

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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8th SB.ENR-MAT (Monitoring of 2024 activities), 3rd of February 2025



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UPPSALA
UNIVERSITET



Aalto University



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 and theoretical simulations of pure and mixed B&PFC layers in different compositions.**



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.



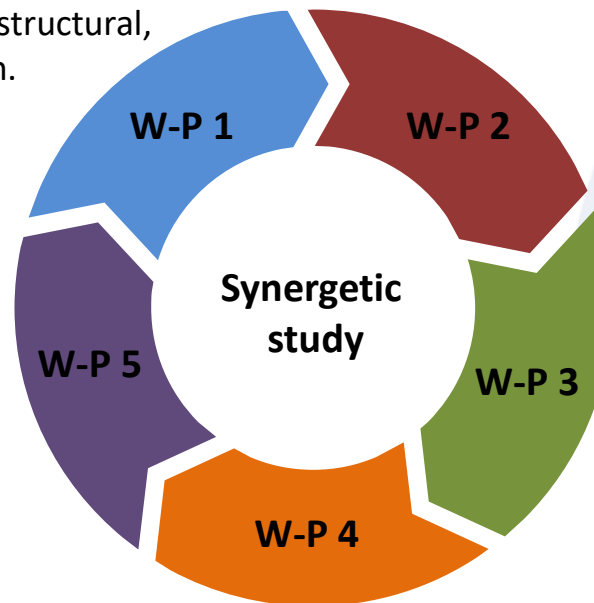
Working-packages

Sample preparation & characterization

Co-depositions in argon/D₂ atmospheres
Chemical, morphological, microstructural,
and mechanical characterization.
Distribution of samples.

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.



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.

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

Ab initio calculations (W₂B, W₂B₅ and WB₄):
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.



Team

VR	ÖAW	VTT (Aalto)
Eduardo Pitthan (PI-UU)	Martina Fellingner (IAP)	Andrea Sand
Daniel Gautam (UU)	Friedrich Aumayr (IAP)	Antoine Clement
Per Petersson (KTH)	Johannes Brötzner (IAP)	Nima Fakhrai
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).





Deliverables proposed 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 initio 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 Ab 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.



Status of Deliverables proposed for 2024

WP 1: Sample preparation & characterization:

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WP 2: Sputtering Yields and BCA simulations

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

WP 3: Ab initio 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 Ab initio calculations of B-W defect energies. (D3.3 - Adhesion between boron and tungsten started earlier)

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

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

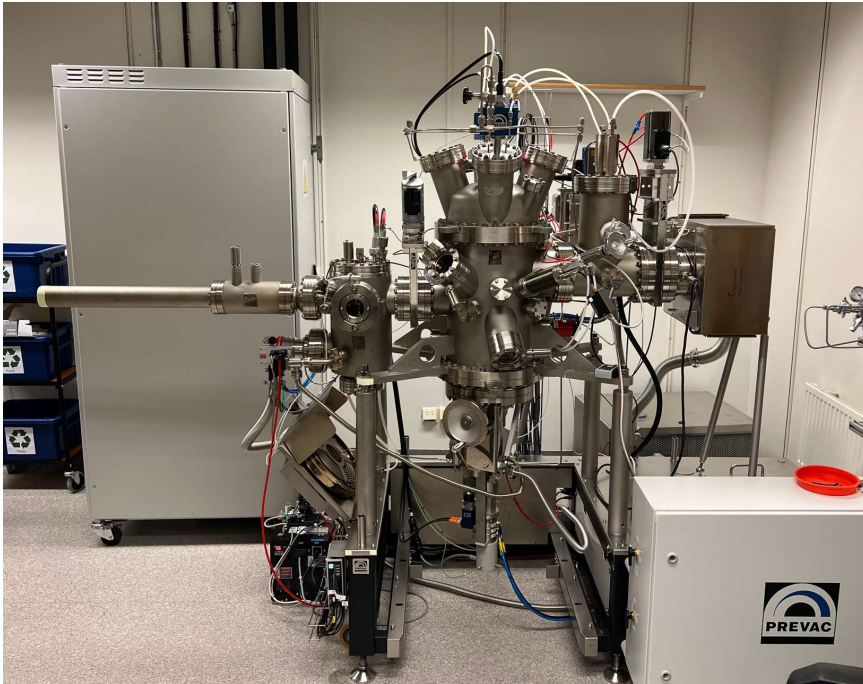
WP 5: Material Modification and Atomic Migration

No activities planned for 2024. → Activities started ahead of schedule.



Tandem Laboratory @ Uppsala University

Sputtering machine for film deposition



Prevac sputtering machine:

4 Magnetrons (2 DC and 2 RF).

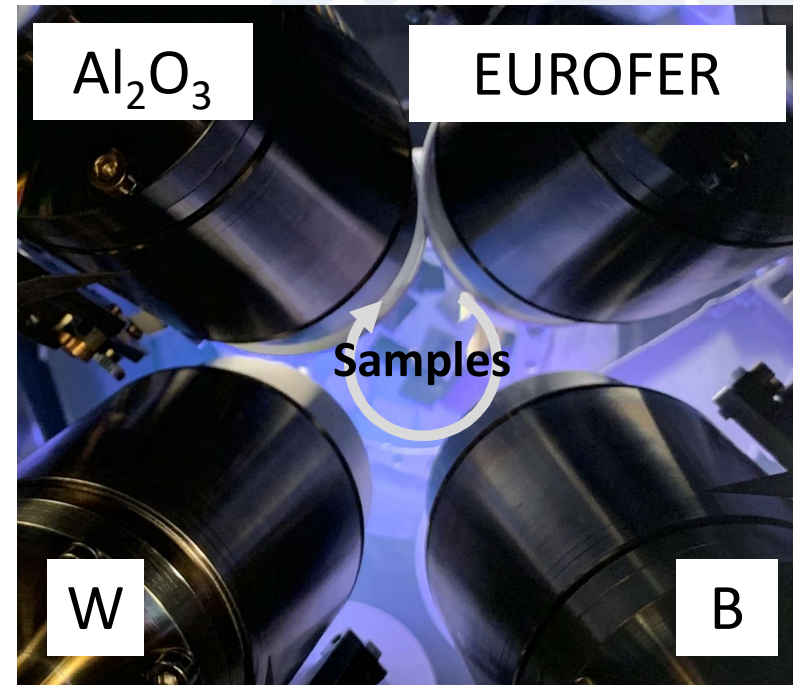
Deposition in Ar and Ar/D₂ mixed plasmas.

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:



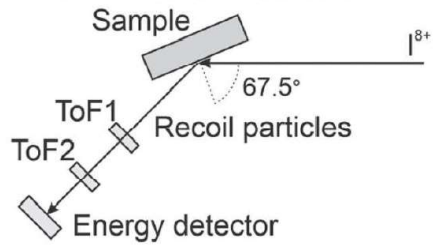
Simultaneous deposition of W and B under different conditions to obtain different ratios of W and B.



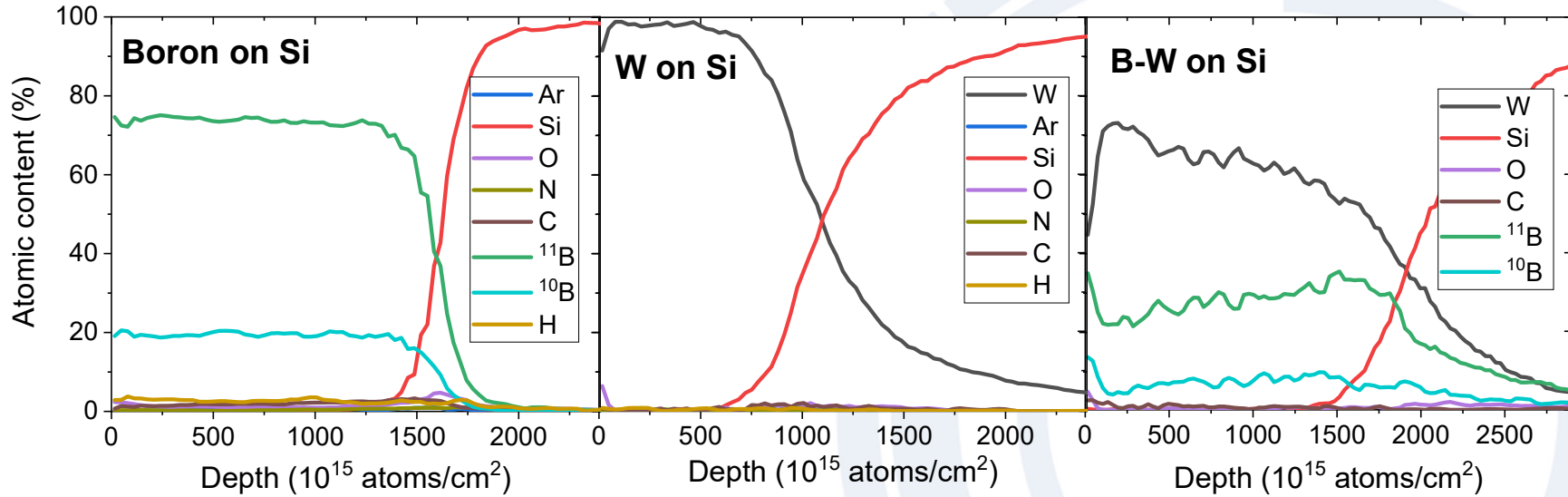
W-B film characterization

Sputtering depositions under argon atmospheres.

Atomic concentration depth profiles by ToF-ERDA:



Average film composition calculated from $150-500 \times 10^{15}$ atoms/cm²

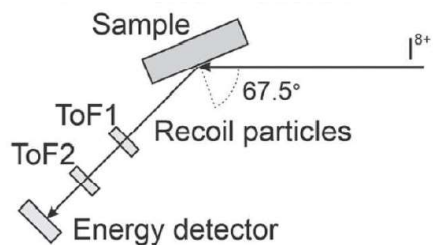




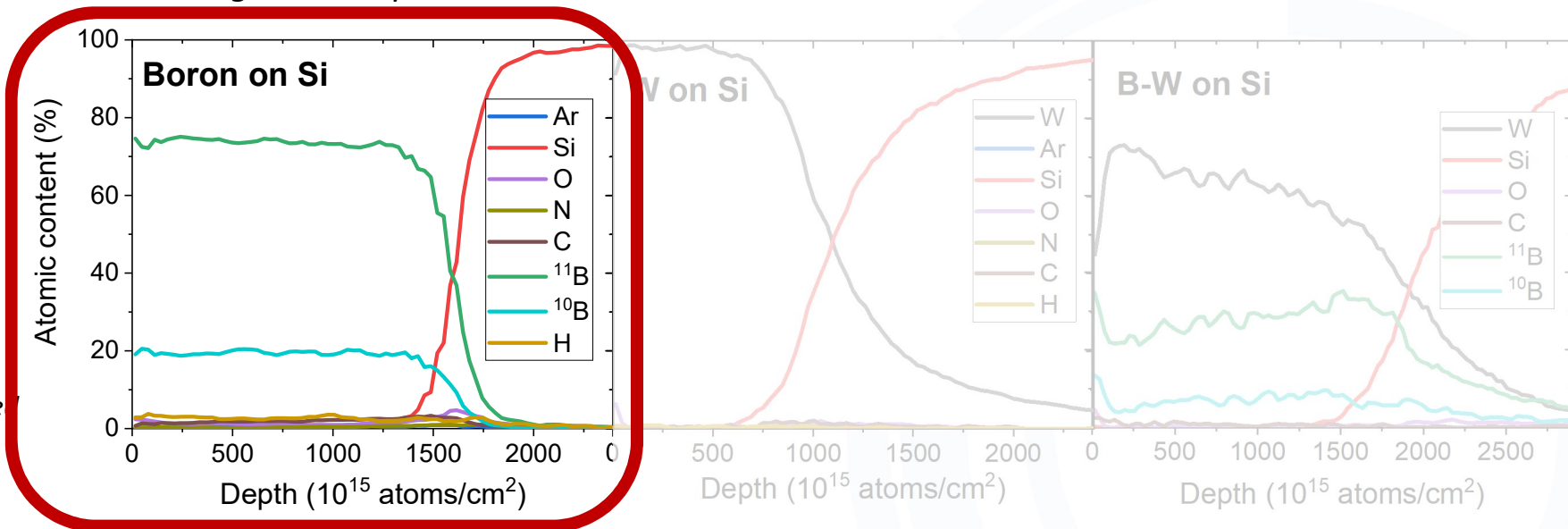
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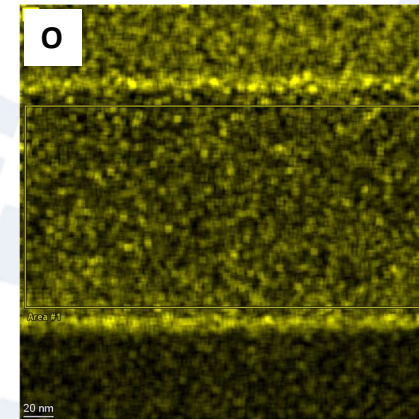
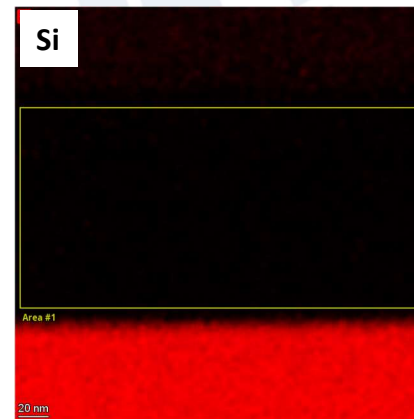
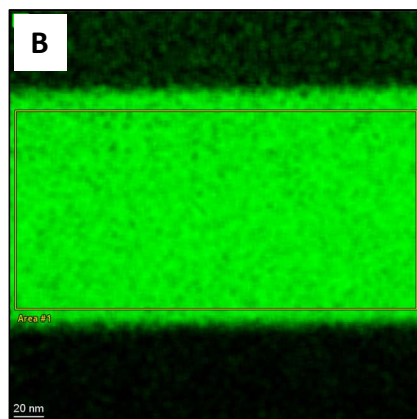
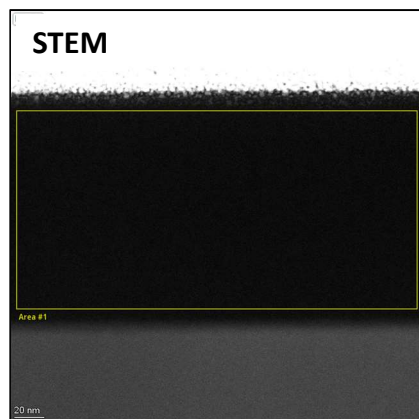


Average film composition calculated from $150-500 \times 10^{15}$ atoms/cm²



Boron on Si:

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

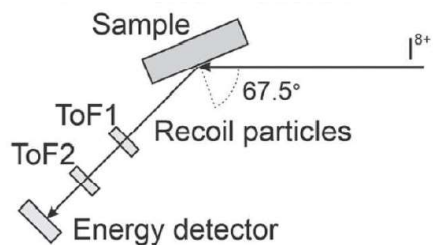




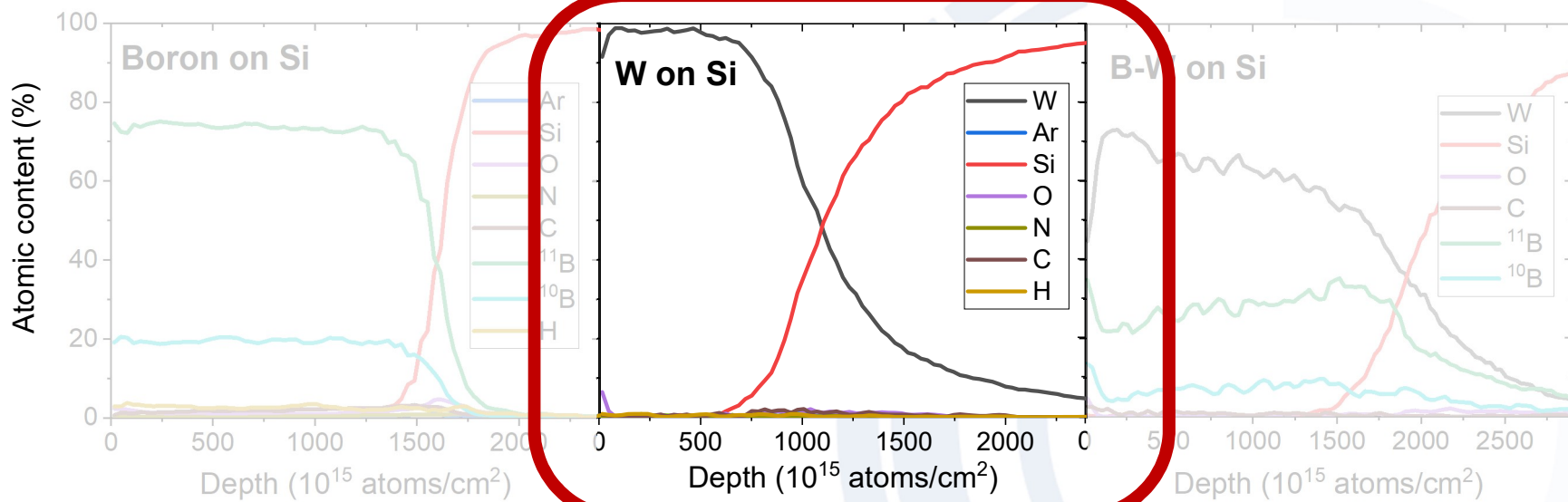
W-B film characterization

Sputtering depositions under argon atmospheres.

Atomic concentration depth profiles by ToF-ERDA:

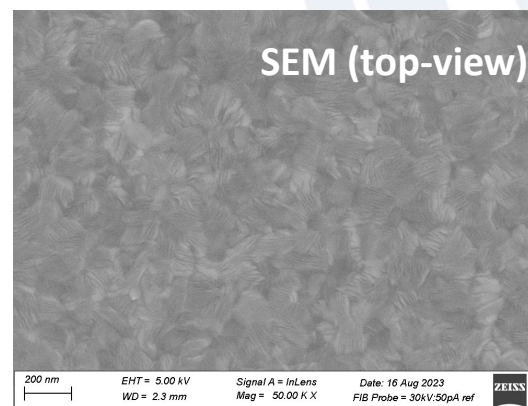
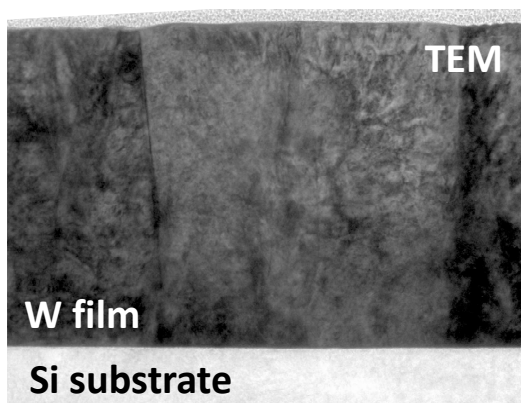


Average film composition calculated from $150\text{-}500 \times 10^{15}$ atoms/cm²



W on Si:

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

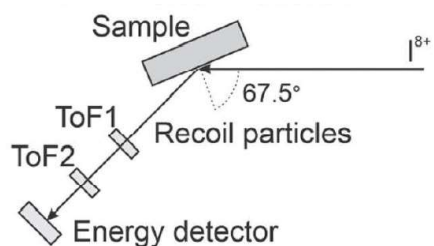




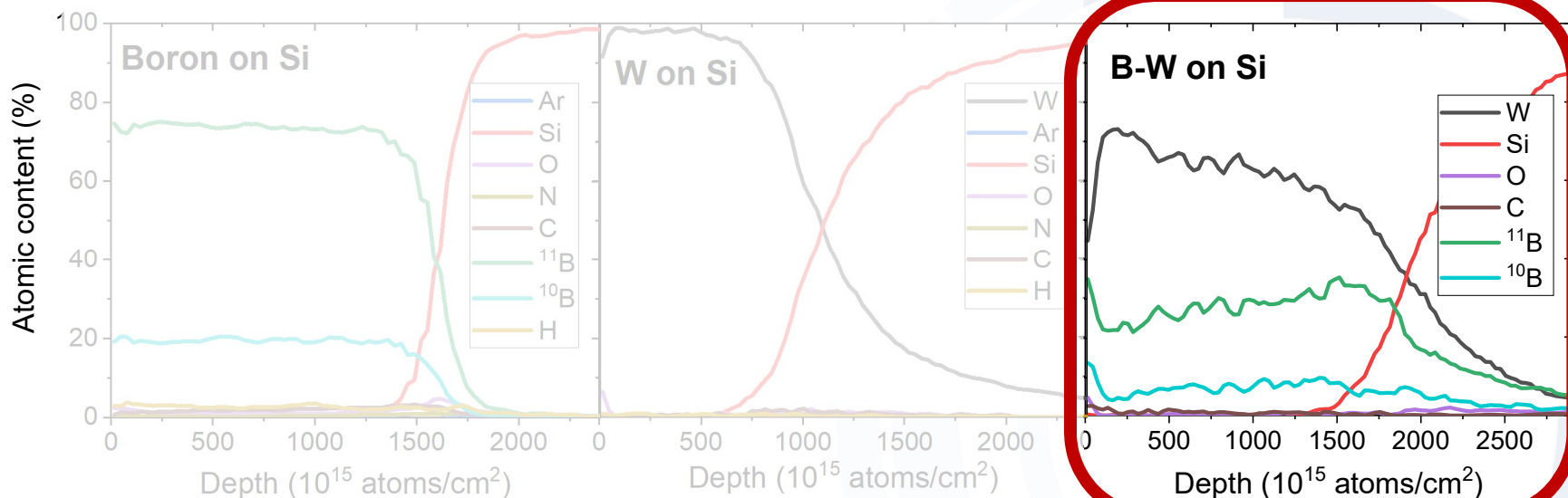
W-B film characterization

Sputtering depositions under argon atmospheres.

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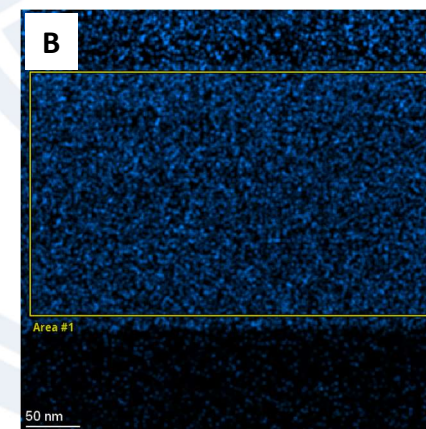
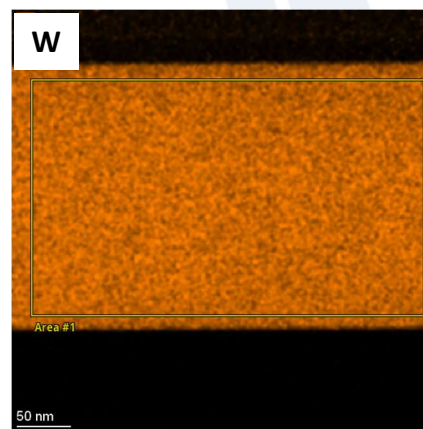
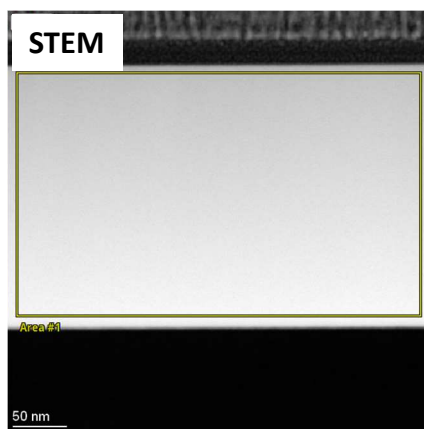


Average film composition calculated from $150\text{-}500 \times 10^{15}$ atoms/cm²



W-B on Si (example):

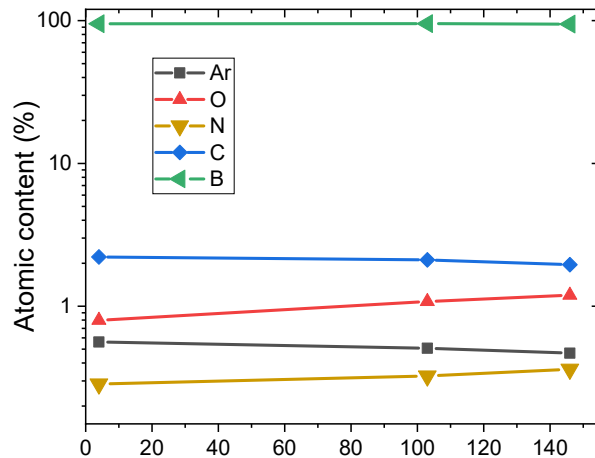
- W:B (2:1);
- Amorphous;
- Low contamination (<1%);
- EDX: no clear signal of B.
- Clear B on ToF-ERDA signal (multiple scattering effect from W).





W-B film characterization: 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 $150-500 \times 10^{15}$ atoms/cm²

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”.

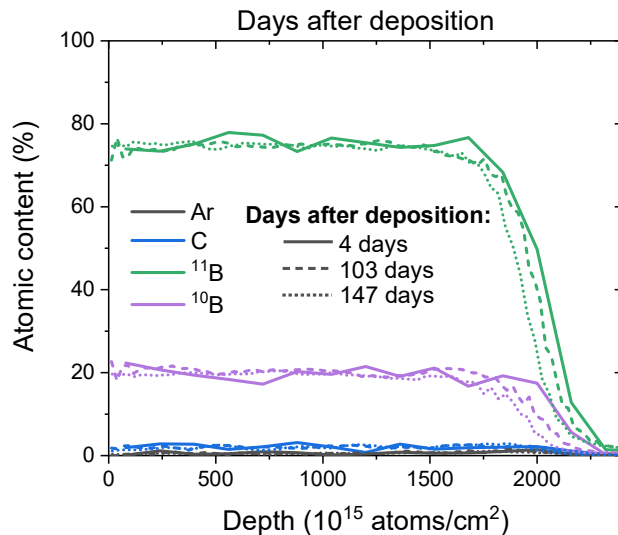
L.B. Bayu Aji et al. Appl. Surf. Sci. 448 (2018) 498.

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.



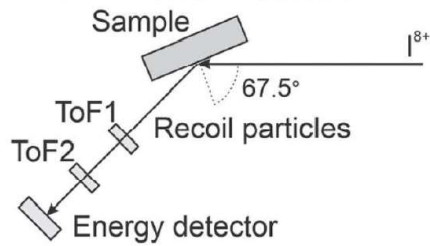


W-B film characterization

Sputtering depositions under argon and deuterium atmospheres:

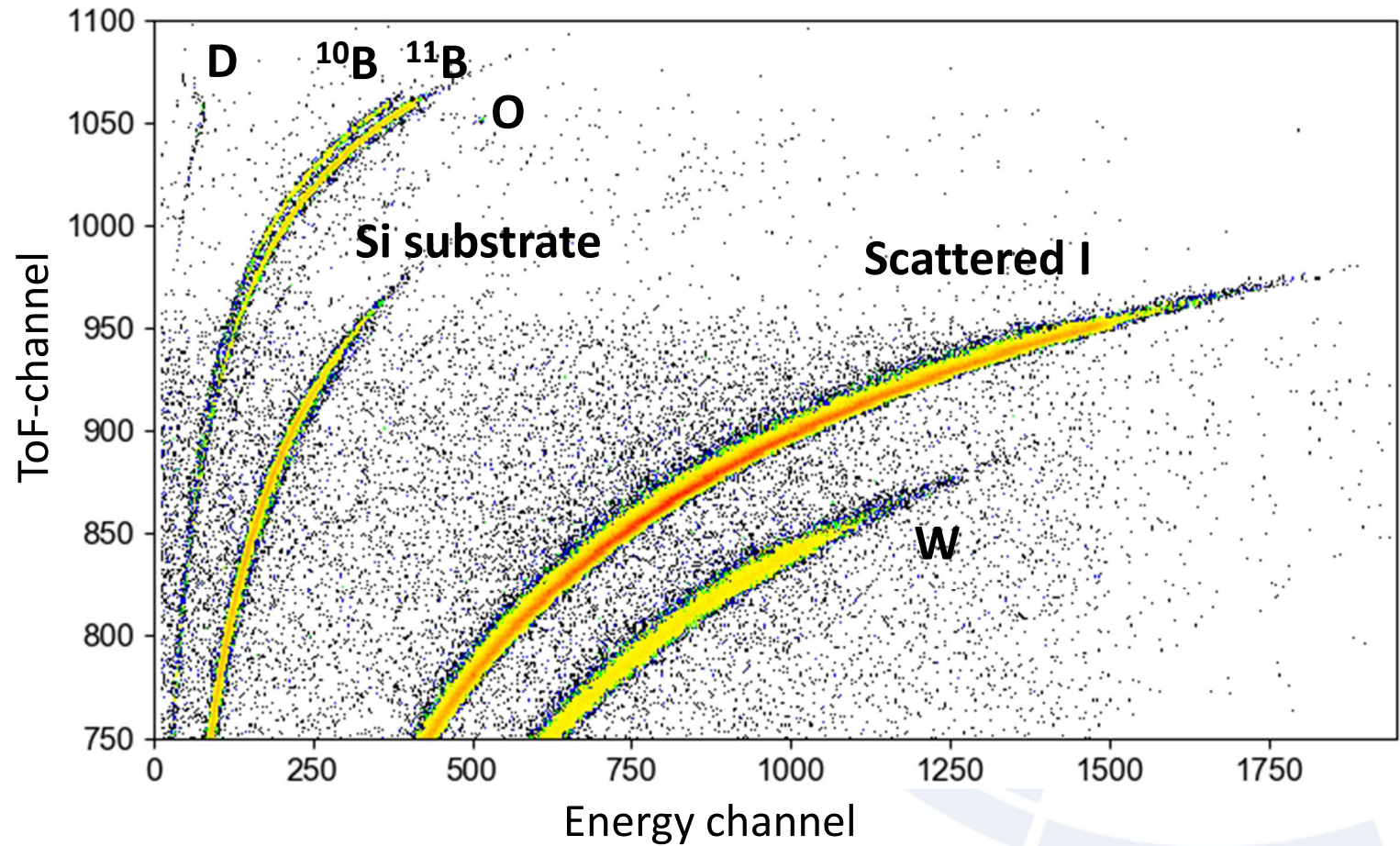
$$f_{Ar} = 10 \text{ sccm}; f_{D_2} = 18 \text{ sccm}; P_{Ar+D_2} = 7 \times 10^{-3} \text{ mbar.}$$

Atomic concentration
depth profiles by ToF-ERDA:



→ H and D detected.

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



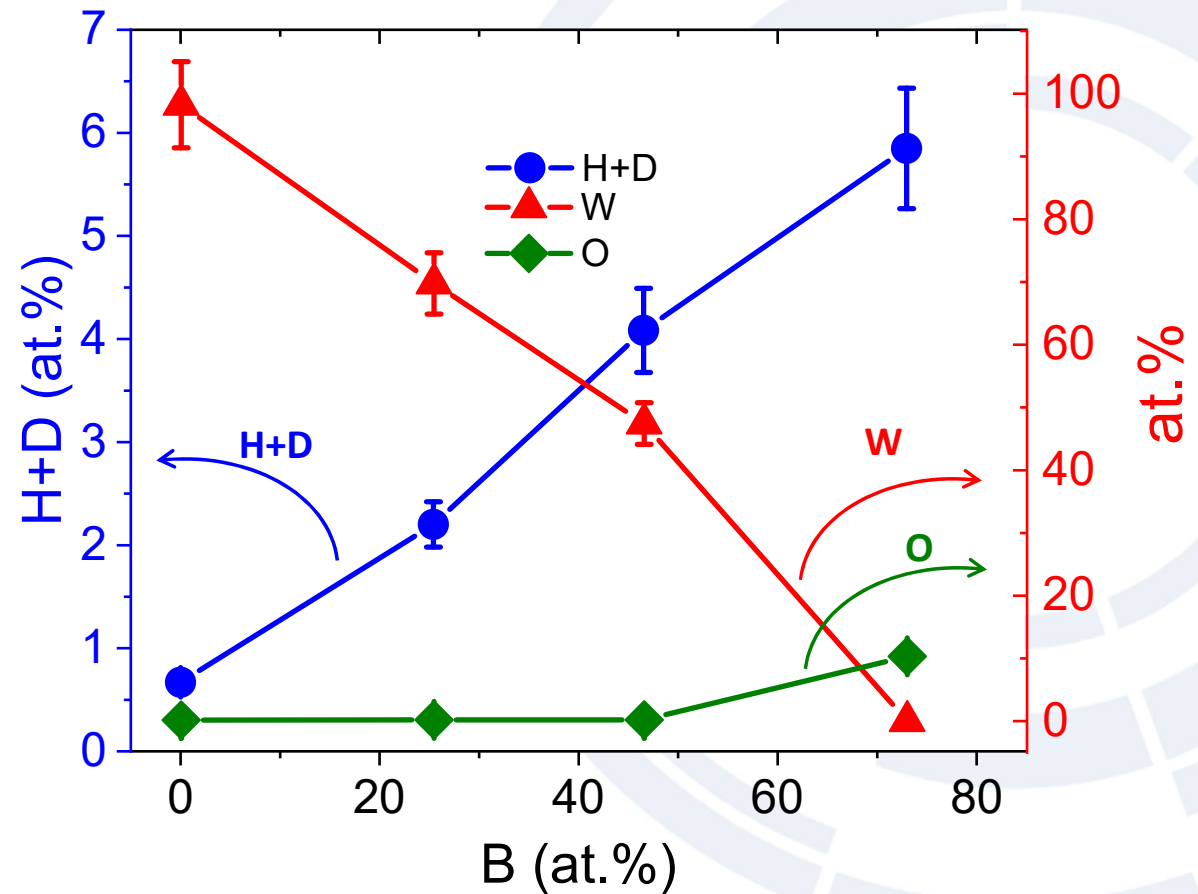


W-B film characterization

Sputtering depositions under argon and deuterium atmospheres:

$f_{Ar} = 10$ sccm; $f_{D_2} = 18$ sccm; $P_{Ar+D_2} = 7 \times 10^{-3}$ mbar.

- f_{Ar} and f_{D_2} 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.





Mechanical properties

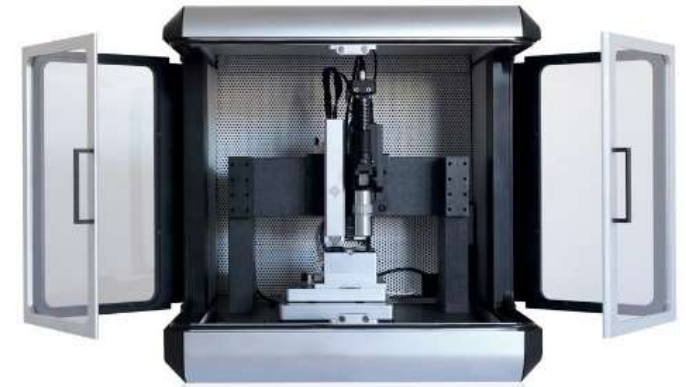
Nanoindentation

Measurement details:

- › Continuous stiffness measurement (CSM) method
- › Nanoindentation was done on each sample with **16 single points**
- › The average values of the obtained

Hardness and Youngs-Moduli were calculated using a Python Script.

- › Measured samples:
- › W-B mixed layers deposited in Ar+D₂ atmosphere on W substrates.
- › **Challenge: thickness of the samples (150-200 nm) still present effect of substrates in the measurements.**
- › *Preparation of thicker films in progress (UU-VR).*
- › *Preparation of thicker films using different approach for comparison (ASCT-ÖAW).*



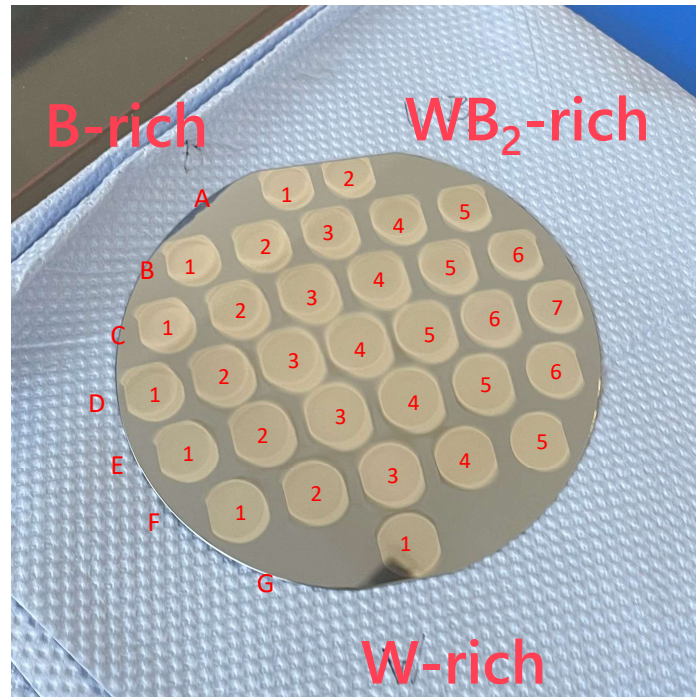
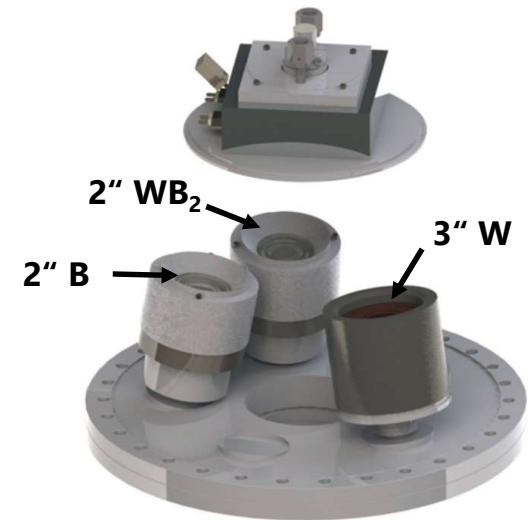


Mechanical properties

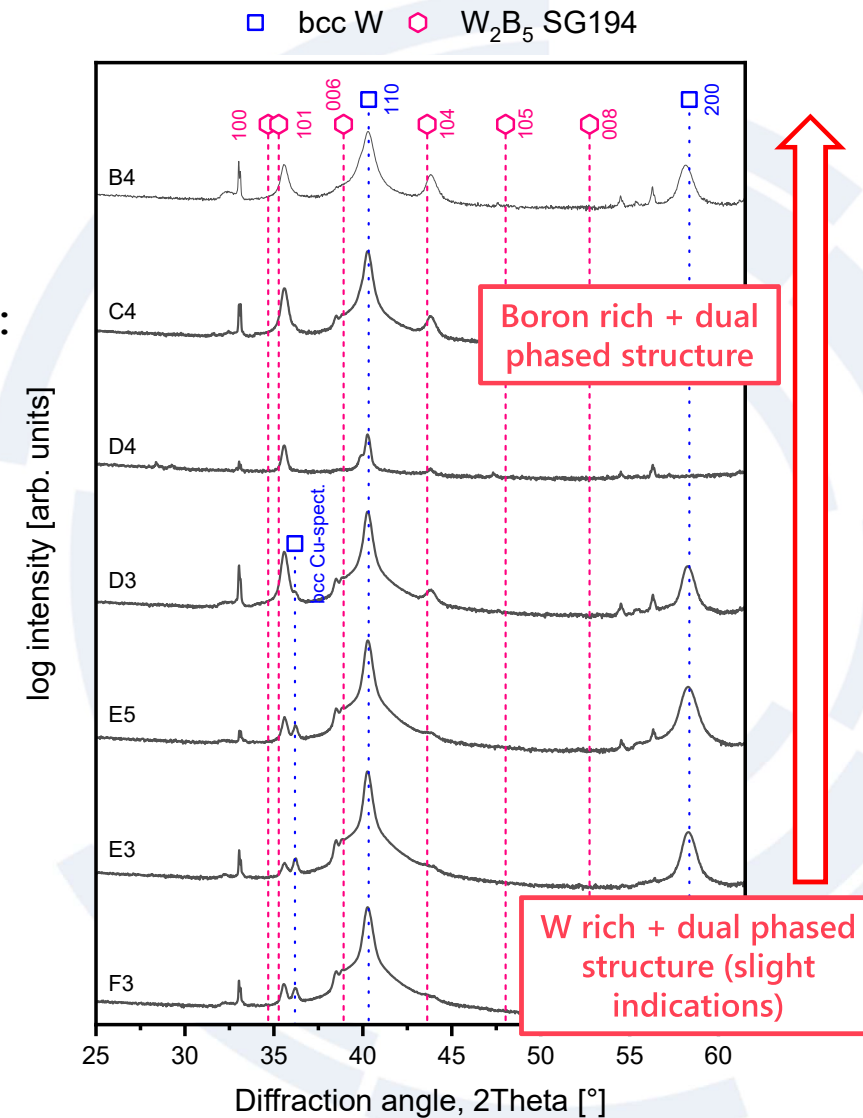
Nanoindentation

Various chemical composition in one deposition run specific sample holder + multi-cathode system required.

Allow comparison with films prepared by different machine/approach:



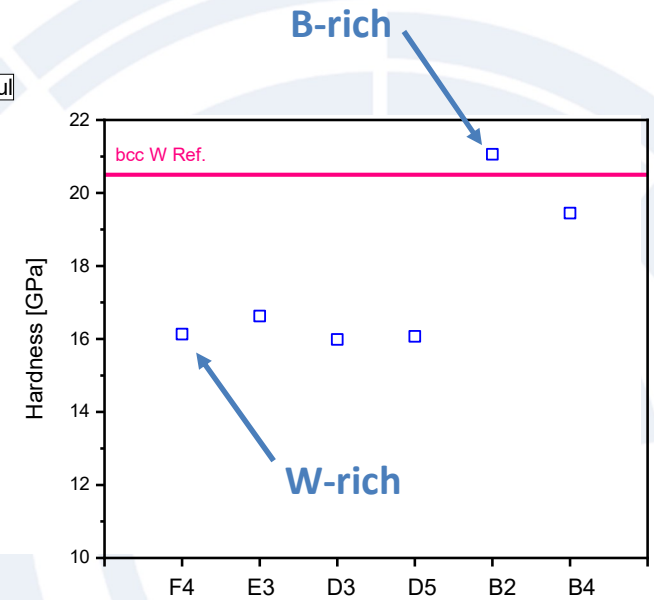
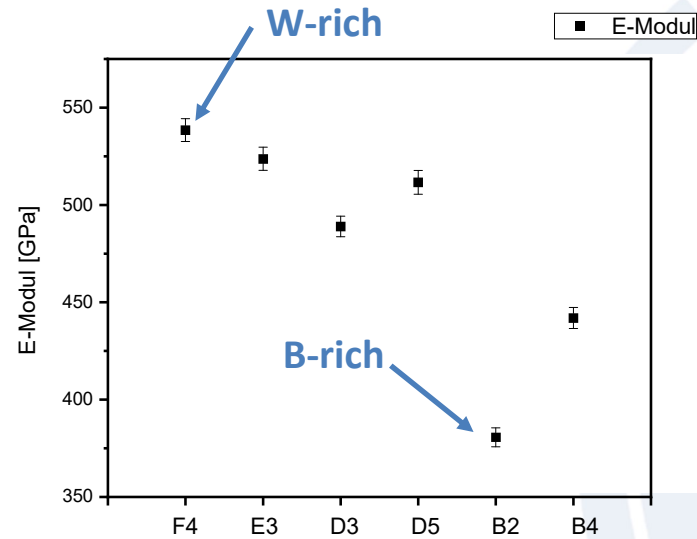
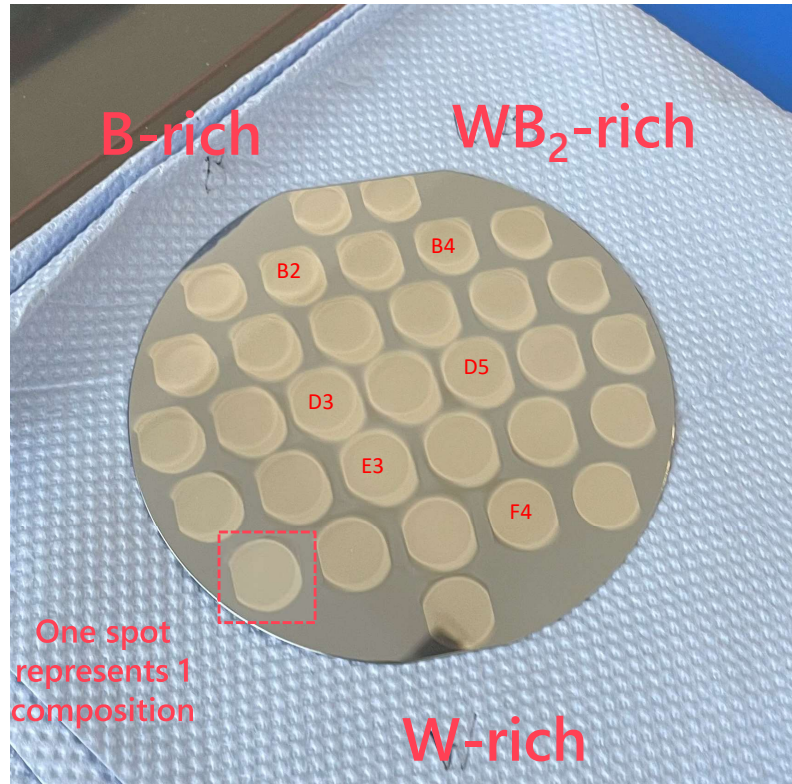
- › Substrate bias potential: -50 V
- › Argon flow rate: 20 sccm
- › Deposition pressure: ~0.4 Pa
- › Substrate temperature: 400 °C





Mechanical properties

Nanoindentation

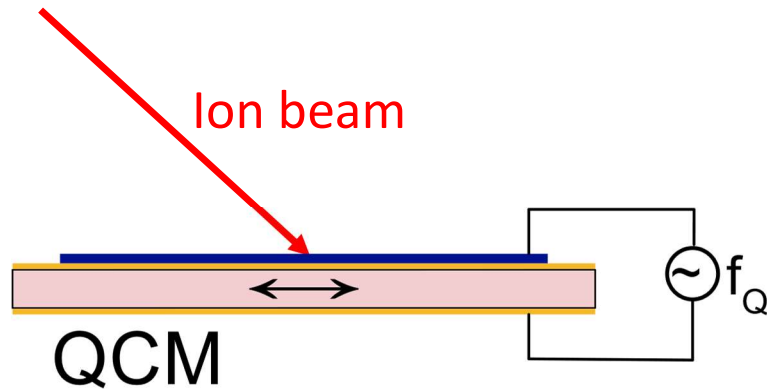


- Lower elastic modulus and higher hardness in B-rich layers.
- Composition of layers still will be verified.
- Results will be compared to reference layers (B and W bulks) and with amorphous (thicker) layers.



Sputtering yields

Experimental measurements



planned measurements for deliverable D2.1 (2024):

- 2 keV Ar⁺ on W and B mixed layers
- 2 keV D₂⁺ on W and B mixed layers
(2 keV D₂ as proxy for 1keV D)

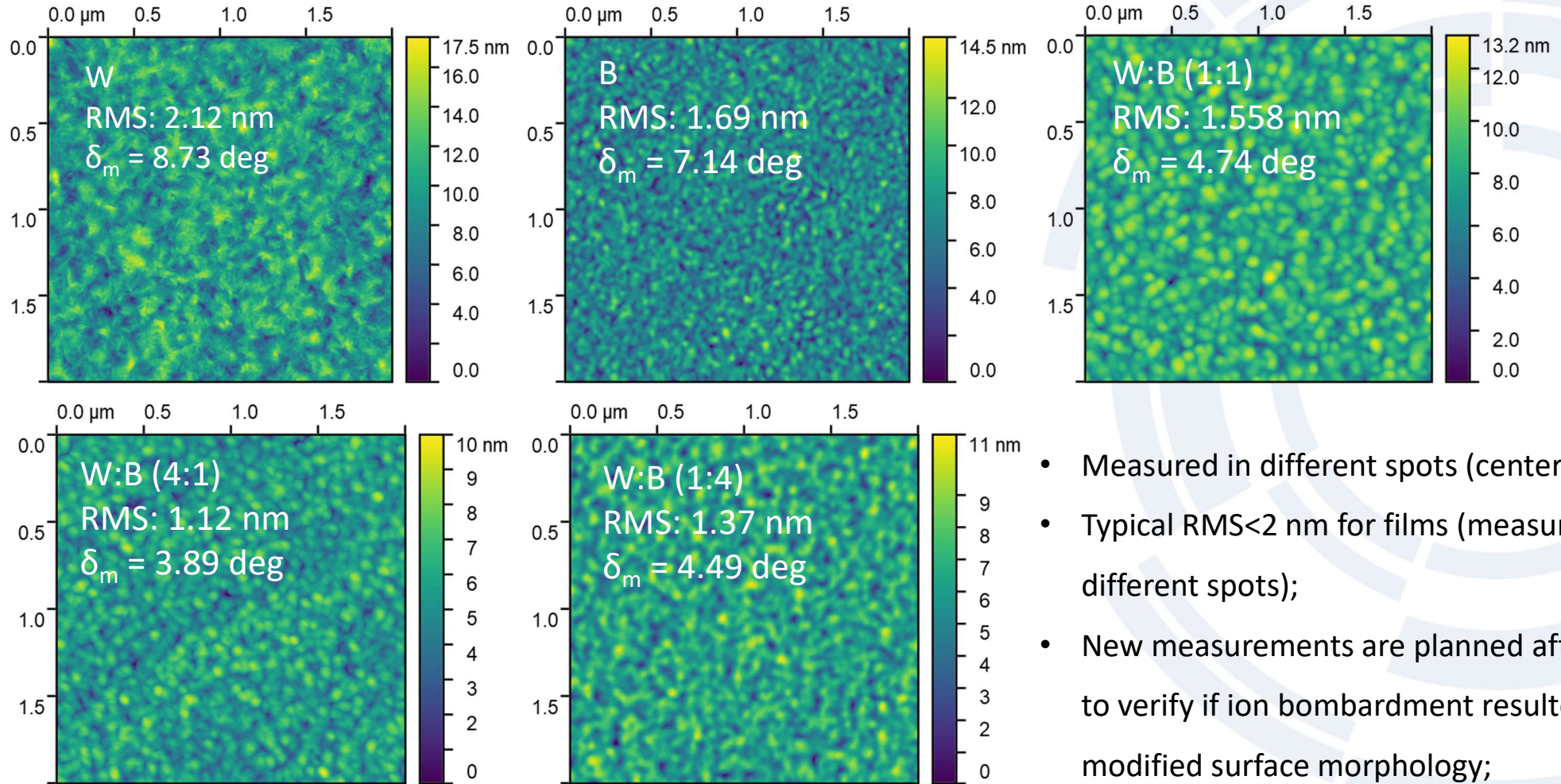
→ W, B and mixed layers need to be prepared for subsequent sputter yield measurements.

→ Initial evaluation of surface morphology to compare with post-irradiation surfaces.



Sputtering yields

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



- Measured in different spots (center and edge);
- Typical RMS < 2 nm for films (measured in different spots);
- New measurements are planned after SY analysis to verify if ion bombardment resulted and modified surface morphology;



Sputtering yields

Experimental measurements and BCA simulations

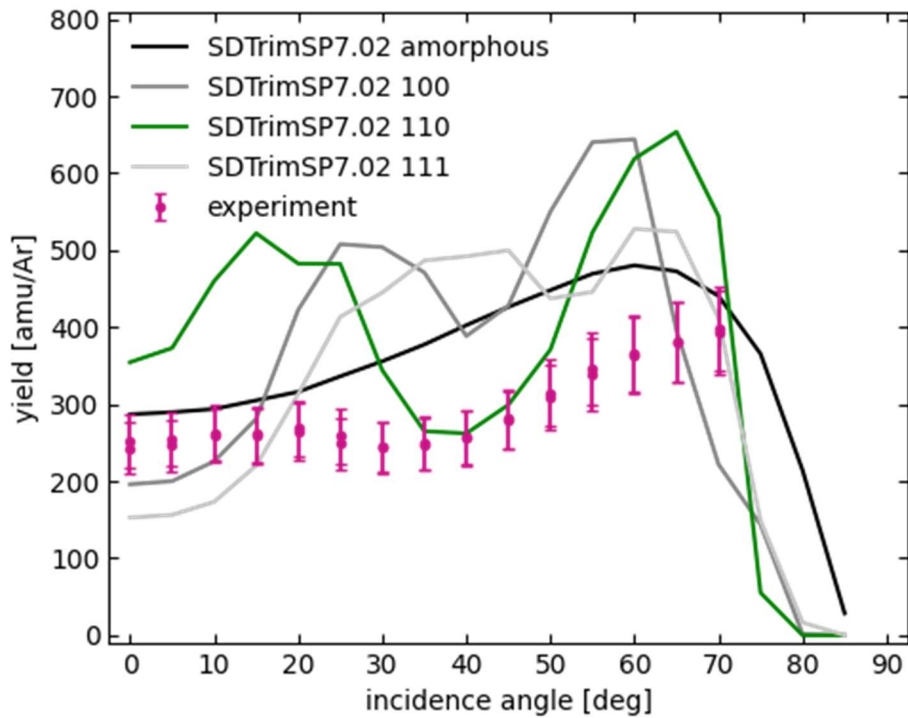
W films on QCM:

Local minimum present suggestion polycrystalline surface.

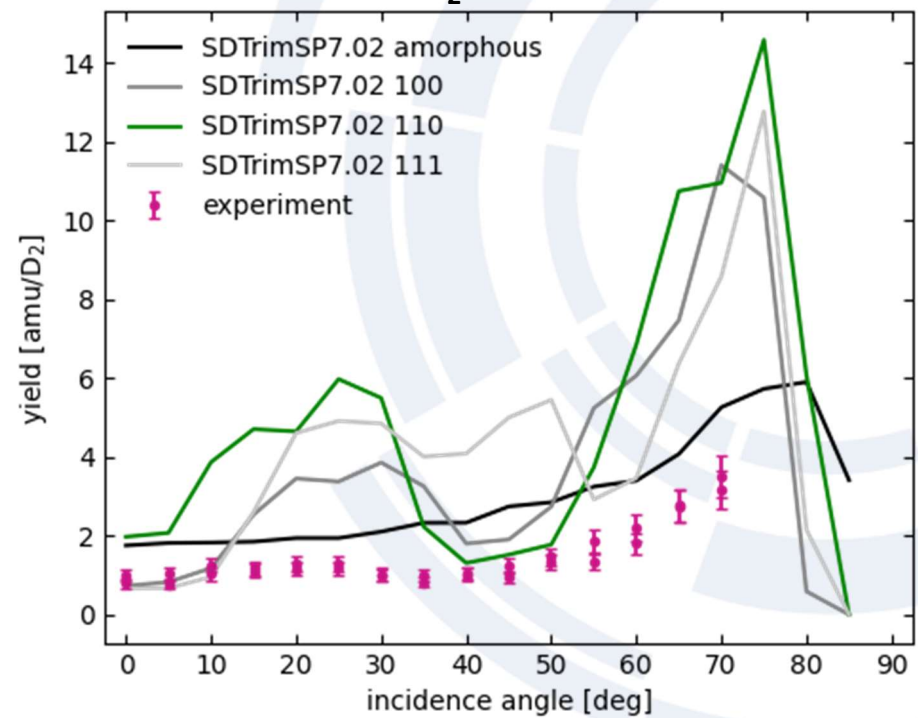
BCA simulations (SDTrimSP 7.02) supports crystal input structures: simulation of different crystallographic orientations.

Results will be used as baseline for comparison with W-B mixed layers.

2 keV Ar⁺ irradiation



2 keV D₂⁺ irradiation





Sputtering yields

Experimental measurements and BCA simulations

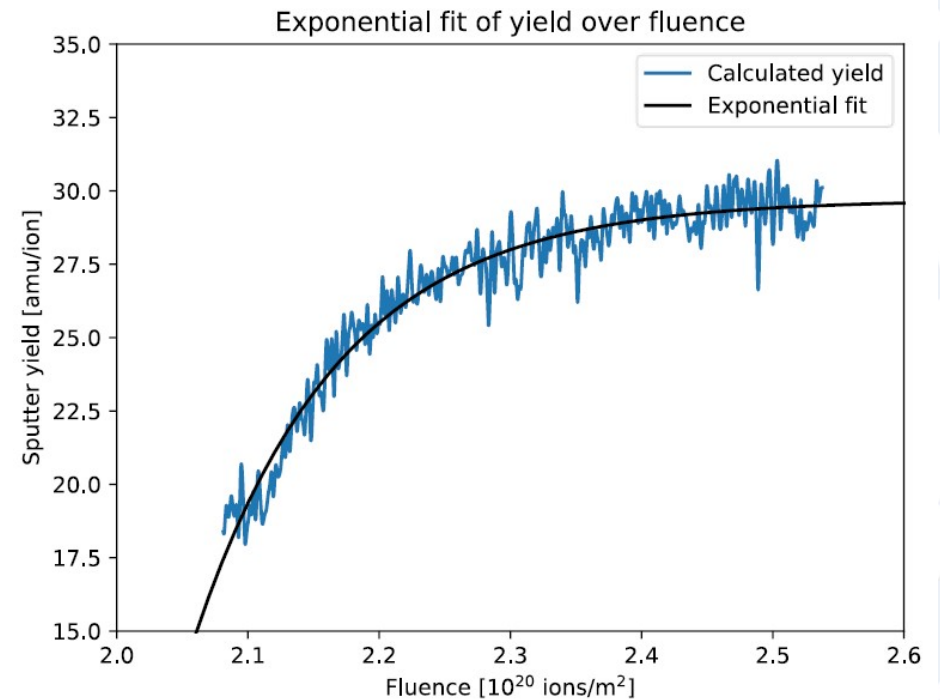
B films on QCM:

During sputtering measurements of Ar on B:

- Yield measurements on pure B turned out to be largely more time consuming than expected due to slow outgassing/implantation saturation.
- Implantation/outgassing saturates exponentially: steady state yield can be obtained by calculated exponential fits to the angle resolved data points.

During sputtering measurements of D_2 on B:

- Performed irradiation experiments at elevated temperature (150°C – there is a minimum in the $f(T)$ curve at this temperature, minimizing the temperature influence on the frequency, which is why we chose this specific value).





Sputtering yields

Experimental measurements and BCA simulations

B films on QCM:

post-processed data is reproducible.

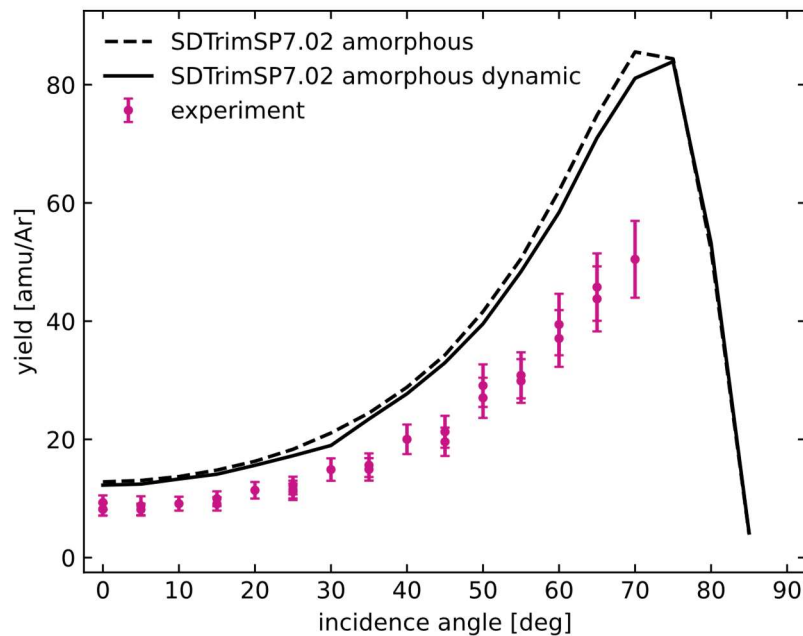
No local minimum (amorphous layer), also observed in SDTrimSP simulations.

Quantitative values of simulation and experiment do not agree (both static and dynamic mode)

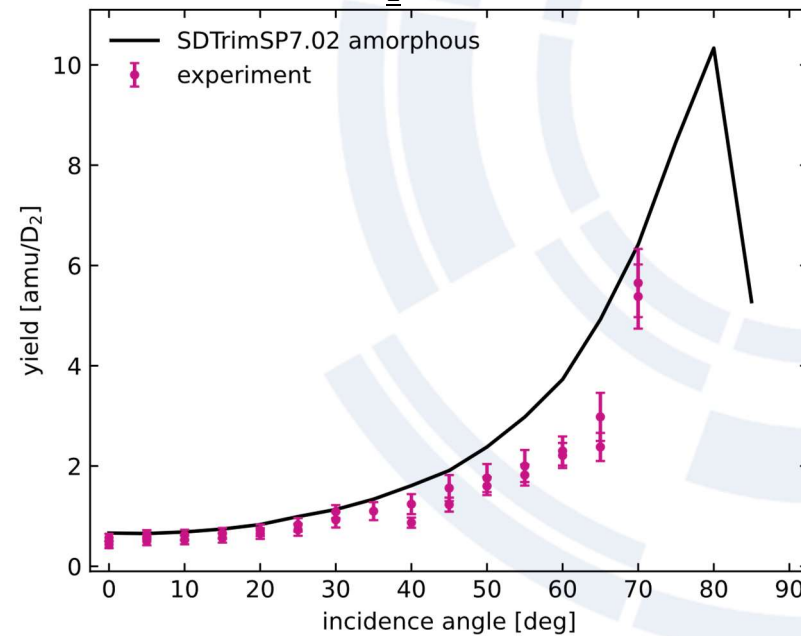
Effect of implantation is largely enhanced during D irradiation → (WP5: deuterium retention studies).

Measurements for W-B mixed layers will be completed soon.

2 keV Ar⁺ irradiation:



2 keV D₂⁺ irradiation:



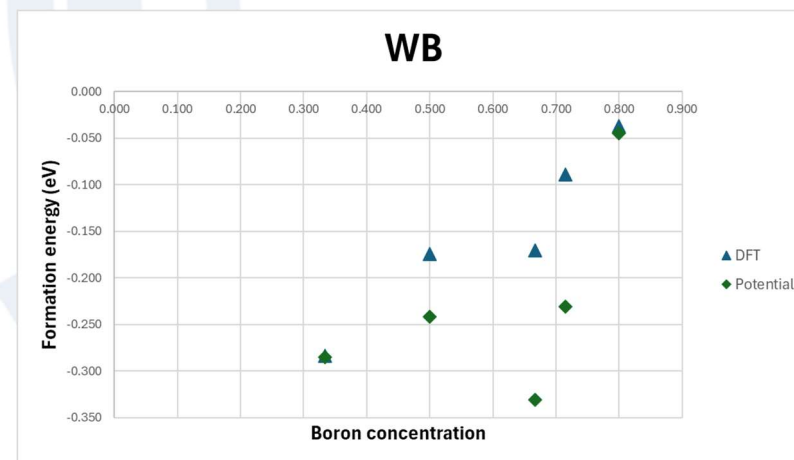
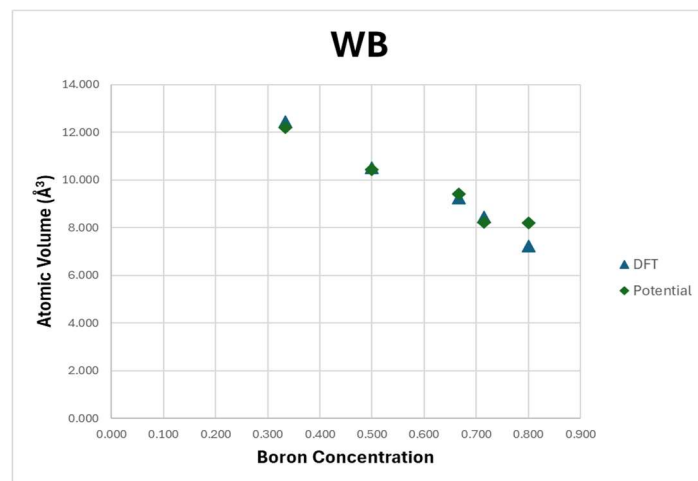
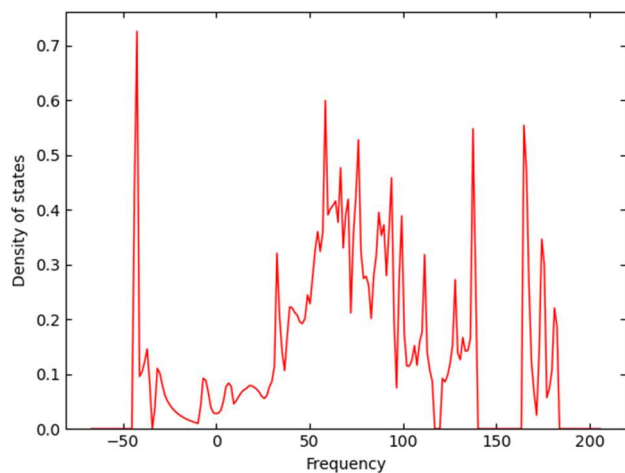


Ab initio calculations of defect energetics and mixed B-W materials

Development of new interatomic potential

- **Develop new semi-empirical interatomic potential for B-W materials, suitable for efficient large scale molecular dynamics (MD) simulations:**
- First version of new semi-empirical interatomic potential, suitable for efficient large scale molecular dynamics (MD) simulations, is complete for pure boron. Proven to be challenging for W-B systems.
- Thermal stability for some stoichiometries and defect properties still to be optimized.
- Will be used for MD predictions of sputtering yields from W-containing surfaces with evolving composition and morphology and comparison to experiment.

Prediction of new potential (boron)

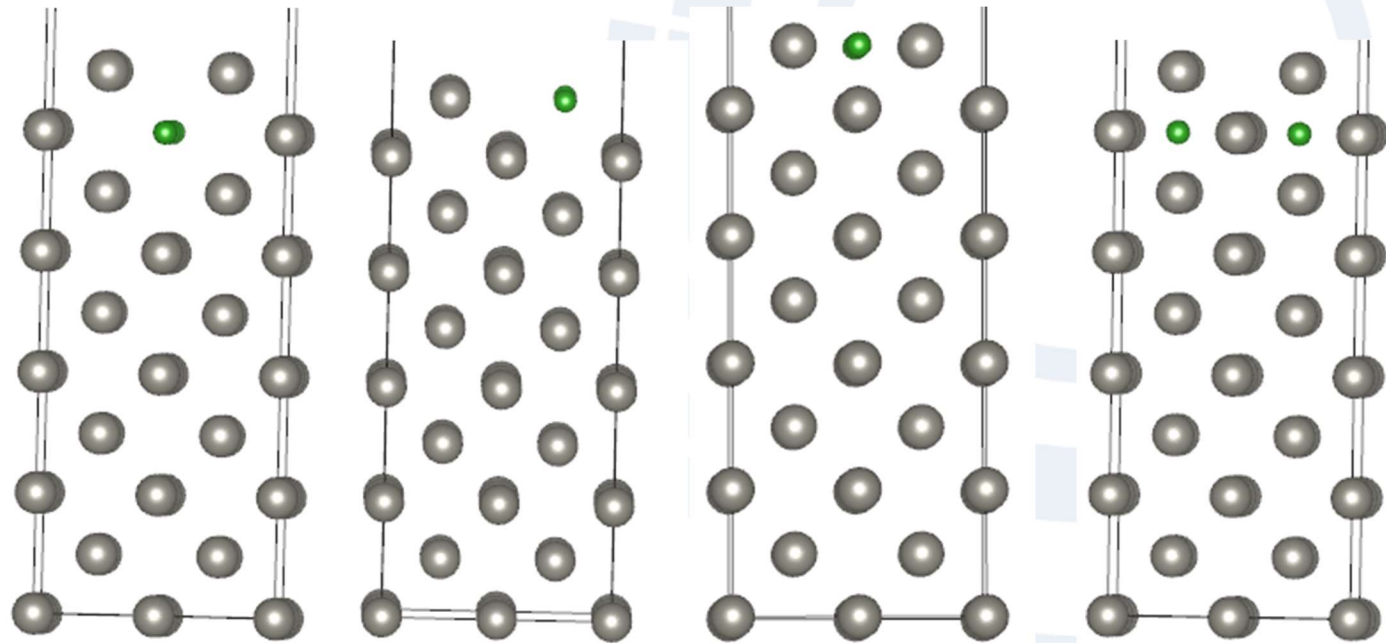
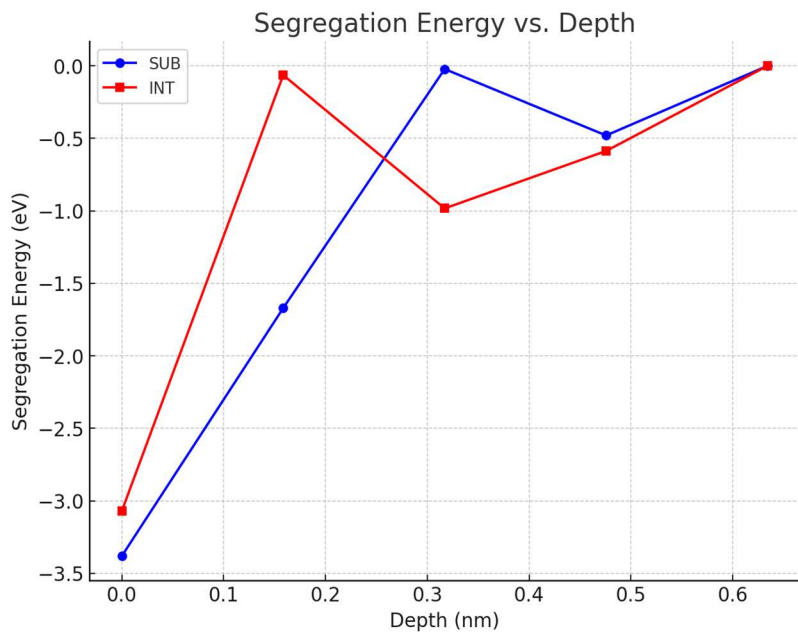




DFT calculations: B adsorption and diffusion on W surfaces

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.

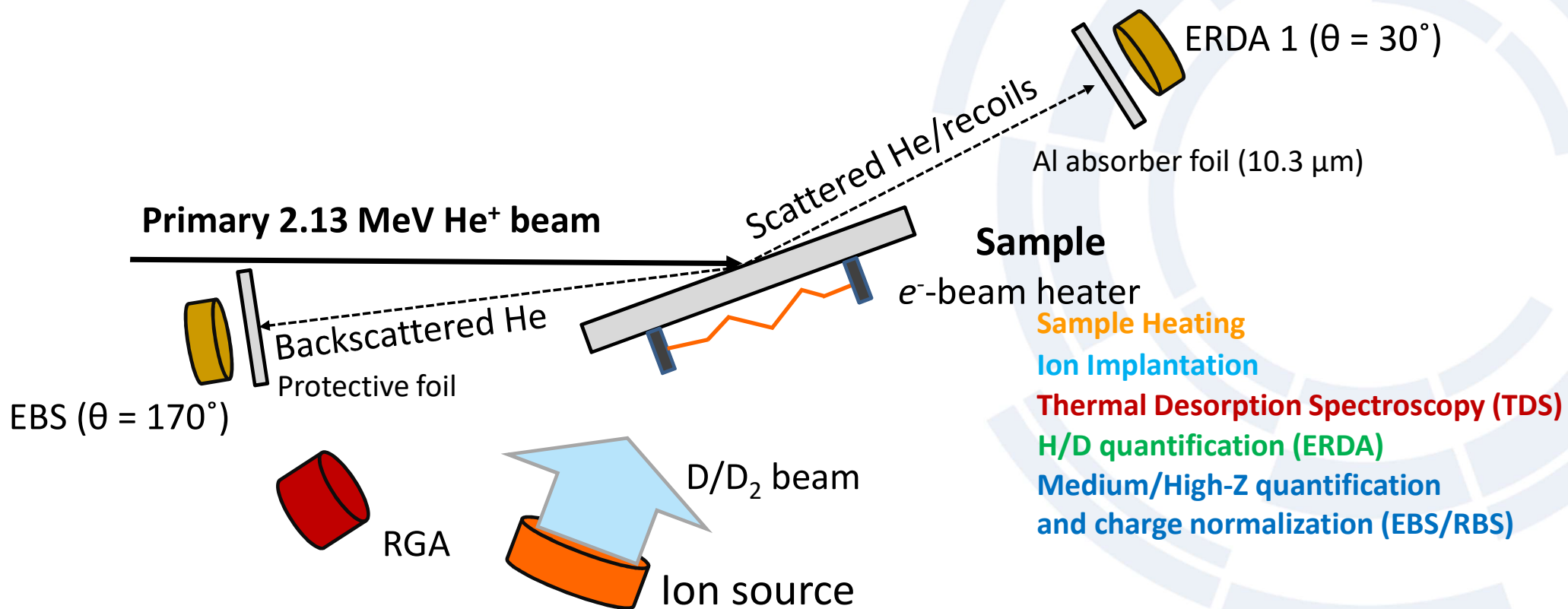




In-situ experiments of hydrogen and deuterium desorption

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

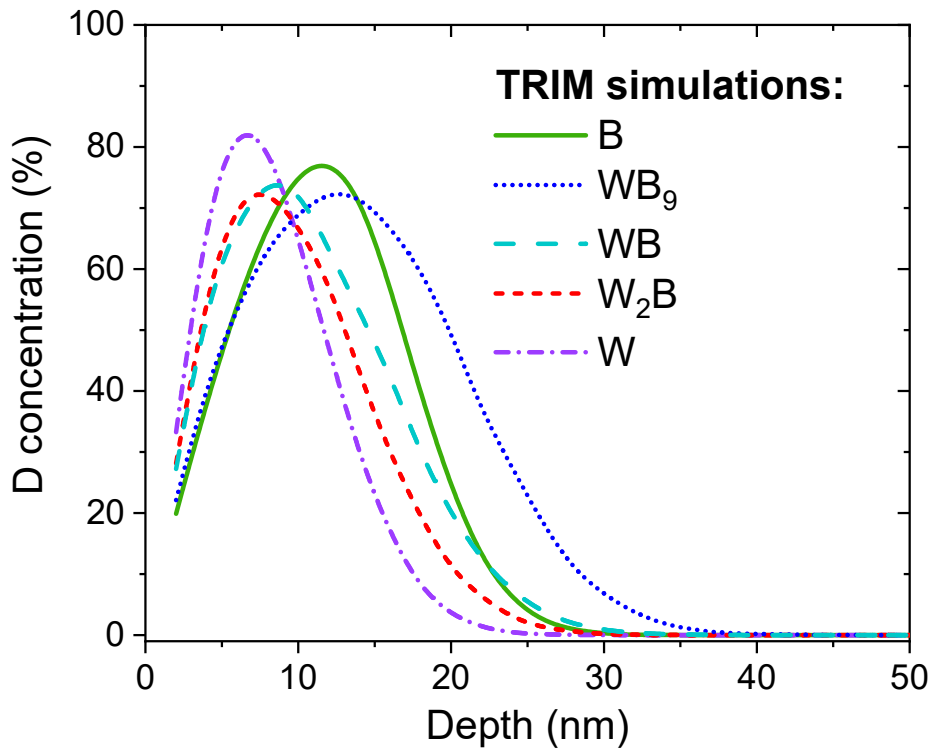
K. Kantre et al. Nuclear Inst. and Methods in Physics Research B 463 (2020) 96–100



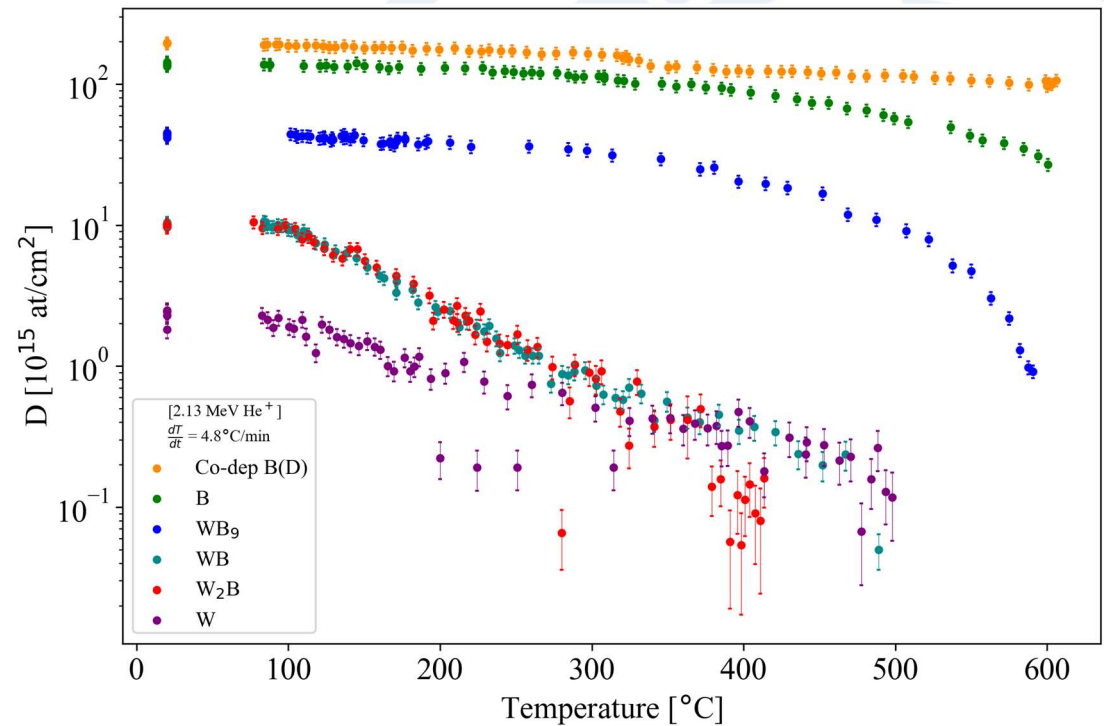


In-situ experiments of hydrogen and deuterium desorption

Deuterium implantations in W-B layers on Si:
1 keV D_2^+ implantation fluence $\approx 6 \times 10^{17}$ at/cm².
Implantation range within 40 nm.



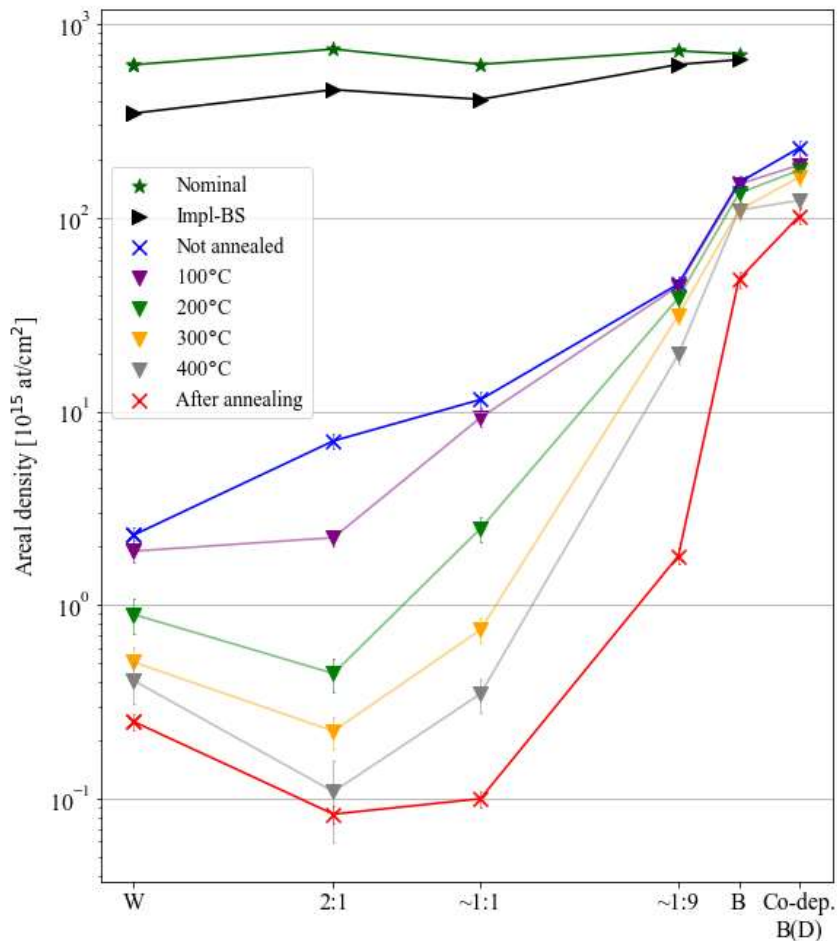
In-situ measurements after implantation:
Monitoring D content during annealing.
Lower deuterium content than nominal values (beyond saturation).





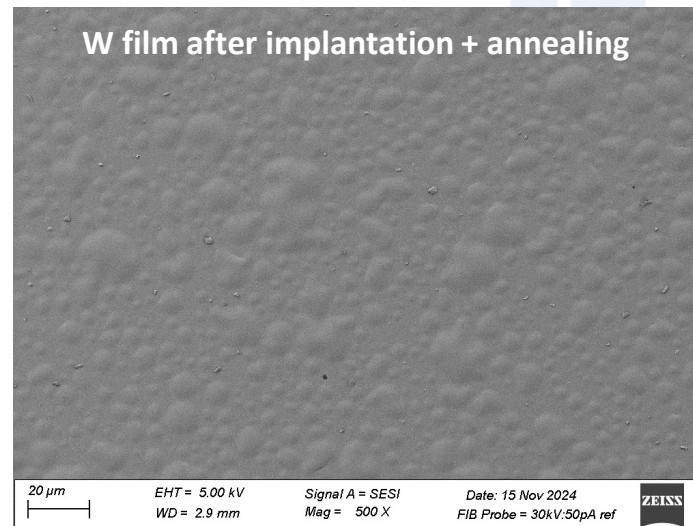
In-situ experiments of hydrogen and deuterium desorption

Simulations (TRIM) and experimental results:



- 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 D₂ atmosphere (different incorporation than implanted).

Ex-situ investigation after D₂ implantation + annealing (highlights):

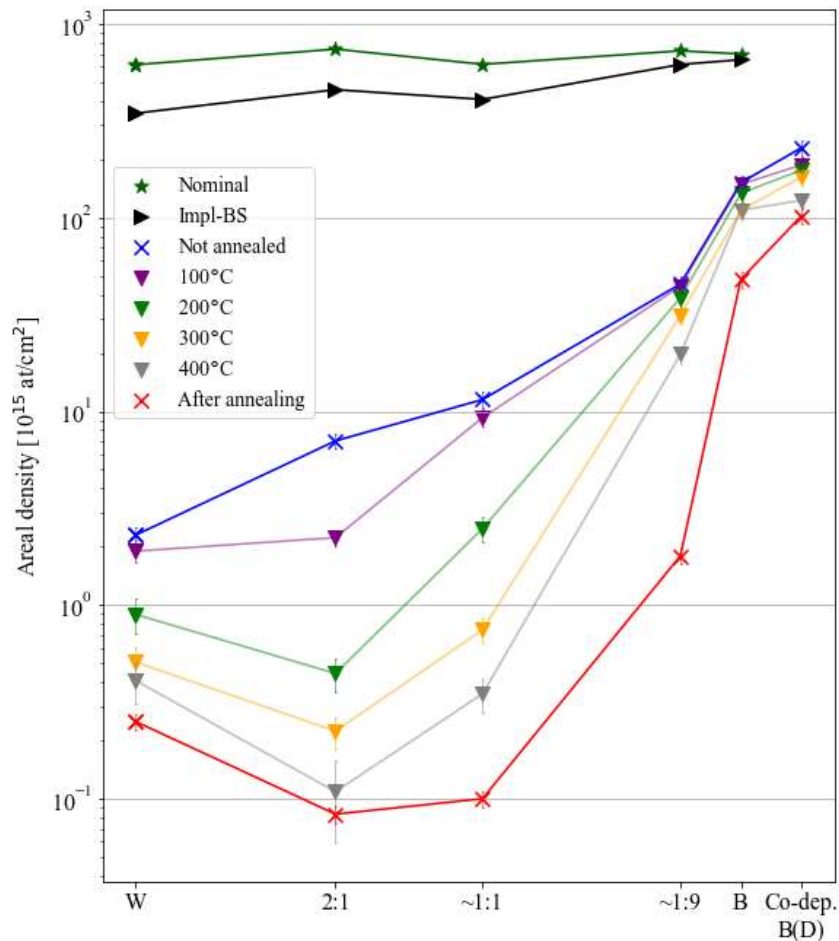


- Formation of bubbles only observed in W sample in D₂ implanted region.



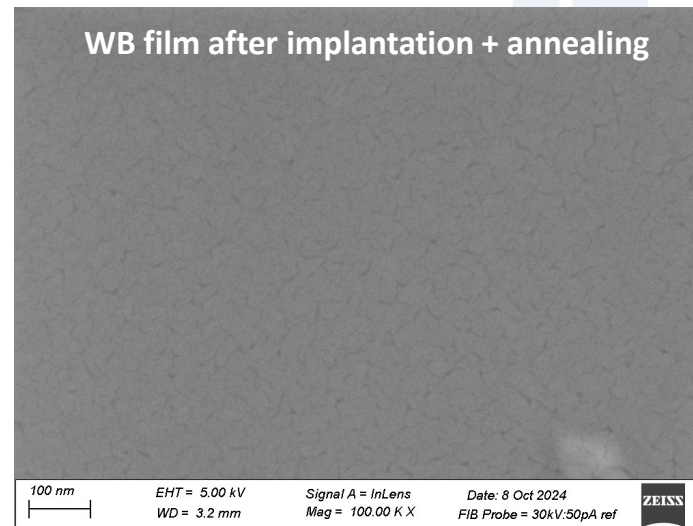
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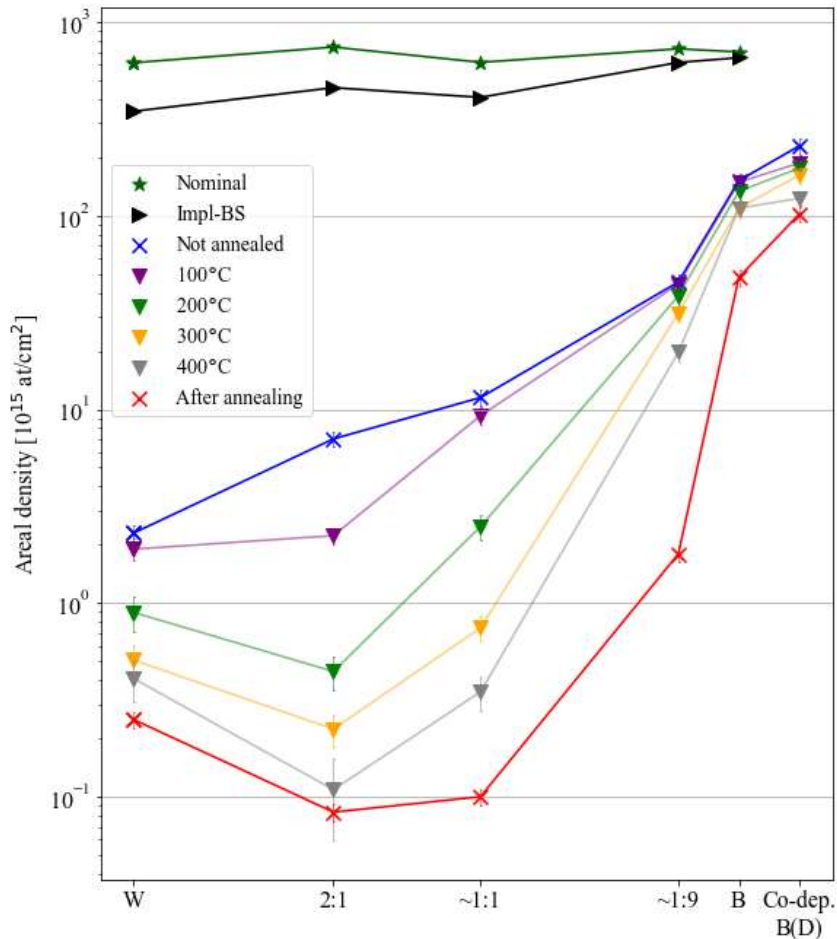


- Formation of bubbles only observed in W sample in D₂ implanted region.
- Formation of cracks in WB films (only in implanted region).



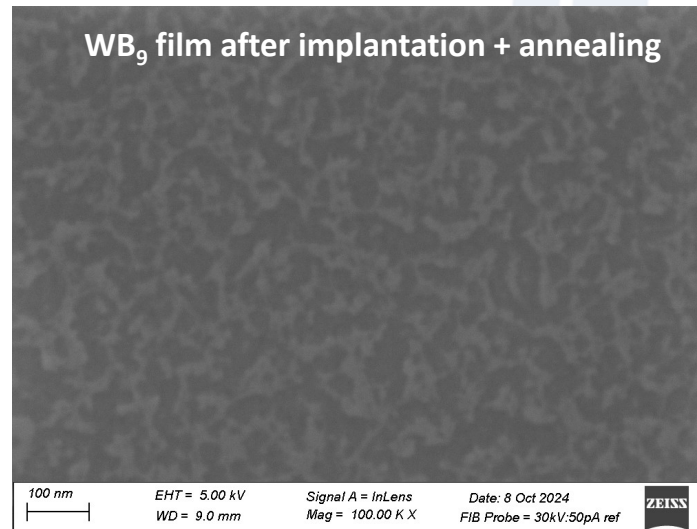
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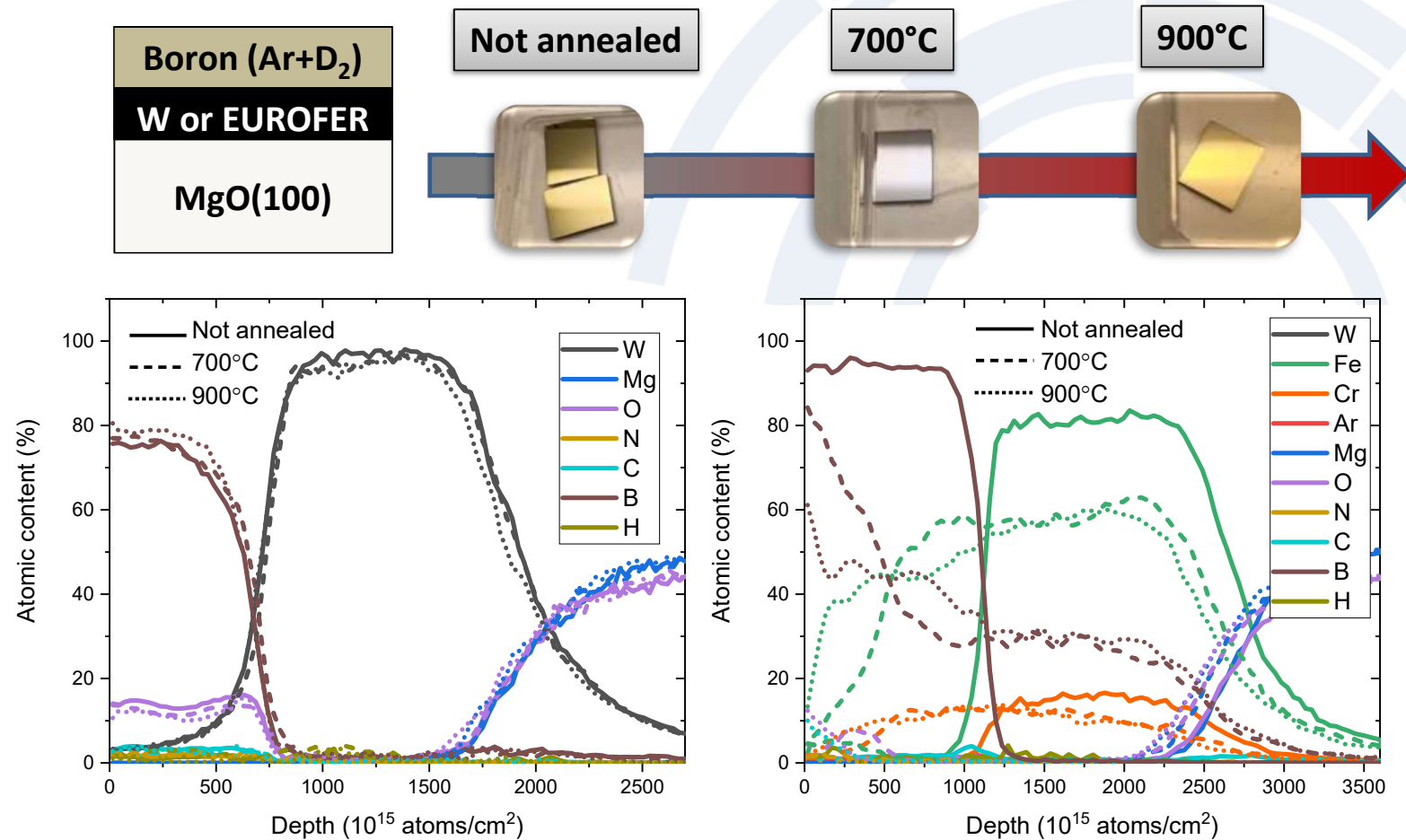
- Formation of bubbles only observed in W sample in D₂ implanted region.
- Formation of cracks in WB films (only in implanted region).
- W surface enrichment from preferential sputtering (confirmed with HR-RBS).



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.





Main activities proposed for 2025

WP 1: Sample preparation & characterization:

Continue deposition and characterization of layers.
Complete mechanical characterization of layers.

WP 2: Sputtering Yields and BCA simulations

Complete the sputtering yields and BCA simulations for W-B layers.
Start sputtering yields and BCA simulations for EUROFER97-B layers.
Sputtering yield in function of surface composition from ion irradiated structures (with WP-5).

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

Ab initio data on B-W defects.

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

MD sputtering yields from W and B surface layers.
MD predictions of sputtering yields from W-containing surfaces with evolving composition and morphology and comparison to experiment (WP2 and 5).

WP 5: Material Modification and Atomic Migration

Exposure of mixed layers to $^{18}\text{O}_2$ and monitoring of oxygen incorporation and depth profile by ion beam analysis.



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 Fellingner (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 initio 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 Fellingner (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 initio 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 18O ₂ 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.



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Thank you!



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

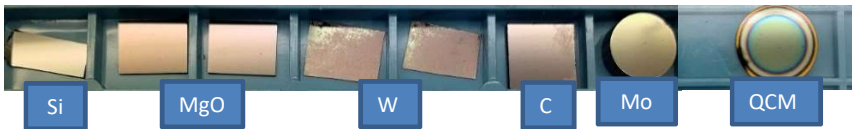
Sputtering depositions under argon atmospheres:

B target (RF-150 W):

0.5 nm/min; 5 hours deposition.

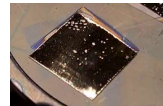
Substrate effect:

- 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.

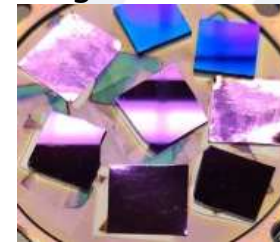


Sputtering depositions under argon Ar+D₂:

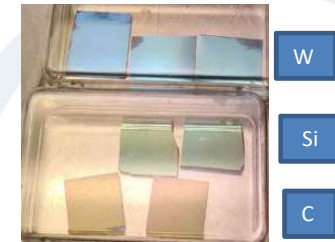
B target (RF-150 W):

$f_{Ar} = 10$ sccm; $f_{D_2} = 18$ sccm; $P_{Ar+D_2} = 7 \times 10^{-3}$ 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):

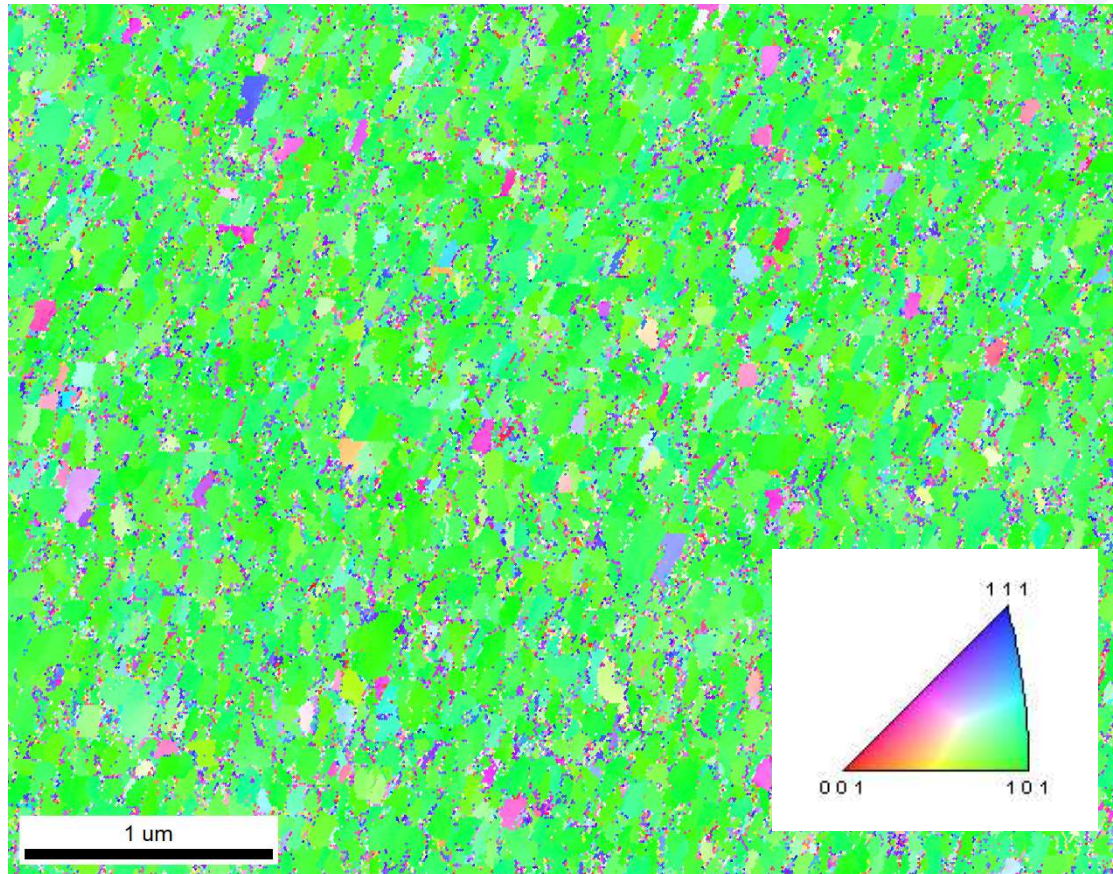
- Similar as in Ar depositions: flat, homogeneous, stable in air.





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

EBSD for W layer

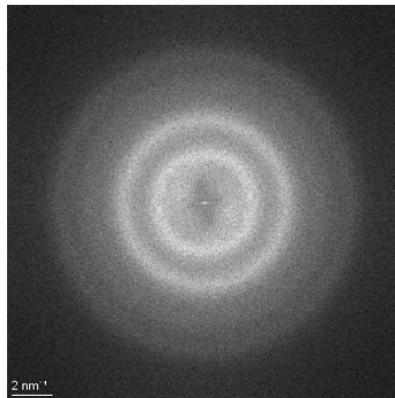
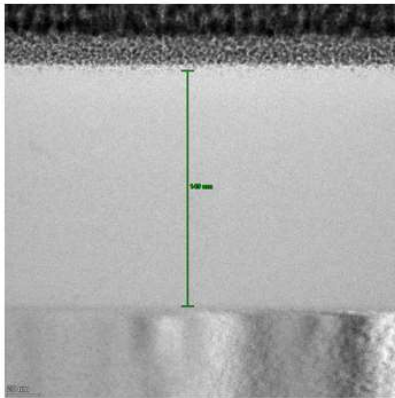




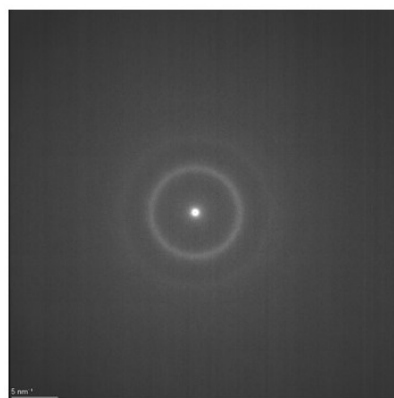
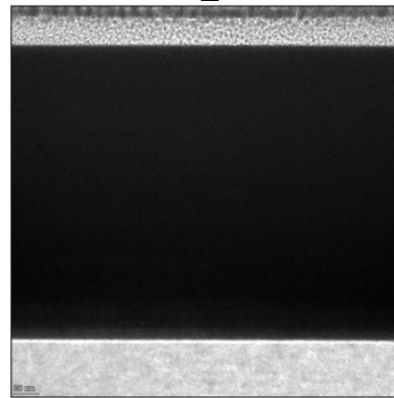
W-B film characterization (100-150 nm films)

TEM for W-B layers

B/Si



W₂B/Si



WB/Si

