is  $\pm 30 \mu\text{gr}$ .

# 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

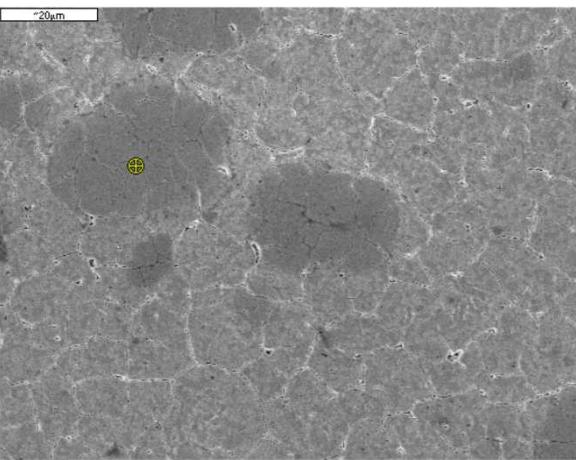
From H. Kurishita (Tohoku U.)



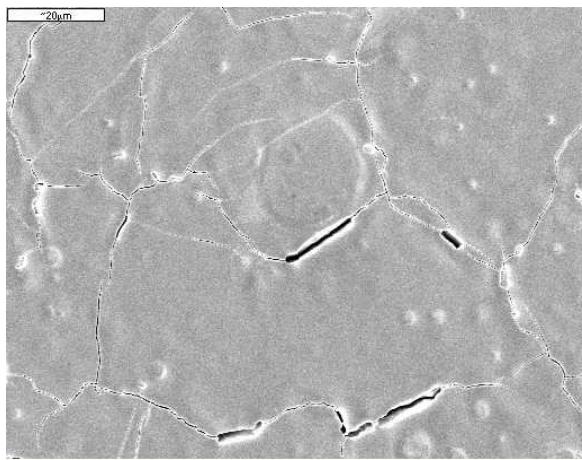


Three forms of Tungsten, treated at ~ same fluence (400 pulses):  
Grain-refinement/strengthening, or below-surface burial  
seem to restrict roughening/mass-loss.

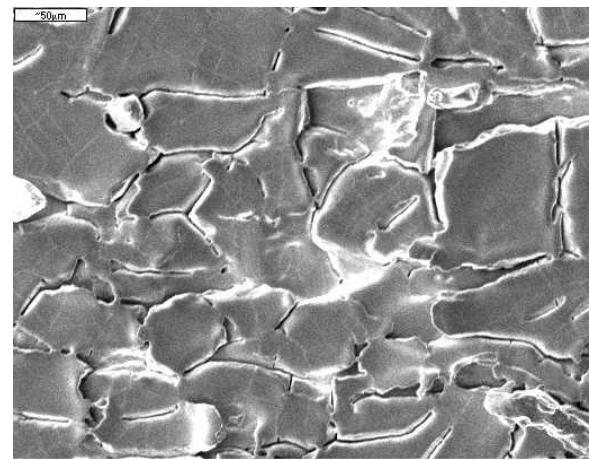
1,000X MAG



1,000X MAG



300X MAG



W-0.5%TiC 1.5 J/cm<sup>2</sup>.  
Ra = 0.04  $\mu$ m

M182Perp ~ 1.25 - 1.5 J/cm<sup>2</sup>  
Ra ~ 0.15  $\mu$ m

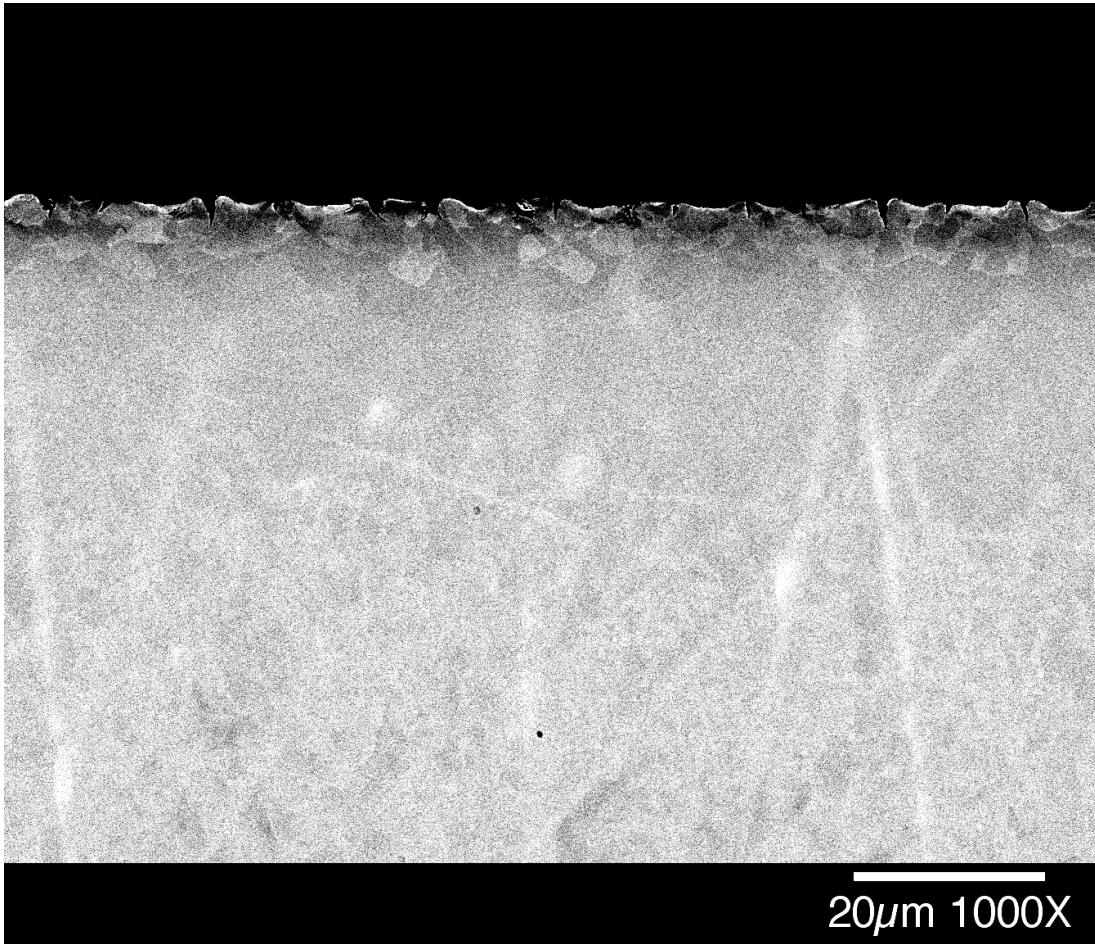
M182Parallel ~ 1.3 J/cm<sup>2</sup>  
Ra ~ 4.5  $\mu$ m

Two on right are SAME material



## Treated N6 2000X (520C) SEM: Clear evidence of recrystallization

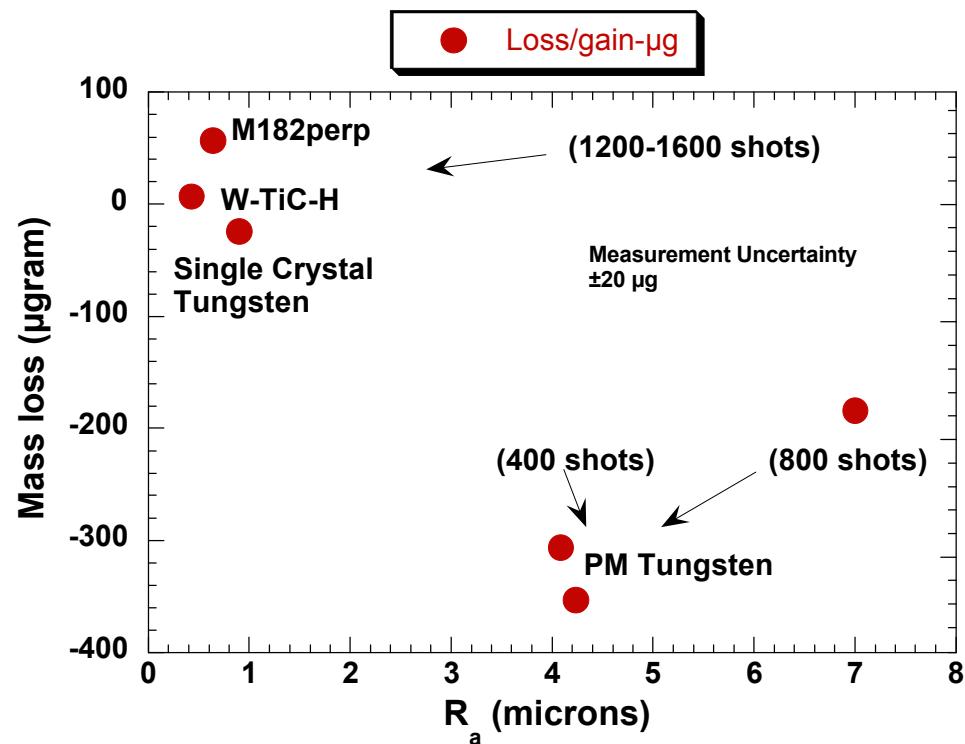
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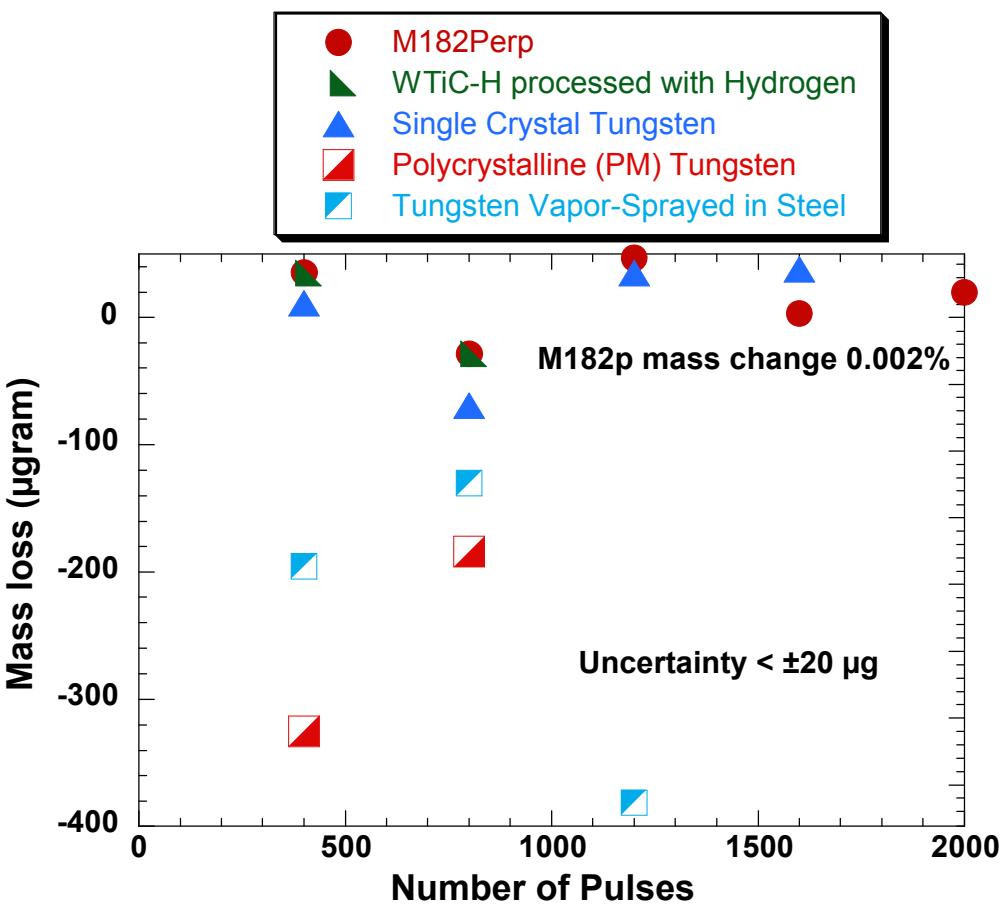
# Mass Loss Measurements

# 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  $\text{J/cm}^2$   
Measurement Uncertainty  $< \pm 20 \mu\text{g}$
- Two samples of polycrystalline (PM) Tungsten lost  $\sim 350 \mu\text{g}$  in 400 pulses, with  $R_a$   $\sim 4 \mu\text{m}$ ; another 400 pulses produced even more roughening,  $-184 \mu\text{m}$  more mass loss
- M182Perp, W-TiC-H, and Single Crystal Tungsten remained  $< 1 \mu\text{m}$   $R_a$ , and suffered little mass loss.

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

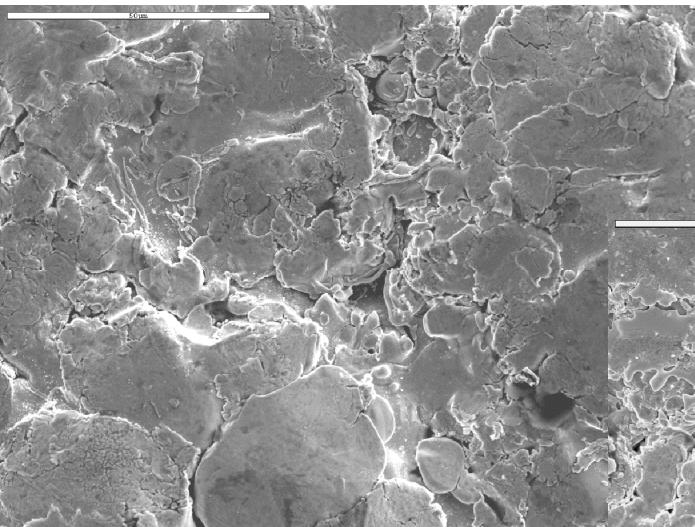


- Mass loss for PM Tungsten terminated after 800 pulses - 350 and 184 µg loss on two 400 shot sets
- Vapor-Sprayed Tungsten (on Ferritic Steel) losses up to 400 µg per 400 pulses
- Mass Gain due to entrained material (Cu) from diode region

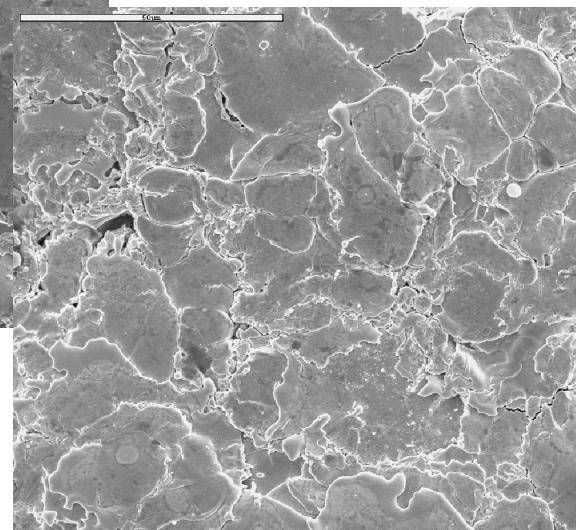


## VPS Tungsten (SEMs): slight roughening, Evidence of scouring/erosion at S6

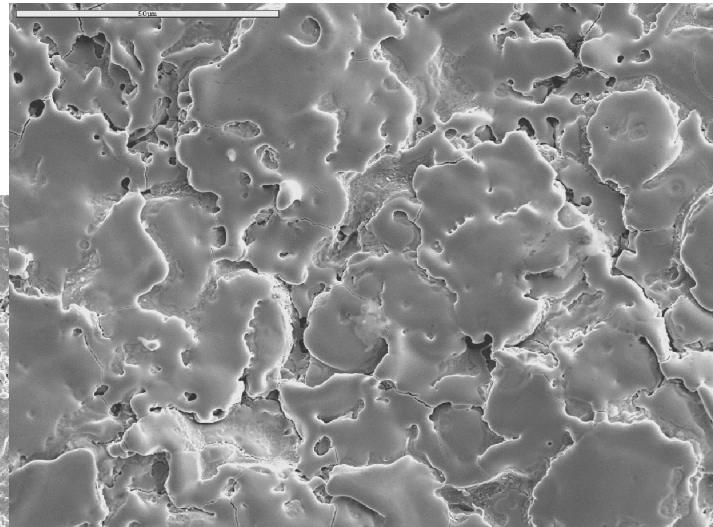
Near S6 No Treat



400 pulses



S8 - 0.6 J/cm<sup>2</sup>



S6 - 1.1 J/cm<sup>2</sup>

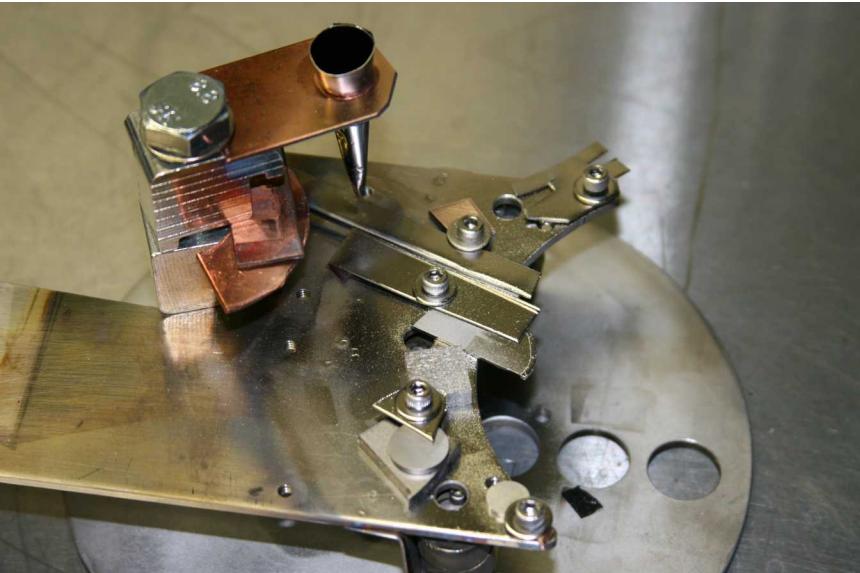
1,000X MAG

R<sub>a</sub> increases from ~3.1  $\mu$ m to 3.3  $\mu$ m at S6

Mass Loss = 195.5 $\mu$ g

# Sample Images after KS2\_2400 Series (Nitrogen)

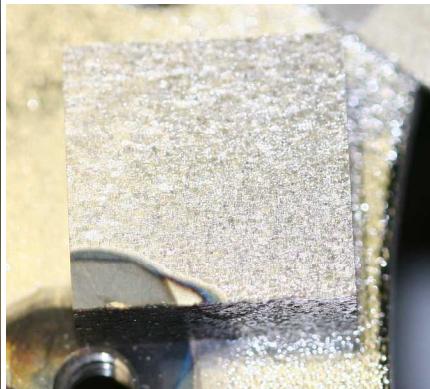
Shown after 400 shots  
Beam Center off to Right



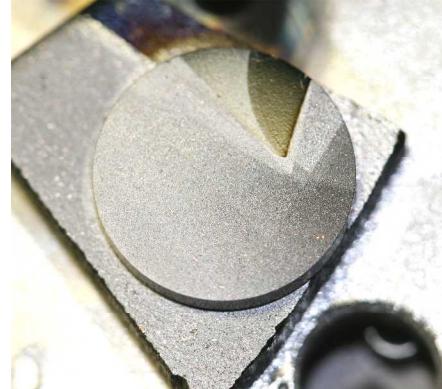
- Samples:
  - M182Perp: ~ 0.75 - 1.15 J/cm<sup>2</sup>
  - W-0.5%TiC-A, R ~ 6cm to 3.5 cm
    - Sample fell off after ~ 100 shots
  - W-0.5%TiC-H, R ~ 6cm to 3.5 cm
  - W25Re: R=4.5 cm to beyond 9cm radius
  - M168 Polycrystalline Tungsten: ~ 1.15 - 1.5 J/cm<sup>2</sup>
  - Ta Cone at large radius
- Only 2 FCups worked: Center (1.7 J/cm<sup>2</sup> ave  $\pm$  30%) and Bottom (1.15 J/cm<sup>2</sup> ave  $\pm$  25%). Sample exposure normalized to closest FCup
- Vacuum ~ 1e-5 Torr. Ta Cone LOST 1 mg. We need to repeat more systematically to understand mass loss numbers better



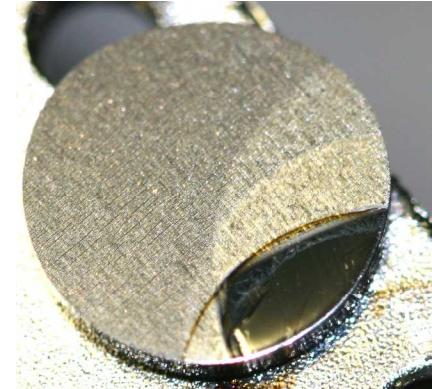
Ta Cone:  
deposition  
on cone  
sides



M168 Poly W



M182Perp (L) Single Crystal (R)



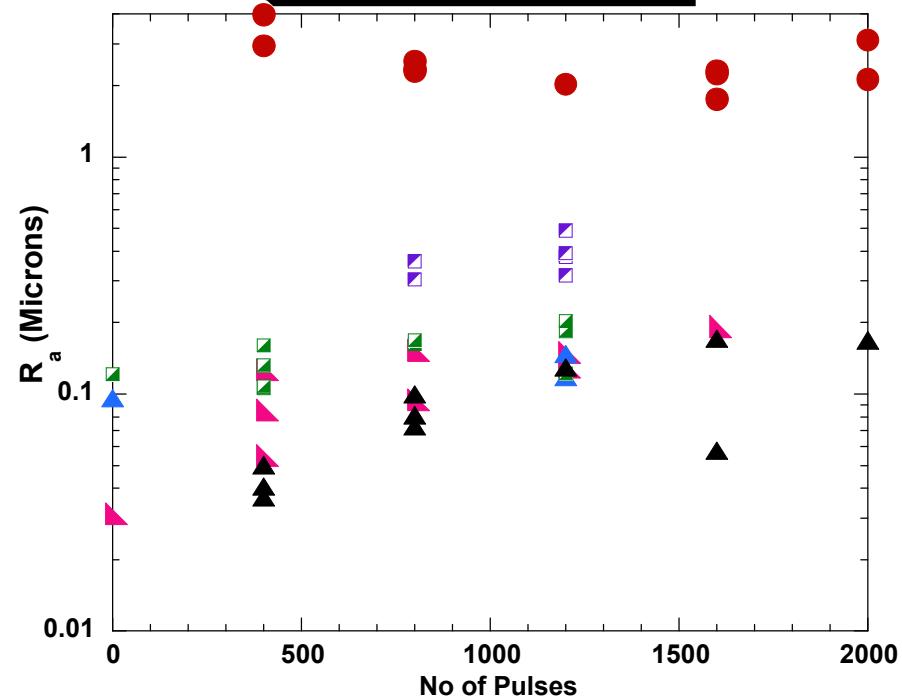


# Roughness Measurements

## RHEPP-1 Surface Roughening, KS2000 Series + KS2-1200

### Evolution of $R_a$ Roughness, PMW + SingXtal, other Ws: PM W is roughening Champ, but only one that tends to saturate

- PMW RT S6
- ▶ WSingXtal RT S6
- ▲ W-0.5TiC S6 Stressed
- M182 Perp unstr-L 0.7-1.0
- M182 Perp unstr-H 1-1.3
- ▲ W25Re RT S6

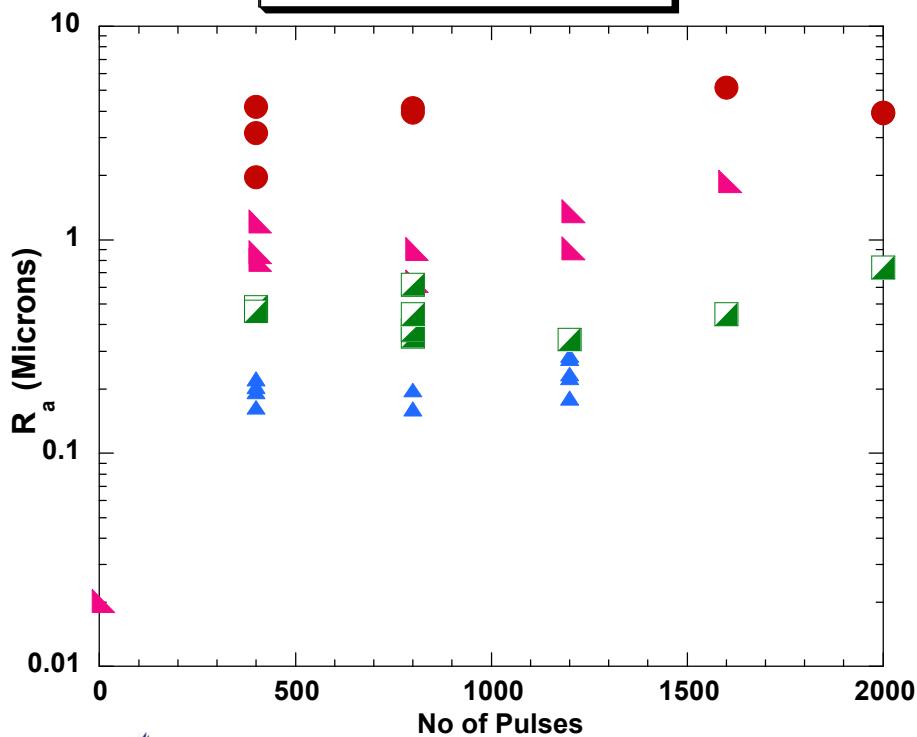


- Red Dots - PMW RT S6, for comparison ( $1.1 \text{ J/cm}^2$ ). Data saturates above 400X
- Pink - SingXtal RT S6:  $R_a$  starts low, but increases w/o saturating
- W-TiC stressed, M182 perp comparable to W25Re.

## RHEPP-1 Surface Roughening, KS2000 Series + KS2-1200

### Evolution of $R_a$ Roughness, PMW + SingXtal, other Ws, S4: PM W is roughening Champ, but only one that tends to saturate

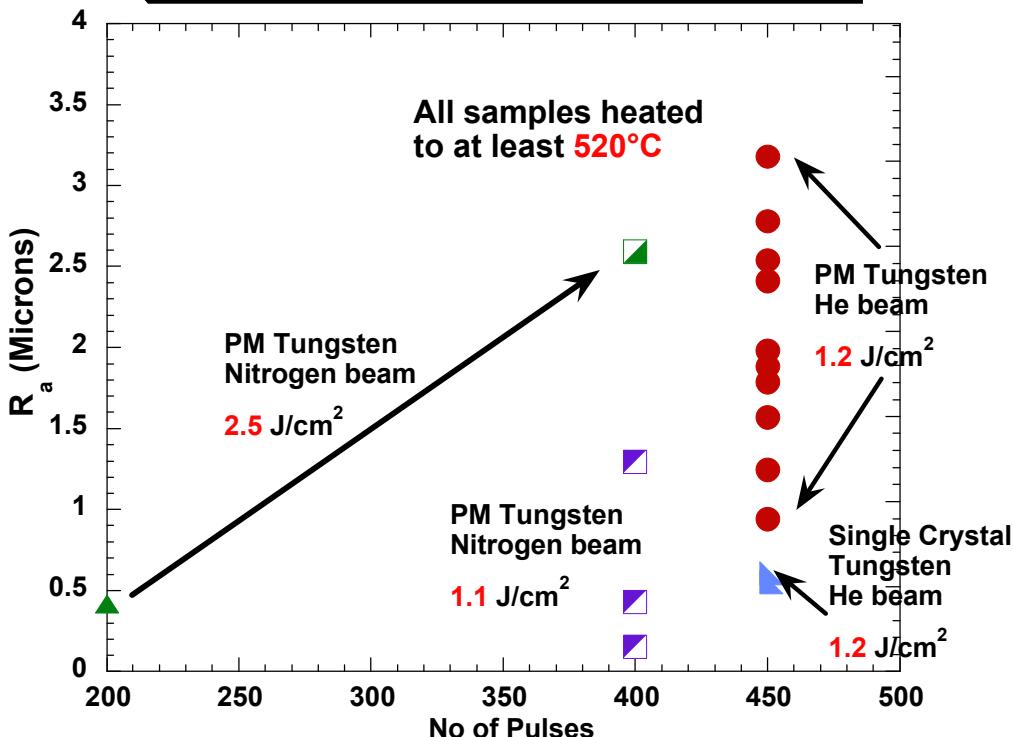
- PMW RT S4
- ▲ WSingXtal RT S4
- ▲ W-0.5TiC S4.2 Stressed
- W25Re RT S4



- Red Dots - PMW RT S4, for comparison ( $1.9 \text{ J/cm}^2$ ). Data saturates above 400X
- Pink - SingXtal RT S4:  $R_a$  much higher than for S6
- W-TiC stressed remains low, W25Re exceeds W-TiC

## 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



# Melting the Surface - Experiments

# 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

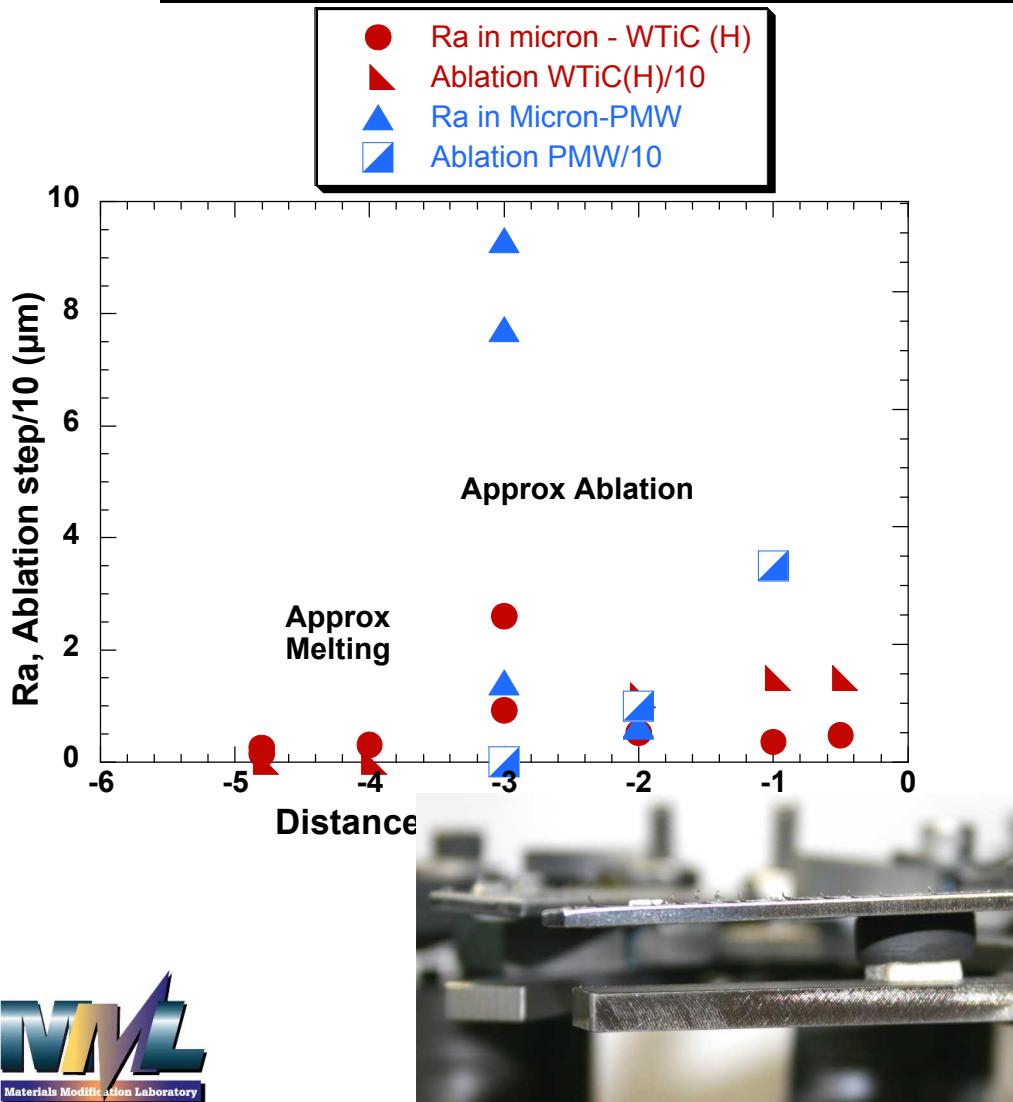


Nitrogen beam

- 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



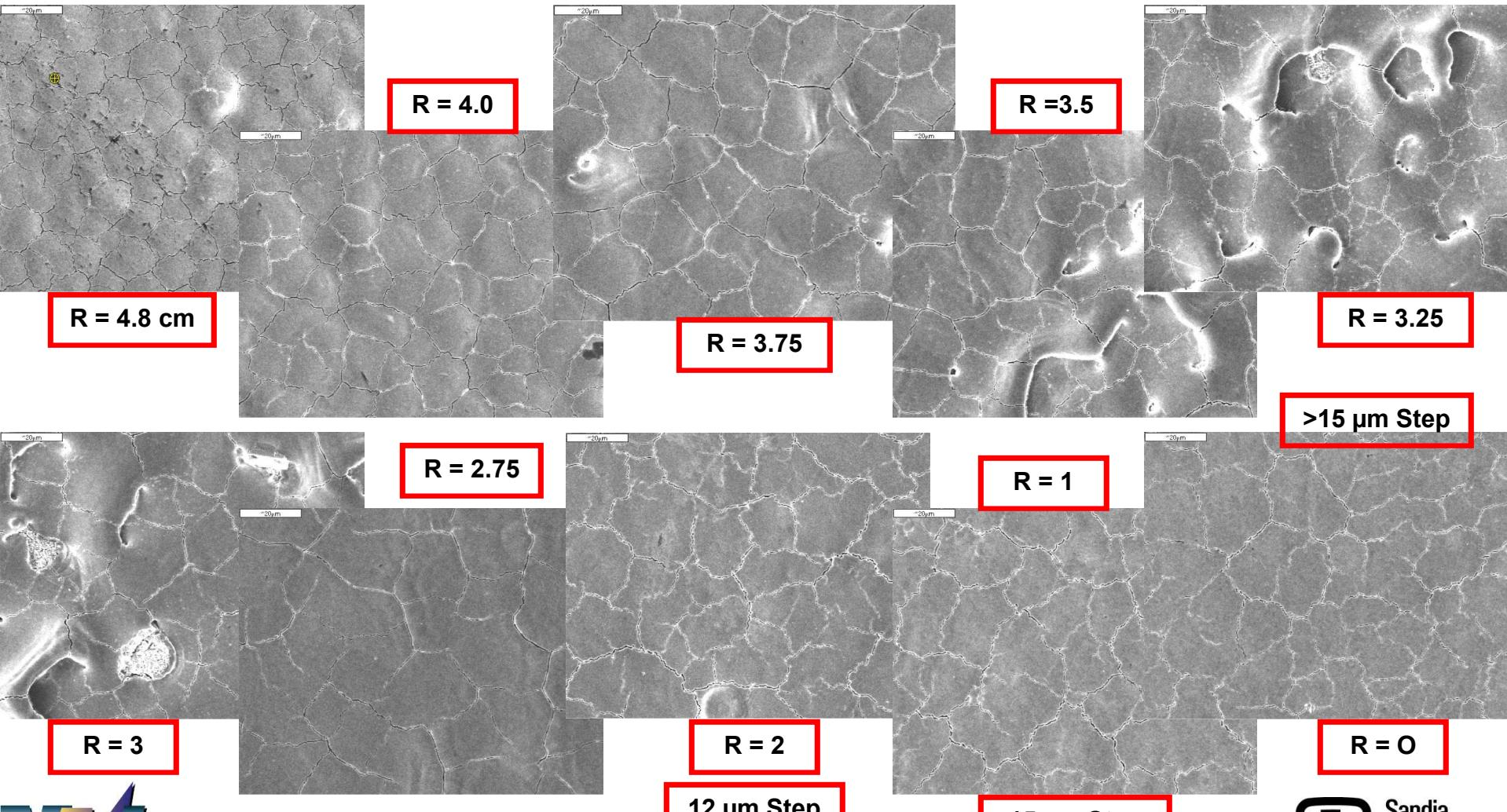
## Roughening behavior of W-0.5%TiC(H), PM W at high fluence: R<sub>a</sub> highest at ablation onset. Melting leads to increased roughness



- 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<sub>a</sub> 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 (> 900  $\text{\AA}/\text{pulse}$  removed)
- BIG surprise: W25Re. Hardly ANY roughening.**
- (LEFT): 'fingers' protruding from W shield

Nitrogen Beam

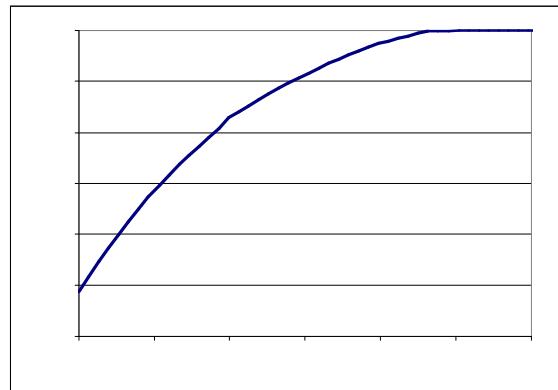
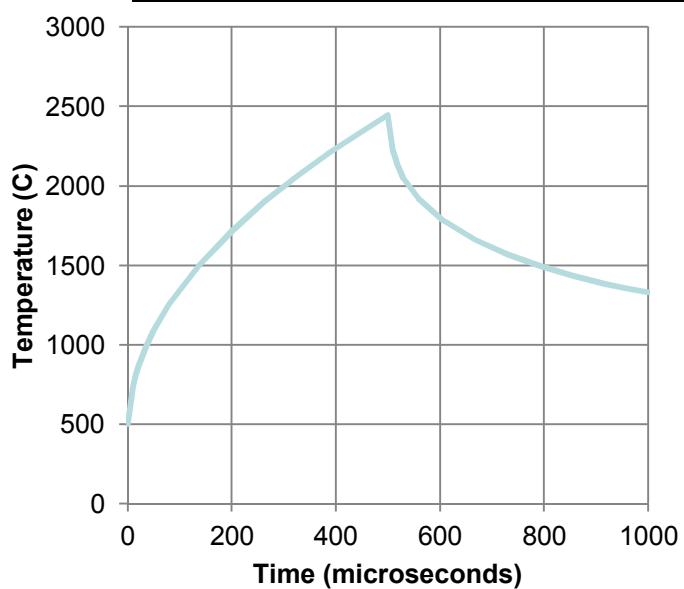
## SEMs, W0.5%TiC (H), S4.8 to BEAM CENTER: Same roughness at both extremes, 15 $\mu\text{m}$ Step at R = 1 cm (400 pulses)





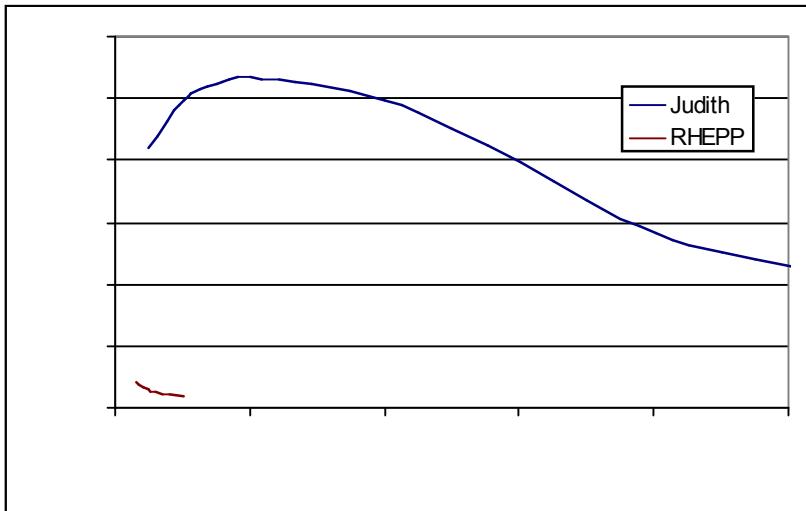
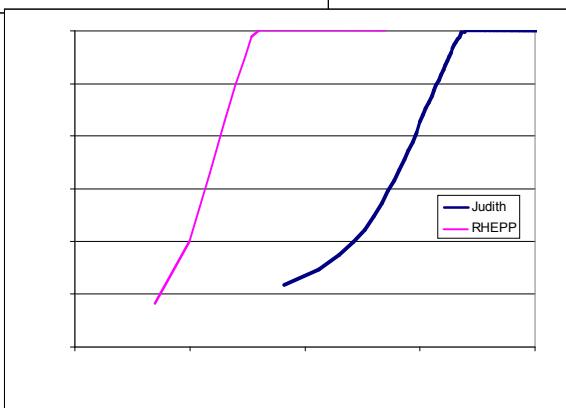
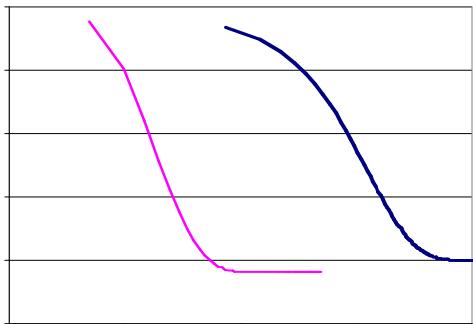
# Stress Modeling (Jake Blanchard)

## 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.  
 $T_{initial} = 500C$ , 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



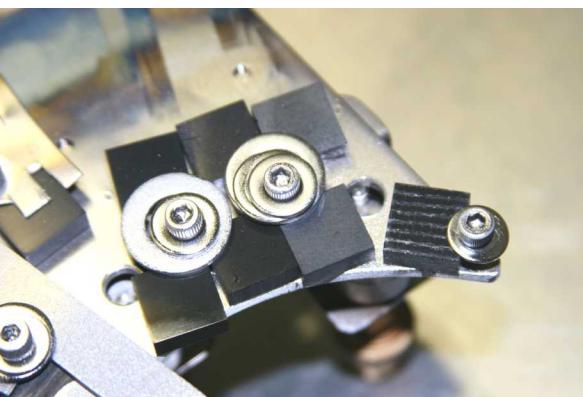
- (Top Left) RHEPP pulses with fluence chosen to reach same surface maximum temperature - 2500C
- (Mid Left) Plastic Strain Curves: Both effects MUCH deeper for the ELM case
- Bottom: Stress Intensity for the 'ELM' case at 25 MPa-m<sup>0.5</sup> - at fatigue cracking threshold for tungsten (20-40 MPa-m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



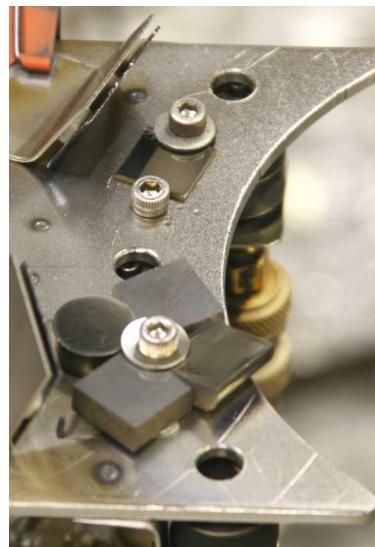
# Carbon and Velvet Materials

## 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

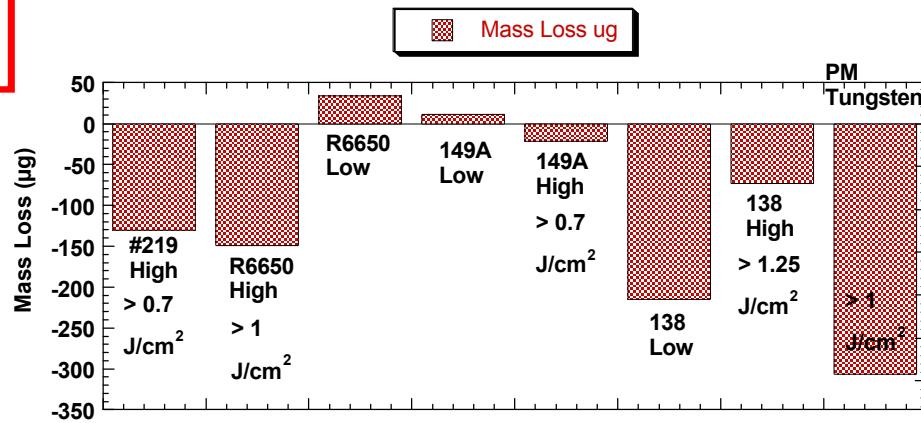


(Above) After 400 shots



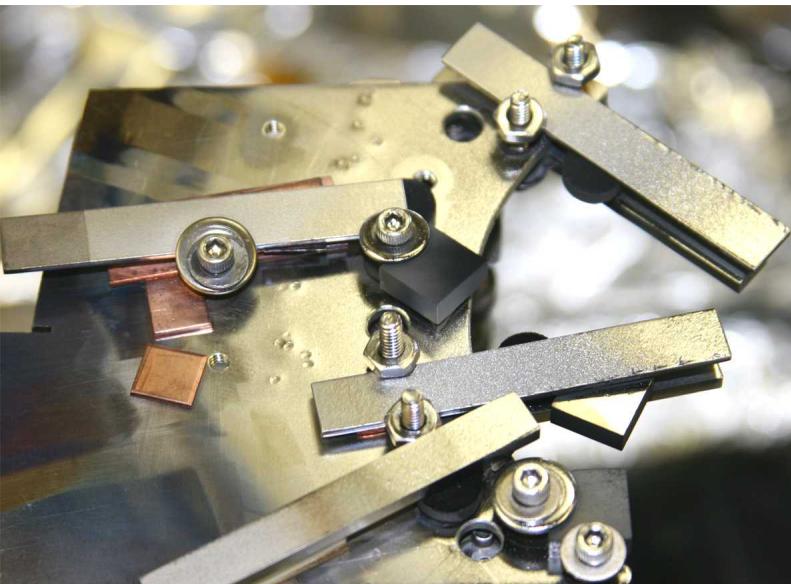
(Above) REFERENCE  
PM Tungsten between  
screws

- 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.



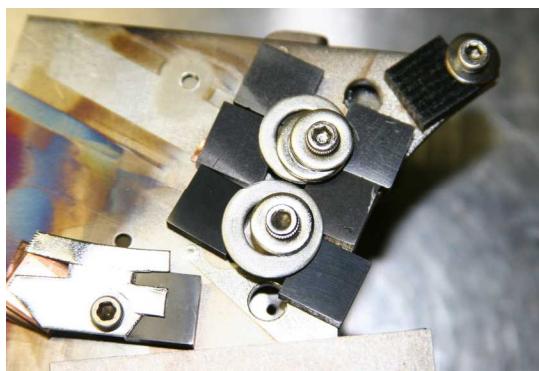
MAP Nitrogen

## 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  $\mu$ m, 2  $\mu$ m step even at 1.3 J/cm<sup>2</sup> (50Å ablative loss/pulse)
  - Pyrolytic: almost 4  $\mu$ 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  $\mu$ m, mass loss likely but not confirmed



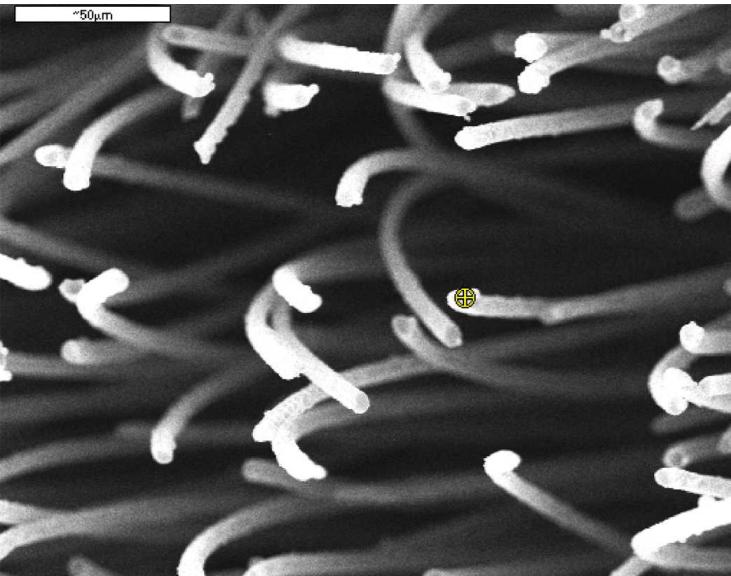
Earlier C400 Series  
(from Previous Slide)

## RHEPP-1 Surface Roughening

# Tungsten-coated Carbon Velvet survives 1600 pulses amazingly well

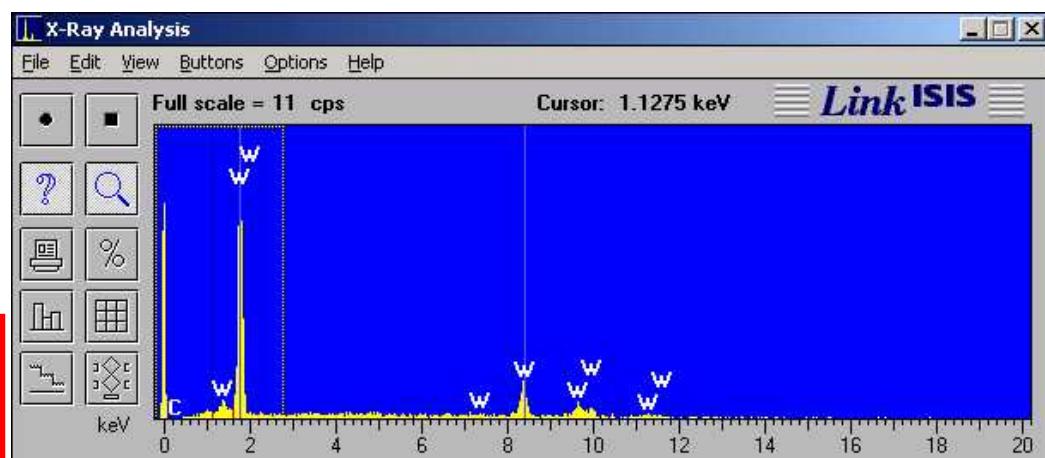
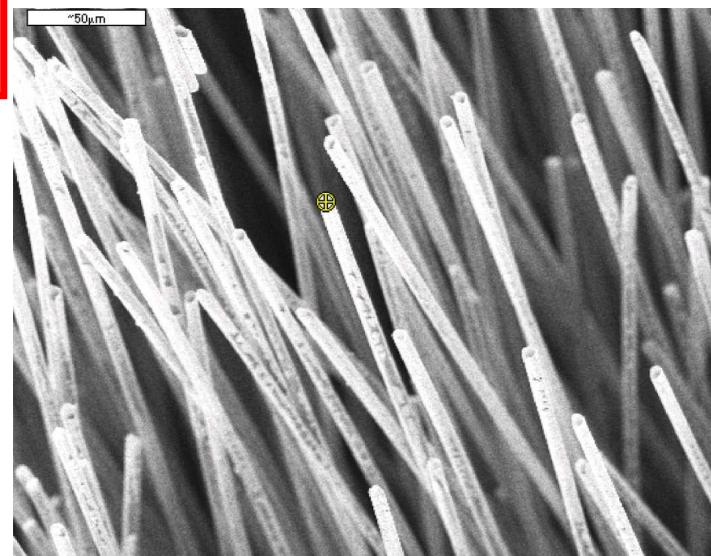
From T. Knowles, ESLI

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



(RIGHT)  
520C (nominal), 1600  
pulses, 1.5  $\text{J}/\text{cm}^2/\text{pulse}$

NOTE: W remaining  
on tips (see below)  
and sides



EDS scan of tip (cross): W rich