Response of W/W alloys and graphite to X-rays (Z) and ions (RHEPP)

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Workshop Agenda

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.

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Outline

- IFE Dry Wall Threat Spectrum - ions and Xrays

- Xrays on Z - description of experiments and results

- Ions on RHEPP-1
- A. Description of experiment
- B. Beam description, roughening behavior
- C. Graphite
- D. W and W alloys

- Engineered surface - graphite ‘velvet’

- Summary

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Examples of R(microns) and F(J/cm^2) for 1 keV x-rays

Property	Carbon	Tungsten
melt (°K)	~ 3500	~3370
specific heat C (cal/g °K)	~0.45	~0.04
latent heat Q (cal/g)	----	45.8
density ρ (g/cm^3)	1.8	19.3
mass abl. coef. μ(cm^2/g)	2,000	3,500
deposition $R = (\mu\rho)^{-1}$	2.8 microns	0.15 microns
threshold $F^{k=0}$	3.3 J/cm^2	0.215 J/cm^2
thermal conductivity k (J/[cm s °K])	~ 0.3	~ 1.05
flux out / flux in $k \Delta T t_0 / (R F^{K=0})$	0.01	10.1
enhancement factor f	1.01	11.1
effective range fR	2.8 microns	1.7 microns
threshold $F^{k \neq 0} = fF^{k=0}$	3.3 J/cm^2	2.4 J/cm^2

x-ray threat spectra for laser dry wall is 0.3 - 1.0 J/cm^2

x-ray problem is near or below threshold

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Examples of R(microns) and F(J/cm^2) for 750 keV C ions

Property	Carbon	Tungsten
melt (°K)	~ 3500	~3370
specific heat C (cal/g °K)	~0.45	~0.04
latent heat Q (cal/g)	----	45.8
density ρ (g/cm^3)	1.8	19.3
range R(g/cm^2)	0.00018	0.00087
deposition R	1.0 microns	0.45 microns
threshold $F^{k=0}$	1.2 J/cm^2	0.65 J/cm^2
thermal conductivity k (J/[cm s °K])	~ 0.3	~ 1.05
flux out / flux in $k \Delta T\, t_0 / (R\, F^{K=0})$	0.5	6.9
enhancement factor f	1.5	7.9
effective range fR	1.5 microns	3.6 microns
threshold $F^{k \neq 0} = fF^{k=0}$	1.8 J/cm^2	5.1 J/cm^2

ion threat spectra for laser dry wall is 8 - 20 J/cm^2

ion problem is more serious

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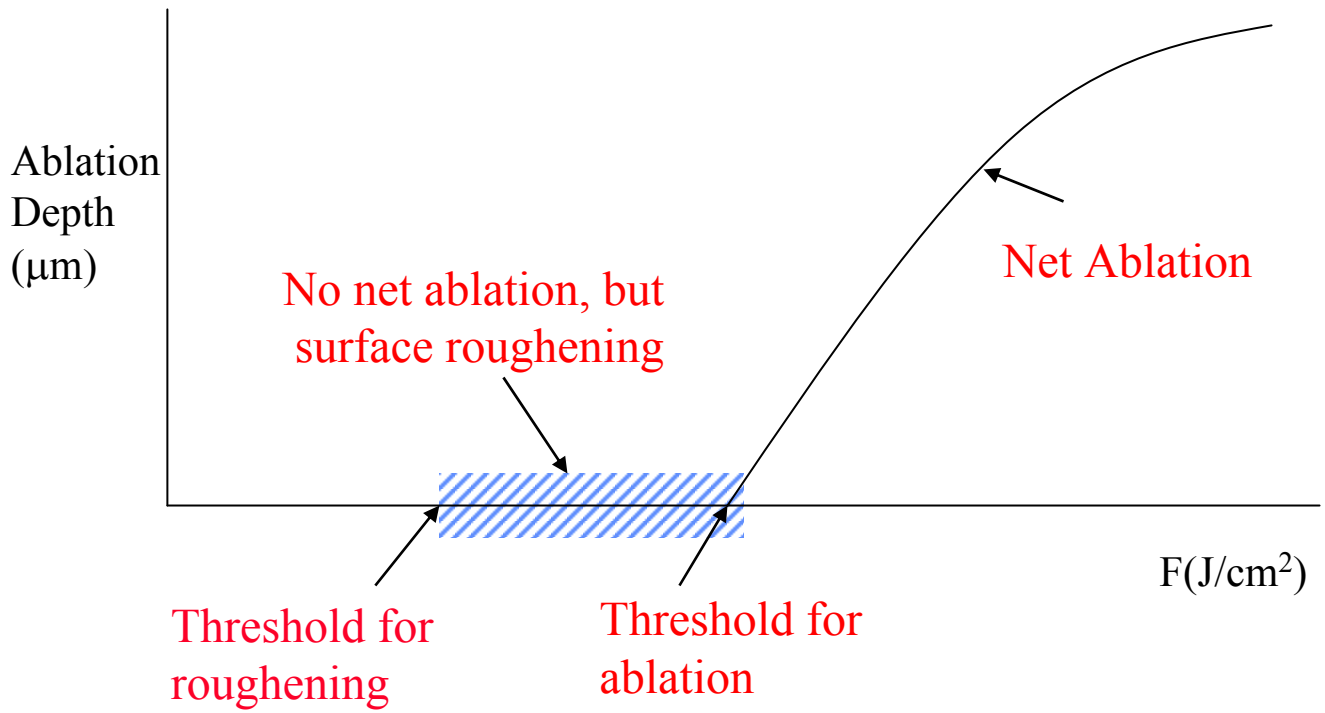
Regimes of IFE Materials Response Studies for x-rays and ions



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

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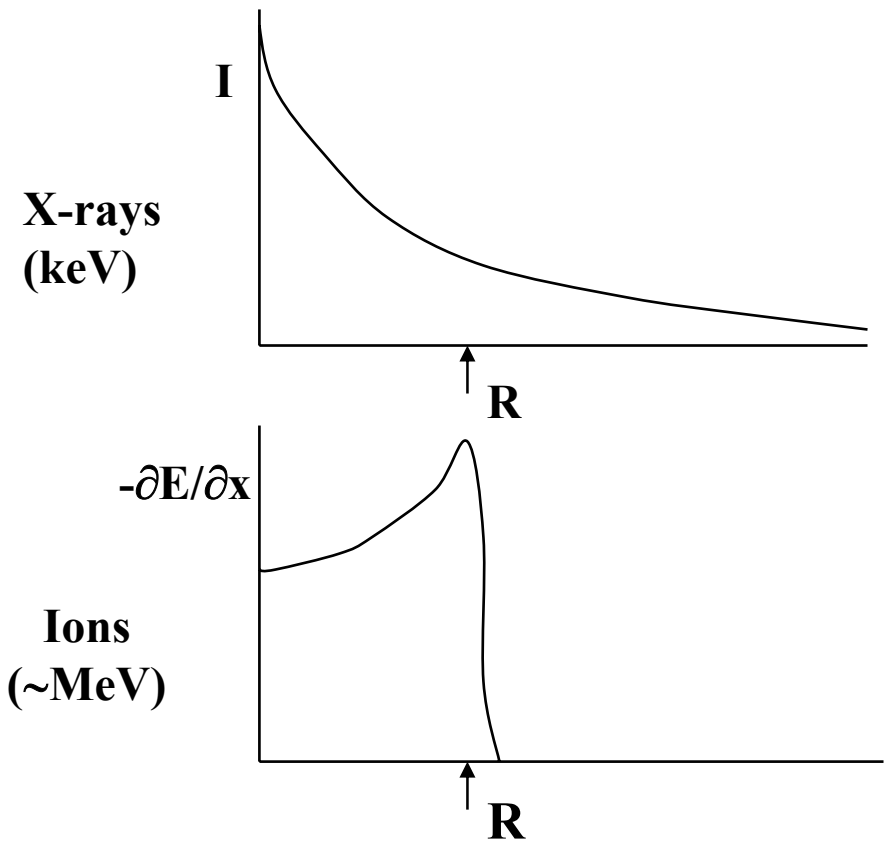
X-rays and ions deposit energy with different spatial profiles



photoelectric absorption of photons

photons removed from flux

I <u>decreases</u> exponentially

absorption length R = $(\mu\rho)^{-1}$

penetrates beyond R

ion collisions with electrons

ions slow down continuously

$|-\partial E/\partial x|$ <u>increases</u> to Bragg peak

range is R

essentially no penetration beyond R

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Materials response testing should accurately reproduce specific energy deposition in candidate materials

Need to reproduce:

(1) specific energy deposition $S = \Delta E/\Delta V \sim F(J/cm^2)/R(g/cm^2)$

(2) temporal profile $S(t)$ [$\partial S/\partial t$, pulse width,…)
rate of rise competes with thermal conductivity

(3) spatial profile $S(x)$ [deposition profile,…]
deposition scale length compared to
grain size, impurity scale size,…
temperature gradients in deposition region

Z and RHEPP can replicate the conditions in IFE chamber walls

- **Though there are some differences between IFE target spectra and Z and RHEPP, the experiments study the most important issues**
- **The surface temperature histories and temperature profiles are similar**

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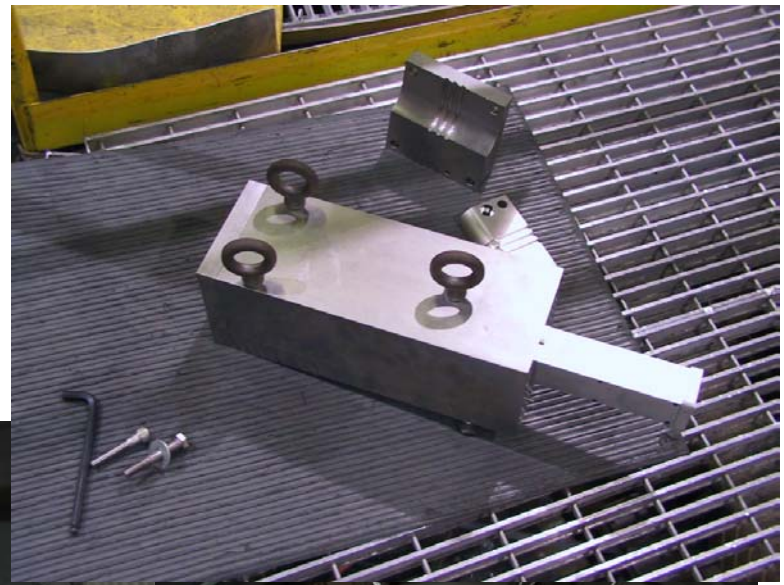
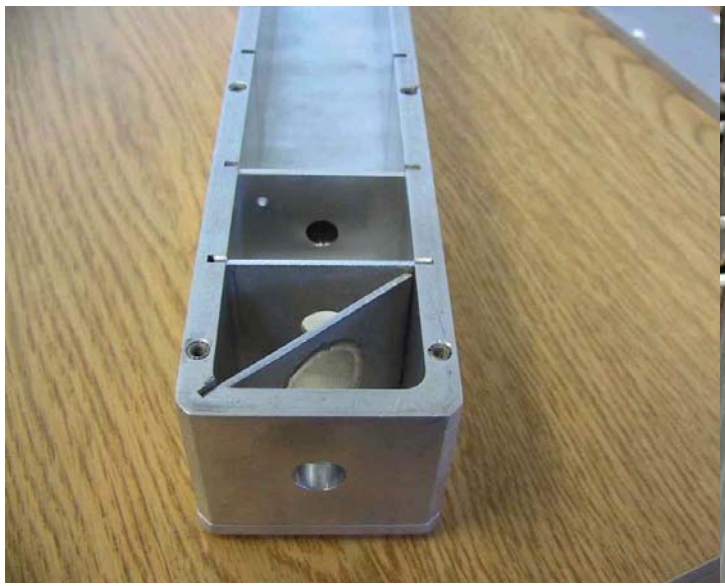
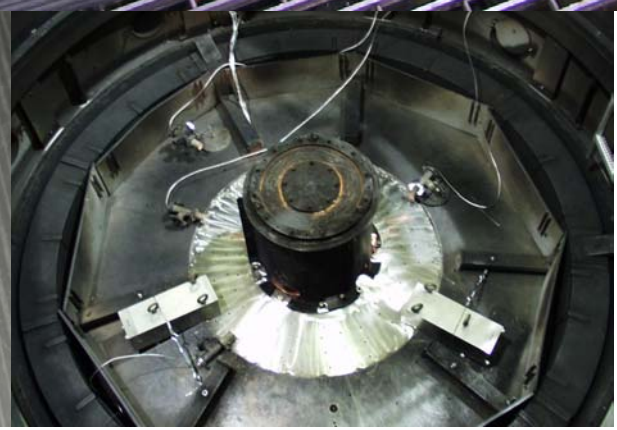
Multipurpose Radiation Box (MPR) used in Z experiments

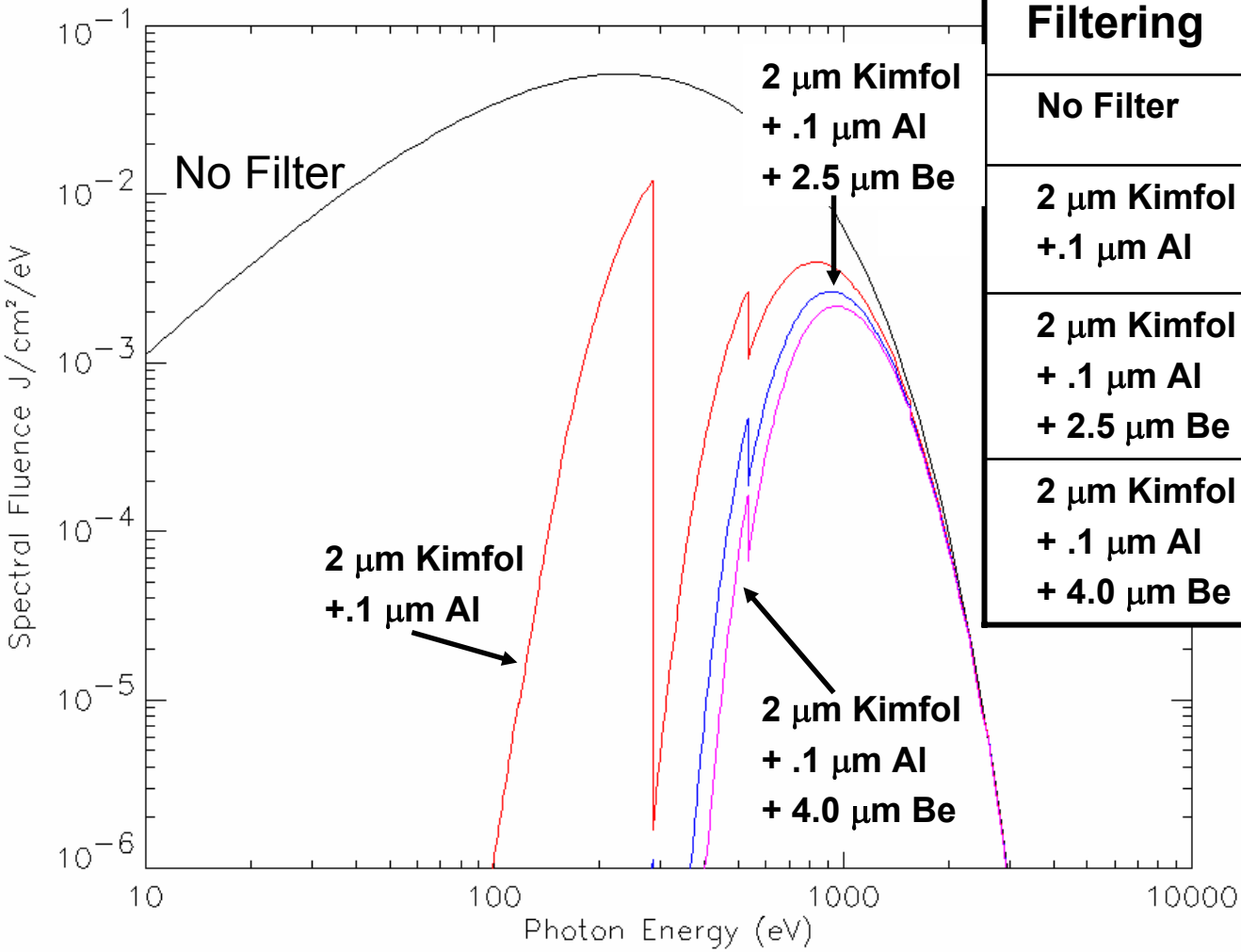
- Piggy back on tungsten wire array Z shots
- High collimation
- Front aperture 50 cm from pinch
- 4 samples per shot
- Filters to approximate NRL target







X-Ray Spectrum and Fluence With Various Filters on Z



Filtering	Fluence	<E>
No Filter	19 J/cm²	443 eV
2 μm Kimfol +.1 μm Al	2.3 J/cm²	830 eV
2 μm Kimfol + .1 μm Al + 2.5 μm Be	1.3 J/cm²	1050 eV
2 μm Kimfol + .1 μm Al + 4.0 μm Be	.98/cm²	1100 eV

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Examples of Data from Z for W and W/Re: Depth of molten layer (ion mill technique)

Fluence (J/cm^2)	Pure W (μm)	75%W/25%Re (μm)
19	2	2
2.3	0.5	1.5
1.3	0	0

Images of samples are shown on next 2 slides

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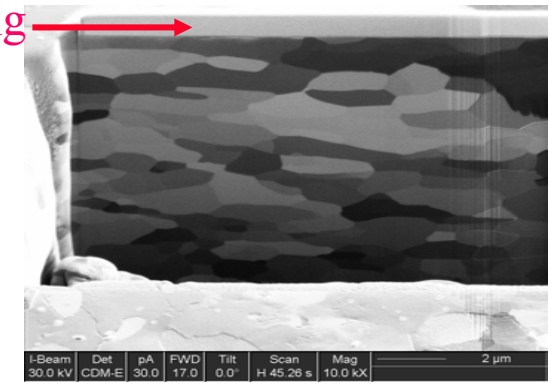
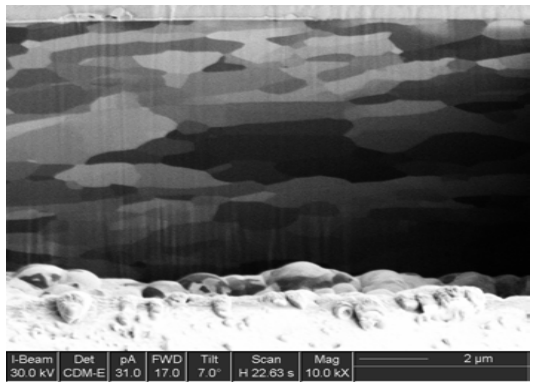
Ion Images of W after Exposure to x-rays on Z

Platinum coating



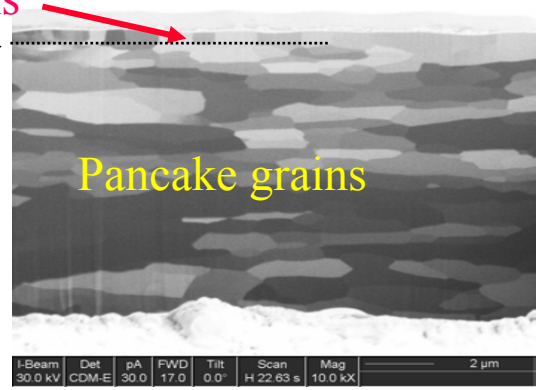
Unexposed



Exposed to 1.3 J/cm^2

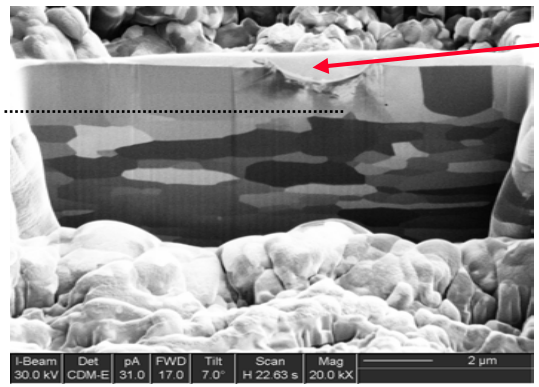
Columnar grains

Bottom of melted region



Exposed to 2.3 J/cm^2

Debris Crater



Exposed to 19 J/cm^2

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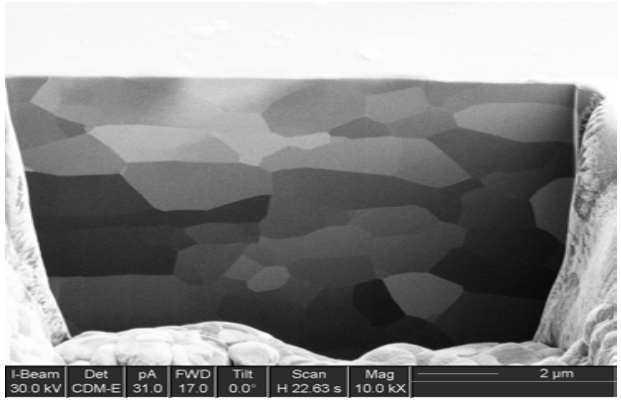
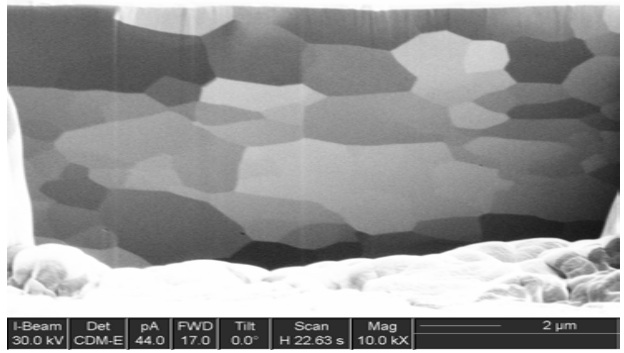
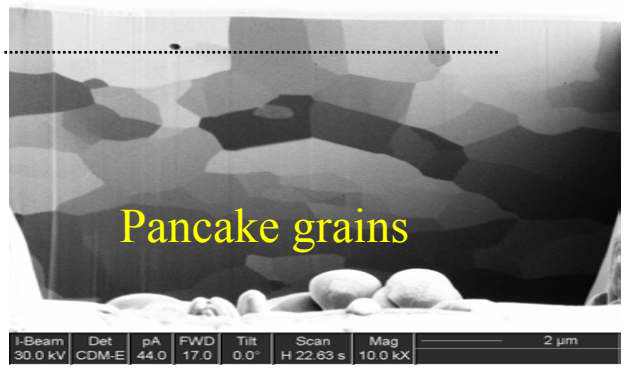
Ion Images of W75%Re25% after Exposure to x-rays on Z



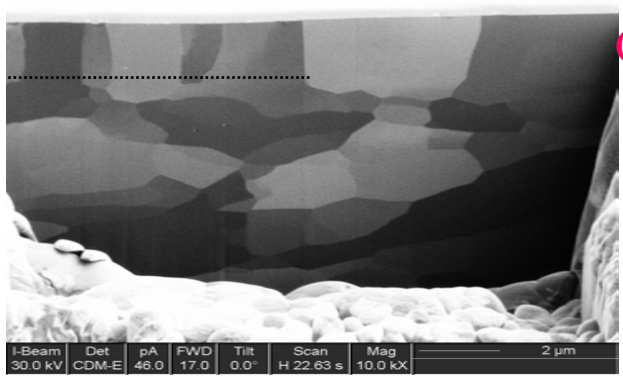
Unexposed sample



Exposed to 1.3 J/cm^2



Exposed to 2.3 J/cm^2



Columnar grains

Exposed to 19 J/cm^2

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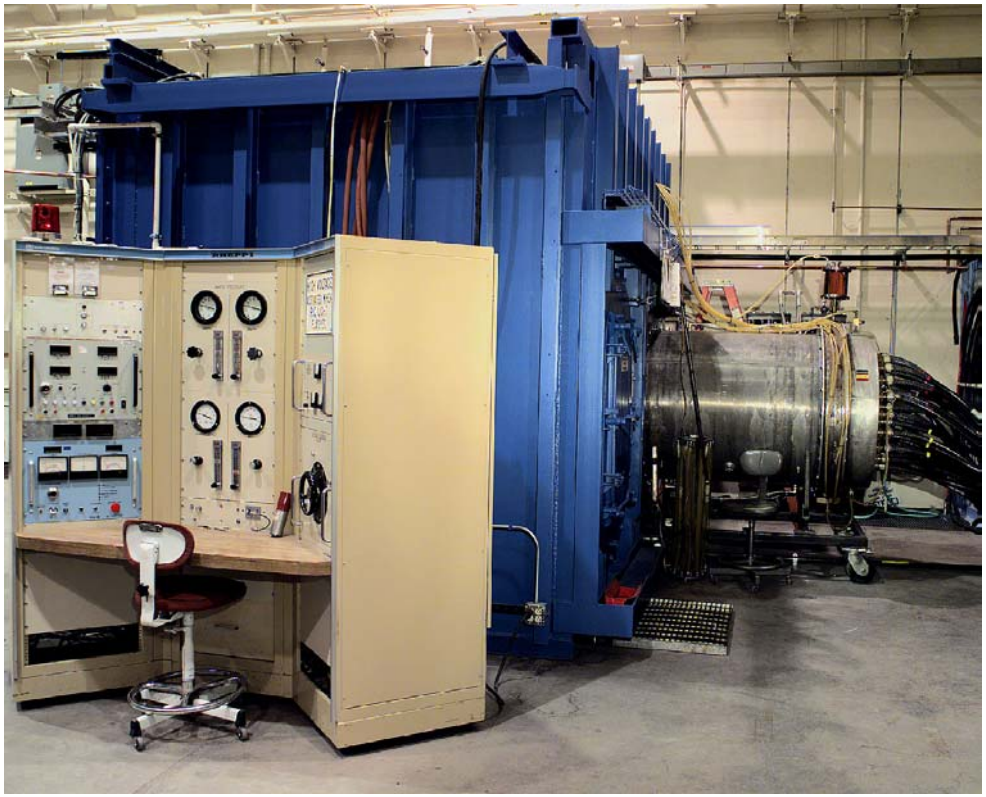
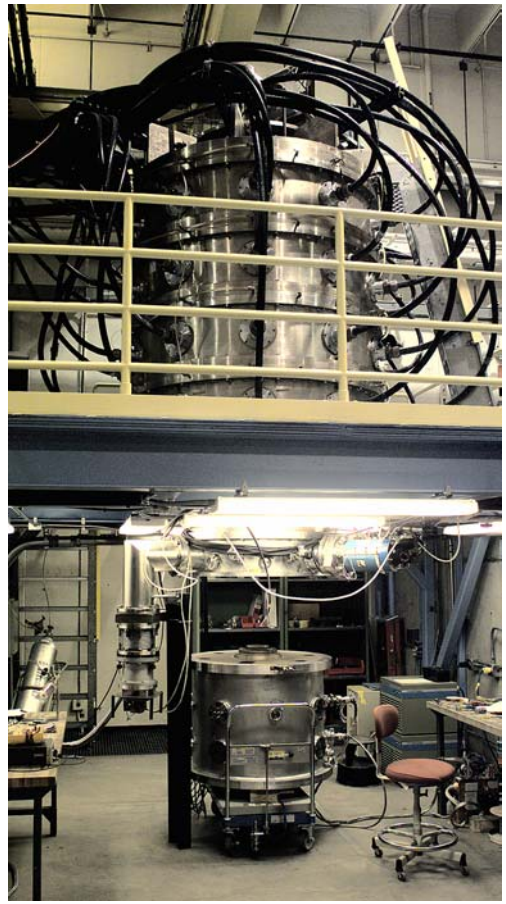
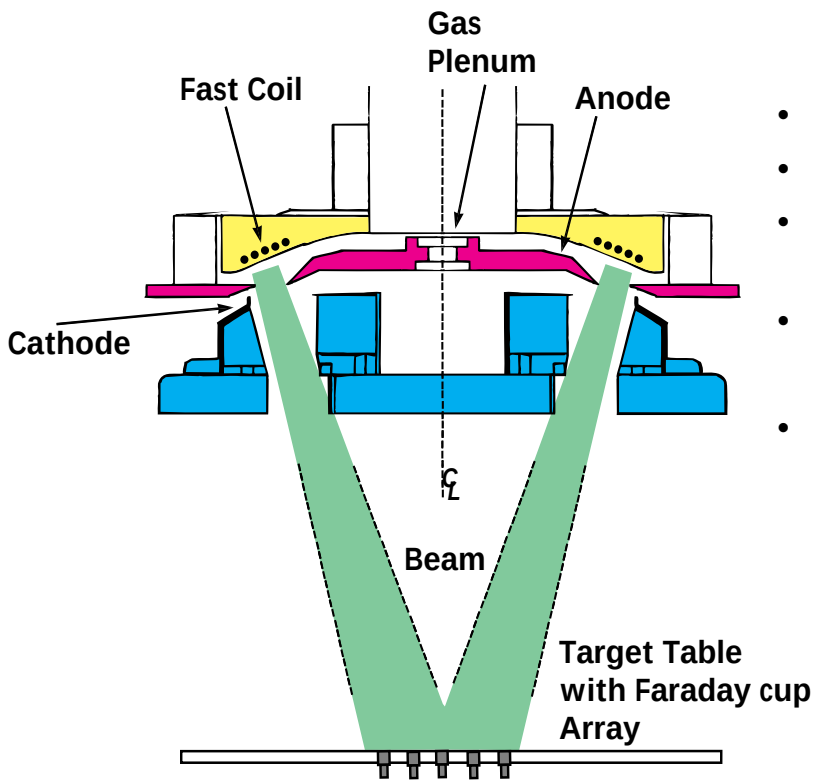
RHEPP-1 combines Repetitive High Energy Pulse Power with a robust and versatile ion source: MAP





Left: Marx tank with pulse-forming line
Right: 4-Stage LIVA and treatment chamber

The MAP (Magnetically Confined Anode Plasma) Ion Source is used for surface modification experiments on RHEPP-1



- 500-700 kV
- ≤ 250 A/cm^2
- Beams from H, He, N_2, O_2, Ne, Ar, Xe, Kr, CH_4
- Overall treatment area ~ 100 cm2
- Diode vacuum ~ 10^{-5} Torr

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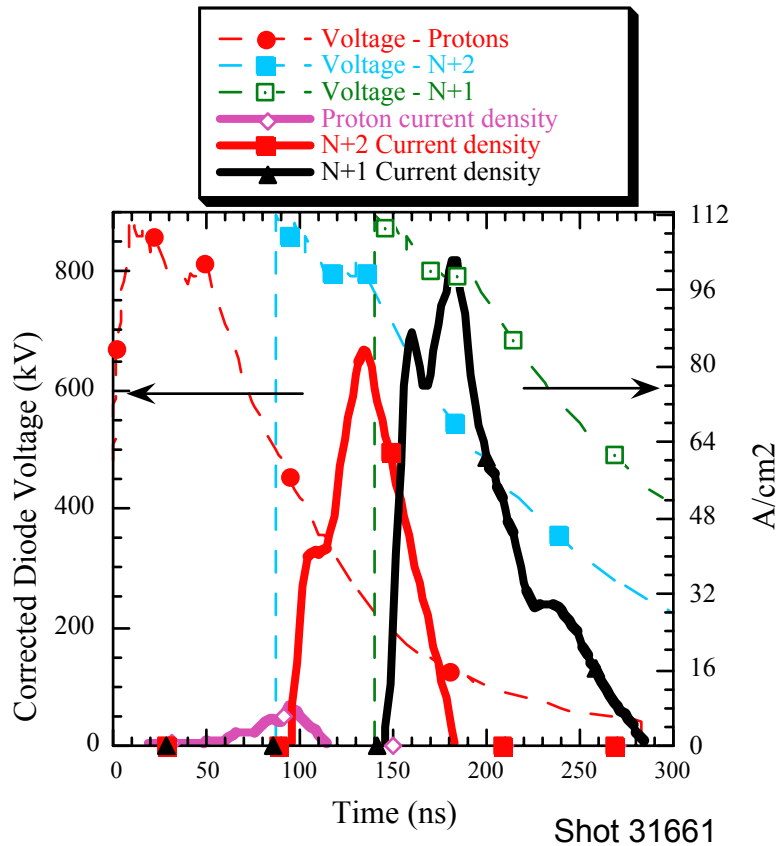
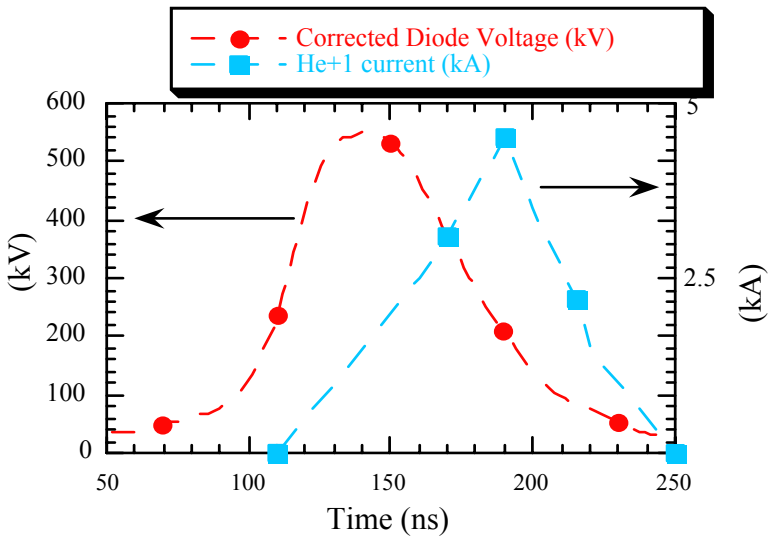
Nitrogen injection into MAP produces 3-component beam of mostly N++, N+



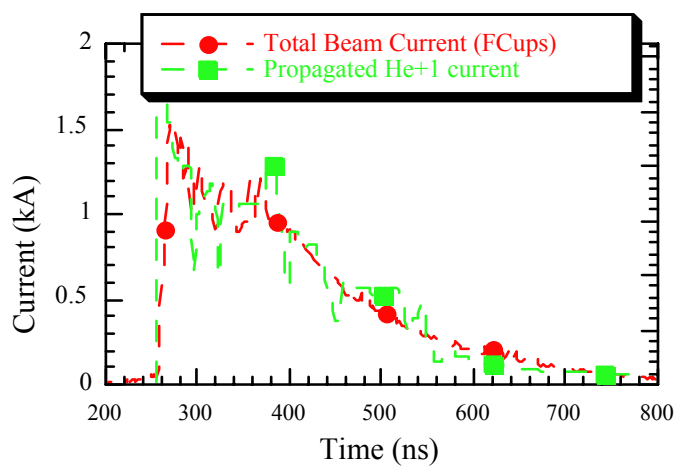
Shot 31661

- Beam predominantly N++ and N+ after small proton pulse at front
- Peak voltage = 850 kV
- Peak current density (total) ~145 A/cm^2
- Total fluence = 7.9 J/cm^2 - 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

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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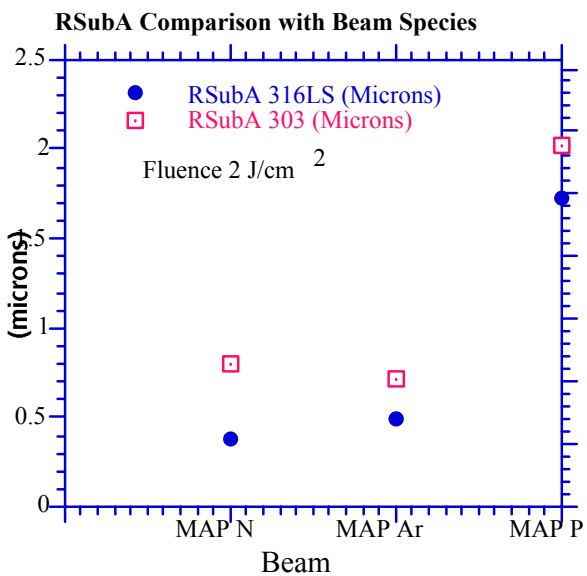
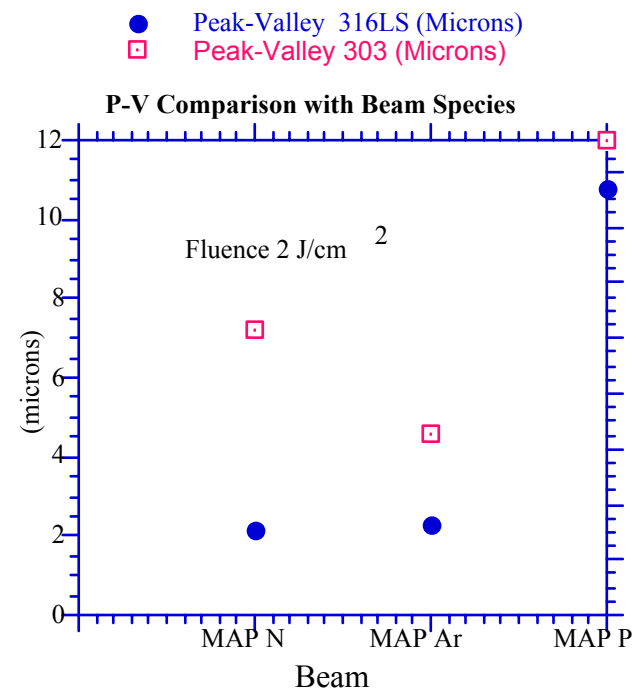
Roughening of SS303, SS316LS varies with ion beam species



- 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



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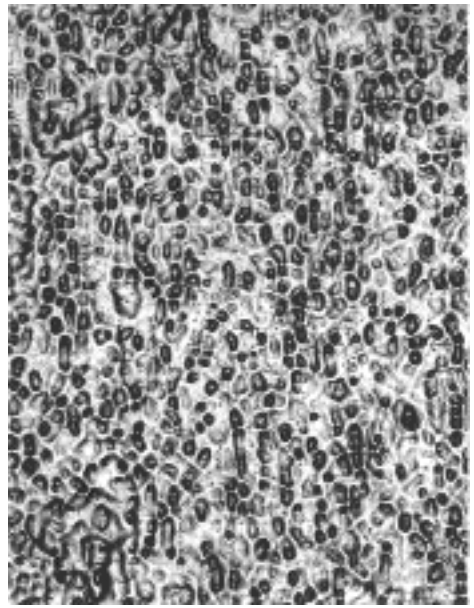
Surface morphology changes on SS samples depend upon treatment ion(s)

Map P 25x

Map N 25x

Map Ar 25x



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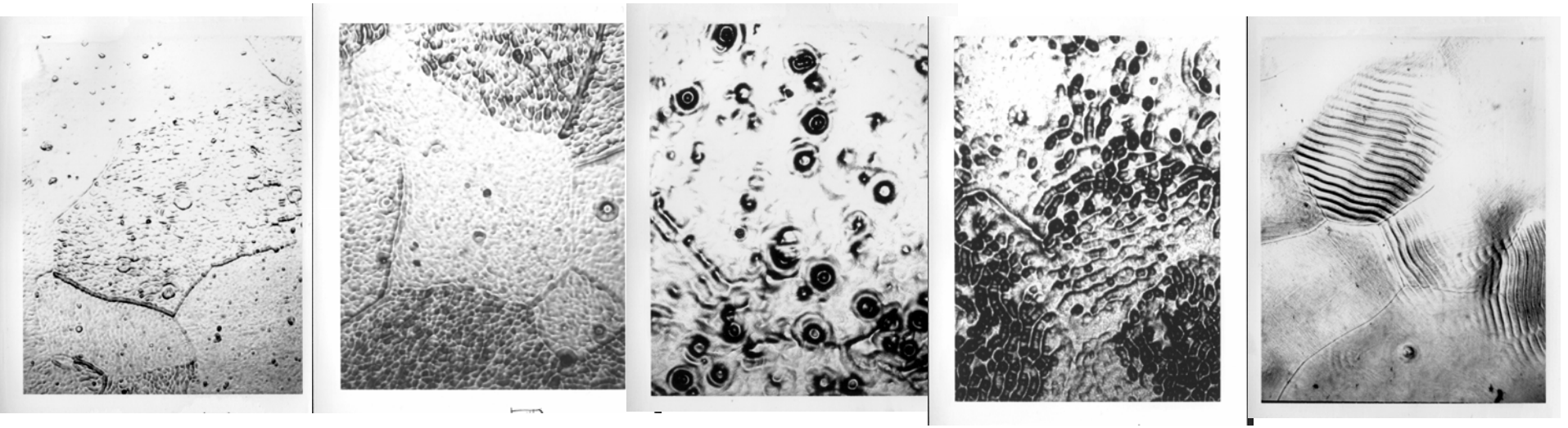
RHEPP-1 on Ti Alloy with 160-pulses O beam: Materials response governed at grain level



~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

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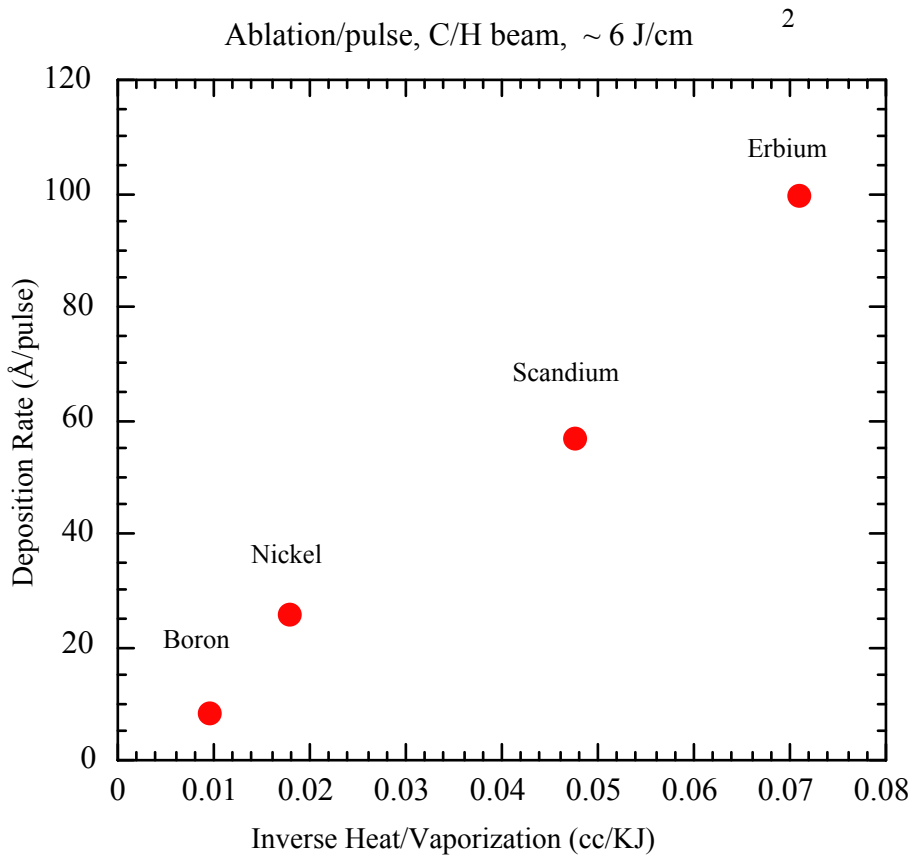
Deposition rate from ion beam ablation appears related to Heat of Vaporization



- **Polished samples of B, Ni, Sc, Er exposed to C/H ion beam at fluence ~ 6 J/cm^2.**
- **Beam-target distance = 40 cm Target - substrate distance = 28 cm**
- **X-axis: inverse heat of vaporization in units cc/kJ**
- **Y-axis: Deposition rate in units Å/pulse**

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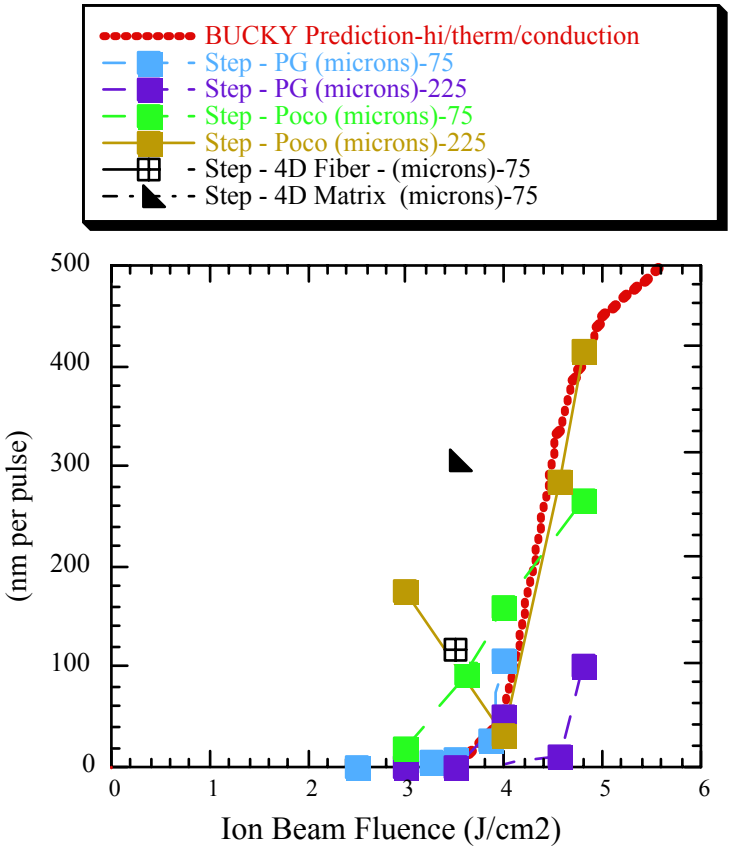
Response of graphite to mixed H - C beam qualitatively confirms BUCKY predictions



- 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)

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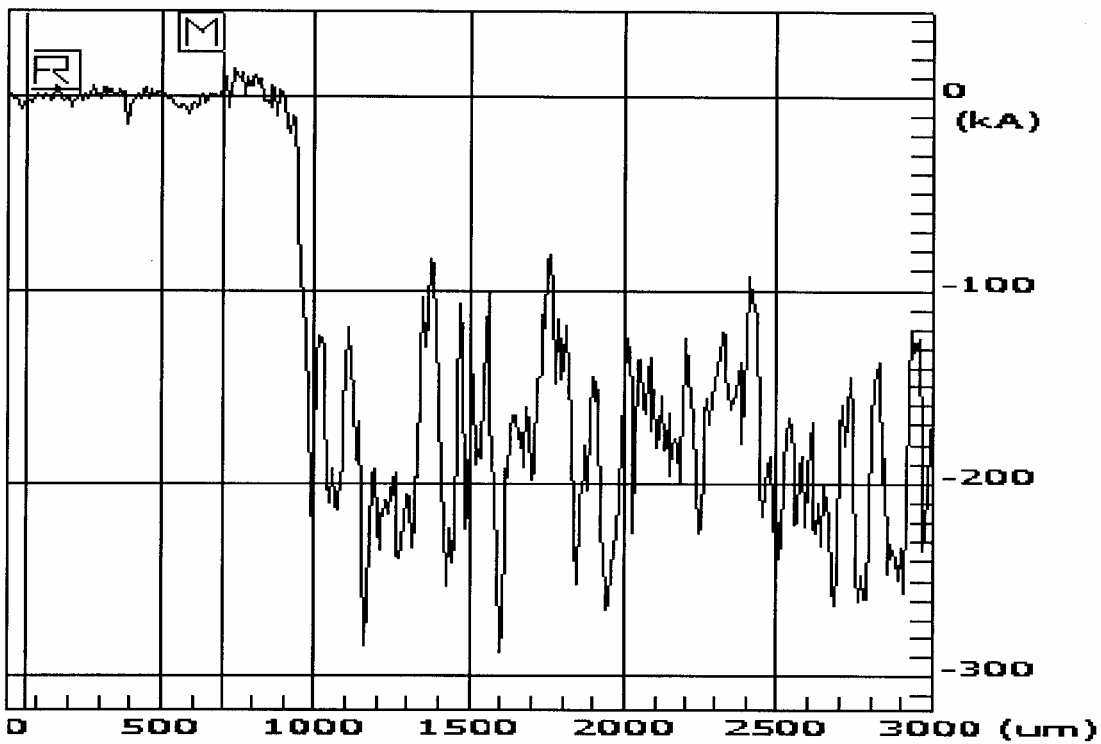
Poco Graphite exposed to 5.5.J/cm^2 carbon/proton beam ablated at rate of 0.3 microns/pulse



- mechanically polished Poco graphite exposed to 75 pulses of 70% C/30% H beam at average dose of 5.5 J/cm^2
- Profilometer scan across interface (left) shows ~ 20 micron step (0.27 μ/pulse)
- Ra (original) = 0.23 microns
- Ra (treated) = 3.6 microns

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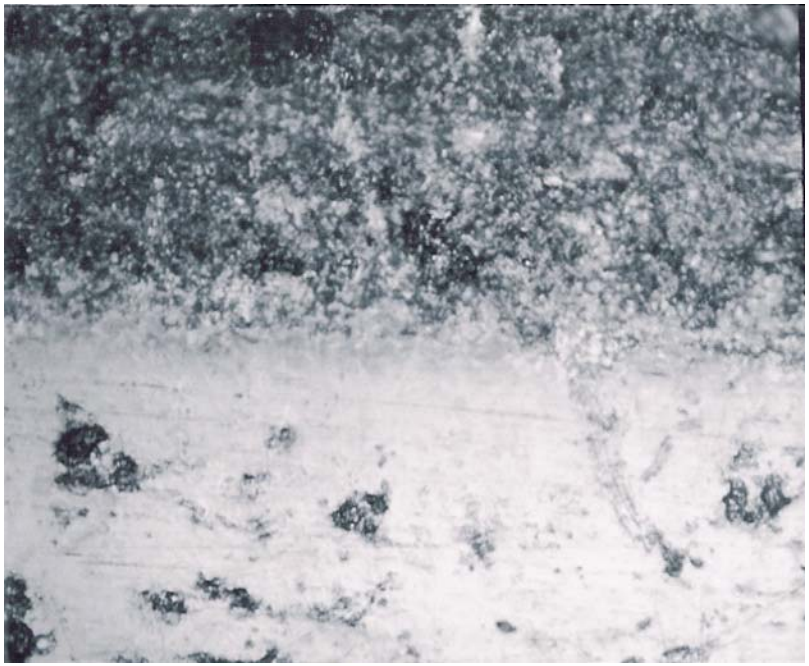
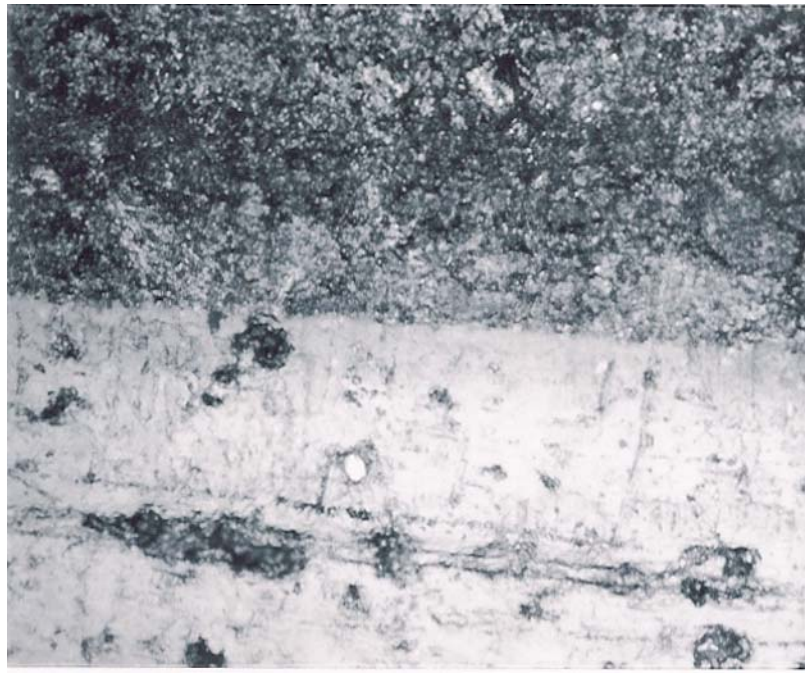
Poco subjected to 400 shots from He beam, Shows visible change down to 0.5 J/cm^2



Interface, untreated Poco (bottom) and 0.25 - 0.5 J/cm^2 (top) - image 20x



Interface, untreated Poco (bottom) and 0.5 - 1 J/cm^2 (top) - image 20x

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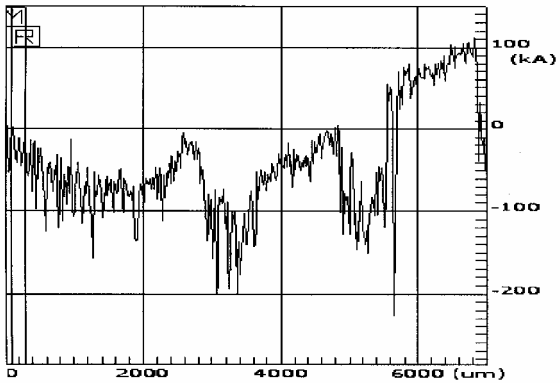
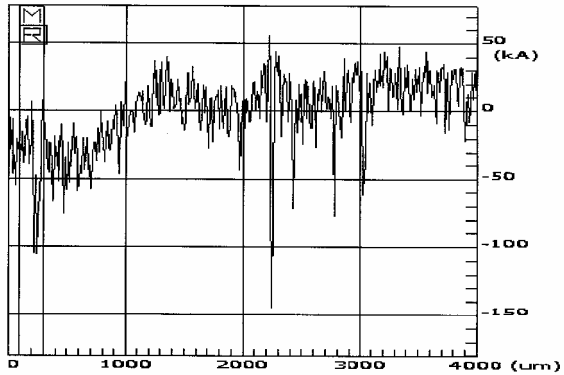
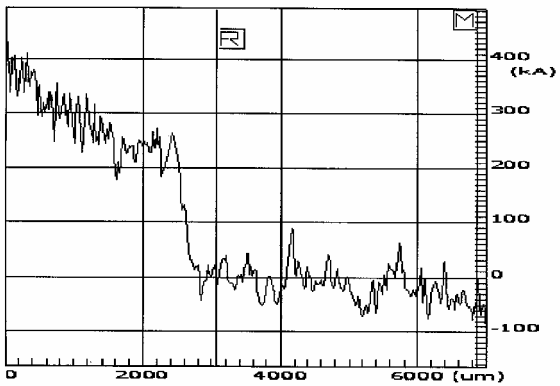
Profilometer scans across interface above and below ablation threshold shows that both fiber and matrix ablate

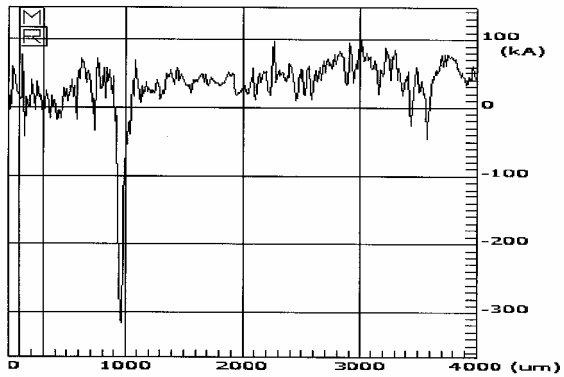


Scan across
Fiber interface





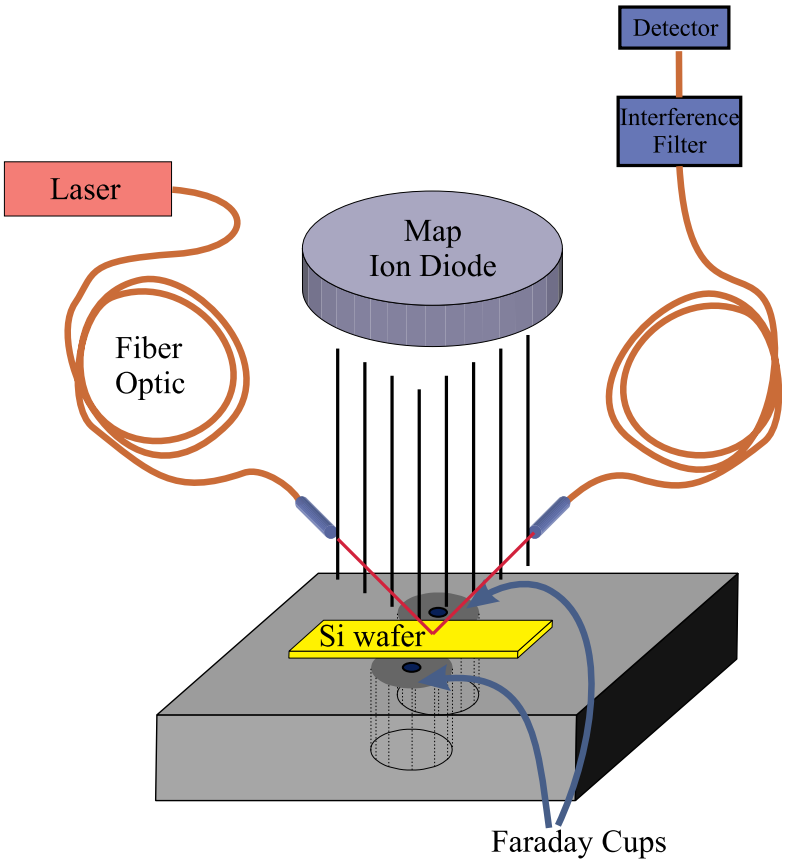
Scan across
Matrix interface



Dose = 3.5 J/cm^2
(Above ablation threshold)
Fiber step = 8.7 microns
Matrix step = 21 microns

Dose = 1.9 J/cm^2
(Below ablation threshold)

We have developed a surface reflectometer to measure surface roughening thresholds and temperature signatures



- Photodiode collects laser light from surface - before/after comparison shows roughening, which is correlated to ion dose from FCups
- Collected photon output (no laser) through InGaAs detector can be unfolded to show T (t)
- Roughening data compared to profilometer

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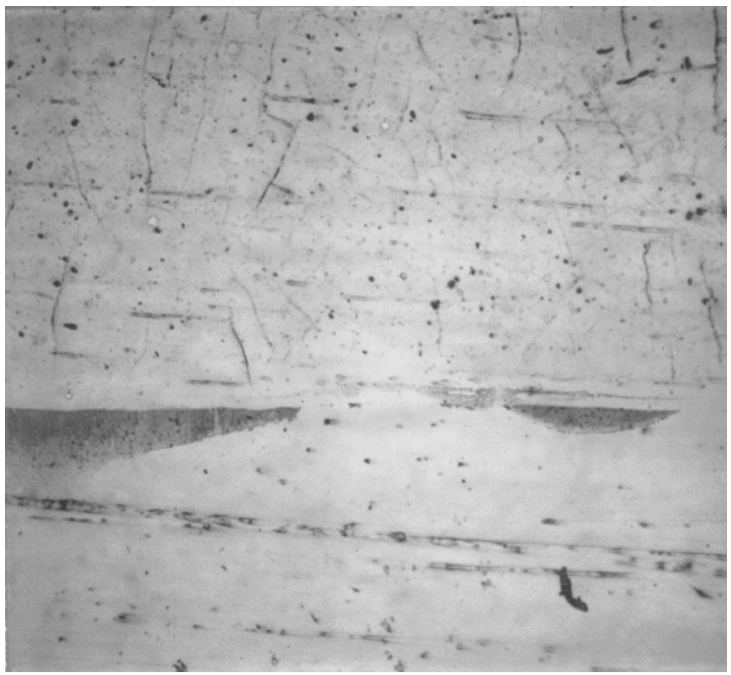
Reflectometer measurements show 99.95% pure W roughens well below melting temp - suspect brittle cracking response

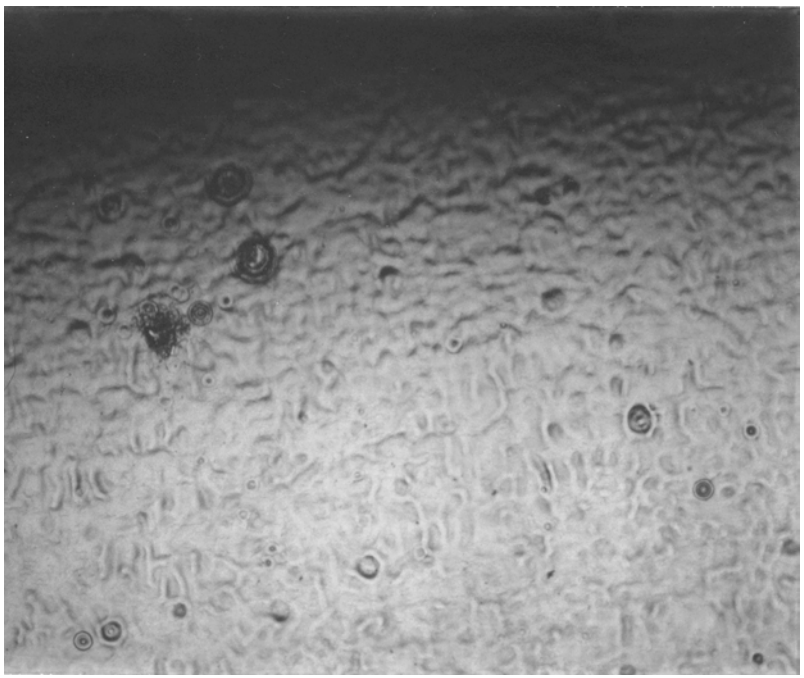
20X Optical Image



- **Polished W exposed to N beam: 0.6 <dose< 1.25 J/cm^2 (53 shots)**
- **Reflectometer shows progressive roughening reached at 1.25 J/cm^2**
- **Simulation: 1.25 J/cm^2 N beam on W yields 1900K max temp (Ablation @ 7 J/cm^2)**
- **Top half - exposed. Note cracks and increased pitting. Suspect thermomechanical cracking from expansion/contraction cycles**
- **Bottom half - masked**
- **Roughening threshold for W/25Re: 3.5 J/cm^2**

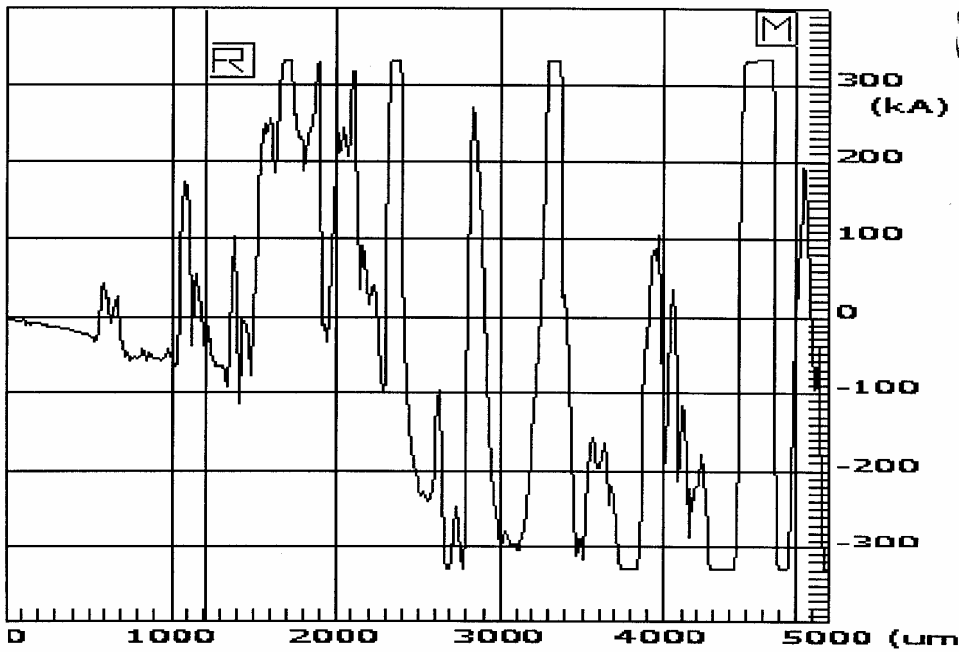
Tungsten w/ 25% Re added exposed to MAP N beam shows much less roughening/surface cracking than pure W

20X Optical Image



- **Polished W25Re exposed to N beam in 6 dose levels - 0.6 to 3.5 J/cm^2. Total of 54 shots**
- **Threshold for progressive roughening reached at 3.5 J/cm^2 as indicated by reflectometer**
- **Optical image shows melt/reflow morphology, with beam debris**
- **R_a (exposed) ~ 0.69 μ (highest)**
- **R_a (unexposed) ~ 0.09 μ**
- **SIM ablation threshold ~ 3.5 J/cm^2**

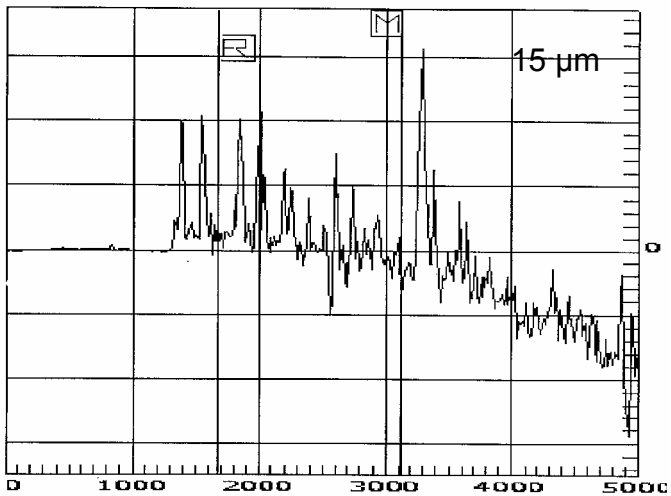
W(99.95% pure) subjected to 400 shots from N beam, shows dramatic roughening at 4.5 J/cm^2



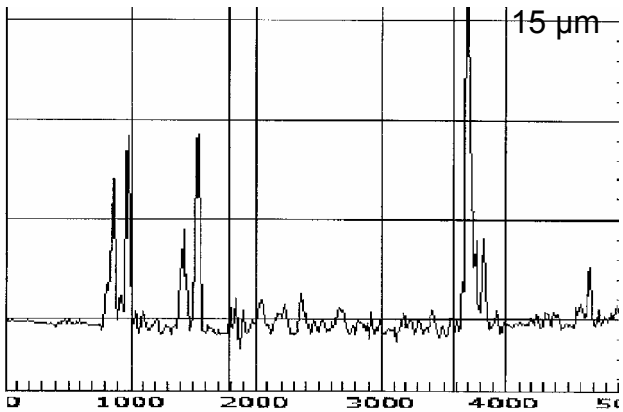
- Polished W exposed to N beam for 400 shots at up to 4.5 J/cm^2
- Profilometer scan (left) of W exposed to 4.5 J/cm^2 (ave)
- Max/min excursions beyond measuring ability
- Indicated R_a = 18.4 μm
- Indicated Max-Min = 66.7 μm

W and W25Re heated 600C, 250 shots @ 3 J/cm^2: W much rougher, but both have 15 µm features

W 99.95%



W25%Re



4 mm

- **Mechanically polished W and W25Re heated to 600C, exposed to 250 pulses of MAP N beam at 3.0 J/cm^2**
- **W R_a ~ 1.8-1.9 µm, W25Re R ~ 0.3 µm, both have 15 µm feature heights**
- **W shows complex topology, W25Re periodic 'lumps'**

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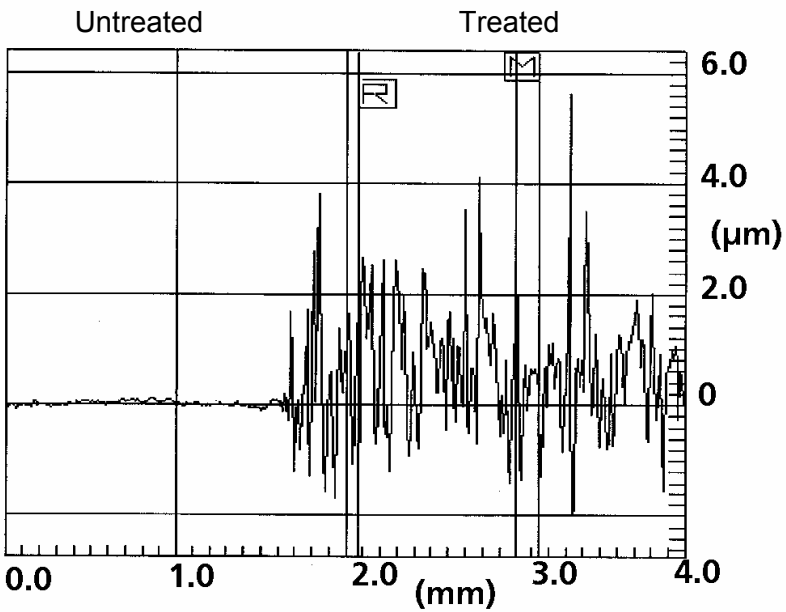
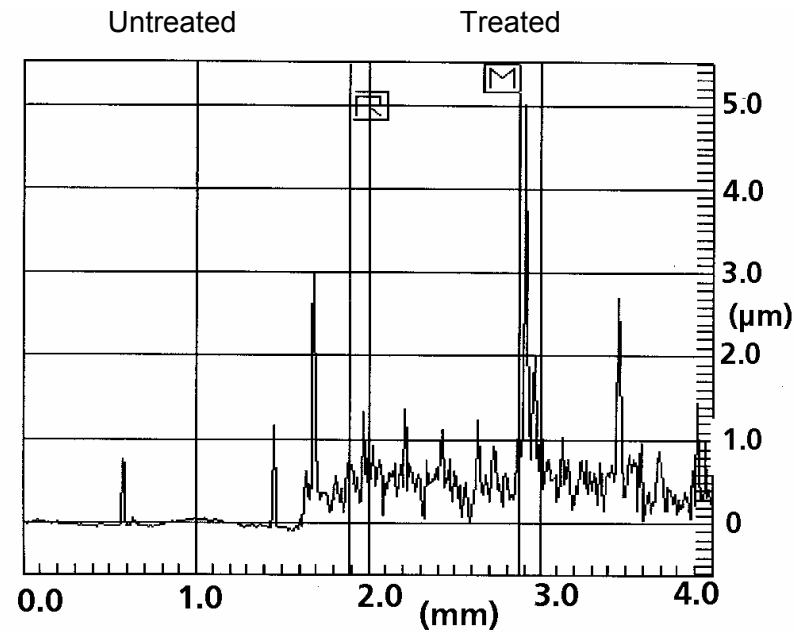
1-D Profilometer scans for 600C Mo and W: W appears smoother but 'expanded' after treatment



Molybdenum
Fluence: 2.5 J/ cm^2
Modeled surface temp: 2900K



Tungsten
Fluence: 2.5 J/ cm^2
Modeled surface temp: 3685K
Note ~ 0.5 µm rise on right

Brittle-Ductile transition:
~ 300C for both Mo and W

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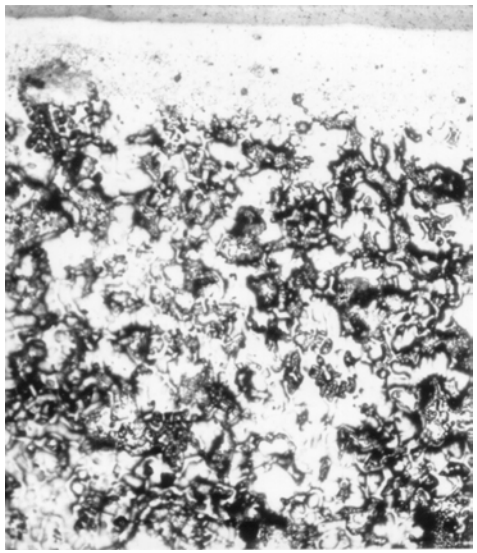
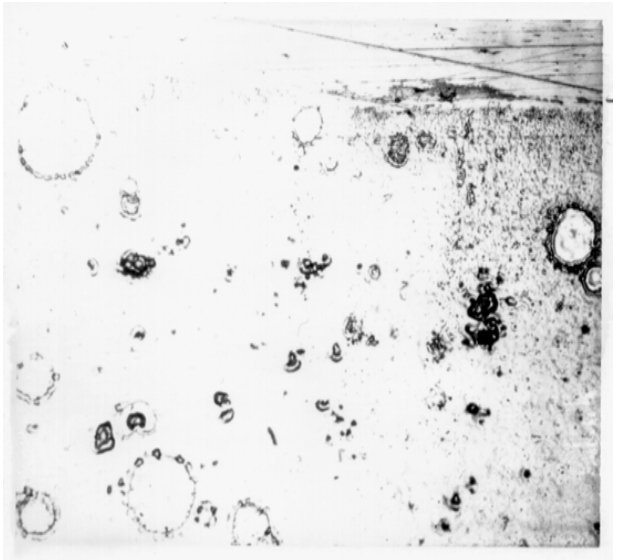
He Beam treatment 450 shots:
600C W roughens w/o melt worse than W/Re with melt





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

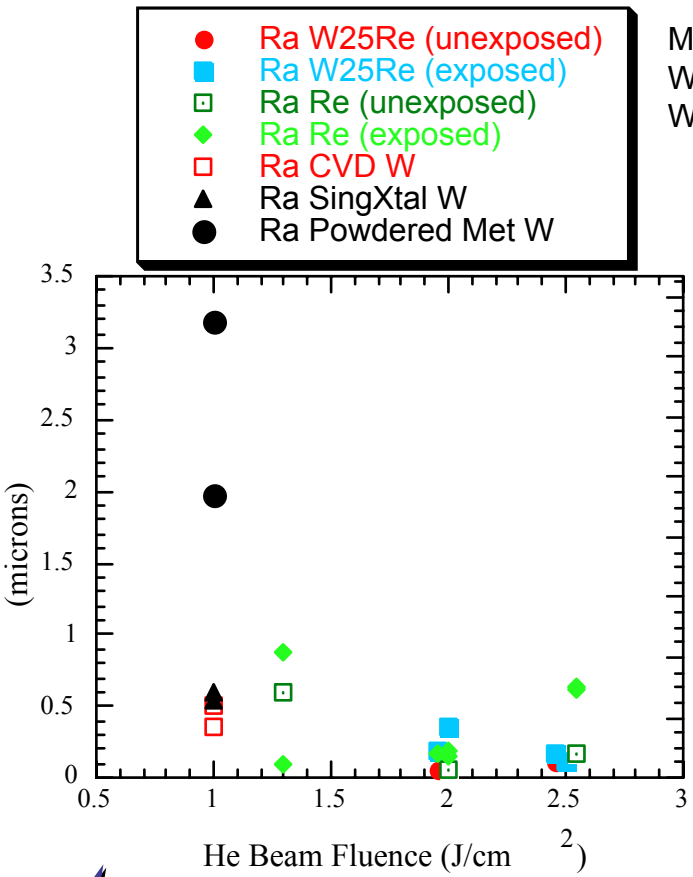
Powder Met W
Same fluence
R_a : 2 - 3 μm

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

These surfaces did not melt

This surface melted

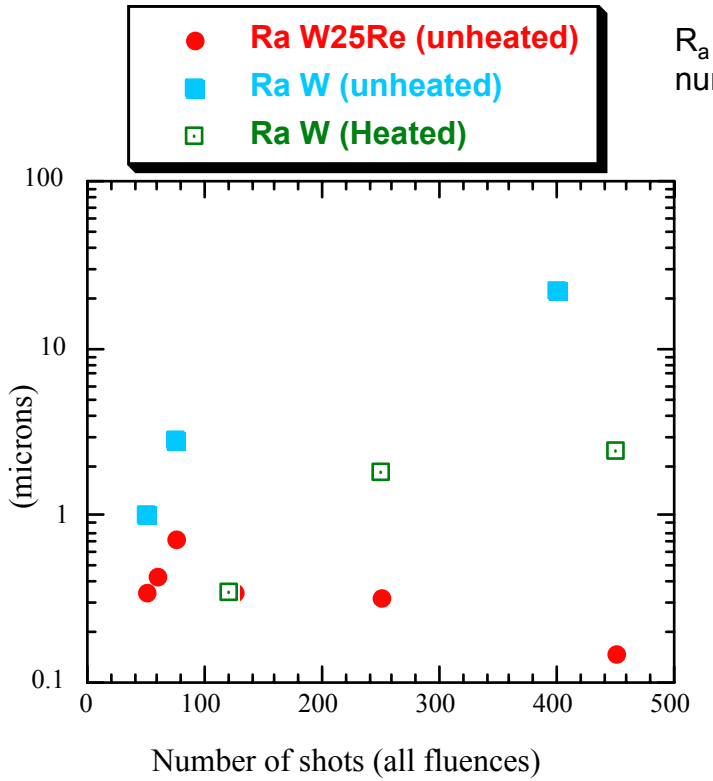
He Beam exposure, 450 shots; Powder Met W has worst roughening, even when heated



Melting fluence:
W/25Re: 2.7 J/ cm^2
W: 3.0 J/ cm^2

- **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 12.5 J/cm^2. Samples at RT**
- **PwderMetW Roughening is significantly worse than CVD or SingXtal 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.**

Summary of roughening behavior of W/Re, W, and W heated surfaces



R_a as function of number of shots

- **Unheated W surface exceeds Ra = microns at 400 shots**
- **Both heated and unheated W roughening appear open-ended**
- **W/25Re shows no Ra > 1 micron, and no upward trend with shot number**

GRAND SUMMARY of experimental data, BUCKY code results, and analytic theory results for IFE candidate first wall materials exposed on Z and RHEPP

	Material	Bucky predicted ablation threshold	Analytic predicted ablation threshold	Experiment measured ablation threshold	Experiment measured roughening threshold	Predicted threat to wall	
						154 MJ target	400 MJ target
X-rays on Z (10 ns exposure)	Poco graphite	4.25 J/cm^2	3.3 J/cm^2	> 8 J/cm^2	grain removal < 8 J/cm^2	0.4 J/cm^2	1.2 J/cm^2
	tungsten	3.5-4 J/cm^2	2.4 J/cm^2	2.3-19 J/cm^2	2.3 J/cm^2		
	tungsten/Re			> 19 J/cm^2	> 2.3 J/cm^2		
	tungsten/La			> 19 J/cm^2	> 2.3 J/cm^2		
Ions on RHEPP (~100 ns exposure)	Poco graphite	3.2 J/cm^2	1.8 J/cm^2	2.5-3.0 J/cm^2	< 1 J/cm^2	8.5 J/cm^2 (1.8 J/cm^2 if $t^{1/2}$ applies)	21.1 J/cm^2 (4.5 J/cm^2 if $t^{1/2}$ applies)
	pyrolitic graphite	1.6 J/cm^2 ⊥ 2.5 J/cm^2 ll	1.2 J/cm^2 ⊥ 3.0 J/cm^2 ll	< 3.0 J/cm^2 3.5-4.0 J/cm^2	2.5 J/cm^2		
	tungsten	6 J/cm^2	5.1 J/cm^2	> 6 J/cm^2	1.25 J/cm^2		
	tungsten/Re			> 6 J/cm^2	3.5 J/cm^2		

X-ray threat does not appear to be a problem
Ion threat does appear to be a problem

Summary

- RHEPP-1 and Z can expose candidate IFE first wall materials to reactor-level ion and Xray fluences
- X-ray fluence levels ~ 1 J/cm2 do not seem a significant threat to graphite and W surfaces
- Ion threat more serious:
  - Graphite: ablates readily above ~ 3.5 J/cm2
  - W: thermomechanical roughening which shows signs of open-ended behavior
  - W/Re alloy: increased ductility may address roughening problem
  - Engineered surface (‘velvet’) shows promise in reducing material loss
- Surface response to ion exposure is dependent upon ion type, microstructure, surface operating temp

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Workshop Agenda