Max-Planck-Institut für Plasmaphysik

Developments at IPP regarding sputtering of EUROFER

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Content:

- Introduction: Sputtering of mixed materials
- Model systems
- Diffusion

EUROfusion

This work has partially been carried out within the framework of the EUROfusion Consortium and has received funding from the Euratom research and training programme 2014-2018 under grant agreement No 633053. The views and opinions expressed herein do not necessarily reflect those of the European Commission. Work performed under EUROfusion WP PFC.

Outline

Commissioning of 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

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 (reduced activation ferritic-martensitic) steel at all?

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

So what is the problem in using steel?

Introduction: Sputter Yields of Fe and W

EUROfusion · IPP

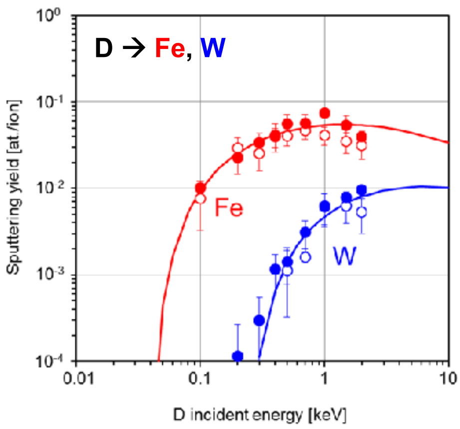
- Energy dependence of sputtering yield of Fe and W measured by weight loss & RBS
  (perpendicular ion incidence)
- Data fitted with Bohdansky formula

$$Y(E) = Q S_n^{KrC}(\varepsilon)\left[1-\left(\frac{E_{th}}{E}\right)^{2/3}\right]\left(1-\frac{E_{th}}{E}\right)^2$$

$$Q(D \rightarrow Fe) = 0.154\ [at./ion]$$
$$Q(D \rightarrow W) = 0.034\ [at./ion]$$
$$E_{th}(D \rightarrow Fe) = 37.5\ [eV]$$
$$E_{th}(D \rightarrow W) = 216\ [eV]$$

- 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.

Introduction

IPP

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 (0.4 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

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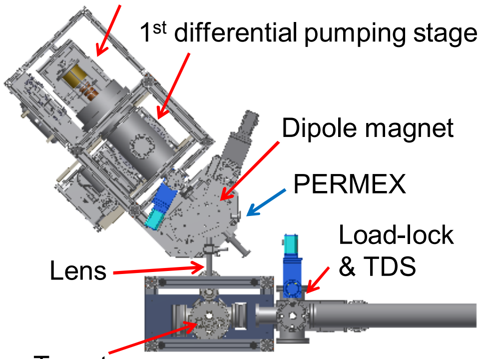
SIESTA (Second Ion Experiment for Sputtering and TDS Analysis): a High Current Ion Source for Sputter Yield Measurements

Overview of SIESTA

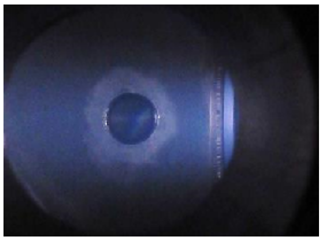


CAD Birds-eye view of SIESTA

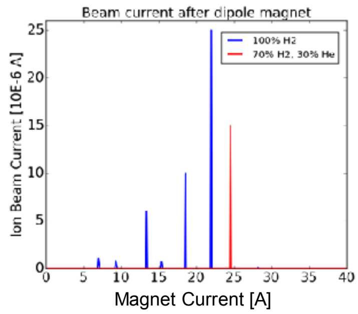
- Ion beam extracted from source
- Neutral gas pumped out in differential pumping stages
- Dipole magnet deflects beam → mass selected ion beam
- Optional ion lens focuses the beam
- Beam impinges on target sample, which can be rotated, heated and weighed in-situ with magnetic suspension balance.
- TDS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

Status of the experimental setup

- Ion source is operational and has been tested with H, D, He and Ar ions
- Dipole magnet enables effective mass filtering of up to 10 keV Ar^+ ions
- The ion beam has been characterized – beam adjustment, current density and emittance have been measured
- All vacuum components have been installed and are in operation. Base pressure at the target of $<10^{-8}$ mbar

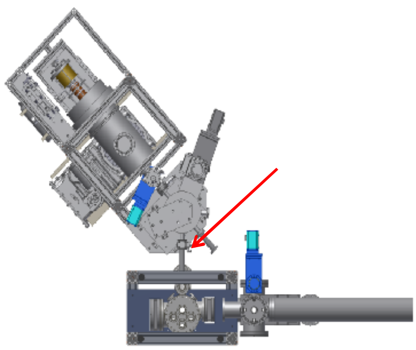


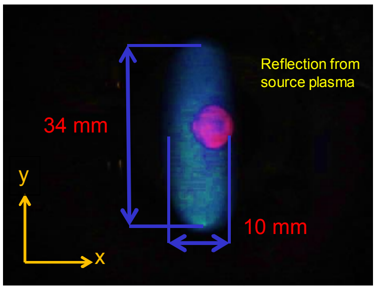
First plasma in SIESTA on Feb. 24th 2016



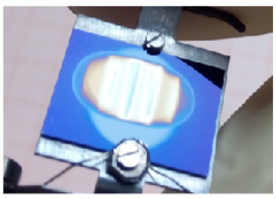
Status of the experimental setup

- Deflection in the dipole magnet induces focusing in the deflection plane (x), forming an image at the target
- Beam reaching the target is astigmatic and inhomogeneous





10 kV beam profile at quartz glass after dipole magnet (light blue color)

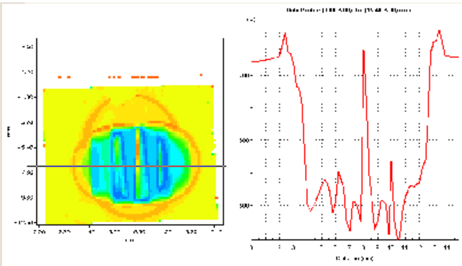


a-C:H sample eroded by inhomogeneous beam

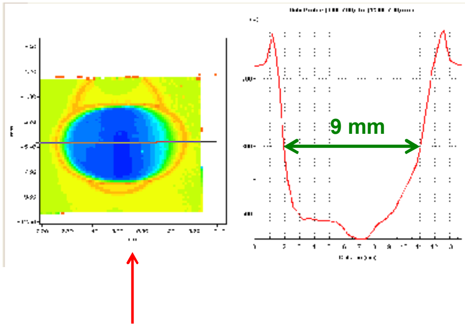
Status of the experimental setup

- The ion beam is “wobbled” at the dipole magnet to homogenize the beam footprint at the target

Without wobbling



With wobbling



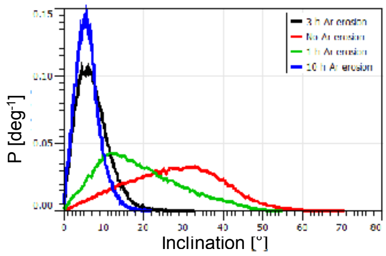
“Wobbled” sample can be considered homogeneous enough

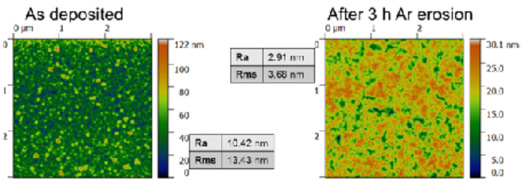
Angular dependence of sputter yield for Fe

Controlled roughness samples

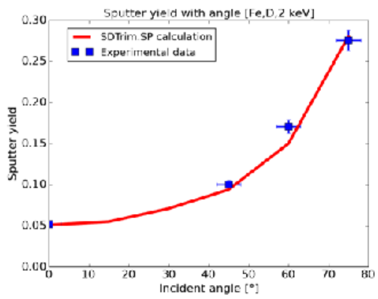
- nm-smooth Fe/Si samples have been prepared and eroded under varying angles of incidence
- Results agree with simulations for a perfectly smooth surface

Slope distribution of Fe on Si sample





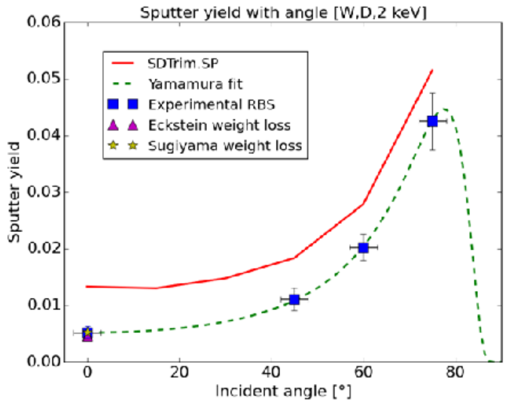
Atomic Force Microscopy of 500 nm Fe layer on Si substrate



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).*

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Introduction: SDTrimSP

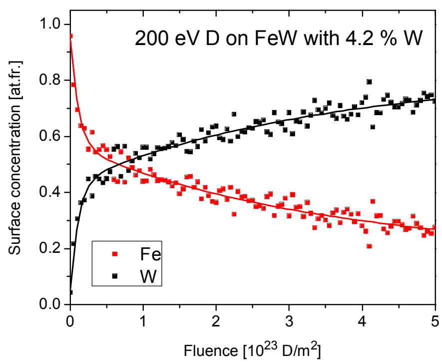
The dynamic surface evolution due to preferential sputtering can be simulated by SDTrimSP

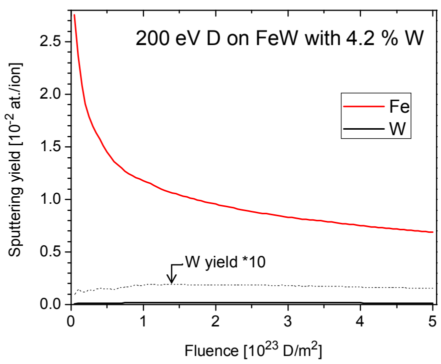
- SDTrimSP: dynamic version of TRIM.SP [1] (an earlier version was called TRIDYN [2])
- TRIM.SP describes the sputtering of surfaces due to impact of energetic species in the binary collision approximation
- TRIM.SP is well established and benchmarked with numerous experimental results
- SDTrimSP takes 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

SDTrimSP results: Dynamic Behaviour

- RAFM steels contain **W** which has a much lower sputter yield than Fe etc.
  - Preferential sputtering leads to W enrichment due to the difference of sputtering yields.
  - Erosion yield is reduced.





Dynamic surface evolution due to preferential sputtering

Preferential Sputtering

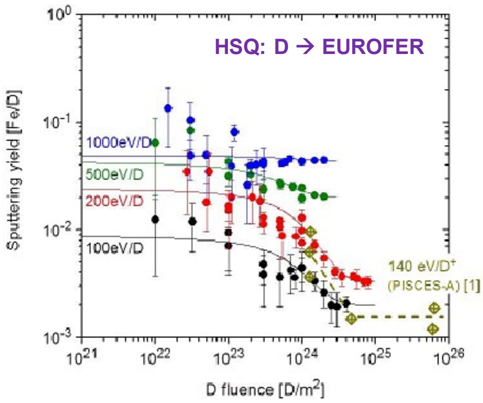
Preferential sputtering

- 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)

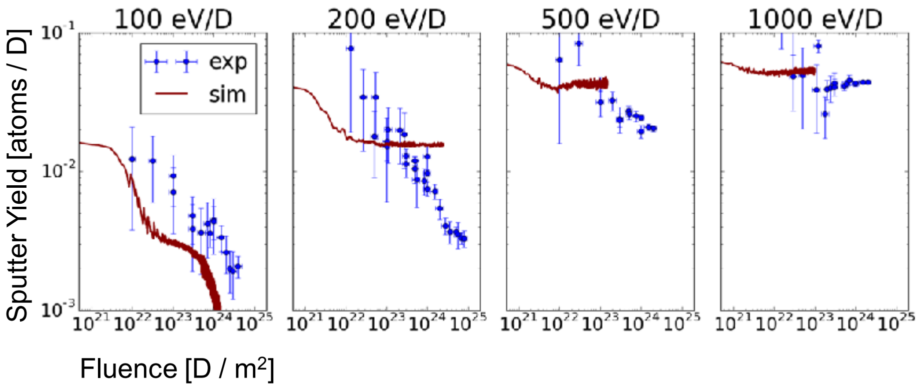
- Yield reduction in the higher fluence range (≥ 10^{23} D/m^2), as well as for Fe/W layer.
- For 200 eV/D steady state seems to be reached for fluence > ~ 5 x 10^{24} D/m^2.
- PISCES-A data[1] at very high fluence and 140 eV/D also indicate steady state for fluence > ~ 5 x 10^{24} D/m^2.



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



→ Experimental sputter yield reduction for lower energies not reproduced

→ Possible reasons: W surface binding energy? Roughness?

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Temperature Dependence

Temperature Dependence

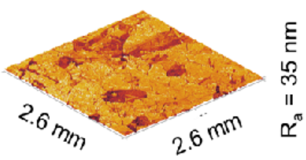
- T dependence of sputter yield
- Onset of diffusion (counteracting enrichment?)
- T dependence of surface morphology

Measuring the interdiffusion coefficient

Sample preparation

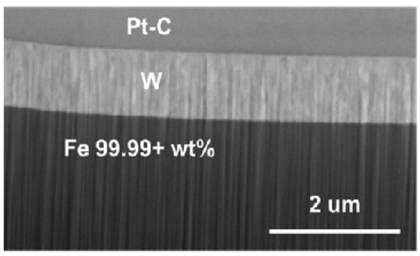
Sputter-deposition of W

Fe substrates recrystallised at 1200 K for 96 h



Confocal laser scanning microscopy image

W on Fe
annealed at 1050 K for 6 h

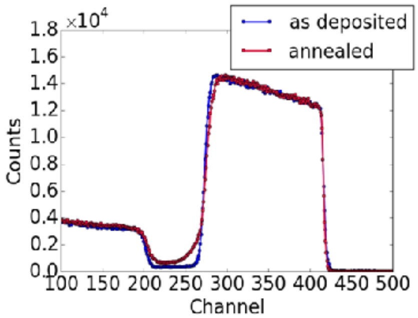


SE image of FIB cross section

Measuring the interdiffusion coefficient

Ion beam analysis → depth profile

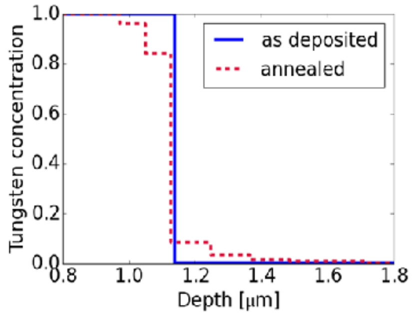
Ion beam analysis before and after annealing
(Caveat: lateral averaging)



Rutherford backscattering (RBS) spectra with ^4He at 4 MeV

→ Noticeable interdiffusion has occurred

Extract depth profiles from RBS data by fitting with SIMNRA

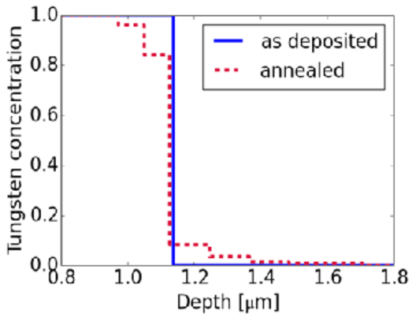


→ Concentrations up to ~10%
→ Fit spline for Boltzmann-Matano analysis

Measuring the interdiffusion coefficient

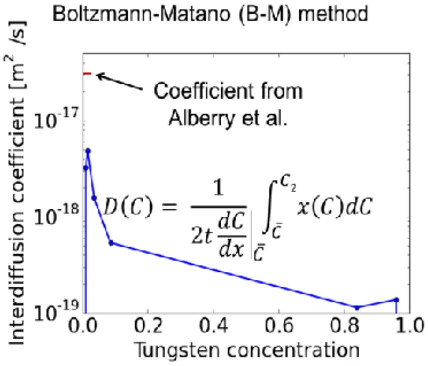
Ion beam analysis → depth profile →D (C)

Extract depth profiles from RBS data by fitting with SIMNRA



→ Concentrations up to ~10%
→ Fit spline for Boltzmann-Matano analysis

Calculate interdiffusion coefficient by Boltzmann-Matano (B-M) method

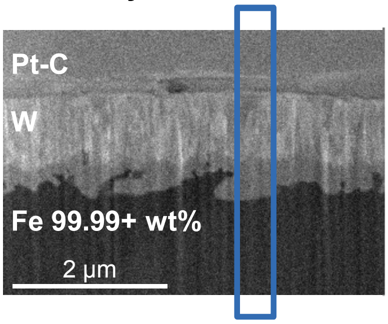


→ Diffusivity decreases with increasing tungsten concentration

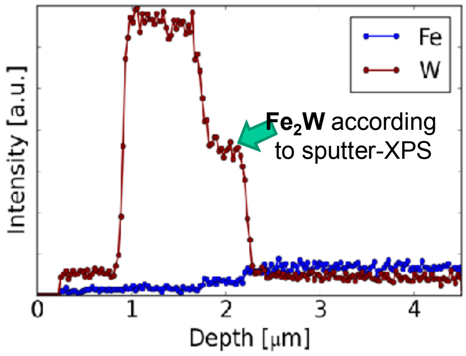
Phase formation at the iron-tungsten interface

SEM analysis

EDX line scan



Secondary electron (SE) image of cross section produced by focussed ion beam (FIB) milling



EDX line scan

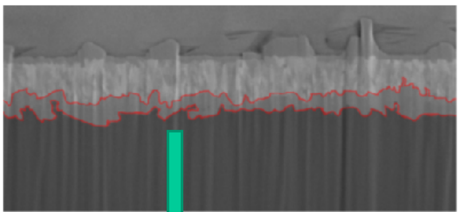
→ Sharply separated zones with different contrast

→ Plateaus in energy dispersive X-ray emission → phase formation

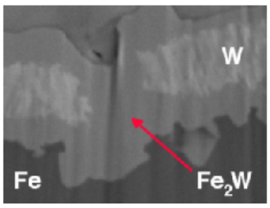
Phase growth at the iron-tungsten interface

SEM analysis

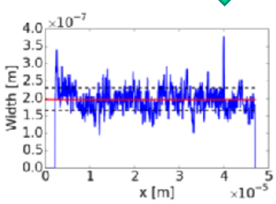
Secondary electron (SE) images of cross section produced by focussed ion beam (FIB) milling



Fe_2W layer (marked in red) after annealing at 1050 K

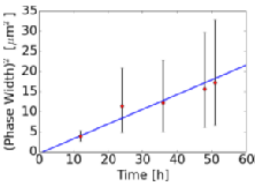


→ Fe can break through W layer at grain boundaries



Thickness distribution of Fe_2W phase

Time series



Quadratic phase growth at 1050 K

→ Interdiffusion coefficient in Fe_2W can be determined

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Projects within EUROfusion in WP PFC

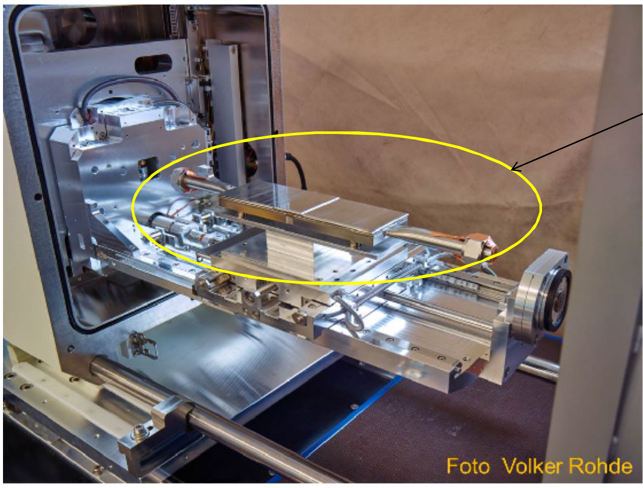
EURO*fusion* tasks

- IPP research on PWI issues is strongly embedded in European cooperation coordinated by the EURO*fusion* consortium
- Issues regarding sputtering of EUROFER are part of WP PFC, Subtask SP2 "PWI Processes I: erosion, deposition and mixing"
- Additional current IPP contributions to this subtask:
  - Preparation and characterisation of model layers (Fe-W) (e.g. for Univ. Vienna, IAP)
  - Influence of roughness on sputtering
  - ToF RBS analysis of eroded EUROFER samples
  - Ion beam exposure of EUROFER samples for MEIS analysis

- Example: Investigation of W enrichment at 450-500°C in GLADIS

EURO*fusion* tasks

- Example: Investigation of W enrichment at 450-500°C in GLADIS



Sample braced on cooling structure for active cooling.

Sample mounted on heavy load sample holder for SEM

EURO*fusion* tasks

Example: Investigation of W enrichment at 450-500°C in GLADIS

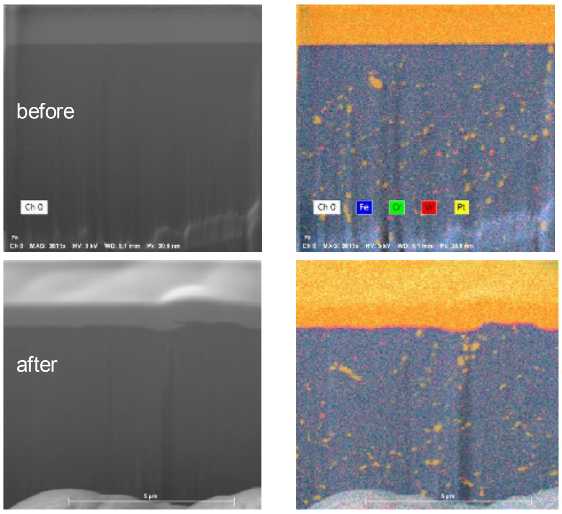
Exposure conditions:

- **H-beam:** 2 MW/m², 17 keV, 1.3×10^{21} H/m²s, 30 sec pulse length
- Species: H^+ : 22% 17 keV
  H_2^+ : 43% 8.5 keV
  H_3^+ : 35% 5.7 keV
- 1st loading GLADIS fluence 10^{24} H/m²
- analysis in Auriga
- 2nd loading GLADIS fluence 10^{25} H/m² (completed)
- analysis in Auriga

Some images after 1st loading

EURO*fusion* tasks

Some images after 1st loading in GLADIS



SEM cross section　EDX　EDX of W

Summary

- Erosion of RAFM steel and model systems was investigated in ion beam experiment and in linear plasma devices
- Surface enrichment of W and reduction of sputter yield were experimentally proven
- Reduction of EUROFER sputter yield by factor 8 (at 200 eV)
- For the model layers reasonable agreement with initial SDTrimSP simulations, but closer analysis shows significant differences → seems reduction cannot be explained by preferential sputtering
- Reduction possibly strongly influenced by surface morphology development → influence of roughness

EURO*fusion*

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