EUROfusion

ENABLING RESEARCH PROJECT ENR-MAT.02.VR

The impact of boron intermixing in PFC on atomic, structural and mechanical features: sputter yields, near-surface morphology, and fuel retention

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Motivation

Effect of boronization in plasma facing components (PFC)

- Potential accumulation of boron in different parts of the reactor.
- Potential mixing of boron with PFC (W and steel).
- **Modification of these materials from plasma-wall interactions?**
- **Influence on hydrogen-isotopes retention? Morphology? Mechanical properties?**
- **Predictability of these properties for mixing B-PFC in different compositions?**
- **This project:**

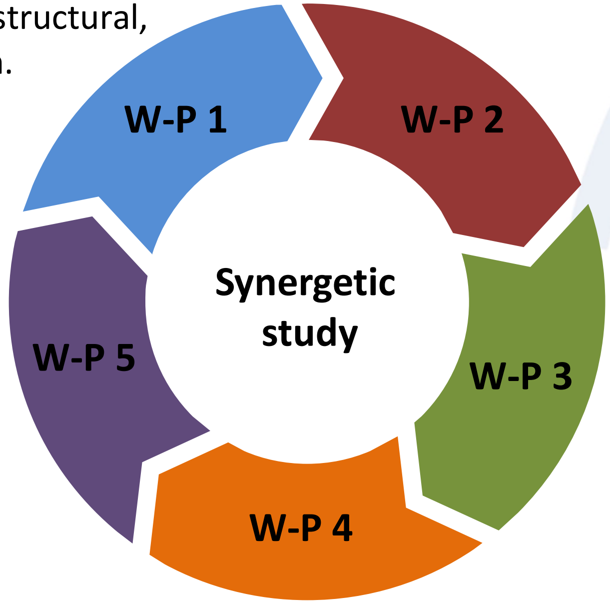
Laboratory scale investigation (deuterium retention, surface morphology, hardness/e-modulus, sputtering yield) and theoretical calculation (sputtering yields, <u>dynamic change of composition</u>) of pure and mixed B&PFC layers in different compositions.

<u>Dynamic change of composition</u>: Use of in-situ and real-time ion beam analysis simultaneous to mass-changes during ion irradiation.

Working-packages

Sample preparation & characterization
Co-depositions in argon/D_2 atmospheres
Chemical, morphological, microstructural, and mechanical characterization.
Distribution of samples.

Sputtering Yields and BCA simulations
Sputtering yield measurements with a QCM (target & catcher configuration).
In-situ dynamic sputtering measurements will be performed with simultaneous IBA (W-P 5).
Comparison to Monte Carlo-based BCA simulations.



Material Modification and Atomic Migration
In-situ monitoring of composition and atomic migration (depth profile) in structures by ion beam analysis: Ion irradiation & Thermal annealing.

Ab inito calculations of defect energetics and mixed B-W materials
Ab initio calculations (W_2B, W_2B_5 and WB_4):
Formation and binding energies of defects.
Development of an interatomic potential.
Adsorption and diffusion behavior of boron atoms along and into mixed B-W surfaces.

Molecular dynamics modelling of sputtering yields and surface modification
A new semi-empirical interatomic potential for B-W materials, development using ab initio data obtained in W-P 3.
Comparison of results with BCA calculations (W-P 2): Sputtering yield & change in composition.

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D1.6 Characterization of mechanical properties of mixed layers with W (from 2024).

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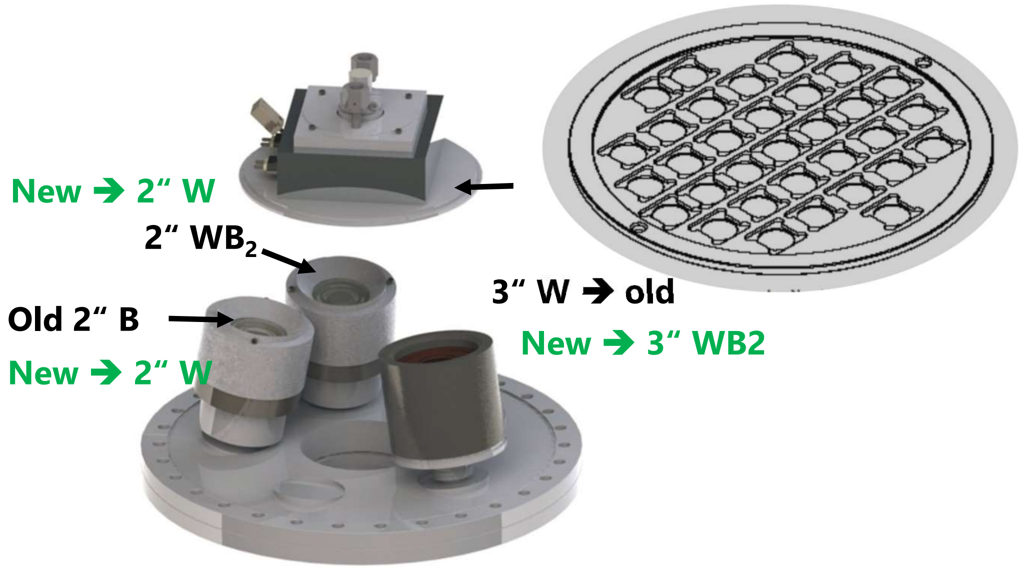
Mechanical properties of mixed W-B layers

Nanoindentation

Experimental Set-up



Goal is to create various chemical composition in one deposition run → specific sample holder + multi-cathode system required

Deposition Parameters

- **AJA Orion 5 deposition plant (Angie)**
  - 3″ WB_2 target power: 200 W (DC)
  - 2″(r) W target power: 60W (DC)
  - 2″(l) W target power: 30W (DC)
- Substrate temperature: 400 °C
- Substrate bias potential: -50 V
- Deposition pressure: 0.4 Pa
- Argon flow rate: 20 sccm
- Deposition time: 22 min
- No substrate rotation

Mechanical properties of mixed W-B layers

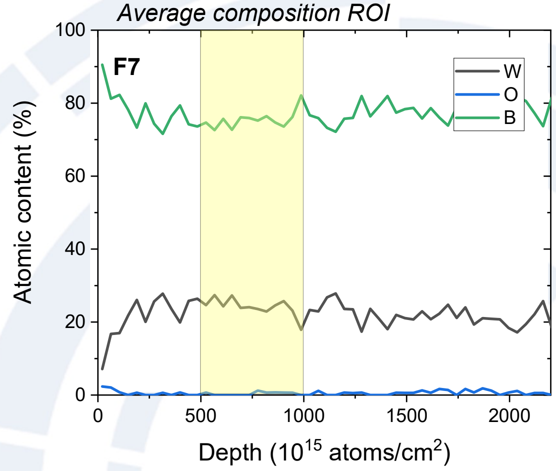
Nanoindentation

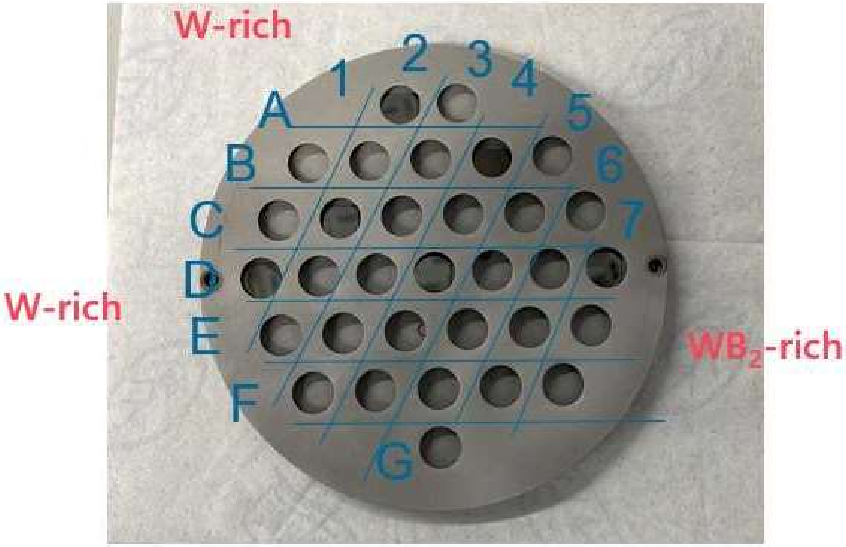
Film composition from Ion Beam Analysis (Tandem Laboratory – UU):

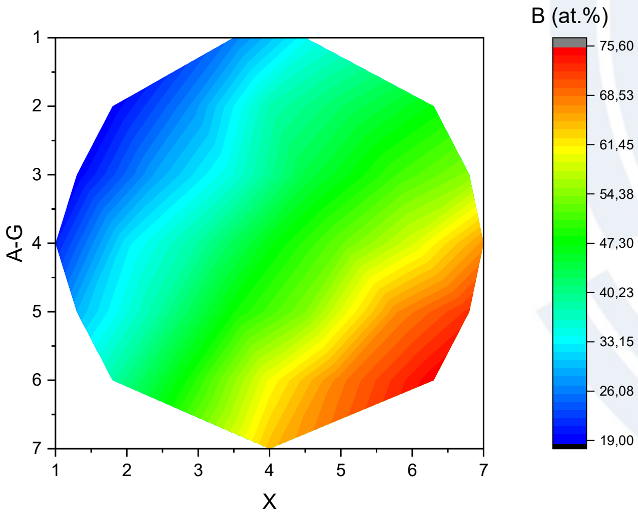
Low concentration of contaminants (H, C, and O) in all samples.

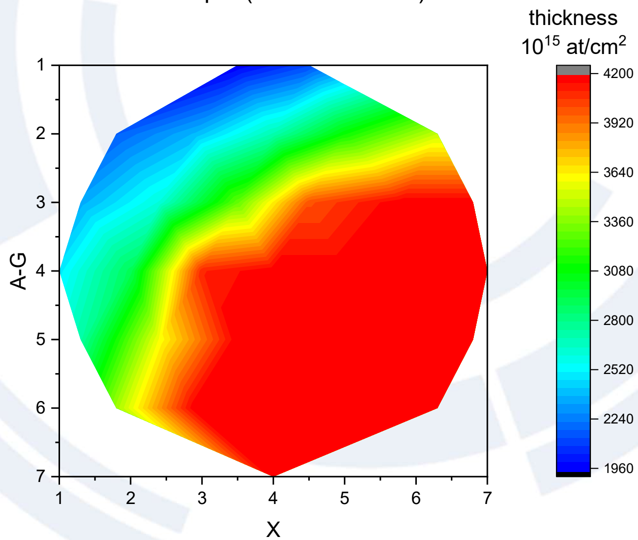
Variation of composition from 19-75 B at.%

ToF-ERDA depth profile:







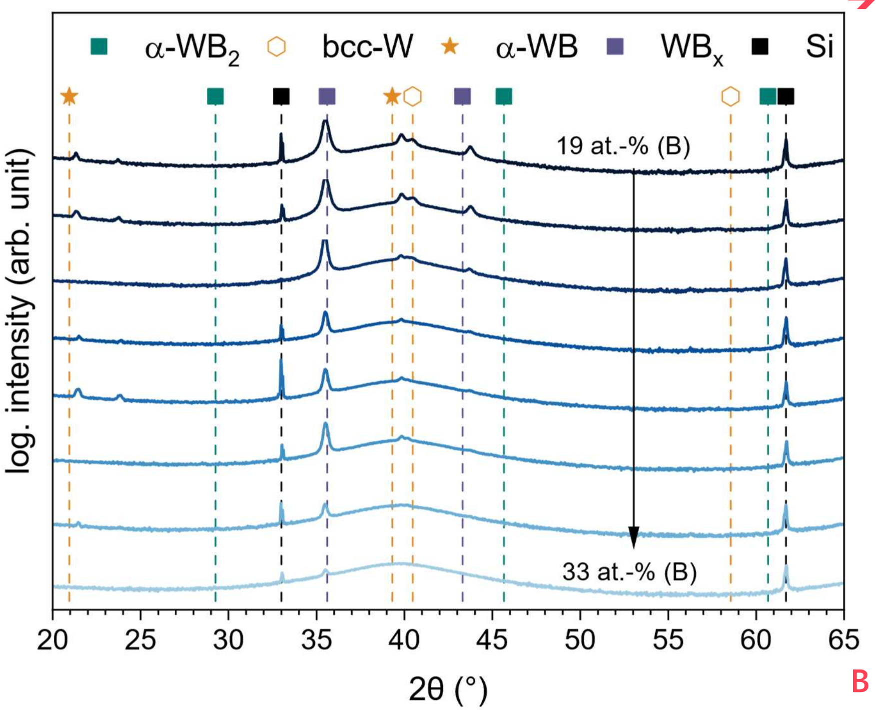


Mechanical properties of mixed W-B layers

Nanoindentation

Phase analysis, XRD

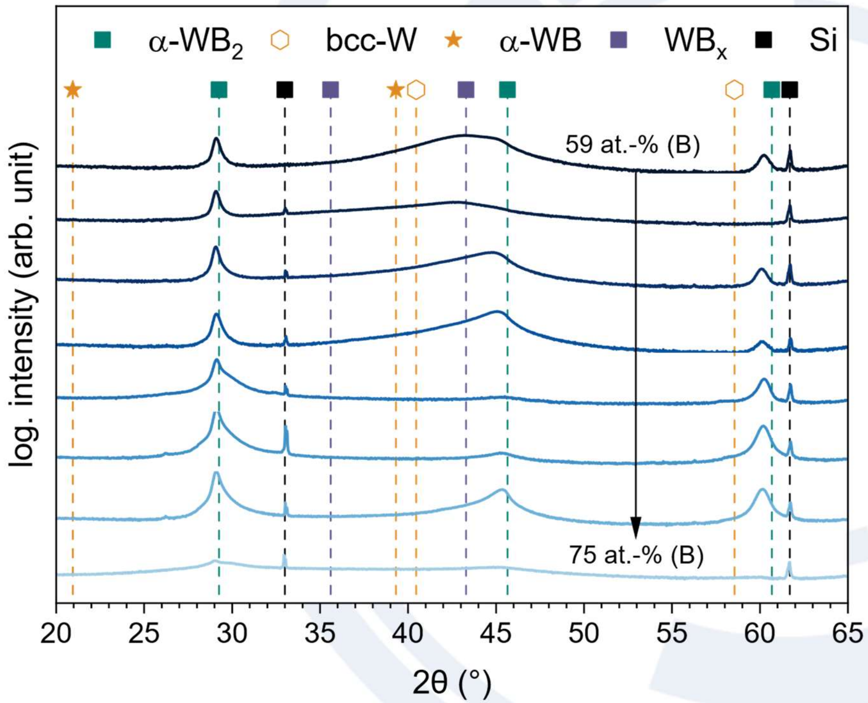
W-rich layers:



Boron depleted ➔ multi phase structure

B enriched ➔ amorphous with small amounts of bcc-W

Boron-rich layers:



Boron = 59 at.% ➔ crystalline WB_2

B enriched ➔ amorphous & WB_2 phase

Mechanical properties of mixed W-B layers

Nanoindentation

Measurement details:

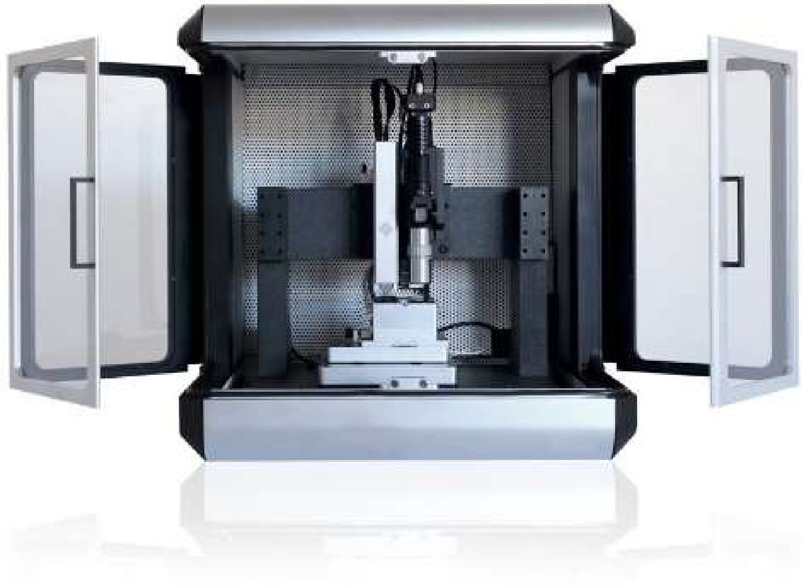
FT-I04 Femto-Indenter

CSM method

Nanoindentation was done on each sample

the average values of the obtained Hardness and Youngs-Moduli

were calculated using a Python Script

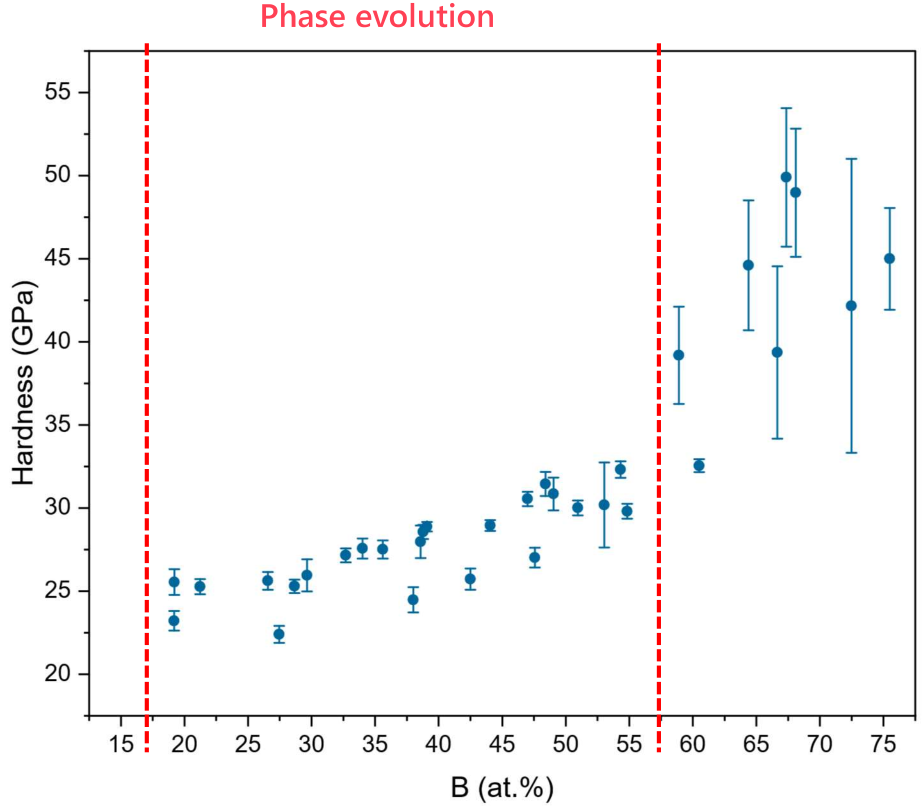


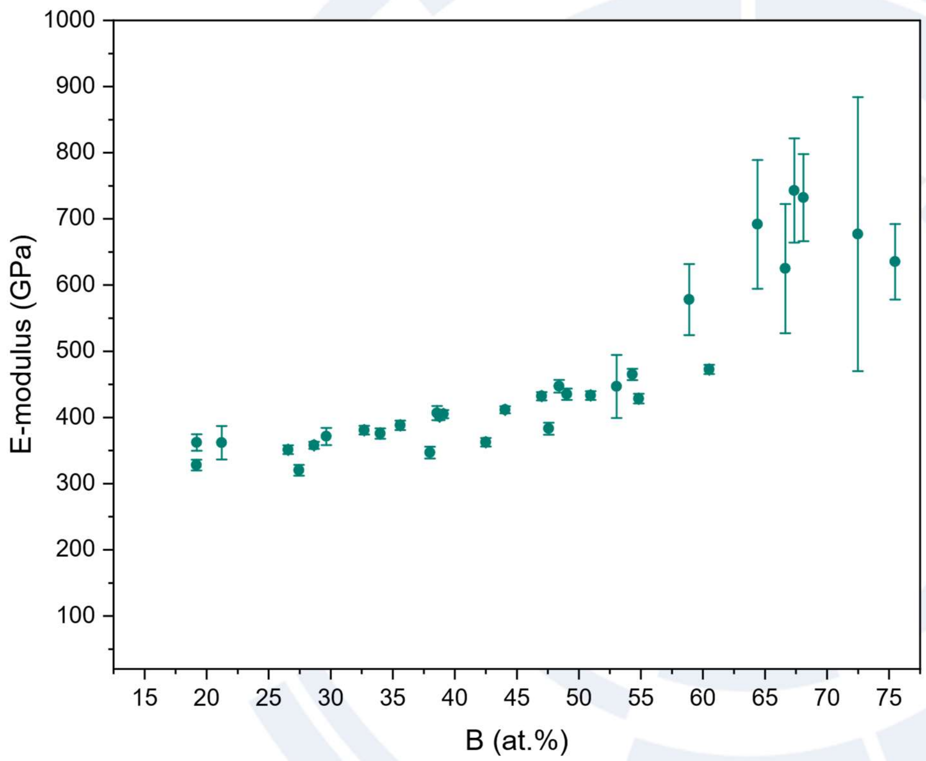
Challenge: thickness of the W1-xBx films

→ 350 to 400 nm is for now lower limit

Mechanical properties of mixed W-B layers

Nanoindentation





Hardness and E-modulus scales with B content. TEM in progress of selected samples (more insight of phase constitution).

Amorphous & WB_2 phase in boron rich layers: larger variation in mechanical properties.

Significant effect of boron in mechanical properties (manuscript in progress).

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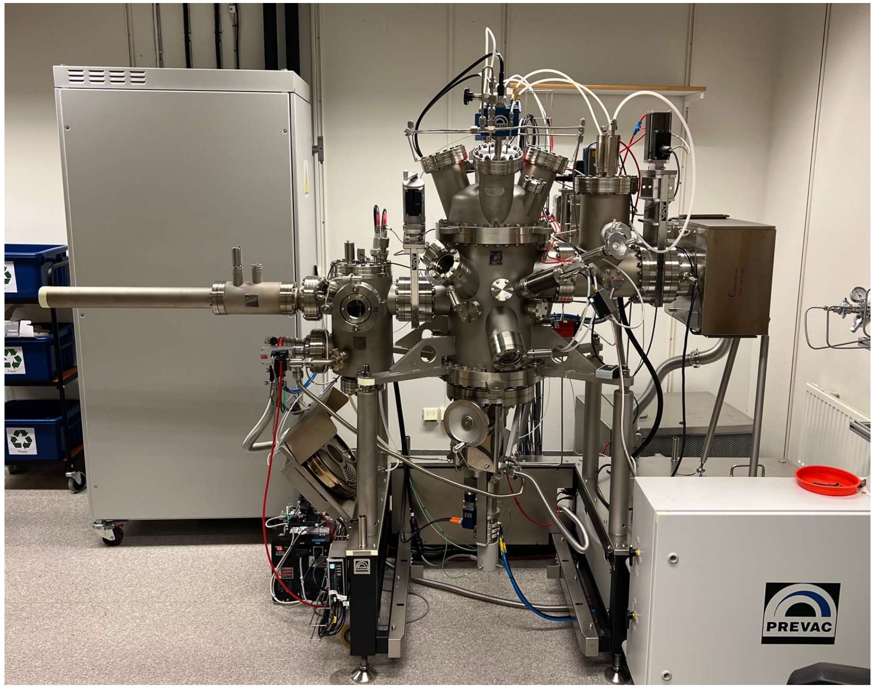
Sputtering yields

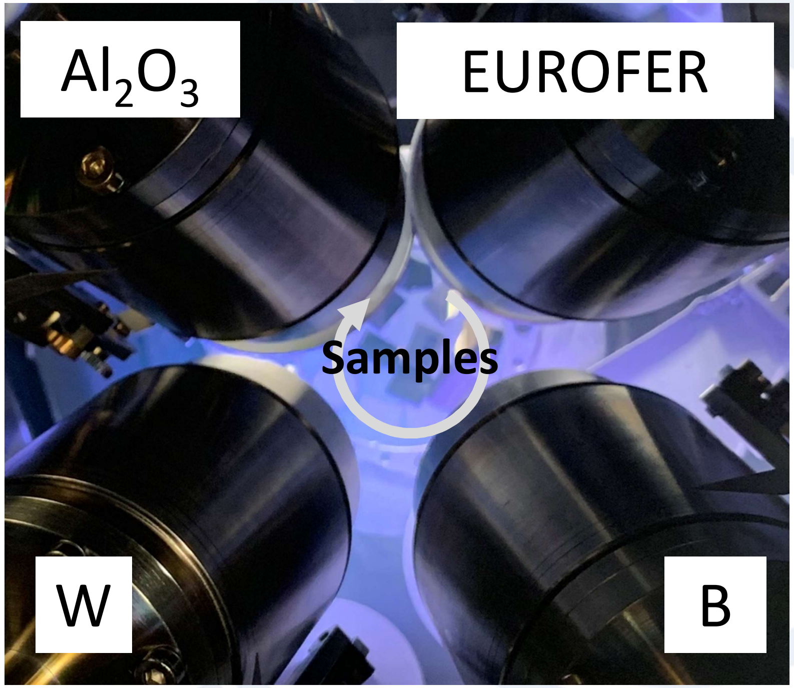
Sample preparation at Tandem Laboratory (Uppsala University)

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).

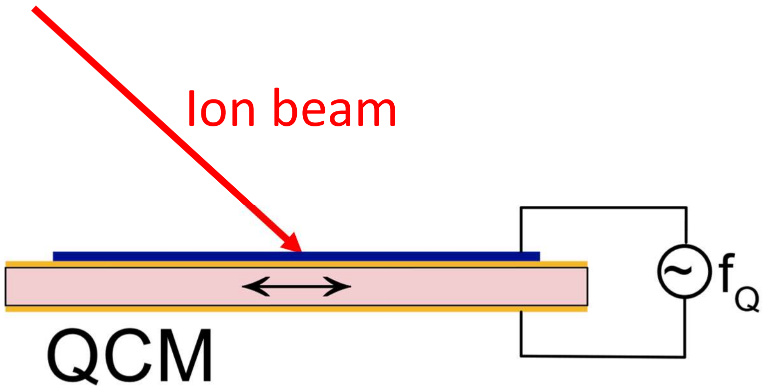
Top-view:



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

Sputtering yields

Experimental measurements



Measurements of Sputter Yield using high-sensitivity Quartz-Crystal-Microbalance (QCM)

Projectiles:

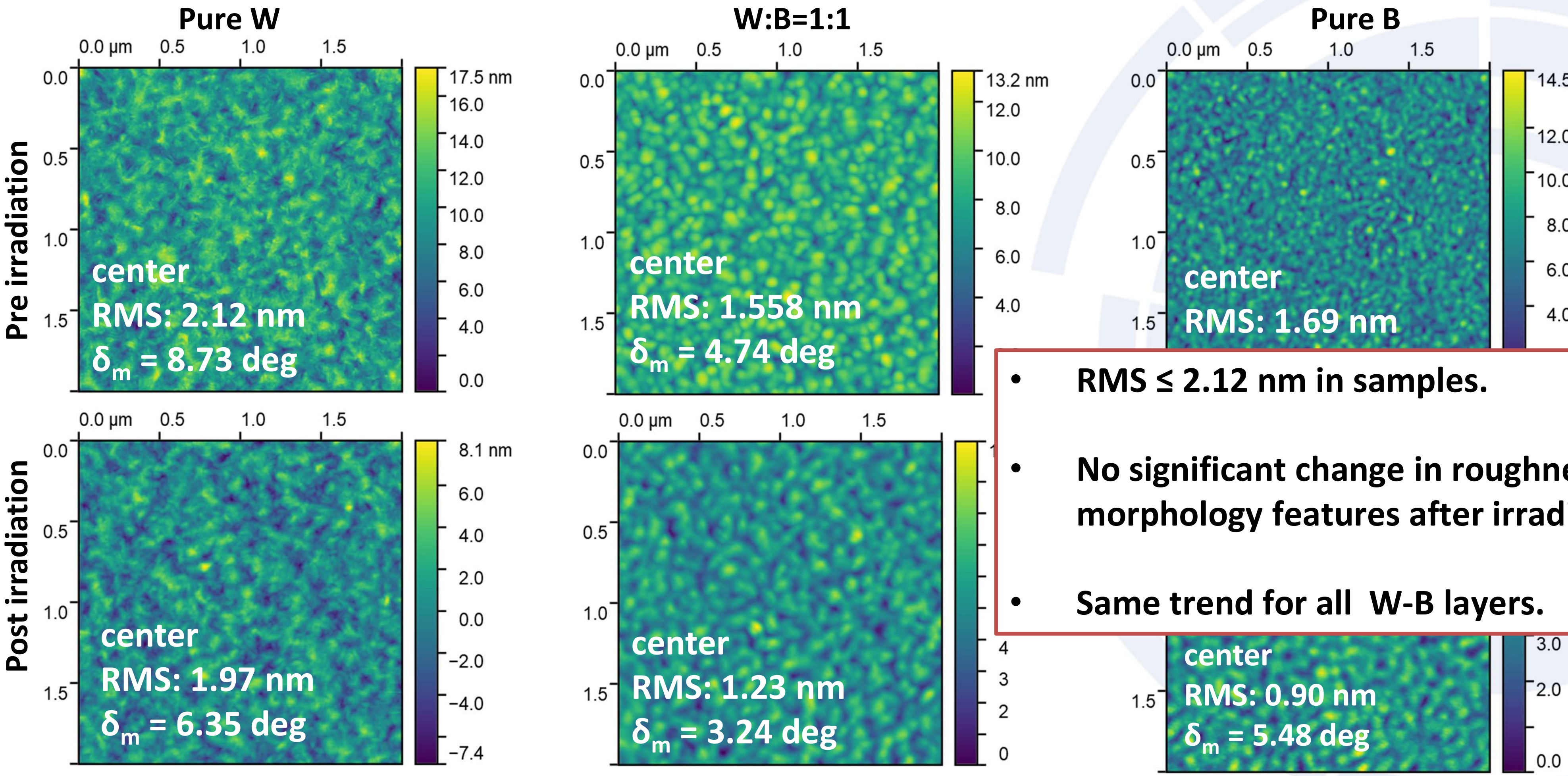
- 2 keV Ar^+
- 2 keV D_2^+

  (2 keV D_2 as proxy for 1 keV D)

- **Target:**
- Pure W and B, and mixed layers.
- Redeposited EUROFER97 and B, and mixed layers.

Sputtering yields

Surface morphology of W-B films on QCM (pre-irradiation and post):



- **RMS ≤ 2.12 nm in samples.**
- **No significant change in roughness and morphology features after irradiation.**
- **Same trend for all W-B layers.**

Sputtering yields

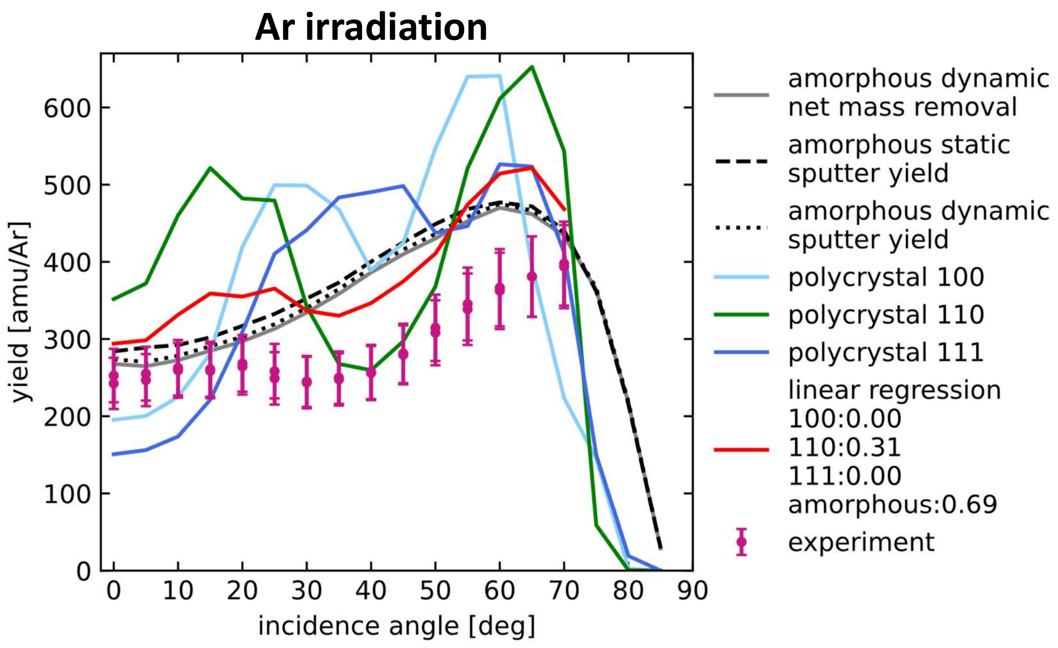
Experimental measurements and BCA simulations

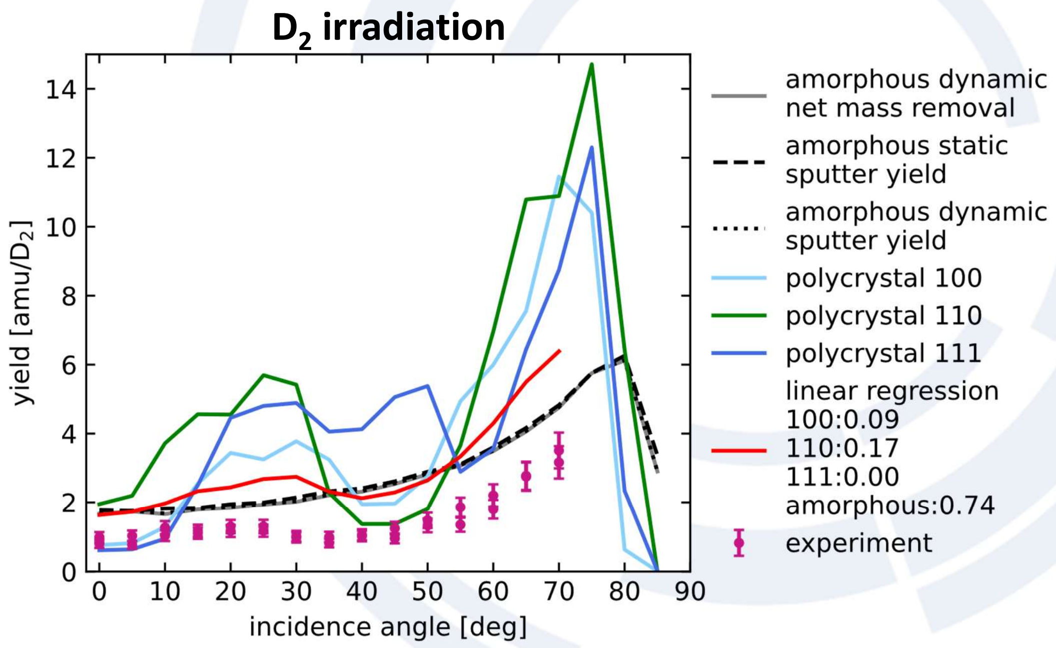
W films on QCM:

Local minimum present suggestion polycrystalline surface.

BCA simulations (SDTrimSP 7.02) supports crystal input structures: simulation of different crystallographic orientations.

→ best fitting simulation: combination of (110) crystal orientation (agreement with EBSD) and amorphous.





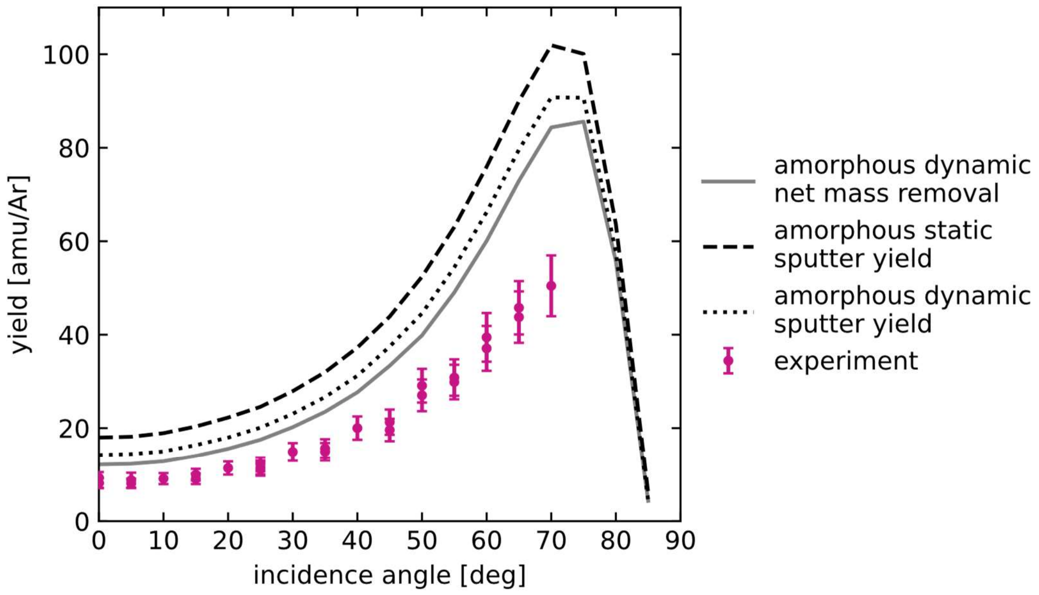
Sputtering yields

Experimental measurements and BCA simulations

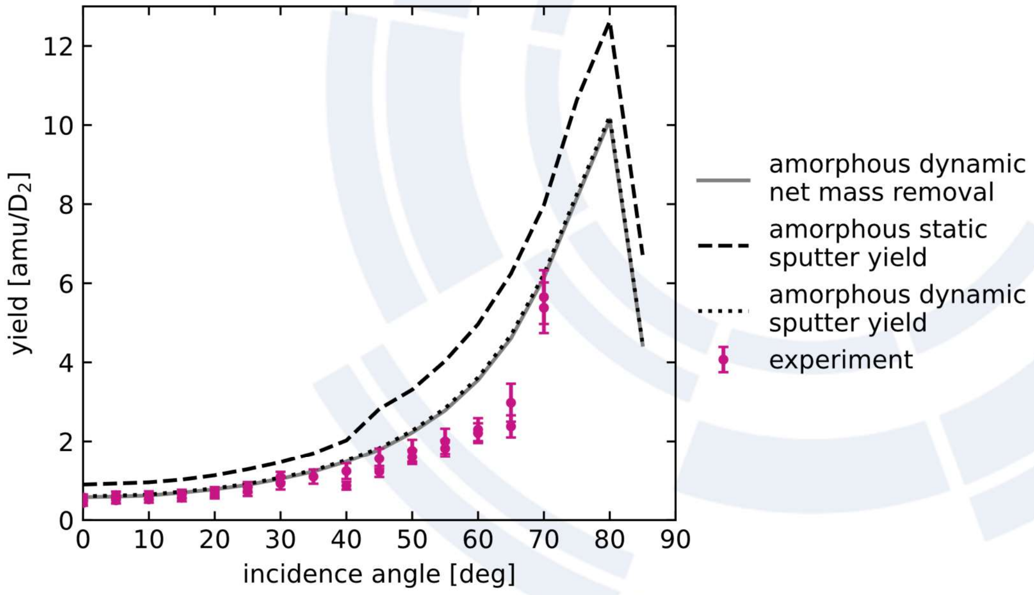
B films on QCM:

performed measurements on pure B at elevated temperature (~150°C –chosen according to minimum in f(T) curve)
No local minimum (**amorphous layer-agreement with TEM-diffraction**), also observed in SDTrimSP simulations.
Quantitative values: simulation and experiment **do not agree** (both static and dynamic mode).
Effect of implantation is largely enhanced during D irradiation → (WP5: deuterium retention studies).

2 keV Ar^+ irradiation:

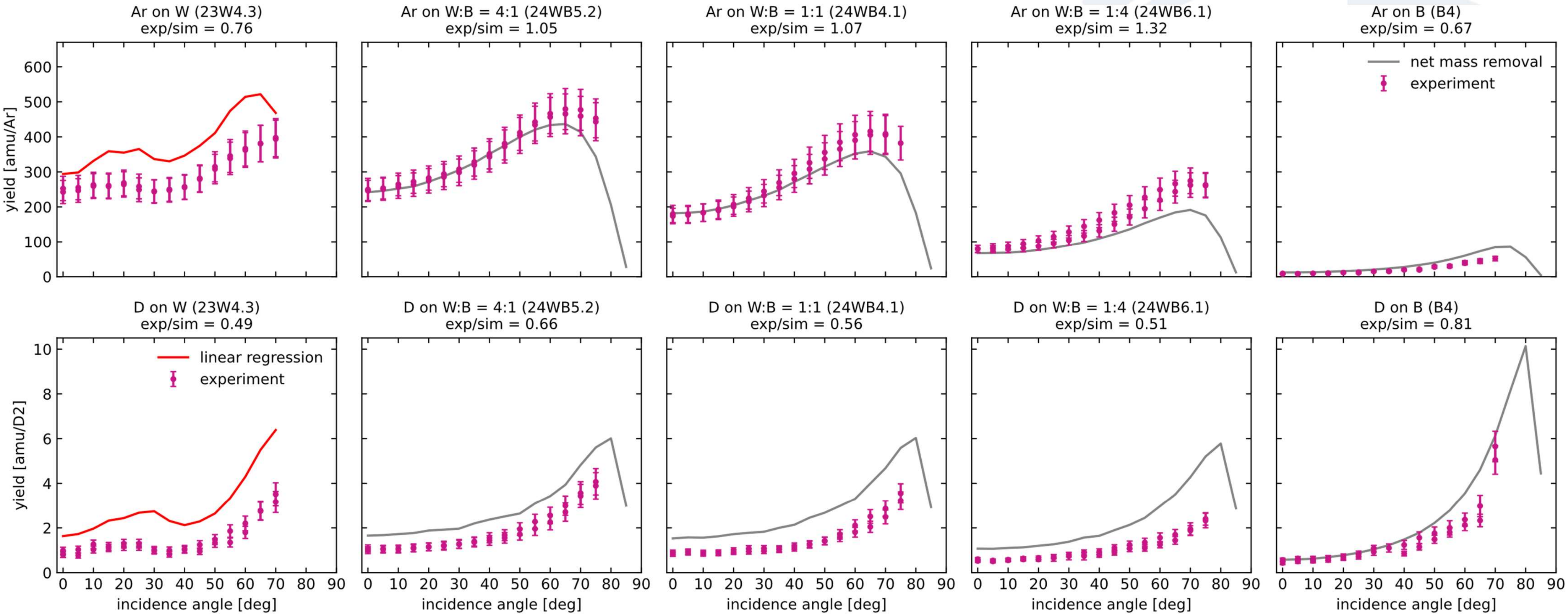


2 keV D_2^+ irradiation:



Sputtering yields

Experimental measurements and BCA simulations



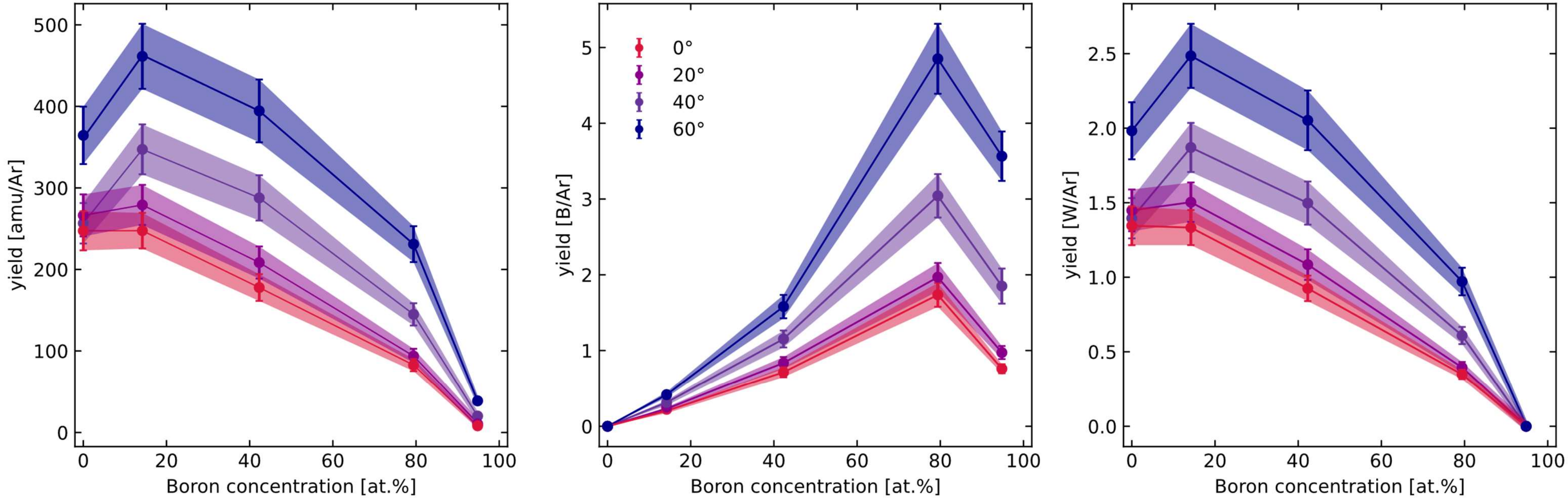
Ar irradiation: BCA simulations present overall good agreement with experiments.

D_2 irradiation: BCA simulations overestimate experiments.

Sputtering yields

Experimental measurements and BCA simulations

comparison – experiments – Ar irradiation



When assuming steady state conditions during sputtering, we can extract the atomic yields of W and B: $\frac{yield_W^{\infty}}{yield_B^{\infty}} = \frac{concentration_W}{concentration_B}$

Clear mass loss reduction in function of boron concentration during irradiation.

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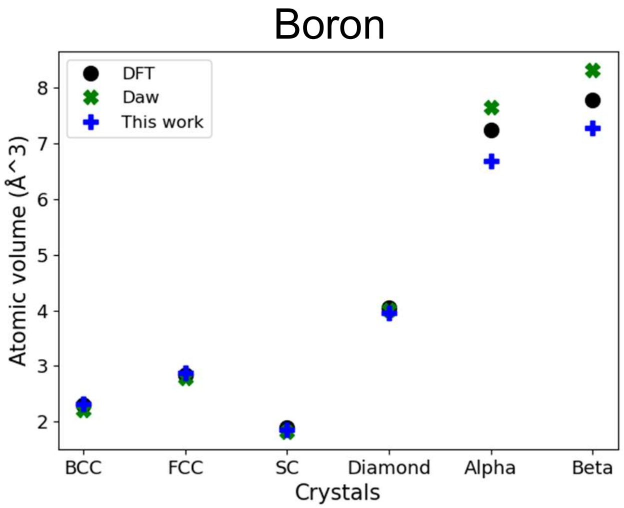
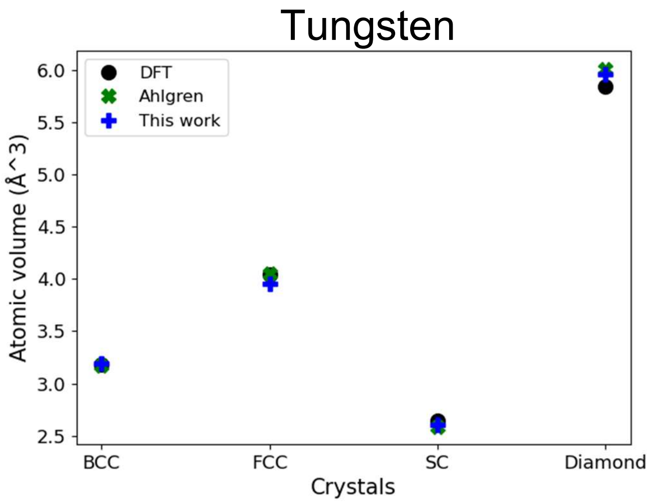
Ab inito calculations of defect energetics and mixed B-W materials

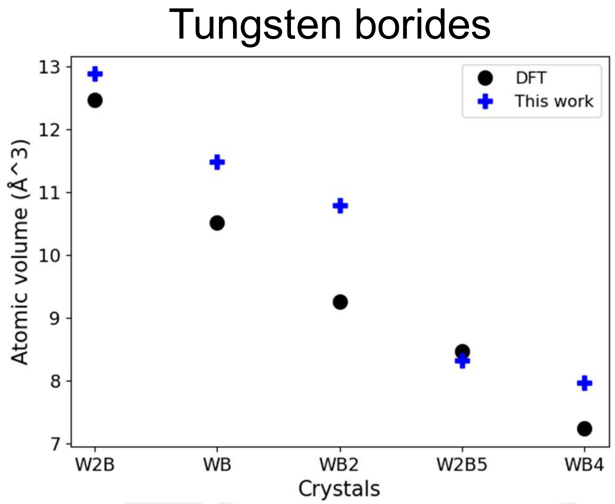
Development of new interatomic potential

- **Develop new semi-empirical interatomic potential for B-W materials:**

- **Final version of new semi-empirical interatomic potential for B-W materials, suitable for efficient large scale molecular dynamics (MD) simulations, is complete: Stable for W, B, W_2B, WB, WB_2, W_2B_5, WB_4.**
- Lattice stability, elastic properties, dimers and molecules (B-B, W-B, W-B, WB_x molecules) optimized.
- **Effective surface binding energies calculated:** Used for MD predictions of sputtering yields from W-containing surfaces with evolving composition and morphology and comparison to experiment.







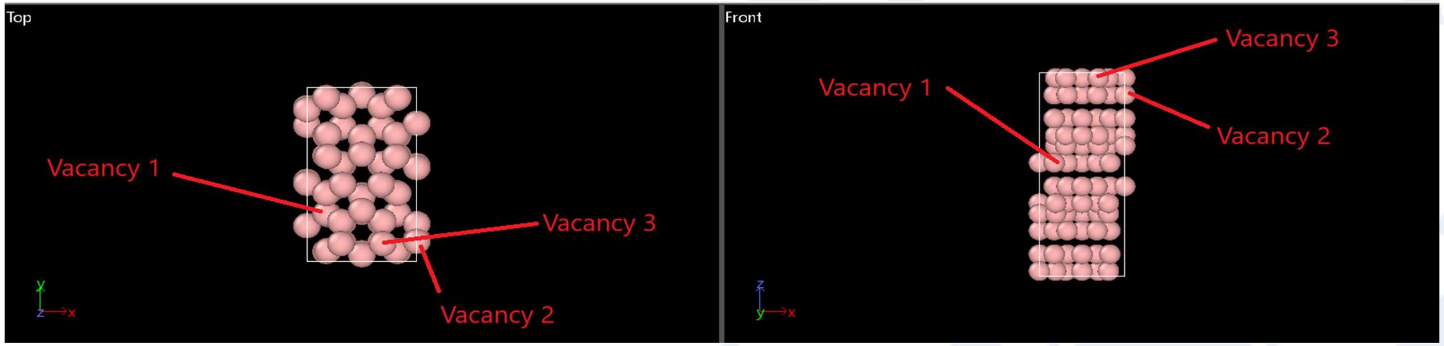
DFT and MD, defect energies in boron

Main observations

- Vacancy defect energies in B computed with DFT and MD
  - Only 3 different energy levels found for different vacancy sites

- Interstitials in B and defects in W-B to be completed with DFT
  - 100 different initial sites tested

		Vacancy 1		Vacancy 2		Vacancy 3	
		DFT	Potential	DFT	Potential	DFT	Potential
B	Energy	3.57	3.39	3.34	3.36	3.34	2.09
	Volume	-9.27	-1.49	-2.44	-1.44	0.00	-0.74



Interstitial 1		Interstitial 2		Interstitial 3		Interstitial 4		Interstitial 5		Interstitial 6		Interstitial 7		Interstitial 8	
DFT	Potential	DFT	Potential	DFT	Potential	DFT	Potential	DFT	Potential	DFT	Potential	DFT	Potential	DFT	Potential
	1.73		1.29		2.38		2.23		1.78		3.33		12.68		2.62

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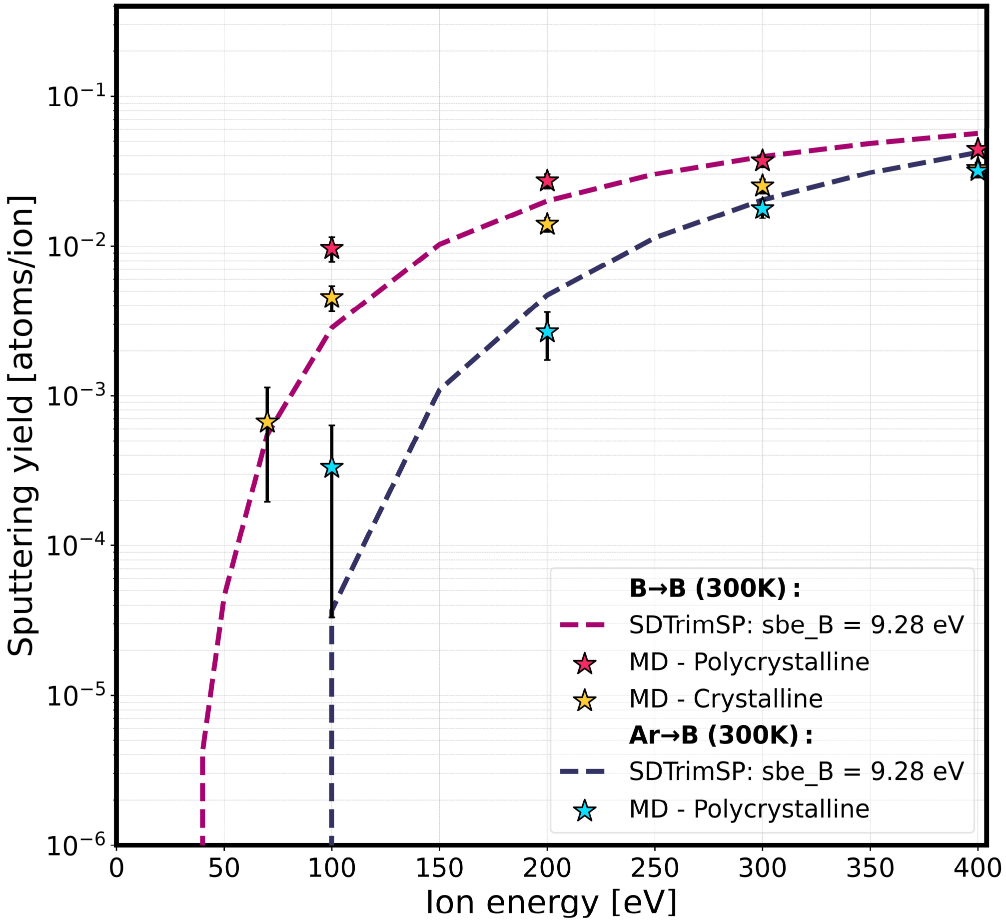
Sputtering from B surface with MD

Development of new interatomic potential



- Comparison with SDTrimSP shows agreement when surface binding energy set to the value predicted by the potential.
- Surface structure affects the yield in MD.
- This provides further validation of the new B potential, which is able to crystallize a random system.
- Surface binding energies somewhat overestimated – affects sputtering yields.
- MD cumulative sputtering of evolving W-B surfaces to be done.
- **Purely physical sputtering in this system.**
- **Future work: adding H/D/T would be interesting.**

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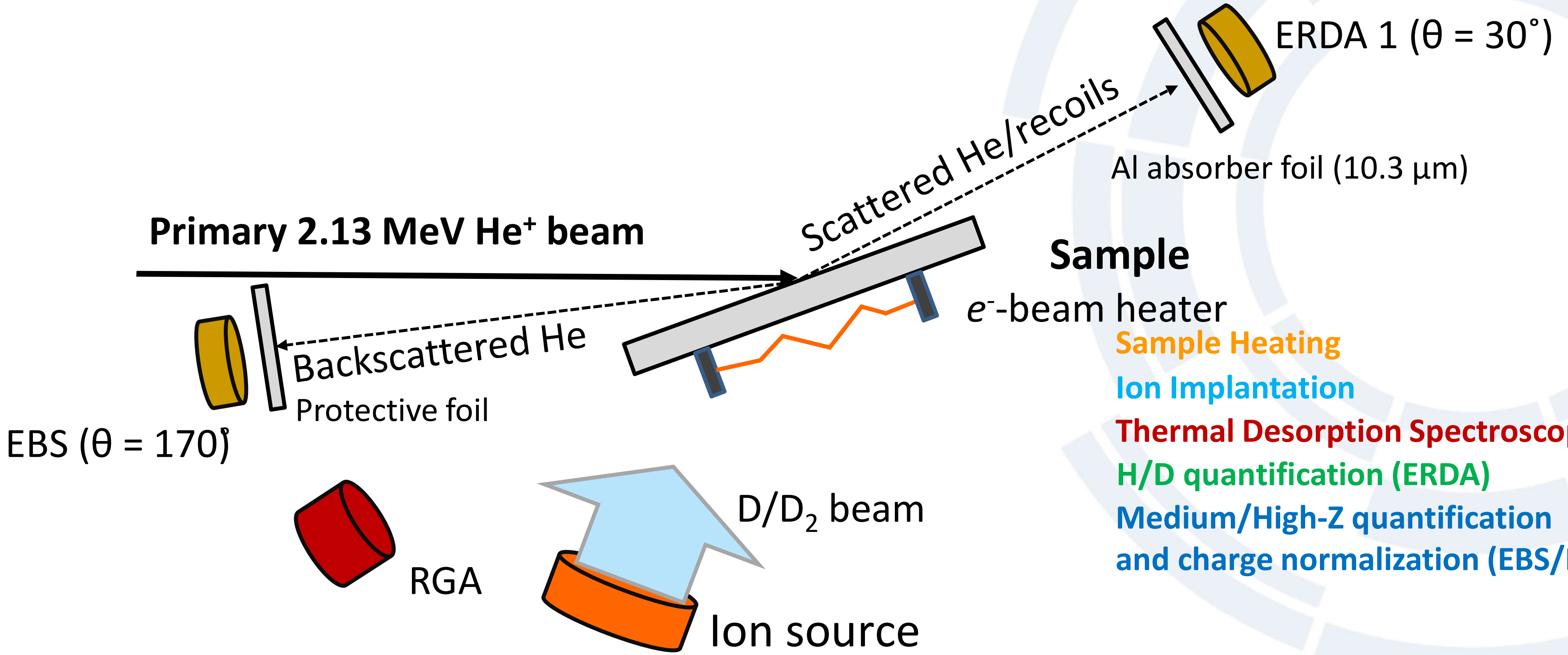
In-situ experiments of hydrogen and deuterium desorption

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



Sample Heating

Ion Implantation

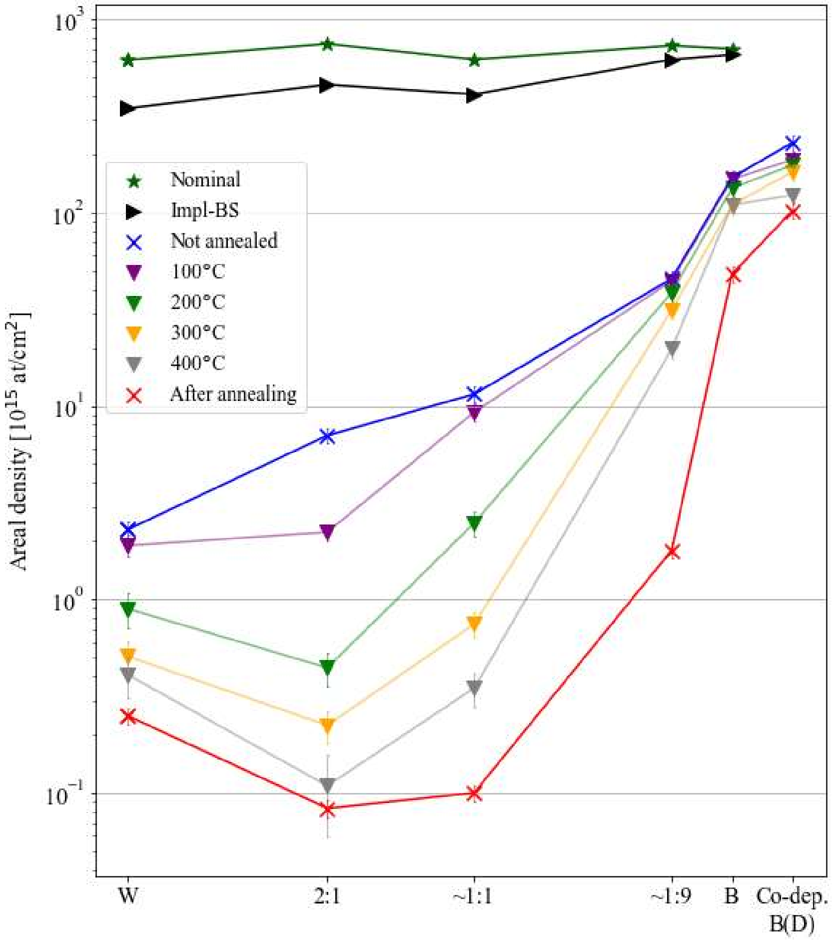
Thermal Desorption Spectroscopy (TDS)

H/D quantification (ERDA)

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

In-situ experiments of hydrogen and deuterium desorption

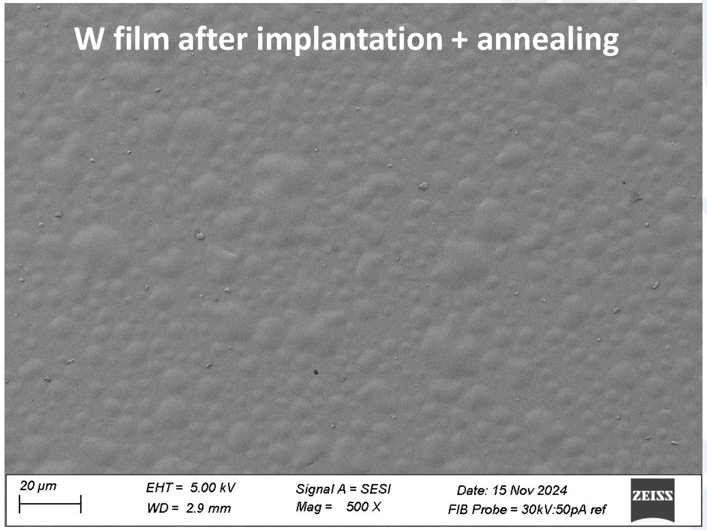
Simulations (TRIM) and experimental results:



D. N. Gautam et al. Nuclear Materials and Energy (2025) 45.

- Deuterium saturation scales with B content (blue line).
- W-B mixed layers present deuterium outgassing at lower temperatures (synergistic effect).
- Highest D content and stability in boron co-deposited in D_2 atmosphere (different incorporation than implanted).

Ex-situ investigation after D_2 implantation + annealing (highlights):



- Formation of bubbles only observed in W sample in D_2 implanted region.

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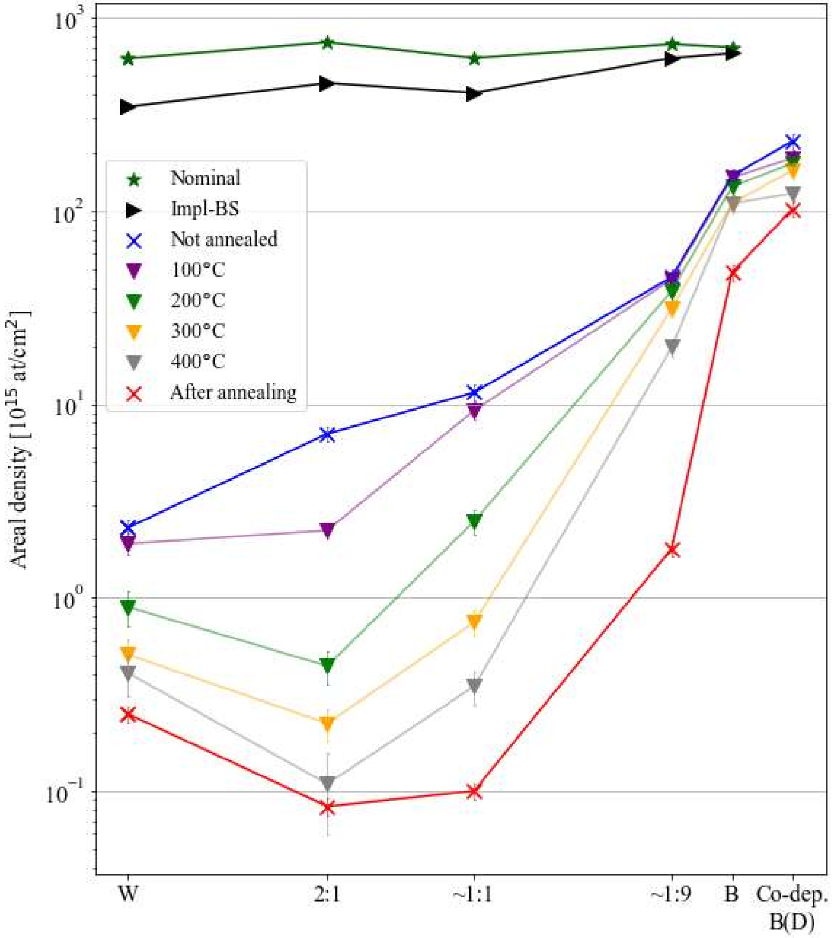

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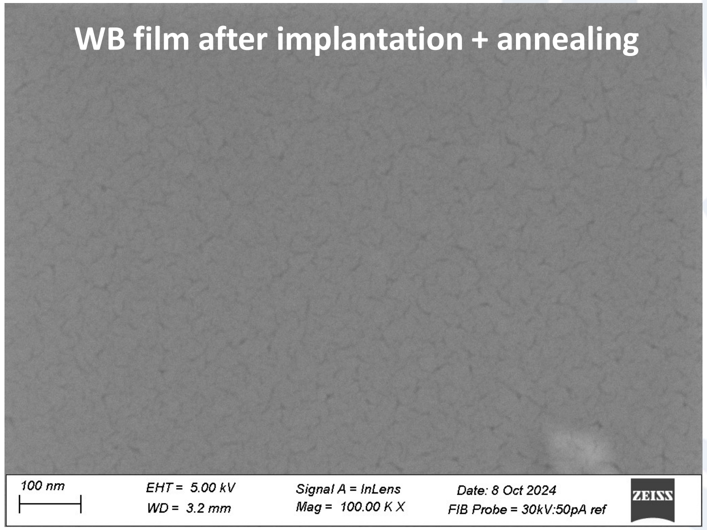
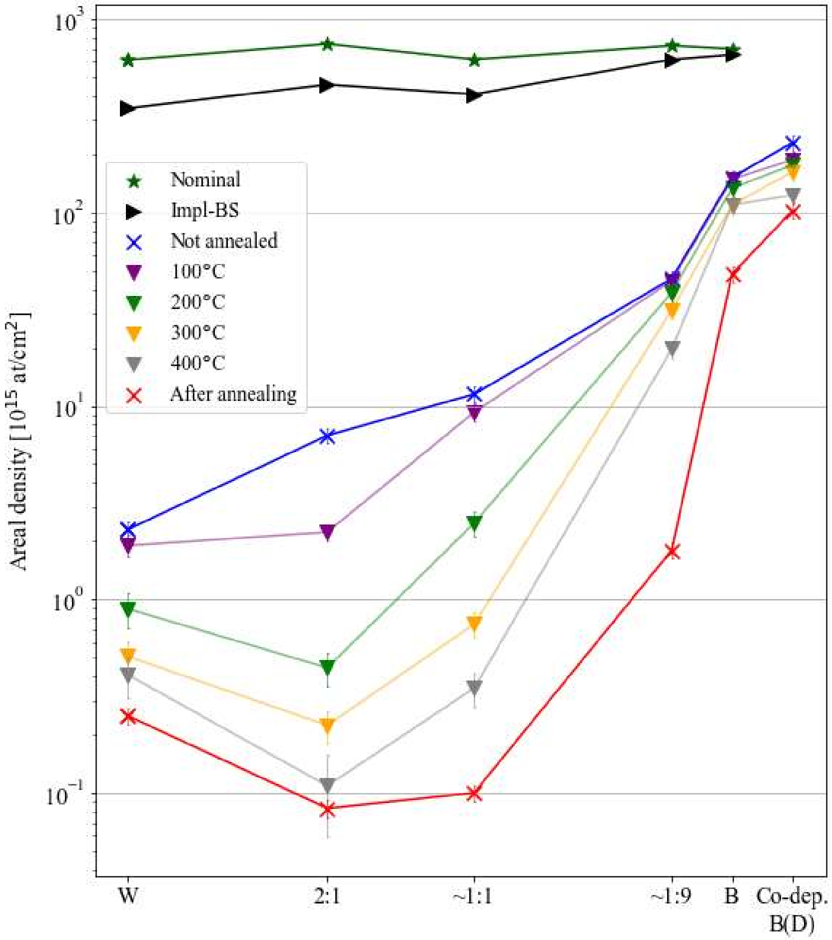


- Formation of bubbles only observed in W sample in D_2 implanted region.
- Formation of cracks in WB films (only in implanted region).

In-situ experiments of hydrogen and deuterium desorption

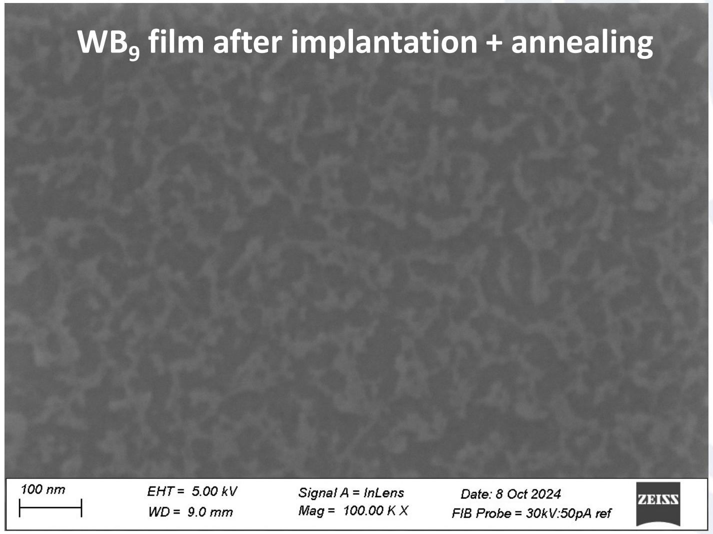
Simulations (TRIM) and experimental results:



D. N. Gautam et al. Nuclear Materials and Energy (2025) 45.

- Deuterium saturation scales with B content (blue line).
- W-B mixed layers present deuterium outgassing at lower temperatures (synergistic effect).
- Highest D content and stability in boron co-deposited in D_2 atmosphere (different incorporation than implanted).

Ex-situ investigation after D_2 implantation + annealing (highlights):

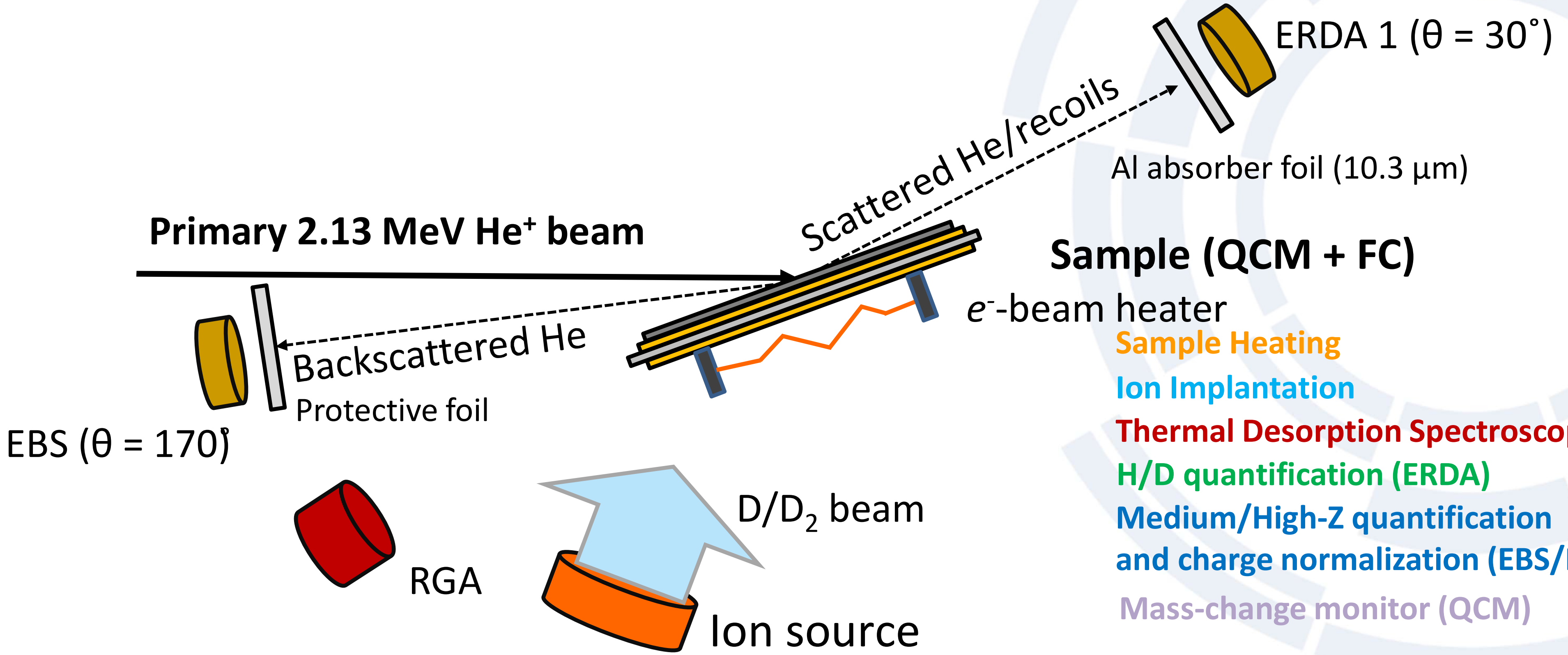


- Formation of bubbles only observed in W sample in D_2 implanted region.
- Formation of cracks in WB films (only in implanted region).
- W surface enrichment from preferential sputtering (confirmed with HR-RBS).

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



Sample Heating

Ion Implantation

Thermal Desorption Spectroscopy (TDS)

H/D quantification (ERDA)

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

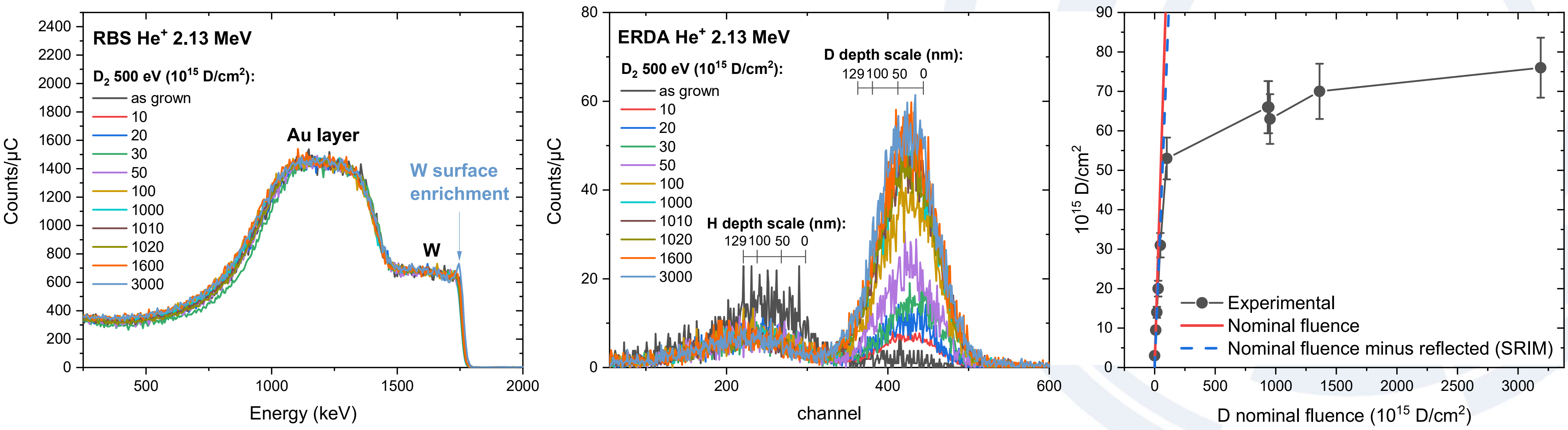
Mass-change monitor (QCM)

In-situ IBA and QCM analysis of a W-B film during deuterium implantation

To correlate real-time mass changes observed via QCM with compositional modifications in a W-B film during deuterium implantation: sputtering effects, deuterium retention and saturation, and tungsten surface enrichment.

Sample: *~130 nm of boron-rich W-B (21.4 at.% W) on a QCM sensor.*

IBA results:

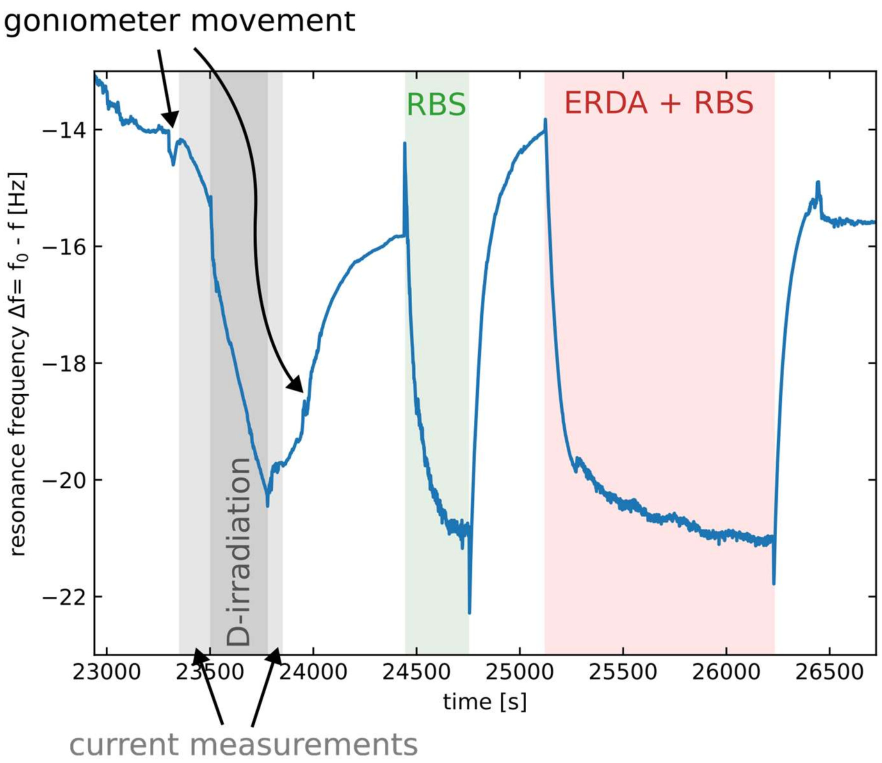


In-situ IBA and QCM analysis of a W-B film during deuterium implantation

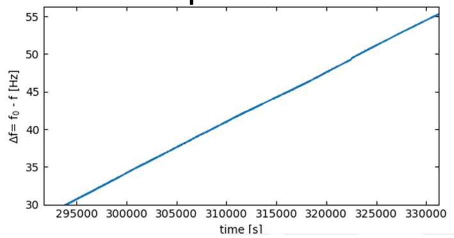
To correlate real-time mass changes observed via QCM with compositional modifications in a W-B film during deuterium implantation: sputtering effects, deuterium retention and saturation, and tungsten surface enrichment.

Sample: *~130 nm of boron-rich W-B (21.4 at.% W) on a QCM sensor.*

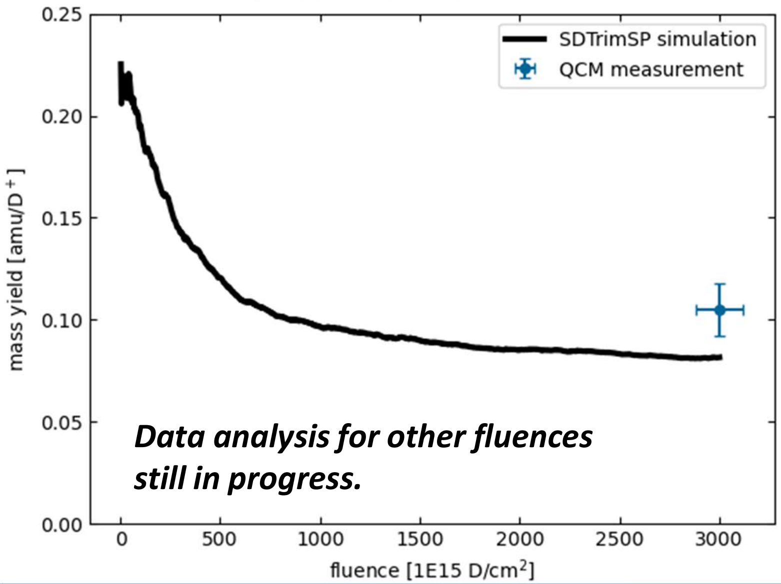
QCM Signal: affected by MeV-beam and goniometer movement.



After an equilibrium is reached, only sputtering remains:



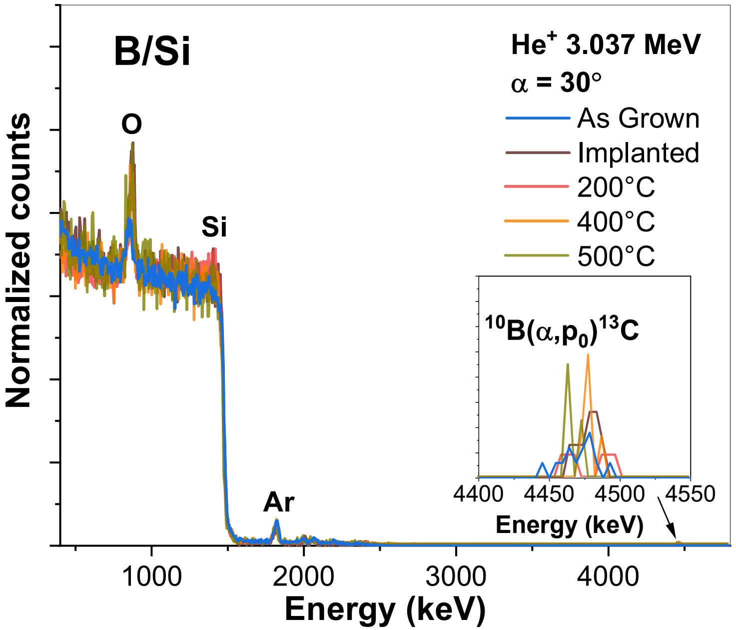
- → Increase in *f*: mass loss.
- → Sputter yield can be measured.
- → Comparison with SDTrimSP dynamic simulation:

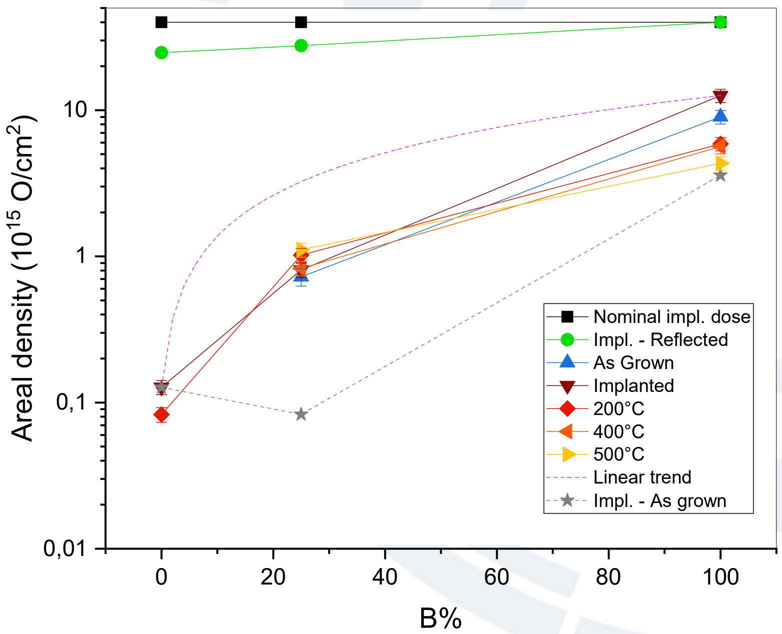


In-situ experiments of oxygen retention in W-B layers

Effect of mixture of W-B in the oxygen gettering properties from boron

O_2^+ implantation (0.5 keV per atom) in B, W, and W-B layer.
In-situ IBA before/during/after thermal annealing: Quantify oxygen trapped and how easy it is do desorb it.
Preliminary results: Less oxygen in W-B mixture as it would be expected from linear interpolation.
Indication that W-B affects reduces oxygen getter properties.
In progress: ^{18}O implantation (differentiate from contamination); Effect of pre-irradiated D_2 in oxygen retention.





Overview of main scientific achievements

WP 1: Sample preparation & characterization:

Deposition routines to grow B and mixed PFC-B (including co-deposition in D_2): Significant contribution to PWIE.
Mechanical characterization of mixed PFC-B: Significant influence of boron in hardness and elastic properties.

WP 2: Sputtering Yields and BCA simulations

Measurement of SY for D_2 and Ar in function of composition and incident angle: discrepancies with BCA models identified.
Successful implementation of QCM+IBA in-situ combination: correlate mass-change processes with change of composition in real-time.

WP 3: Ab inito calculations of defect energetics and mixed B-W materials

Vacancy defect energies in B computed with DFT and MD.
Binding energies indicate a stronger adhesion between boron and tungsten for (110) surface orientation than for (100, 111) orientations.

WP 4: Molecular dynamics modelling of sputtering yields and surface modification

Development of new semi-empirical interatomic potential for B-W materials completed.
MD sputtering yields from W and B surface layers can be performed (to be completed before the end of project).

WP 5: Material Modification and Atomic Migration

In-situ quantification of deuterium retention in mixed W-B layers in function of the composition: W-B layers present lower retention in comparison to simple extrapolations from pure W and B.
Oxygen presence in W-B is lower than pure B: possible consequences in oxygen getter properties.
B/W structures are highly stable, while strong interdiffusion is observed in redeposited B/EUROFER structures.

Acknowledgement

UPPSALA UNIVERSITET

Daniel N. Gautam
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Daniel Primetzhofer

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Marek Rubel

TU WIEN

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Raphael Gurschl
Benjamin B. Domazet
Helmut Riedl
Friedrich Aumayr

Aalto University

Antoine Clement
Nima Mofrad
Andrea E. Sand

Thank you!

EUROfusion

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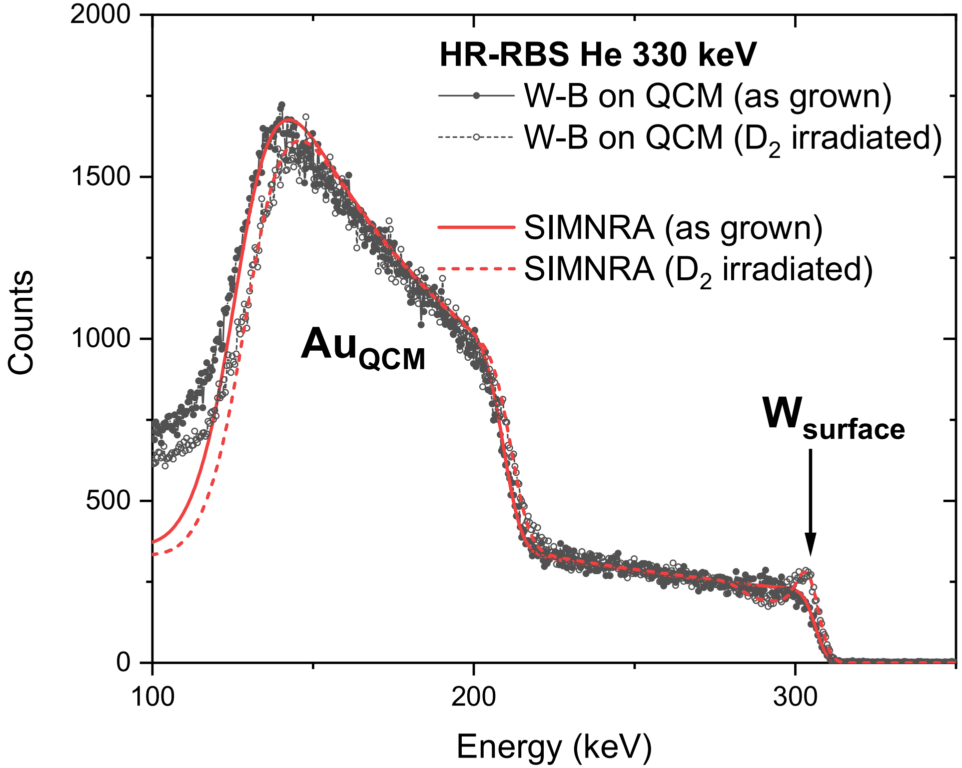
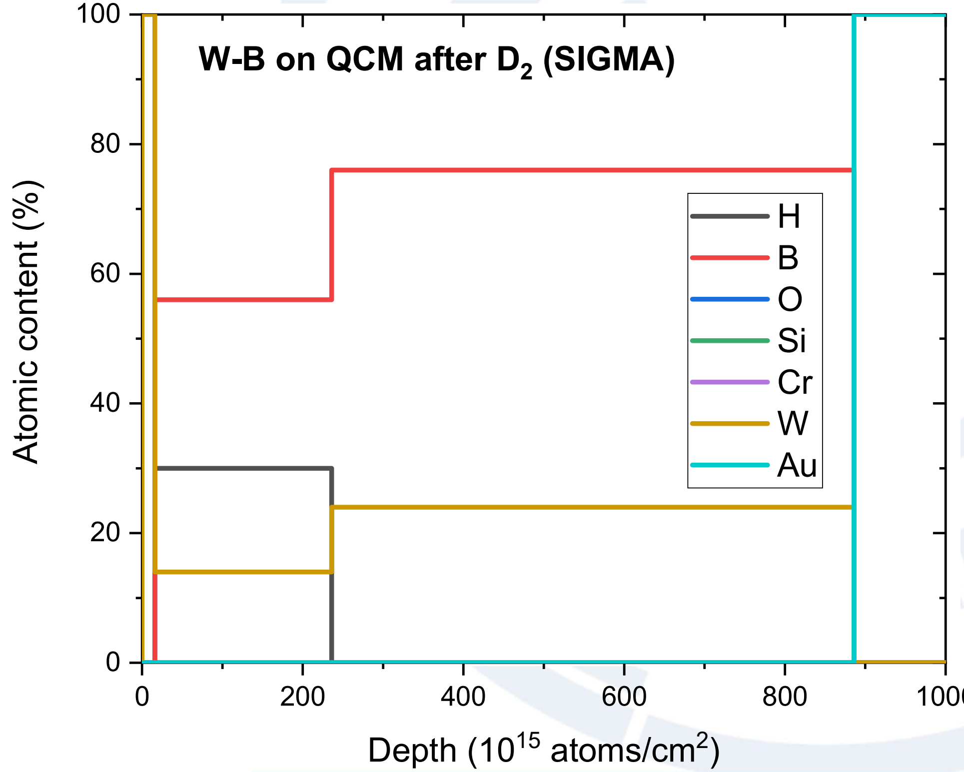
In-situ IBA and QCM analysis of a W-B film during deuterium implantation

To correlate real-time mass changes observed via QCM with compositional modifications in a W-B film during deuterium implantation: sputtering effects, deuterium retention and saturation, and tungsten surface enrichment.

Sample: *~130 nm of boron-rich W-B (21.4 at.% W) on a QCM sensor.*

Ex-situ characterization: HR-RBS of sample after D_2 implantation (3×10^{18} D/cm²). W surface enrichment of ~2.4 nm.





Team

VR	ÖAW	VTT (Aalto)
Eduardo Pitthan (PI-UU)	Martina Fellinger (IAP)	Andrea Sand
Daniel Gautam (UU)	B. Burazor Domazet (IAP)	Antoine Clement
Per Petersson (KTH)	Raphael Gurschl (IAP)	Nima Fakhrayi
Collaborators (UU)*	Helmut Riedl (ASCT)	Akseli Aro

VR main tasks: Sample preparation and characterization, in-situ experiments.
**Tuan Tran (SEM, TEM, EDX); Dmitry Moldarev (XRD); Rajdeep Kaur (AFM).*

ÖAW main tasks: Sputtering yield measurements, and BCA-based simulations. Film deposition and mechanical characterization.

VTT main tasks: Computational modelling (binding energies and interatomic potential).

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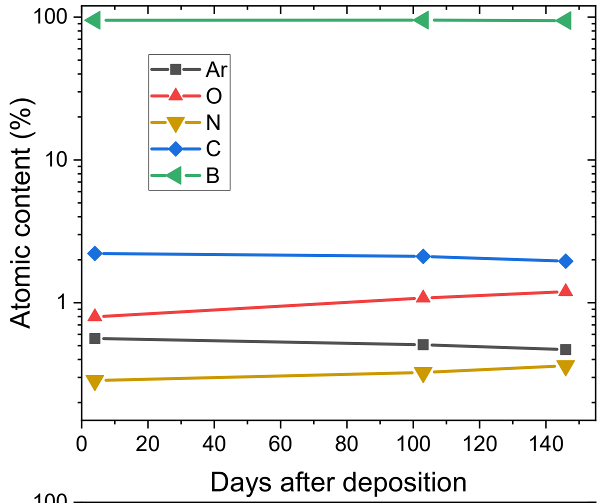
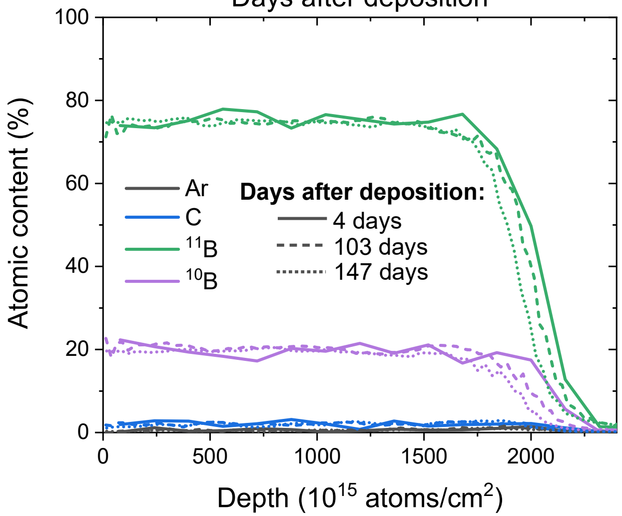
W-B film characterization: aging effect

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).

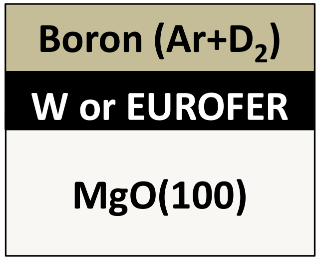
W-B mixed layers presented good stability overtime: no change of composition and thickness.

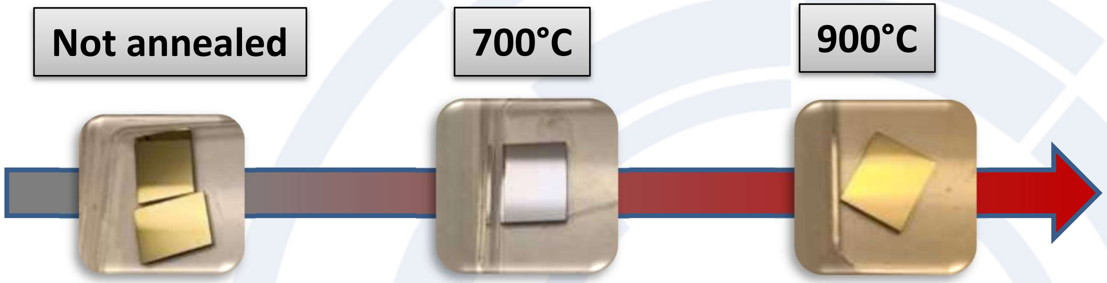
Sample characterization immediately before/during each experiment.

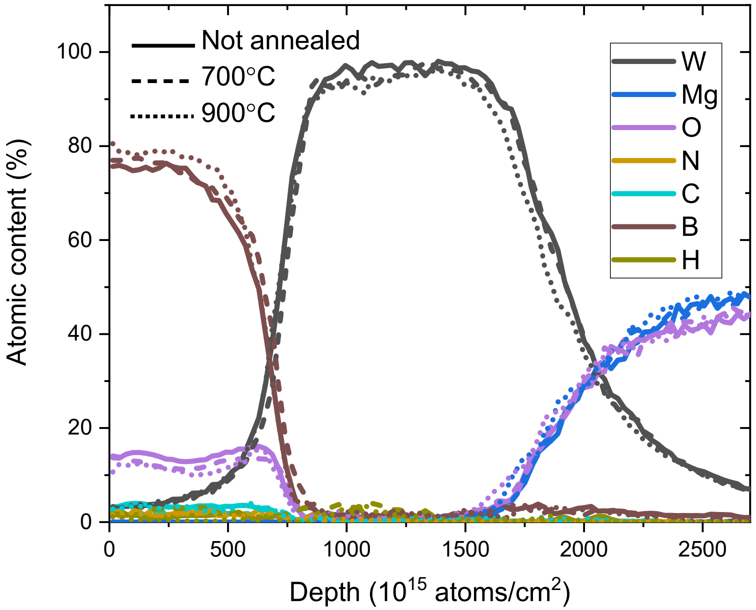
Boron migration in re-deposited PFC

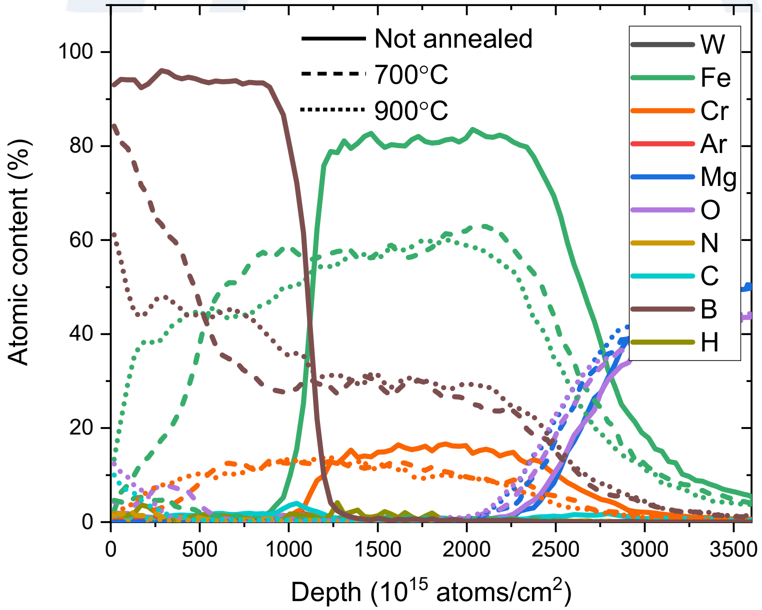
Effect of annealing in B/PFC stack layers

- Effect of annealing at higher temperatures was performed and analyzed ex-situ in different structures to evaluate atomic migration of boron on re-deposited PFC (W&EUROFER97).
- Significant temperature dependence in the intermixing of boron with sputter-deposited layers from EUROFER97.









Schedule 2024

Deliverables and Milestones for each year per working package

W-P 1	Sample Preparation and Characterization
Responsible	Eduardo Pitthan Filho (VR)
Deliverable	D1.1 Deposition of mixed layers of W and B in Ar atmosphere. D1.2 Deposition of mixed layers of EUROFER97 and B in Ar atmosphere. D1.3 Deposition of mixed layers of W and B in Ar and D2 atmosphere. D1.4 Deposition of mixed layers of EUROFER97 and B in Ar and D2 atmosphere. D1.5 Chemical and morphological characterization of mixed layers. D1.6 Characterization of mechanical properties of mixed layers with W. D1.7 Characterization of mechanical properties of mixed layers with EUROFER97.
Milestone	M1.1 Completion of preparation (formation and characterization) of W and B mixed layers in comparison to pristine bulk materials. M1.2 Completion of preparation (formation and characterization) of EUROFER97 and B mixed layers in comparison to pristine bulk materials. M1.3 Completion of preparation (formation and characterization) of W and B mixed layers with D2. M1.4 (2025) Completion of preparation (formation and characterization) of EUROFER97 and B mixed layers with D2.

W-P 2	Sputtering Yields and BCA simulations
Responsible	Martina Fellinger (ÖAW)
Deliverable	D2.1 Measurement of sputtering yields of mixed layers with W.
Milestone	M2.1 Sputtering Yields of W and B mixed layers.

Schedule 2024

Deliverables and Milestones for each year per working package

W-P 3	Ab inito calculations of defect energetics and mixed B-W materials
Responsible	Andrea Sand (VTT)
Deliverable	D3.1 Ab initio calculations of mixed B-W bulk materials with varying stoichiometry. D3.2 An initio calculations of B-W defect energies
Milestone	M3.1 Ab initio data for bulk W-B structures with varying stoichiometry M3.2 Ab initio data on B-W defects

W-P 4	Molecular dynamics modelling of sputtering yields and surface modification
Responsible	Andrea Sand (VTT)
Deliverable	D4.1 Validated B-W potential for MD simulations.
Milestone	M4.1 (beginning of 2025) Validated interatomic potential for W-B systems suitable for MD simulations.

No deliverable/milestone proposed for W-P 5 in 2025.

Other information:
WIKI pages project documenting area
Document : team , deliverables, scope, meetings, results, WPs links, use of experimental data, reports, publications.
https://wiki.euro-fusion.org/wiki/WPENR_wikipages:_Enabling_Research_Work_Package

INDICO – Meetings & Presentations
Materials uploaded there will remain for the entire Horizon Europe framework: https://indico.euro-fusion.org/category/405/

Schedule 2025

W-P 2	**Sputtering Yields and BCA simulations**
Responsible	Martina Fellinger (ÖAW)
Deliverable	D2.2 Measurement of sputtering yields of mixed layers with EUROFER97. D2.3 In-situ measurement of sputtering yields in function of surface composition of mixed layers.
Milestone	M2.2 Sputtering Yields of EUROFER97 and B mixed layers. M2.3 Sputtering yield in function of surface composition from ion irradiated structures.
W-P 3	**Ab inito calculations of defect energetics and mixed B-W materials**
Responsible	Andrea Sand (VTT)
Deliverable	D3.3 Ab initio calculations of B adsorption and diffusion on B-W surfaces
Milestone	M3.3 Ab initio calculations of B adsorption and diffusion on B-W mixed surfaces
W-P 4	**Molecular dynamics modelling of sputtering yields and surface modification**
Responsible	Andrea Sand (VTT)
Deliverable	D4.2 MD sputtering yields from W and B surface layers D4.3 MD predictions of sputtering yields from W-containing surfaces with evolving composition and morphology and comparison to experiment
Milestone	M4.2 MD-predicted sputtering yields of W and B mixed layers. M4.3 MD sputtering simulations of yields as function of surface composition and comparison to experiment.
W-P 5	**Material Modification and Atomic Migration**
Responsible	Eduardo Pitthan Filho (VR)
Deliverable	D5.1 In-situ annealing of W and B mixed layers monitoring atomic modifications in chemical composition and depth profiling. D5.2 In-situ annealing of WEUROFER97 and B mixed layers monitoring atomic modifications in chemical composition and depth profiling. D5.3 Exposure of mixed layers to 18O2 and monitoring of oxygen incorporation and depth profile by ion beam analysis.
Milestone	M5.1 Characterization of modifications of W and B mixed layers submitted to annealing. M5.2 Characterization of modifications of EUROFER97 and B mixed layers submitted to annealing. M5.3 Comprehension of the influence of composition of mixed-layers in the oxygen in-take by in-situ measurements.

Objectives

i. to deposit thin films from PFC candidate materials (i.e. W and EUROFER97) simultaneously with boron under argon and isotopically-enriched atmospheres to obtain mixed layers of these materials.

ii. To characterize the composition, microstructure, mechanical properties, deuterium retention, and sputtering yields of these layers and evaluate quantitatively the deviations from its original bulk counterparts.

iii. to model sputtering yields by molecular dynamics (MD) and binary collision approximation simulations to evaluate dynamic changes of sputtering rates due to change of composition and compare to the ones obtained experimentally.

iv. to model changes in surface nano-structure by MD due to ion/plasma exposure and evaluate possible changes in sputtering yields.

v. to investigate the modifications of these materials caused by exposure to ion fluxes and thermal annealing by means of in-situ and ex-situ experiments and with focus on high-sensitivity surface characterization.

In-situ experiments of hydrogen and deuterium desorption

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

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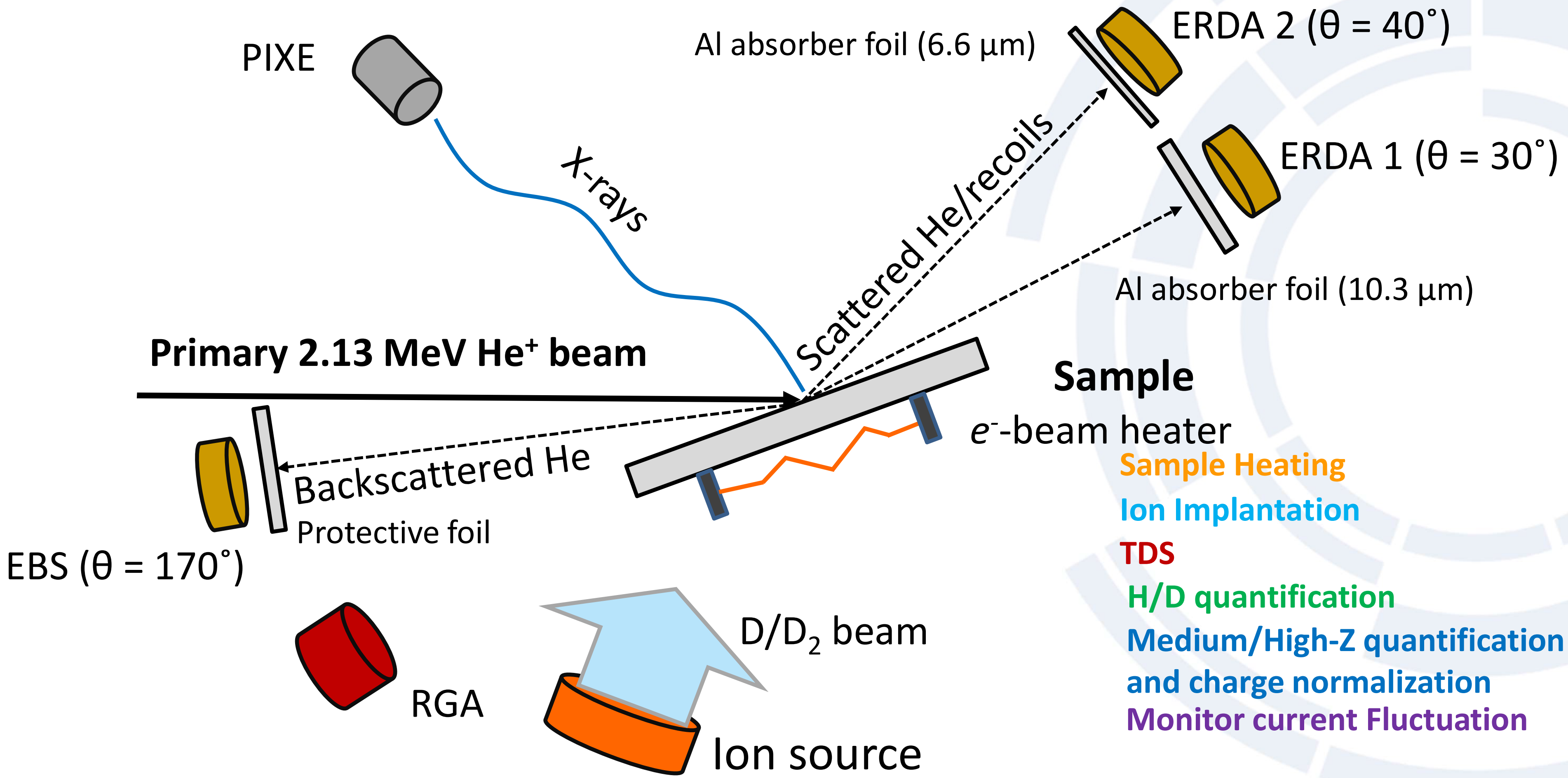


W-B film characterization (100-150 nm films)

Sputtering depositions under argon atmospheres:

B target (RF-150 W):

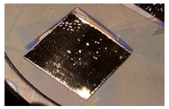
0.5 nm/min; 5 hours deposition.

Substrate effect:

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

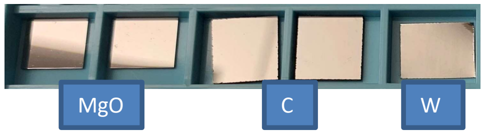
Improved on W substrates by:

- 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.

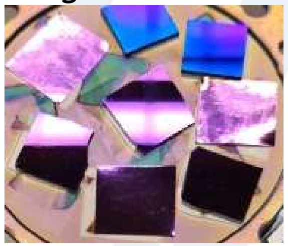


Sputtering depositions under argon Ar+D_2:

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

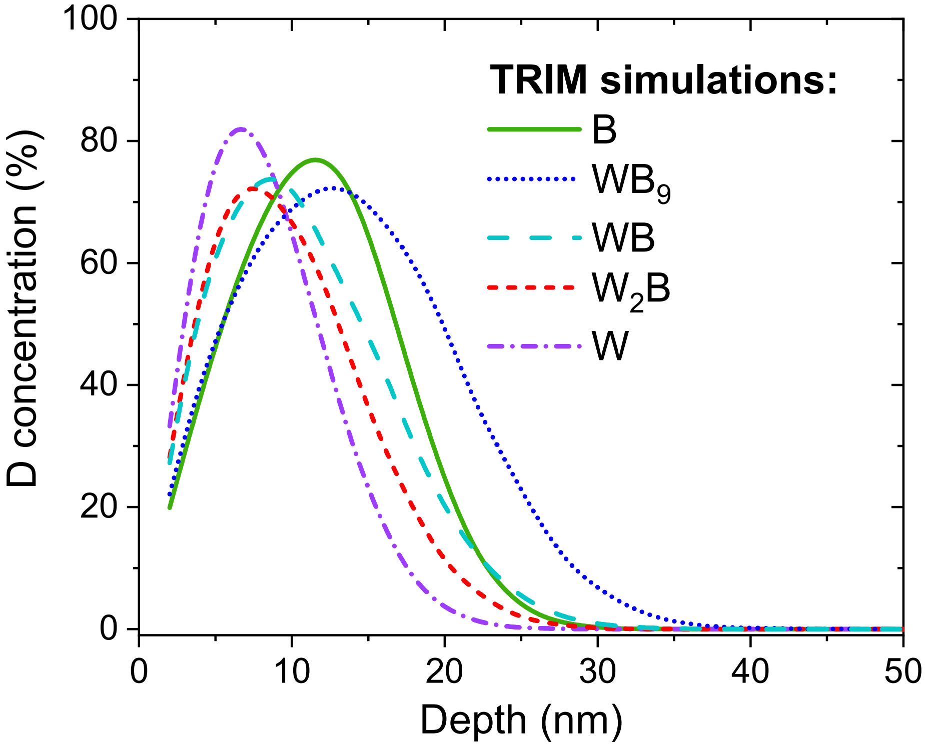
- Layers are stable in vacuum, flat and homogeneous.
- Change of color within minutes in all substrates.
- Layers remain flat on all substrates.

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

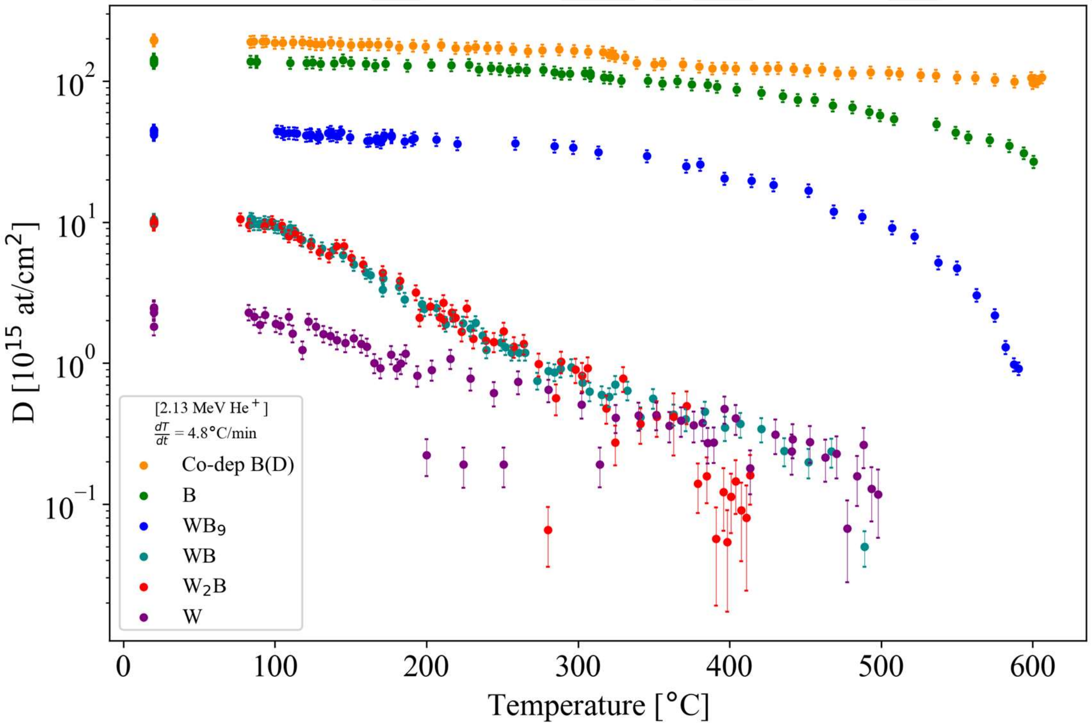
- Similar as in Ar depositions: flat, homogeneous, stable in air.

In-situ experiments of hydrogen and deuterium desorption

Deuterium implantations in W-B layers on Si:
1 keV D_2^+ implantation fluence ≈ 6×10^{17} at/cm^2.
Implantation range within 40 nm.



In-situ measurements after implantation:
Monitoring D content during annealing.
Lower deuterium content than nominal values (beyond saturation).



Theoretical Results (WP3 and WP4)

Main objectives

Objectives of WP 3

- Ab initio calculations using VASP for solid tungsten borides with varying stoichiometries, including the experimentally synthesized W_2B, W_2B_5 and WB_4, as well as different experimentally observed phases of WB.
- Formation and binding energies of B-W defect complexes in bulk W and mixed B-W materials.
- Ab initio calculations of the adsorption and diffusion behaviour of boron atoms along and into mixed B-W surfaces for varying surface composition and structure.

Objectives of WP 4

- Develop new semi-empirical interatomic potential for B-W materials, suitable for efficient large scale molecular dynamics (MD) simulations.
- High through-put MD simulations of sputtering yields of mixed B-W layers using the new potential, using as initial composition input the stoichiometry obtained experimentally.
- Comparison of results with BCA calculations, and to experimental data obtained in W-P 2.
- Modelling of changes in composition due to preferential sputtering will be carried out, and the changes in morphology of surfaces due to cumulative impacts predicted from different initial composition, compared with morphologies obtained experimentally from W-P 1.

Deliverables for 2024

WP 1: Sample preparation & characterization:

D1.1 Deposition of mixed layers of W and B in Ar atmosphere.
D1.2 Deposition of mixed layers of EUROFER97 and B in Ar atmosphere.
D1.3 Deposition of mixed layers of W and B in Ar and D2 atmosphere.
D1.4 Deposition of mixed layers of EUROFER97 and B in Ar and D2 atmosphere.
D1.5 Chemical and morphological characterization of mixed layers.
D1.6 Characterization of mechanical properties of mixed layers with W.
D1.7 Characterization of mechanical properties of mixed layers with EUROFER97.

WP 2: Sputtering Yields and BCA simulations

D2.1 Measurement of sputtering yields of mixed layers with W.

WP 3: Ab inito calculations of defect energetics and mixed B-W materials

D3.1 Ab initio calculations of mixed B-W bulk materials with varying stoichiometry.
D3.2 An initio calculations of B-W defect energies

WP 4: Molecular dynamics modelling of sputtering yields and surface modification

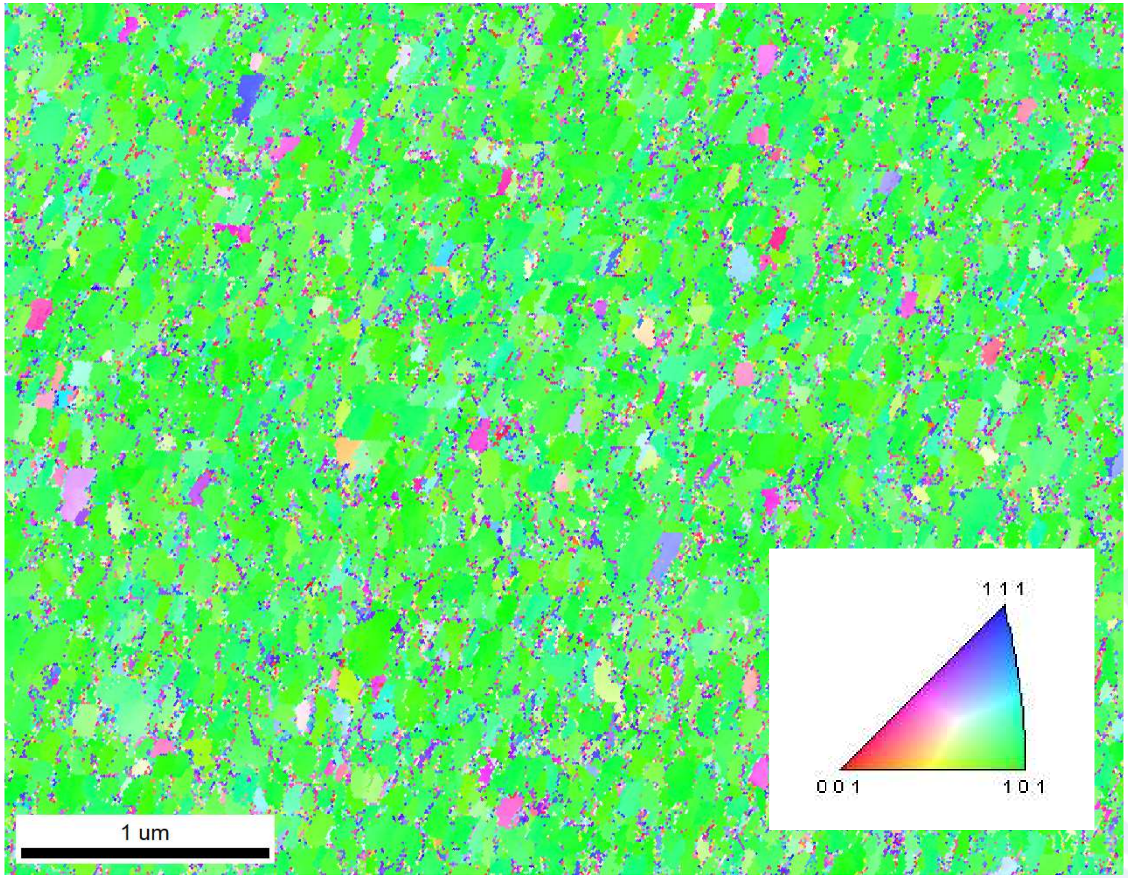
D4.1 Validated B-W potential for MD simulations.

WP 5: Material Modification and Atomic Migration

No activities planned for 2024.

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

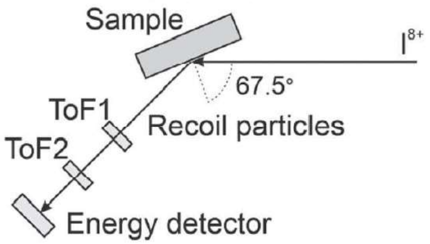
EBSD for W layer



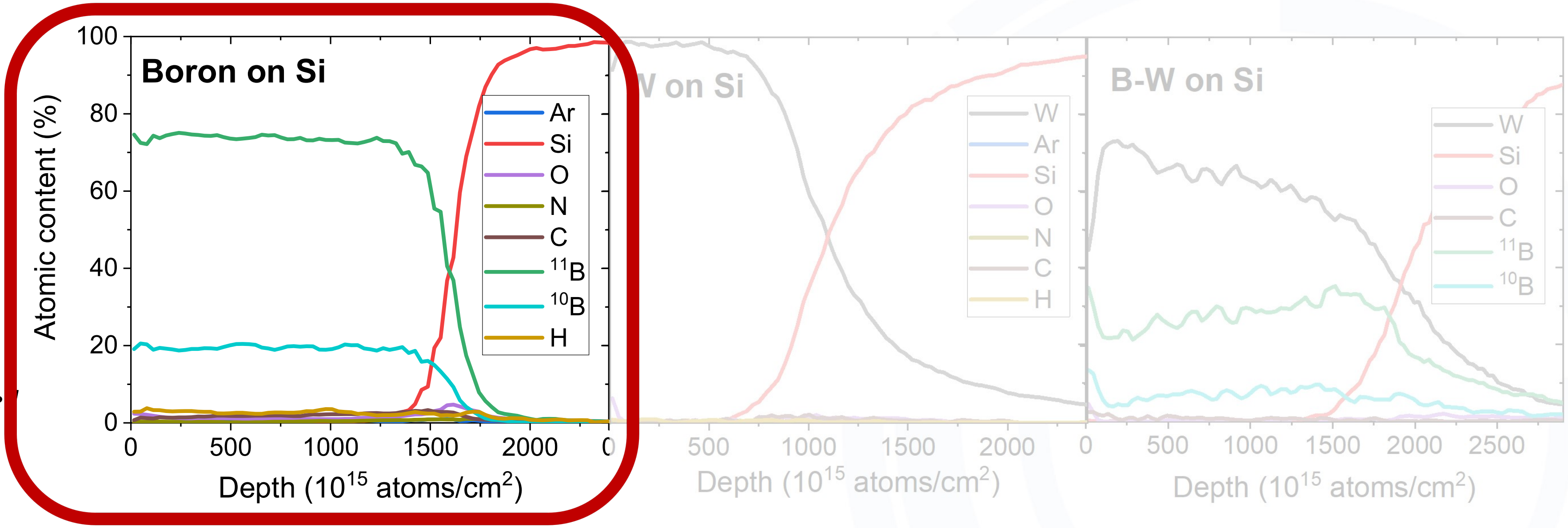
W-B film characterization

Sputtering depositions under argon atmospheres.

Atomic concentration depth profiles by ToF-ERDA:

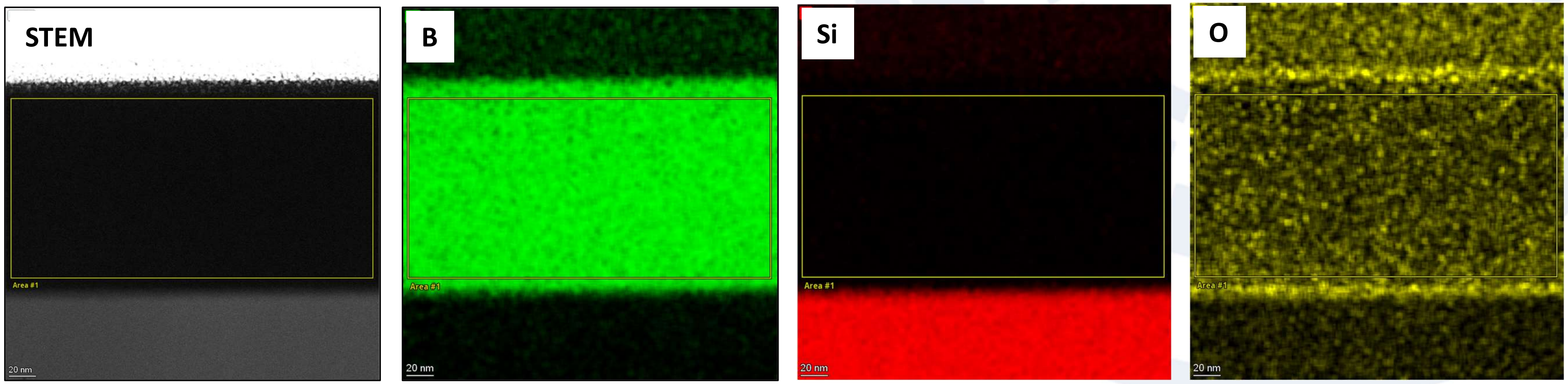


Average film composition calculated from 150-500×10^{15} atoms/cm^2



Boron on Si:

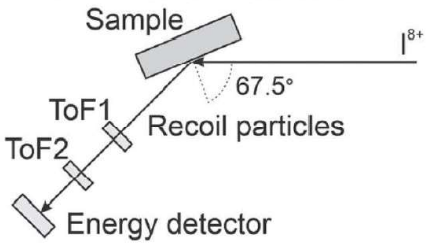
- B at. % >95;
- amorphous (TEM);
- 96% of bulk density; (RBS+TEM);
- EDX → $O_{surf/interf}$



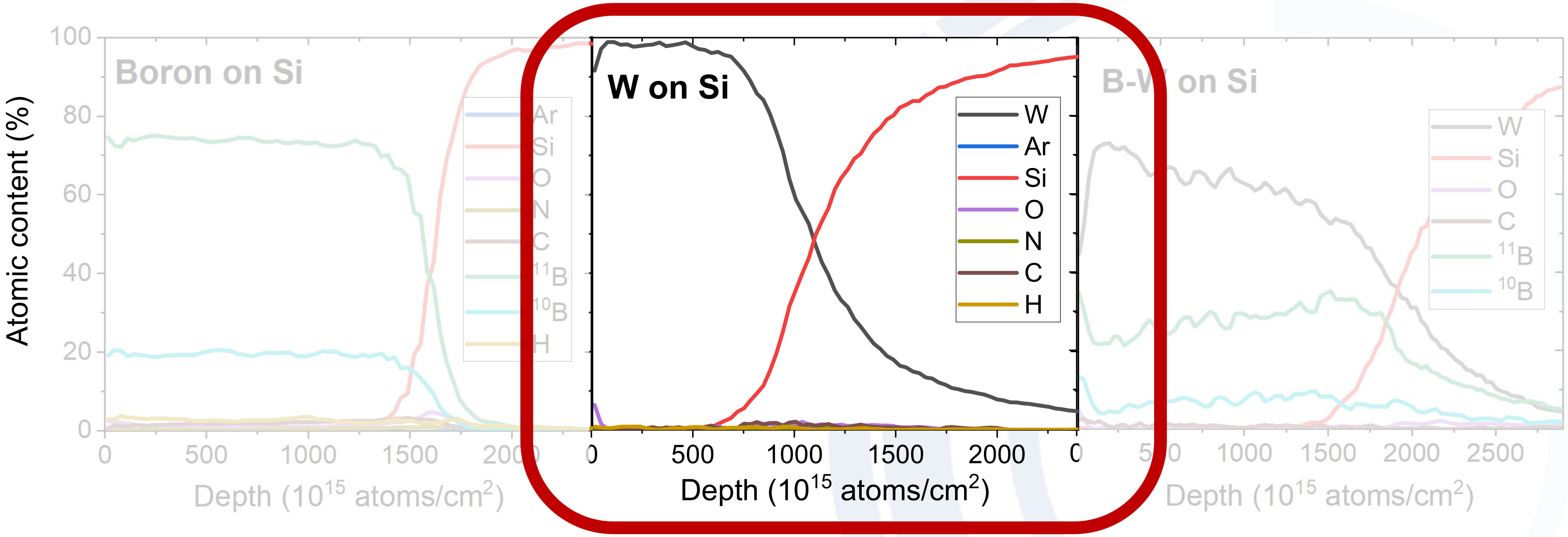
W-B film characterization

Sputtering depositions under argon atmospheres.

Atomic concentration depth profiles by ToF-ERDA:

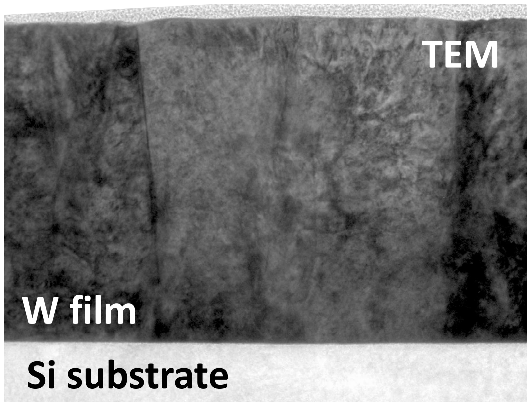


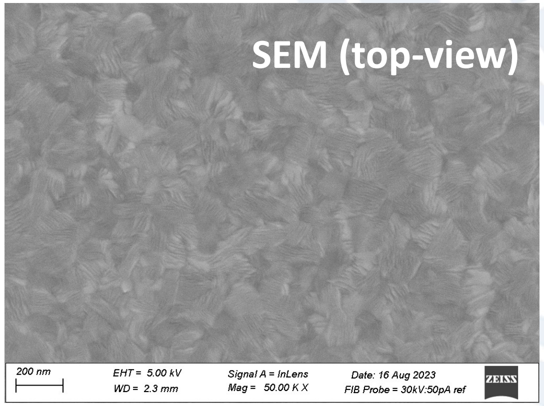
Average film composition calculated from 150-500×10^{15} atoms/cm^2



W on Si:

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

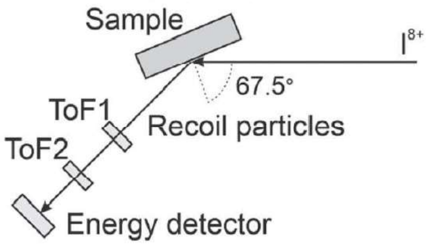




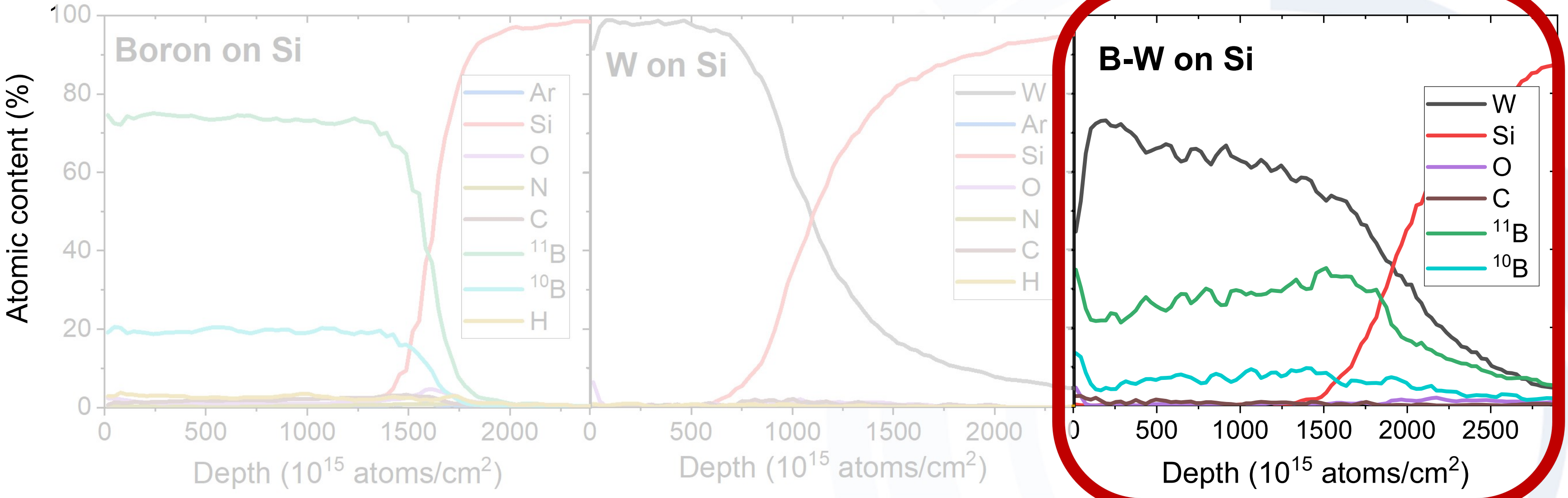
W-B film characterization

Sputtering depositions under argon atmospheres.

Atomic concentration depth profiles by ToF-ERDA:

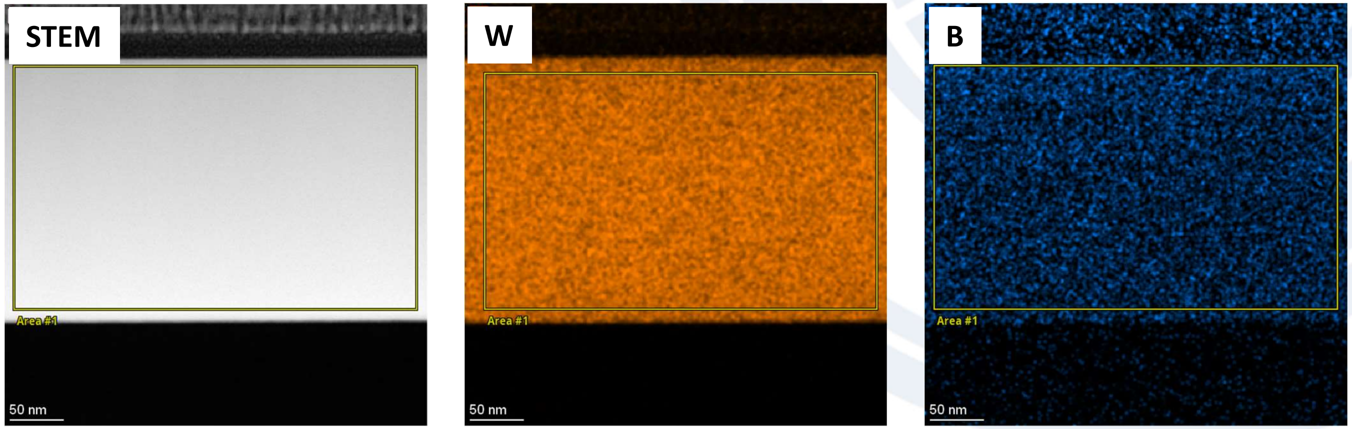


Average film composition calculated from 150-500×10^{15} atoms/cm^2



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).



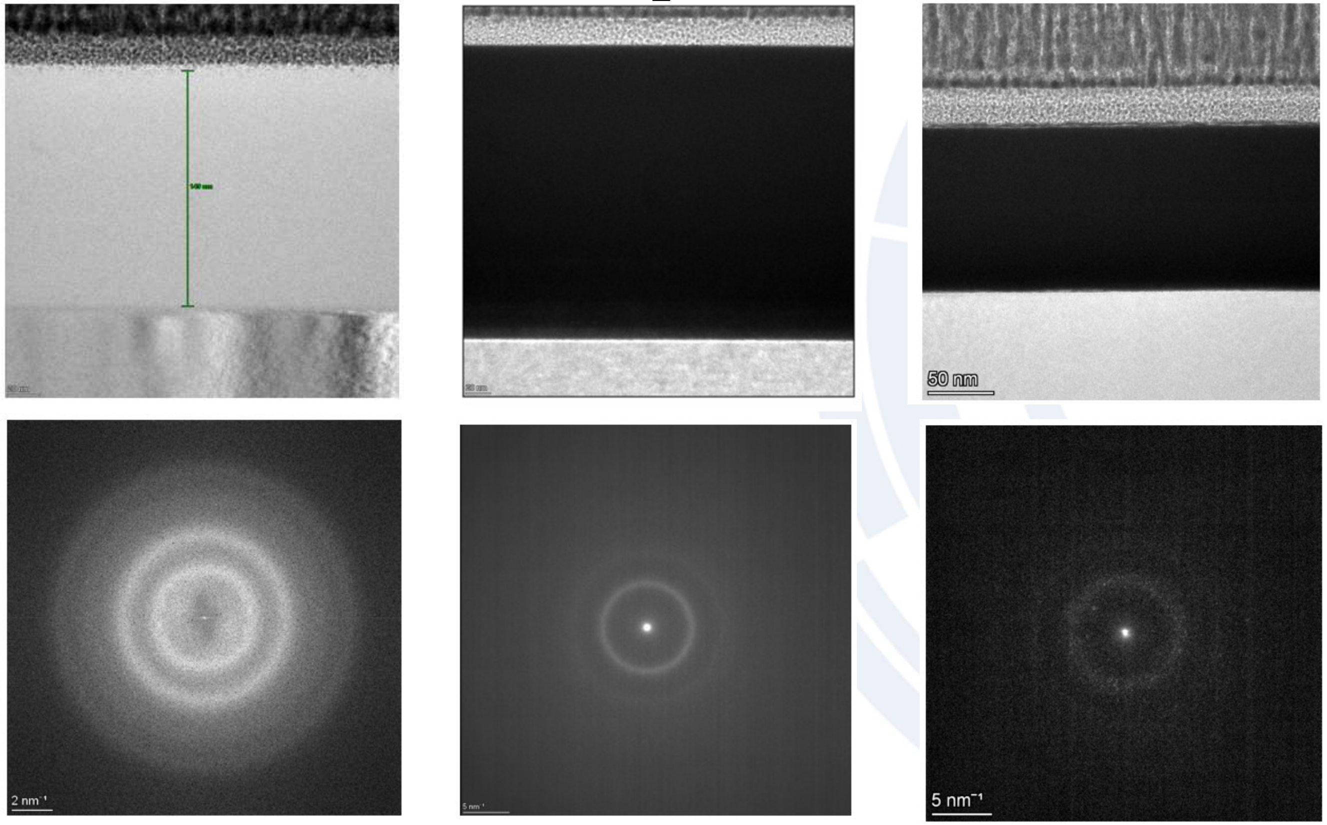
W-B film characterization (100-150 nm films)

TEM for W-B layers

B/Si | W_2B/Si | WB/Si

TEM

Electron-diffraction of selected regions

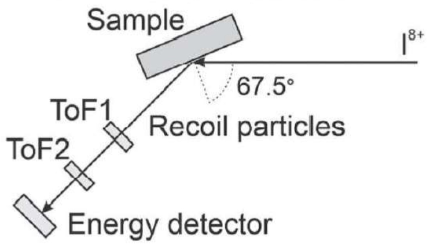


Amorphous B and W-B layers

W-B film characterization

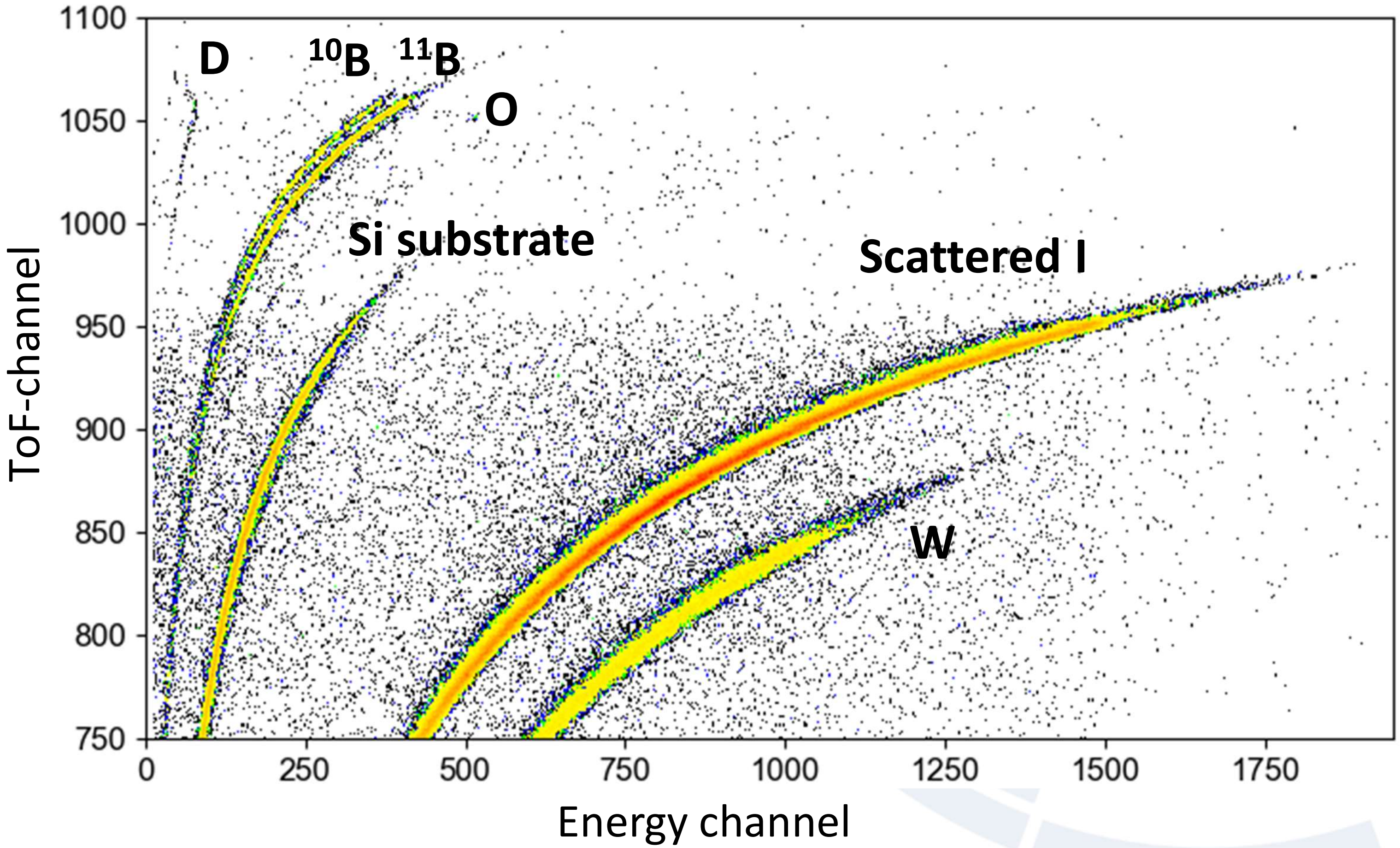
Sputtering depositions under argon and deuterium atmospheres:
f_{Ar} = 10 sccm; f_{D2} = 18 sccm; P_{Ar+D2} = 7×10^{-3} mbar.

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.**



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.

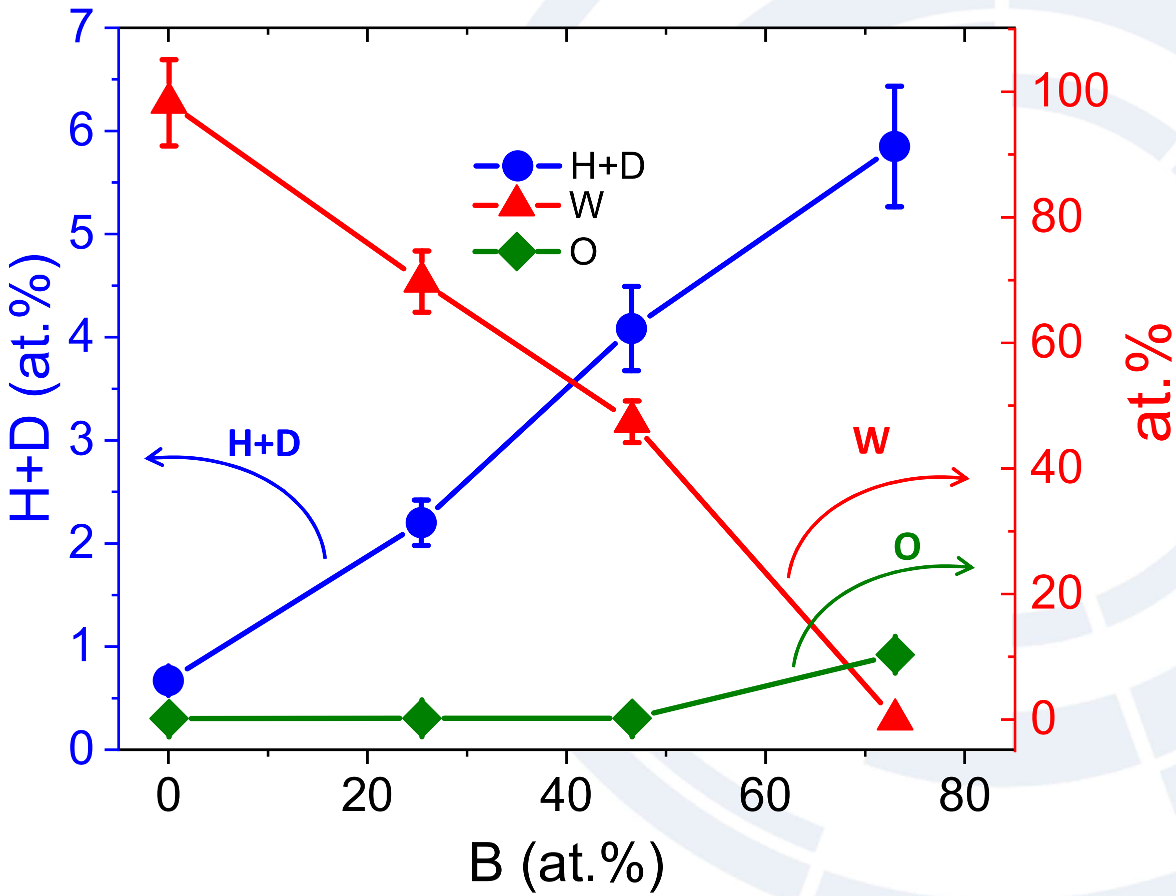
→ **f_{Ar} and f_{D2} fixed;**
→ **W and B magnetron power varied to obtain different B/W ratios.**

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

→ **Hydrogen and deuterium variation attributed to isotopic exchange from air exposure and different aging of samples.**

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

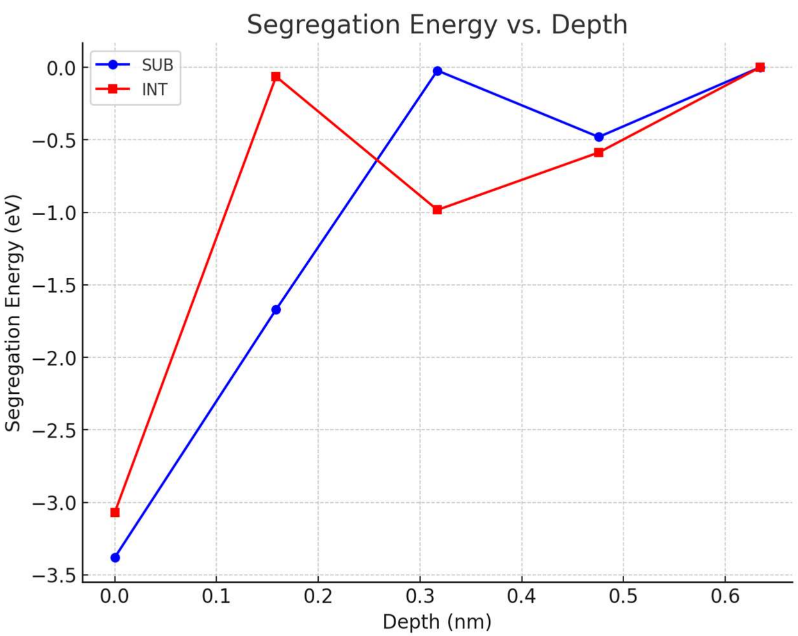
→ **Oxygen around 10 at.% only at high B/W:**
Presence of W might suppress oxygen incorporation.

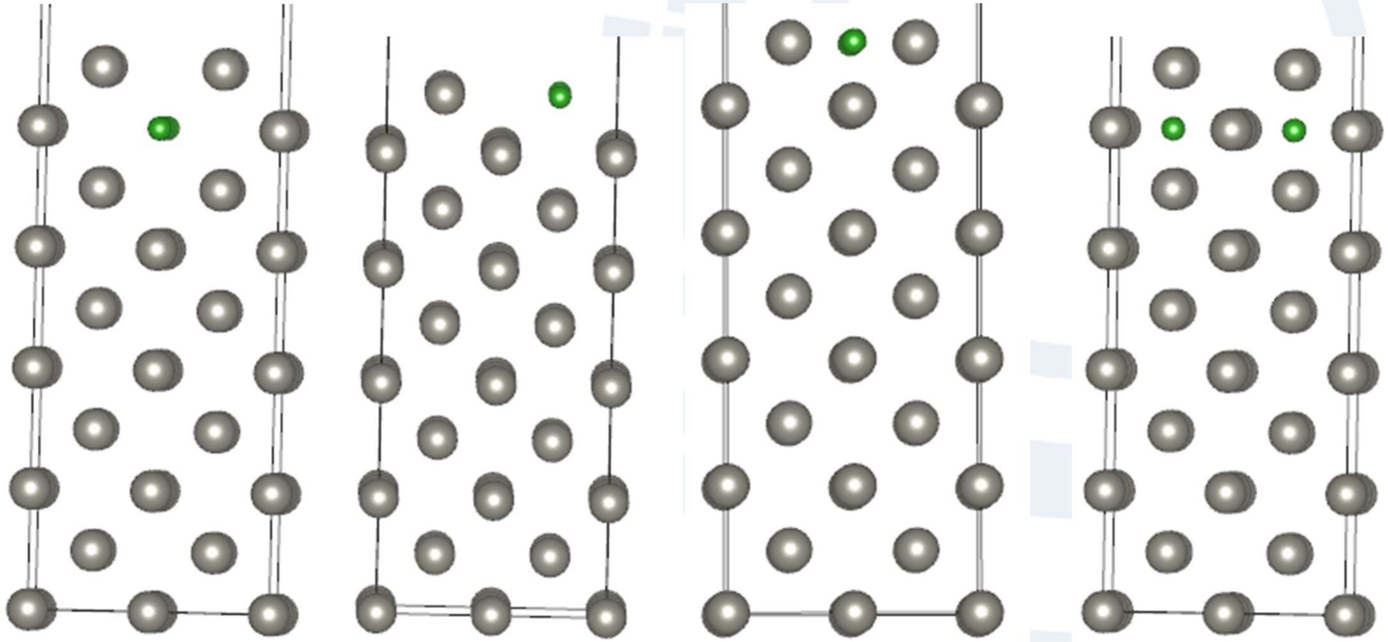


DFT calculations: B adsorption and diffusion on W surfaces

Main observations

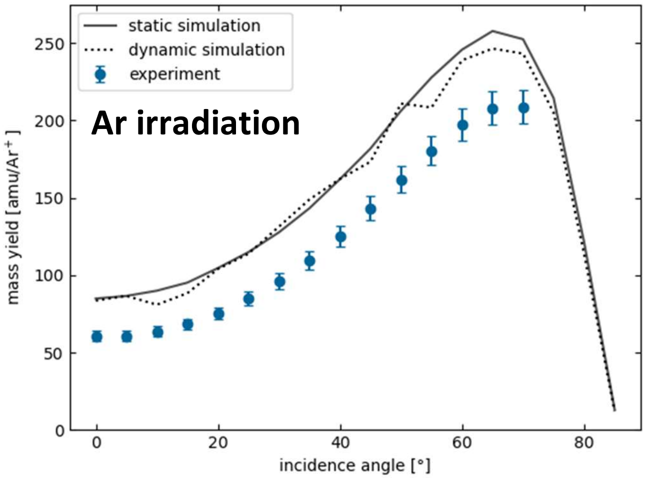
- **Investigation of B surface and bulk segregation in W different orientations:**
- Binding energies indicate a stronger adhesion between the coating (boron) and tungsten for (110) surface orientation than for (100, 111) orientations.
- Surface Segregation: Boron prefers tungsten surfaces, and it is more stable in substitutional position in W(100).
- Bulk Segregation: Positive energies show boron is less stable deeper in the bulk.
- 110 has much stronger adhesion between the coating (boron) and tungsten.

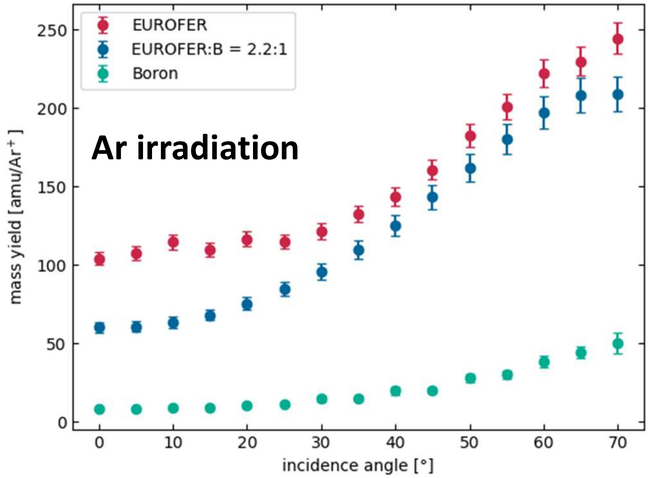


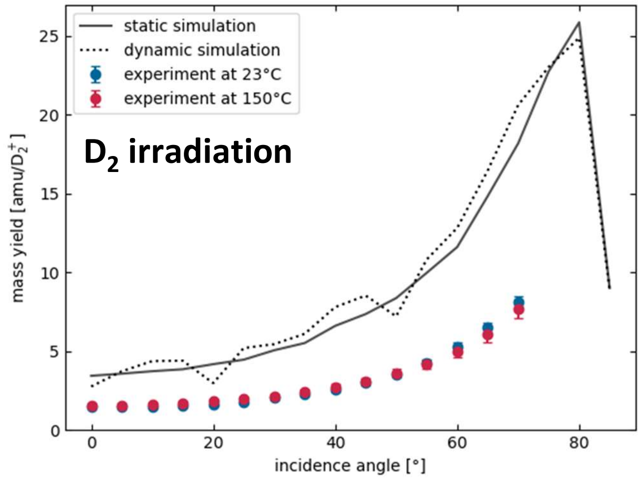


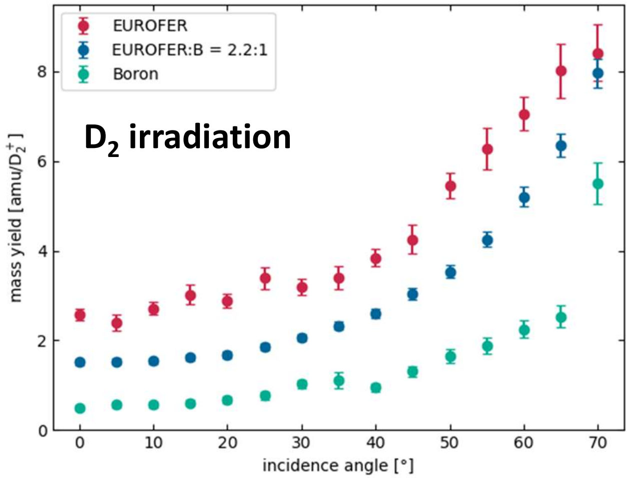
Measurement of sputtering yields of mixed layers with EUROFER97

EUROFER97:B=2.2:1 (25-Eu97-B-1.1)



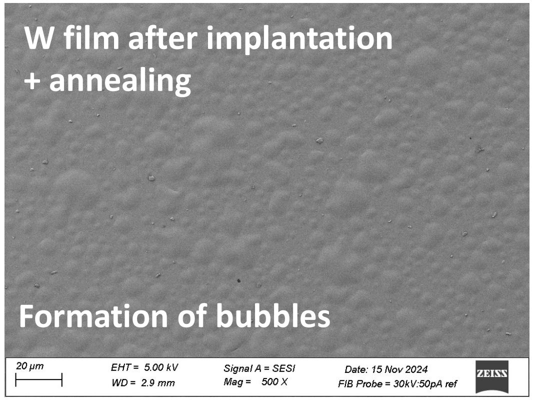


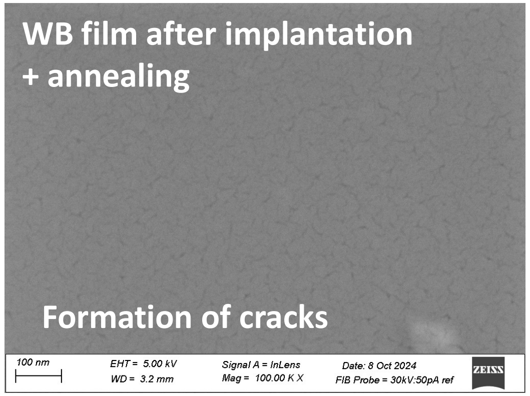


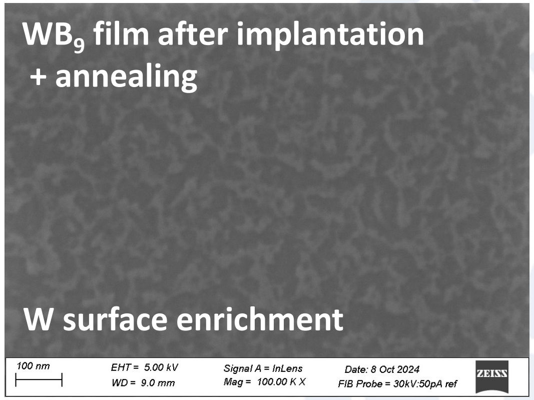


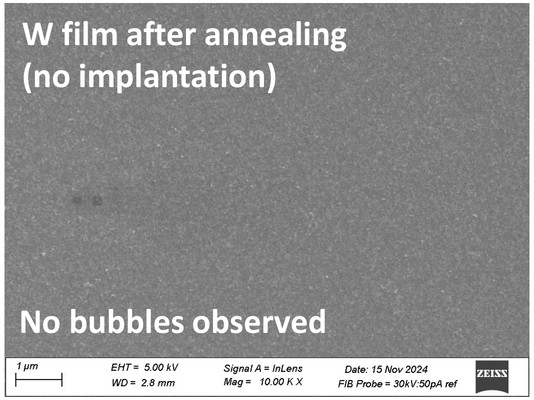
Ex-situ characterization after implantation/annealing

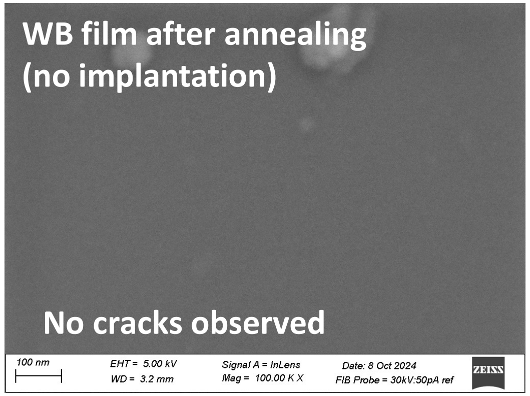
SEM:

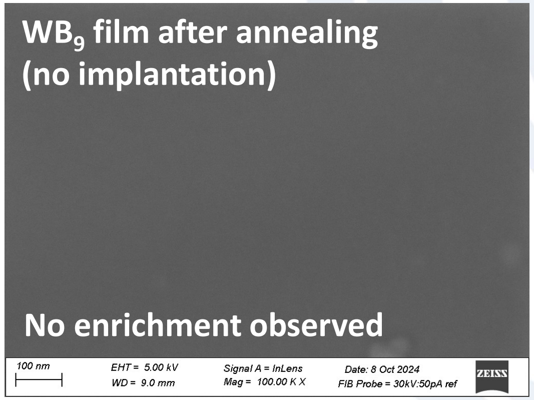




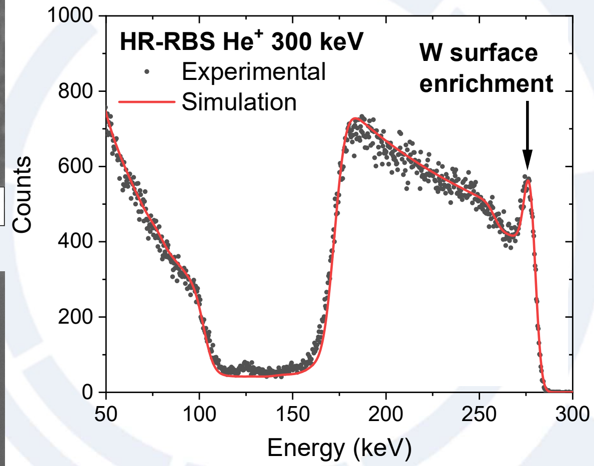






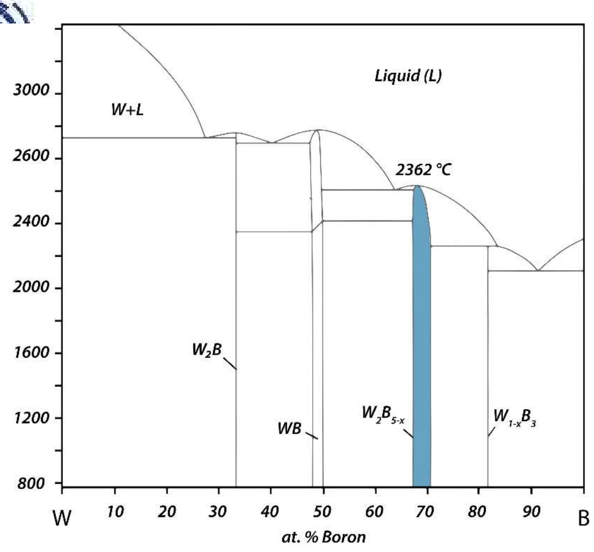


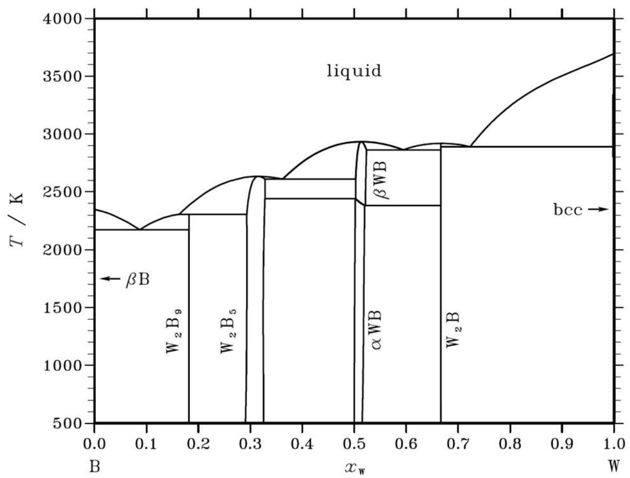
Confirmation using high-resolution RBS:



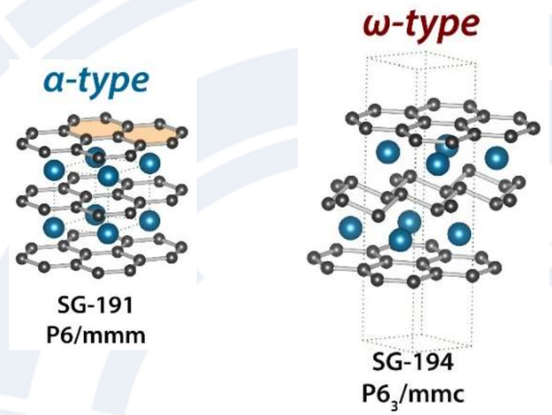
Overall observation:
Significantly different behavior after implantation for each composition.

Phase analysis, XRD





- Metastable phases formed during PVD ➔ equilibrium type phase diagrams not perfectly useable
- For high B contents competing phases for WB_2 structures



$WB_{2\pm z}$ *W_2B_5 or W_2B_4*

Defects are essential for SG-191 α-structured WB_{2-x}

M. Frotscher, et al., **M2B5 or M2B4? A Reinvestigation of the Mo/B and W/B System**, Z. Anorg. Allg. Chem. 633 (2007) 2626–2630.
J. Palisaitis, et al., **Where is the unpaired transition metal in substoichiometric diboride line compounds?**, Acta Materialia. 204 (2021) 116510.