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EUROPEAN SPALLATION SOURCE

Pulsed Heavy Ion Irradiation of Tungsten

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Purpose of experiment is to study:

- Mechanical integrity of W under dynamic beam loads
- Radiation induced changes
  - of mechanical properties
  - in electrical and thermal conductivities
- Stability of W-oxide layer
  - Sample pre-oxidized at 500°C in air for 24h
- Beam pulse induced dynamic response of W foil and its change due to irradiation

M3-beamline of GSI UNILAC

Beam conditions at GSI

- Uranium beam
  - Beam energy: 4.8 MeV/u
  - Ion flux: ~1e10 ions/cm^2/pulse
  - Pulse length: 100 µs
  - Repetition rate: 1 Hz

ESS beam

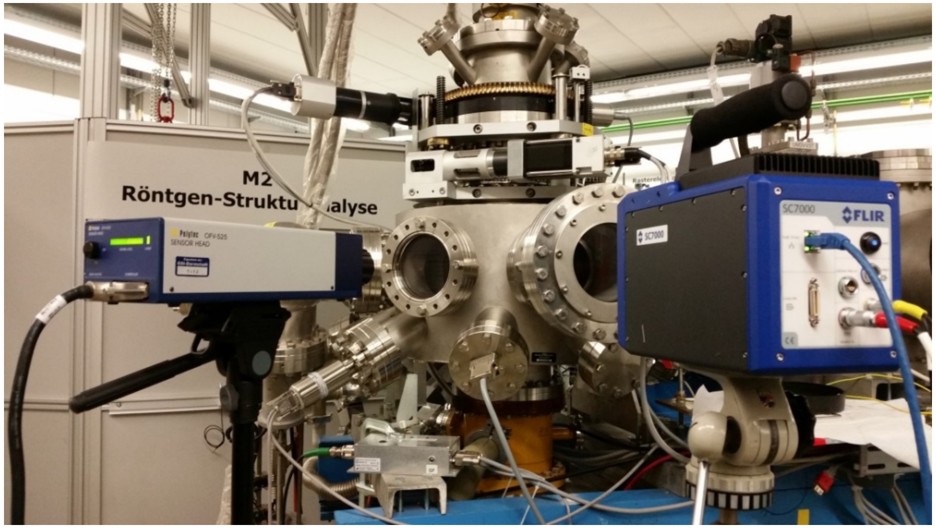
- Energy - 2.5 GeV
- Rep. rate - 14 Hz
- Pulse length - 2.86 ms

Two types of rolled tungsten specimens

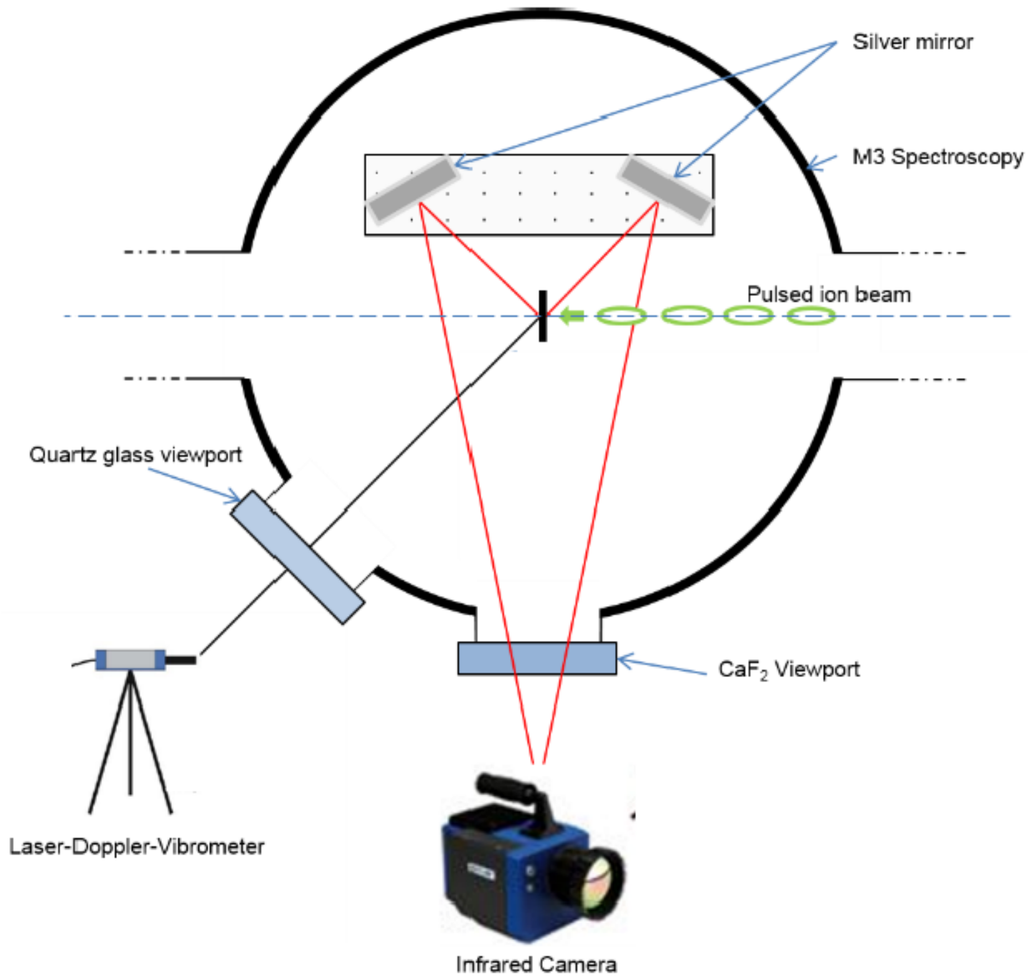
- Coin:
  - 20 mm dia., 3 mm thick
  - Beijing Tianlong Tungsten & Molybdenum Co., Ltd
  - Beam induced cyclic shear stress
- Foil:
  - 20 mm dia., 0.026 mm thick
  - Plansee Metall GmbH, Austria
  - Tensile and compressive stress waves

Online measurements

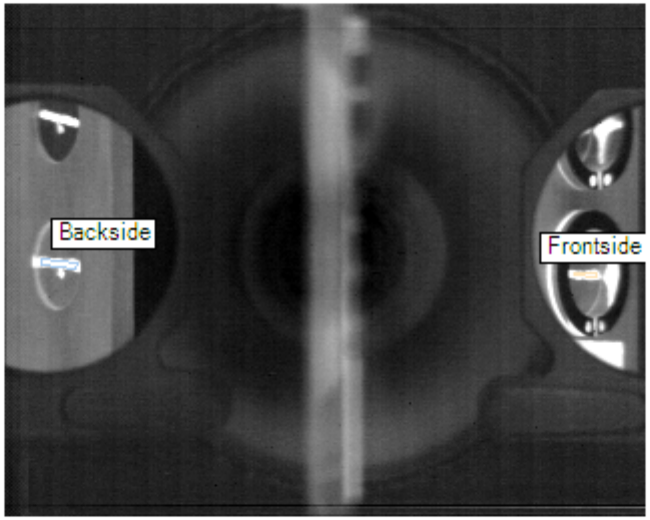
- Laser Doppler Vibrometer (LDV)
  - Monitors vibrations on surface induced by beam pulses
- IR Camera FLIR Systems SC7500
  - Measures temperature on both sides of sample
- Optical Camera
  - Surface failure detection



Irradiation set-up



Mirrors for temperature measurements



Response of High-Power Accelerator Materials to a short pulse 4.8 MeV/u Uranium beam, Pascal Simon, Advanced Research Lab summary report, January 2016, GSI Darmstadt.

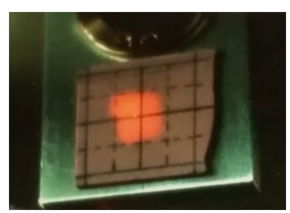
Experimental set up – Sample holder

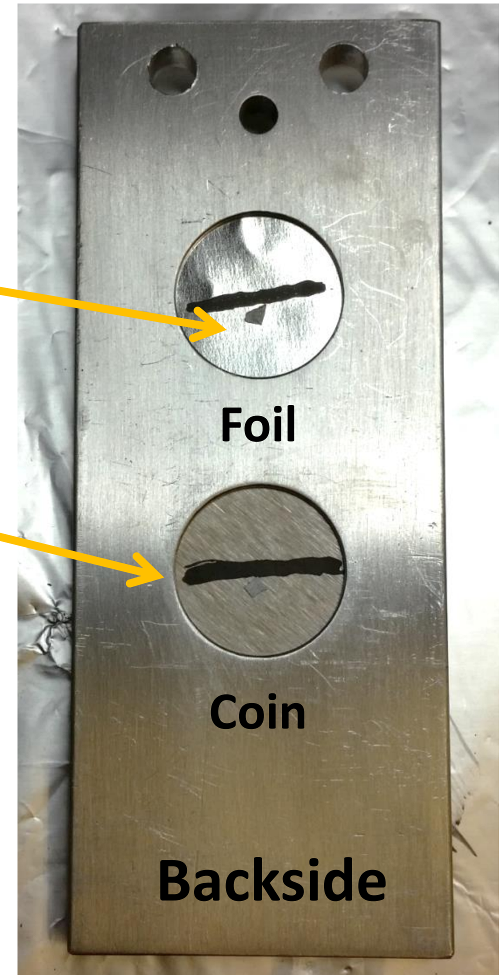
Copper rings fix the samples

Reflective tape for vibration measurements

Graphite used for temperature measurements

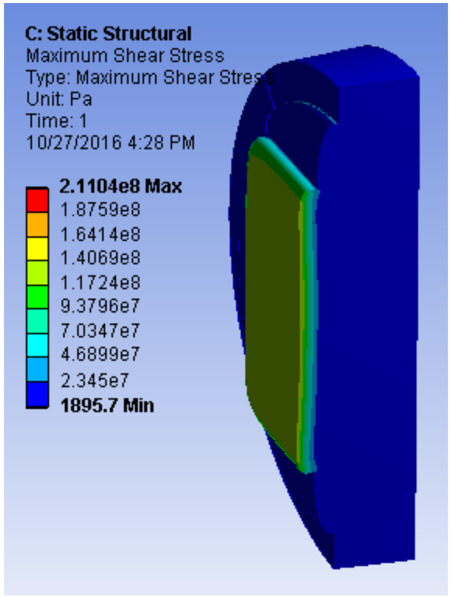
Luminescence target
Beam spot ~1cm^2

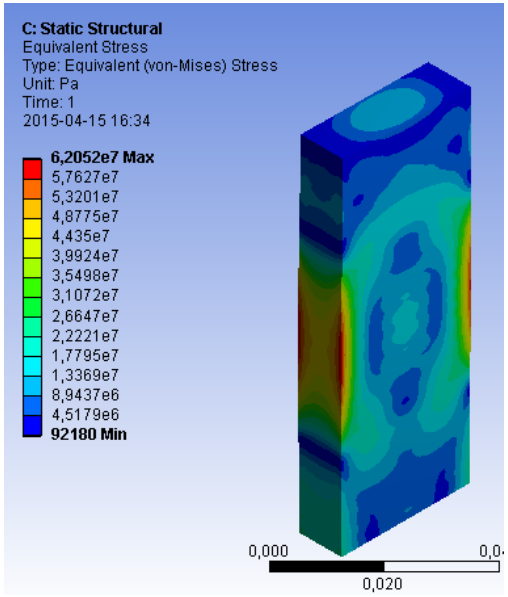


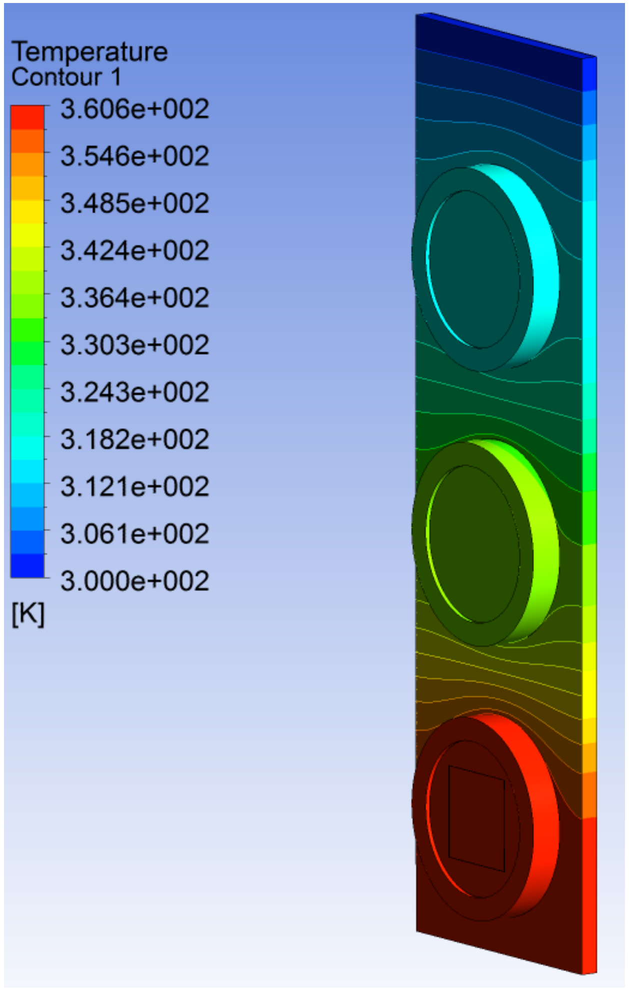


Calculations on uranium beam irradiation Assuming max. U-beam flux

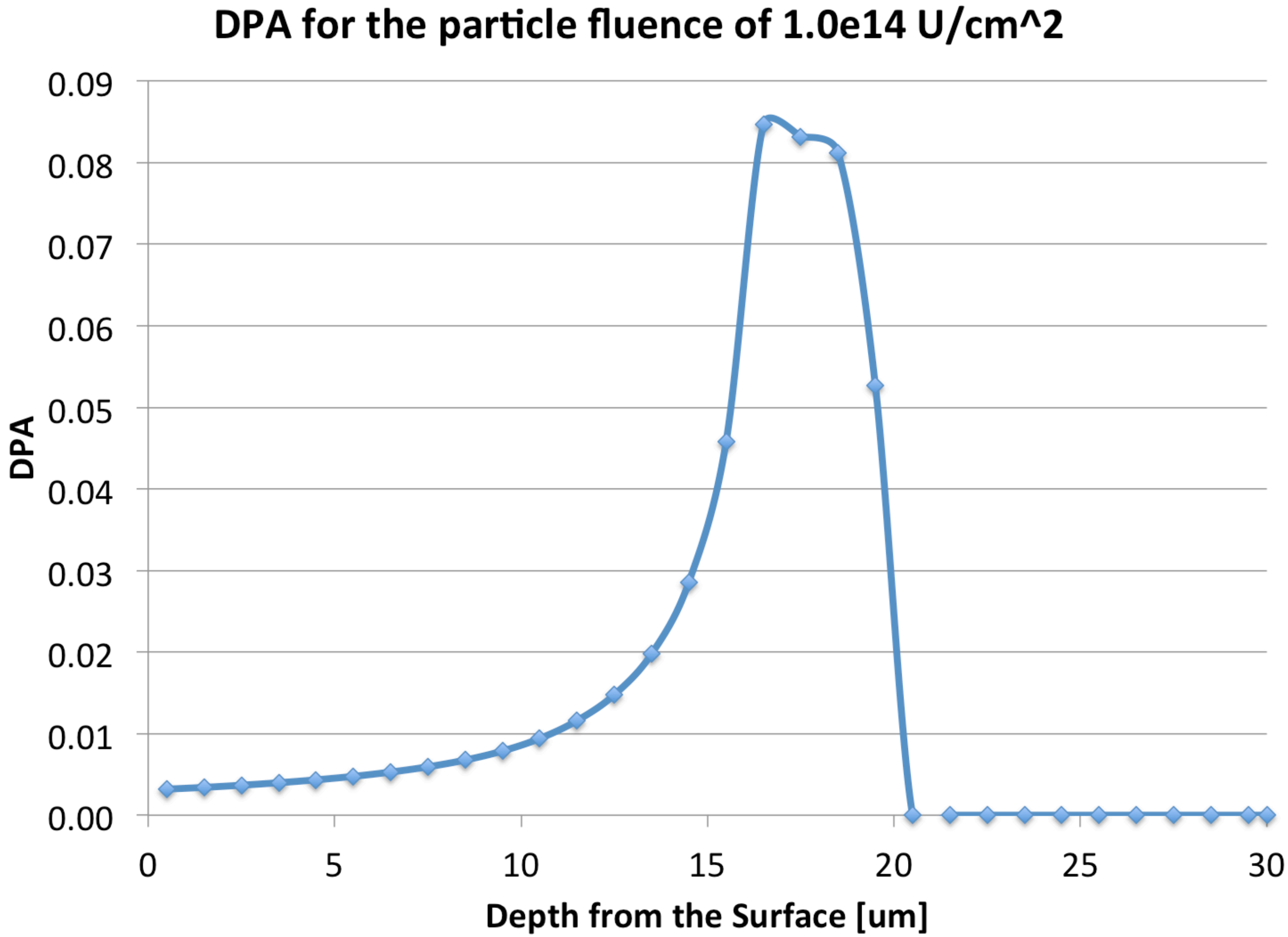
- Steady temperature: 87°C
- Temperature range during pulse: 113°C
- Max. post-pulse temperature: 200°C
  - ESS block: Max. 447°C
- Max. post-pulse shear stress on sample surface: 130 Mpa
  - ESS block: Max. 110 MPa von Mises stress





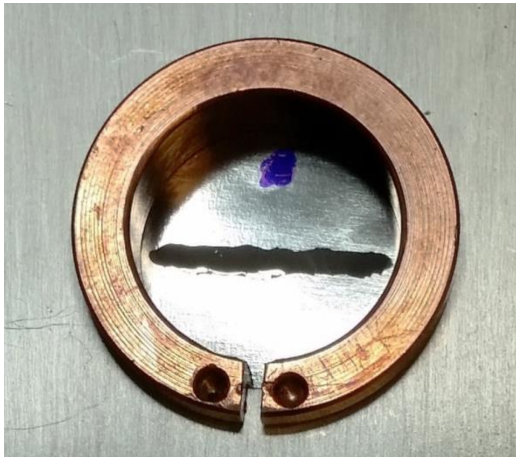


Damage map



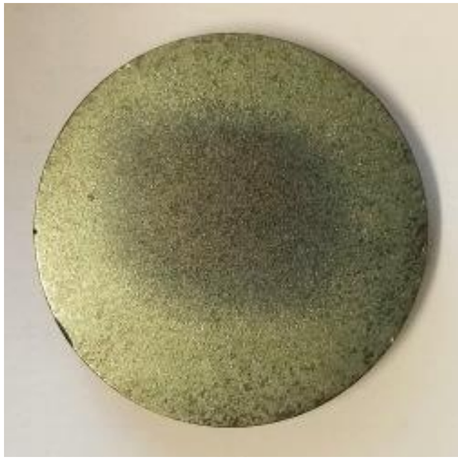
DPA for the particle fluence of 1.0e14 U/cm^2
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0.00
DPA
0
5
10
15
20
25
30
Depth from the Surface [um]

Samples for post irradiation examination



Foil 26 µm thick
Flux ~1e10 ions/s/cm^2
Fluence 1e14 i/cm^2

Coin 3 mm thick
Oxidized – 500°C, 2h in air
Clearly visible beam print
Flux ~1e10 ions/s/cm^2
Fluence 5e12 i/cm^2



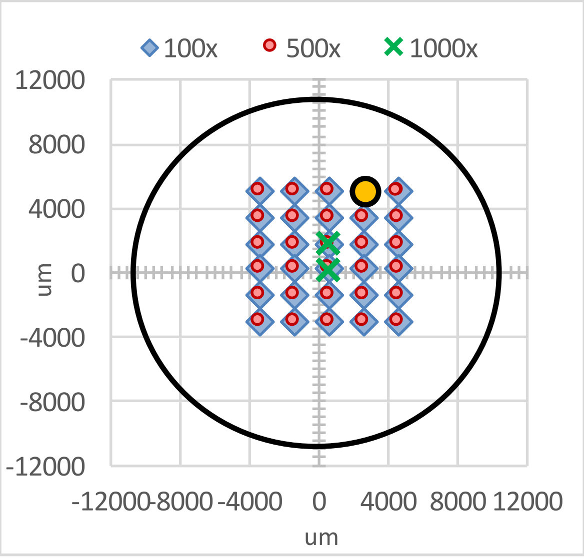
Coin 3 mm thick
Flux ~1e10 ions/s/cm^2
Fluence 1e14 i/cm^2

SEM images reveal damaged oxide layer

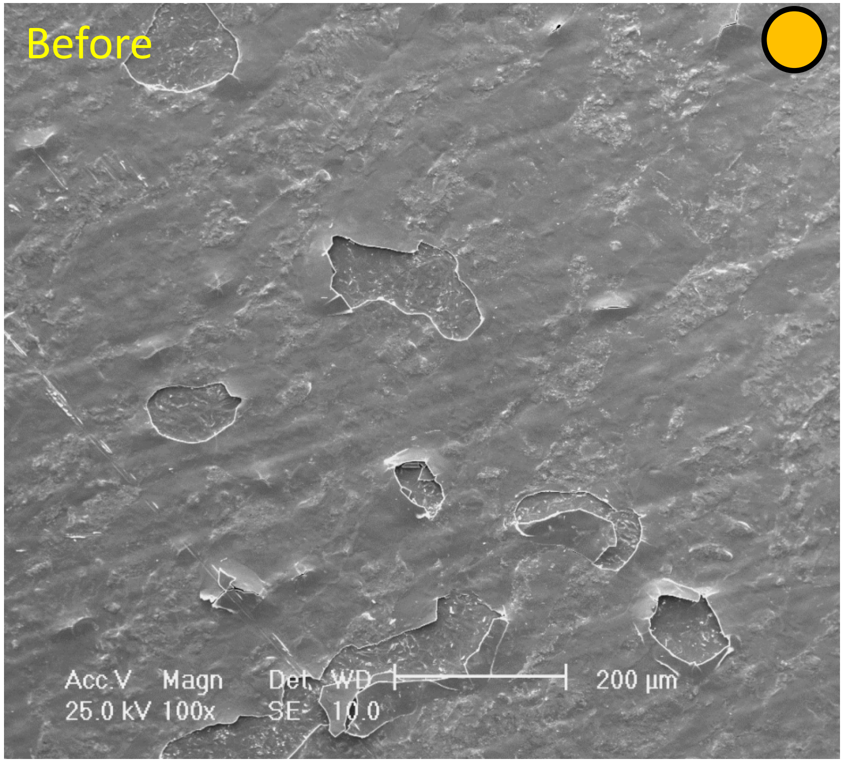
Oxidized 2h in air at 500°C

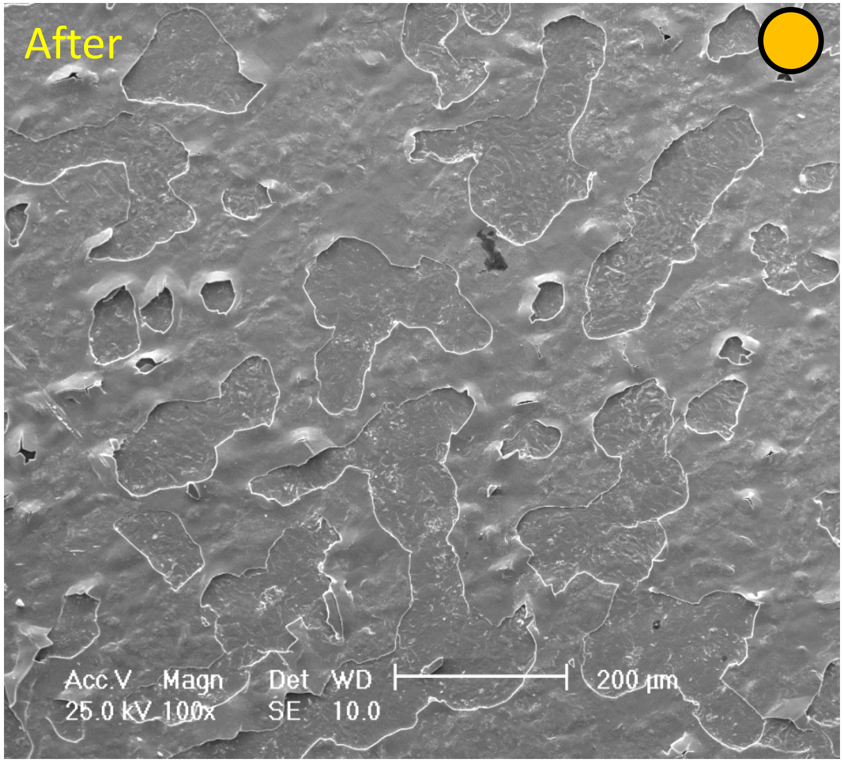
Before and after image (100x) of the same position, top right corner of irradiated zone

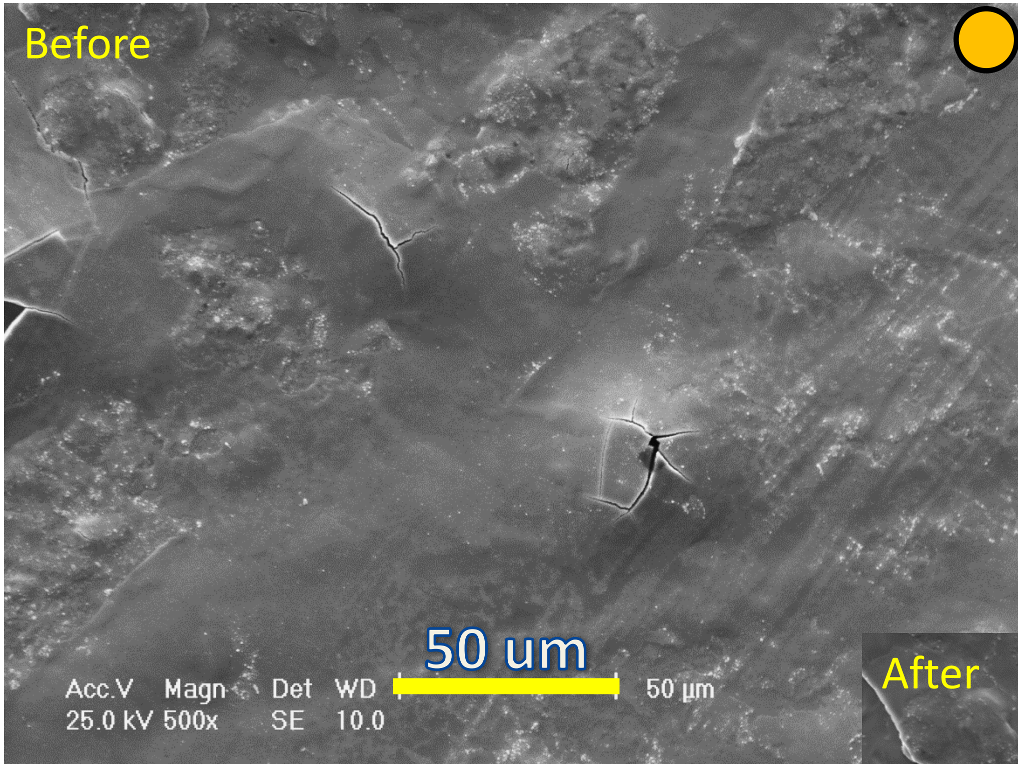
Images taken at 3 different magnifications, 30 positions

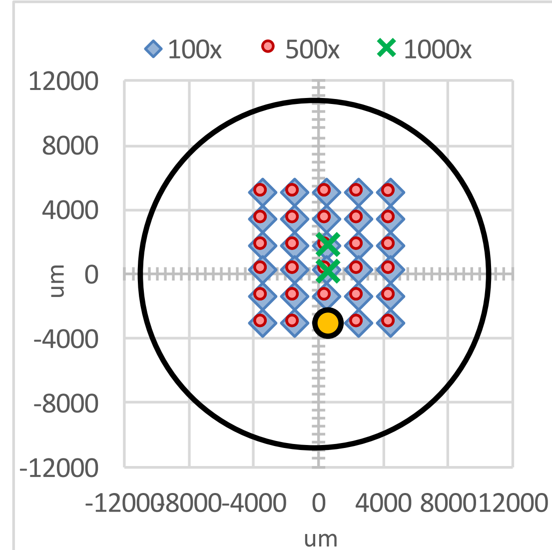


Map of SEM image positions





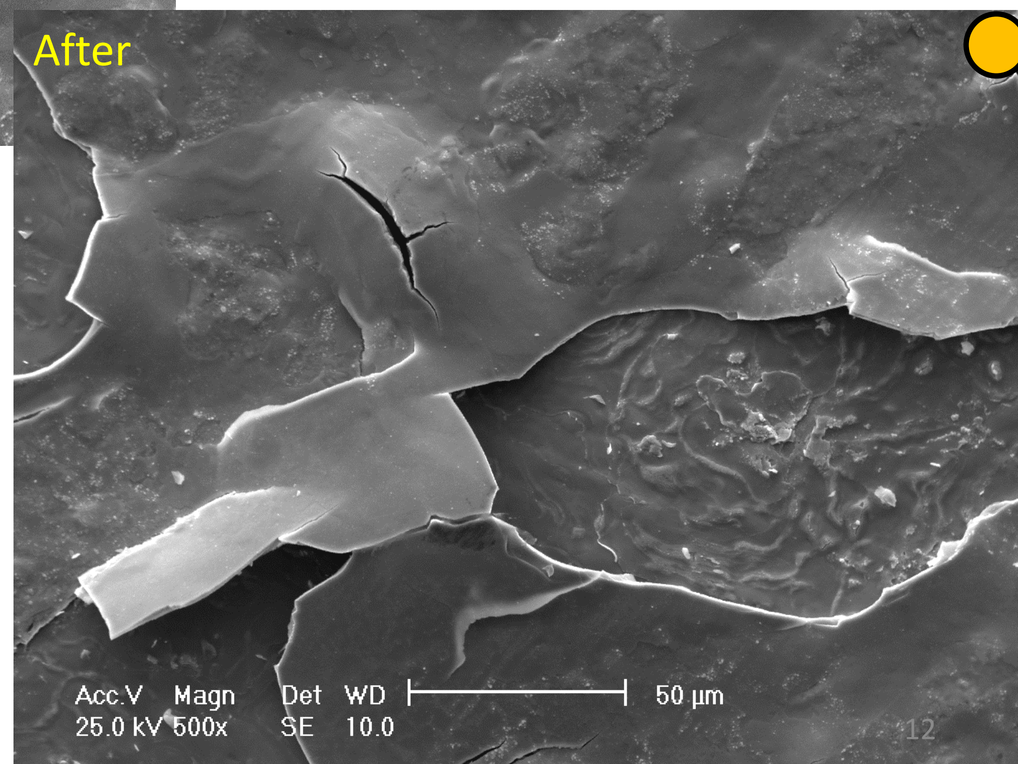




Map of SEM image positions

Higher mag. image of bottom/center position of same sample

Porous outer oxide scale clearly damaged

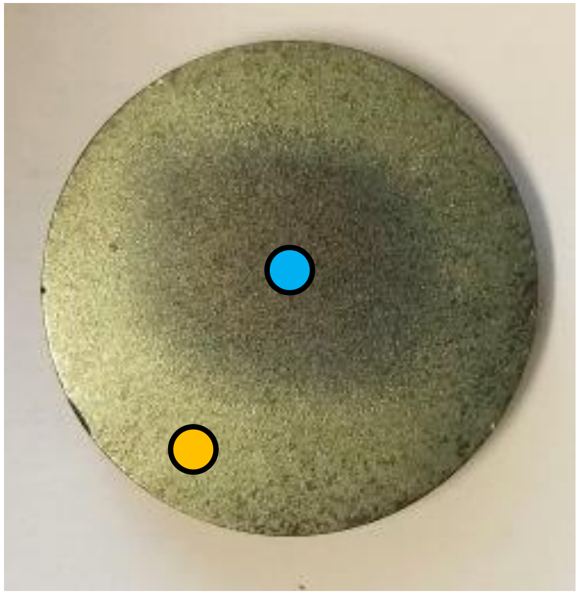


Auger Electron Spectroscopy (AES) at Dalarna University, Sweden

- Surface analysis of oxidized sample
- Depth profile of W and O determines oxide thickness
- 55 nm/min removed by argon ion sputtering

<u>Removed</u>

- (blue) 1870 nm
- (yellow) 3080 nm



+0.373 mg/cm^2 mass change due to oxidation

AES: non-irradiated area

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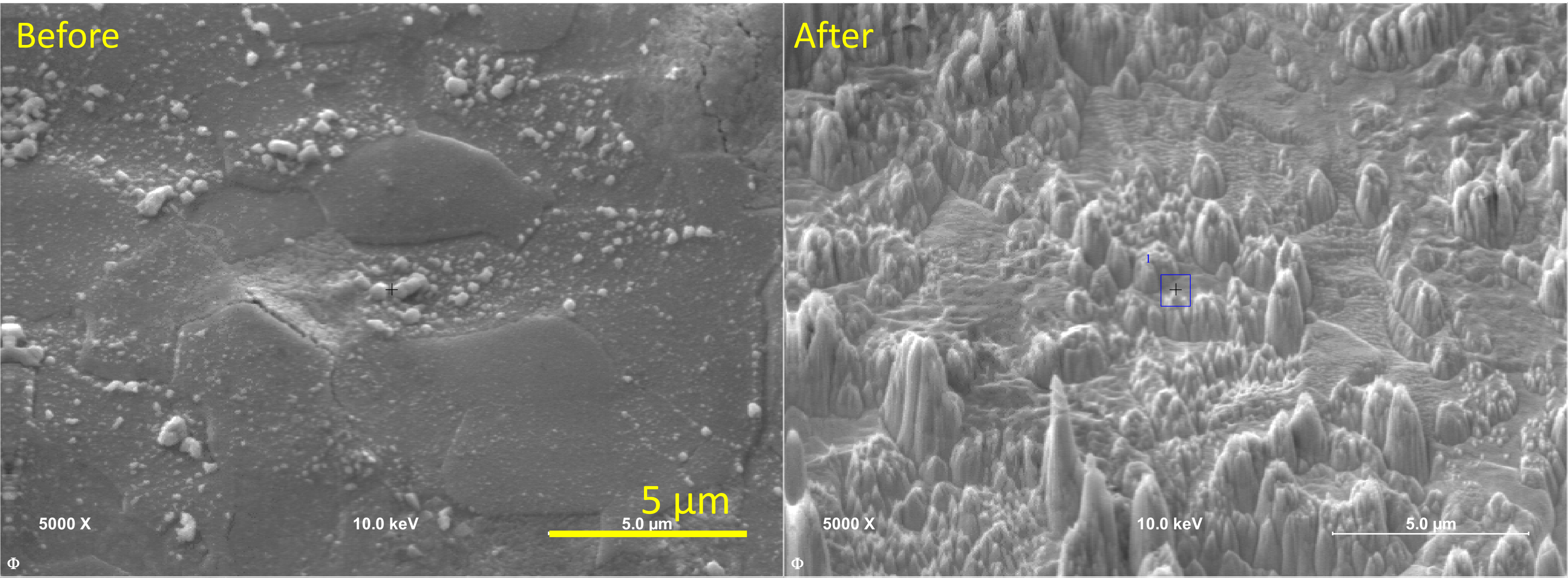
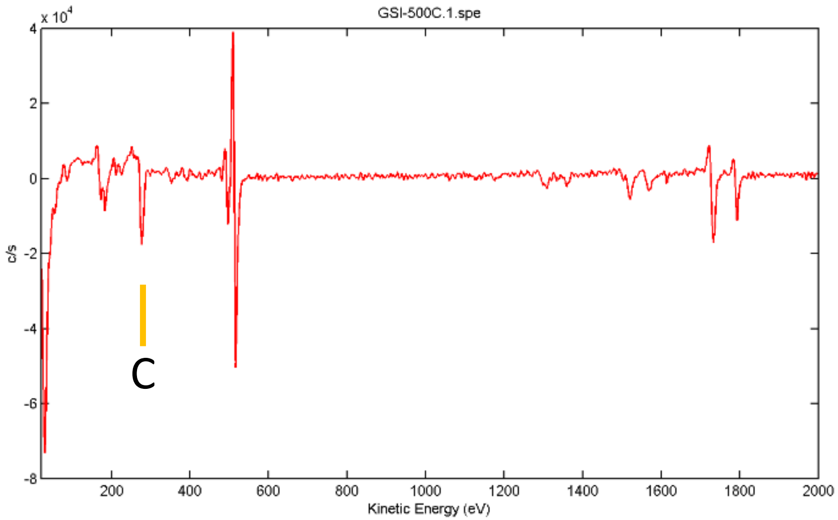
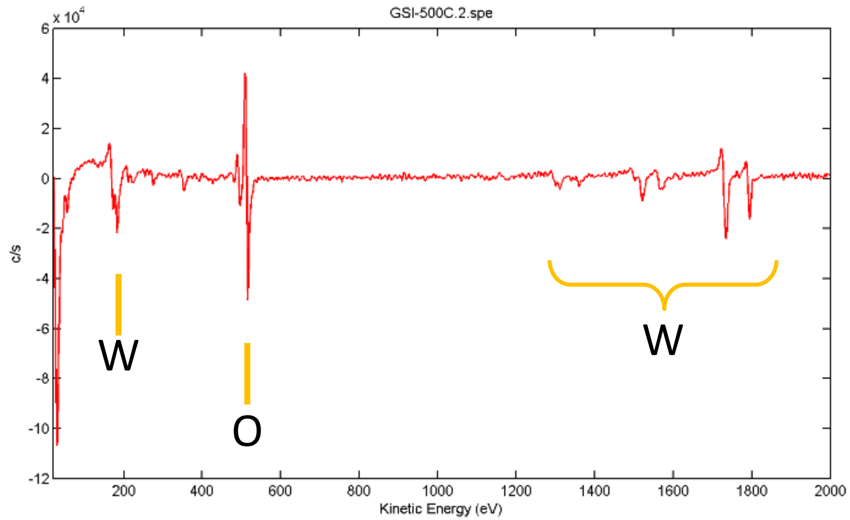


Outer non-irradiated area of oxidized coin
After 56 min of sputtering

Surface and depth analysis of non-irradiated oxidized area



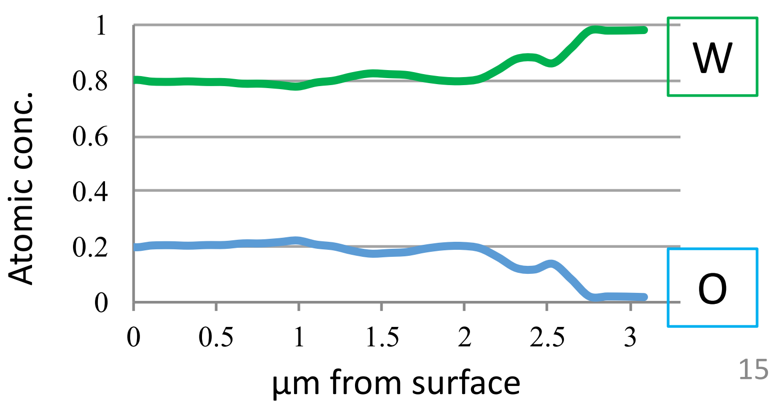
Before pre-sputtering



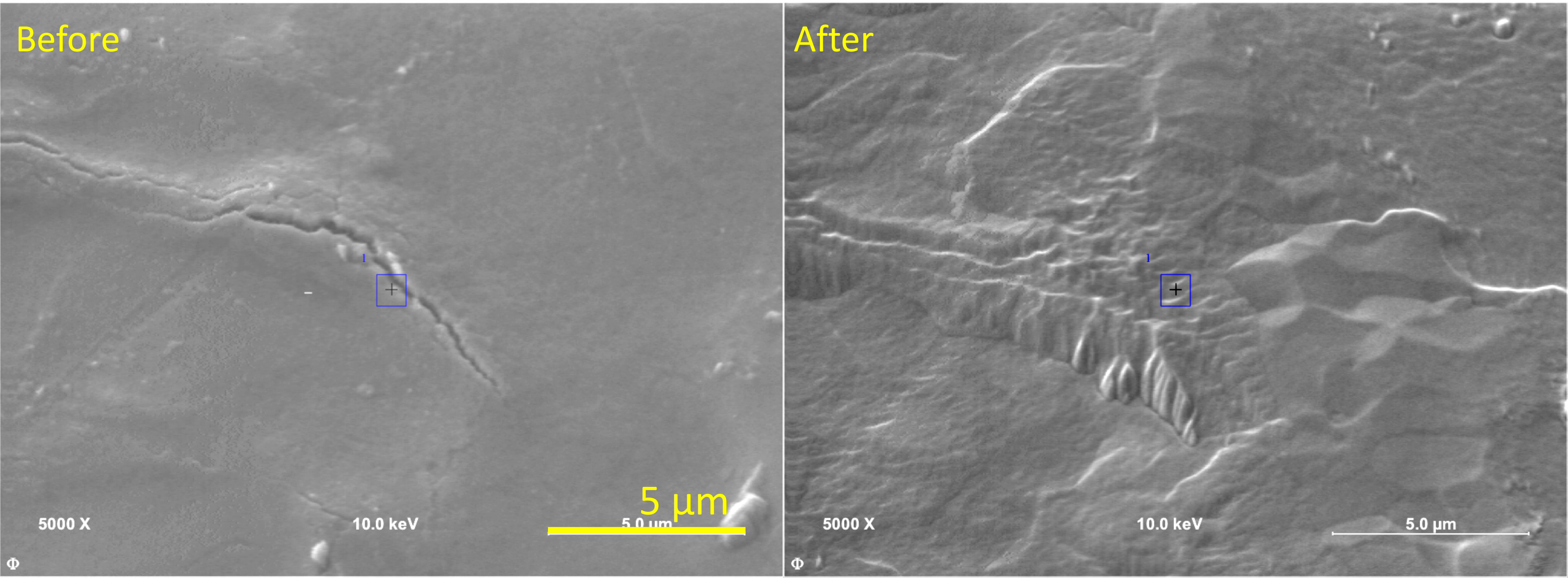
After pre-sputtering

- Oxide layer thickness: ~2.75 µm
- Pre-sputtering to remove thin carbon layer on surface

Depth analysis of O and W

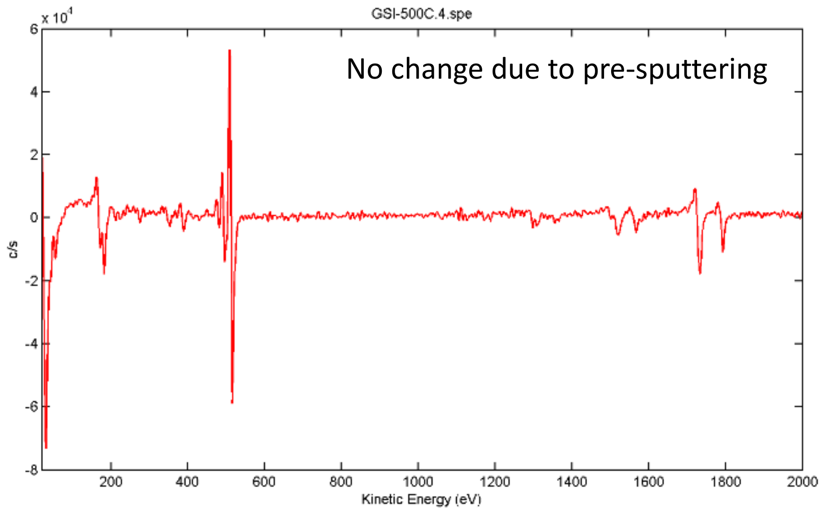


AES: irradiated area

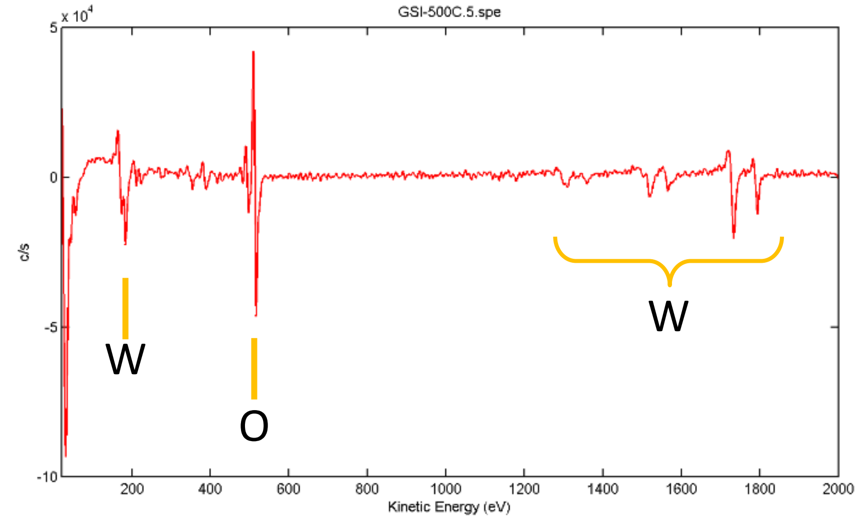


Irradiated area of oxidized sample, center
After 34 min of sputtering

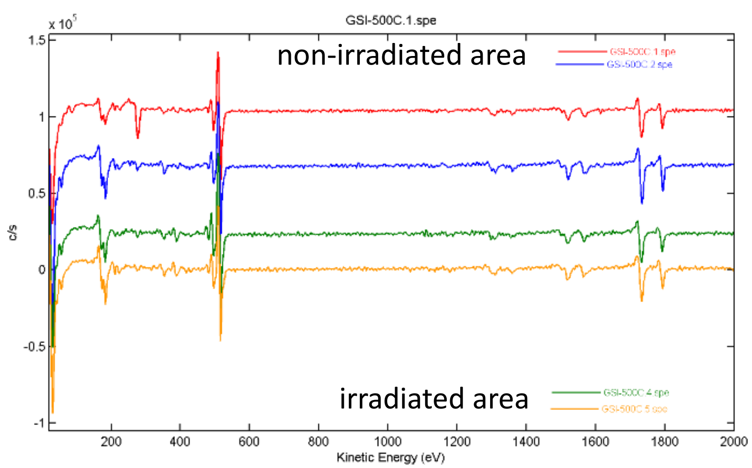
Surface and depth analysis of irradiated oxidized area

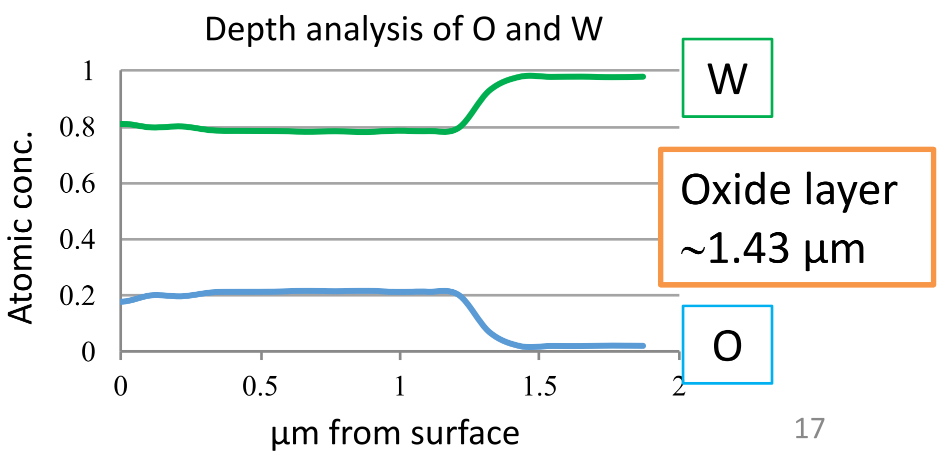


Before pre-sputtering



After pre-sputtering

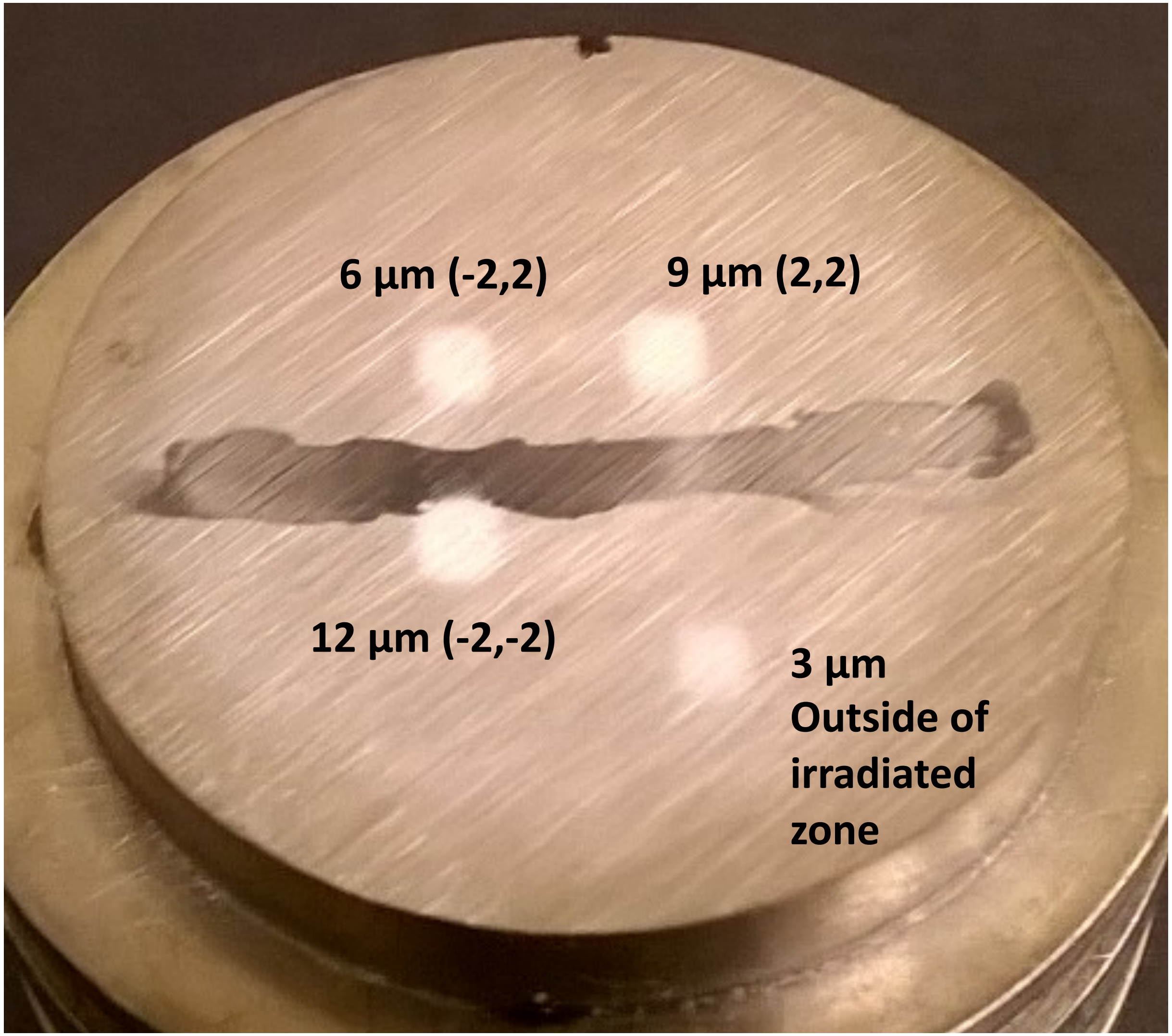




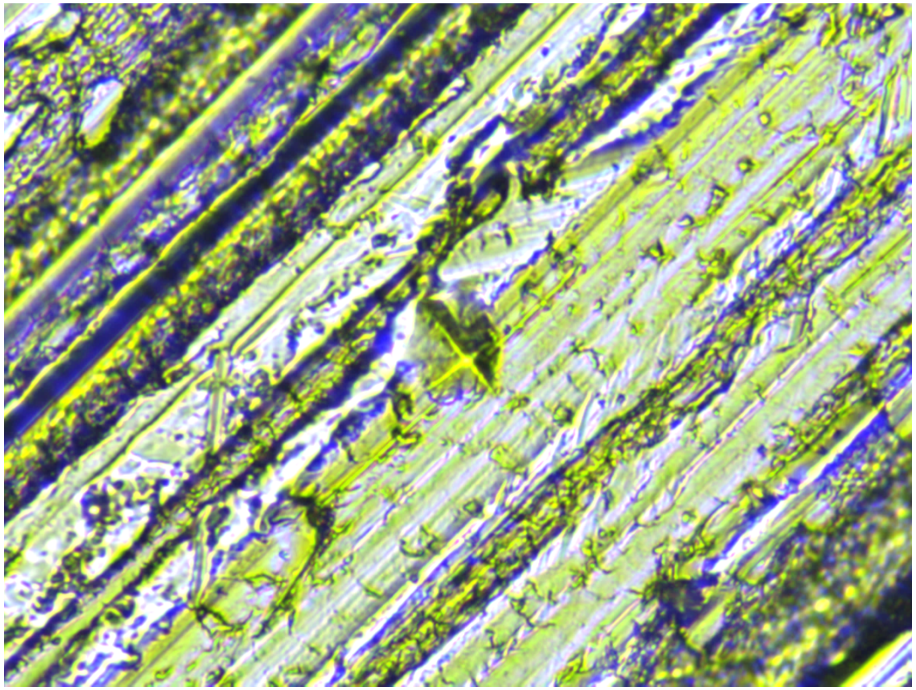
In-depth hardness of non-oxidized coin

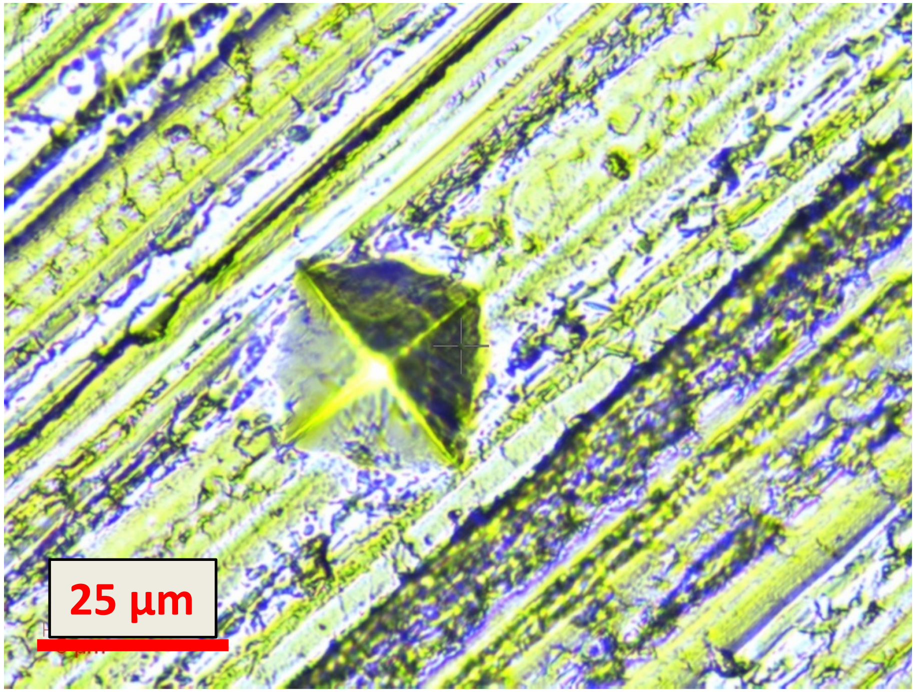
- CSM instruments Combi Micro/Nano Tester
  - Surface too rough for pure nano-indentaion
- Loads of 100-500g
- Comparing surface hardness of irradiated and non-irradiated zone
- Auger sputtering down to 6, 9, 12 µm for in-depth hardness

Map of indentations



100g vs 500g indent



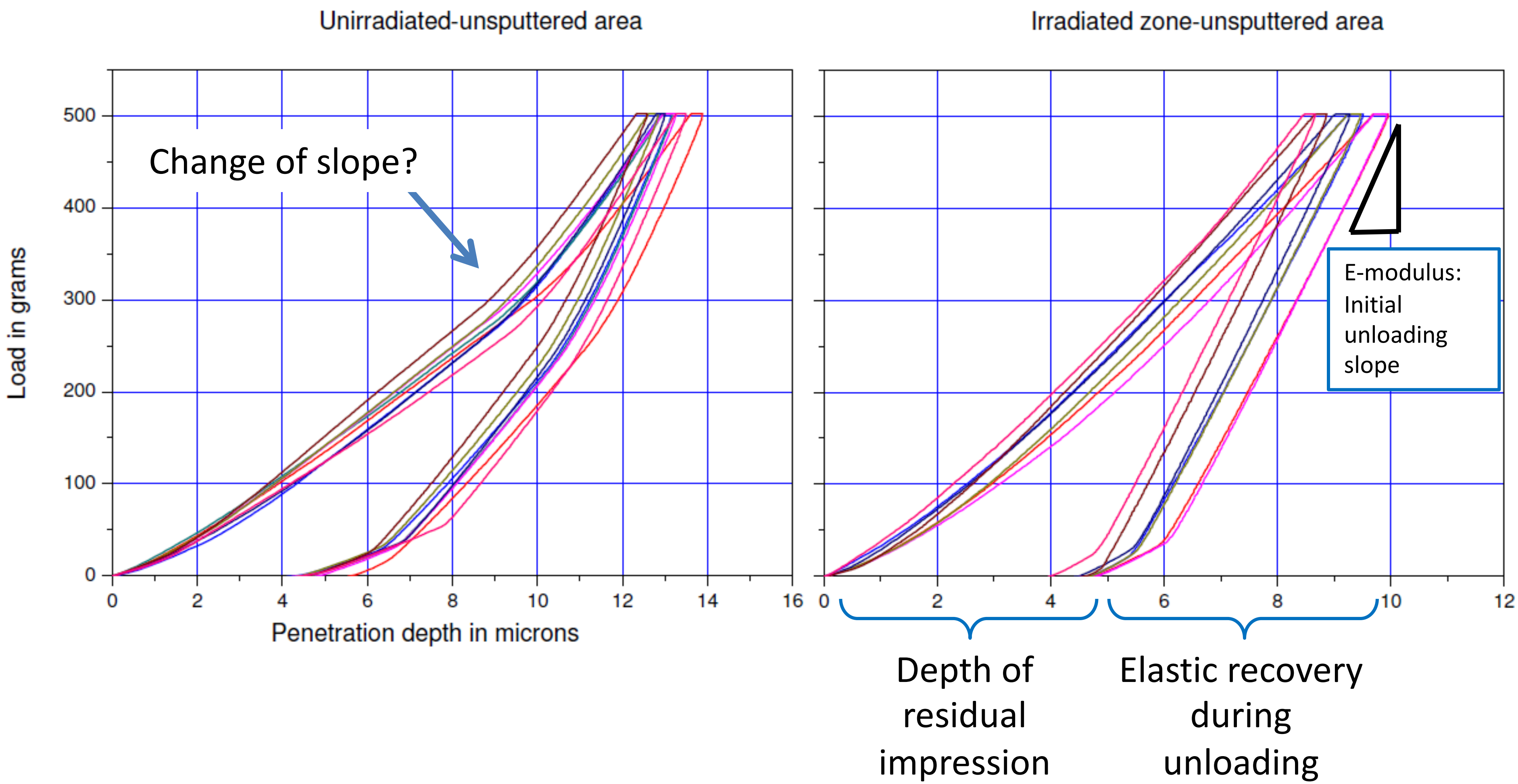


Final testing parameters

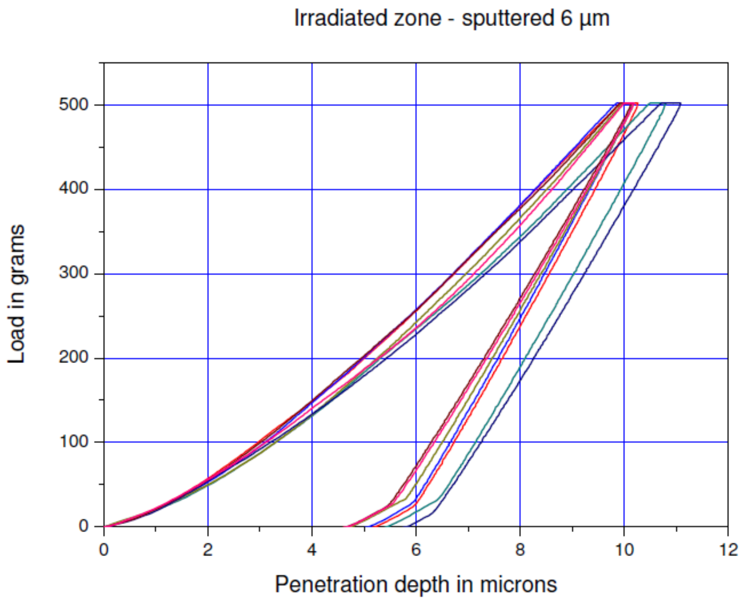
Indenter: diamond, Vickers
Max. load: 500g
Loading rate: 1000.00g/min
Unloading rate: 1000.00g/min

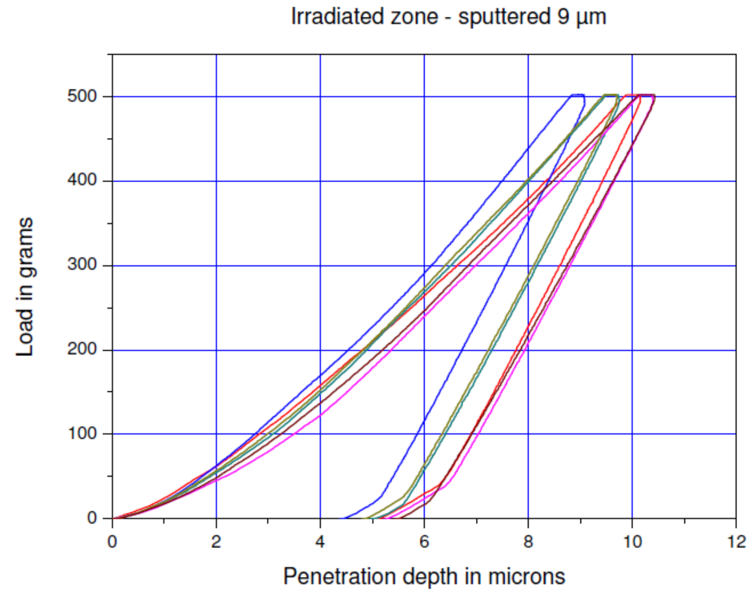
Hold time: 15.0s
Contact force: 3g
Approach speed: 500µm/min

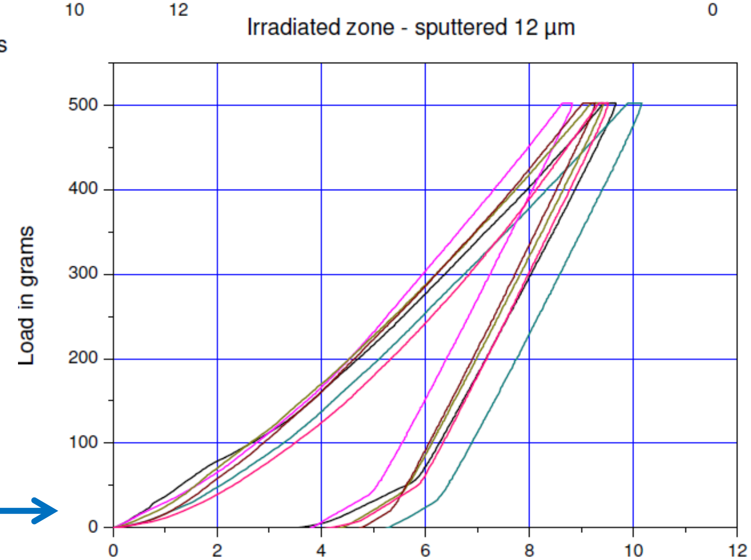
Penetration depth at surface



Penetration depth at sputtered points: 6, 9, 12 µm below surface



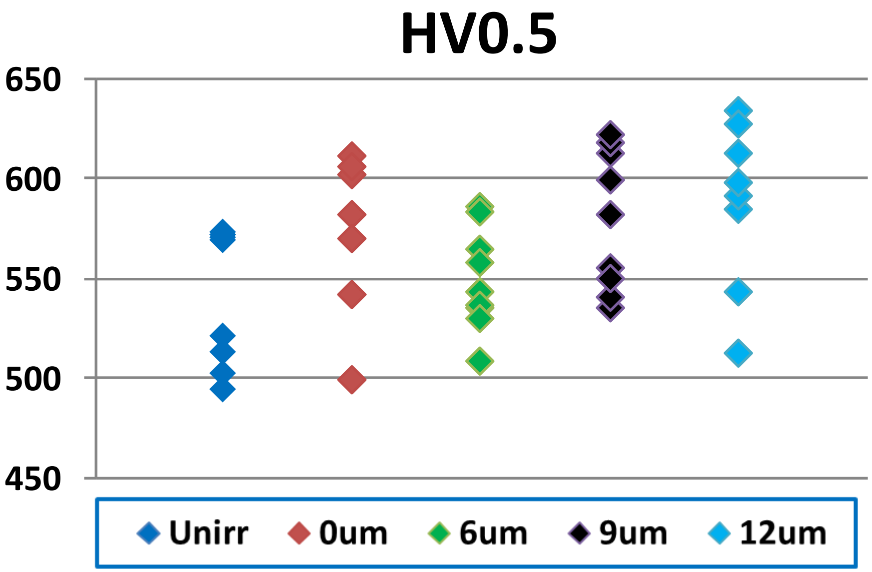


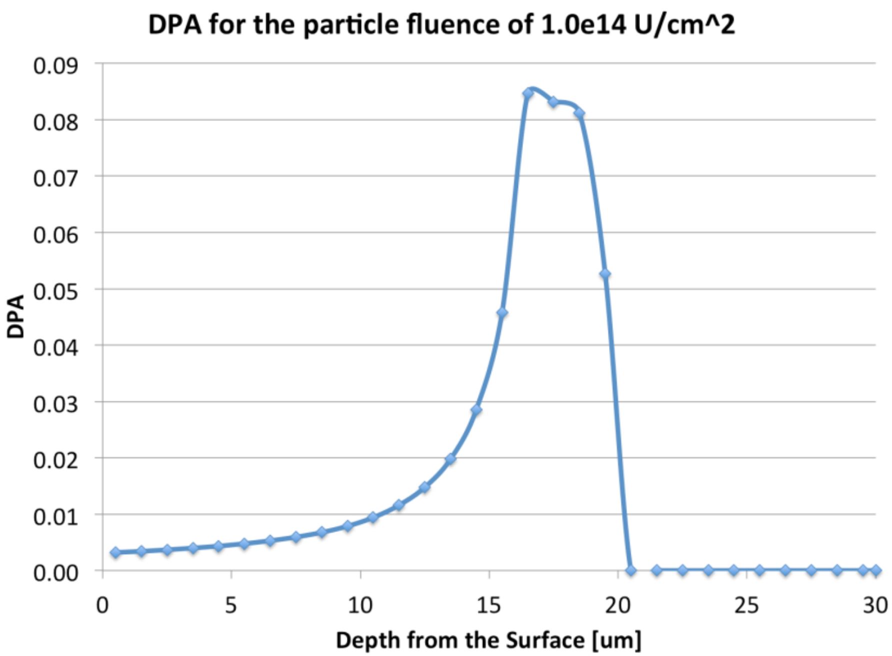


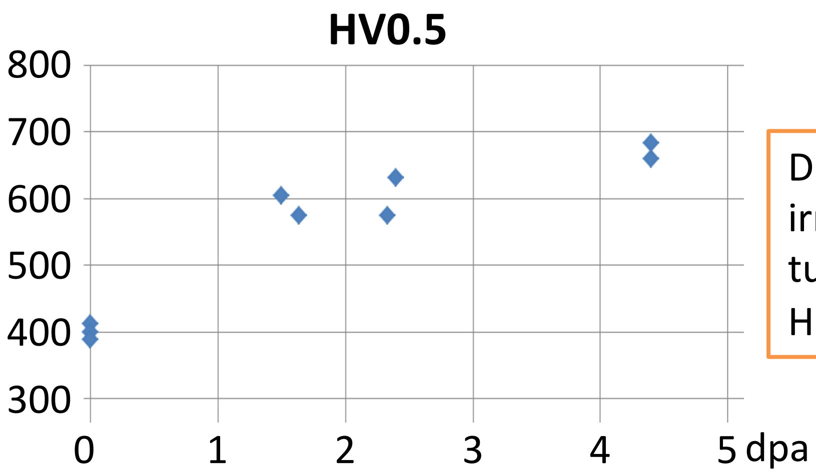
Indentation depth decreases from surface inwards

4 µm here is actually
12+4 µm below surface

Hardness increases with depth

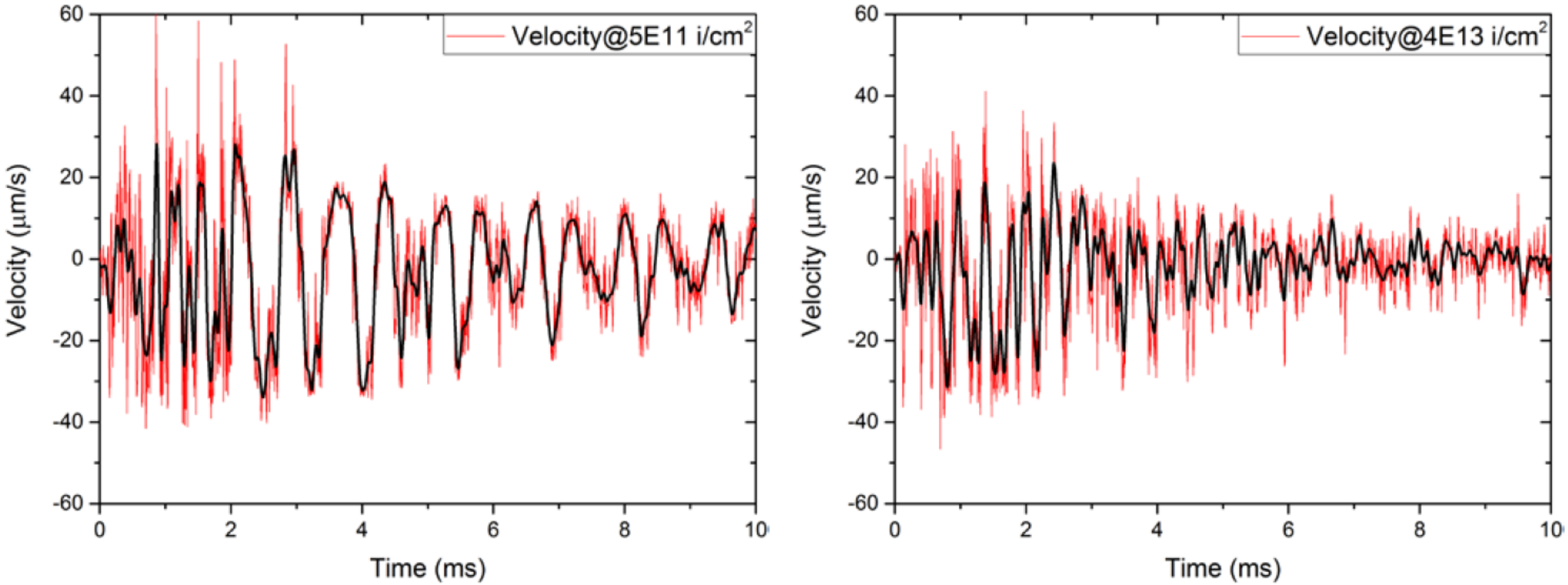






Data from proton irradiated STIP-V tungsten (Y. Dai , J. Habainy)

Vibrational analysis of foil at 1e10 U/(s·cm²) - Preliminary results



Surface velocity of W foil as a function of time at an accumulated fluence of $5e10^{11}$ U/cm² (left) and $4e10^{13}$ U/cm² (right)

Frequency of vibrations increased with irradiation – stiffening of irradiated area due to change of vibration mode?

By Pascal Simon, GSI

Response of High-Power Accelerator Materials to a short pulse 4.8 MeV/u Uranium beam, Pascal Simon, Advanced Research Lab summary report, January 2016, GSI Darmstadt.

Work under progress

- Correlation between radiation induced hardening and observed dynamic vibration mode
- Thermal and electrical resistivity measurements
- Nano-hardness on cross-section of coin
- Microstructural examination using TEM
- Surface analysis at 20 µm – can uranium ions be detected?

Summary

- Hardness tends to increase with depth, from 540 to 590 on average. 590HV0.5 corresponds to peak damage.
- Frequency of vibration increased with irradiation
- Beam induced dynamic stress caused damage to oxide, ~1.3µm lost
- Obtained stress with GSI beam comparable to ESS W stress
- Temperature during irradiation: base 40°C (87°C calc.), range 130°C (113°C calc.) Difference explained by graphite blackening and higher flux at times.
- Advantage of ion irradiation – sample can be collected immediately