Production, characterization, and exposure of B reference samples

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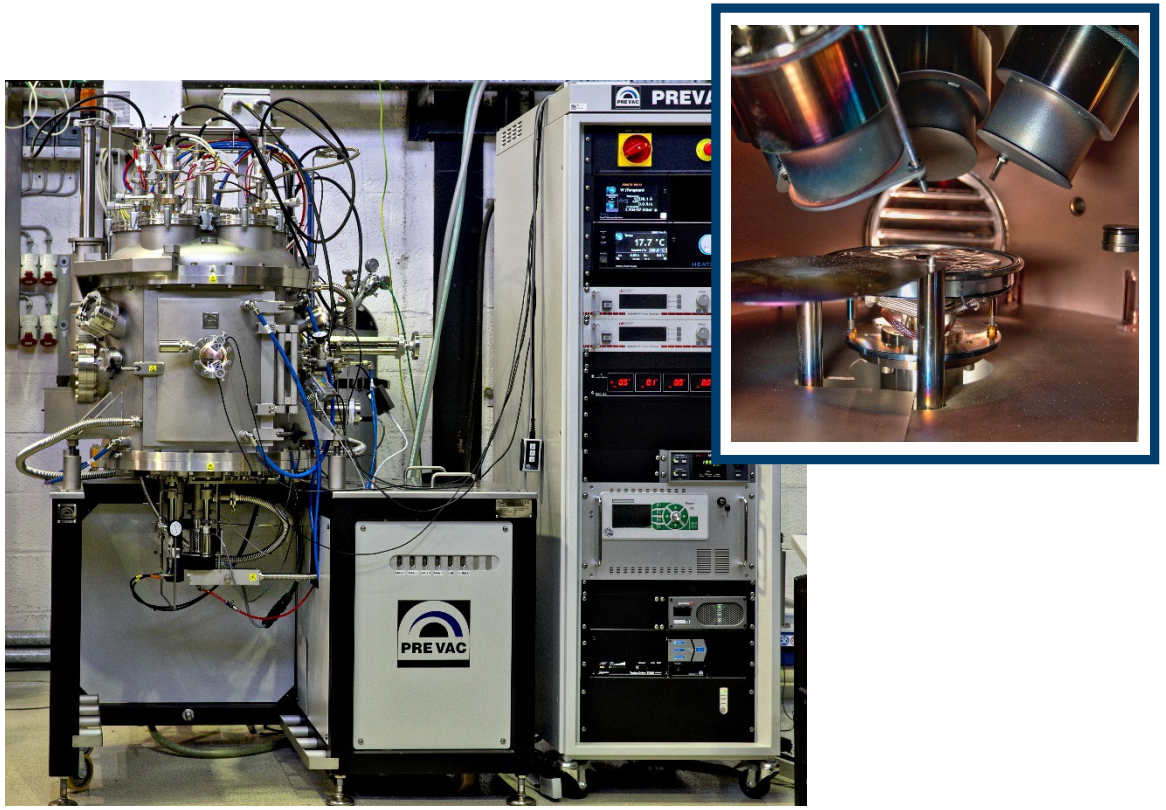
B layer fabrication

Boron layer fabrication in laboratory: Magnetron sputter deposition



- 4 magnetrons, simultaneous operation possible
- Base pressure 10^{-7} mbar
- Rotating sample stage -> homogeneous layer
- Biased and heatable sample stage
- Gas injection ring close to sample for reactive sputtering (oxygen, hydrogen, nitrogen)
- Large sample stage

RF-mode, Ar plasma, pure boron target

→ **Pure, amorphous boron layers** (reliable process parameter)

→ B:D layers (first tests performed)

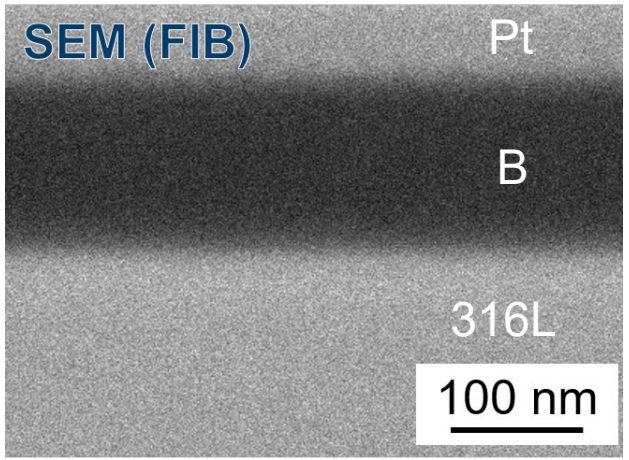
→ B:W layers (foreseen for the next months)

Substrates: polished W and 316L-IG steel

B layer fabrication and characterization

Boron layer fabrication in laboratory: Magnetron sputter deposition

- Pure boron, 5.5 h deposition -> ~100 nm thickness, W and steel substrates
- Annealing at 1000°C (W) or 550°C (steel) in vacuum → stable, no peeling, most cases no change of layer

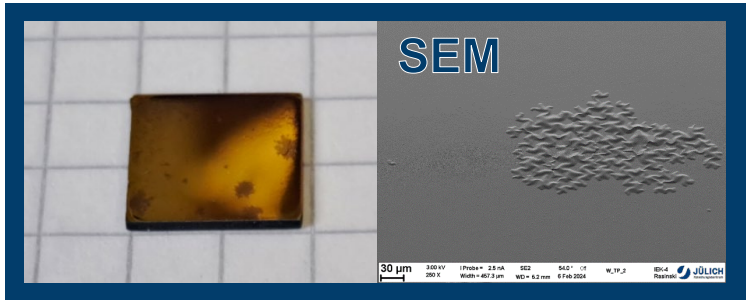


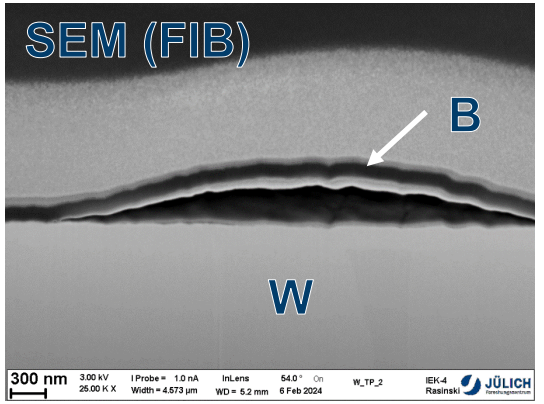
Characterization of B layer after deposition:

- XRD: amorphous
- SEM(FIB): smooth, dense, homogeneous layer
- EDX: oxygen on interface to W or steel, less in B layer, but hard to interpret
- XPS: pure B (no B_2O_3)
- Ongoing: further improvement of characterization techniques (B:D, B:H and B:O)

Interesting feature of B on W:

- B on W: after some weeks: purple dots/whole area purple -> detachment of B layer from substrate -> ‘blister’, dependent on W substrate surface before deposition, especially W oxide
- B layer properties do NOT change (XPS: no change of B signal, no increase in O)
- **Annealing** of W substrates before deposition and **keep under vacuum!**





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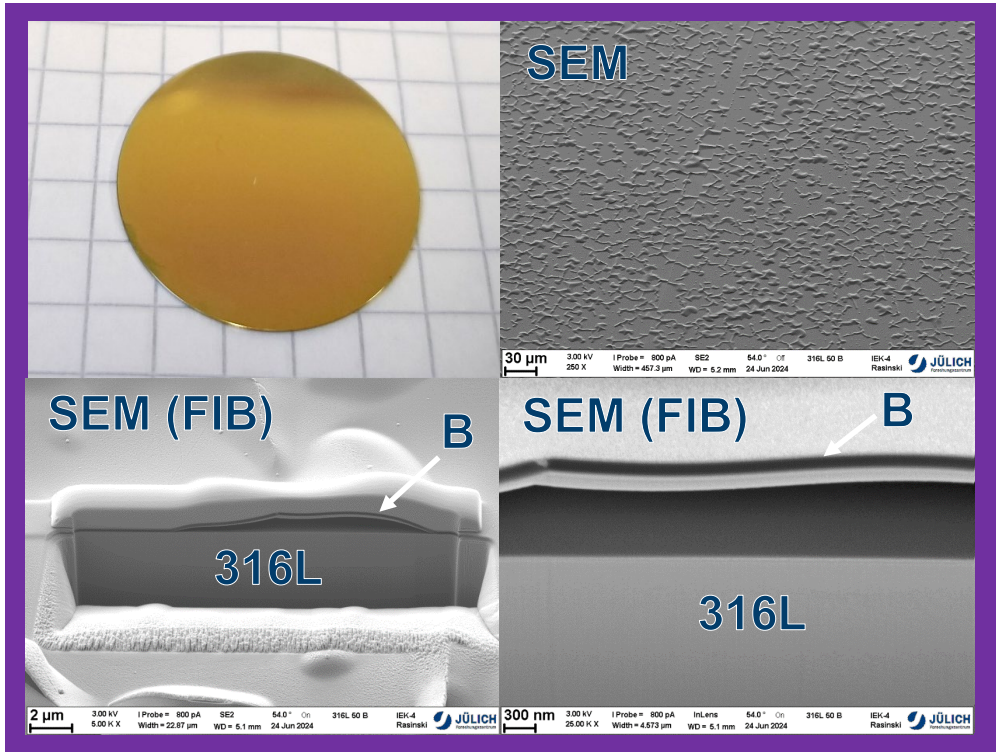
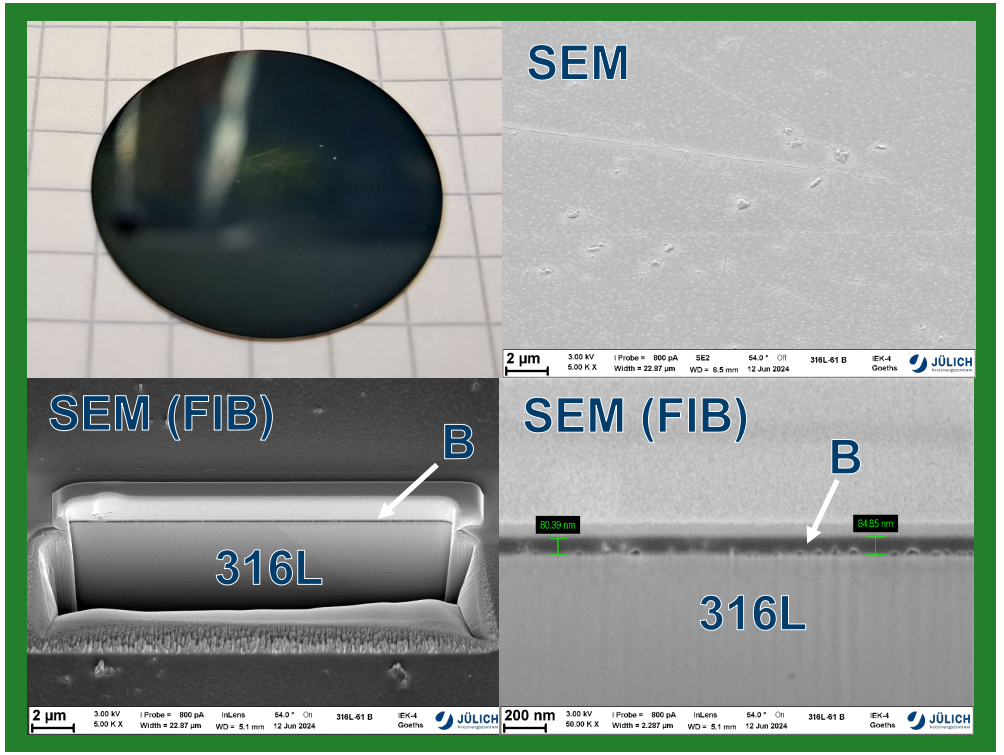
B layer fabrication and characterization

Interesting features of B on 316L steel:

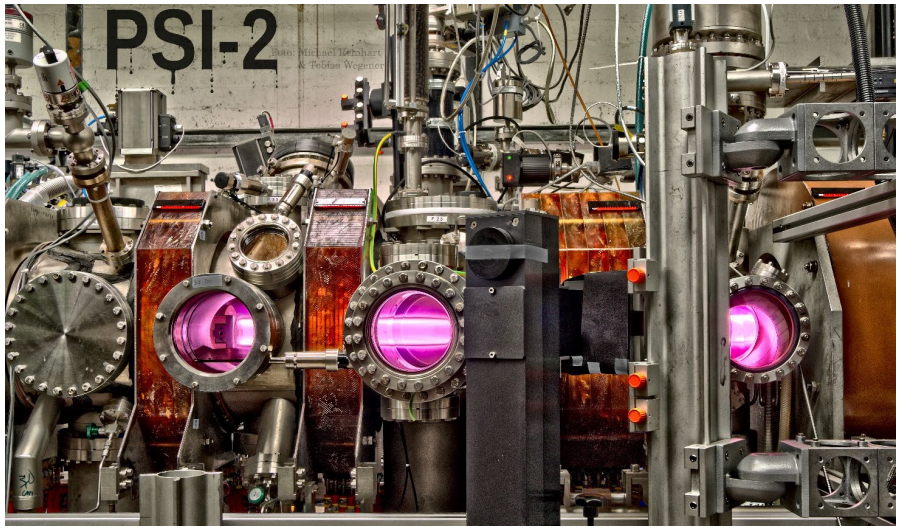
- ‘Blister’ formation after longer time (~ several months) in air (humidity?)
  → B on steel samples should be kept **under vacuum** as well!
- Change of color and B layer thickness during annealing at 550°C on **some** samples
  → First assumption: oxidation, but no change of B and O signal in XPS observed so far
  → XPS shows Fe signal → Fe in the B layer? Why on some samples only? Interface?





→ Influence of sample substrate and interface: substrate preparation and characterization has to be done carefully!
→ **Keep substrate surface clean and sample under vacuum!**

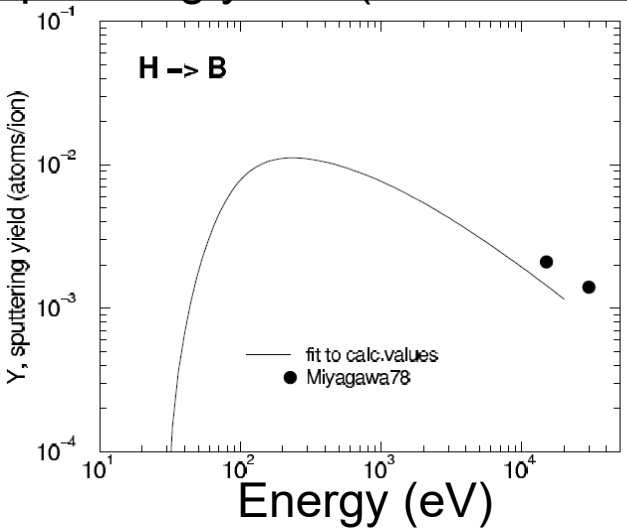
Erosion of B layers by D/H plasma

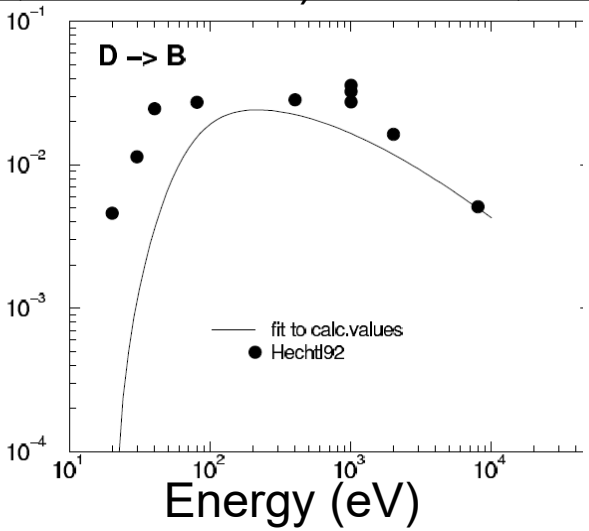


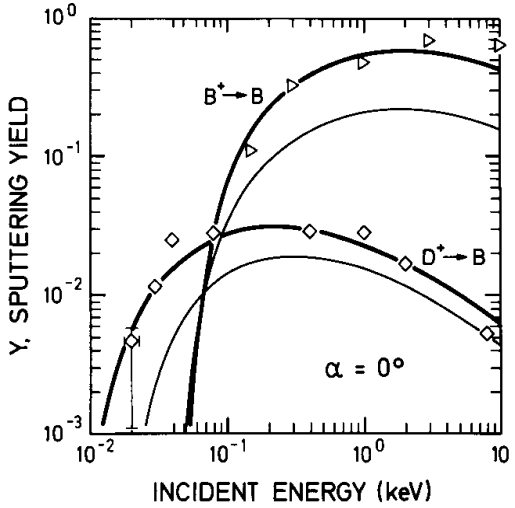
Plasma

PSI-2: → Linear plasma device, various gases are possible (D, H)
→ Incident ion energy: ~10 - 300 eV, flux: up to 10^{22} $m^{-2}s^{-1}$
→ First try: B layer was removed completely
→ Second try: successful, see next slide

Sputtering yields (Behrisch 2006, Hechtl 1992) H/D -> B, O/W -> B?, chem. Erosion?







Erosion of B layers by D plasma

Boron layer fabrication: Magnetron sputter deposition at FZ Jülich

- RF-mode with pure boron target and Ar plasma, ~ 6 h deposition -> ~115 nm thickness on polished and annealed W PSI-2 substrates -> Fig. 1

Deuterium plasma exposure at PSI-2 for B erosion studies:

- Deuterium plasma: ion energy: ~43 eV; density: 6e21 $m^{-2}s^{-1}$
- Simultaneous imaging spectroscopy for boron, calibrated
- Non-cooled PSI- sample holder with Mo mask (Fig. 2)
- Two exposures: identical plasma conditions, identical samples, different exposure times: a) 70 s, b) ~26 min

Comparison: B erosion time from FIB/SEM analysis and spectrometer data:

- Case a) 70 s: first try to see B by spectrometer: visible remains of boron layer, PSI-2 profile visible (Fig. 3) -> FIB/SEM studies
- Case b) ~26 min: no remains of boron layer, detailed imaging spectrometer signal data -> Analysis of spectrometer data

Estimation of B layer erosion rate possible!

Fig. 1

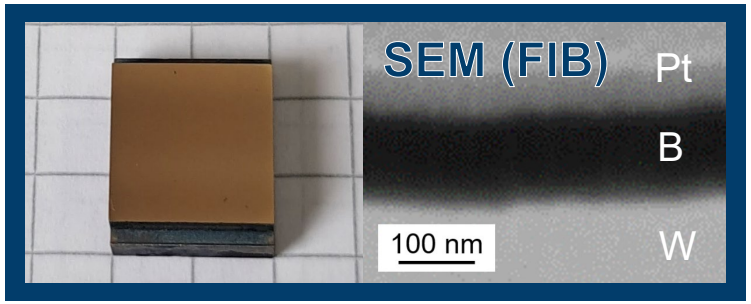


Fig. 2 W with B

Other 7 sample: W dummy

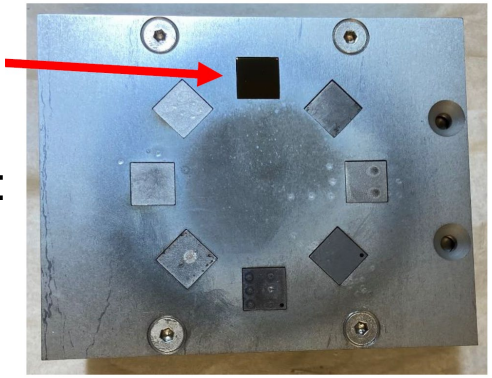
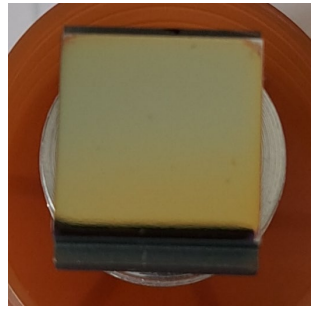


Fig. 3



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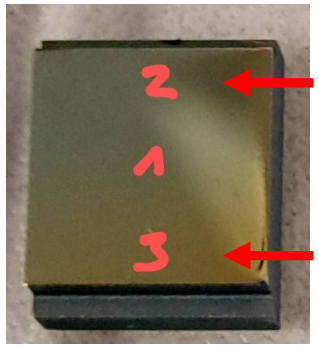
Erosion of B layers by D plasma

Comparison: B erosion time from FIB/SEM analysis and spectrometer data:

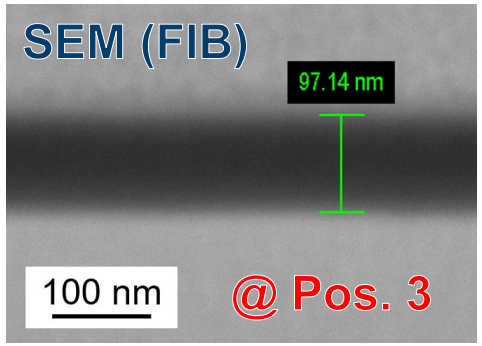
Case a) 70 s plasma exposure

- Measurement of the remaining B layer thickness at three different positions by FIB/SEM:



higher erosion

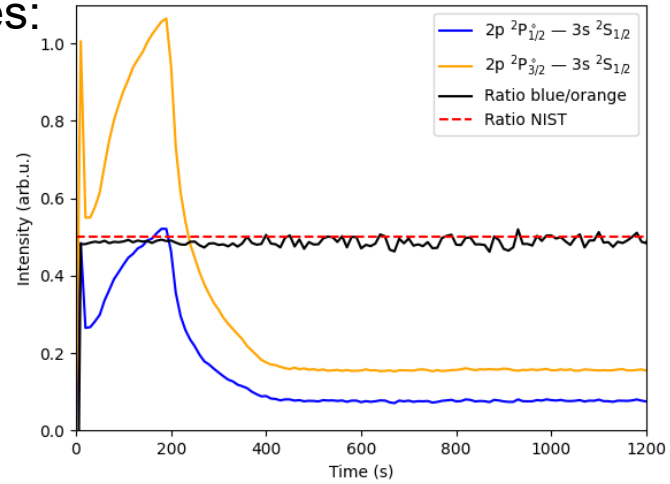
lower erosion



- Higher erosion region: Pos. 2: ~85 nm B -> erosion rate: **~0.43 nm/s**
- Lower erosion region: Pos. 1 and 3: ~97 nm B -> erosion rate: **~0.26 nm/s**

Case b) ~26 min plasma exposure

- B I line intensity versus exposure time:
- Interpretation: 3 zones:
  1) up to 200 s: high erosion zone
  2) 200 s to 400 s: low erosion zone
  3) from 400 s: background signal



- Higher erosion: zone 1 -> erosion rate: **~0.575 nm/s**
- Low erosion: zone 2 -> erosion rate: **~0.29 nm/s**

- Please note: Preliminary results, further analysis and estimation of errorbars is ongoing!

Very good agreement of erosion rate between the values obtained by FIB/SEM and spectroscopy!
+ It is possible to observe the boron (erosion) by spectroscopy!

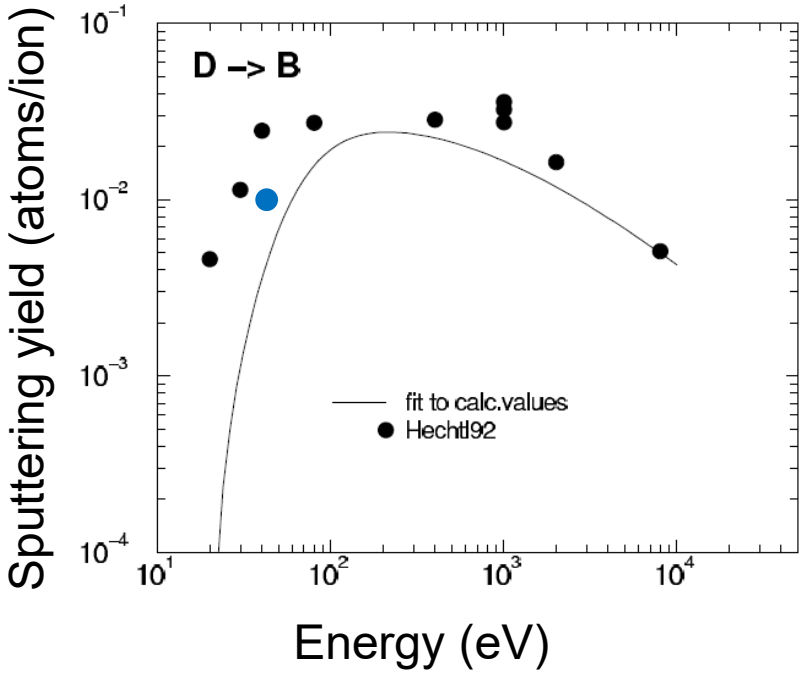
Erosion of B layers by D plasma

Comparison: B erosion by deuterium with published data

- Calculation of the sputter yield from Case a) for higher erosion region:
  - Erosion rate: ~0.43 nm/s
  - Boron density: 2.4 g/cm^3
  - Ion energy: ~43 eV
  - Ion density: 6e21 m^{-2}s^{-1}

Sputtering yield: 0.01 atoms/ion

Sputtering yield (Behrisch 2006, Hechtl 1992)



- Please note: Preliminary results, further analysis and estimation of errorbars is ongoing!

Outlook:
→ Other ion energies and temperatures at PSI-2
→ Enhancement of the spectroscopy measurements
→ Hydrogen isotope retention measurements (TDS/NRA)
→ Investigation of erosion rate of ‘mixed’ B layers