Max-Planck-Institut
für Plasmaphysik

Sputtering of EUROFER: The Role of Roughness

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Content:

- Introduction:
- SIESTA
- Benchmarking of SDTrimSP-3D
- Sputtering of EUROFER: New results
- Summary

EUROfusion

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Outline

SIESTA: Our new high current ion source

SDTrimSP simulations for sputtering of EUROFER

Bechmarking of SDTrimSP-3D

Sputtering of EUROFER

What do we need to answer the question:

Can we use RAFM steels at some areas of the first wall of a future fusion power plant?

Certainly, steel is not an option for areas receiving a high power load and high particle flux.

And probably also not for areas receiving a non-negligible ion (plasma) flux.

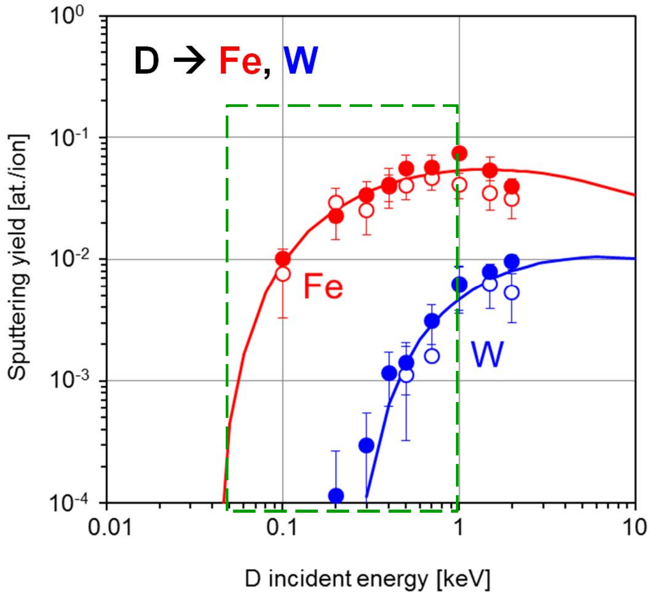
Why should we use RAFM steel at all?

- Blanket modules for the first wall blankets are made of RAFM steel
- Presently it is foreseen to clad BM with W
- Technologically it would be much easier and less expensive to use plain steel wall
- H retention in RAFM steels is low, even lower than in W

So what is the problem in using steel?

- Energy dependence of sputtering yield of Fe and W measured by weight loss & RBS
  (perpendicular ion incidence)
- Data fitted with Bohdansky formula
- Fe has lower sputter threshold and higher yield
- In relevant E region (50 to 1000 eV)
  $Y_{Fe} > 10 * Y_W$

→ **Fe (steel) not useable as PFM**



Sputtering yields of Fe and W due to D bombardment as a function of D energy.
- Open circle: determined by weight-loss measurement,
- Closed circle: determined by RBS (Rutherford Backscattering Spectrometry).
- The curve is derived from the fitting by Bohdansky formula.

Data from: K. Sugiyama et al., Nucl. Mater. Energy 8, (2016) 1

Sputtering of pure Fe (the main component of steel) **is too high!**

But: steel is not pure Fe

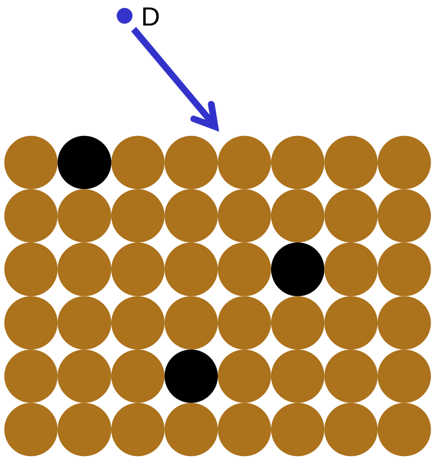
RAFM steels (EUROFER, RUSFER, F82H)
contain small amounts (0.3 to 1.0 at.%) of W

Sputter yield of W, Y_W, is much lower than Y_{Fe}
→ W enrichment / Fe depletion at the surface

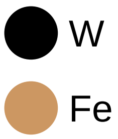
This phenomenon is called "***preferential sputtering***"

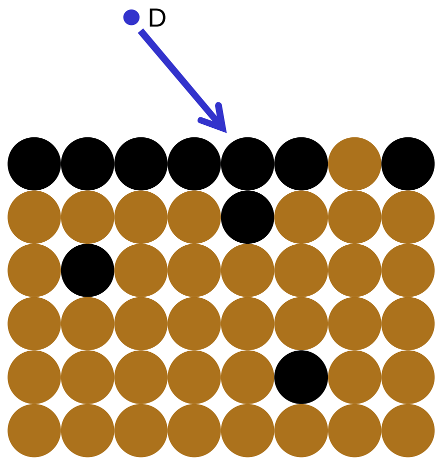
Preferential sputtering will lead to a continuous change of the sputtering behavior

Preferential Sputtering



Homogeneously distributed
W in Fe matrix





In steady state (after substantial erosion):
W enriched at the surface

Outline

SIESTA: Our new high current ion source

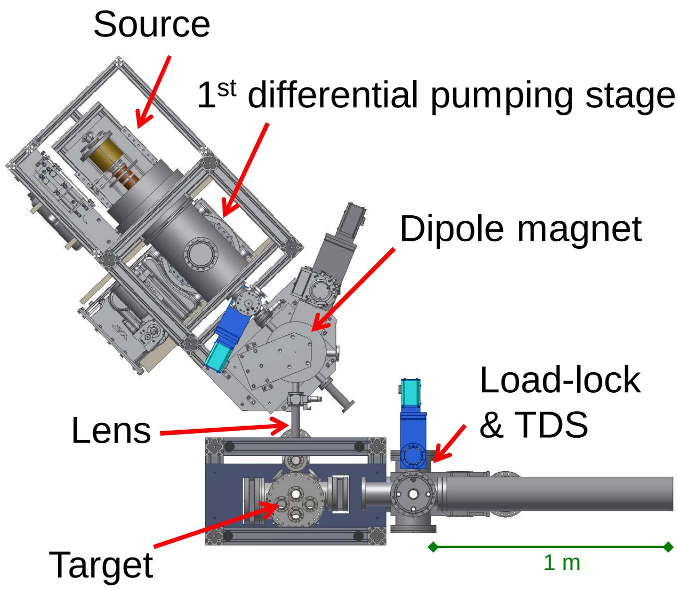
SDTrimSP simulations for sputtering of EUROFER

Temperature dependence: Diffusion of Fe in W and vice versa

Projects within EUROfusion in WP PFC

SIESTA (Second Ion Experiment for Sputtering and TDS Analysis):
a High Current Ion Source for Sputter Yield Measurements

Overview of SIESTA



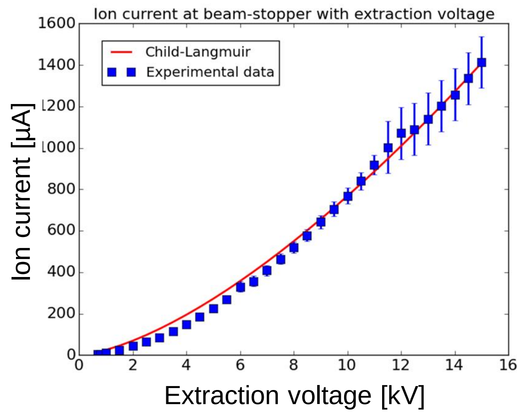
CAD Birds-eye view of SIESTA

- Ion beam extracted from source
- Neutral gas pumped out in differential pumping stages
- Dipole magnet deflects beam → mass-selected ion beam
- Optional ion lens focuses the beam
- Beam impinges on target sample, which can be rotated, heated and weighed in-situ with magnetic suspension balance.
- TDS can be performed in-vacuum

Status of the experimental setup

- High ion fluxes at low impinging ion energies are obtained by positively biasing the target up to +6 kV
- Sample temperatures of 1300 K have been achieved via electron impact heating on the sample backside
- An improved temperature measurement system has been installed, increasing reliability
- Ion fluxes to the target of ~4×10^{19} D/m²/s have been measured

SIESTA is in operation since Q2 2017!

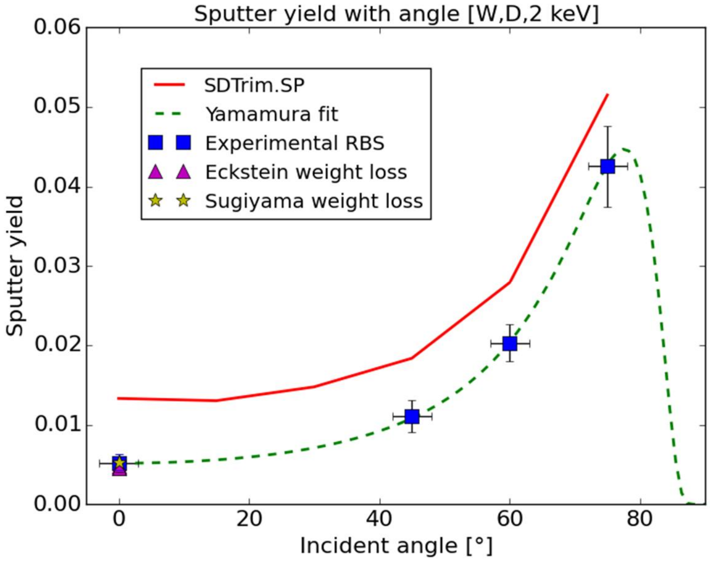


a-C:H sample heated to 950 K by electron impact heating

Angular dependence of sputter yield for W

Controlled roughness samples

- Results agree with experimental data at 0° incidence and can be fitted well with Yamamura’s formula
- Mismatch of D on W with SDTrimSP using standard parameters is a known issue*
  (SDtrimSP value is a factor of 2 higher than experimental data)



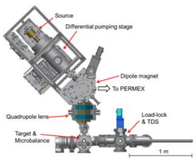
*

*Behrisch-Eckstein Sputtering by Particle Bombardment, Topics of Applied Physics, **Vol 110***

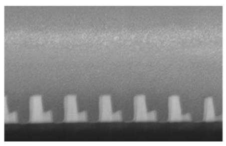
*K. Sugiyama et al., „Sputtering of iron, chromium and tungsten by energetic deuterium ion bombardment” Nuclear Materials and Energy **8**, 1–7 (2016).*

Summary

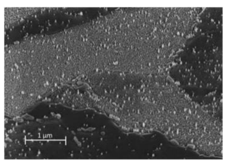
- SIESTA good for: →
  - Well-characterized erosion studies
  - In-vacuum retention studies



- SIESTA capabilities: →
  - Flux density of several 10^{19} at/m²/s
  - H, D, He, Ne, Ar ions
  - Energies of 200 eV to 10 keV
  - In situ weight measurement
  - In-vacuum TDS



- Used in this work for: →
  - Influence of roughness on sputtering
  - Sputtering behaviour of EUROFER under D ion bombardment



The dynamic surface evolution due to preferential sputtering can be simulated by SDTrimSP

- SDTrimSP: dynamic version of TRIM.SP [1] (an earlier version was called TRIDYN [2])
- TRIM.SP describes the sputtering of surfaces due to impact of energetic species in the binary collision approximation
- TRIM.SP is well established and benchmarked with numerous experimental results
- SDTrimSP takes into account dynamic changes at the surface during sputtering, for example those due to preferential sputtering [3] *(SDTrimSP fka TRIDYN)*
- Important for extrapolation to conditions not (easily) accessible to experiments (e.g. sputtering by tritium)

[1] W. Eckstein, Springer Series in Materials Science, Springer, Berlin, 1991
[2] W. Möller, W. Eckstein, J. P. Biersack, Comput. Phys. Comm. 51 (1988) 355
[3] Mutzke et al., IPP Report #12/8 “SDTrimSP, Version 5.00“, 2011

Preferential sputtering

- Leads to enrichment of one component (transient phase until steady state)
- Reduces total sputter yield
- Effect increases with difference of sputter yield of the 2 components
- Occurs for all energies, but is strongest in the region between the 2 threshold energies

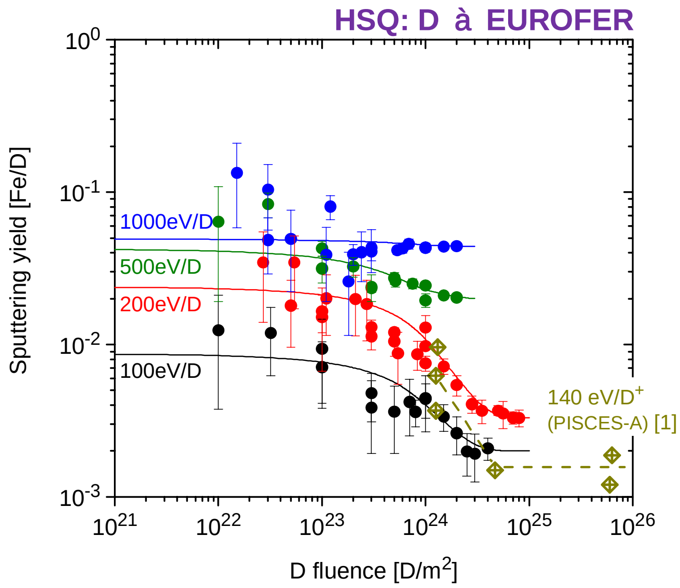
SDTrimSP can simulate the dynamic surface evolution due to preferential sputtering

Erosion of EUROFER (data from 2015)

- Yield reduction in the higher fluence range (≥ 10^{23} D/m^2), as well as for Fe/W layer.

- For 200 eV/D steady state seems to be reached for fluence > ~ 5 x 10^{24} D/m^2.

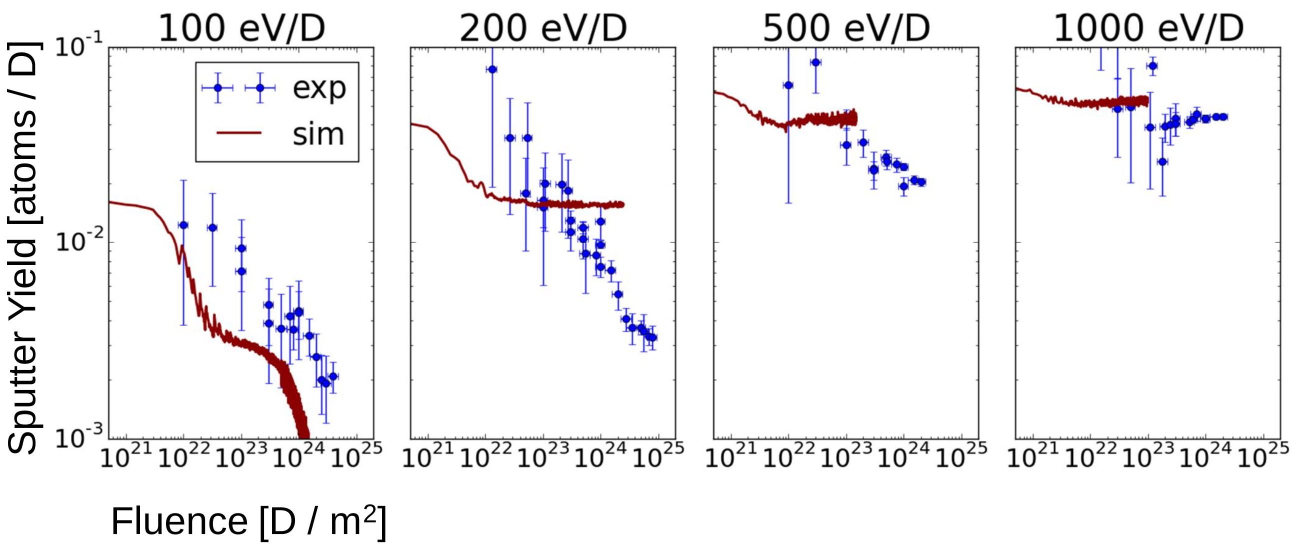
- PISCES-A data[1] at very high fluence and 140 eV/D also indicate steady state for fluence > ~ 5 x 10^{24} D/m^2.



Sputtering yield of EUROFER steel by D ion irradiation with different D energies as a function of D fluence (320 K)

[1] J. Roth et al., J. Nucl. Mater. **454** (2014) 1

Comparison experiment vs SDTrimSP

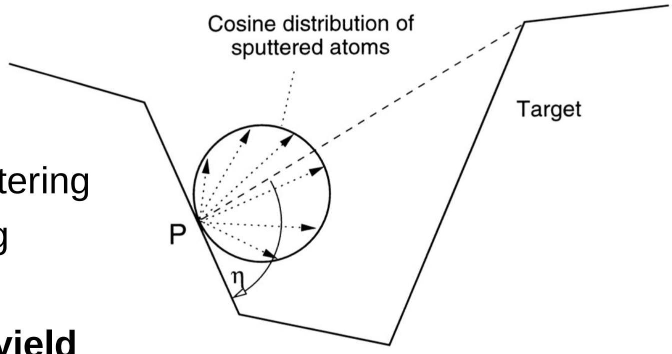


à Experimental sputter yield reduction for lower energies not reproduced

à Possible reasons: W surface binding energy? Roughness?

Influence of roughness on erosion

Surface roughness has a significant influence on the net sputter yield:



- o Oblique angles à increased sputtering
- o Re-deposition à lower sputtering

Due to surface roughness, **sputter yield can vary by a factor of 2 or more**

M. Küstner et al., "The influence of surface roughness on the angular dependence of the sputter yield", Nucl. Instrum. Methods Phys. Res., Sect. B 145 (1998)

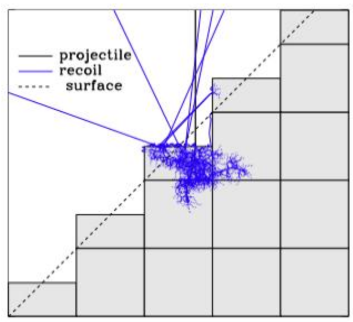
Up to now, most simulations of the sputter yield ignored roughness
à further study is required

SDTrimSP-3D has been developed to simulate transport of ions in 3-D targets

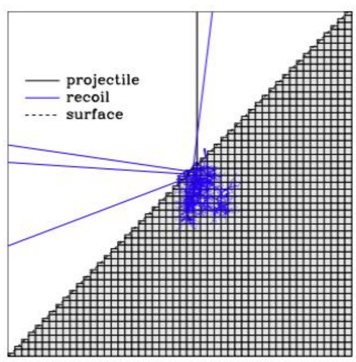
SDTrimSP 2D & 3D

SDTrimSP has been expanded to include 2D / 3D targets (essentially, parallelized SDTrimSP)

Instead of layers, now we have 3D cubes



Important:
Requires small enough cell resolution (nm)
Drawback:
Computation time (scales with N_y x N_z)



1D 5 keV Ar à Si
50×10^{20} Ar/m²
dx = 2.5 nm
Processors: 32
Time: **15 min**

3D 5 keV Ar à Si
50×10^{20} Ar/m²
dx = dy = dx = 2.5 nm
100 x 100 x 200 nm
Processors: 32
calculation-time: **52 h**

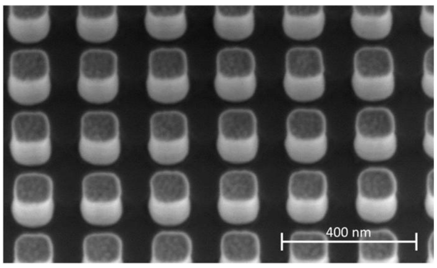
Benchmarking of SDTrimSP-3D

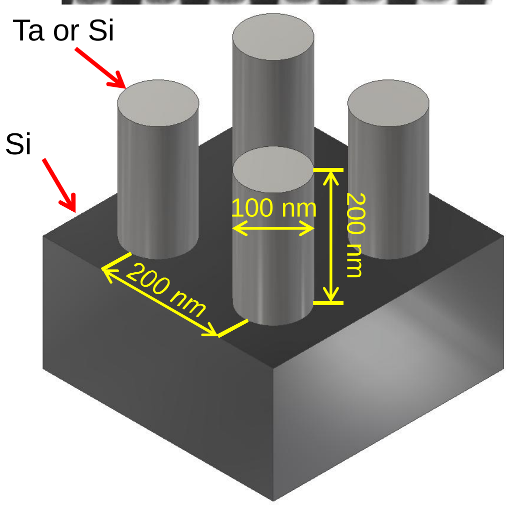
- Use of a well-defined structured sample
- Erosion with a well-defined, monoenergetic, mass-selected ion beam (5 keV Ar) under well-defined conditions in SIESTA
  (variation of angle of incidence and applied ion fluence)
- Comparison of resulting surface morphologies with model predictions

nm-sized columnar structures eroded with Ar under varying angles of incidence:

- 1x Ta used as W surrogate
- 3x Si to benchmark the code with previous versions

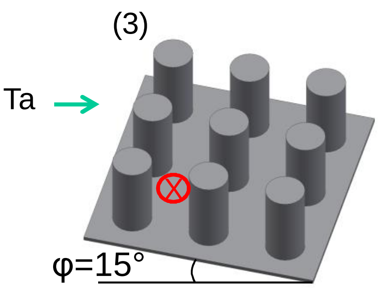
Eroded sample morphology studied under SEM & FIB cut and compared to SDTrimSP-3D





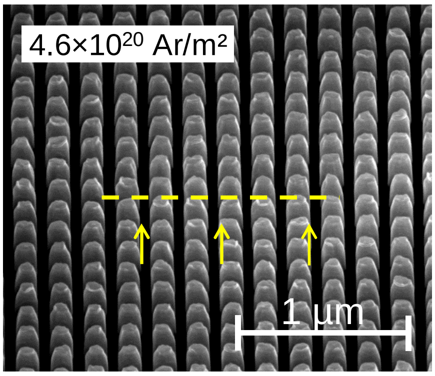
(1) Θ=0°,φ=0°
(2) Θ=45°,φ=0°
(3) Θ=45°,φ=15°

⊗= view of ion beam

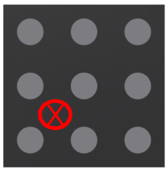


Si columns – normal incidence

SDTrimSP-3D Si columns eroded by 5 keV Ar under 0° incidence

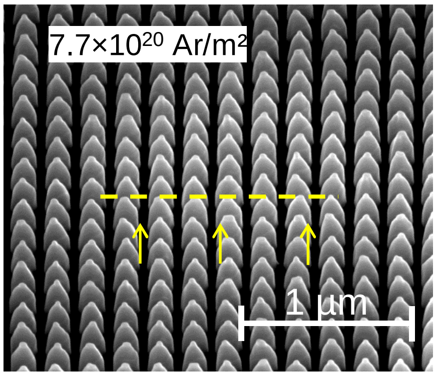


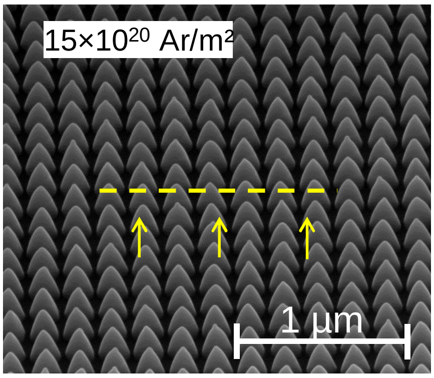
(1)

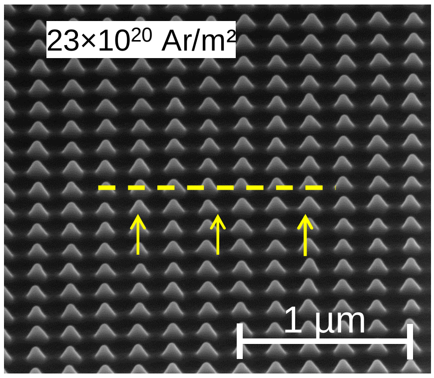


Ar beam is not homogeneous

Various fluences on same sample

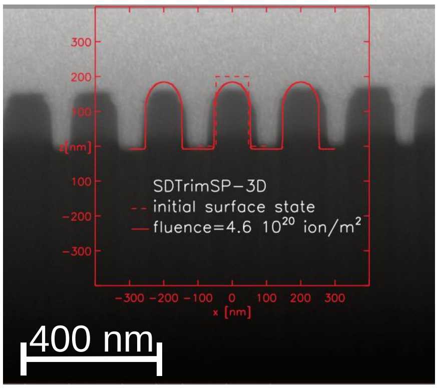




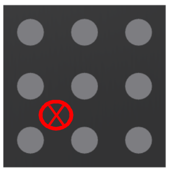


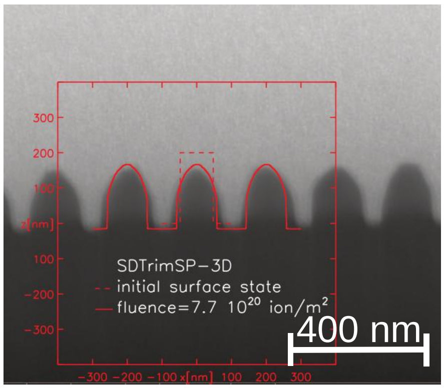
Si columns – normal incidence

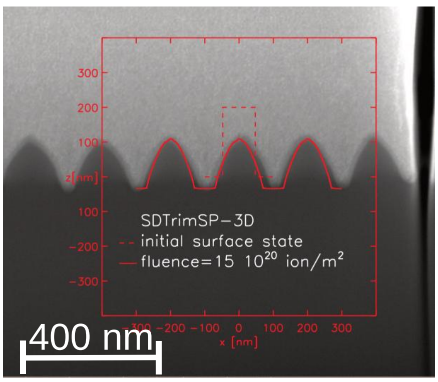
SDTrimSP-3D Si columns eroded by 5 keV Ar under 0° incidence

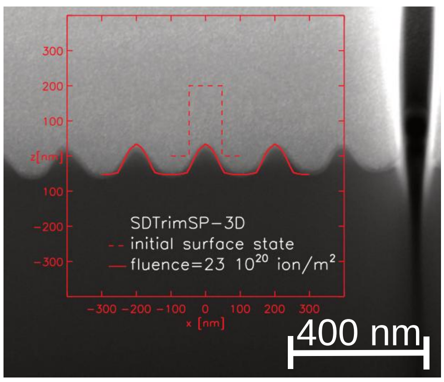


(1)



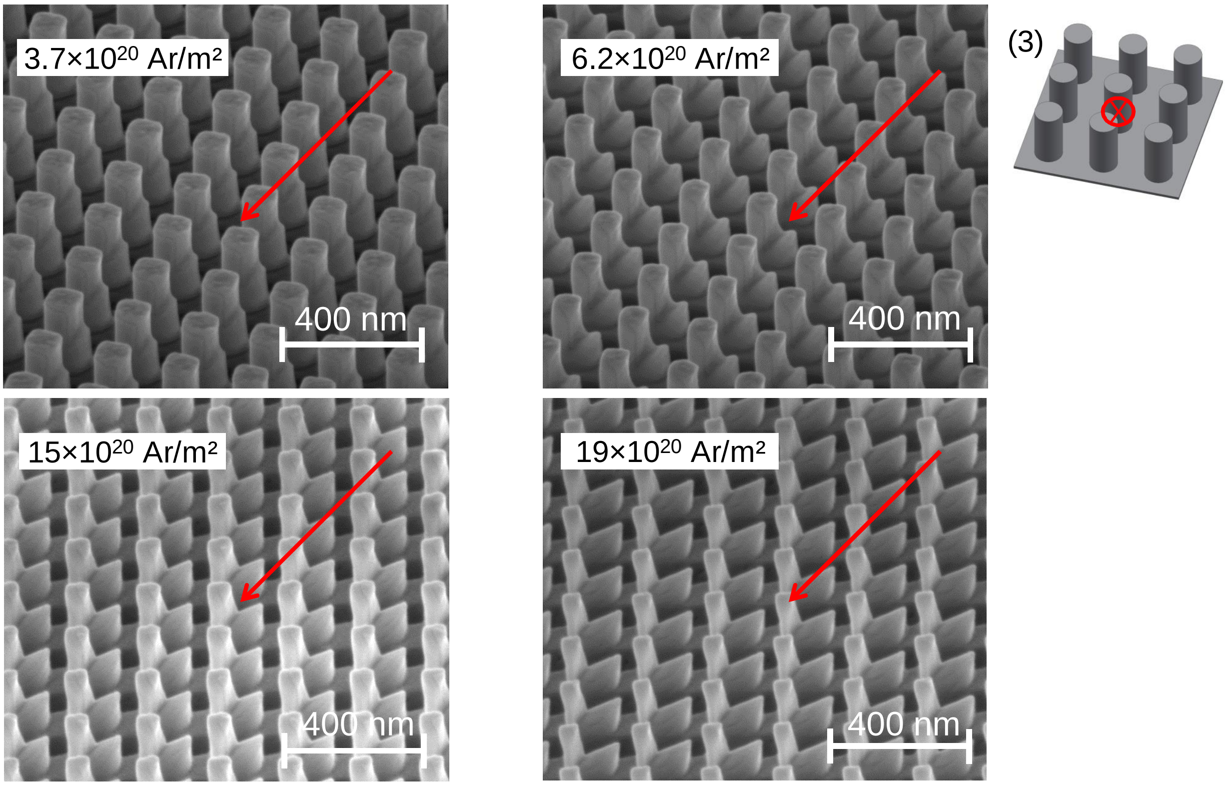






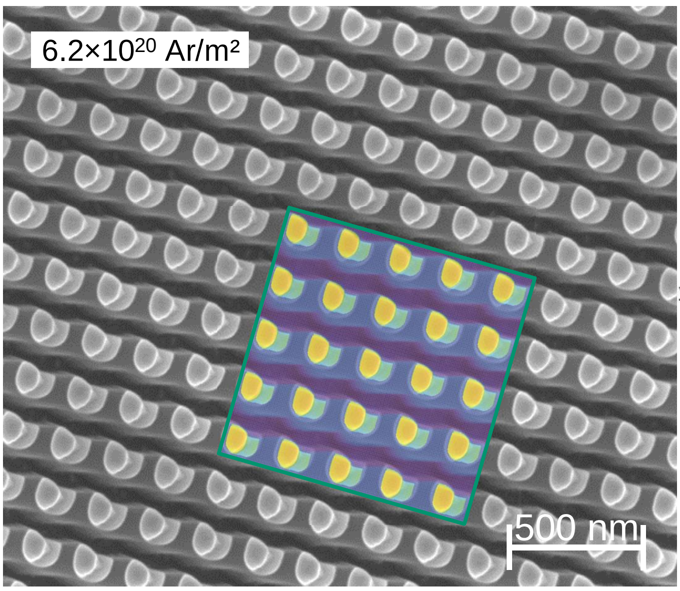
Ta sample after exposure

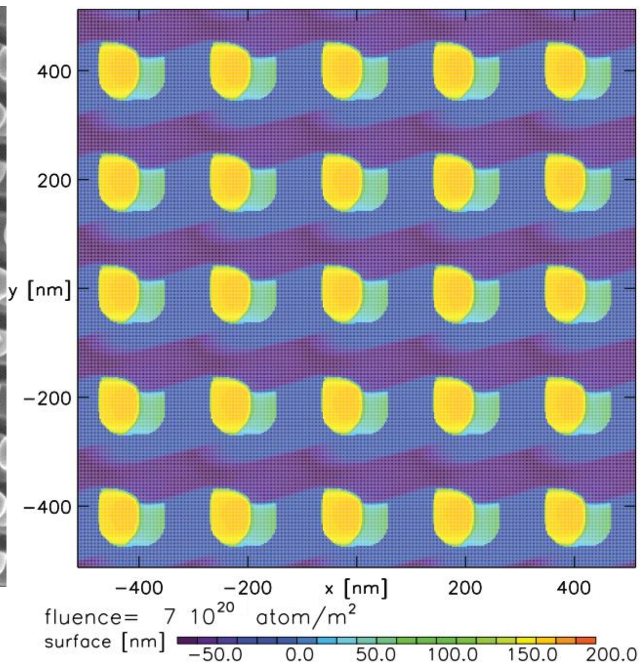
SDTrimSP-3D Ta columns eroded by 5 keV Ar under 45° incidence, 15° rot.



Comparison with SDTrimSP-3D

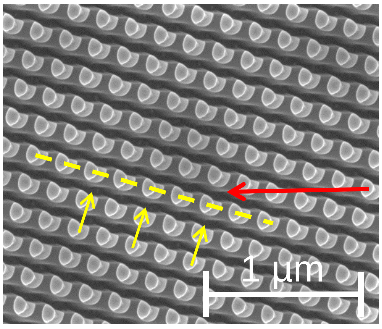
SDTrimSP-3D Ta columns eroded by 5 keV Ar under 45° incidence, 15° rot.

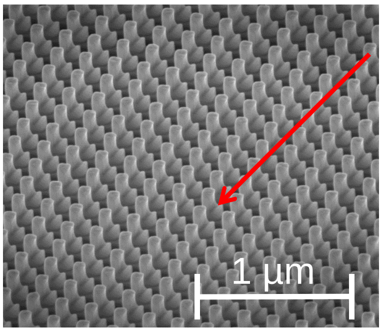




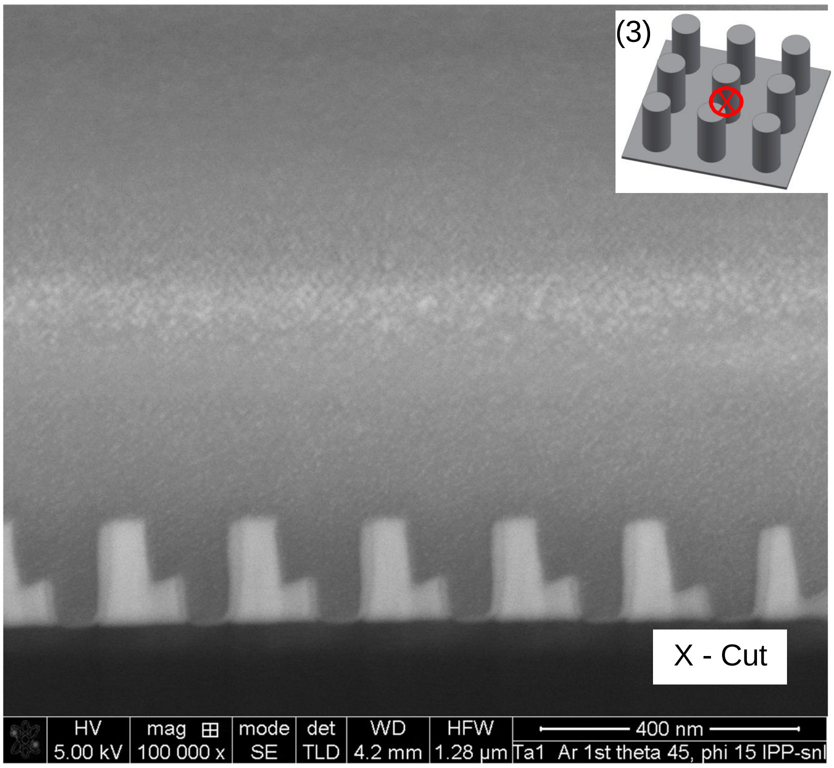
FIB cross-sections

SDTrimSP-3D Ta columns eroded by 5 keV Ar under 45° incidence, 15° rot.



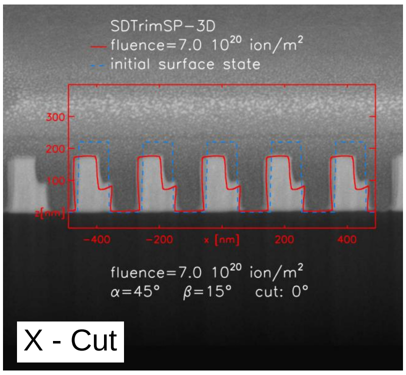


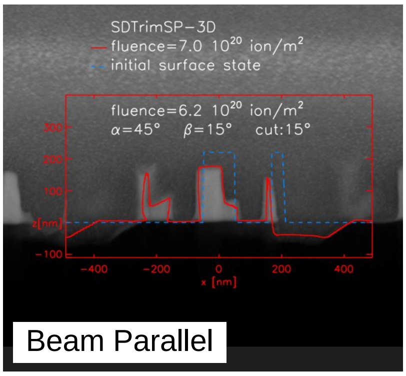
6.2×10^{20} Ar/m²

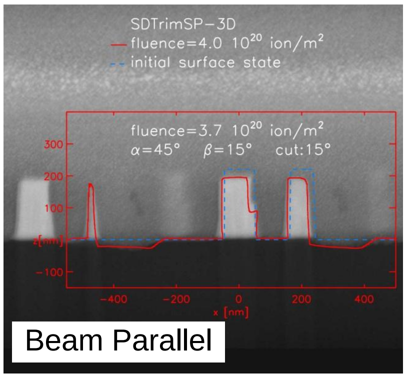


Comparison with SDTrimSP-3D

SDTrimSP-3D Ta columns eroded by 5 keV Ar under 45° incidence, 15° rot.







7×10^{20} Ar/m²

4×10^{20} Ar/m²

Overall very good agreement
Validation of SDTrimSP-3D successful

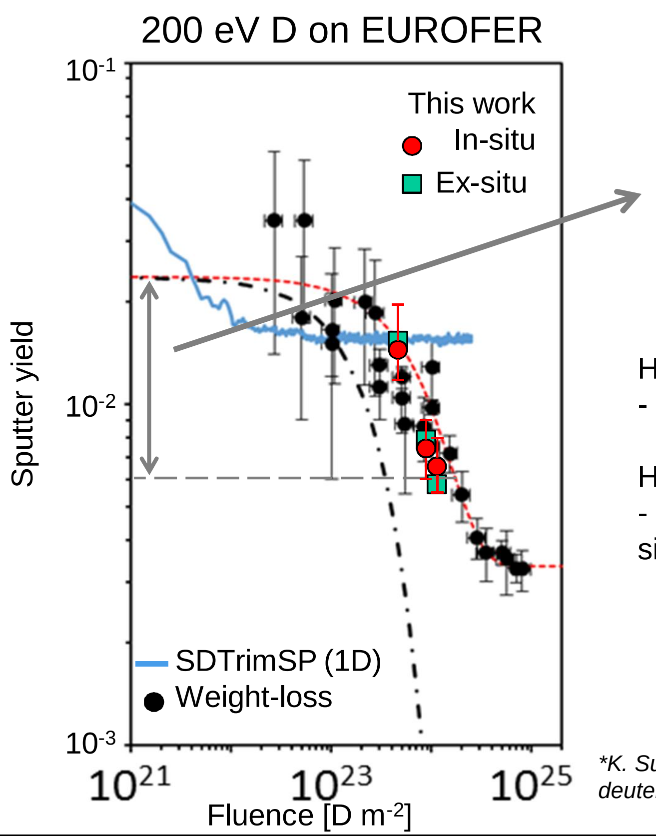
New experiments in SIESTA*

Sputtering of EUROFER: New results (PhD Thesis R. Arredondo)

- Development of SDTrimSP-3D by Udo von Toussaint
- Benchmarking of SDTrimSP-3D by R. Arredondo
- Exposure of well prepared and pre-characterised EUROFER sample to defined D ion flux: **200 eV, 320 K, fluence = 10^{24} Dm^{-2}**
- Simulation of influence of measured surface morphology (SIESTA & AFM) with SDTrimSP-3D (periodic-2D model surface with truncated cos^2)
- Reduction factor due to surface morphology is about 0.58±0.06 (determined from measurements; SEM & FIB cross sections)
- Calculated reduction factor due to preferential sputtering = 0.64±0.03 (calculated with SDTrimSP-1D based on XPS sputter depth profile after SIESTA exposure)
- $SY_{Combined} = (0.37\pm0.05)*SY_{SDTrimSP}$
- Measured: $SY_{SIESTA} = (0.29\pm0.05)*SY_{SDTrimSP}$
- Acceptable agreement, but still many open questions, e.g.:
  - Strong grain-dependent sputtering
  - Less sputtering on smooth grain surfaces (contrary to expectation)

* **SIESTA** = new IPP high current ion source,
see: R. Arredondo et al., “SIESTA: A High Current Ion Source for Erosion and Retention Studies”,
Rev. Sci. Instrum. 89, 103501 (2018). doi: 10.1063/1.5039156

Experimentally determined SY reduction



Experiments show sputter yield reduction

Reduction measured by weight loss:

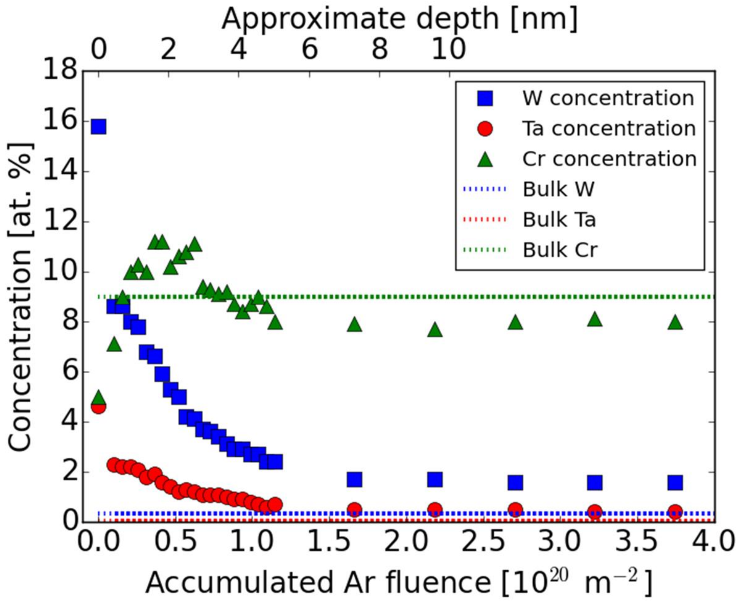
- Ex-situ = 75% ± 2%
- In-situ = 71% ± 5%
- Literature* = 69% ± 4%

How much is W surface **enrichment**?
- Enrichment measured by XPS

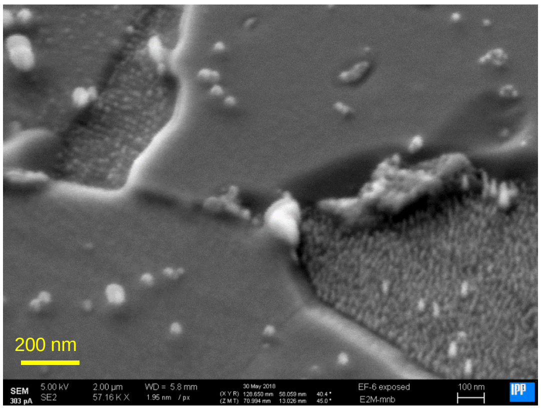
How much is **surface morphology**?
- Effect of roughness from static SDTrimSP-3D simulations with measured surface morphology

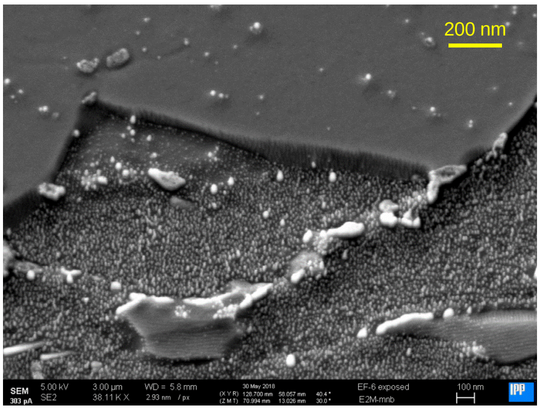
**K. Sugiyama et al., “Erosion of EUROFER Steel by mass-selected deuterium ion bombardment”, Nuclear Materials and Energy 16 (2018)*

XPS sputter depth profiling: W and Ta enrichment in top 2-3 nm

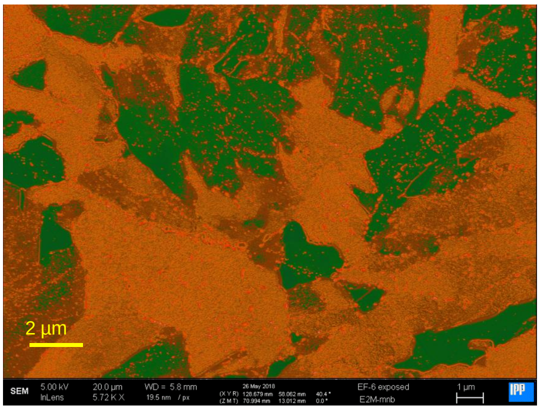


SDTrimSP simulation yields: Reduction by **36 %** in SY (i.e., $SY_{enriched}$ » $0.64*SY_{bulk}$)



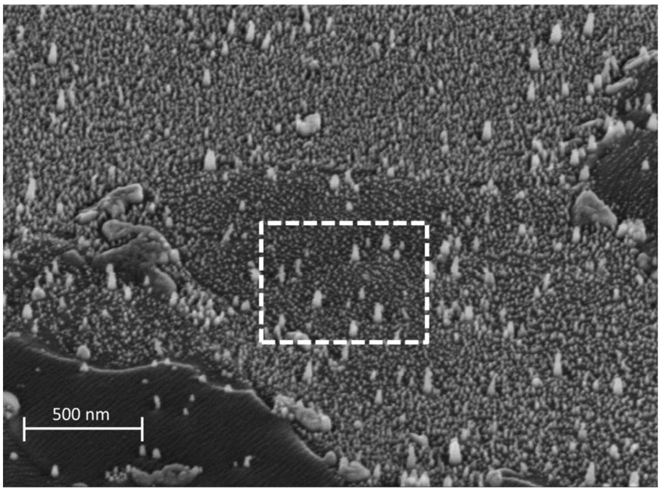


EUROFER after sputtering: flat and rough grains



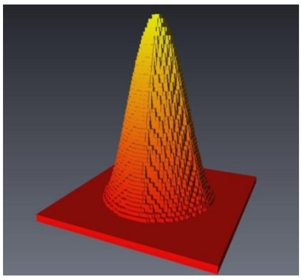
about 71 % of the surface area is rough

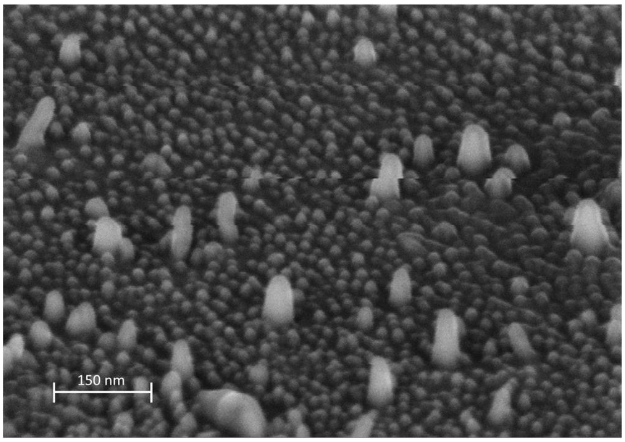
SEM: Rough surface morphology



70% of surface area is rough
30% is smooth

Sputter yield reduction: 24%
(i.e., SY_{rough} » $0.76*SY_{flat}$)





Summary

- Erosion of RAFM steel (EUROFER) was investigated in dedicated ion-beam experiments
- Surface enrichment of W and reduction of sputter yield were experimentally proven
- Reduction of EUROFER sputter yield by factor up to 8 (at 200 eV/D, fluence about 10^{25} Dm^{-2})
- SDTrimSP-3D was developed and benchmarked
- New data for EUROFER show significant grain-dependent sputtering
- For investigated conditions: contribution of enrichment and morphology are roughly comparable
- H retention in steel is low (even lower than in W)

EUROfusion

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