



WP PFC SP2 - PWI Processes I: erosion, deposition and mixing

Elodie Bernard – Emmanuelle Tsitrone



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- SP2.1: Eurofer
- SP2.2: Ammonia qualification and destruction
- SP2.3: PWI related issues of new materials envisaged as PFM in DEMO with D, He and D seeded discharges
- SP2.4: Impact of Surface Roughness on erosion and retention
- SP2.5: High fluence exposure of W in MAGNUM PSI



SP2.1: Measure the activation energy for W diffusion in steels with higher W content, similar to the experiment done with EUROFER in 2017

H. R. Koslowski, FZJ



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Key results in 2020



- Experiments are delayed because the LEIS apparatus was occupied for work on smart alloys
[Nucl. Instr. Meth. B 479 42-46 (2020) and Nucl. Mater. Energy 22 100736 (2020)]
- “Hyperfer” samples with a geometry for PSI-2 experiments (thickness 5 mm) and W contents of 4 wt% and 6 wt% are available and need to be cut in 1 mm slices for the LEIS apparatus
- Experimental procedure and analysis will follow
Nucl. Mater. Energy 16 (2018) 181-190 “Temperature-dependent in-situ LEIS measurement of W surface enrichment by 250 eV D sputtering of EUROFER”
- At present LEIS measurements of pure W are in the works in order to better characterize the W scattering peak and the re-ionisation background which will help to determine the enriched layer thickness of the Hyperfer



SP2.1 Erosion of Eurofer and advanced steels (2019)

F. Ghezzi, A. Uccello (ENEA)



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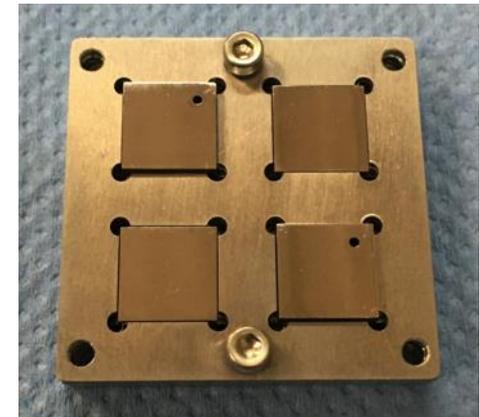
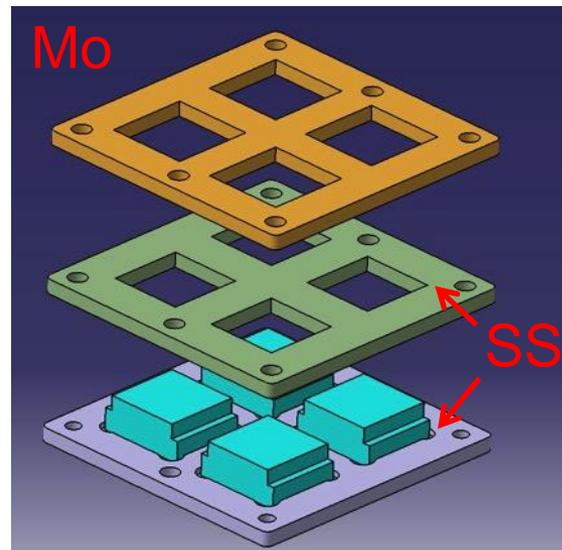
SP2.1 Erosion of Eurofer and advanced steels (2019)



Exposure of HiperFer and CroFer from FZJ to a deuterium plasma in GyM up to a fluence of 10^{25} ions/m² for different bias voltages.

- Mass loss for sputtering yield evaluation
- LEIS (IFP-CNR) + RBS (@RBI) for W surface enrichment

A new sample carrier was designed and manufactured to arrange Eurofusion standard samples



- **9 HiperFer** (1.1 at% W, 0.7 at% Nb) and **9 CroFer** (0.58 at% W, 0.3 at% Nb) samples arrived at late July 2019 (at.% compositions are the results of RBS analysis carried out by FZJ)
- Exposure conditions:
 - Plasma exposure time: 3 h, 5 h, 7 h, 15 h → **4.3E24, 7.2E24, 1E25, 2.16E25** ions•m⁻²
 - Bias voltage: -80 V and -180 V → **100 eV** and **200 eV** taking into account the plasma potential

SP2.1 Erosion of Eurofer and advanced steels (2019)

Activity carried out:

- Procurement of new sample carrier
- Weight measurement for 8 virgin sample CroFer and HyperFer (average of 5)
- XPS and LEIS of 1 virgin CroFer and 1 virgin HyperFer.
- Exposure of CroFer and HyperFer samples to the GyM plasma.

Activity still to be done:.

- Weight measurements of eroded sample (and Erosion Yield vs fluence).
- XPS and LEIS on exposed sample for surface enrichment.
- RBS (@RBI) of virgin and exposed sample for surface enrichment.

- Difficulties:
- Interpretation of surface enrichment on steel based on the ToF-SIMS , XPS and SEM-EDX analyses on Eurofer exposed sample carried out at JSI
- (on voluntary basis) revealed thermal diffusion to the surface of many elements
- (Cr, Mo , Mn ...), surface diffusion of W enhanced by irradiation .

SP2.1 Qualification of EUROFER in comparison with other advanced steels as plasma-facing material (2020)



Expose Fe films, along with a gold strip, to deuterium plasma in GyM (flux $<4 \cdot 10^{20}$ ions /m² s, -200 V bias) up to a fluence of 10^{25} ions/m² to address the contribution of re-deposition during EuroFer, HiperFer and CroFer exposures.

Activity still to be done:

- Procurement of gold stripes
- Expose the samples to GyM Plasma
- Surface analysis (XPS, LEIS) for re-deposition on gold (late 2020 beg. 2021)



SP2.1: Influence of hydrogen on the segregation of W in iron

M. Balden, Max-Planck-Institute for Plasma Physics

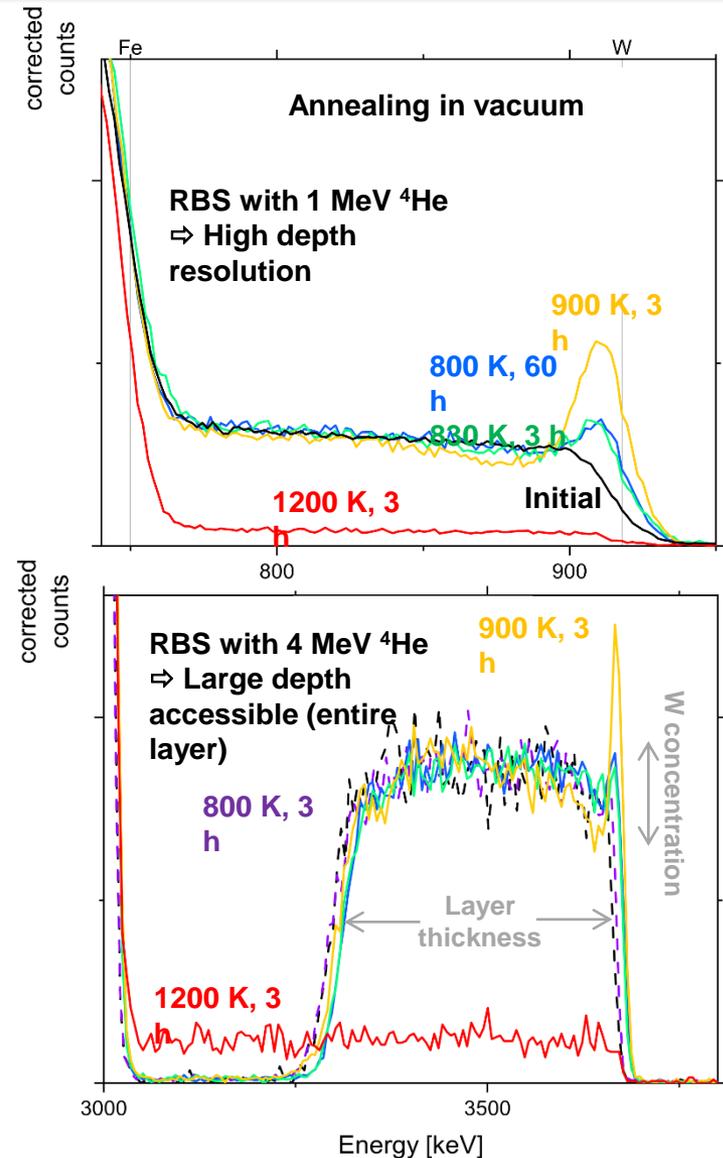


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Key results in 2020



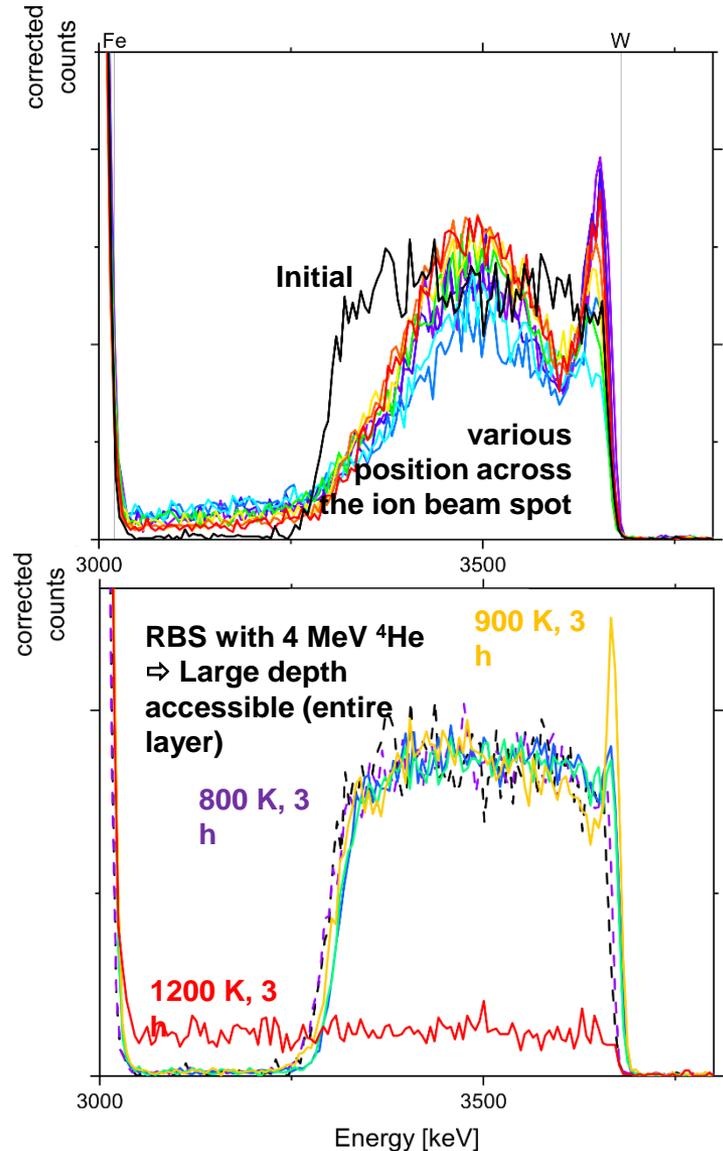
- Produce Fe-W layers, anneal them to different temperatures (and times), expose them to deuterium plasma/ion beams (and probably rare gas plasma/ions such as neon), analyse with TOF-RBS the segregation of W/Fe, check the morphology of the layers (SP2.1-2020-1)
- Fe-W layers produced by magnetron-sputtering on Fe substrate with sufficient thickness and composition homogeneity (~400 nm, 2.4 at% W in Fe with <5 at% O and <1 at% C)
- W-enrichment in top 20 nm of layer by annealing in vacuum always below a factor of two compared to deposited layer (up to 900 K, up to 60 h)



Key results in 2020



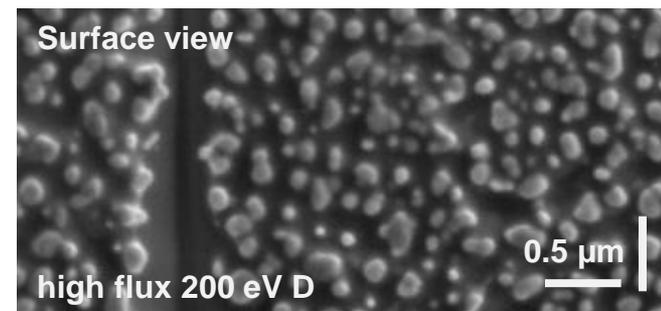
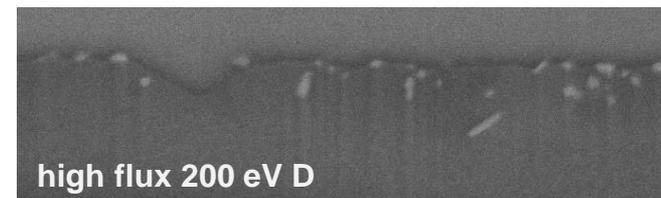
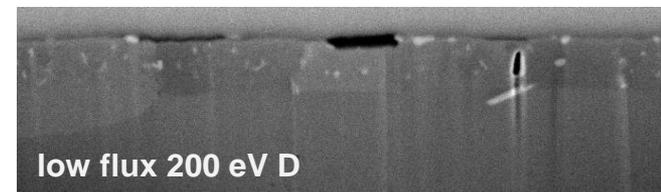
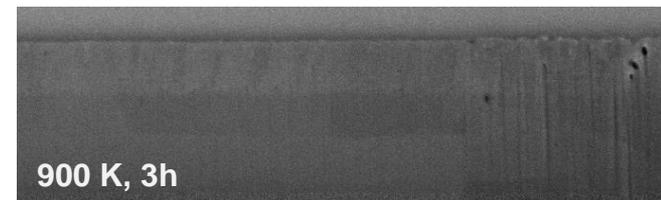
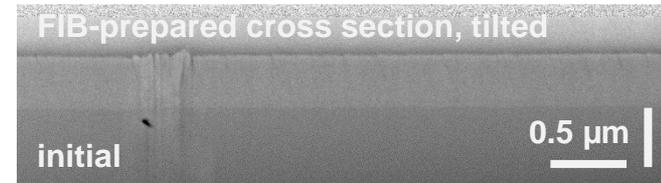
- Soft plasma exposure (<10 eV D ion energy) at 800 K for 3 h: no enhanced surface W enrichment compared to annealing
- 200 eV D ion beam exposure at 800 K for 3 h
 - ⇒ strong rearrangement of W
 - ⇒ stronger W enrichment at surface than by annealing even to higher temperatures
 - ⇒ solving W into the Fe substrate (see 1200 K annealing)



Key results in 2020



- Soft plasma exposure (<10 eV D ion energy) at 800 K for 3 h: no enhanced surface W enrichment compared to annealing
- 200 eV D ion beam exposure at 800 K for 3 h
 - ⇒ strong rearrangement of W
 - ⇒ stronger W enrichment at surface than by annealing even to higher temperatures
 - ⇒ solving W into the Fe substrate (see 1200 K annealing)
 - ⇒ W agglomeration visible in SEM images on FIB-prepared cross-section (not for annealing only)
 - ⇒ interface between layer & substrate not flat anymore
- **Final conclusion: Strong effect by hydrogen presence and/or ion impact**
- Task completed
- No publication jet (except a Bachelor thesis in German)



SP2.1: Pre- and post-characterization of selected samples with HIERDA and/or RBS

I. Bogdanovic Radovic , IRB

No sample received to perform IBA



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SP2.2: Isotopic exchange of NH_3 with deuterated surfaces of tungsten

T Angot, R Bisson, A Dunand, M Minissale (PIIM-AMU-CNRS)



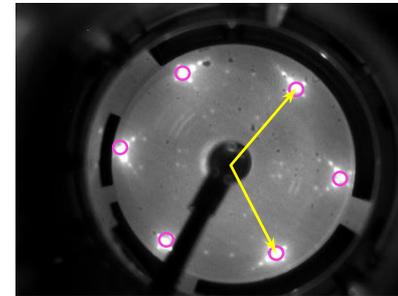
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Key results in 2020

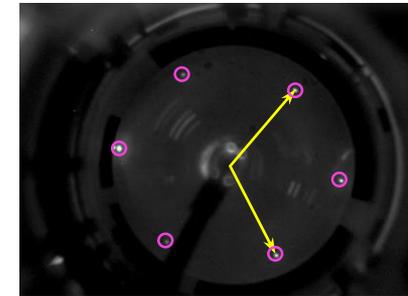


- **Goals (2019):** measure the production of NH_xD_y species when a beam of NH_3 molecules impinges on a surface of W with controlled D coverage (no task in 2020)
- **Results (2019):** a new method for cleaning W surfaces has been used (laser heating) but O and C contamination remained in unknown quantities
- **Major results obtained until now (02/11/20):** Truly clean W surfaces have been obtained. Controlled D coverage have been obtained thanks to sequential co-adsorption of O and D on initially truly clean W.
- **Plan for completing the work (delay due to COVID-19):** by the end of 2020, a beam of NH_3 (operational) will be aimed at the W surface with controlled D coverage (operational) and NH_xD_y species will be measured as a function of D surface coverage

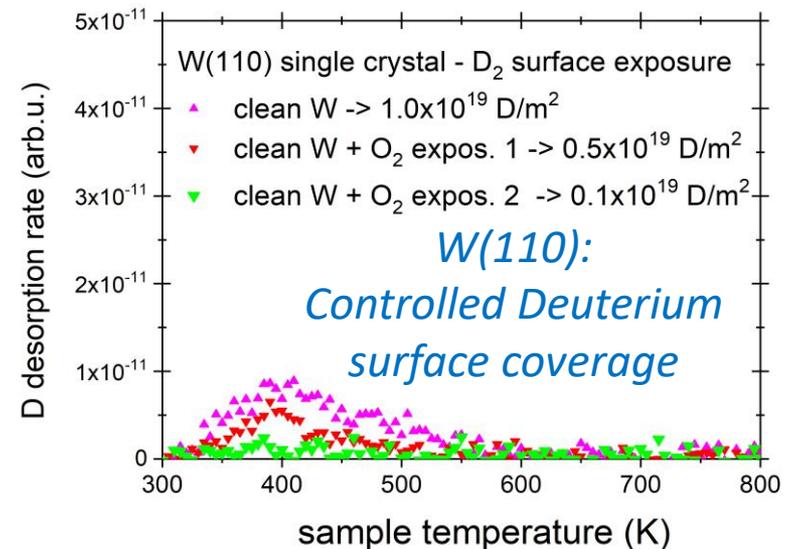
W(110)
"native oxide"



W(110)-(1x1)
truly clean



Low energy electron diffraction results

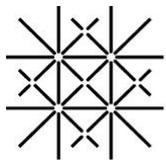




SP2.2.T003: Qualification of ammonia production mechanisms, its quantification and potential ways of mitigation/destruction

Rodrigo Antunes, Roland Steiner, Laurent Marot, Kunal Soni, Fabien Sanchez and Ernst Meyer

Department of Physics, University of Basel, Department of Physics, CH-4056 Basel, Switzerland



**University
of Basel**



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Pending 2019 – 2020 tasks



Task	Year	Status
Study isotopic exchange rate between NH_3 and D_2	2019	Ongoing. To be completed by end of 2020
Study NH_3 production rate on W surfaces previously contaminated with N_2 , H_2 , O_2 , and boron	2019	Completed
Study the low-temperature decomposition of NH_3 on novel catalysts	2020	Not started. To be completed by mid of 2021 (due to COVID-19)

Publication to be submitted soon to ACS Catalysis:

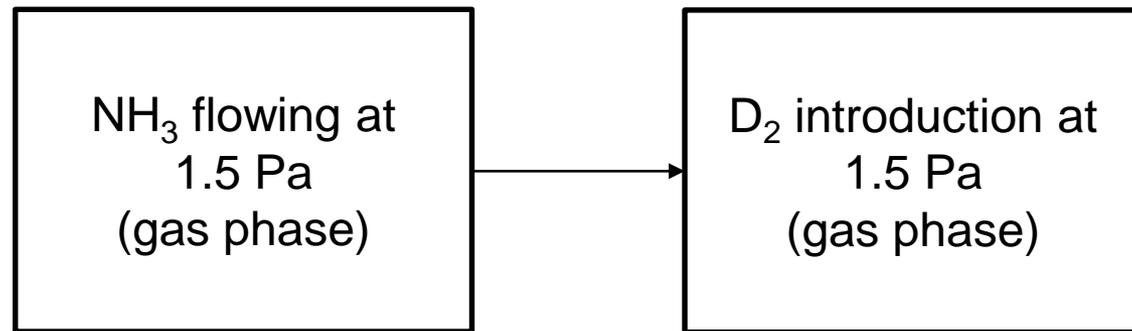
R. Antunes et al. *Plasma-assisted catalysis of ammonia using tungsten as catalyst: a parametric study*

Key results Task 2019: Isotope exchange

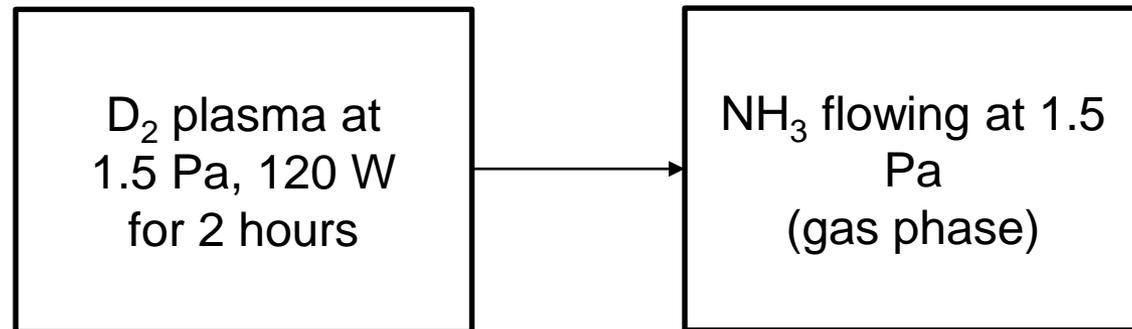


Up to now only experiments **without catalyst** done in our metal-free setup (quartz surface) at room temperature

Experiment 1



Experiment 2



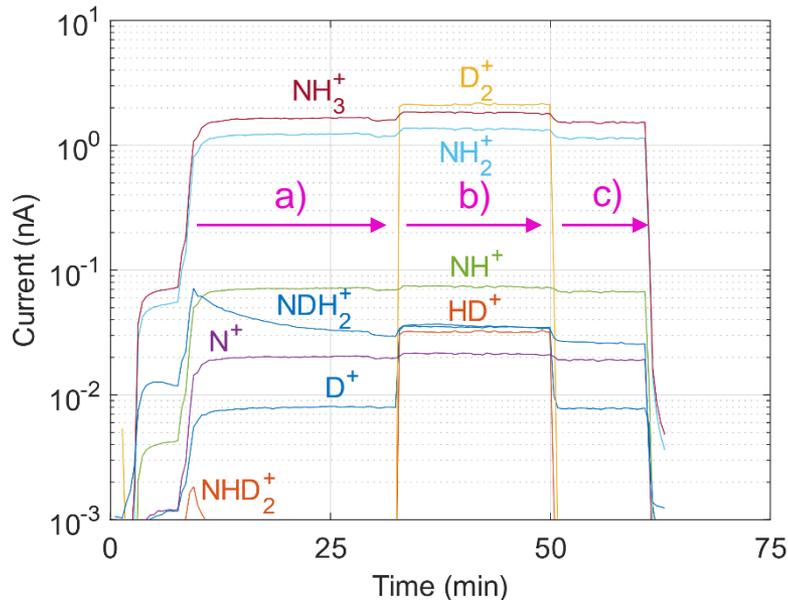
(See setups on last slide)

Key results Task 2019: Isotope exchange



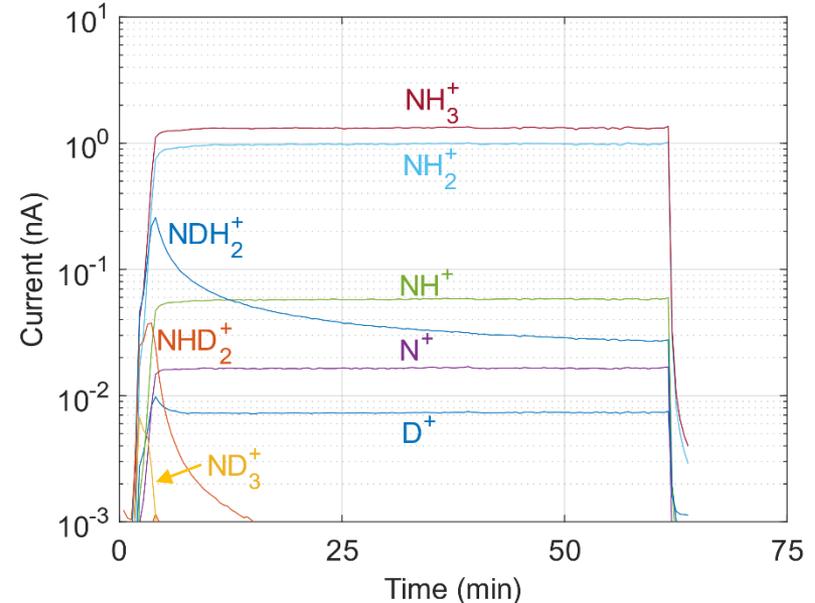
Experiment 1

a) NH_3 only, b) $\text{NH}_3 + \text{D}_2$, c) NH_3 only



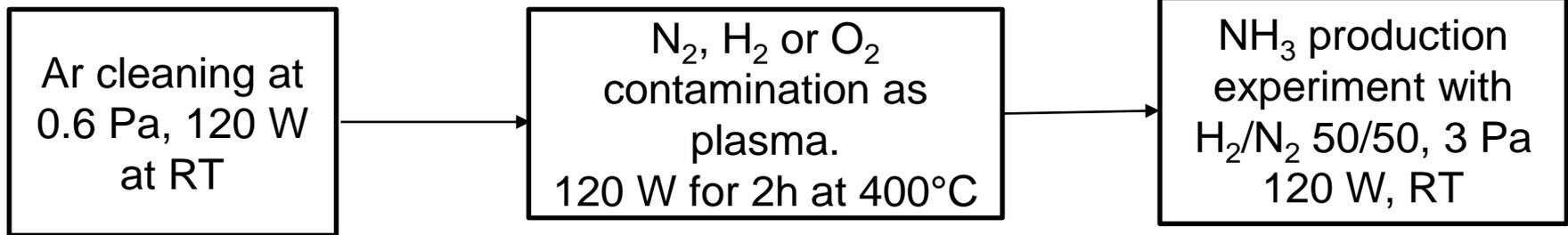
Experiment 2

NH_3 flowing after D_2 plasma

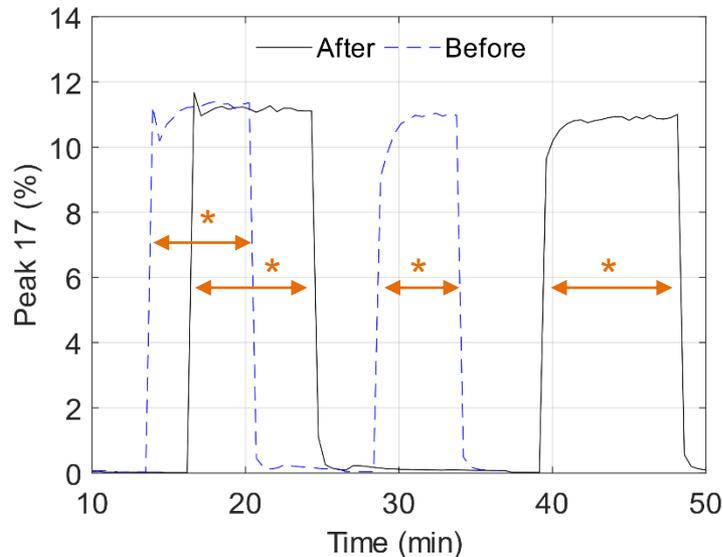


- **No isotopic exchange** occurs on $\text{D}_2 + \text{NH}_3$ gas phase (the signals of NQ_3 remain fairly unchanged when D_2 is introduced)
- After D_2 plasma, we see a **significant increase of NDH_2 , NHD_2 , ND_3** . NDH_2 is dominant
- Ar cleaning after Experiment 2 **confirms the isotopic exchange** occurred: D_2 is lowest and the D and HD signals are higher than after D_2 plasma

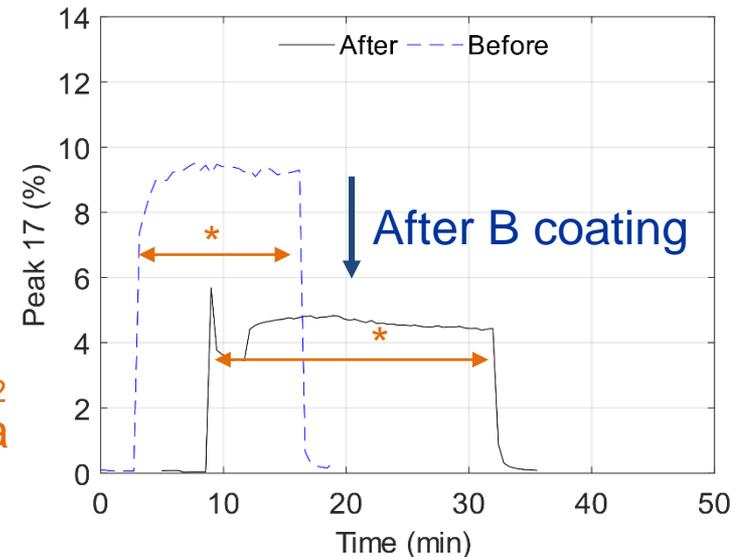
Key results Task 2019: W contamination



NH₃ peak in % after 400°C O₂ plasma



NH₃ peak in % after Boron coating



- **Exposing** the W surface to H₂, N₂ or O₂ **did not affect** NH₃ production
- Coating with Boron **led to a decrease** in NH₃ production **by a factor of 1.7**
- Note that two different W foils were used explaining the difference in 17% “Before”



SP2.2: EDX measurements samples exposed on N-seeded plasma

Timo Dittmar, FZJ



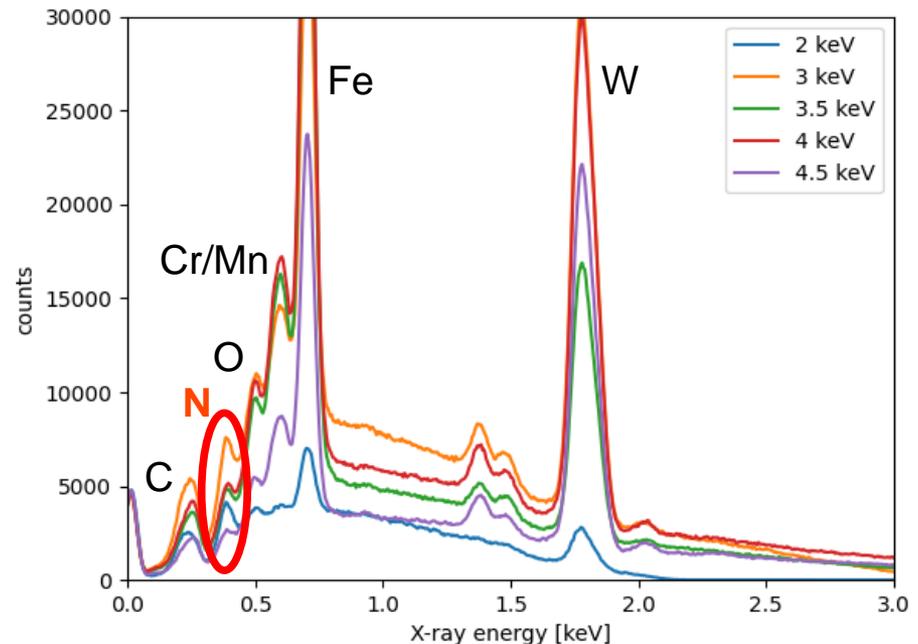
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Key results in 2020



Task: Surface nitrogen content as function of plasma parameters, seeding levels and sample temperatures.

- P92 steel and W samples were exposed in PSI-2 to pure and N-seeded D- and He-plasma.
- after exposure the sample were analysed with in-vacuo EDX with different electron energies
- all samples show **stable nitrogen layer** after exposure (so formation of volatile NH compounds below detection limit)
- absolute quantification challenging and ongoing (hopefully finished by March 2021)





SP2.2: Ammonia decomposition

L. Laguardia, F. Ghezzi, G. Gervasini

**Institute for plasma science and technology (ISTP)
ENEA –CNR Milan Italy**



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Key results in 2020 (1)

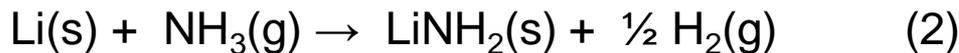


Goals:

1. Study the variable-temperature NH_3 decomposition efficiency on ruthenium (Rh) as catalyst

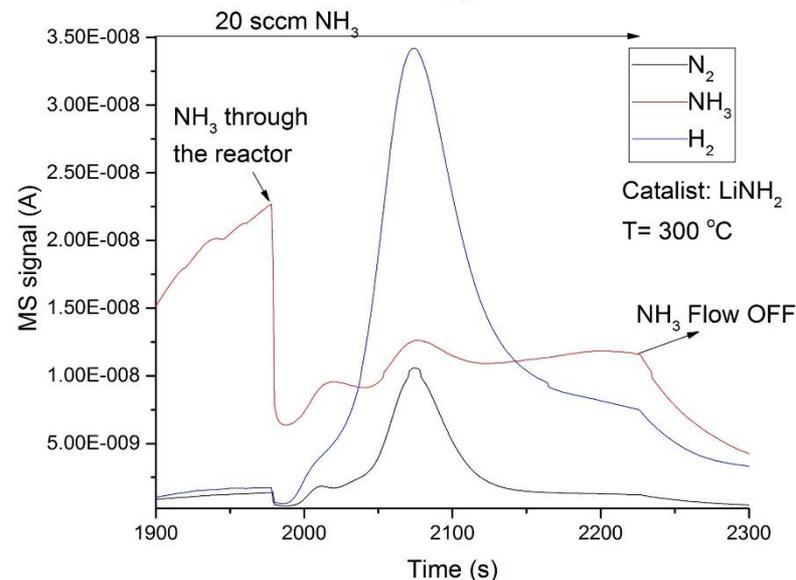
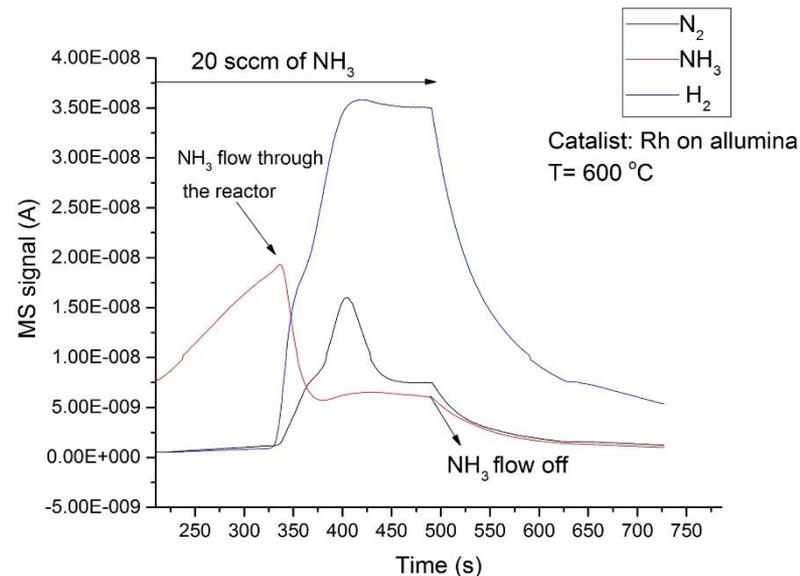


2. Testing ammonia (NH_3) decomposition activity across a lithium amide (LiNH_2) as catalyst at low temperatures (less than 600°C).



Major results:

1. At 200°C , with Rh, catalysis reaction starts even if the conversion efficiency is low.
2. At 200°C , with LiNH_2 , catalysis reaction don't starts. Higher activation energy is required.
3. With LiNH_2 at moderate temperatures (300°C) catalysis reaction produces the same increase of the H_2 signal that was achieved with Rh at high temperature (600°C).



Key results in 2020 (2)

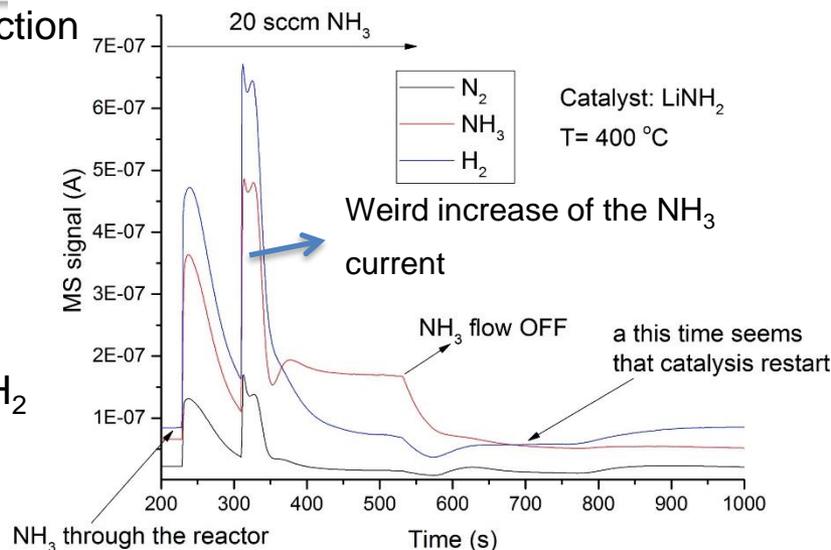


4. Experiment performed at 400°C with LiNH₂, shows, as reaction starts, a very high increase in the H₂ signal, and in the same time, a weird increase of the NH₃ signal.

5. LiNH₂ shows a reversible thermal decomposition to form lithium imide (Li₂NH).

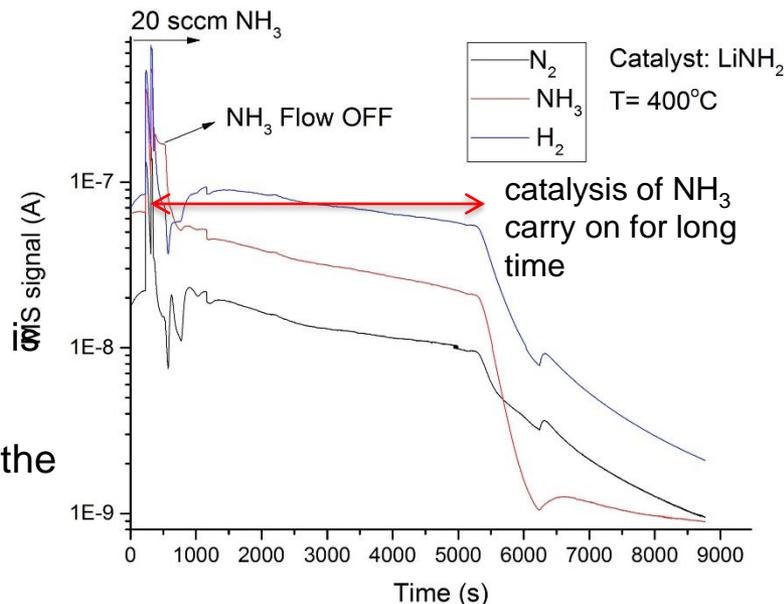


6. Making same quantity of NH₃ flow, with LiNH₂ as catalyst, H₂ production proceeds for very long time.



Final conclusions for completed task:

1. Rh catalyst shows superior performance at lower temperatures.
2. The LiNH₂ system shows superior NH₃ decomposition activity to Rh based catalysts.
3. Activation energy within the empirical relation of Arrhenius is not the only determining factor of the reaction kinetics.
4. LiNH₂ has a more complex interaction with ammonia than the simple surface catalysis.
5. LiNH₂ melts at around 370 °C.





SP2.2: Injection of ammonia into GyM plasmas to determine effective D/XB values

L. Laguardia, A. Cremona, A. Uccello, F. Ghezzi

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Key results in 2020



• **Goal:** Injection of NH_3 into GyM plasmas to determine effective D/XB values.

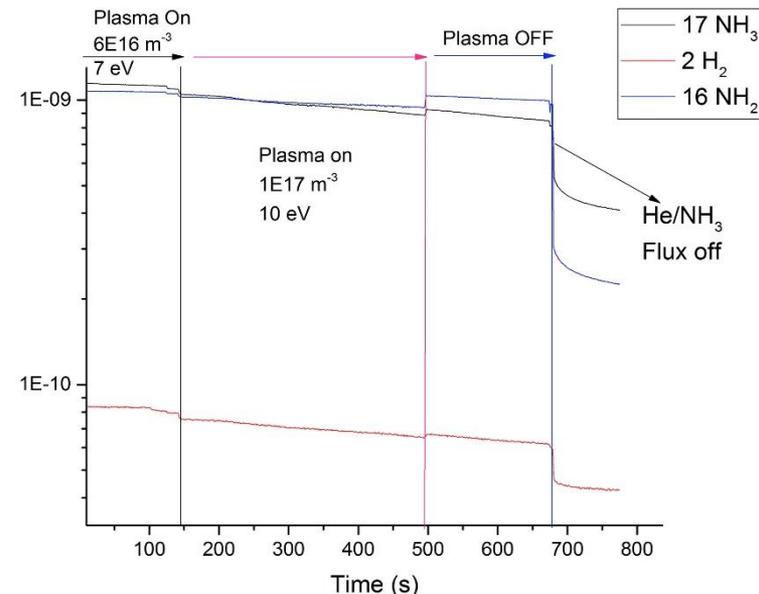
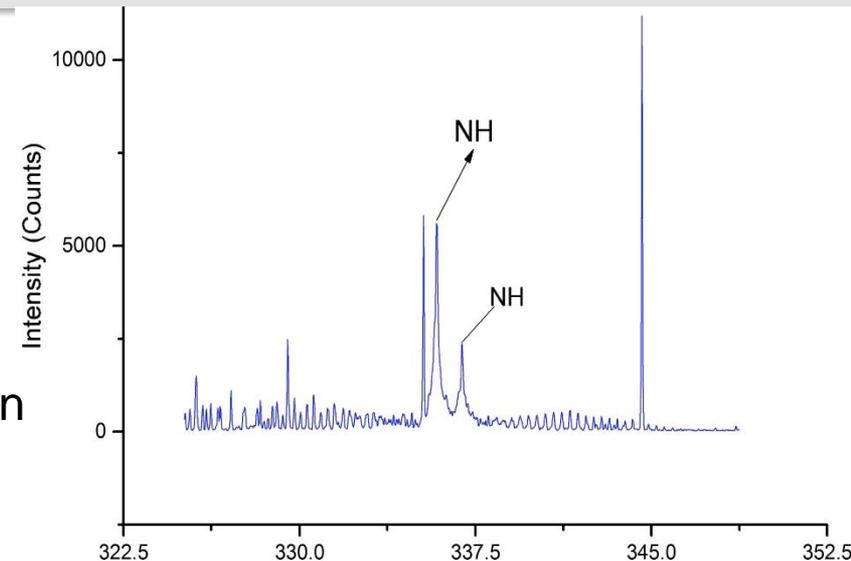
• **What was done:** Until now we have done experiments by injection of He/ NH_3 mixture ($\text{NH}_3 = 0,5\%$ mol) at $T_e = 10\text{ eV}$ $n_e = 1.4\text{E}17\text{ m}^{-3}$.

• **Major results obtained:**

- NH_3 injected into GyM plasmas is dissociated in NH and H_2 as shown by OES and MS results
- NH band intensity increases with T_e and n_e
- Some particles of NH_3 are retained at the surface of the wall as demonstrated from NH band identified in post NH_3 injection argon plasmas.

• **A plan for completing the work:**

- Simulation, that taking in to account rate coefficients involved in the dissociation and excitation, necessary to obtain the total NH photon flux!
- To make a modelling to estimate the number of particles which can escape out of your plasma volume.



SP2.3: Self-passivating tungsten alloy

H. Maier
Max-Planck-Institut für Plasmaphysik Garching

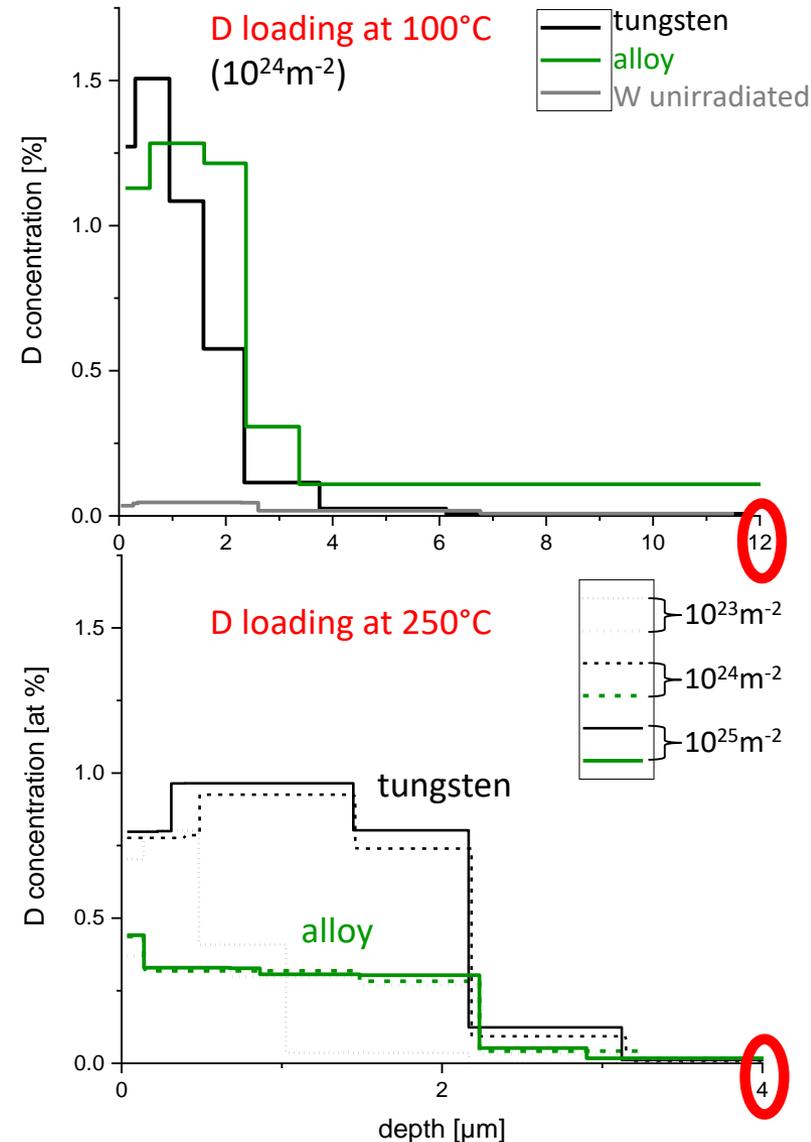


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Key results in 2020



- Expose powder metallurgical self-passivating alloy W-10Cr-0.5Y implanted with 20 MeV tungsten to D plasma at 38 eV/deuterium at 250°C substrate temperature up to fluences of several 10^{24} D/m². Measure D retention by NRA and TDS.
 - As in pure W radiation damage increases D retention substantially
 - At elevated temperature (250°C) **the retention after irradiation is significantly lower** than in W
 - Neutron irradiation would affect the whole volume instead of only ~ 2 microns (20 MeV W)
- **T retention in the whole volume would be lower**
- ⇒ Further investigation of temperature dependence





SP2.3.T003 and T004: Erosion of W_f/W

Johann Riesch, Rodrigo Arredondo
Max-Planck-Institut für Plasmaphysik, Garching, Germany

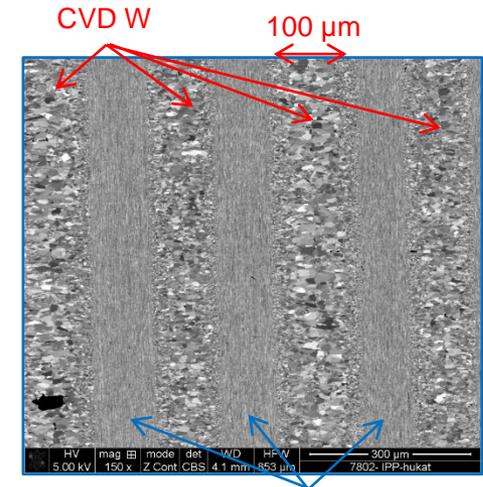


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Aim and results



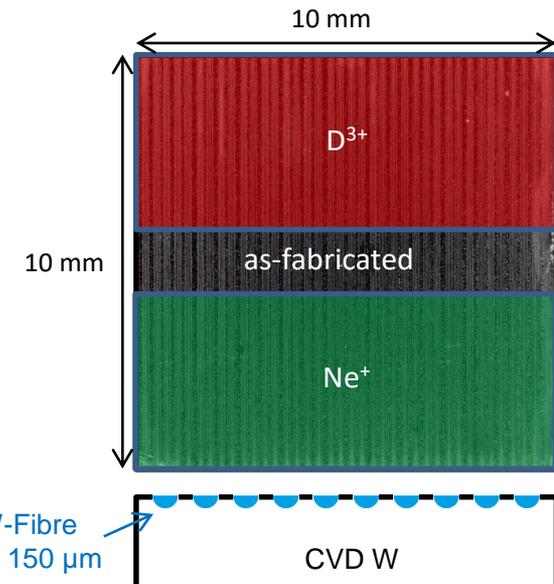
- Expose W_f/W tungsten produced by CVD to mass selected ion beam to measure erosion to gain initial information on **sputter yield** and **surface morphology**.
- Use model system with W fibres (highly deformed) and CVD W (defect free) at the surface and expose to D^{3+} and Ne^+ in SIESTA
 - 2 keV/D D^{3+} ions to 4×10^{24} D/m²
 - 5 keV Ne^+ ions to 3×10^{22} Ne/m²



W-Fibre \varnothing 150 μ m

Sputter yield determined by:

- Ex-situ weight loss (WL)
 - D^{3+} : 0.0078 (Literature 0.005; SDTrimSP 0.01)
 - Ne^+ : **0.24** (Literature 0.8; SDTrimSP 1.18)
→ needs to be double checked
- Eroded layer thickness (CLSM)
 - D^{3+} : CVD W: 0.021
W-Fibre: 0.0096
(Literature 0.005; SDTrimSP 0.01)
 - Ne^+ : CVD W: 1.90
W-Fibre: 1.25
(Literature 0.8; SDTrimSP 1.18)



Conclusion and Outlook

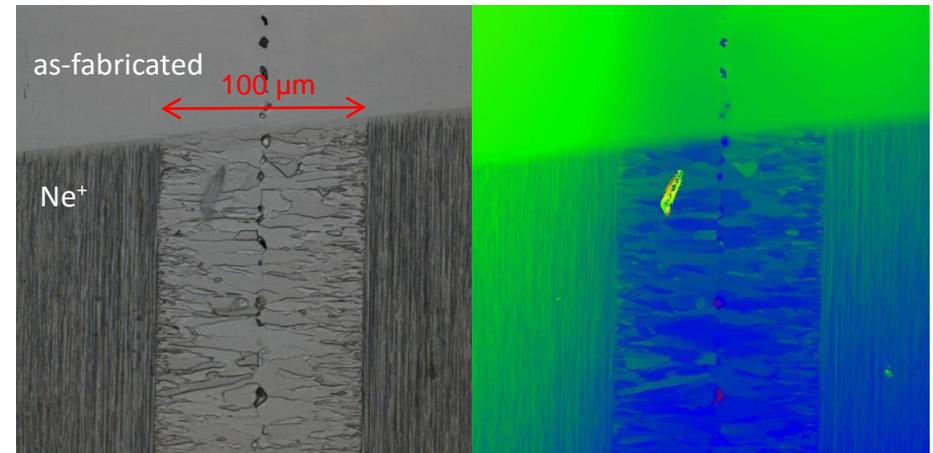


Conclusion

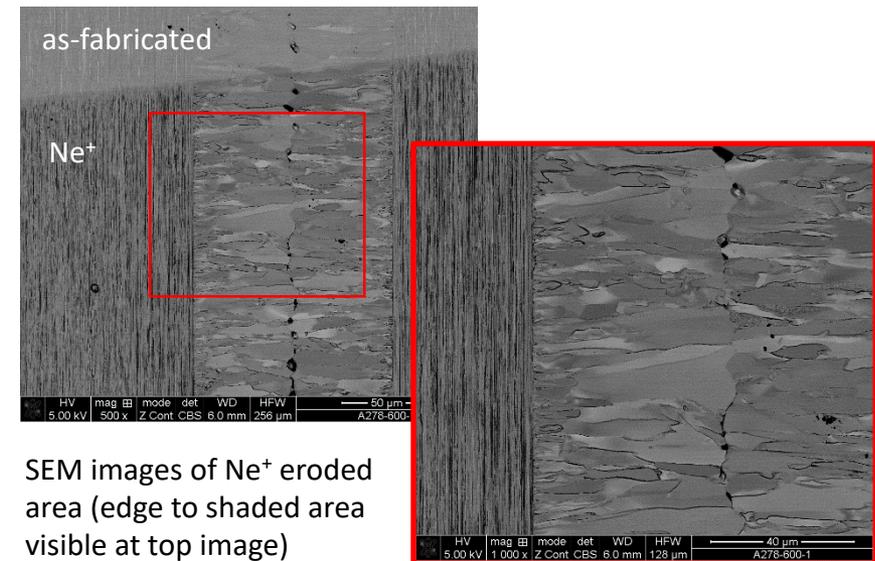
- Sputter yield for W-Fibre close to literature and theoretical values, for CVD W significantly higher
- Grain dependant sputtering in both W-Fibre and CVD W visible, may be more pronounced in the latter

Open Questions/Ideas for future work

- Role of texture/grain orientation
 - Evaluate EBSD measurements
- Role of microstructure
 - Study effect of annealing (defined change of microstructure)



Light microscopy image (left) and corresponding height image (right)



SEM images of Ne^+ eroded area (edge to shaded area visible at top image)



SP2.3 task #3: D retention in W-W2C composites and pure WC produced by field assisted sintering (FAST)

**Sabina Markelj, Matic Pecovnik, Anze Abram,
Collab. K7 dep. from WP MAT: Sasa Novak, Petra Jenus, Matej
Kocen**



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Key results in 2020



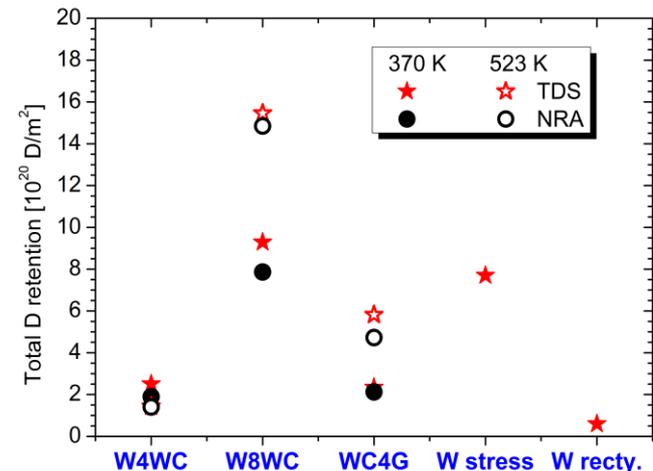
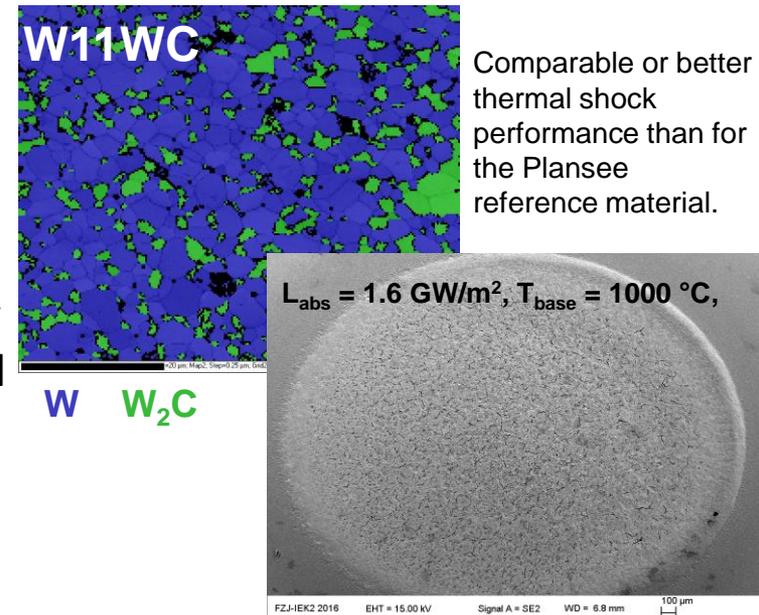
- Expose samples composed of pure W, or W-W₂C composites and pure WC produced by field assisted sintering (FAST) that were displacement-damaged by 20 MeV tungsten implantation to D plasma at elevated temperature.
- Study erosion by mass loss and surface change. Study deuterium retention by NRA and TDS. Microstructural analysis before and after.

Development of new samples - four new samples were produced in October - delay due to covid-19:

- **W7WC (W-W₂C mix) = candidate for high heat flux material**
- **W11WC = best response on thermal shocks**
- **W4WC = W reference**
- **mWC: WC4G = tungsten carbide**

W ion damaging, D exposure and D retention analysis still need to be done in collaboration MPG.

Publication in preparation for the results from last years (D retention shown on the figure bellow).



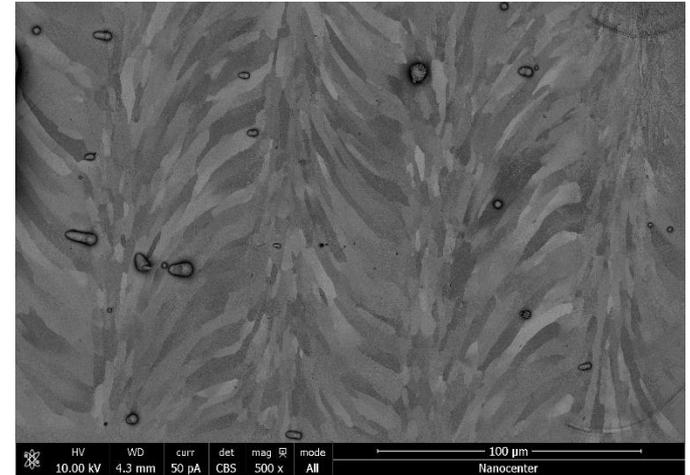
Plans for the future



Study deuterium retention in W-W₂C, WC and W₂C samples in comparison to reference W:

- Study of deuterium retention in additively manufactured (Laser Powder Bed Fusion-LPBF) W and W-W₂C
- Expose LPBF produced W and W-W₂C samples that were displacement-damaged by 20 MeV tungsten implantation, to D plasma at elevated temperature.
- Study erosion by mass loss and surface change. Study deuterium retention by NRA and TDS. Microstructural analysis before and after.

S. Markelj



Surface microstructure of LPBF W11WC



WP PFC SP 2.3

Exposure of doped W produced via PIM to PSI-2 deuterium plasmas at different bias voltage and particle flux. Surface characterization via SEM-EDX

Robert Krug, B. Unterberg (FZJ)

Further delayed



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WP PFC SP 2.3

Exposure of smart alloys to PSI-2 deuterium Ar and Ar/He seeded plasmas at different bias voltage and particle flux

Andrey Litnovsky (FZJ)



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- Exposure of smart alloys to PSI-2 deuterium plasmas at different bias voltage and particle flux. Surface characterization via SEM-EDX (FZJ)

Experiment [3]	Ion energy, [eV]	ion flux [ions/m ² s]	ion fluence [ions/m ²]	Sample temperature [C]
DAr120P Composition:	120 0.99D+0.01Ar	2.8x10 ²¹	0.2x10 ²⁶	620-700
DArHe120P Composition:	120 0.94D+0.05He+0.01Ar	5.7x10 ²¹	0.5x10 ²⁶	620-700
D220P (for comparison)	220 D	5.5x10 ²¹	1.0x10 ²⁶	620-670

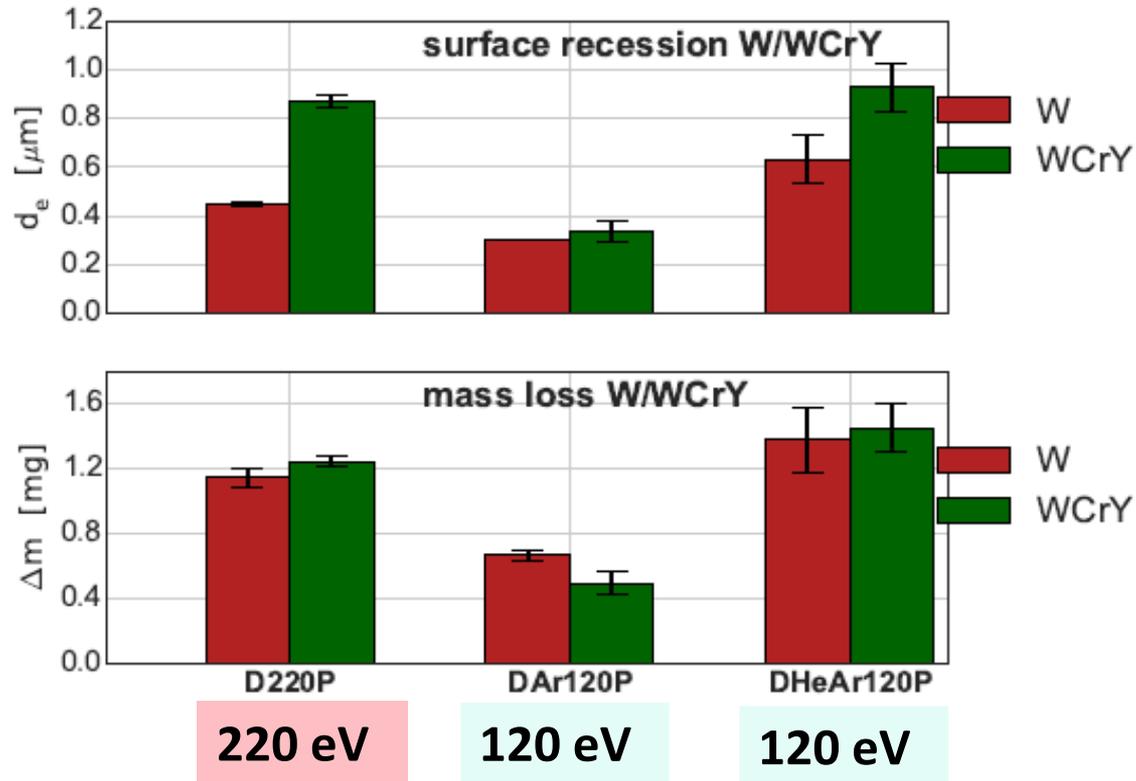
120 eV: regular operation of power plant

220 eV: limiter regime (during e.g. startup)

[3] J. Schmitz, A. Litnovsky, F. Klein et al., Phys. Scr. T171 (2020) 014002

FIB data on surface recession

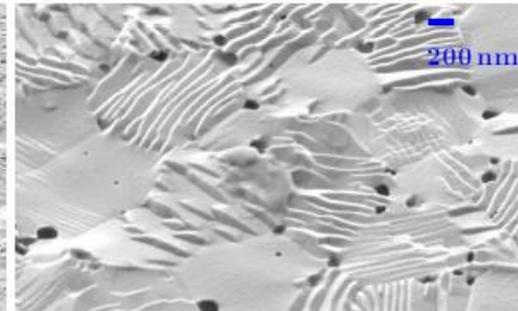
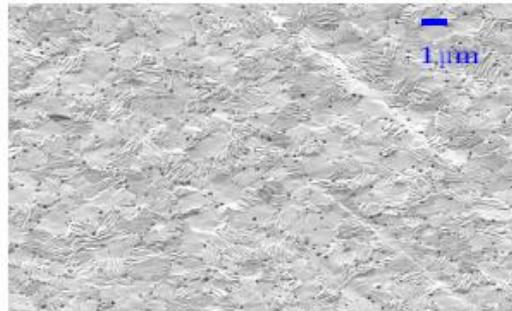
Mass loss data: microbalance



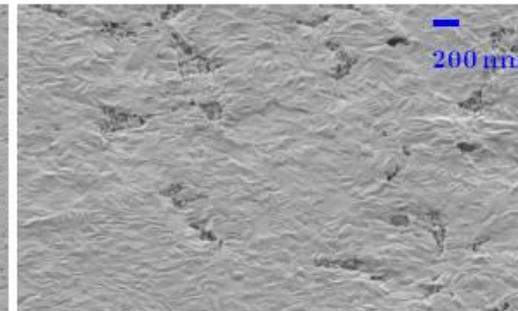
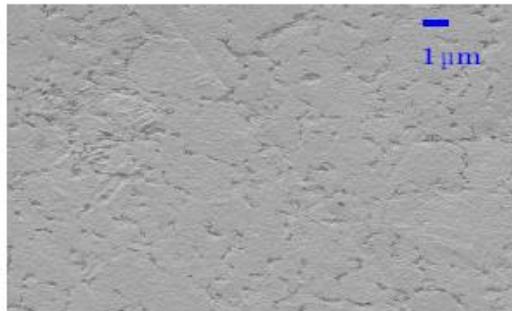
- Similar sputtering of W and SA in case of Ar seeding
- Enhanced sputtering of W and SA in case of Ar+He seeding
- Generally: seeded plasmas sputtered both W and SA severely

SEM

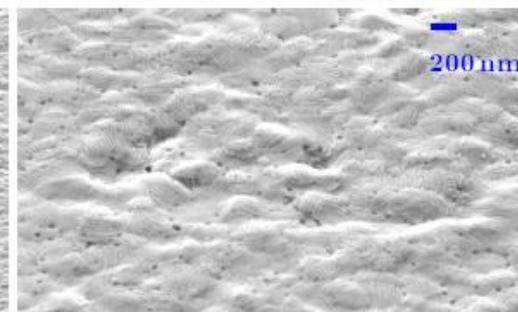
D⁺220 eV



**D⁺120 eV
+ 1% Ar**



**D⁺120 eV
+ 1% Ar
+ 5% He**



Grain structures are much more pronounced for sputtering by D ions only



SP2.4:

Study the temporal surface roughness evolution under plasma exposure and its impact on the erosion and deposition balance

C. Cupak¹, P.S. Szabo¹, H. Biber¹, R. Stadlmayr¹,
C. Grave¹, F. Troneberger¹, W. Möller² and F. Aumayr¹

- 1) Institute of Applied Physics, TU Wien, Austria
EUROfusion partner: Fusion@ÖAW
- 2) Institute of Ion Beam Physics and Materials Research
Helmholtz-Zentrum Dresden-Rossendorf (HZDR), Germany



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Key results SP2.4, ÖAW task 1



Goal: Quantify synergistic effects on the sputter yield from concurrent ion beam (D₂ and Ar) irradiation

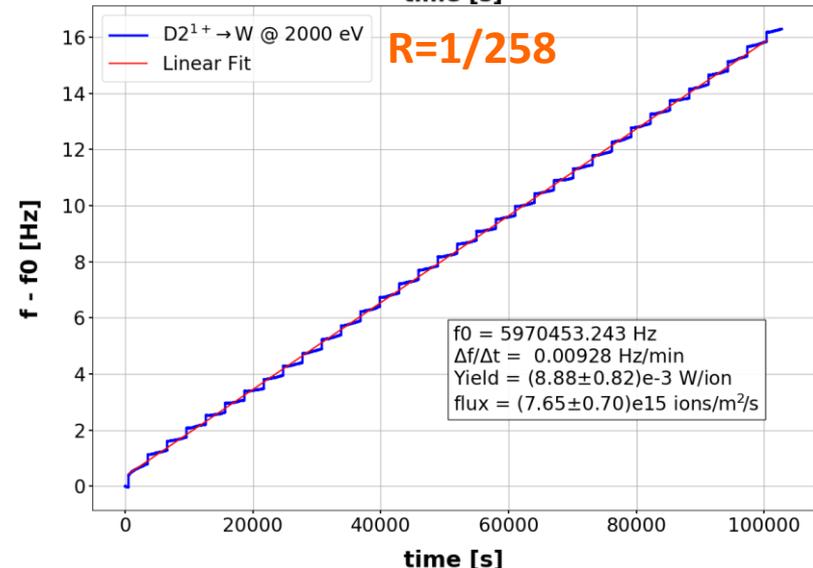
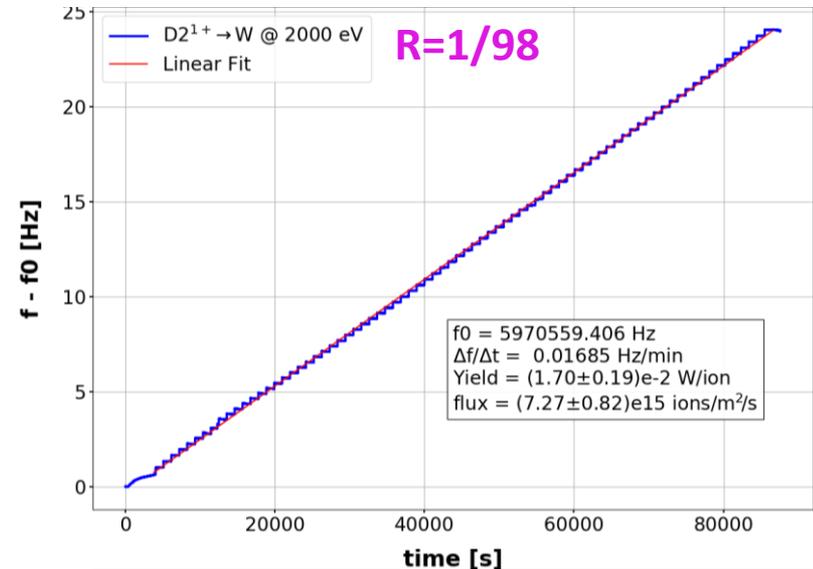
- Approach: Experiment upgrade with a controllable Wien filter.
 - > allows to rapidly alternate ion species during irradiation
 - > 2keV Ar / 2 keV D₂ ions under 0° incidence
 - > W coated QCM for determination of net mass change
 - > f increase = mass decrease
- First, reference sputter yields evaluated for Ar, D₂ only
- Then, quasi-concurrent Ar + D₂ irradiations performed:
 - > desired fluence ratios R = fluence Ar / fluence D₂ (see right graphs) via different step times
 - > evaluation of mean sputteryield via fit through data
 - > comparison to a rule-of-thumb formula, which scales reference sputter yields via the individual fluence ratios

$$Y(R) = Y_{Ar,ref} * R + Y_{D_2,ref} * (1 - R)$$

$$Y\left(R = \frac{1}{98}\right) = 2.02 * 10^{-2} \text{ [W/ion]}; Y_{exp} = 1.70 * 10^{-2} \text{ [W/ion]}$$

$$Y\left(R = \frac{1}{258}\right) = 9.86 * 10^{-3} \text{ [W/ion]}; Y_{exp} = 8.88 * 10^{-3} \text{ [W/ion]}$$

- Reasonable agreement of estimated to experimental values



Key results SP2.4, ÖAW task 1



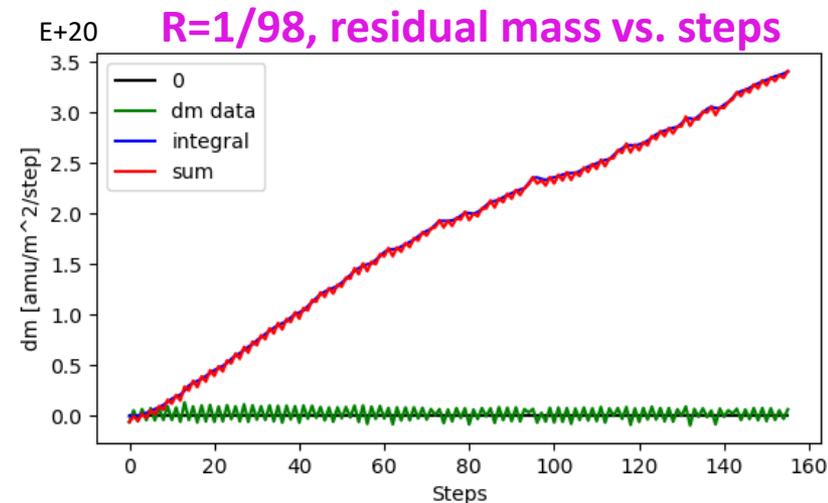
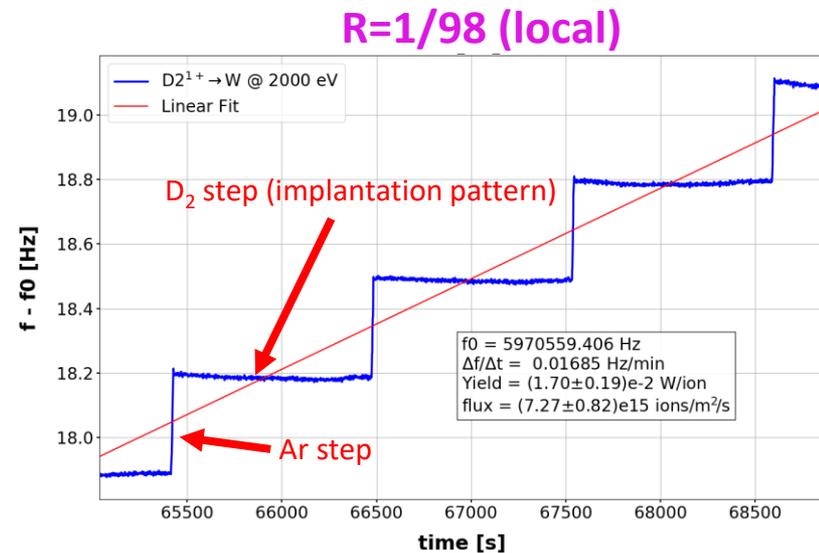
Goal: Quantify synergistic effects on the sputtering yield from concurrent ion beam (D_2 and Ar) irradiation

- Still, the exp. sputter yields are slightly systematically lower
- Local investigation of QCM signal reveals implantation pattern after short Ar steps (right top figure)
- Via subtracting the pure sputtering contribution from the frequency signal, residual stepwise mass changes were calculated, revealing an almost linear net mass increase for rising Ar + D_2 step numbers
- The total mass difference corresponds well to the observed difference in sputter yield values
- **Hypothesis:** Ar enhances D implantation via defect creation
- ToF-ERDA investigations at Uppsala University in near future!

Key result regarding the stipulated task: COMPLETE

Experimental sputter yields fit reasonably well to rule-of-thumb formula, which indicates no severe synergistic sputtering effects for concurrent Ar + D_2 irradiation of model films

- Publication in preparation (2021)



Key results SP2.4, ÖAW task 2



Goal: Validation of SDTrimSP-3D and/or TRI3DYN. (see task 2019)

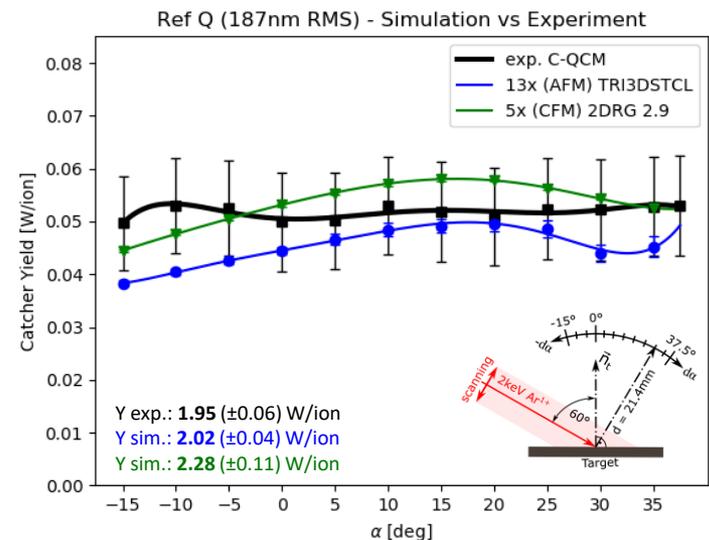
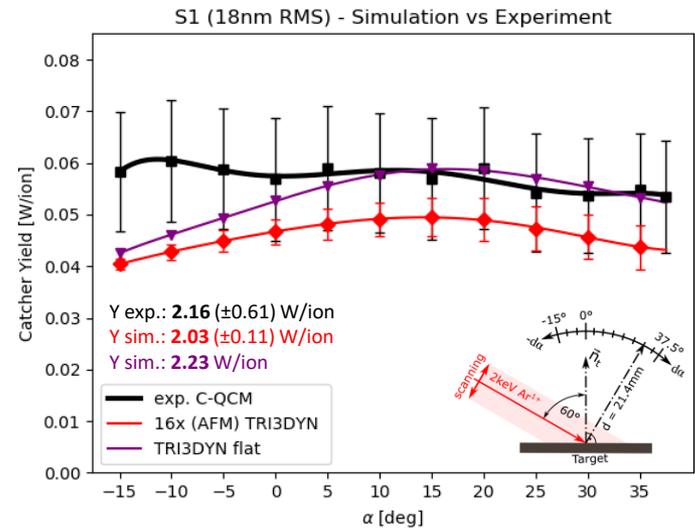
- This task is connected to our 2019 task, which was to investigate sputtering effects from argon ions on corrugated W samples via a Catcher QCM setup.
- Experimental signal obtained by the Catcher QCM (mass increase by deposited W atoms) was reconstructed by consideration of simulation data for sputtered atom and reflected ion trajectories. Further, simulated sputter yields can be compared to the experimentally obtained values.
- The experimental samples RMS values ranged from about 20nm to 1500nm. Surface images obtained via AFM or CFM.
- Following codes were tested (for 2keV Ar → W, 60deg):
 - TRI3DYN (for small RMS)
 - TRI3DSTCL (special TRI3DYN for higher RMS dev. by W. Möller for this task)
 - 2DRG (raytracing code, developed in-house for this task, no topography limit)

1) TRI3DYN for smooth W surfaces (RMS = 18nm, top right fig.)

Good agreement of results for the ideal flat simulation and the 3D surface implementation (16 AFM images considered here). No fit parameters used.

2) TRI3DSTCL+2DRG for rougher W (RMS = 187nm, right bottom)

TRI3DYN not applicable for this roughness, other codes show good agreement. 13 AFM surface images were used for TRI3DSTCL, 5 CFM images for 2DRG. No fit parameters were used.



Key results SP2.4, ÖAW task 2



Goal: Validation of SDTrimSP-3D and/or TRI3DYN. (see task 2019)

3) 2DRG for roughest W surface (RMS = 1500nm, top right)

BCA codes not applicable for such rough textures. 5 CFM images were used for the 2DRG simulations. 2DRG reproduces tendency well, but an offset between the data sets is found. No fit parameters used.

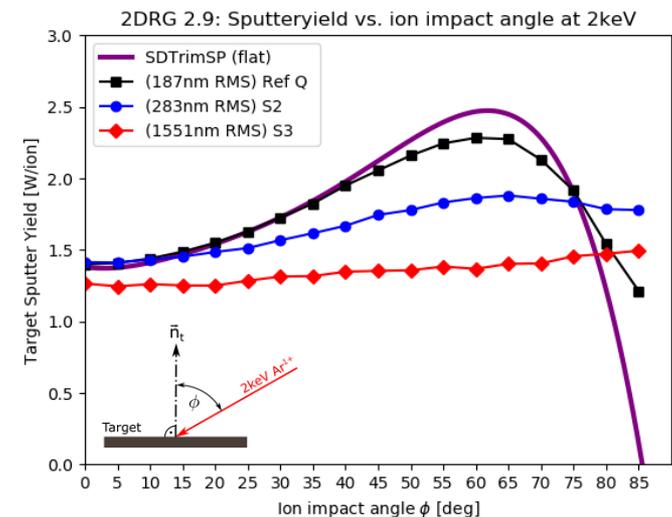
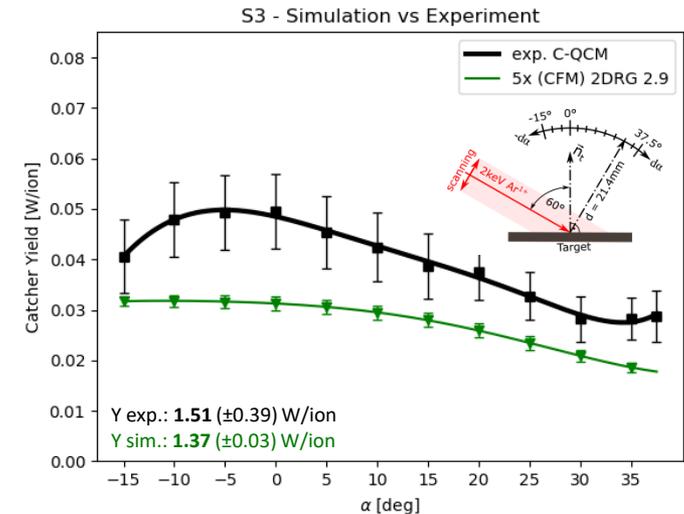
• How works the new developed code 2DRG?

No BCA code. Here, generic TRI3DYN data sets (obtained for flat W surfaces under ion incidence between 0-85°) are considered. Individual ion hit points on the surface are calculated, from which sputtered atom and reflected ion trajectories are launched. Effects like shadowing, redeposition and local ion incidence angles are intrinsically considered via a raytracing approach. There is no limitation regarding the roughness of surface input. Sweep studies are also possible, i.e. for various ion impact angles on a surface (right bottom figure).

• Key result regarding the stipulated task: COMPLETE

- **TRI3DYN** allows to predict sputter yields only for surfaces which are rough on the nanoscale.
- **TRI3DSTCL** is a more applicable tool for rougher textures
- **2DRG** allows a prediction of the experimental data also for more corrugated samples.

• Publication in preparation (2020/21)





SP2.4: LEIS measurements to determine ion reflection of 2018 model films with different roughness

H. R. Koslowski, FZJ

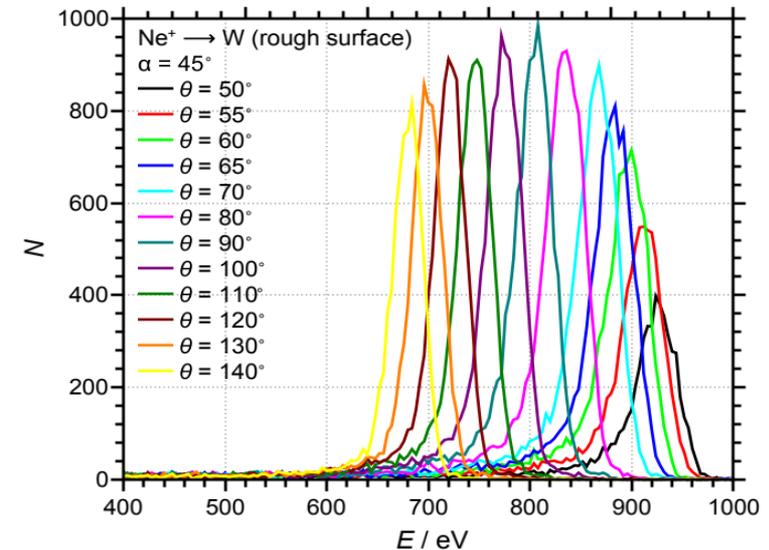
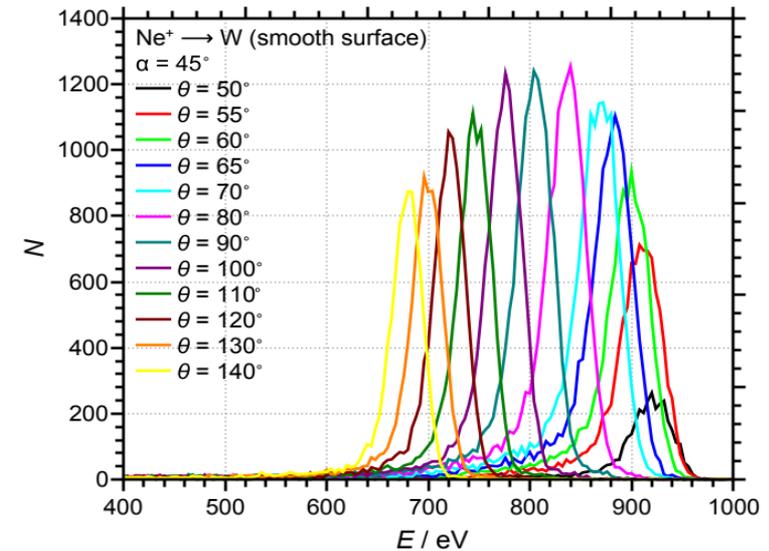


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Key results in 2020



- Measure low energy ion scattering (LEIS) spectra from smooth and rough surfaces
- Derive specific changes and compare to code calculations (e.g. MARLOWE)
- Decompose scattering spectra from rough surfaces in series of ideal scattering spectra from smooth surfaces (“eigenfunctions”) under various angles (which correspond to surface normal vectors, i.e. effective scattering angles)
- Surface roughness will be characterised as distribution of surface normal vectors



Key results in 2020 (cont'd)



- Previously measured scattering spectra had problems with normalisation and needed to be measured again in 2020
- Results (figures on previous slide) of measurements taken under fixed angle of incidence and various scattering angles show that intensity of backscattered ions is lower for rough surfaces, but dependence on scattering angle is less pronounced
- MARLOWE calculations were started and modelling of experimental spectra is in progress
- If successful this method could probably be extended to sputtered particles, too
- Eigenfunction approach could be applied to other codes (e.g. Trim etc.) and may simplify inclusion of surface roughness
- The work has been delayed because the LEIS apparatus was occupied for work on smart alloys [Nucl. Instr. Meth. B 479 42-46 (2020) and Nucl. Mater. Energy 22 100736 (2020)]



WP-PFC SP2.4 Study the temporal surface roughness evolution under plasma exposure and its impact on the erosion and deposition balance

F. Ghezzi, L. De Vecchi, E. Vassallo, M. Pedroni, F. Dell'Era

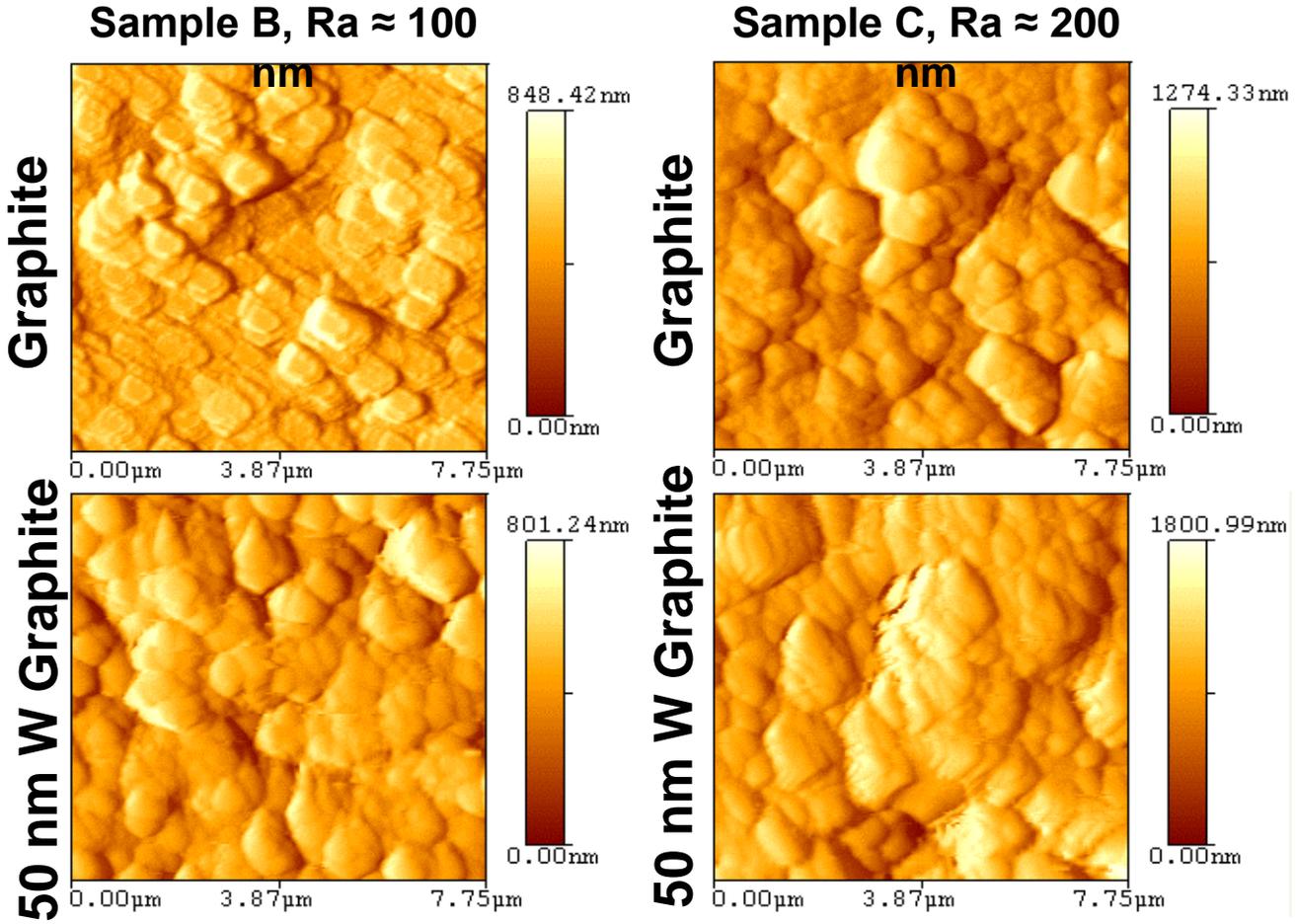


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Task 2019: Deposition of thin W on graphite with different roughness but constant aspect ratio. Characterization with LEIS and comparison with flat surface

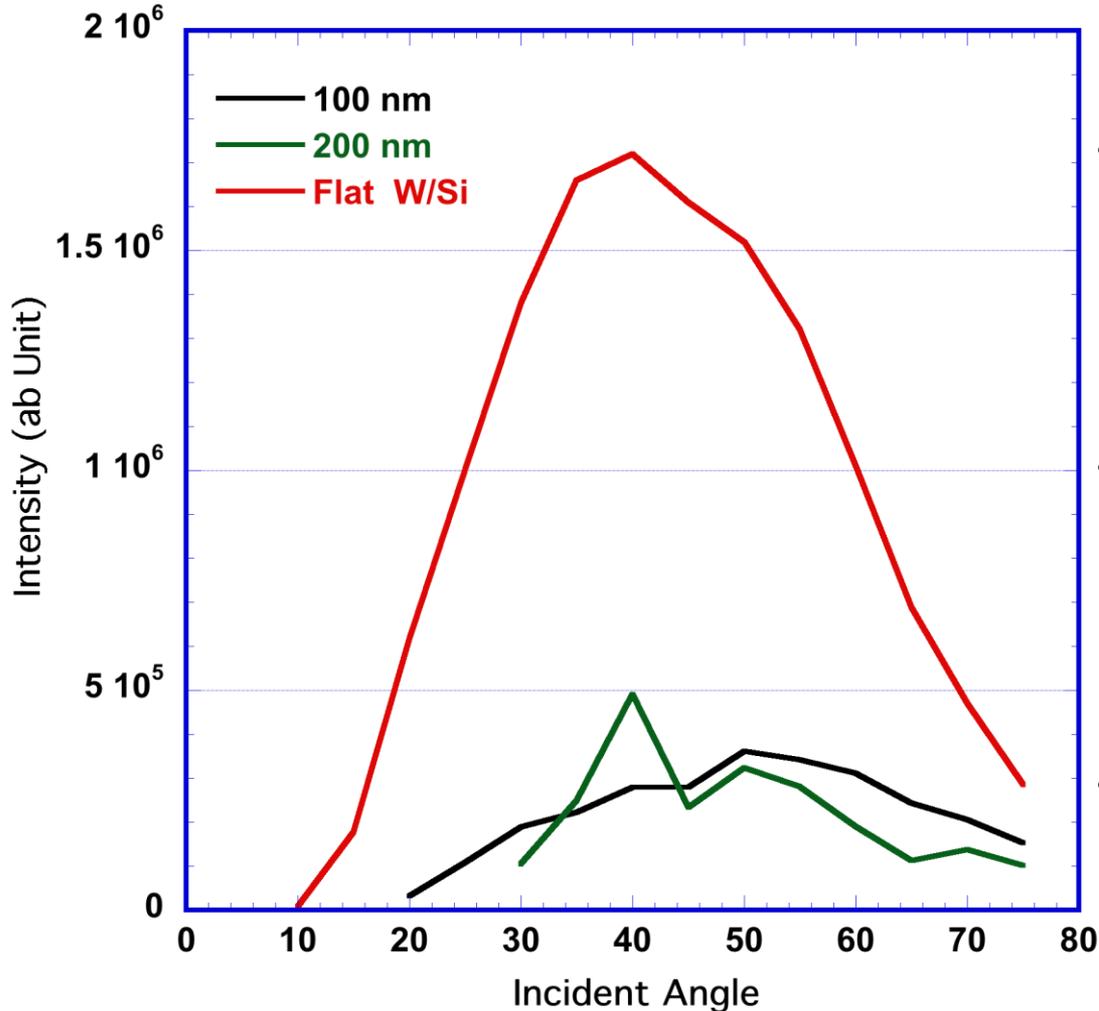
Nanostructurization of graphite by RIE and W deposition (samples for LEIS)





LEIS Results

Dependence of reflection on surface roughness



- For these values of roughness the reflection coefficient is already reduced by orders of magnitude
- No difference between the two values of surface roughness ($R_a \approx 100$ and 200 nm).
- Counts are spread out at different azimuthal angles.

Task 2020: produce samples with various aspect ratio and a constant average roughness



Attempts:

1. Metallic mask via FIB drilling and successive grow by MS deposition
2. Si nanostructurization with defined conical tip via FIB and successive deposition of a thin metallic layer
3. Metallic mask via LASER drilling and successive grow by MS deposition
(hole too large, shape not good).

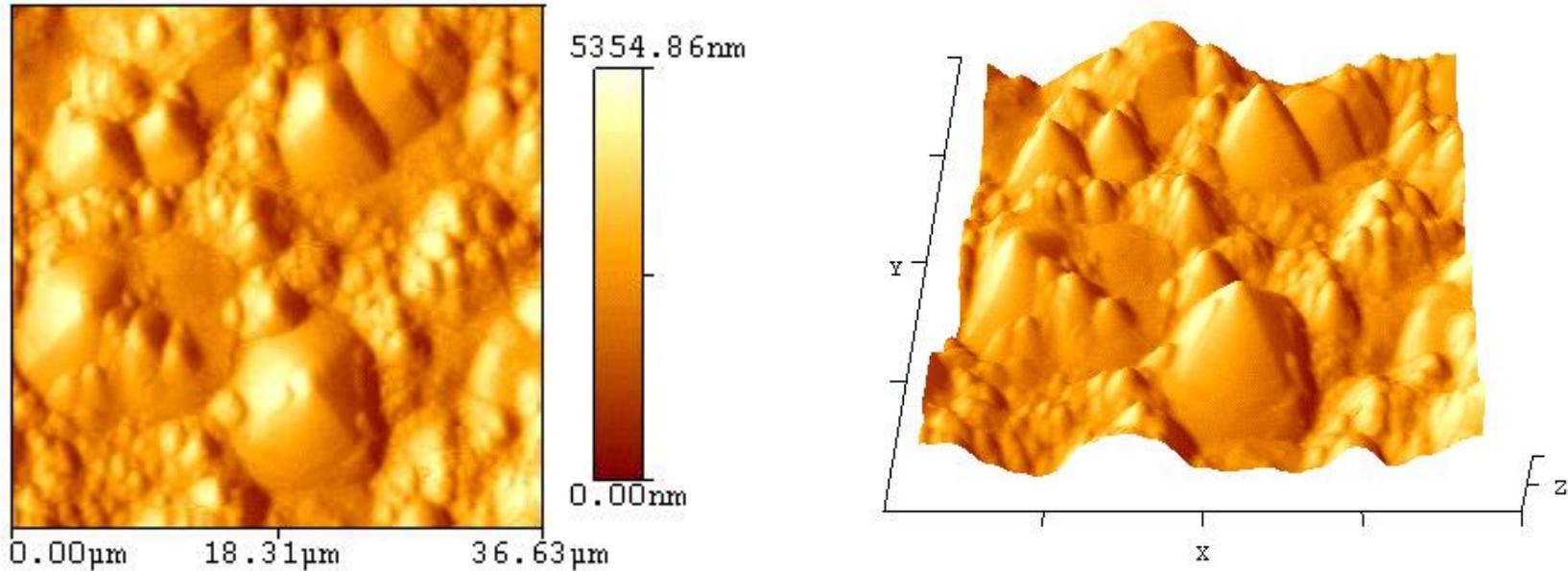
All the three techniques are too expensive.

For example, the production by FIB of a substrate 5 x 5 mm > 3000 € each sample (at least 10 samples needed)

4. Si chemical etching



Si nanotexturing by means of chemical etching (maskless) KOH + IPA + ΔT



- Promising results but damaged AFM. Impossible to optimize the process without AFM feedbacks
- New AFM purchase planned for 2021



SP2.5: High fluence exposure of W in MAGNUM PSI

T.W. Morgan¹, Y. Li^{1, 2}, M. Balden³, S. Brezinsek⁴, G. De Temmerman⁵

¹Dutch Institute for Fundamental Energy Research, Eindhoven, The Netherlands

²Eindhoven University of Technology, The Netherlands

³Max Planck Institute for Plasma Physics, Garching, Germany

⁴Forschungszentrum Julich, Germany

⁵ITER Organization, Cadarache, France



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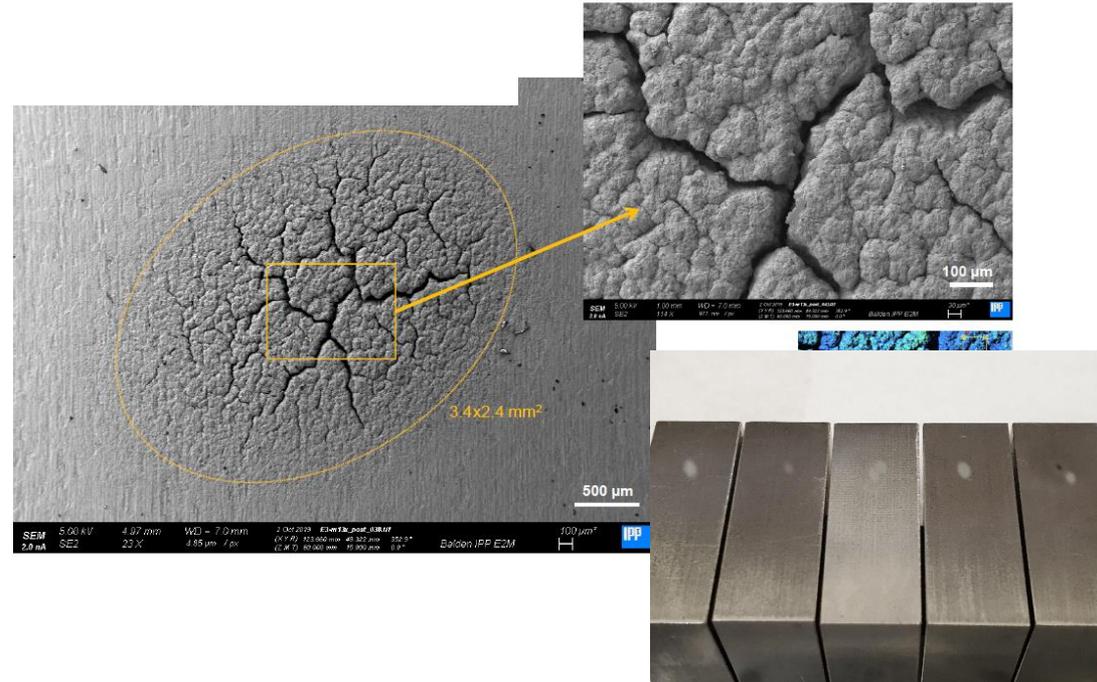


- Simulate ITER steady-state and transient plasma exposure in Full Plasma Operation (FPO) in pure D and He and Ne
- Publications:
 - T.W. Morgan; M. Balden; T. Schwartz-Selinger; Y. Li; T. Loewenhoff; M. Wirtz; S. Brezinsek; G. De Temmerman , **ITER monoblock performance under lifetime loading conditions in Magnum-PSI** , Phys. Scr. 95 (2020) 014065
 - M. Balden; S. Elgeti; T.W. Morgan; S. Brezinsek; G. De Temmerman , **Scanning electron microscopy analyses of an ITER plasma-facing unit mockup exposed to extreme ion fluences in Magnum-PSI** , Phys. Scr. 95 (2020) 014026

Key results in 2020



- Previously exposed ITER monoblocks to very high steady-state fluence in Magnum-PSI to simulate FPO
- Goal to investigate influence of ELMs on W mechanical properties when simultaneous plasma (with seeding) and ELM-like loading close to damage threshold performed on ITER monoblocks at base temperature of 750 °C

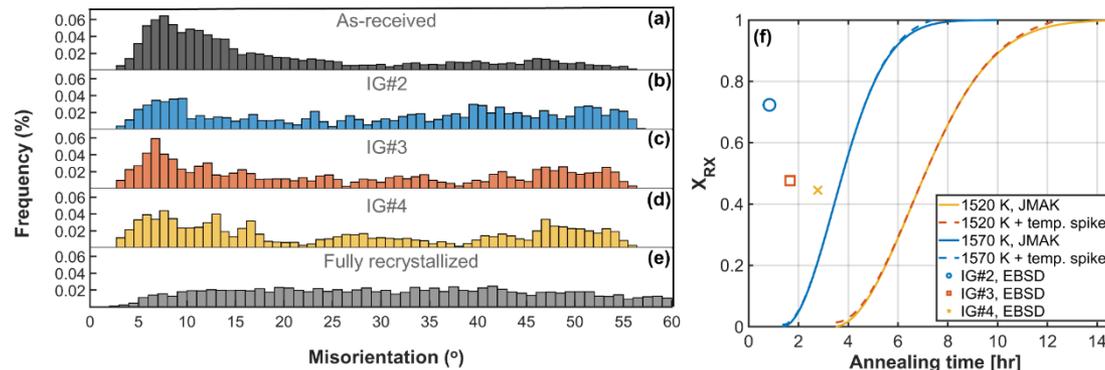


- ITER monoblocks exposed to 12 different loading scenarios in terms of pulse number, seeding rate (pure H, H+Ne, H+He, H+He+Ne) and pulse energy using high power laser

Plans for the future



- Proposed to investigate at higher base temperature:
 - Does lower thermomechanical properties at higher temperature lead to different results?
 - Do recovery and recrystallization play a dynamic role in cracking?
 - Can ELM-like transients drive recrystallization, even below the Rx boundary for the steady-state loading?



Experiments on ELM-like loaded W materials show accelerated Rx below the region exposed to the transient loading compared to JMAK model expectations (Y. Li submitted to Nucl. Fusion (2020))

SP2.5: High fluence exposure of W in MAGNUM PSI

**M. Balden, Max-Planck-Institute for
Plasma Physics**

**M. Balden et al., Phys.Scr. T171 (2020)
014026, "Scanning electron microscopy
analyses of an ITER plasma-facing unit
mockup exposed to extreme ion fluences in
Magnum-PSI"**

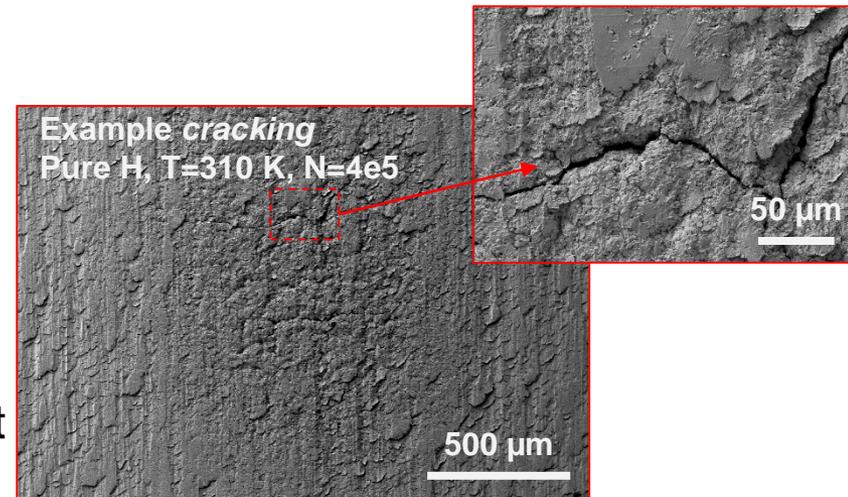
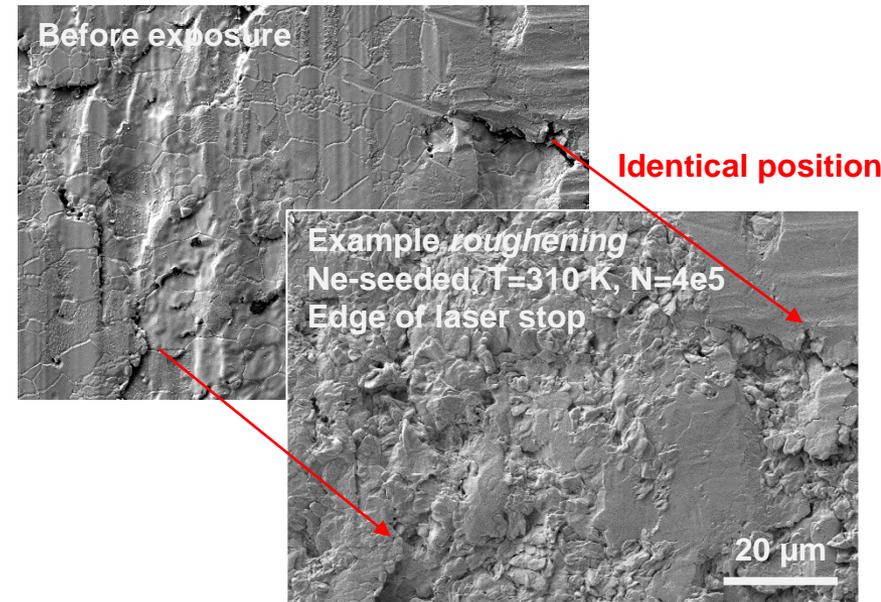


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Key results in 2020



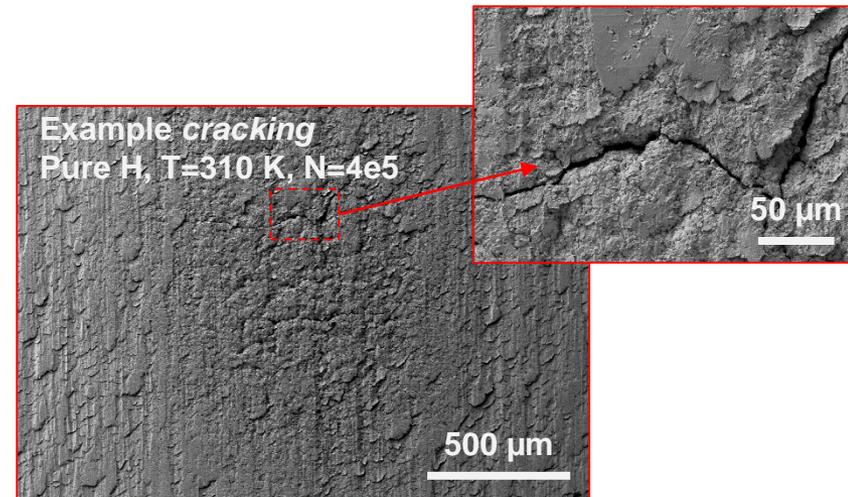
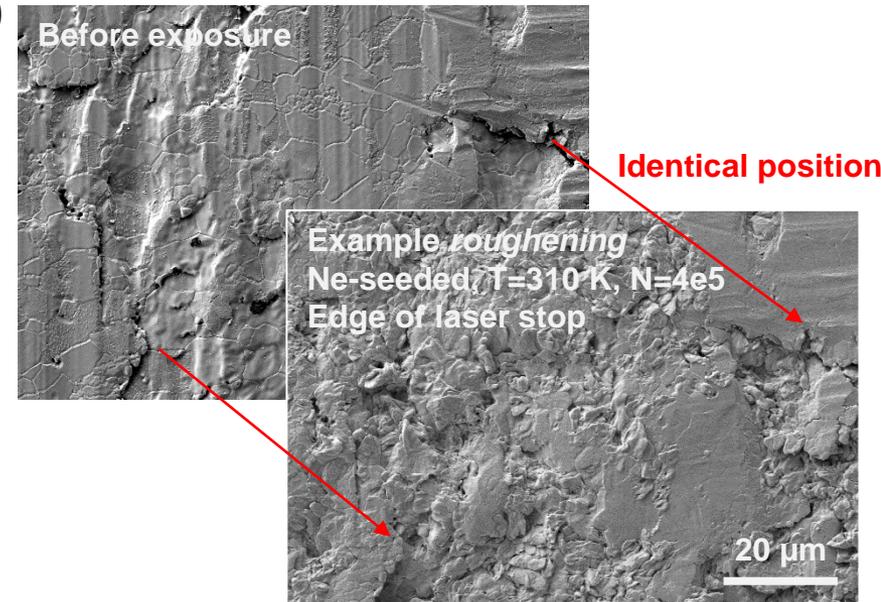
- Simulate ITER steady state and transient plasma exposure in FPO in pure D and He and Ne
- A W-monoblock chain was exposed to pure H and He- and Ne-seeded H plasma beams in MAGNUM-PSI. To simulated transients, additional laser pulses were applied.
- **Microscopic analyses** (mainly with SEM) allow to specify the changes of the W monoblocks, which are ranging from *no damage* via differently strong *roughening* to *cracking* (*melting* not observed) \Rightarrow **subject of IPP task**
- Pre- and post-characterisation of 16 laser spots finished; 5 further position pre-char.; waiting for next exposure (November 2020)
- Attributing the exposures to HHF and compare this to data from electron beam test are the content of the task of DIFFER



Key results in 2020 / Plans for the future



- Last analyses envisaged for December 2020 (slight delay due to slowing down due to COVID-19) ⇒ then task completed
- Publication: T. Morgan et al., *ITER monoblock performance under lifetime loading conditions in Magnum-PSI*, Phys.Scr. T171 (2020) 014065, <https://doi.org/10.1088/1402-4896/ab66df>
- Contribution at upcoming PSI
- **Plans for the future**: Enlarge parameter space of exposure conditions (by vary e.g. base temperature and temperature increase by laser pulses)
- Benefit from amylases capabilities of studying entire W-monoblock chains





- **SP2.1: Eurofer**

- W-enrichment: similar effect of annealing and soft D plasma exposure (always below a factor of two compared to deposited layer), but strong rearrangement of W, enrichment and agglomeration under 200 eV D ion exposure

- **SP2.2: Ammonia qualification and destruction**

- Isotopic exchange of NH_3 with W deuterated surfaces: new method developed for obtaining truly clean W surfaces. Controlled D coverages have been obtained.
- Isotopic exchange for NH_3 flowing after D_2 but not in the $\text{D}_2 + \text{NH}_3$ gas phase exposure.
- Coating with Boron led to a decrease in NH_3 production by a factor of 1.7.
- Ammonia decomposition: superior performance at lower temperatures. Of Rh catalyst but superior NH_3 decomposition activity with LiNH_2 system. Activation energy within the empirical relation of Arrhenius is not the only determining factor of the reaction kinetics.



- **SP2.3: PWI related issues of new materials envisaged as PFM in DEMO with D, He and D seeded discharges**
 - Self-passivating alloy W-10Cr-0.5Y: 20 MeV W irradiation damage increases D retention substantially at 100°C but at elevated temperature (250°C), the retention after irradiation is significantly lower than in W.
 - W_f/W : Sputter yield for W-Fibre close to expected values, for CVD W significantly higher
 - W-Cr-Y smart alloys (SA) and pure W exposed to DEMO-relevant conditions (1% Ar seeding and 1%Ar + 5% He seeding): severe sputtering for both materials, selective sputtering of alloying elements less pronounced in seeded exposures, low D retention for both after seeded exposures



- **SP2.4: Impact of Surface Roughness on erosion and retention**

- Temporal surface roughness evolution under plasma exposure:
 - Experimental sputter yields fit reasonably well to rule-of-thumb formula: no severe synergistic sputtering effects for concurrent Ar + D₂ irradiation of model films
 - TRI3DYN allows to predict sputter yields only for surfaces with roughness of the order of the nanoscale, TRI3DSTCL is a more applicable tool for rougher textures. 2DRG allows a prediction of the experimental data also for more corrugated samples.
- LEIS measurements to determine ion reflection of 2018 model films with different roughness: intensity of backscattered ions is lower for rough surfaces
- Thin W on graphite with different roughness: reflection coefficient reduced by orders of magnitude.

- **SP2.5: High fluence exposure of W in MAGNUM PSI**

- No crack+melting at HFF~12: plasma rounds off edges so no edge melting
- Roughening vs small cracks seen with e-beam: not seen due to initial technical finish or due to more freedom for plastic deformation
- Surface modifications such as corrugation and erosion lines seen: plasma + laser synergy?