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Hydrogen Isotope Interaction with Boron Layers

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Motivation

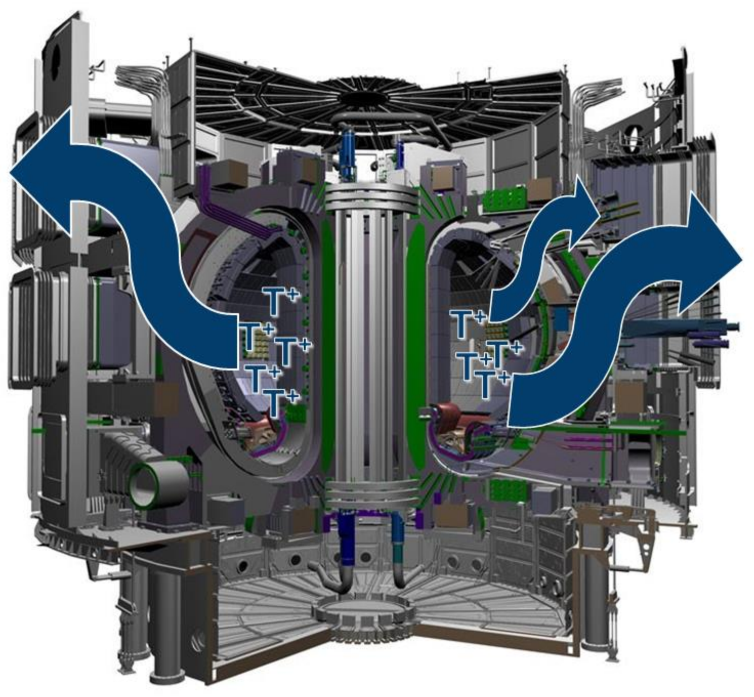


Figure (edited): http://www.iter.org/

Hydrogen isotope permeation and retention in fusion devices:

- Fuel loss and unpredictable fueling
- Tritium accumulation

⬇

The study of hydrogen interaction with fusion materials is necessary!

Due to the ITER re-baseline: Fabrication and investigation of thin boron coatings.

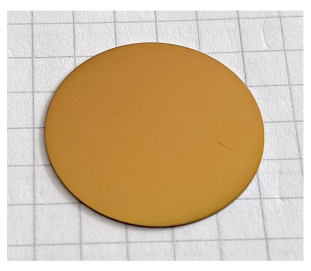
Samples

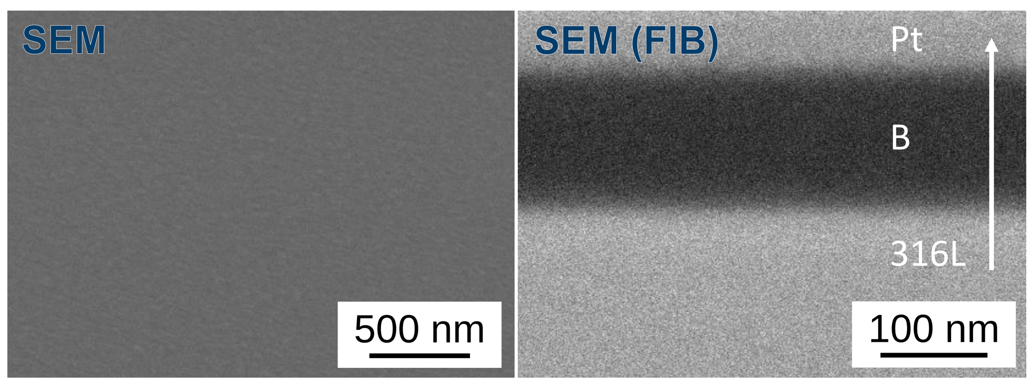
Sample preparation:

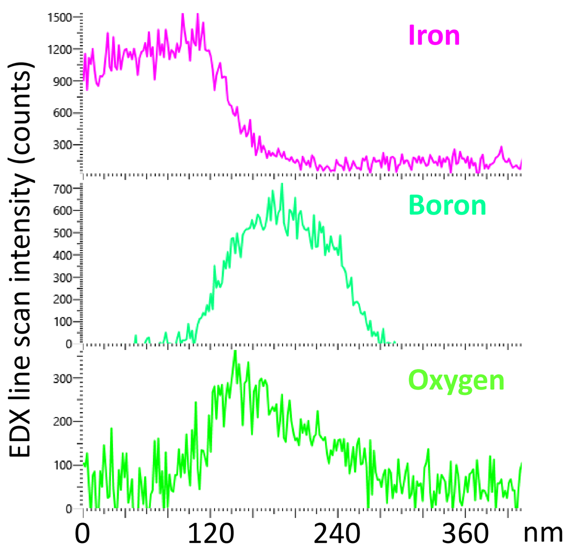
→ Magnetron sputter deposition: RF-mode with pure boron target and Ar plasma
→ Coated on both sides with same deposition parameter (5.5 h deposition)
→ Substrates: polished 316L-ITER Grade, 24 mm in diameter, 0.3 mm thickness (and polished W (Plansee))
→ Annealing at 550°C in vacuum → stable, no peeling, no change of layer

Sample characterization:

→ SEM: smooth, dense layer
→ SEM(FIB): - layer thickness: 110 nm per side
- homogeneous layer
→ EDX: oxygen on interface to steel, less in B layer
→ XRD: amorphous

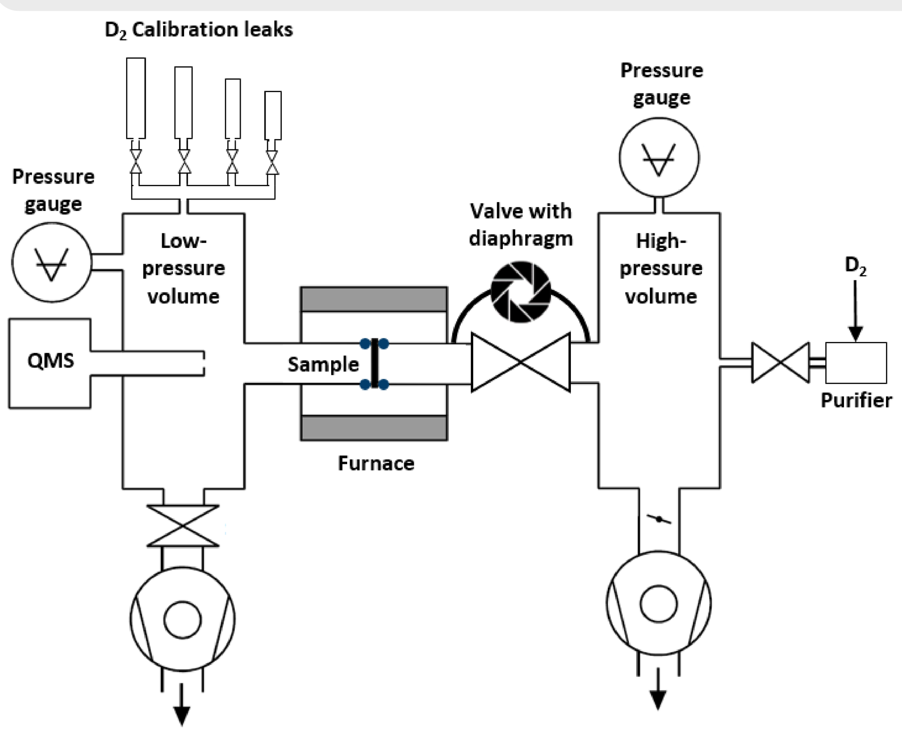






→ B layer on W: see **Excurse – Boron Layers**

Permeation Measurement

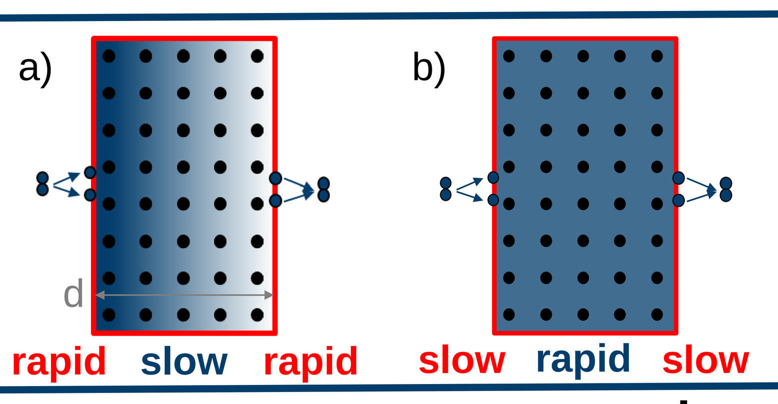


Device specifications:

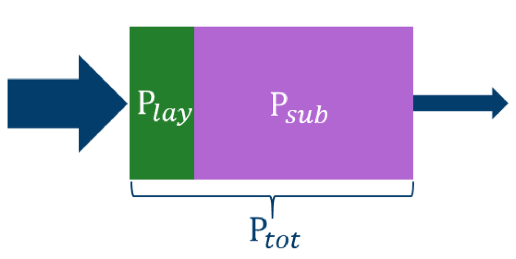
- D_2 purifier → no oxidation of sample during measurement
- Installation of a valve with baffle
  → rapid D_2 inlet → 'Lag-time'
  → determination of diffusion (D)

Measurement procedure:

- Evacuation (10^{-9} mbar), calibration
- Temperature (T) range: 300 - 550°C, up/down (sample stability)
- Pressure (p) range: 25-800 mbar
- Lag-time measurement



Pressure dependence (p): 25-800 mbar: permeation control mechanism
(J_P = permeation flux):
a) $J_P \sim \sqrt{p}$: diffusion limited, dependent on sample thickness (d)
b) $J_P \sim p$: surface limited, independent on d



Layer permeability:

- Substrate and layer thickness independent
- Valid in diffusion limited regime

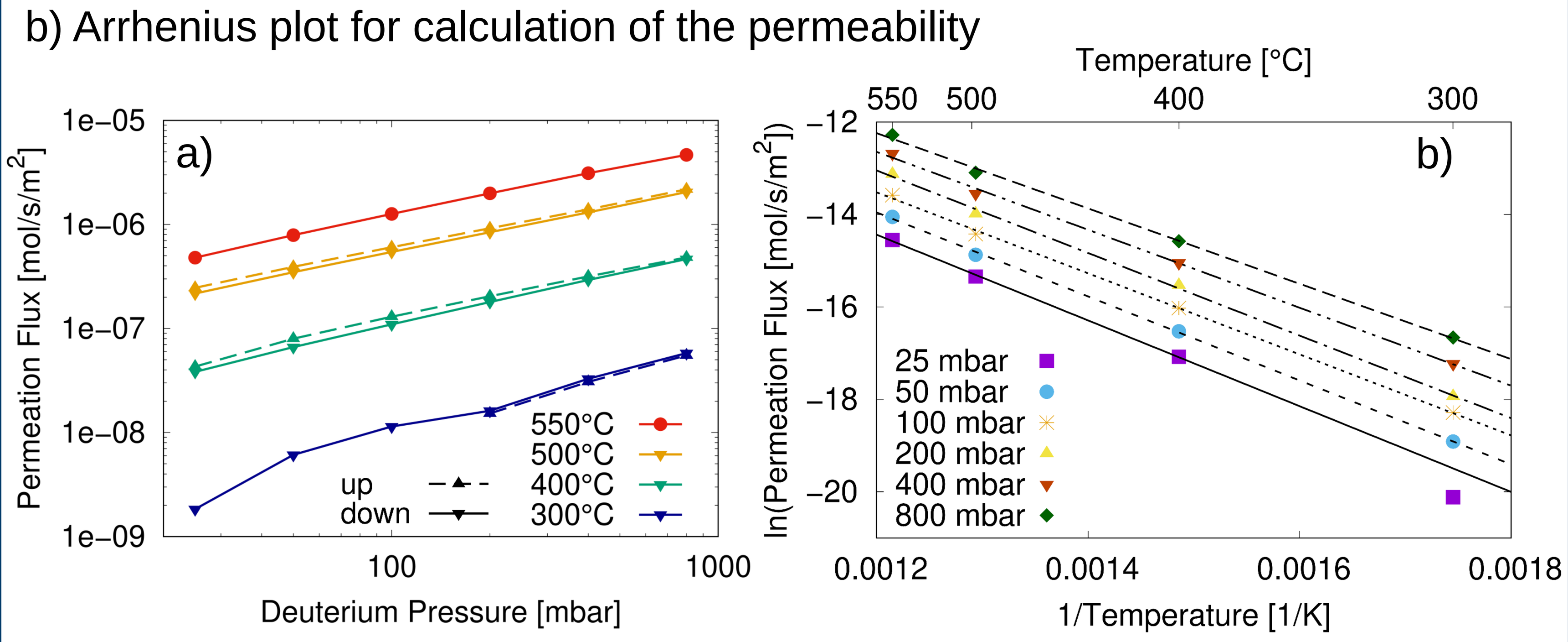
$$P_{lay} = \frac{d_{lay}}{\left(\frac{d_{tot}}{P_{tot}} - \frac{d_{sub}}{P_{sub}}\right)}$$

lay: layer
tot: total
sub: substrate

Results

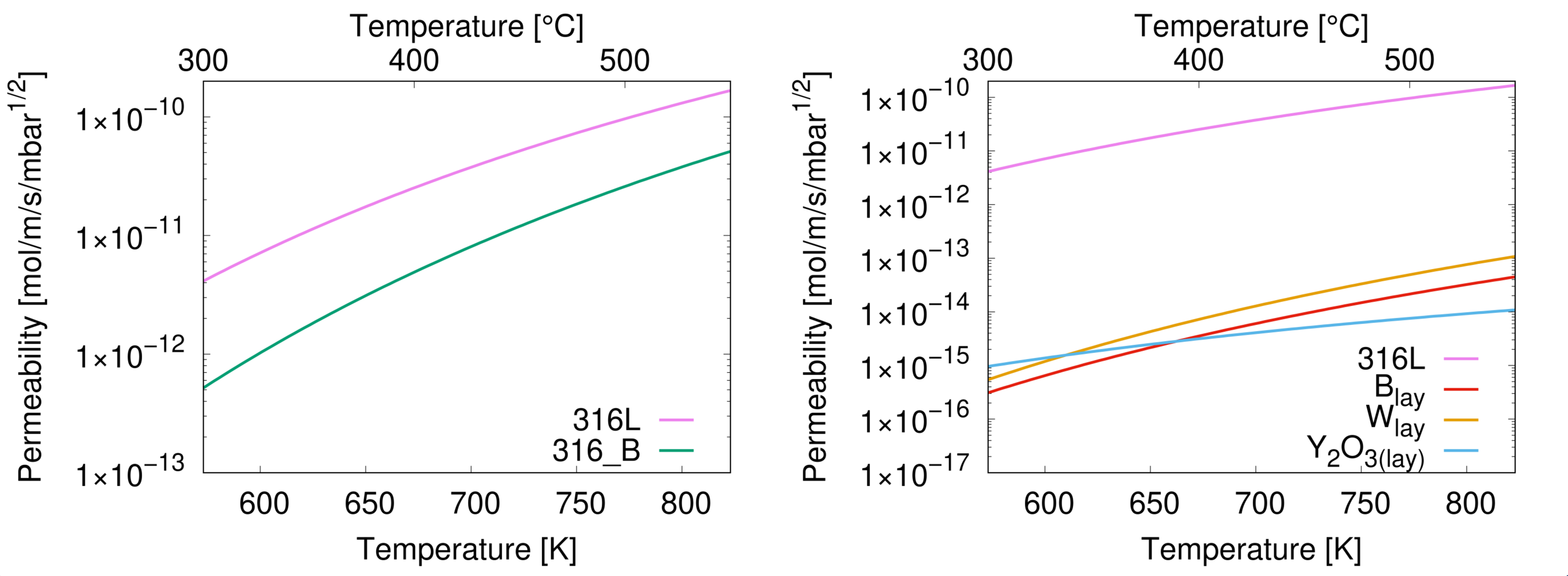
Permeation flux (J_P) measurement through the coated sample:

a) Permeation flux vs. applied deuterium pressure for all temperatures
b) Arrhenius plot for calculation of the permeability



Comparison up/down measurement: No change of sample
Pressure dependence: $J_P \sim p^{-0.65}$: Diffusion limited to intermediate regime
p/T-dependent measurement: $J_P = \frac{P_0\sqrt{p}}{d} e^{\frac{-E_P}{RT}}$ → permeability + layer permeability

Sample	$P_{0(tot)}\left[\frac{mol}{ms\sqrt{mbar}}\right]$	$E_{P(tot)}\left[\frac{kJ}{mol}\right]$	$P_{0\,(lay)}\left[\frac{mol}{ms\sqrt{mbar}}\right]$	$E_{P(lay)}\left[\frac{kJ}{mol}\right]$
316L	$8(1) \cdot 10^{-7}$	58(1)	-	-
316L_B	$1.9(5) \cdot 10^{-6}$	72(2)	$4 \cdot 10^{-9}$	78



Conclusion and Outlook

Conclusion:

- The permeation flux is reduced by about one orders of magnitude due to thin (~220 µm) B coating compared to a non-coated 316L substrate
- B_{lay} permeability around four orders of magnitude lower as permeability of 316L substrate
- B_{lay} permeability in the same order of magnitude as W_{lay} and the permeation barrier $Y_2O_{3(lay)}$ permeability
- **Low hydrogen permeability of pure, amorphous B layers**

Outlook:

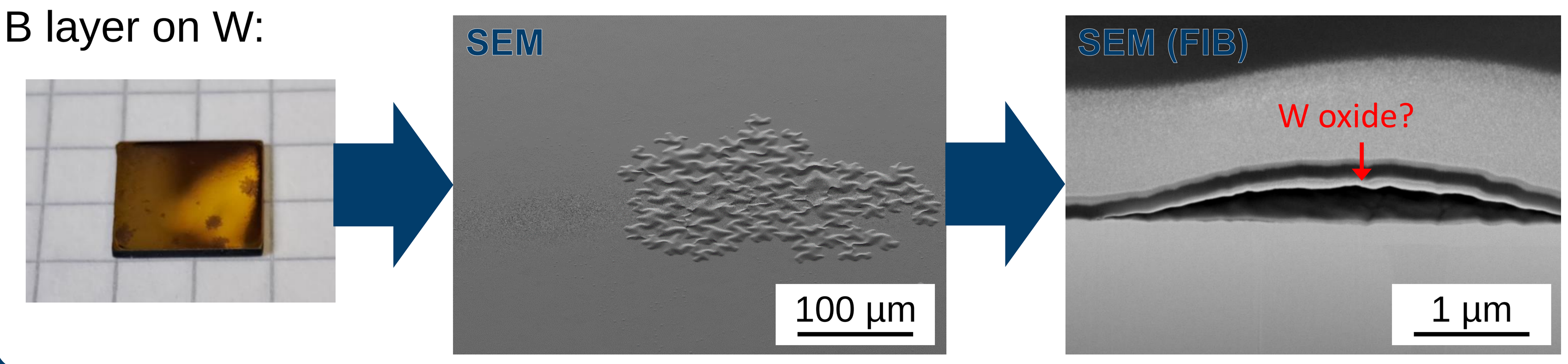
- Ongoing study of permeability of one side B coated 316L substrate
- Investigation of permeability of mixed B layers, e.g. with W, O, D
- Deuterium retention measurements on D_2 filled B layers on W and 316L substrates
- D_2 filling of B coated samples by gas, plasma and ions
  → Investigation of sputter rate from D plasma and ions on B coating

Excurse – Boron Layers

Magnetron sputter deposition of pure boron layers on W substrates:

- Polished and annealed W samples: 1 x 1 cm² and PSI-2 geometry
- Same deposition parameter and B layer characteristics as on steel substrates
- BUT: after some time (~weeks) development of purple dots/blister: W oxide?
  → study ongoing (EDX, XPS), keep under vacuum after polishing / annealing
- Annealing up to 1000°C → no change of sample state

B layer on W:



Related publications: A. Houben *et al.*, NME **19** (2019), 55-58; A. Houben *et al.*, Plasma and Fusion Research **15**, 2405016 (2020); A. Houben *et al.*, NME **37** (2023), 101518

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