IFE Materials Response: Long-term exposure to nitrogen and helium beams on RHEPP

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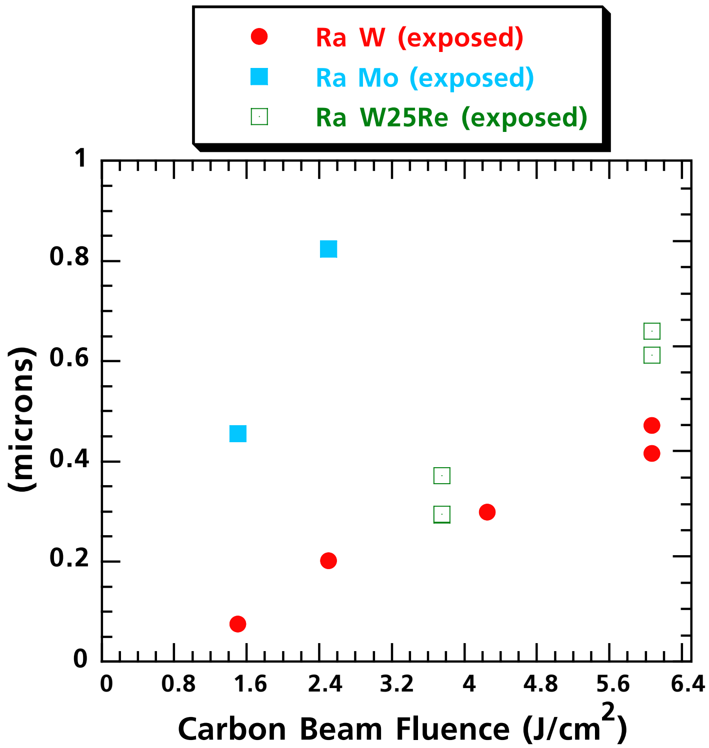
High Average Power Laser Workshop
Madison, WI
September 24-25, 2003

Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under contract DE-AC04-94AL85000.

Roughening threshold for 600C W predicted ~ 1.25 J/cm^2; Looks like W/25Re has similar roughening, Mo worse



Melting fluence:
Mo: 2.2 J/ cm^2
W: 2.5 J/ cm^2

- Smoothed W and Mo samples, provided by Lance Snead, were exposed to 250 MAP N pulses from ~ 1.5 to 6.1 J/cm^2
- Sample temperature held to 580 < T < 600C - above brittle-ductile transition temp for both Mo and W
- Roughening threshold for pure W appears to be ~ 1.25 J/cm^2 , same as previously measured at RT by reflectometer. Similar scaling with fluence for W25Re
- Mo exposed at 1.5 - 2.5 J/ cm^2 roughens worse than heated W
- Both W and Mo appear to roughen below melt

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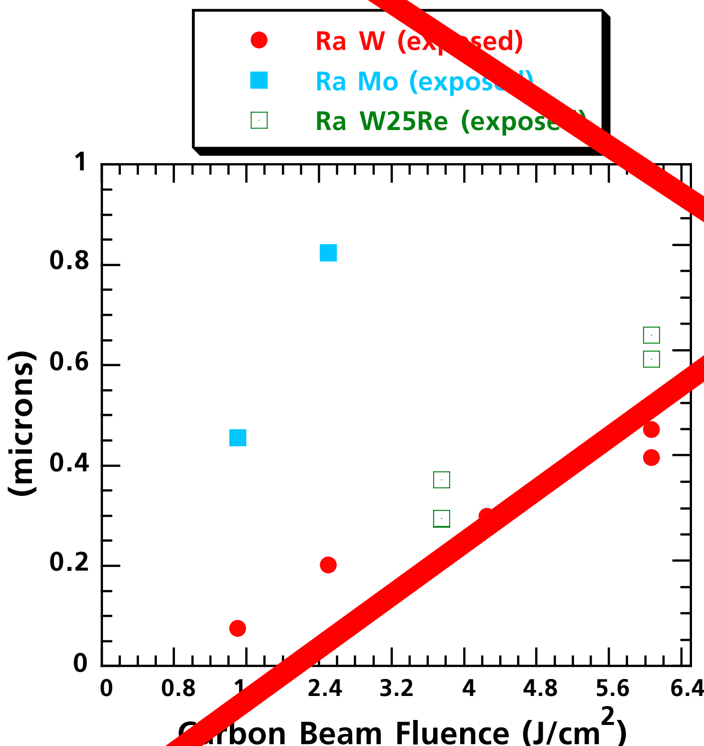

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Executive Summary

- Materials response to pulsed ion exposure is affected greatly by grain structure. Roughening appears species-dependent for same dose, - He ions roughen worse than N.
- Roughening: is worst for PowderMet W, heated or not. Response varies significantly amongst metals tested, - Cu roughens hardly at all, for example. Roughening evolves over hundreds of pulses, and appears open-ended. Smaller-grain PowderMet W might help.
- Deep-lying cracking evident with W, W25Re, and Re. Depths are in the tens to hundreds of microns, and appear consistent with fatigue/stress cracking. Too deep for DekTak/WYCO to see.
- CFC Graphite (222-FMI) suffers material loss over 1000 pulses at the 1.6 J/cm^2 level, well below predicted ablation threshold.
- Foams seem to suffer material loss over 800 pulses, but hard to tell exactly.
- C-C-W Velvet (Knowles) subjected to 200 pulses up to 7 J/cm^2, survives amazingly well.
- Copper contamination an issue, but does not change major conclusions

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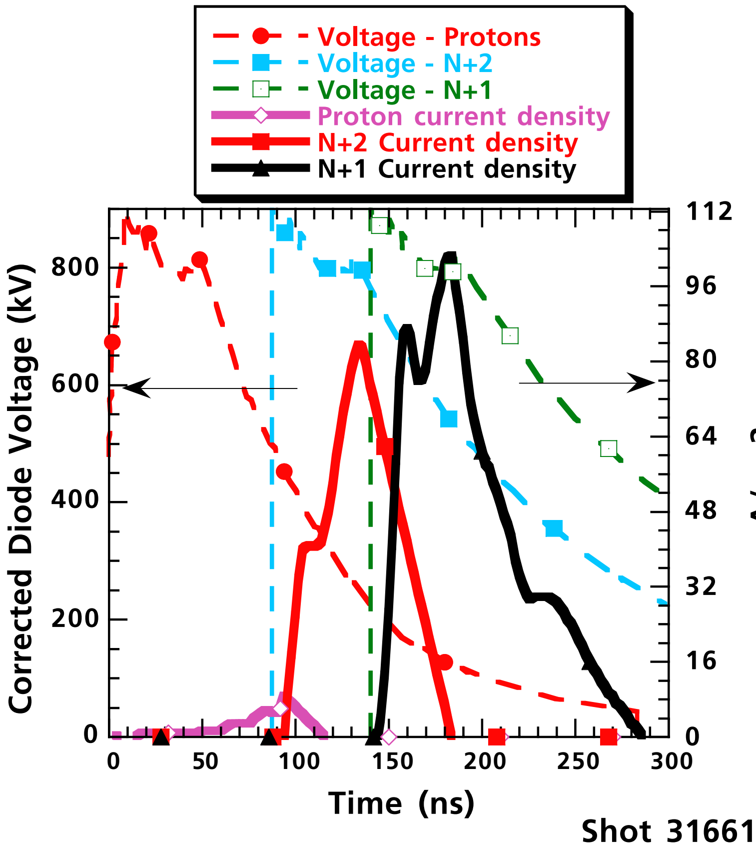
Extended shot series taken since June 2003

- 450x Helium Series:
  W, Mo Powder Met W
  CVD W
  SingXtal, all 600C
  W25Re, Re unheated

- 1000x Nitrogen KS Series: W,
  Mo Powder Met 600C
  W25Re, Re CFC,
  Foams Ti-2,
  Al1100, Cu

- 600x Nitrogen series:
  W, Mo Powder Met 600
  C CFC, Foams higher fluence

Nitrogen injection into MAP produces 3-component beam of mostly N++, N+



- Beam predominantly N++ and N+ after small proton pulse at front
- Peak voltage = 850 kV
  Peak current density (total) ~145 A/cm²
- Total fluence = 7.9 J/cm² - will ablate almost all materials
- Total pulse width ~ 200 ns
- Ion range (TRIM):
  - N+ 0.9 µm, N++ 1.2 µm
- Oxygen, Neon beams similar

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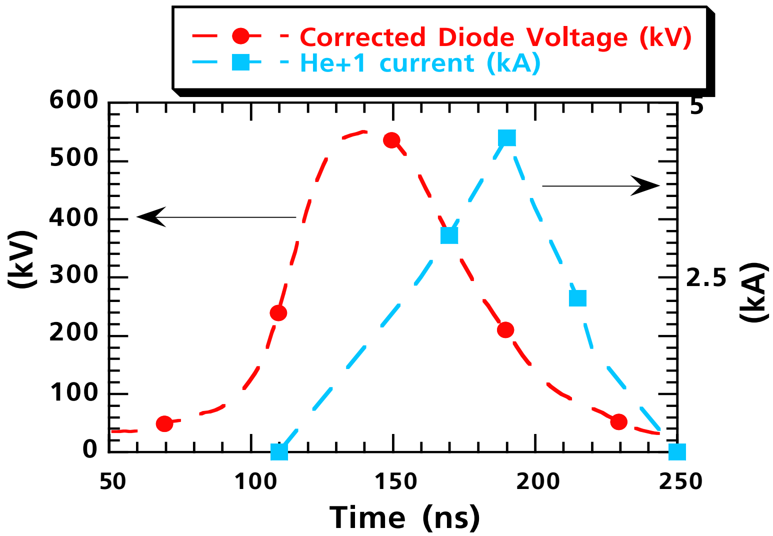
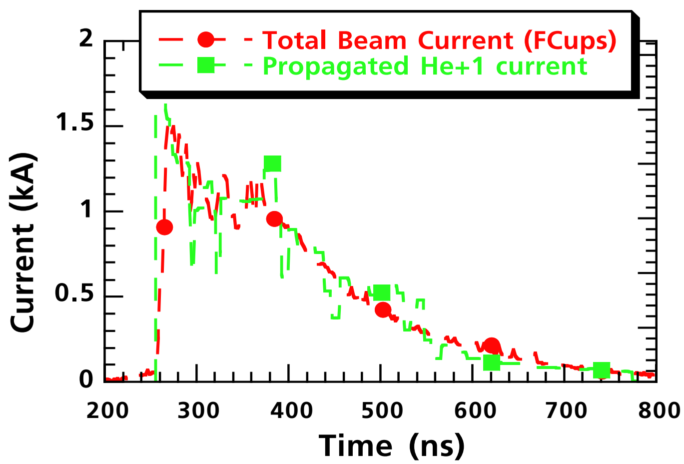
He beam (HE^{+1}) pulsewidth is longer than N or C beam



- He current lags voltage, leading to debunching
- Current pulse width at 63 cm is almost 0.5 µsec long
- Beam here was intentionally attentuated
- He range in W (TRIM) ~ 0.9 µm
- Range similar to N beam because singly charged



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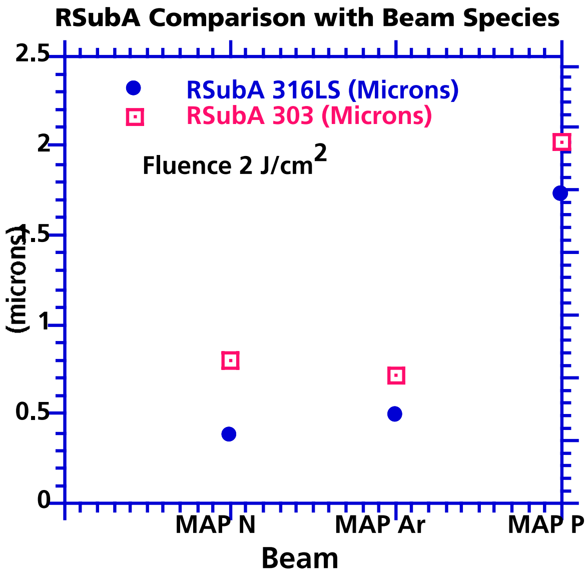
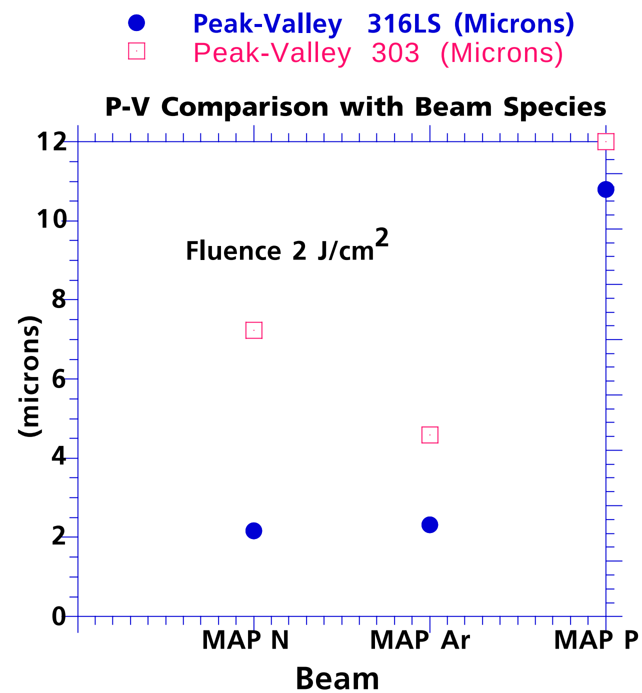
Roughening of SS303, SS316LS varies with ion beam species

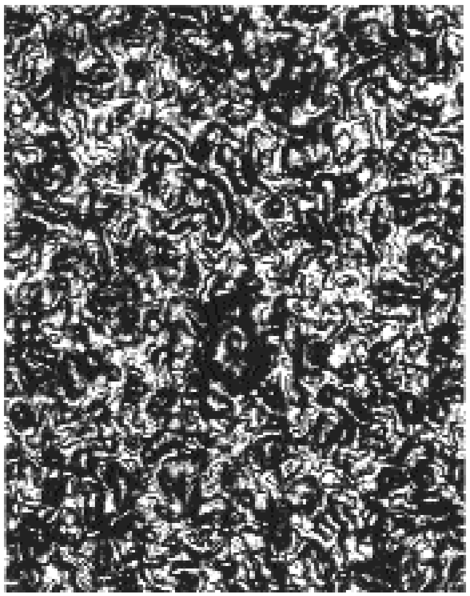


- Polished SS303, 316LS exposed to 25 pulses each of 3 beams at 2 J/cm^2:
  - proton
  - nitrogen
  - argon
- Proton beam produces most roughening (R_a and peak-valley)
- 303 roughens more in all cases then 316LS
- MAP N and MAP Ar produce similar roughening, Ar smaller peak-valley



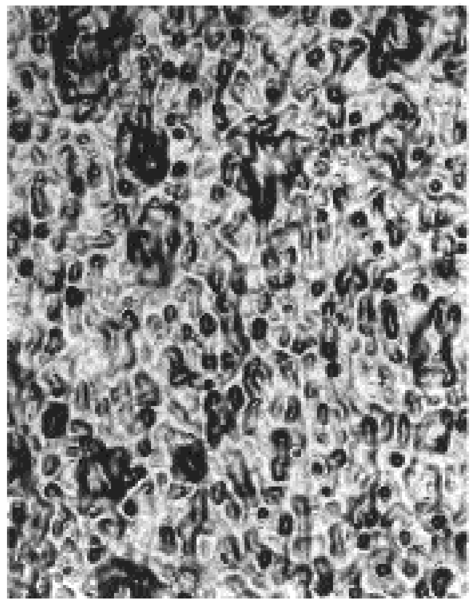
Surface morphology changes on SS samples depend upon treatment ion(s)

Map P 25x



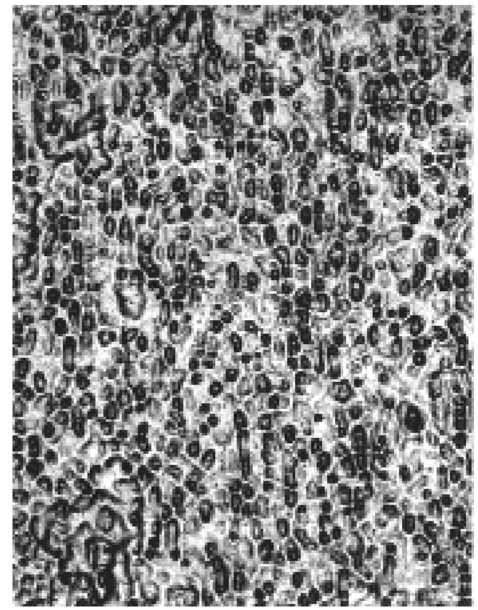
Modeling (Fe):
Max Temp: 1658K
No melting

Map N 25x



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

Map Ar 25x



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

- SS303 exposed to 25 shots each Map P, Map N, Map Ar @ 2 J/cm^2
- 'Finer' surface features with N, Ar
- Periodicity ~ 50 μm for N, Ar surface, ~ 70 μm for proton-treated surface

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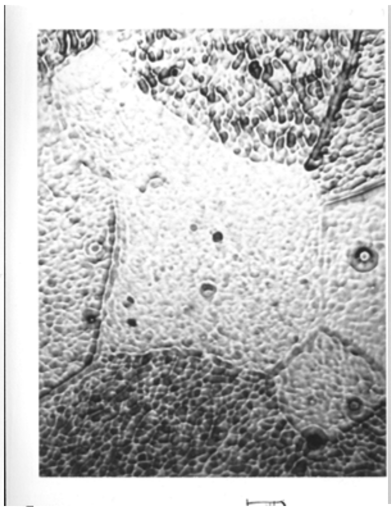
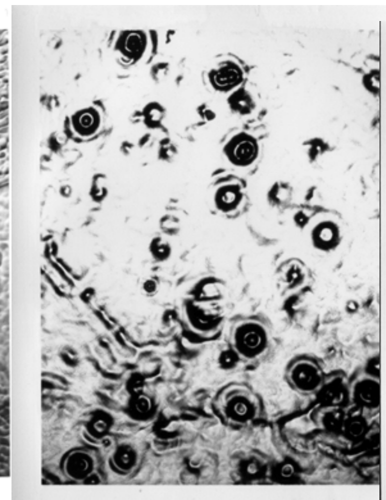
RHEPP-1 on Ti Alloy with 160-pulses O beam: Materials response governed at grain level

~2.0 J/cm^2
R_a ~ 0.15 μm
Ablation:
Minimal
Melt depth:
1.5 μm



~2.3 J/cm^2
R_a ~ 0.25 μm
Ablation:
~25 Å/pulse
Melt depth:
1.75 μm



~2.7 J/cm^2
R_a ~ 1 μm
Ablation:
~300 Å/pulse
Melt depth:
~2.25 μm

~4.0 J/cm^2
R_a ~ 2 μm
Ablation:
~2000 Å/pulse
Melt depth:
3.25 μm

~6.0 J/cm^2
R_a ~ 0.6 μm
Ablation:
>3000 Å/pulse
Melt depth:
> 4 μm

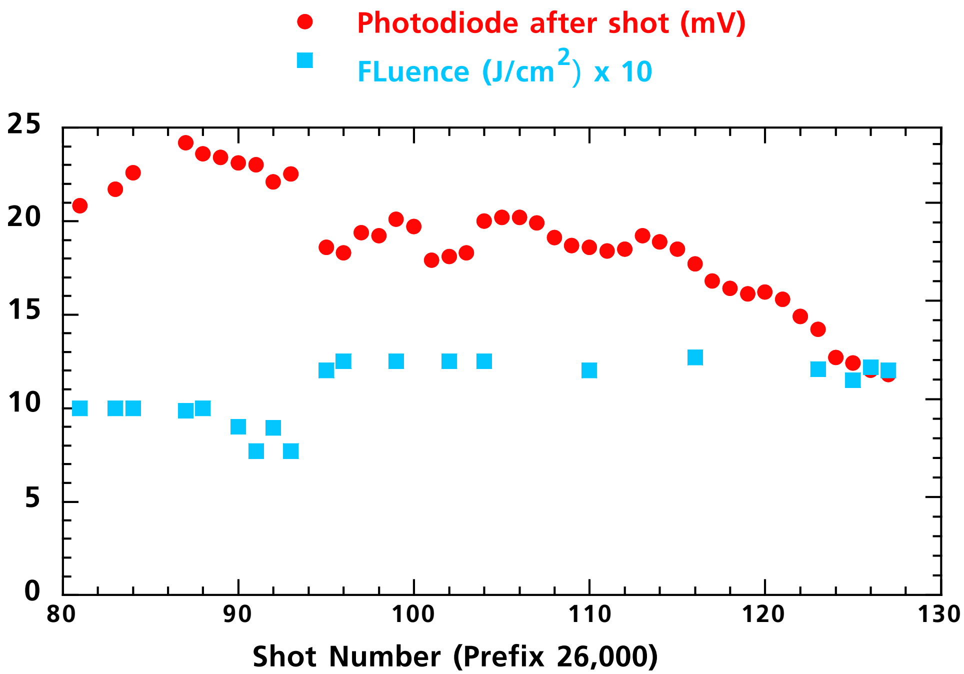
- 600-800 kV, up to 200 A/cm^2 (center-peaked)
- Cratering, roughening worst at intermediate dose

5X microscope images
Grains ~ 1 - 3 mm

1000 shot KS Series: Test heated W, Mo at low level, Materials Tests of W25Re, Re, Cu, Ti-2, Al1100, Foams, CFC

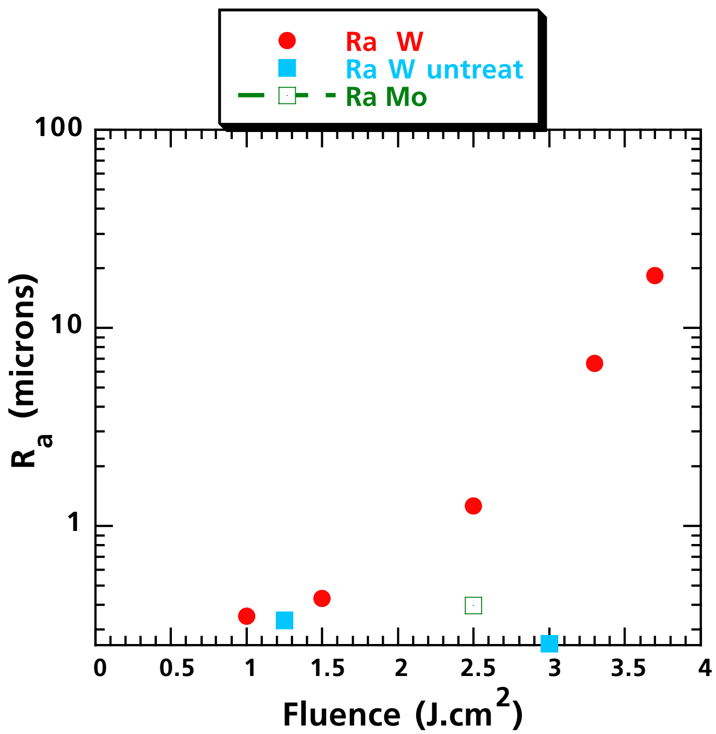
- Photo shows plate with heated W, Mo on right. Goal: melting temp (max 2.5 J/cm^2)
- Other (unheated) samples arrayed on left. CFC and Foams at 1.6 J/ cm^2, rest up to 4 J/ cm^2
- Samples shot 200X, Ra measured by 1-D Dektak, then reloaded for another 200X (not Foams and CFC)
- SEMS, WYCO 2-D profilometry after 1000X (800X for foams and Cu)
- W/Mo + Foams and CFC exposed at higher dose on next 600 shot series
- Foams: W, Re, Nb, Mo

Tungsten roughening (Room Temperature): Detailed History of Reflectometer measurements



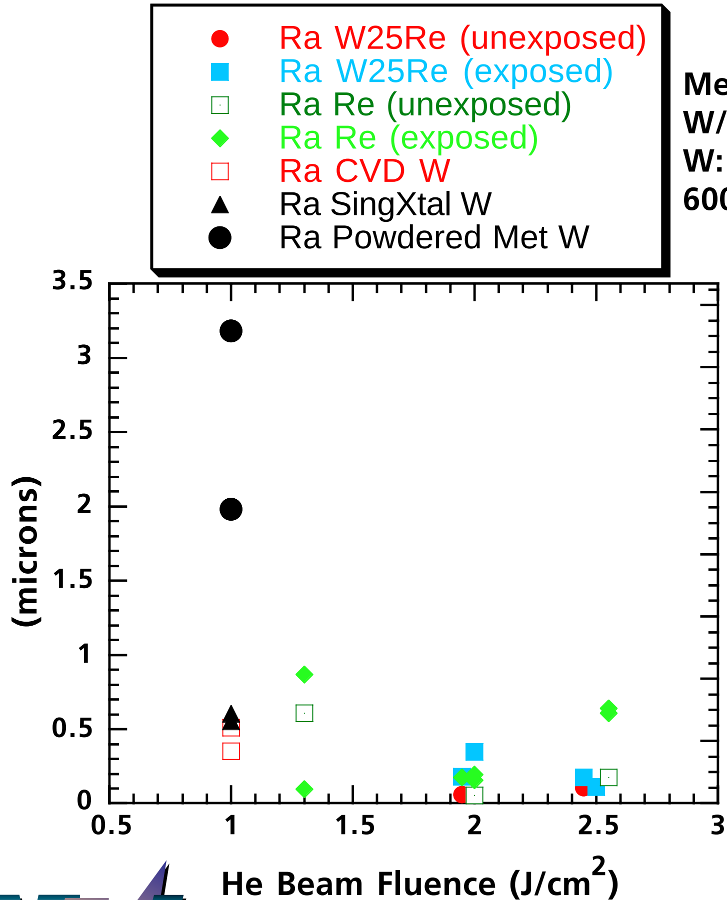
- Polished W exposed to N beam: 0.6 <dose< 1.25 J/cm^2 (53 shots)
- Reflectometer photodiode signal (red) plotted as function of shot number (26081 - 26127)
- Initial exposure at 1 J/cm^2 or less: photodiode remains above 20 mV
- Note progressive signal decrease after shot 26,112. Fluence is ~ 1.25 J/cm^2

400 shot Map N shots on Tungsten (Room Temperature): Roughening only above threshold



- Polished W exposed to 400 shots N beam: 1.0 < fluence < 3.7 J/cm²
- Room Temp (RT) exposure
- Roughening occurs above 1.25 J/cm², consistent with single-shot reflectometer roughening threshold
- Powder Met Mo (one point at 2.5 J/cm²): roughness stays near unexposed value
- Above threshold, roughening is a severe function of fluence. Maximum R_a exceeds 22 μm, with P-V height above 70 μm

He Beam exposure, 450 shots;
Powder Met W has worst roughening, even when heated

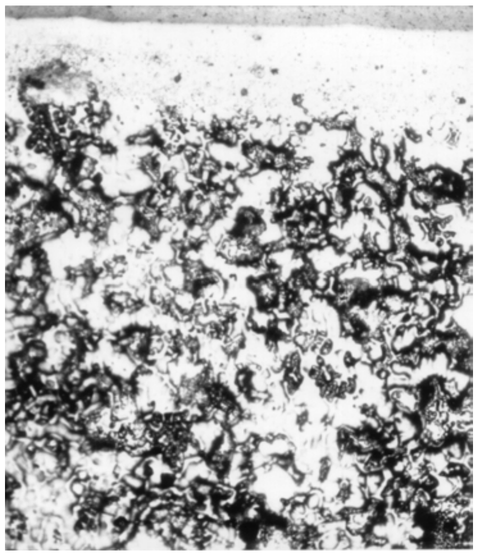


Melting fluence:
W/25Re: 2.7 J/ cm^2
W: 2.3 J/ cm^2 with 600C heating

- Smoothed PowderMetW, CVDW, and single XtalW, provided by Lance Snead, were exposed to 450 MAP He pulses ~ 1.0 to 1.3 J/cm^2. Heated to 600C, heated 3X including heater failure
- W/25Re and Re samples also treated, from 1.3 to 2.5 J/cm^2. Samples at RT
- PwderMetW Roughening is significantly worse than CVD or Single Crystal W. Latter are similar to powderMet W/25Re and Re. None are roughening appreciably compared to untreated.
- Surface morphology for SingXtal and CVD W different from PowderMetW.

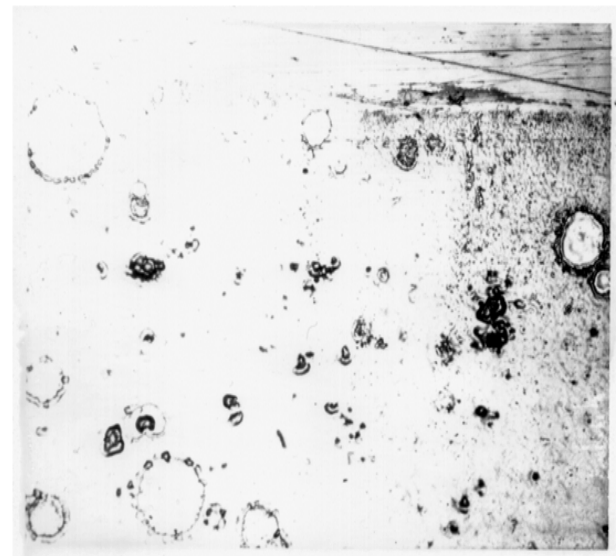
He Beam treatment 450 shots: 600C W roughens w/o melt worse than W/Re with melt

Single Xtal W (Snead)
Fluence: 1 - 1.3 J/ cm^2
Max Temp (Code): 1900K
R_a: 0.5 - 0.6 µm



Powder Met W
Same fluence
R_a : 2 - 3 µm

These surfaces did not melt



W/25Re
Fluence: up to 4 J/ cm^2
R_a : < 0.2 µm
(Circles are melted Cu)

This surface melted

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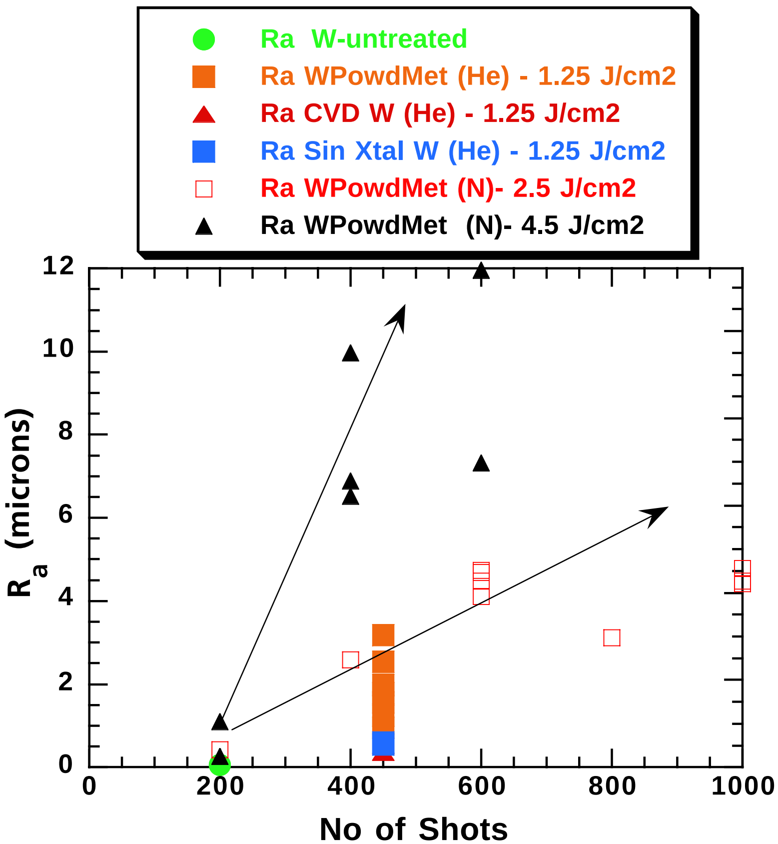
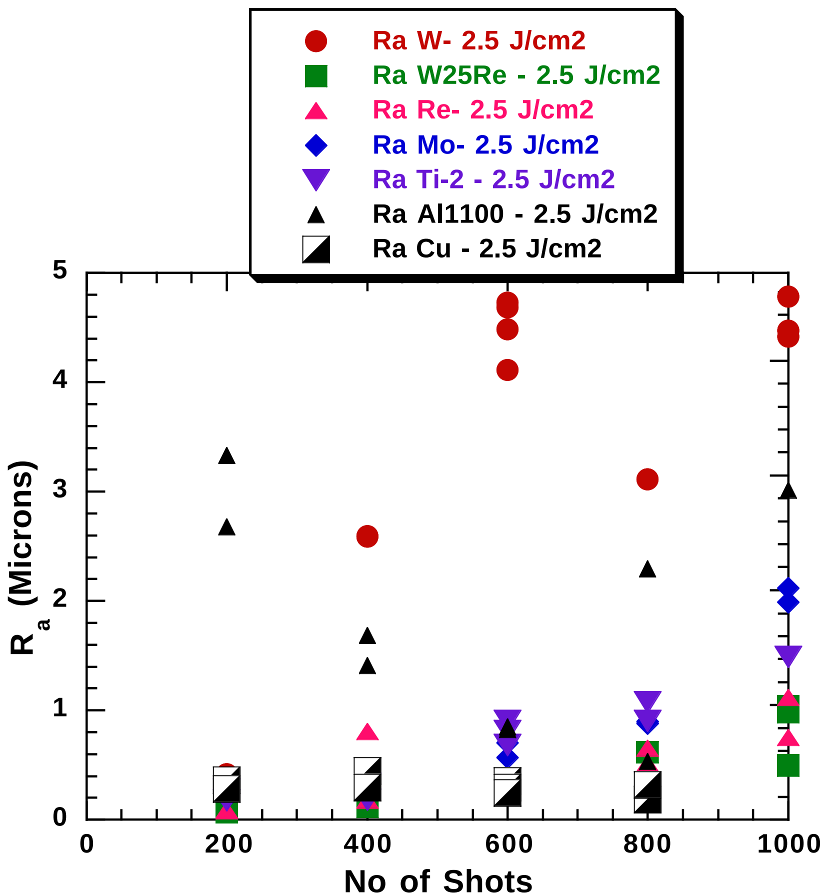
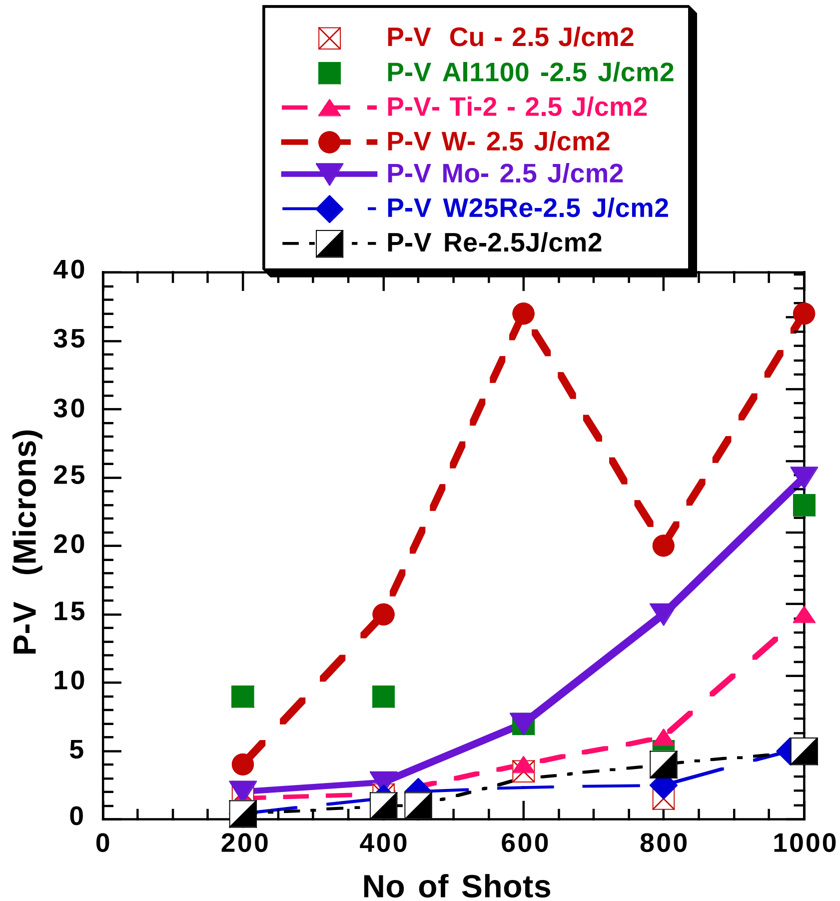
Evolution of R_a Roughness for W: PowderMet, CVD, SingXtal - He and N beams, up to 1000 shots



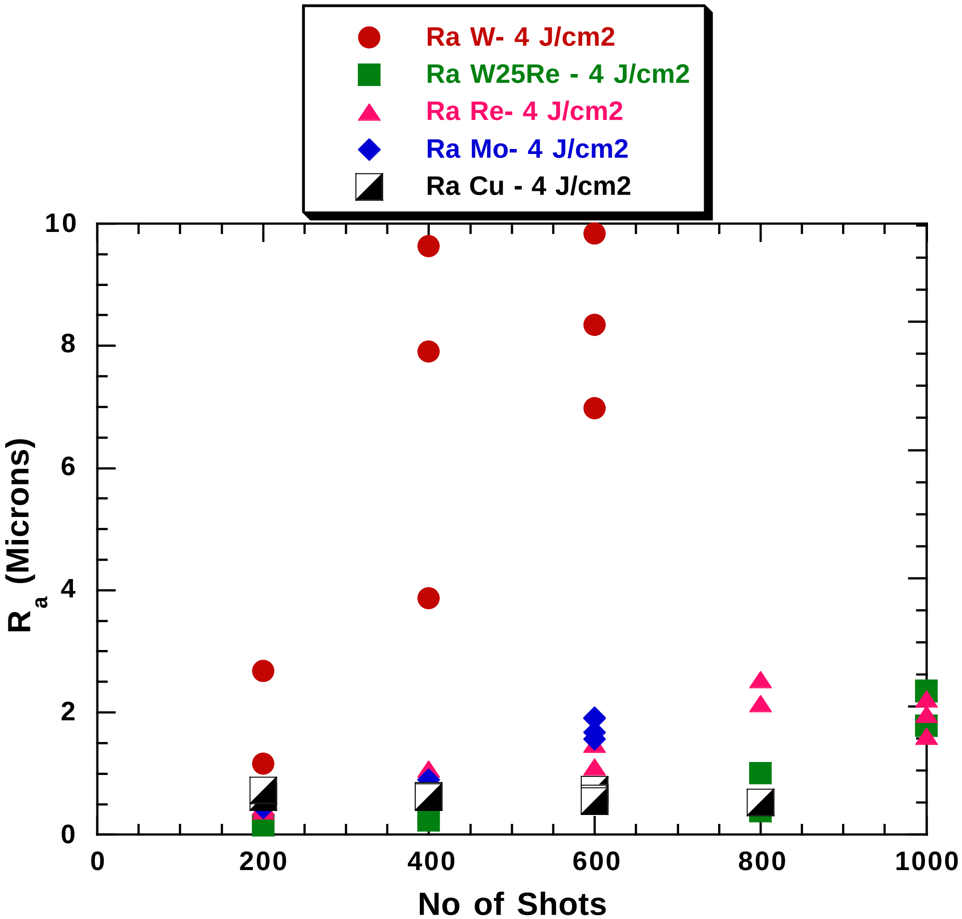
- Polished W samples exposed to 450 shots He, and 600/1000 shots N beam: 1.25 (He) < fluence < 2.5-4.5 J/cm^2 (N)
- All samples exposed at 550-600C
- PowderMet W roughens much worse than CVD or SingXtal (one data pt)
- Roughening with shot number up to 600-100 shots for PowderMet. Slope for 4.5 J/ cm^2 twice that of 2.5 J/cm^2
- Roughening with He beam at 1.25 J/cm^2 comparable to N at 2.5 J/cm^2
- Peak-Valley plot almost identical with R_a plot, except max = 80 μm

Evolution of R_a, Peak-Valley Roughness at 2.5 J/cm^2: WPowderMet is worst, then Al1100, Mo and Ti-2





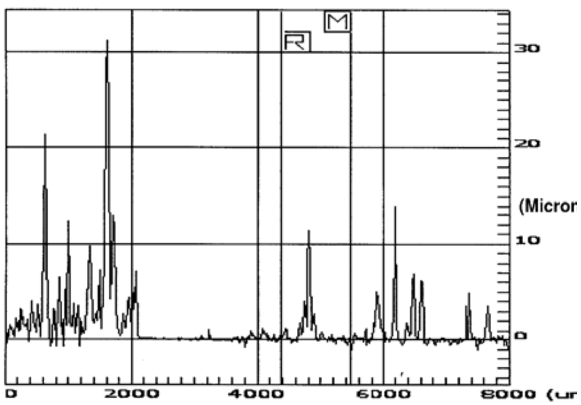
Evolution of R_a Roughness at 4.0 J/cm^2: WPowderMet, then everything else. Cu does NOT roughen



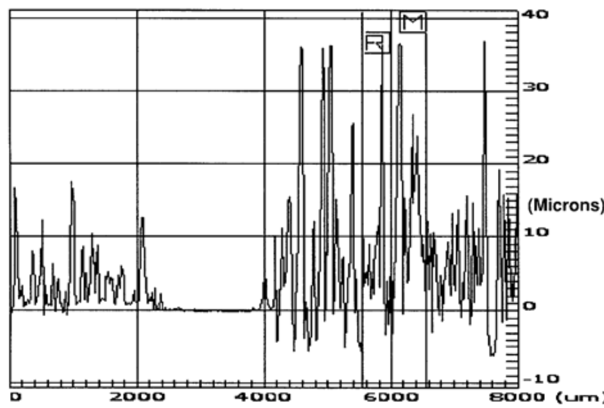
- 4 J/cm^2 is near or above ablation level for most metals shown
- Polished W and Mo are heated to 550 - 600C
- W roughens beyond 10 µm R_a at 400-600 shots (only 600 taken)
- W25Re, Re reach 2 µm R_a at 1000 shots, but Cu remains below 1 µm
- Ti-2 (not shown) roughens steadily to 1000 shots
- W Peak-Valley exceeds 70 µm at 600 shots

W (Powder Met) roughening: R_a as function of Number of Nitrogen pulses @ 4.0 J/cm^2

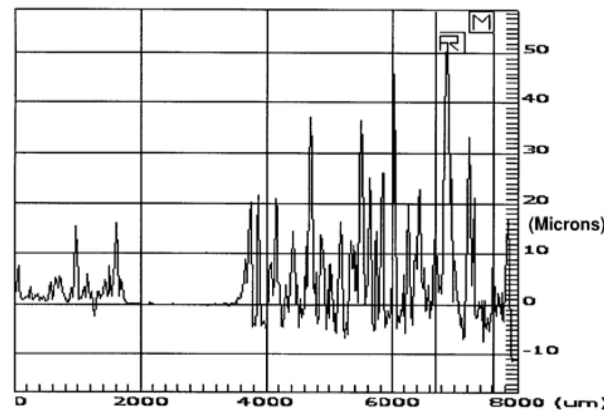
I-D 8 mm Profilometer Scans, 450x He (Left) and Nitrogen (Right)



(He) (None) (N 200X)



(He) (None) (N 400X)



(He) (None) (N 600X)

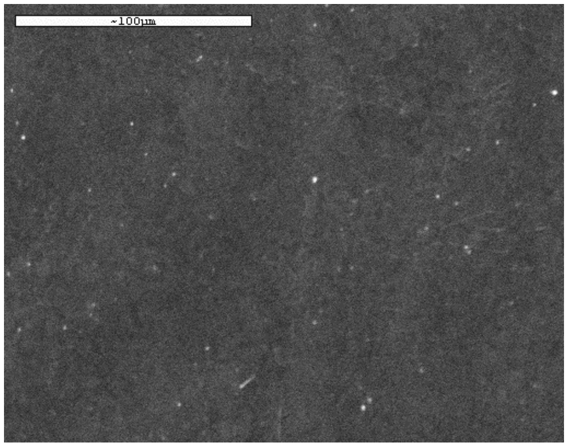
Ra ~ 1-3 μm
P-V ~ 10-30 μm

Ra ~ 1-3 μm
P-V ~ 5-15 μm

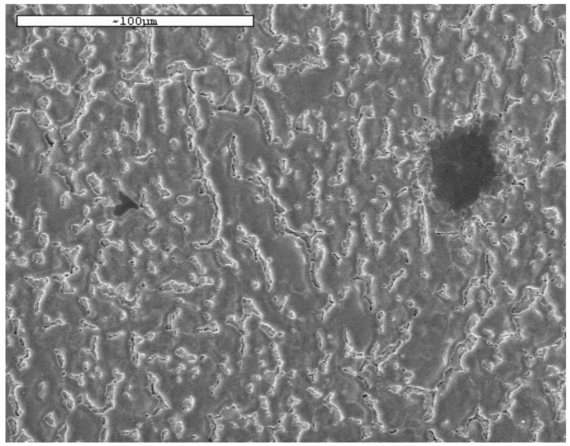
Ra ~ 4-9 μm
P-V ~ 20-35 μm

Ra ~ 7-10 μm
P-V ~ 50-70 μm

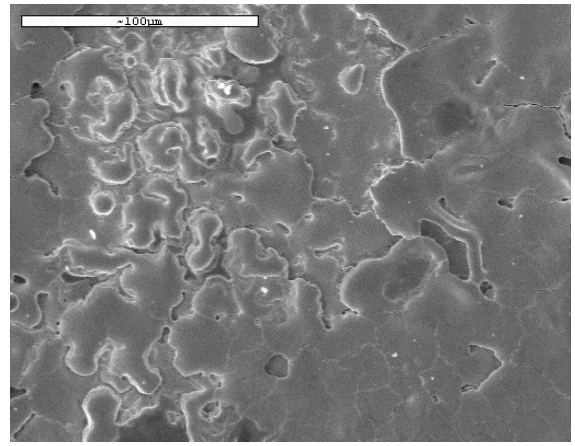
W (Powder Met) roughening: SEM images as function of shot number: 60 to 1000X



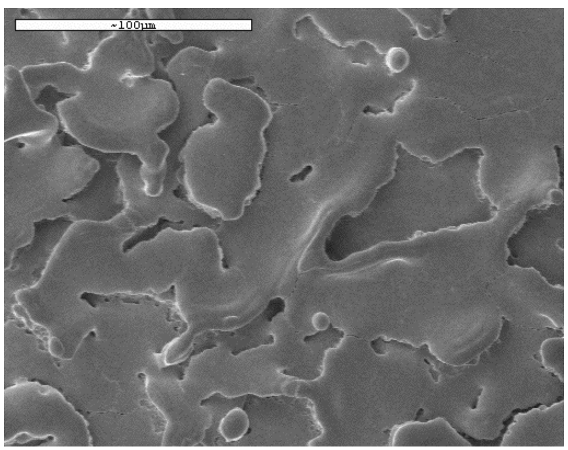
(N 60X @ 6 J/cm^2)



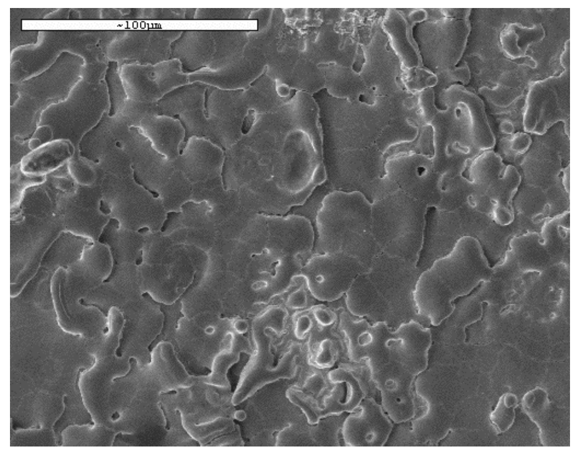
(N 250X @ 2.5 J/cm^2)



He 450X @ 1-1.3 J/cm^2)

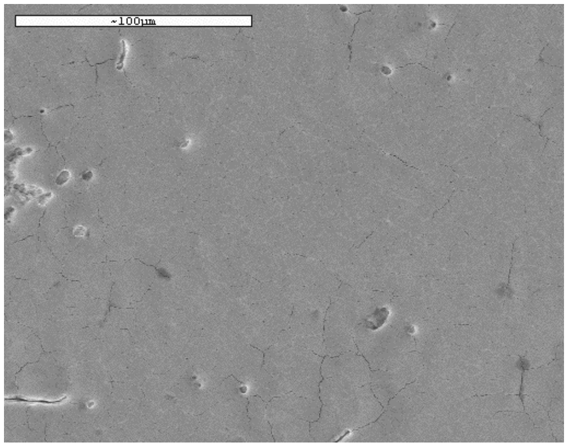


(N 600X @ 4.0 J/cm^2)

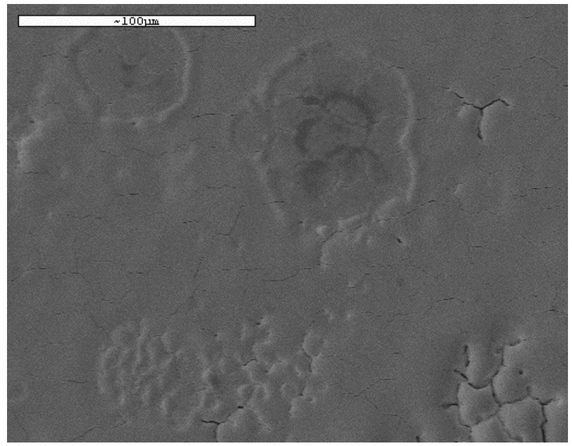


(N 1000X @ 2.5 J/cm^2)

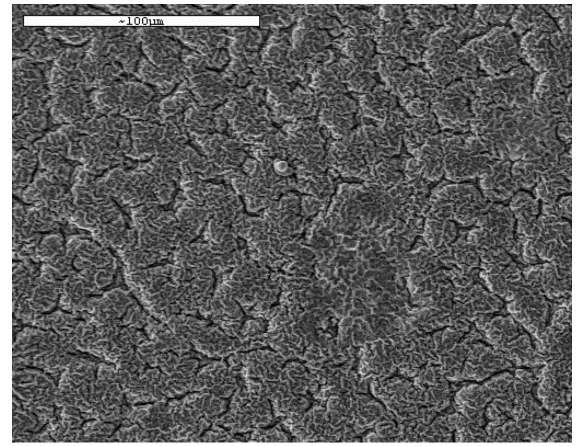
SEM images of different metals @ 500X Mag: 450XHe to 1000X N



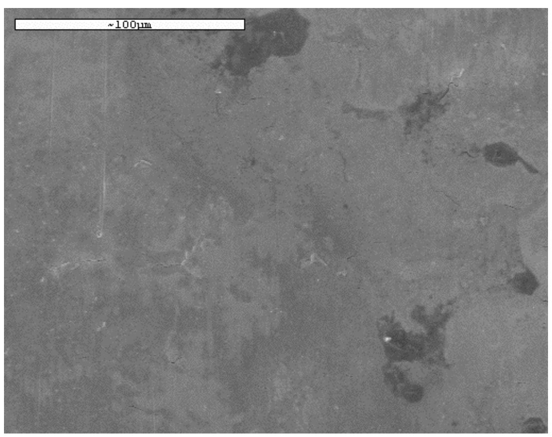
(W25Re 450X He @ 2.5 J/cm^2)



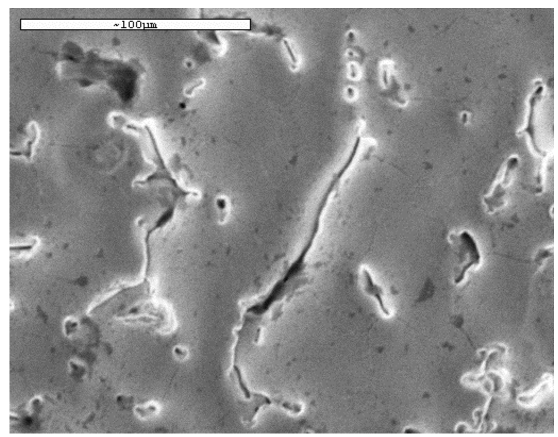
(W CVD 450X He @ 1-1.3 J/cm^2)



Ti-2 1000X @ 2.6 J/cm^2)

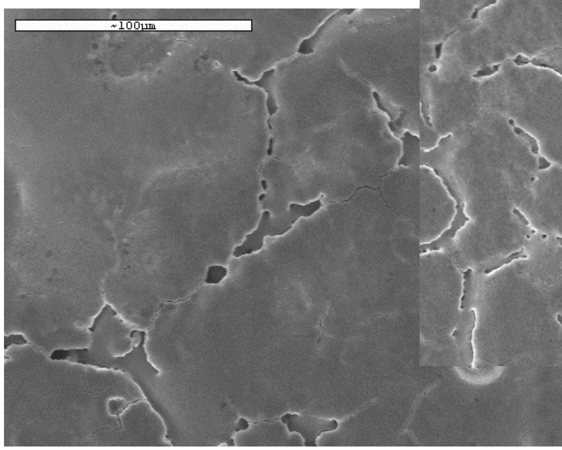


(Cu 1000X @ 2.6 J/cm^2)

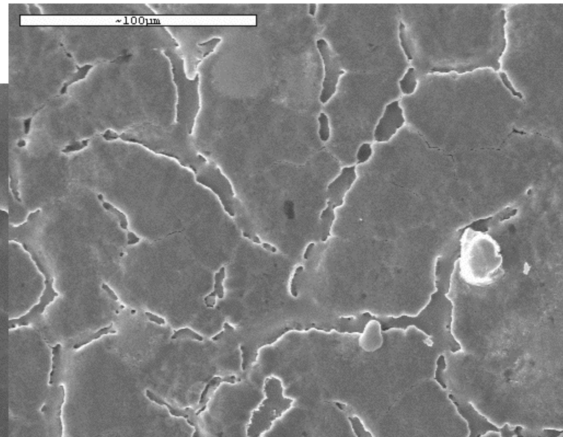


(Re 1000X @ 2.6 J/cm^2)

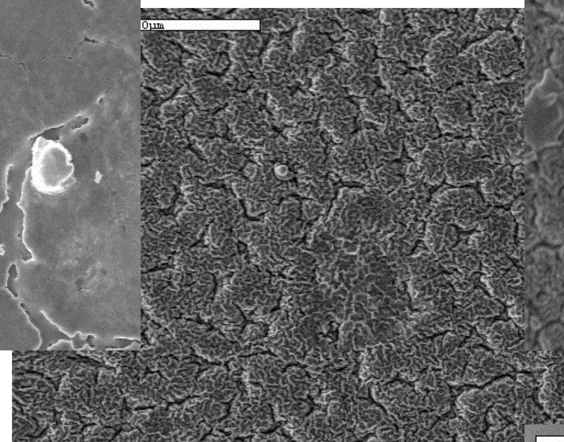
SEM images of Ti-2 and Re:
Lateral scale length with fluence increase



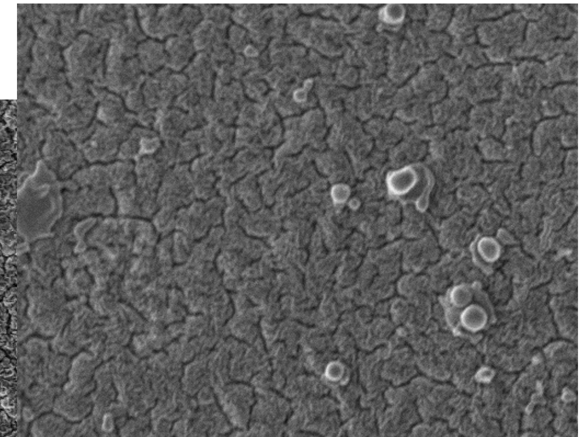
(Ti-2 @ ~ 1.0 J/cm^2)



Ti-2 @ 1.6 J/cm^2)

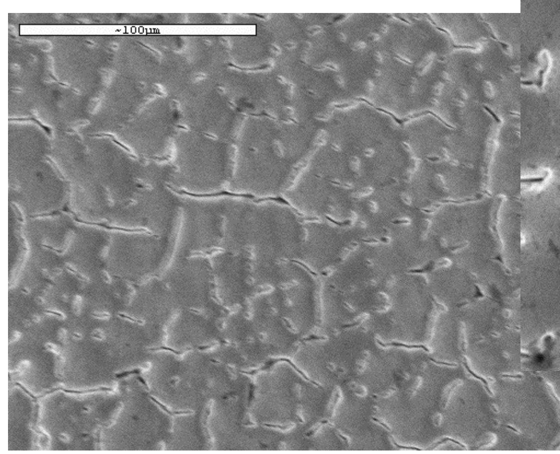


Ti-2 @ 2.6 J/cm^2)

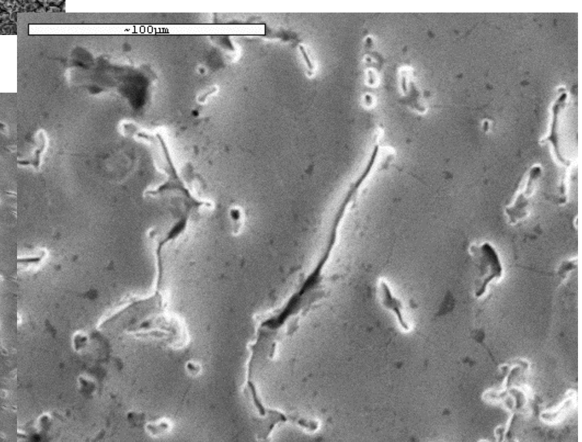


Ti-2 @ 4.0 J/cm^2)

Length scale *decreases* for Ti-2, *increases* for Re

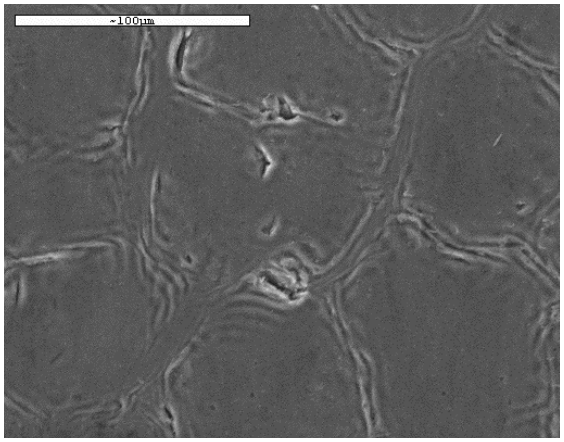


(Re 1000X @ 2.6 J/cm^2)

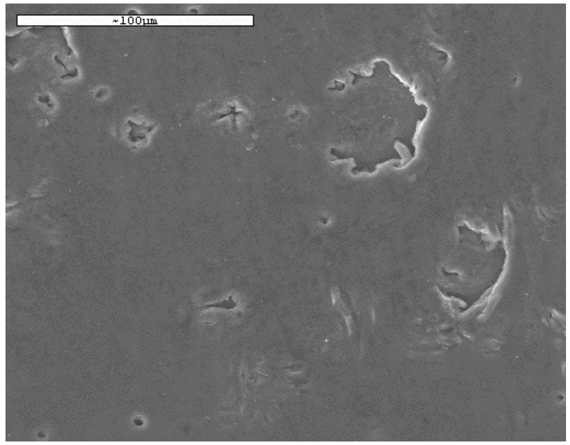


(Re 1000X @ 4.0 J/cm^2)

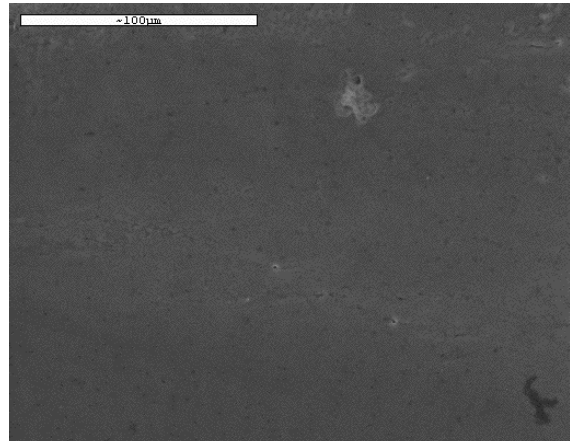
(Upper) SEM : Al1100, 1000x Map N @ 500X Mag
Lower: Cu 1000x Map N @ 2.5 J/cm^2



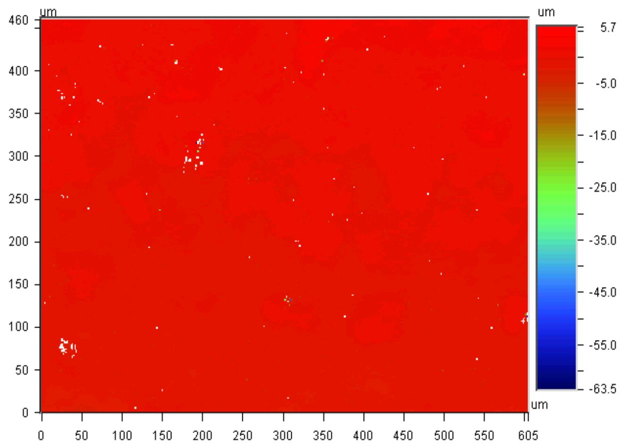
Al1100 1000xN @ 0.85 J/cm^2



Al1100 1000xN @ 1.6 J/cm^2

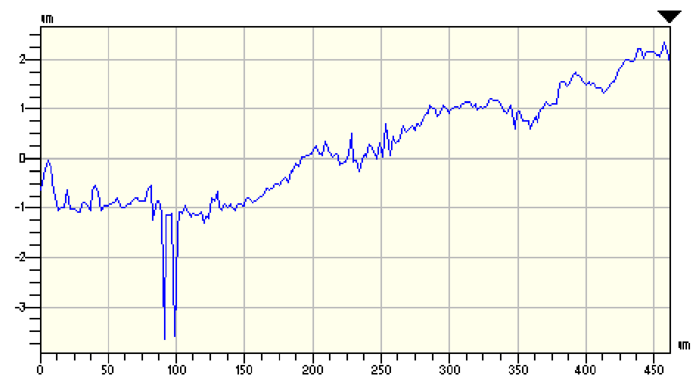


Al1100 1000xN @ 2.5 J/cm^2



(Cu 1000X @ 2.6 J/cm^2)

Y Profile



(Re 1000X @ 2.6 J/cm^2)

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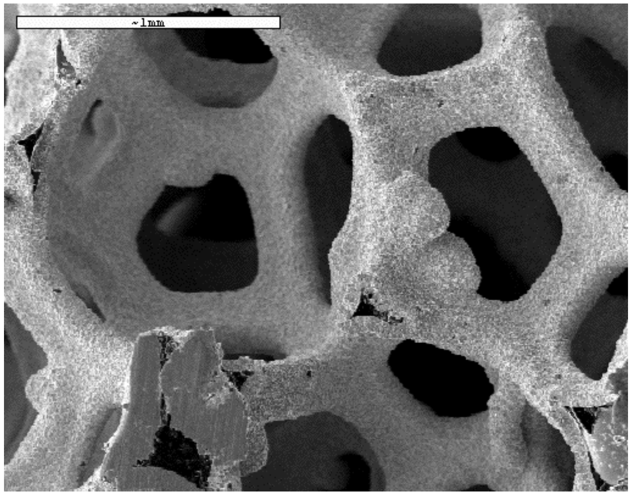
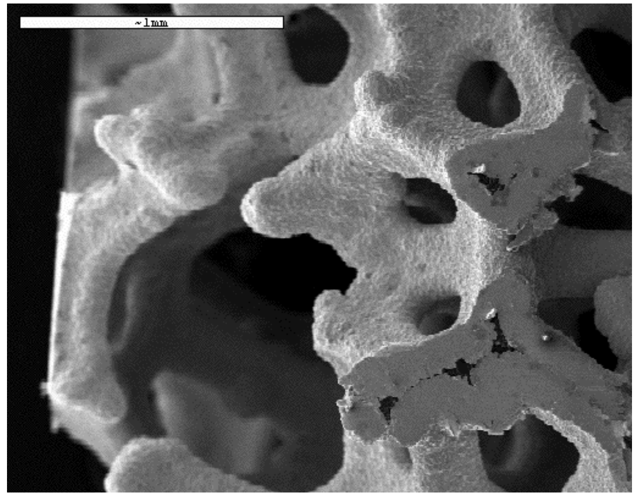
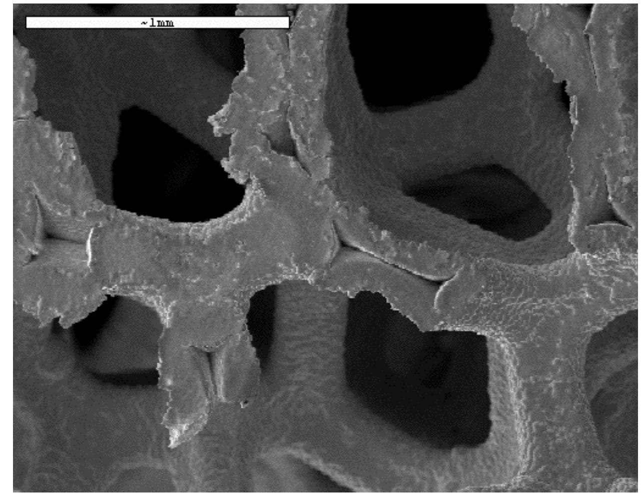
W and Mo Foam SEM images as function of fluence



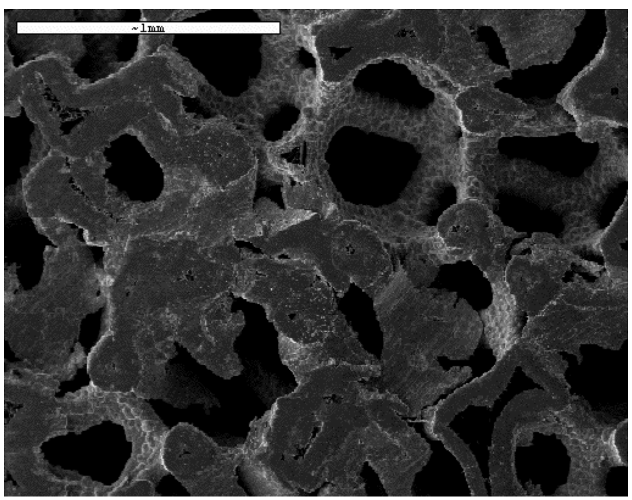
W Untreated



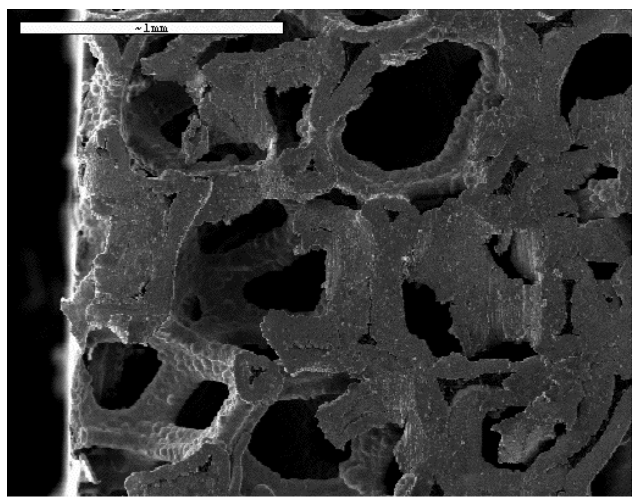
W Treated at 1.6 J/cm^2 600 shots



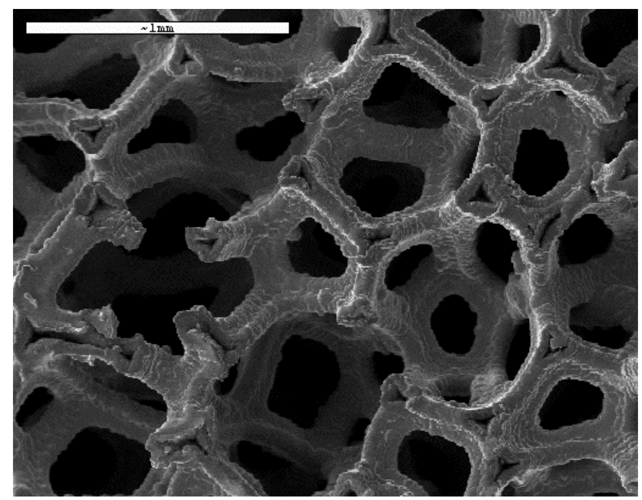
W Treated at 3.0 J/cm^2 600shots



Mo Untreated



Mo Treated at 1.6 J/cm^2 600 shots



Mo Treated at 3.0 J/cm^2 600 shots

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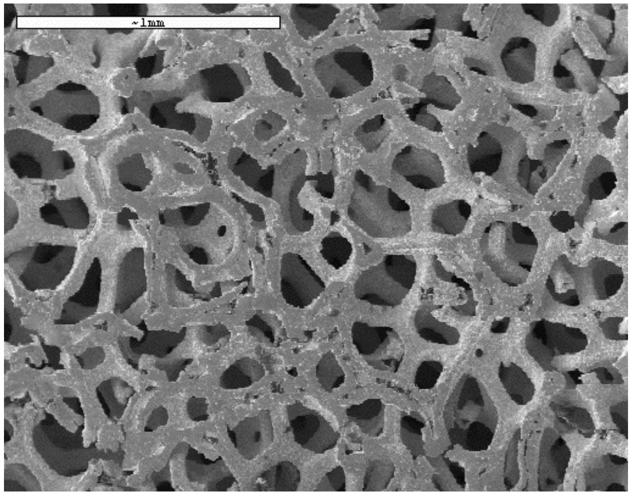
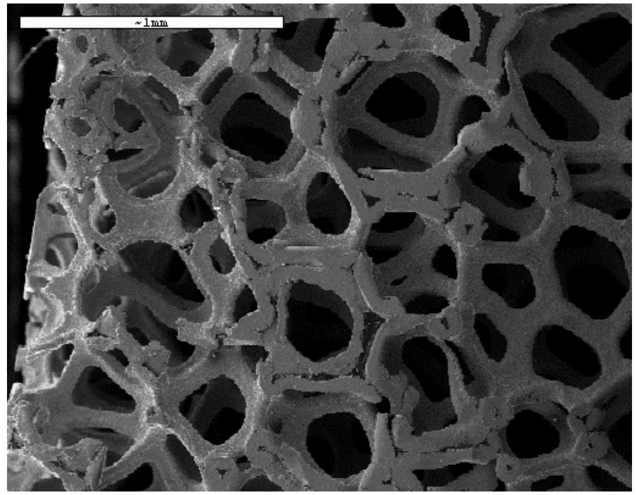
All images 50X magnification

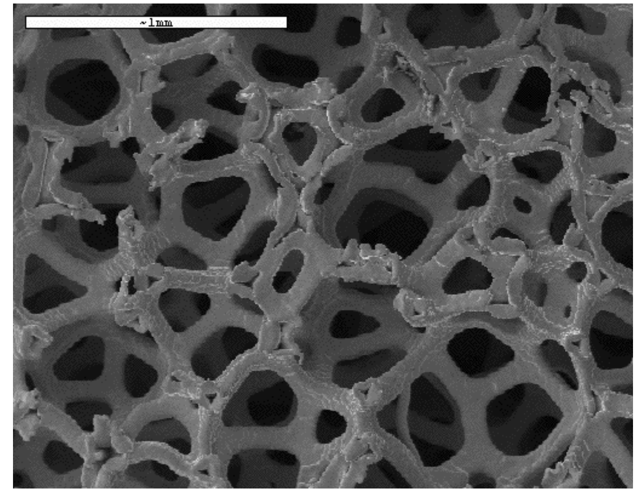
Re and Nb Foam SEM images as function of fluence



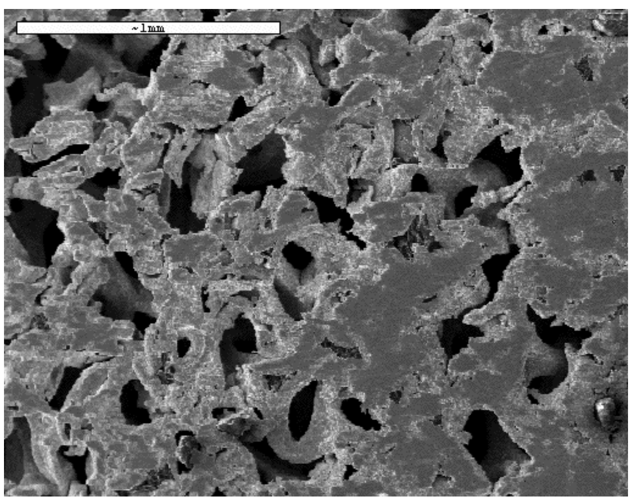
Re Untreated



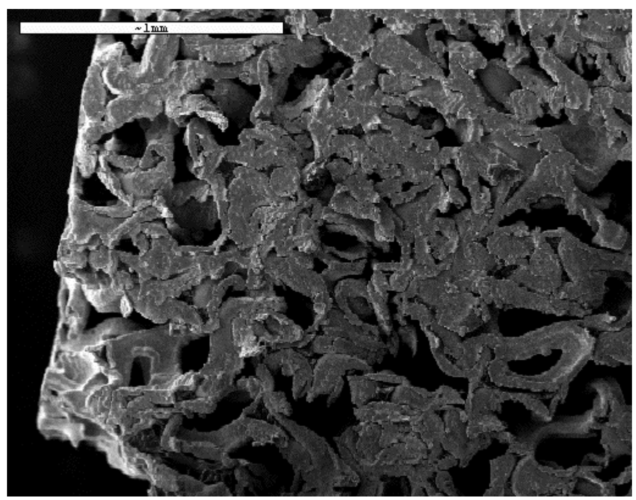
Re Treated at 1.6 J/cm^2 600 shots



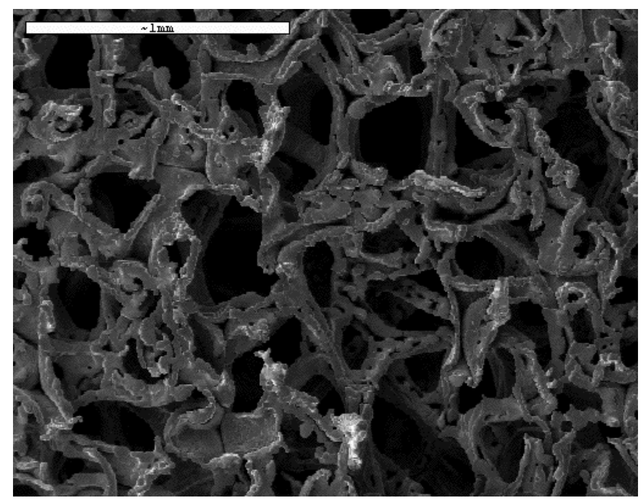
W Treated at 3.0 J/cm^2 600 shots



Nb Untreated



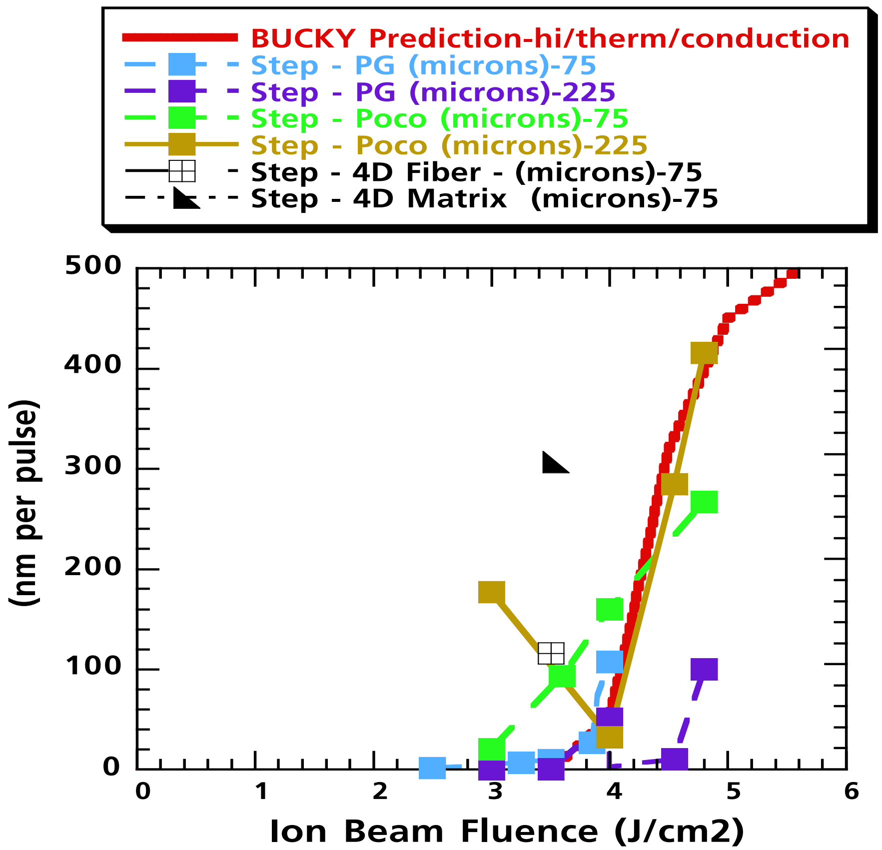
Nb Treated at 1.6 J/cm^2 600 shots



Nb Treated at 3.0 J/cm^2 600 shots

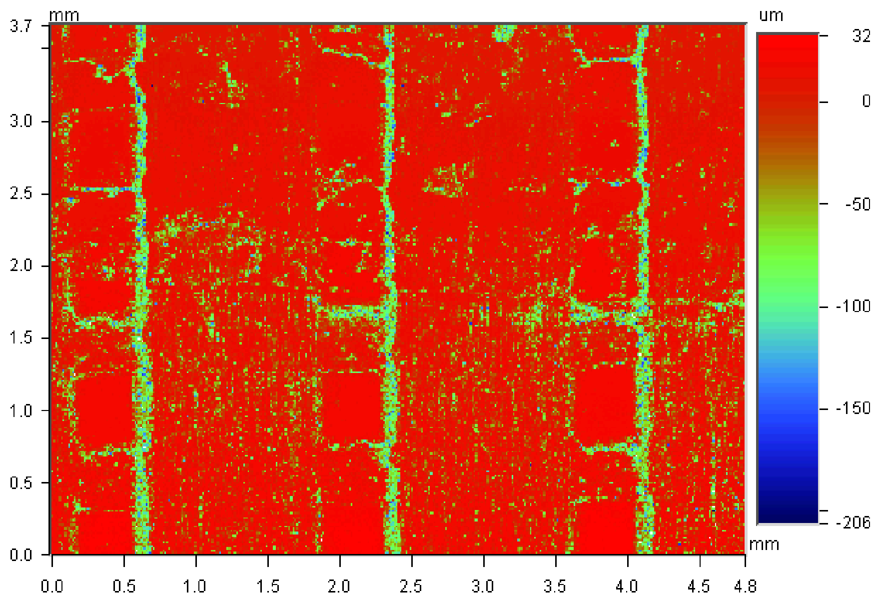
All images 50x magnification

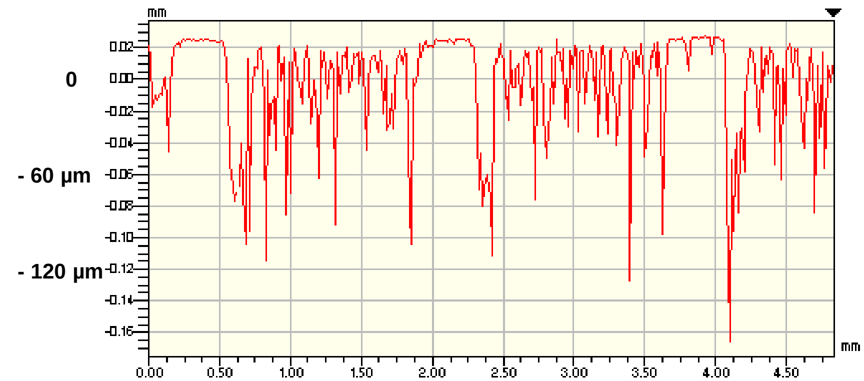
Response of graphite to mixed H - C beam qualitatively confirms BUCKY predictions



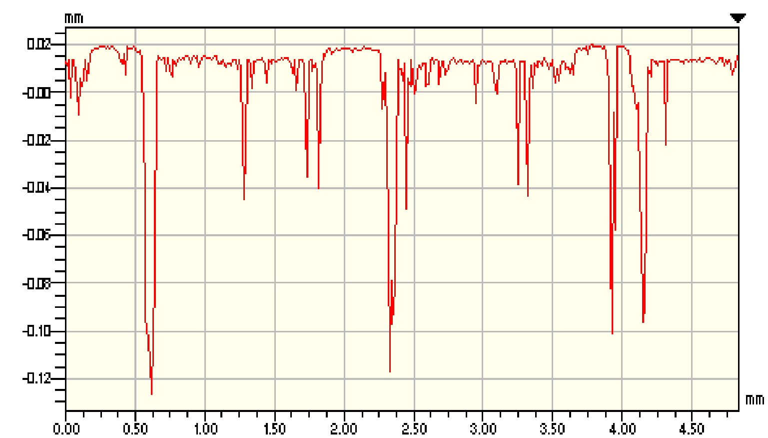
- Mechanically polished pyrolitic graphite (PG), Poco, and 4D carbon composite weave exposed to 75 pulses/225 pulses of 70% C /30% H beam at doses of 1.9 to 5 J/cm^2
- PG ablation threshold ~ 4 J/cm2
- Poco ablation threshold ~ 3 J/cm2
- Above threshold, rapid increase in ablated material per pulse with 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 1000X at 1.6 J/cm²: Significant erosion of matrix





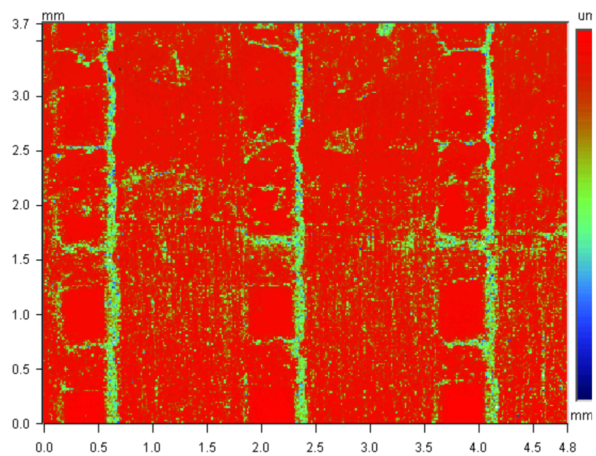
Treated at 1.6 J/cm²

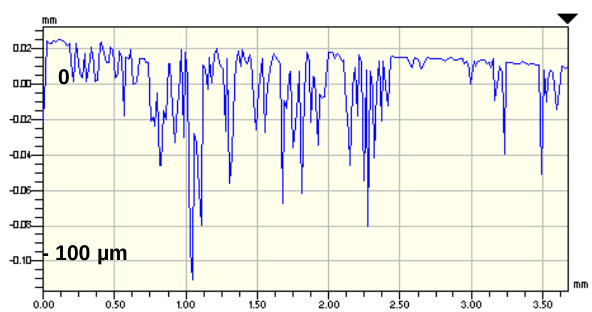


Untreated

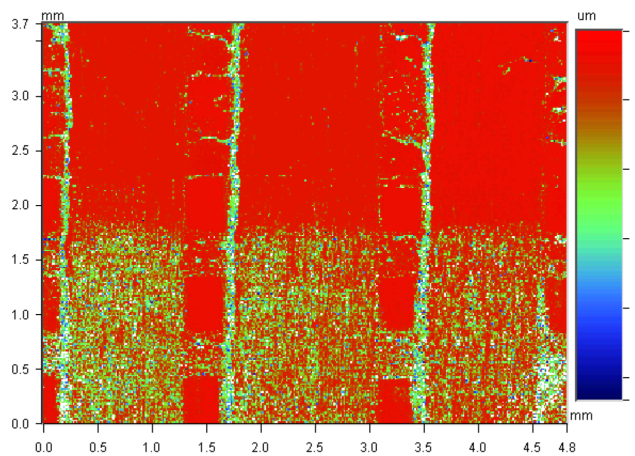
FMI-222 Fiber ends appear ablation-resistant; Matrix loss ~ 0.3 µm/pulse at 4.0 J/cm^2:

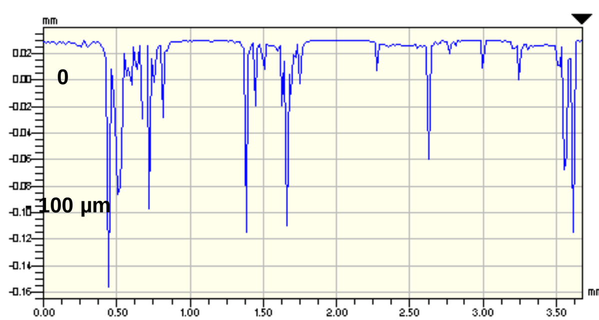
Treated at 1.6 J/cm^2 1000X



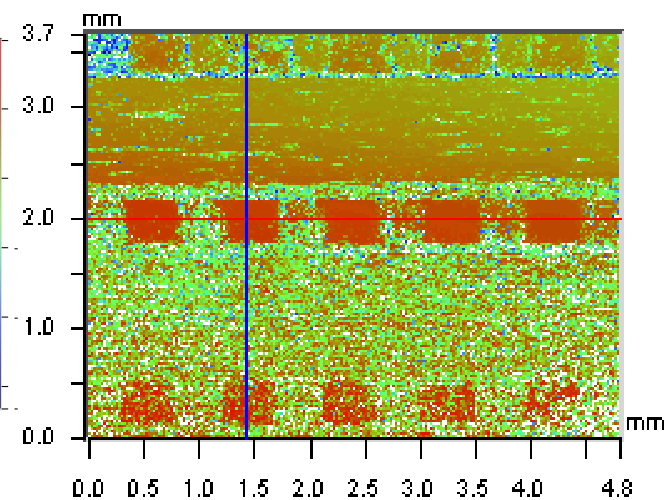


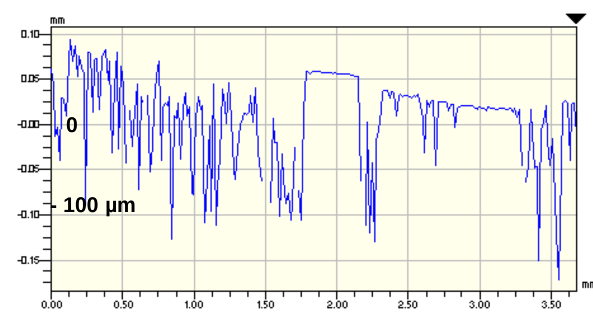
Treated at 2.6 J/cm^2 600X



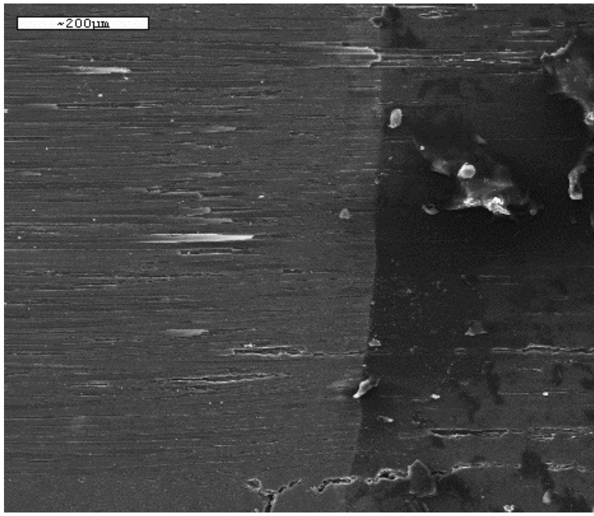


Treated at 4.0 J/cm^2 600X

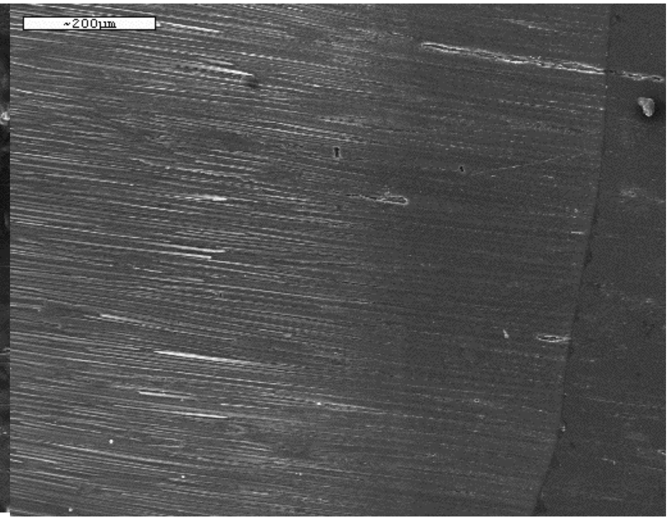




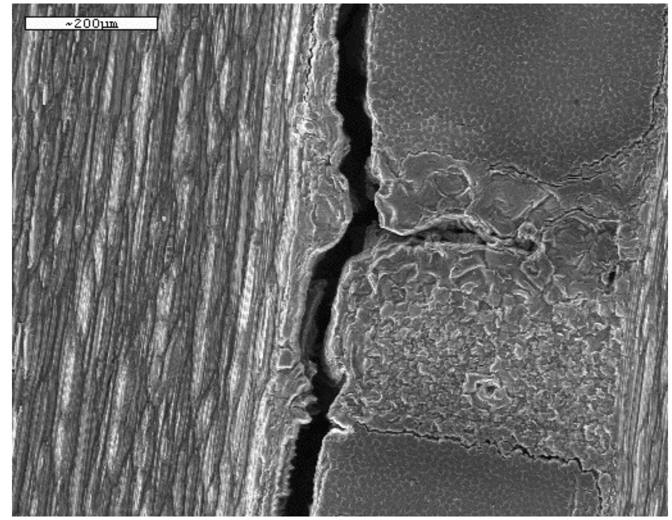
FMI-222 CFC SEM images as function of fluence



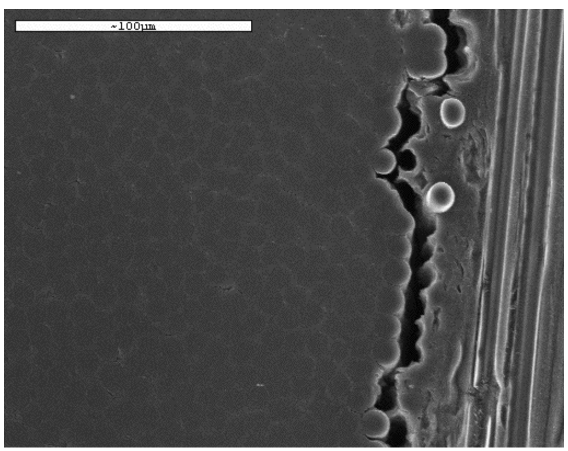
Treated at 1.6 J/cm^2 1000X 120MAG



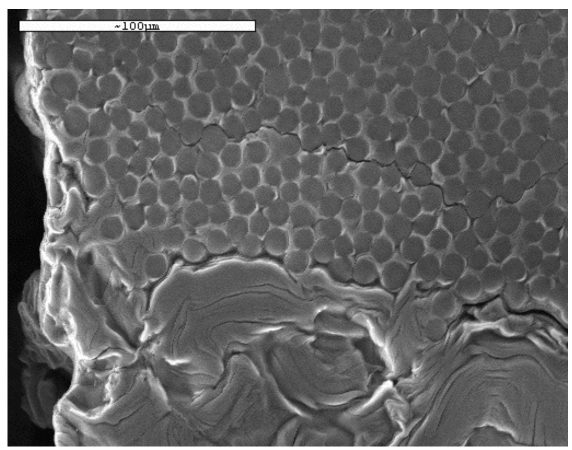
Treated at 2.6 J/cm^2 600X 120MAG



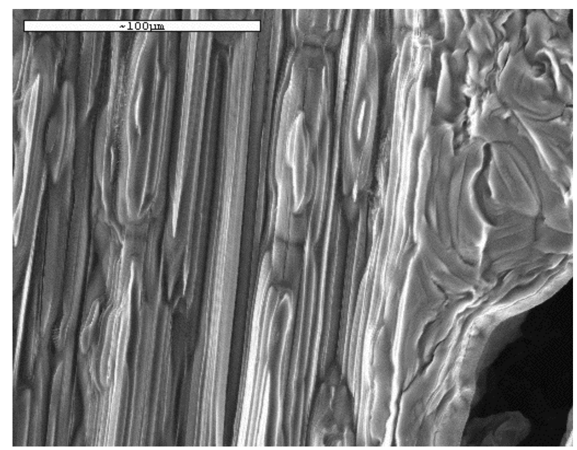
Treated at 4.0 J/cm^2 600X 120MAG



Untreated fiber end



Fiber end 4.0 J/cm^2 600X



Matrix 4.0 J/cm^2 600X

Bottom images all 500MAG

ESLI CCV/W Test Specimen

ESLI Carbon-Carbon Velvet (CCV) with sputtered-W Coating

- Carbon fiber volume packing 1.9%
- Sputtered-W film thickness 1.6 µm over area
  - Thick (1.6 µm) caps on fiber tips; thin (<0.1 µm) film on fiber shafts (thickest at top)

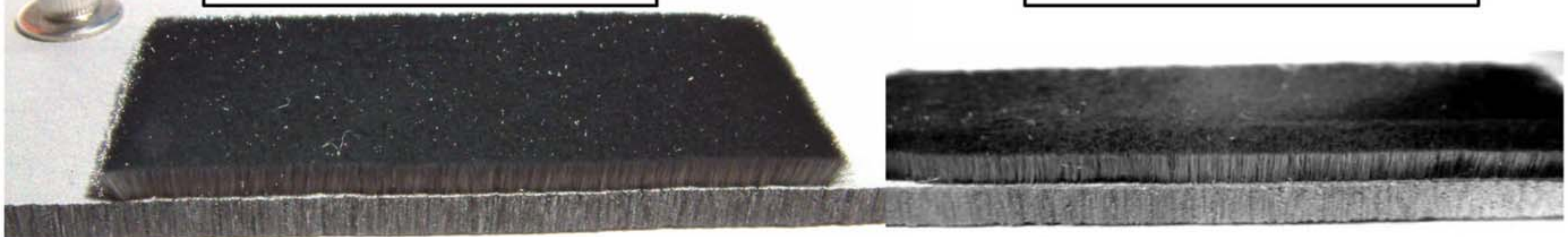
Design for RHEEP incidence angle (0.3-0.5 radian)

- Fluence distribution

On flat fiber tops	100% of incident fluence
On fiber shafts	< 25% of incident fluence (max near top)
On graphite substrate	<5% of incident fluence

Before RHEPP Exposure

After RHEPP Exposure



50 x 25 mm^2 fiber area
1/8” Graphite Substrate

50 x 6 mm^2 fiber area exposed
(remainder masked by Ta foil)

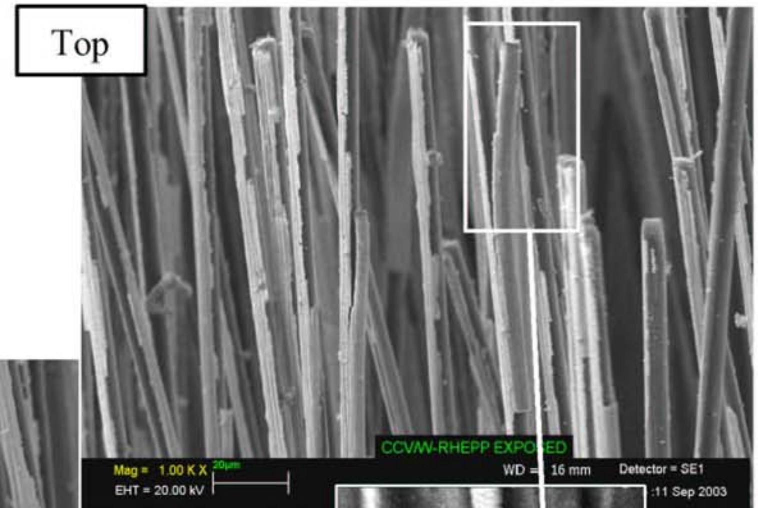
RHEPP Exposure

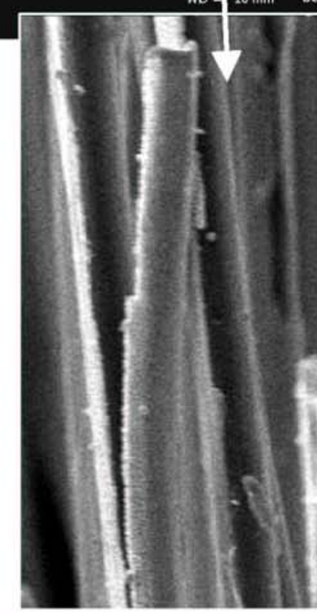
- Specimen was exposed to 200 shots on RHEPP
  - Exposed at SNL, August 2003
- A Ta mask covered a portion of the specimen
  - The exposed portion appears blacker, cleaner than the masked portion
  - Horizontal fibers are eroded entirely
- Fluence gradient along 50-mm strip of specimen
  - High fluence end ~ 6-7 J/cm^2
    - Expect to ablate solid W at this fluence
  - Low fluence end ~ 1 J/cm^2
    - Expect 1500 K max T on solid W at this fluence

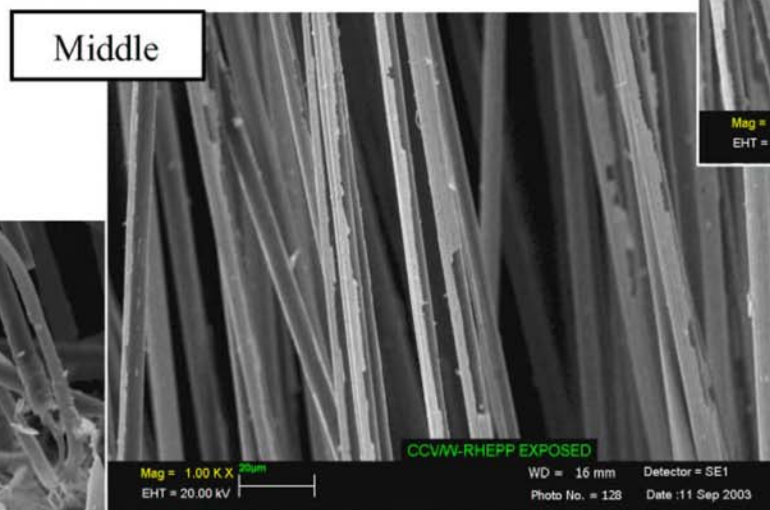
CCV/W at Low Exposure (Side)

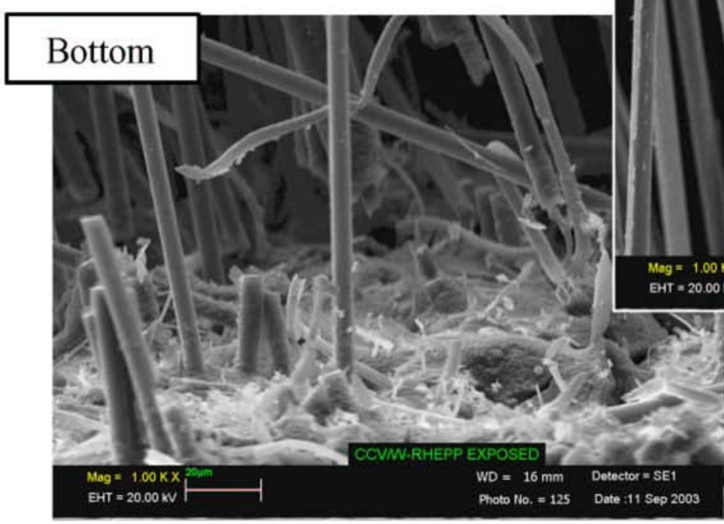
Observe

- Tips have lost W caps
- Peeling of W films from fibers
- W coating smooth





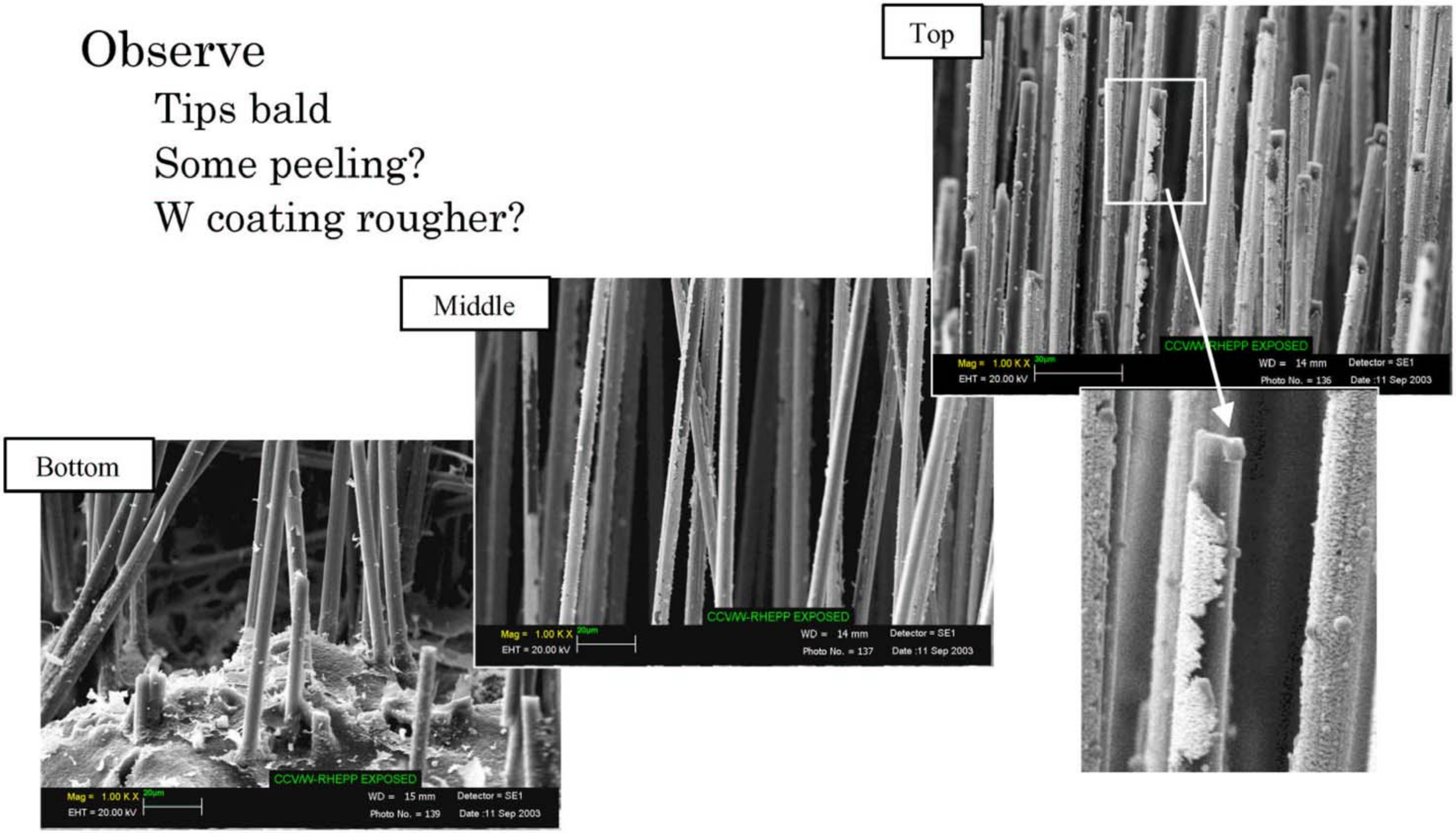




CCV/W at Medium Exposure

Observe

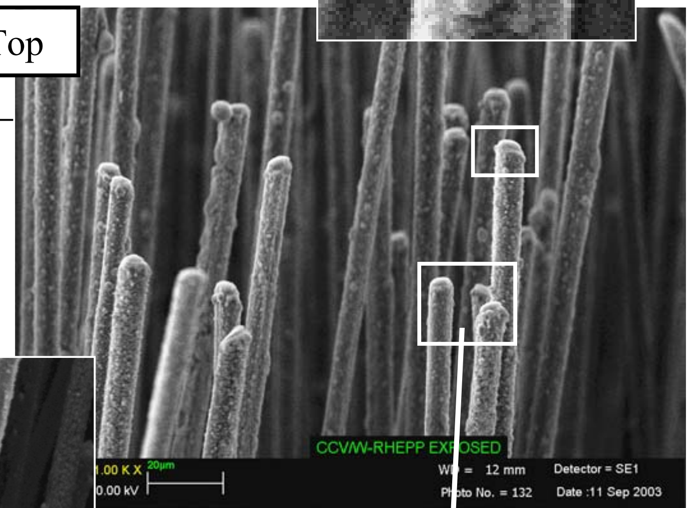
- Tips bald
- Some peeling?
- W coating rougher?

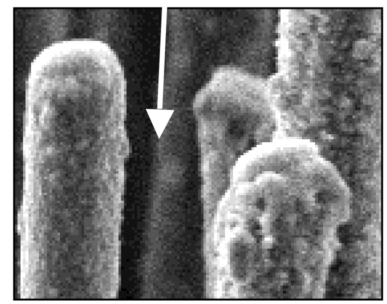


CCV/W at High Exposure

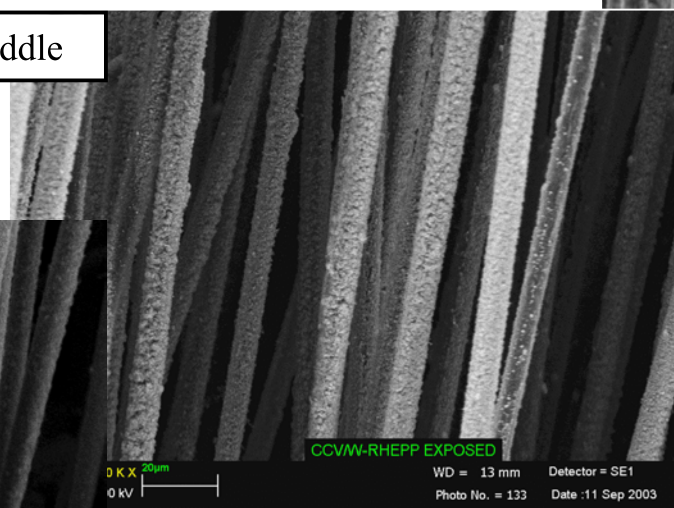
- Observe
  - Tips eroded, but still coated?
  - No peeling of W film; substrate clean
  - Surfaces roughened

Top

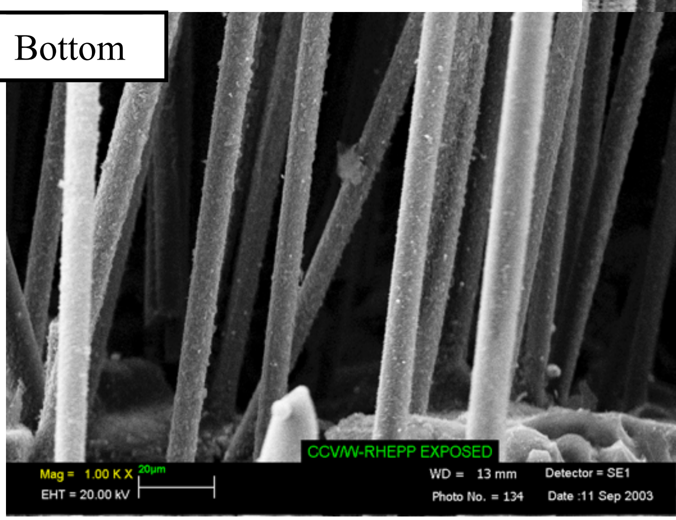




Middle



Bottom



Summary

- Tungsten: surface roughening excessive (PowderMet), with fatigue cracking below surface maybe even worse. Mitigating: CVD/SingXtal, alloying with Re, heating. Cracking looks like enough to compromise performance as armor coating.
- Roughening occurs over hundreds of shots. Topology may stabilize for W by 1000 shots, looks to increase for other metals beyond this.
- Below the roughening threshold (1.25 J/cm^2), W may be topologically stable to repeated ion exposure.
- If there is a material loss threshold for CFC Graphite (222-FMI), it is below the 1.6 J/cm^2 level.
- Performance of foams needs further study.
- Most promising performers? C-C-W Velvet, foam possibly. Most 'unroughened': Cu

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Conclusions

- CCV/W can be fabricated with controlled packing density, fiber orientation, W film coating suitable for RHEPP exposure
- <u>Low fluence response shows damage to coating</u>
  - W films peel off fibers, accumulate at base
- <u>High fluence response shows much less damage</u>
  - No apparent damage to substrate – velvet protects substrate from RHEPP exposure
  - No W debris accumulating at substrate
  - Fiber shafts are slightly roughened (<1 μm)
  - Fiber tips show some erosion
- High fluence heating appears to join W to C