EUROfusion

SP B monitoring meeting 2024

SP B.4 - production and characterization of B reference samples

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Objectives and strategy

- **SP-B4:** Characterization of atomic composition, thicknesses, and depth profiles of sputter-deposited B and W+B using Ion beam Analysis from samples produced in different facilities (not this presentation).

- Synthesis in-house (Tandem Laboratory at Uppsala University) of sputter-deposited layers (MAT-ENR 2024-2025 project):

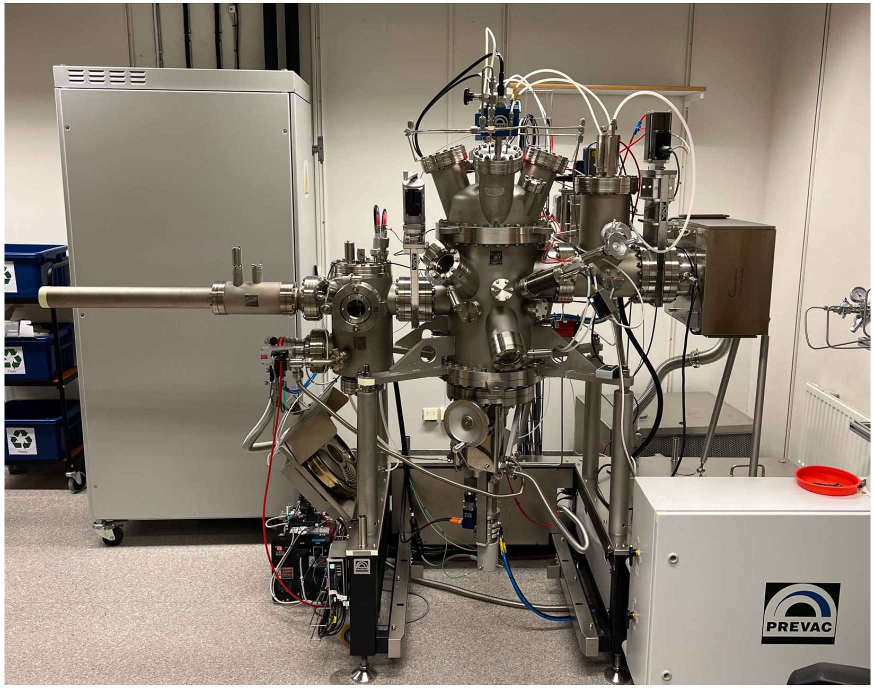
- Preliminary investigations of sputtering yields for B layers on QCM (TU-Wien).
- Explore the use of **in-situ ion beam analysis** in the investigation of the real-time modifications:
  - Analyze atomic composition and distribution in samples before/during/after ion irradiation and/or annealing without air exposure.
  - Identify surface enrichment, material segregation and H/D retention.

First steps:

- Growth and characterization of sputter-deposited layers from B and W targets (co-deposition) in Ar and $Ar+D_2$ atmospheres in different substrates (ex: W, QCM, Si, C, and MgO).

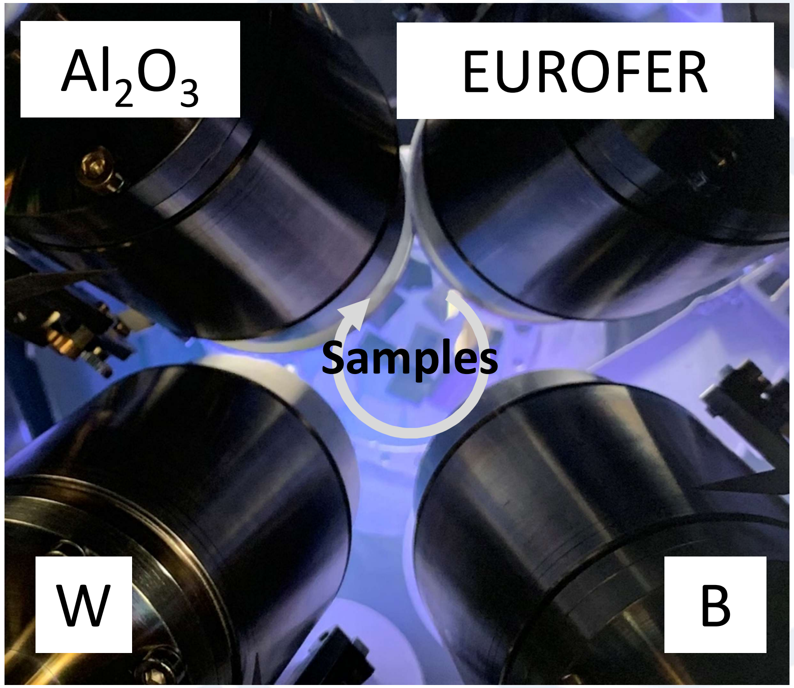
Tandem Laboratory @ Uppsala University

Sputtering machine for film deposition



Prevac sputtering machine:
4 Magnetrons (2 DC and 2 RF).
Deposition in Ar and Ar/D_2 mixed plasmas.
Base pressure < 10^{-7} mbar.
Possibility of annealing during deposition (up to 1000°C).
E. Pitthan et al. Nucl. Mater. Energy. 34 (2023).

Top-view:



Simultaneous deposition of W and B under different conditions to obtain different ratios of W and B.

W-B film characterization (100-150 nm films)

Sputtering depositions under argon atmospheres:

B target (RF-150 W):

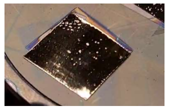
0.5 nm/min; 5 hours deposition.

Substrate effect:

- <u>Layers are stable in vacuum.</u>
- After air exposure (within minutes): roughness on W substrates.

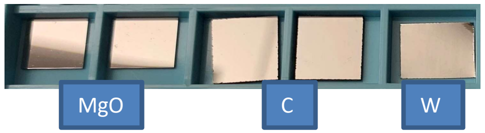
Improved on W substrates by:

- W layer deposition (20 nm) before B deposition (no air exposure).
- Optimization still needed (including annealing before air exposure).



B target (RF-150 W) + W target (RF-50 W):

- Layers are stable in vacuum and in air for all substrates.

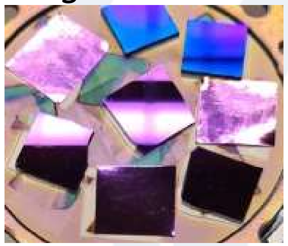


Sputtering depositions under argon Ar+D_2:

B target (RF-150 W):

f_{Ar} = 10 sccm; f_{D2} = 18 sccm; P_{Ar+D2} = 7×10^{-3} mbar.

As-grown



20 minutes in air

- Layers are stable in vacuum, flat and homogeneous.
- Change of color within minutes in all substrates.
- Layers remain flat on all substrates.

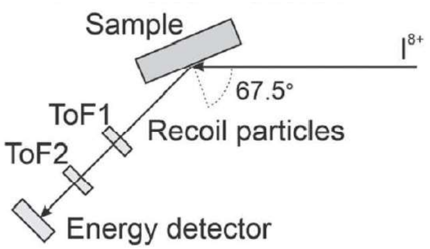
B target (RF-150 W) + W target (RF-50 W):

- Similar as in Ar depositions: flat, homogeneous, stable in air.

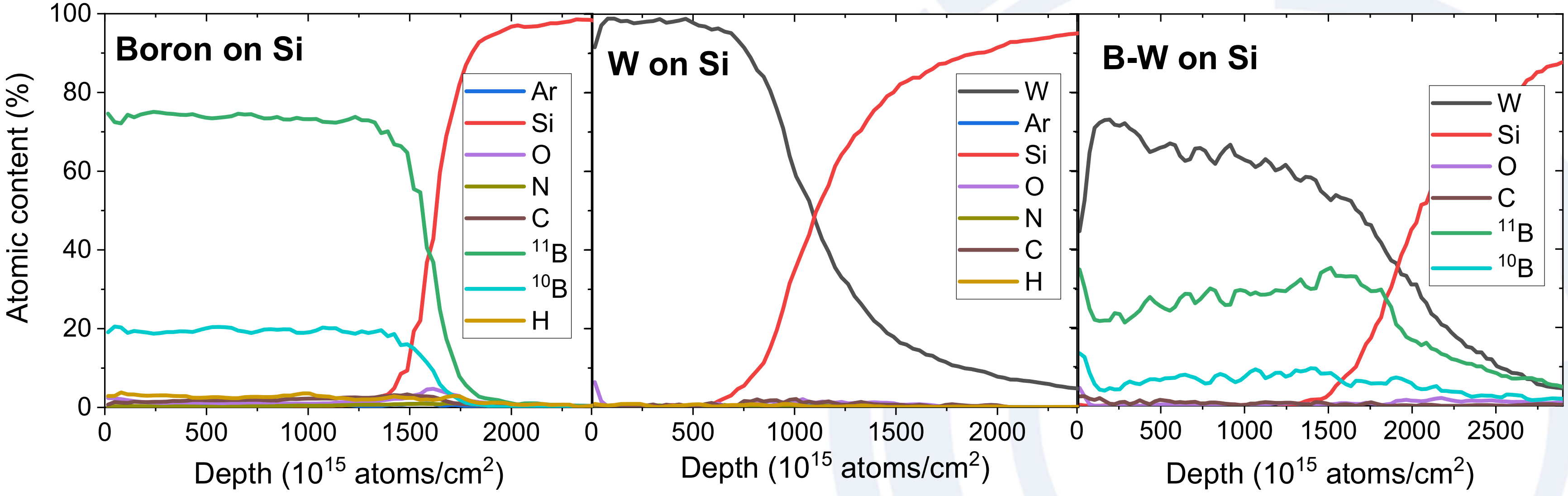
W-B film characterization

Sputtering depositions under argon atmospheres.

Atomic concentration depth profiles by ToF-ERDA:



Average film composition calculated from 150-500×10¹⁵ atoms/cm²



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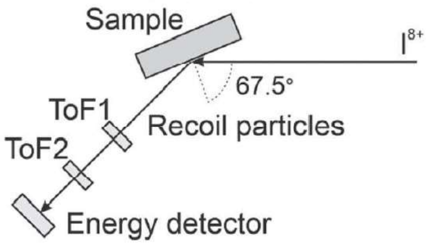
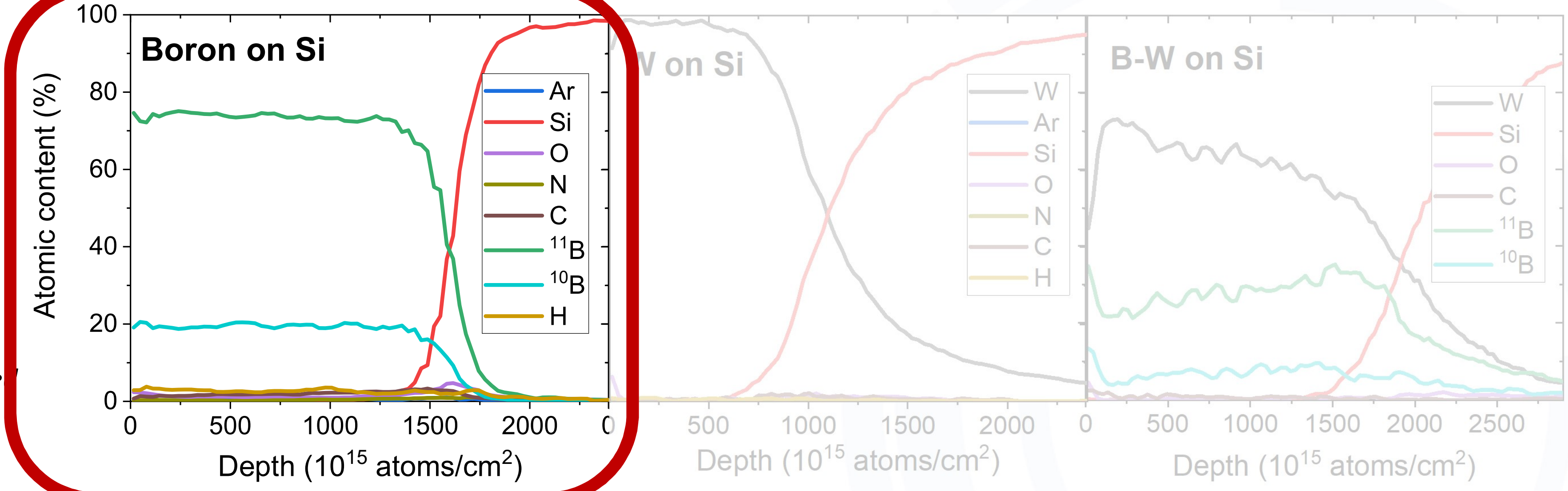
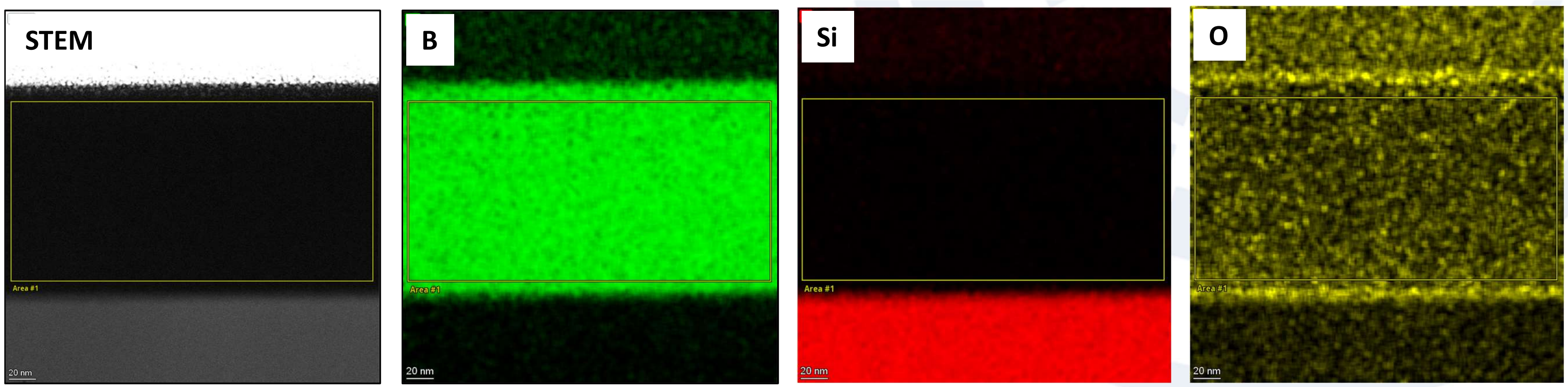


Average film composition calculated from 150-500×10^{15} atoms/cm^2



Boron on Si:

- B at. % >95;
- amorphous (TEM);
- 96% of bulk density; (RBS+TEM);
- EDX → $O_{surf/interf}$



W-B film characterization

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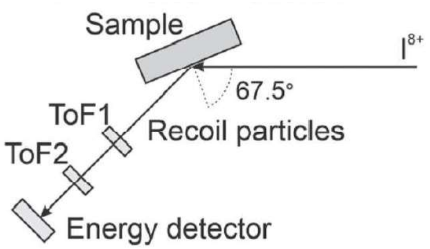

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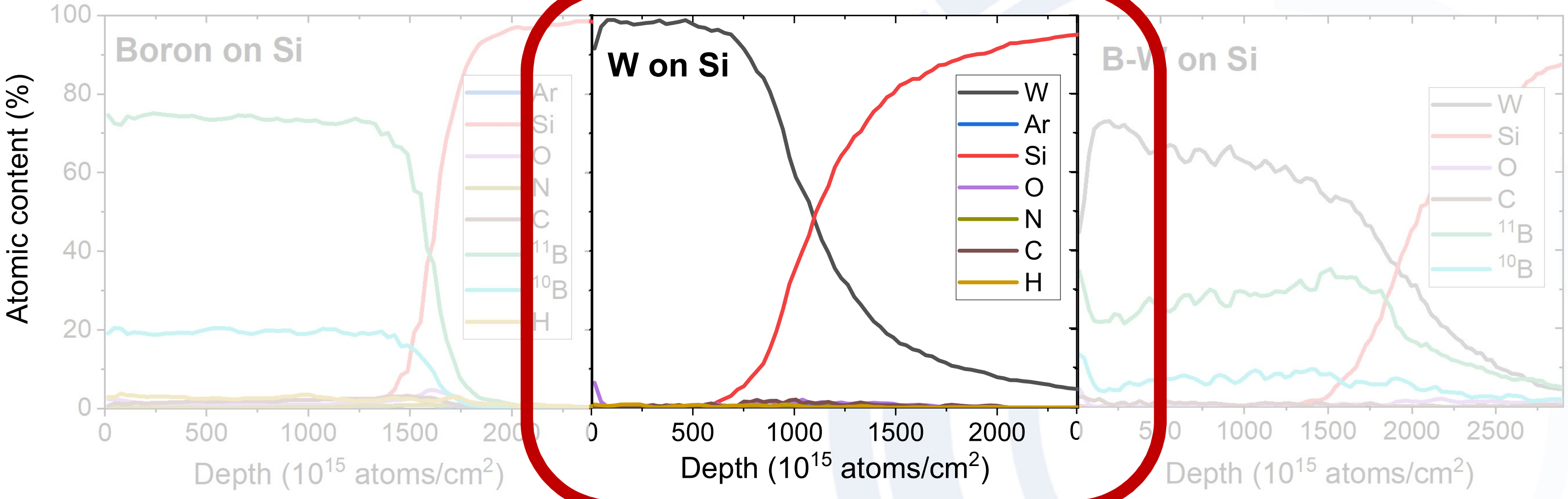
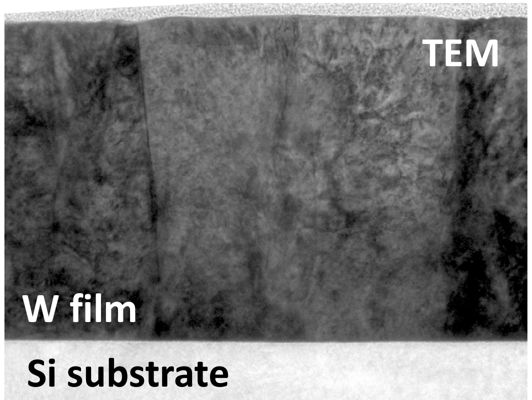
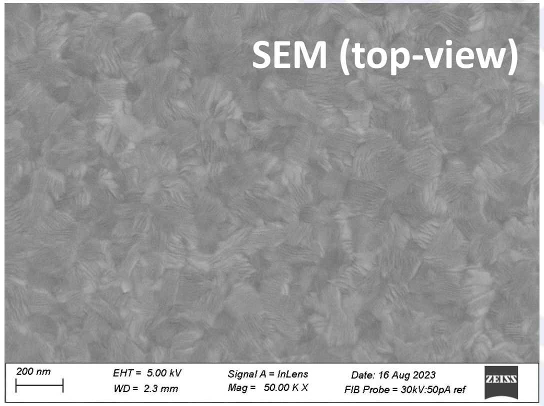


W on Si:

- W at. % >98;
- Polycrystalline;
- Columnar µ-structure;
- 95% of bulk density; (RBS+TEM);





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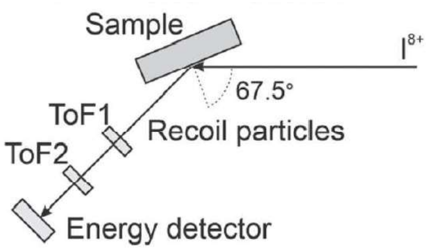

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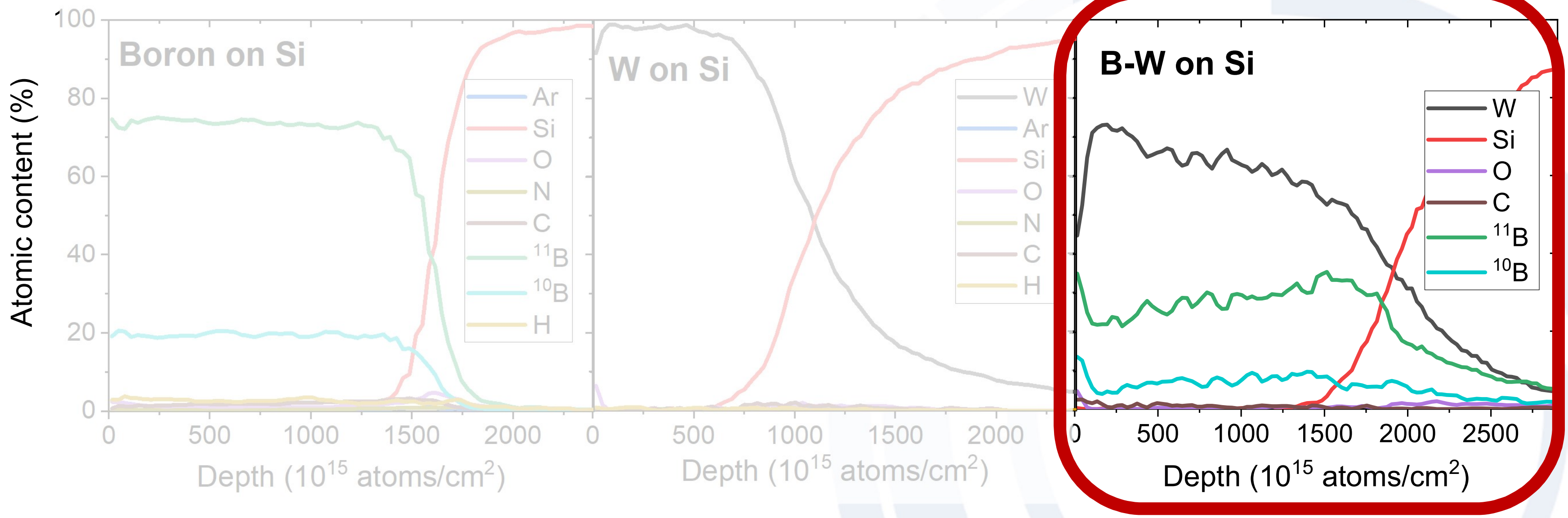
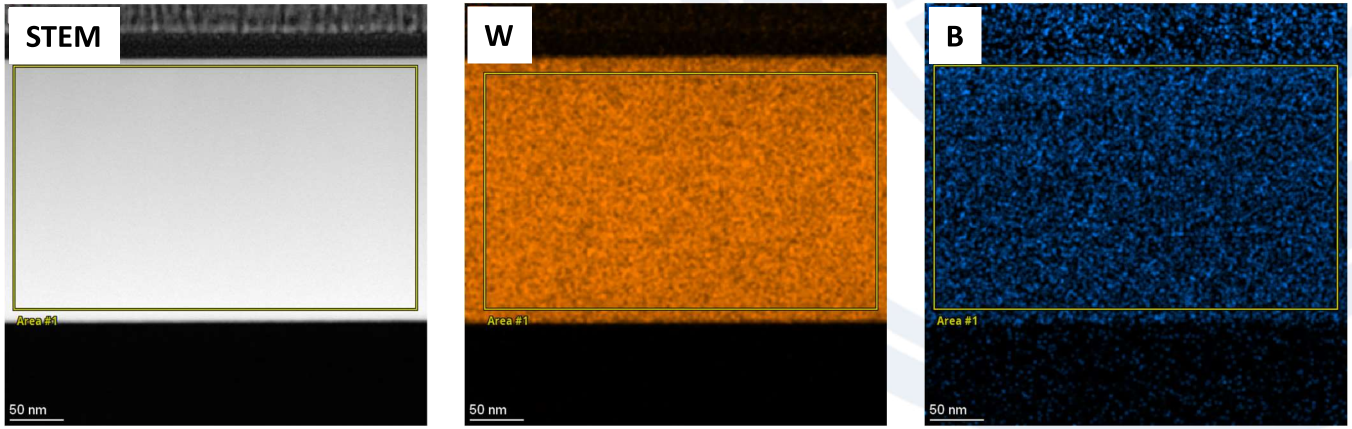


W-B on Si (example):

- W:B (2:1);
- Amorphous;
- Low contamination (<1%);
- EDX: no clear signal of B.
- Clear B on ToF-ERDA signal (multiple scattering effect from W).



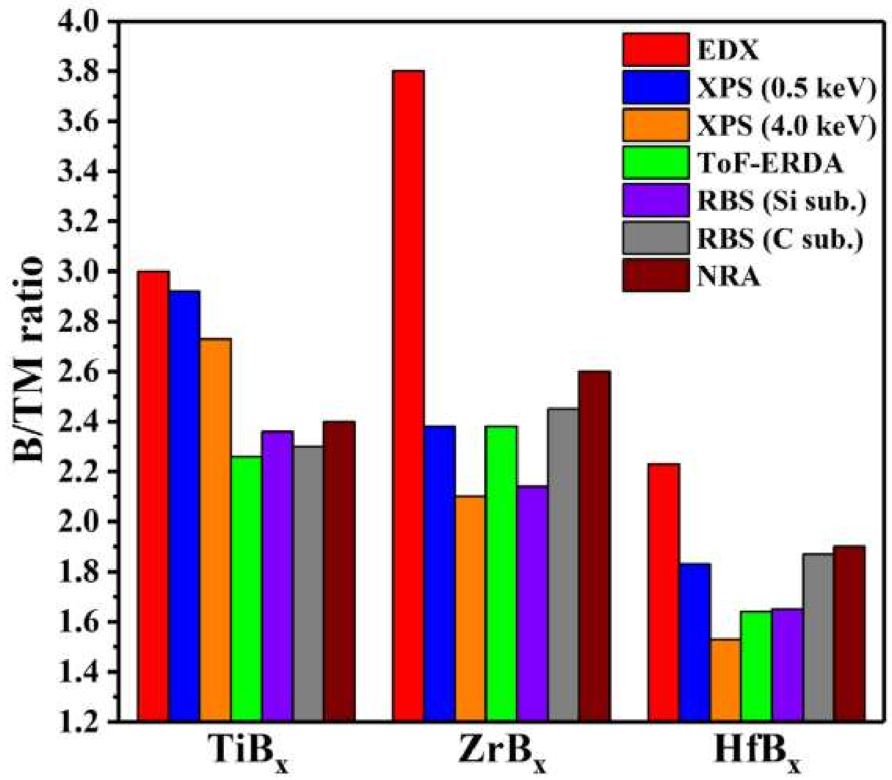
W-B film characterization

Sputtering depositions under argon atmospheres.

Systematic compositional analysis of sputter-deposited boron-containing thin films

B. Bakhit, D. Primetzhofer, E. Pitthan, M. A. Sortica, E. Ntemou, et al.
*J. Vac. Sci. Technol. A **39**, 063408 (2021)*

- Significant overestimation of B/TM ratio from EDX.
- Overall good agreement between different IBA techniques.



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Assessing boron quantification and depth profiling of different boride materials using ion beams

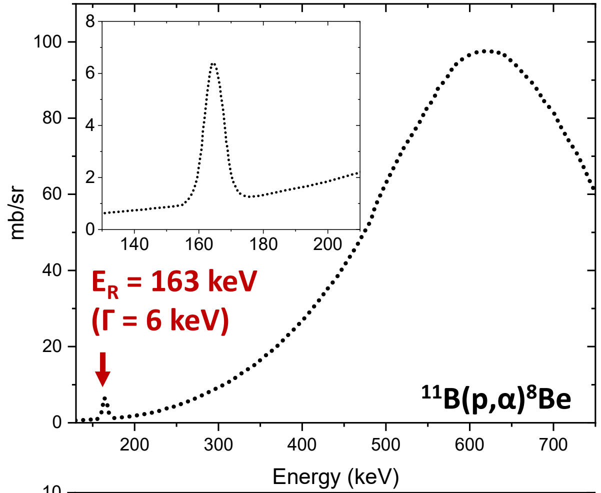
E. Pitthan, M.V. Moro, S. A. Corrêa, D. Primetzhofer,
*Surf. Coat. Tech. **417**, 127188 (2021)*

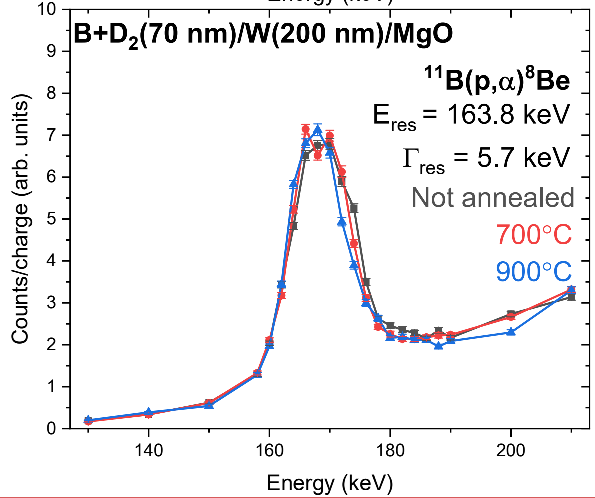
- Multiple scattering on high-Z species can affect depth profiles can hamper homogeneity assessment in ToF-ERDA.
- Combination of multiple IBA techniques is recommended.

A multipurpose set-up using keV ions for nuclear reaction analysis and high-resolution backscattering spectrometry

S. A. Corrêa, E. Pitthan, M.V. Moro, D. Primetzhofer,
*Nucl. Instrum. Methods Phys. Res. B **478**, 104 (2020)*

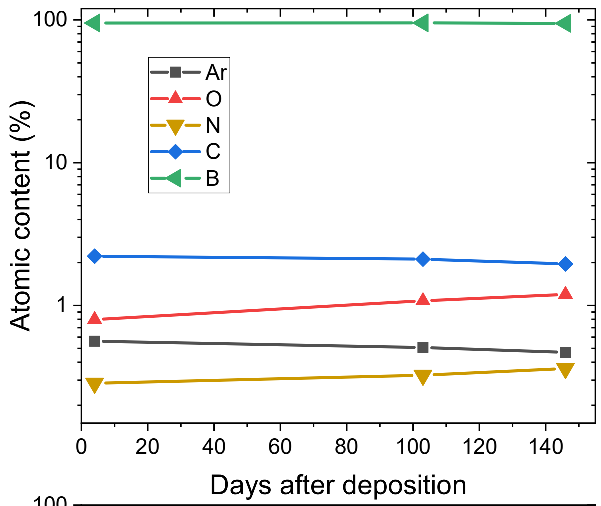
- Resonant ^{11}B-NRA (selectivity and high sensitivity) combined with (HR)-RBS can be used to assess homogeneity and stoichiometry.

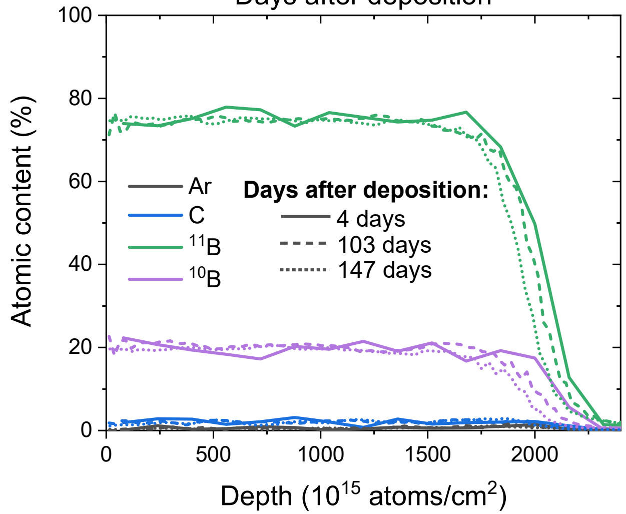




W-B film characterization: aging effect

Stability of boron layers over time (deposited in argon).





No significant change in composition in the bulk of layers over time.
Average film composition calculated from 150-500×10^{15} atoms/cm^{2}

Agreement with L.B. Bayu Aji et al.:
"thicknesses of ≥55 nm have expected excellent corrosion resistance during storage in laboratory air at room temperature over several months".
L.B. Bayu Aji et al. Appl. Surf. Sci. 448 (2018) 498.

Boron loss over time (thickness reduction) not accompanied by significant change of film composition.

L.B. Bayu Aji et al.: Boron loss from surface oxidation → formation of boric acid (evaporation).

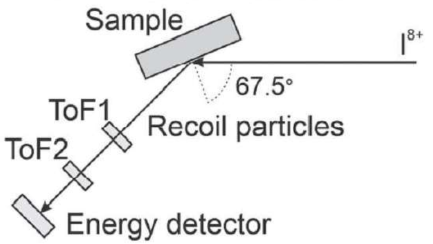
W-B mixed layers presented good stability overtime: no change of composition and thickness.

Sample characterization immediately before/during each experiment.

W-B film characterization

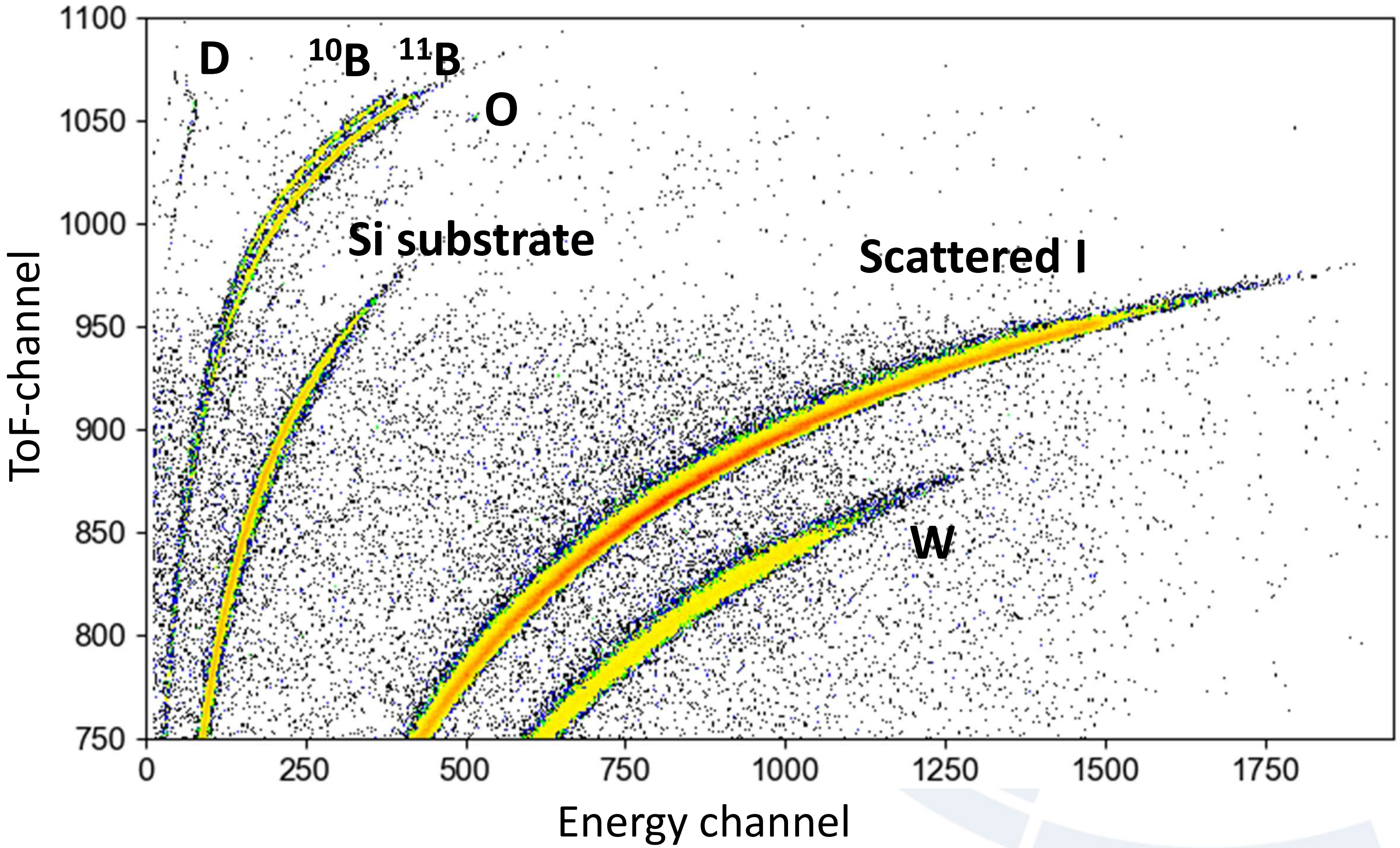
Sputtering depositions under argon and deuterium atmospheres:
f_{Ar} = 10 sccm; f_{D2} = 18 sccm; P_{Ar+D2} = 7×10^{-3} mbar.

Atomic concentration depth profiles by ToF-ERDA:



→ **H and D detected.**

→ **D amounts verified by NRA using D(^{3}He,p)^{4}He at 2 MeV.**



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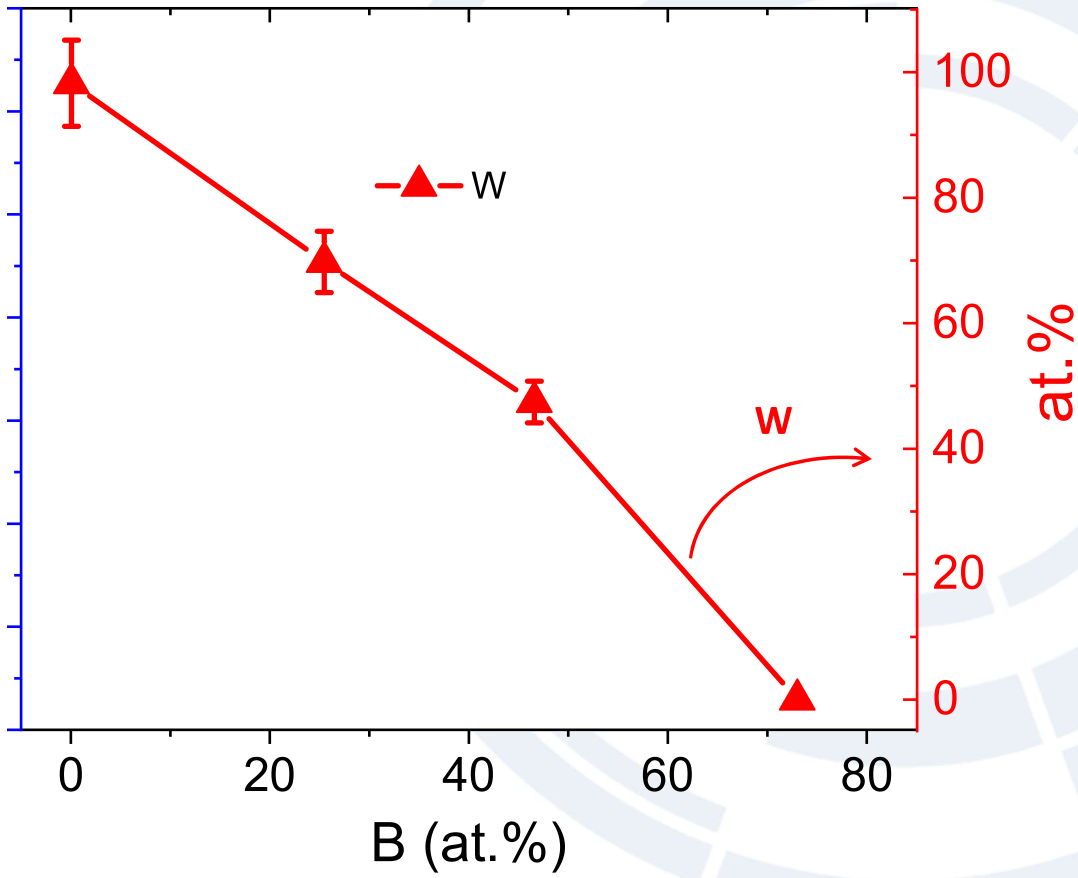
f_{Ar} = 10 sccm; f_{D2} = 18 sccm; P_{Ar+D2} = 7×10^{-3} mbar.

→ **f_{Ar} and f_{D2} fixed;**
→ **W and B magnetron power varied to obtain different B/W ratios.**

→ **Average composition from bulk of films.**



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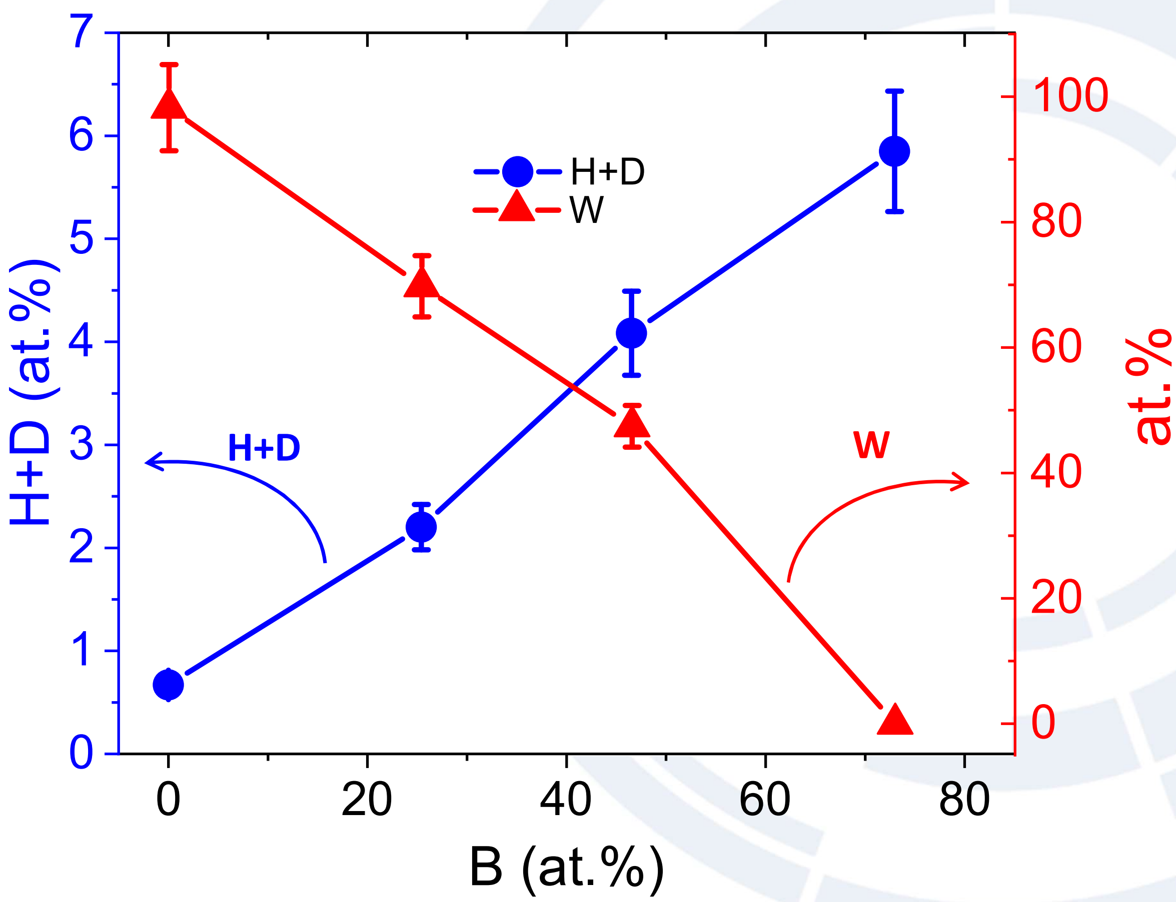
→ **Average composition from bulk of films.**

→ **Hydrogen and deuterium variation attributed to isotopic exchange from air exposure (dedicated experiments in progress) and different aging of samples.**

→ **Hydrogen and deuterium atomic content scales with boron in B+W mixed layers.**



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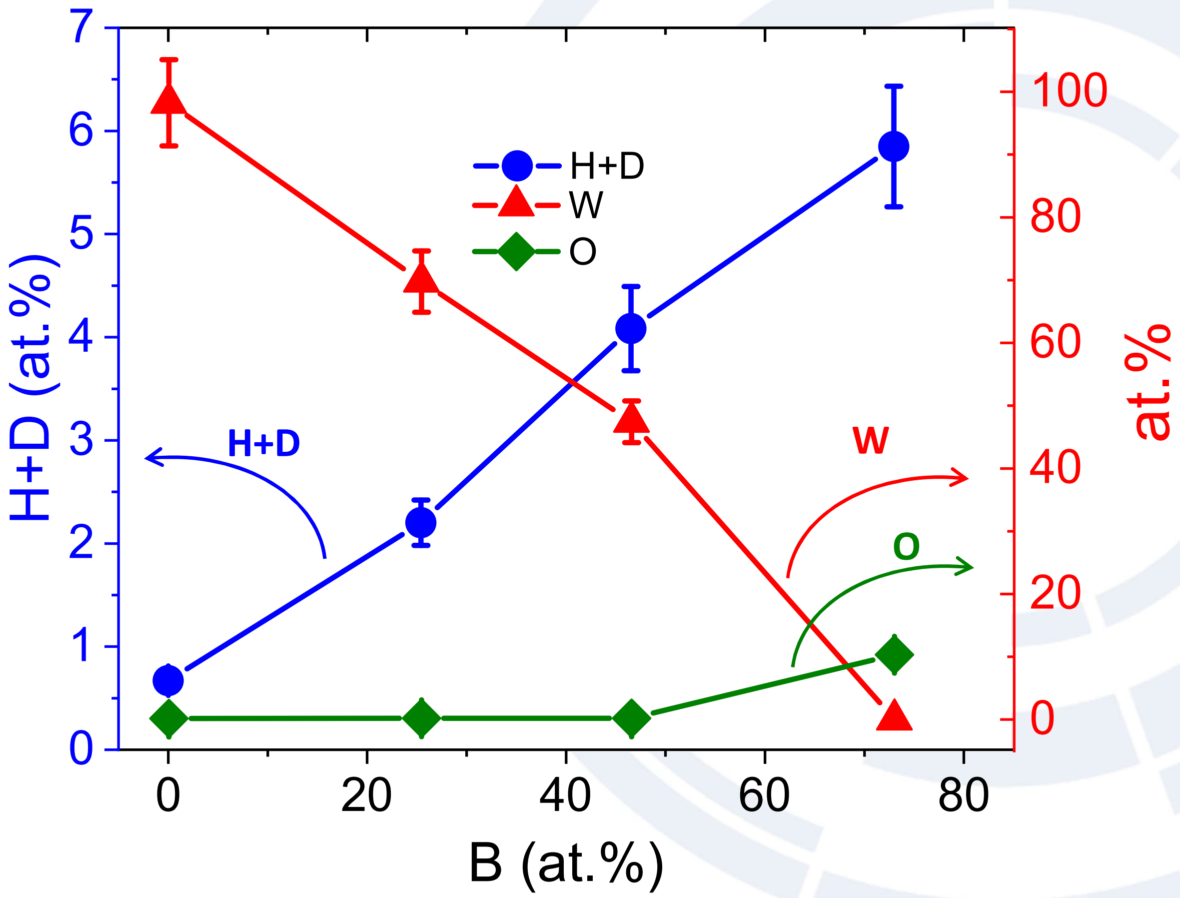
→ **Hydrogen and deuterium variation attributed to isotopic exchange from air exposure (dedicated experiments in progress) and different aging of samples.**

→ **Hydrogen and deuterium atomic content scales with boron in B+W mixed layers.**

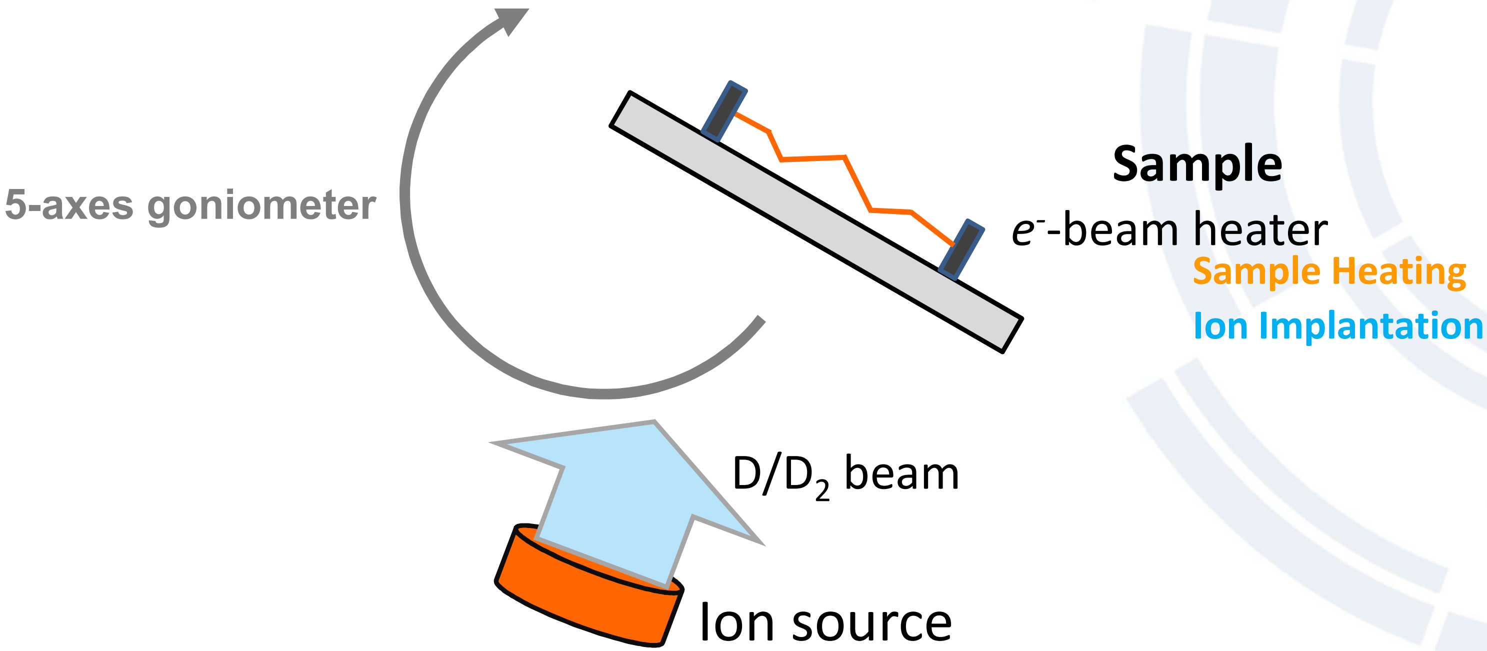
→ **Oxygen around 10 at.% only at high B/W:**
Presence of W might suppress oxygen incorporation.



In-situ experiments of hydrogen and deuterium desorption

SIGMA: Set-up for In-Situ Growth, Material modification and Analysis

K. Kantre et al. Nuclear Inst. and Methods in Physics Research B 463 (2020) 96–100



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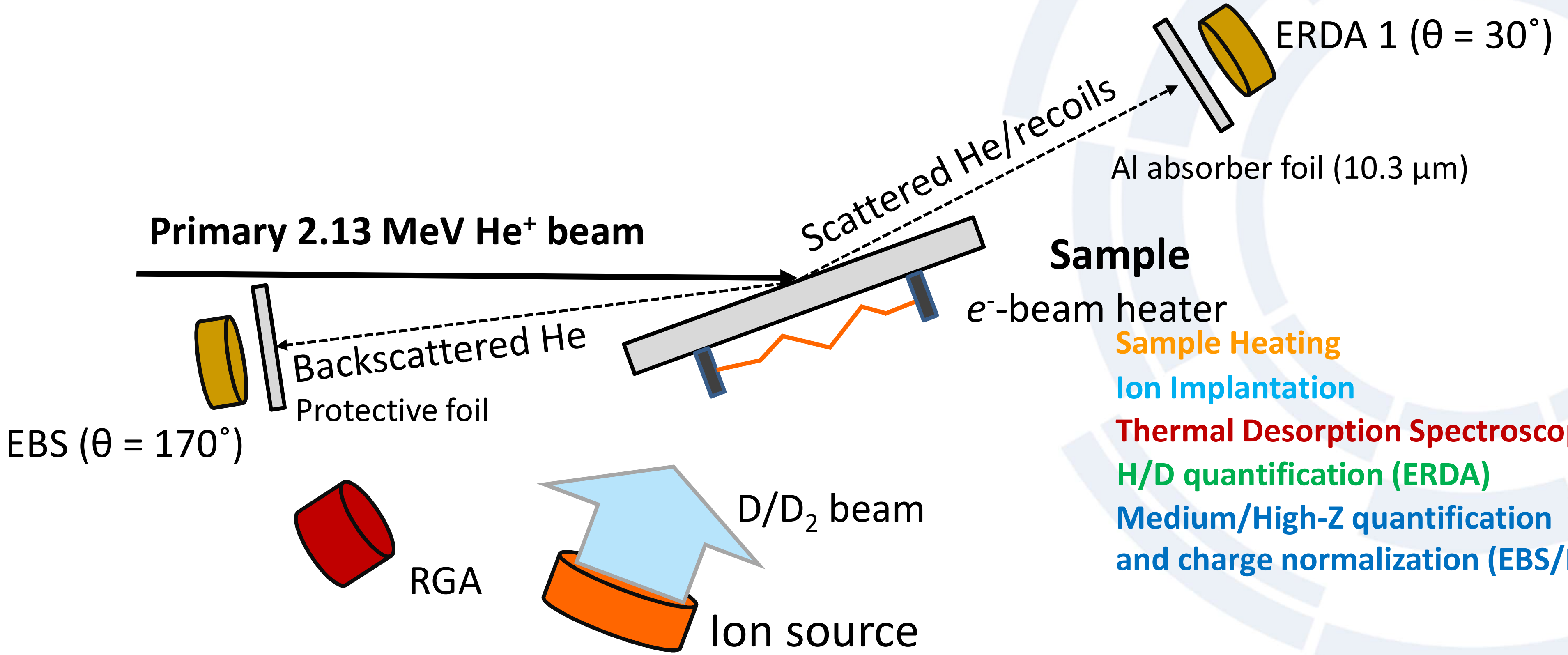


Sample Heating

Ion Implantation

Thermal Desorption Spectroscopy (TDS)

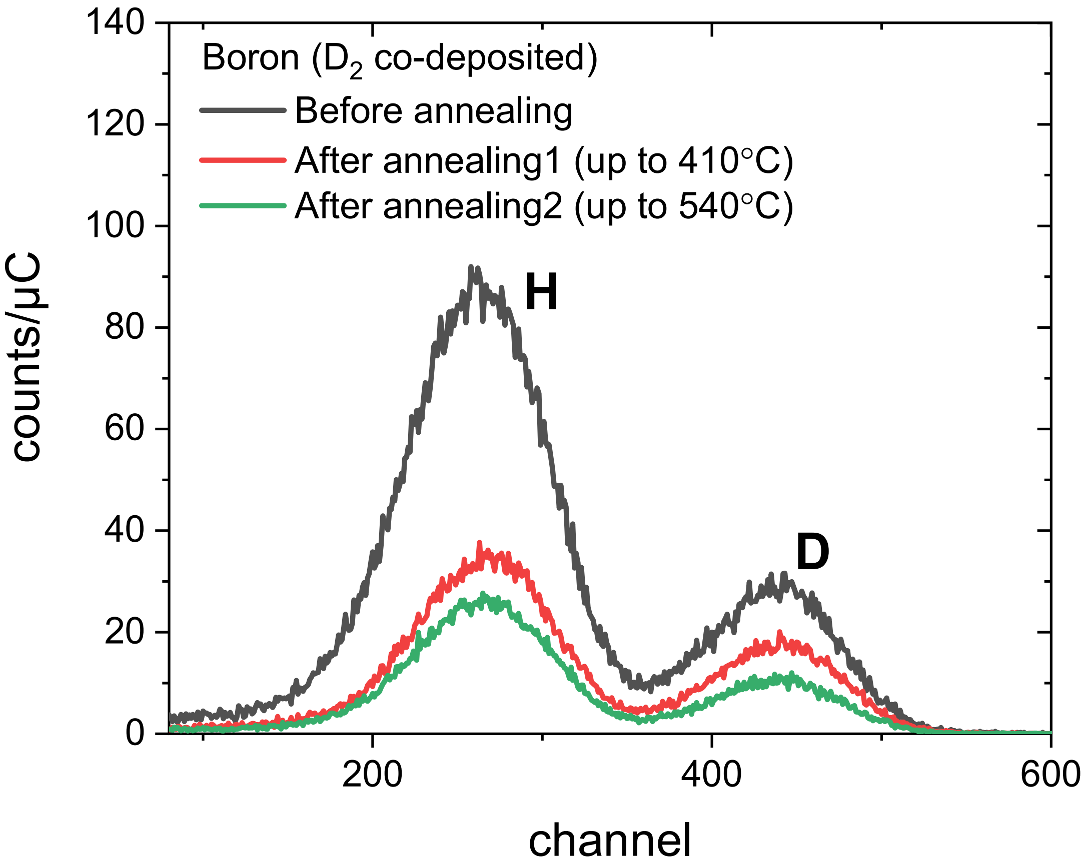
H/D quantification (ERDA)

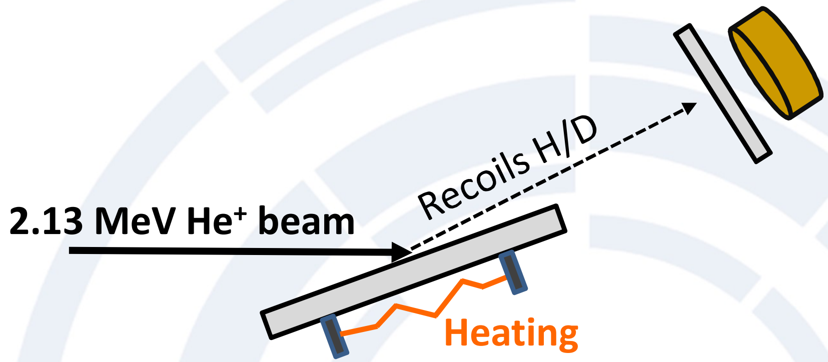
Medium/High-Z quantification and charge normalization (EBS/RBS)

In-situ experiments of hydrogen and deuterium desorption

Sample: Boron (Ar+D_2 atmosphere) on W substrate

ERDA He^+ 2.13 MeV



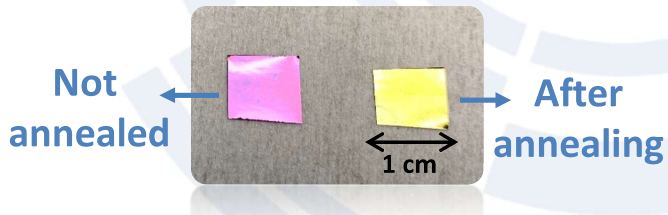


Quantification of H and D using the 2.13 MeV resonance in the $^2H(\alpha,d)^4He$ cross-section.

V. Quillet et al. Nucl. Instrum. Methods in Physics Res., Sect.B, 83, (1993) 47.

In-situ annealing

- Ramp of around 0.1°C/s and keep for 30 min at final T.



- H and D content is also be monitored during annealing.

Overview and Conclusions

- **B and B+W deposited in Ar atmosphere:**
  - Low presence of contamination (B>95 at.%); amorphous.
  - Stability in vacuum; peeling on W substrates in air for B layer;
  - Stable (no peeling) for B+W in any ratio on W substrates.

- **B and B+W deposited in Ar+D_2 atmosphere:**
  - D concentration scales with B/W ratio (up to 5.8 at.%).
  - No peeling observed for any film/substrate.
  - Boron layer (no W) present the highest O content (10 at.%).

Combined RBS/ERDA in-situ analysis can be used to obtain H&D distribution during annealing.

***e^-*-beam evaporator also available in the SIGMA beamline: possibility to grow B films in different atmospheres in-situ.**

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EUROfusion

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