

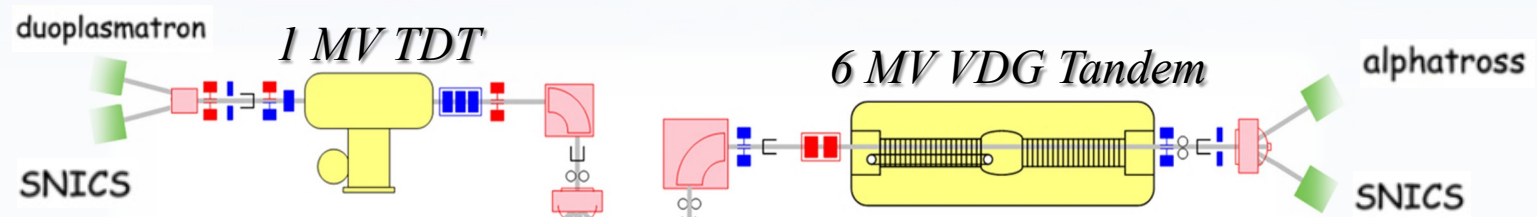
# A SEM, EDS and $^3\text{He}$ -NRA synergy in the micro analysis of AUG dust particles

Georgios Provatas

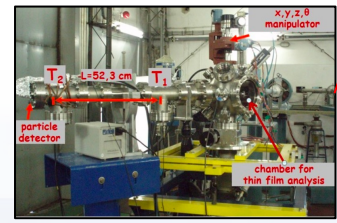
S. Fazinić, I. Bogdanović Radović, I. Božičević Mihalić, T. Dunatov, K. Ivanković Nizić

M. Balden, V. Rohde

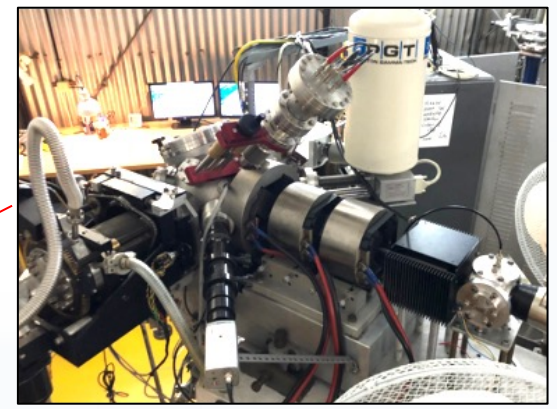
# The RBI accelerator facility infrastructure



Dual Microprobe

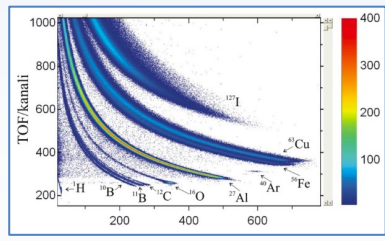
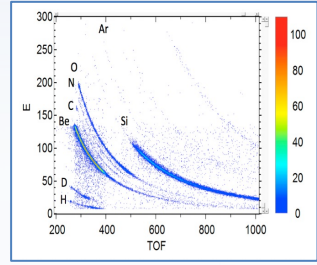
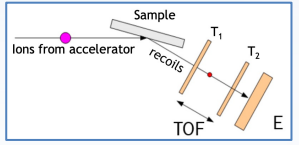


ToF ERDA



Microprobe

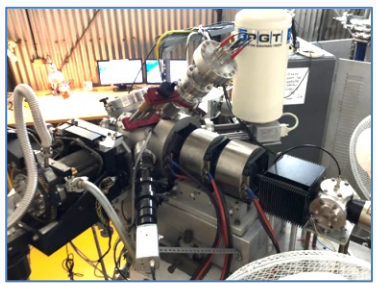
### ToF-ERDA



- Mass resolution is better than 1 for  $M < 40$
- Sensitivity 0.1 at%
- For films containing heavy elements (like W) TOF-ERDA can measure concentrations and depth profiles of light elements in first 100-200 nm

Beam spot of few mm  
Not appropriate for dust analyses

### uNRA, uPIXE



Si(Li) X-Ray detector  
30 mm<sup>2</sup>, 12.5 μm Be window

<sup>3</sup>He beam, 4 MeV  
Focused ≈ about 6 μm  
Current < 70 pA

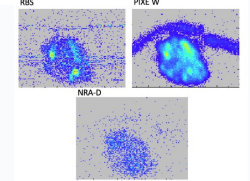
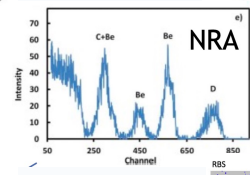
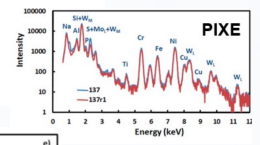
PDSSB Detector  
ORTEC BA-022-300-2000  
Depletion depth = 2000 μm  
Active area 300 mm<sup>2</sup>  
Collimated to 230 mm<sup>2</sup>  
Foil: mylar 11.9 μm + Ti 10.5 μm  
θ = 135 deg extended to ± 19 deg

Ni mesh 50 μm  
Area ≈ 350x350 μm

Sample holder

### IBA analysis of dust particles:

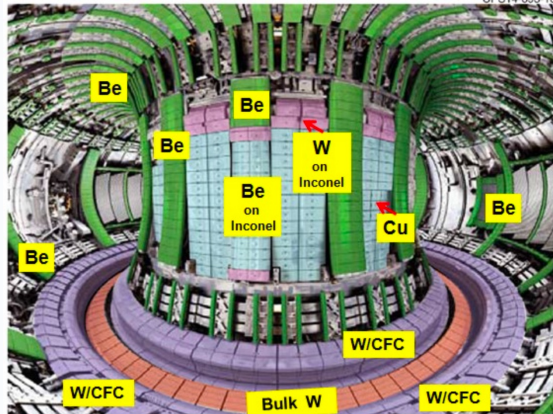
- Focused ion beam: 2.7 - 4 MeV <sup>3</sup>He ≈ 6 μm
- Ion beam currents about 70 pA
- scanned areas: 350x350 μm
- NRA: light elements (D, B, Be, C, N)
- PIXE: Na and heavier elements
- EBS: C, O, N, etc.
- analysis to the depth of about 15 μm in Be rich samples





- Dust particles from the JET-ILW studied at RBI

Previously  
on RBI



■ Beryllium   ■ CFC tungsten coated   ■ Inconel tungsten coated  
■ Tungsten   ■ Inconel beryllium coated

J.C. Flanagan et al., Plasma Phys. Control. Fusion 57 (2015) 014037

2

### Ion Microbeam Analyses of Dust Particles and Codeposits from JET with the ITER-Like Wall

Stjepko Fazinić<sup>\*,†</sup> Tonči Tadić<sup>†</sup> Marin Vukšić<sup>†</sup> Marek Rubel<sup>‡</sup> Per Petersson<sup>‡</sup>  
Elżbieta Fortuna-Zaleśna<sup>§</sup> and Anna Widdowson<sup>||</sup>

2018

### Micro-analyses of dust particles generated in the JET tokamak with the ITER-like wall

Stjepko Fazinić<sup>1</sup>, Iva Božičević-Mihalić<sup>1</sup>, Georgios Provas<sup>1</sup>, Tonči Tadić<sup>1</sup>,  
Marek Rubel<sup>2</sup>, Elżbieta Fortuna-Zaleśna<sup>3</sup>, Anna Widdowson<sup>4</sup> and JET contributors<sup>5</sup>

2020

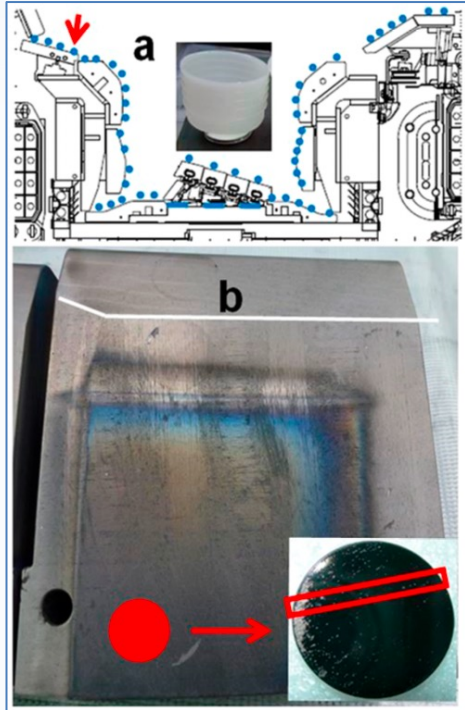
Article

### Dust Monitors in JET with ITER-like Wall for Diagnosis of Mobilized Particles and Co-Deposited Layers

Stjepko Fazinić<sup>1,\*</sup>, Georgios Provas<sup>1,\*</sup>, Iva Božičević Mihalić<sup>1</sup>, Tonči Tadić<sup>1</sup>, Marek Rubel<sup>2</sup>,  
Justyna Grzonka<sup>3</sup>, Per Petersson<sup>2</sup>, Anna Widdowson<sup>4</sup>, Sunwoo Moon<sup>2</sup> and Elżbieta Fortuna-Zaleśna<sup>5</sup>

2022

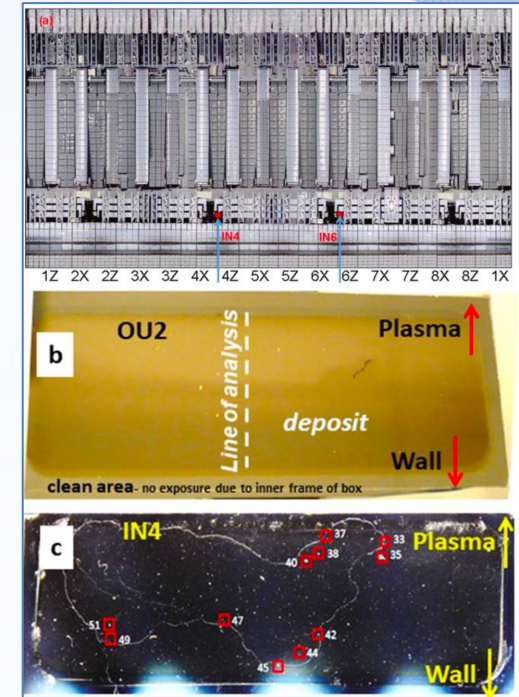
# Micro-analyses of JET-ILW particles @ RBI



Dust sampled using stick carbon pad from the inner divertor tile after ILW-2 (2013-2014)

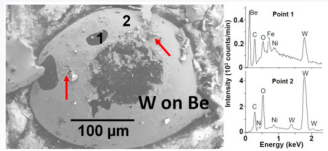


Samples collected from vacuum cleaner after the ILW-3 in 2015-2016

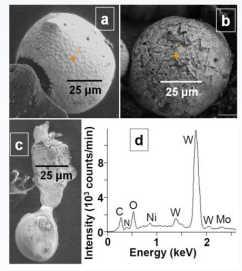
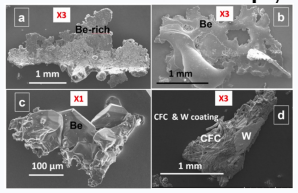


Dust particles collected ILW-2 and ILW-3 on Si plates-monitors, wall probes for erosion and deposition studies.

# Micro-analyses of JET-ILW particles @ RBI



SEM / EDX microscopy



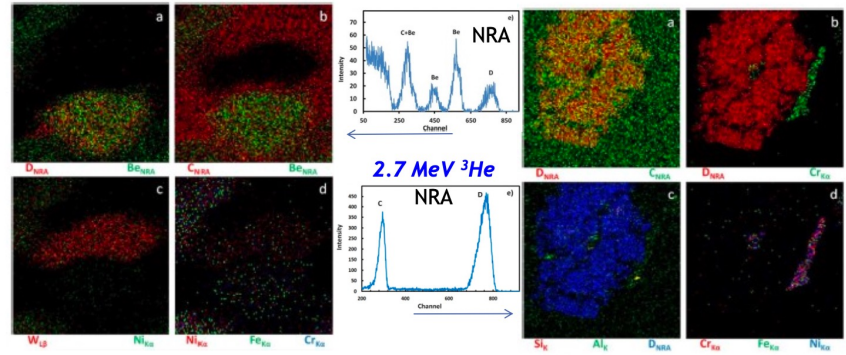
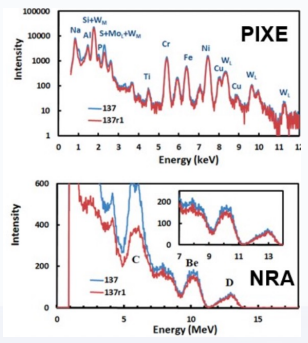
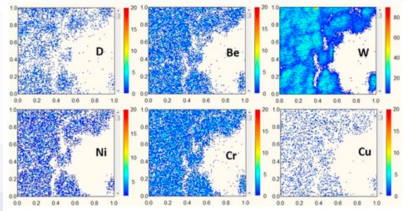
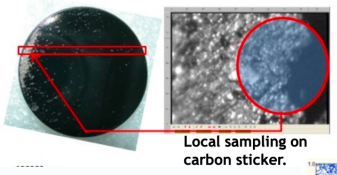
Initially, particles interesting for further analysis are characterized and selected by means of SEM/EDS microscopy.

**Warsaw University of Technology**



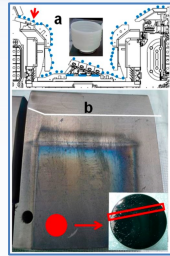
EDS not sensitive to D  
Not accurate in light elements quantification

Selected particles were further studied by NRA, PIXE microscopy





# Findings from micro-analyses of JET-ILW particles @ RBI



Two types of dust particles were found

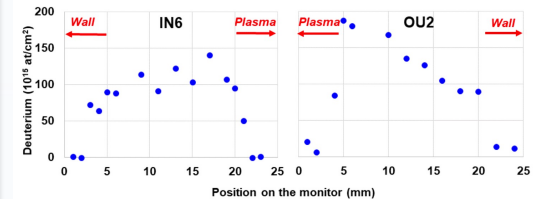
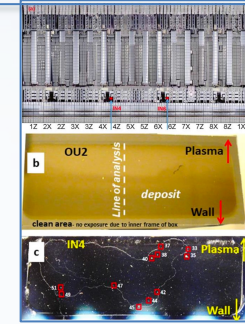
- 1) Larger Be-rich >90 at% with D up to 3.4 at% also Ni (1–3 at%), Cr (0.4–0.8 at%), W (0.2–0.9 at%), Fe (0.3–0.6 at%), and Cu and Ti.
- 1) small particles rich in Al and/or Si that were in some cases accompanied by other elements, such as Fe, Cu, or Ti or W and Mo.



- 1) Areas of  $\sim 120 \times 120 \mu\text{m}$  rich in Be, W and steel or Inconel components and NRA spectrum with Be and D presence.
- 2) Carbon particles with high D-content



Clear evidence that fuel retention is predominantly associated with Carbon.



Different types of particles were found.

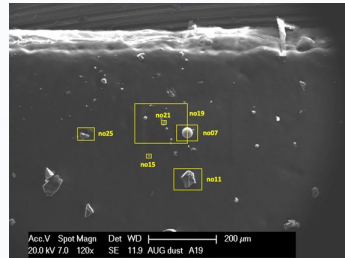
Deuterium content in co-deposits on the monitor did not exceed  $2 \times 10^{17} \text{cm}^{-2}$ .



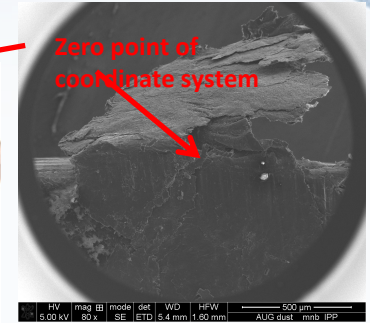
Low fuel retention and a very small amount ITER-relevant dust generated in JET-ILW

# Micro-analyses of particles from AUG @ RBI

On 2020 M. Balden sent to RBI dust particles for AUG on two sticky carbon pads



Coordinates of 2<sup>nd</sup> particle at edge (-9.11 / 4.37)



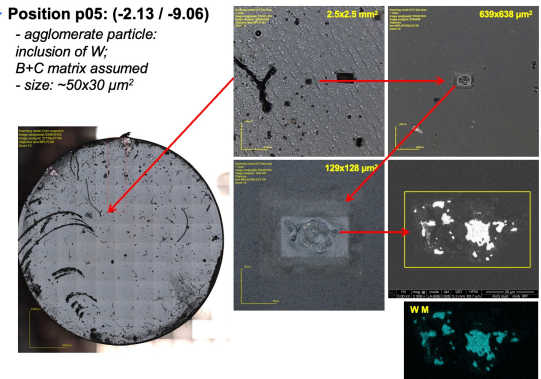
The ROIs were initially prioritized by means of EDS microscopy.

Detailed SEM images with coordinates of interesting particles highlighted for further u-IBA analysis.

## AUG dust: HS12-2009 – priority 1

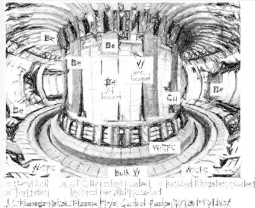


► Position p05: (-2.13 / -9.06)  
 - agglomerate particle:  
 inclusion of W;  
 B+C matrix assumed  
 - size: ~50x30 μm<sup>2</sup>

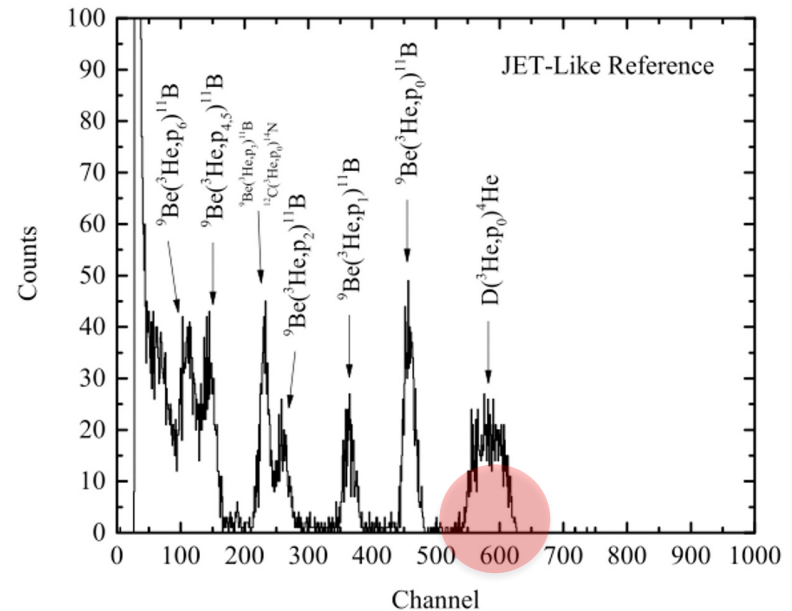
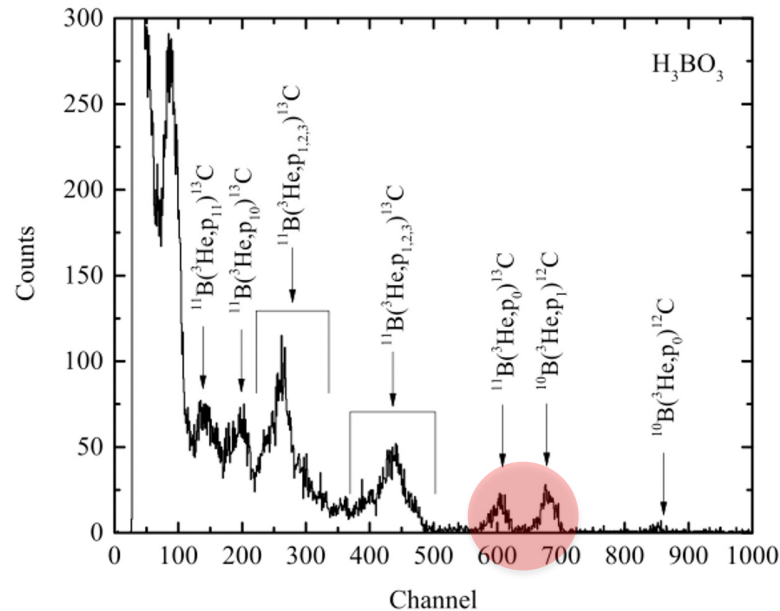




# Micro-analyses of particles from AUG @ RBI

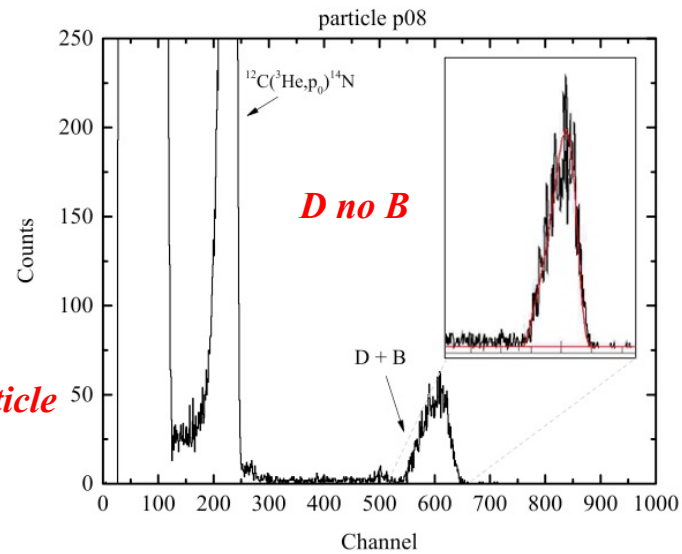
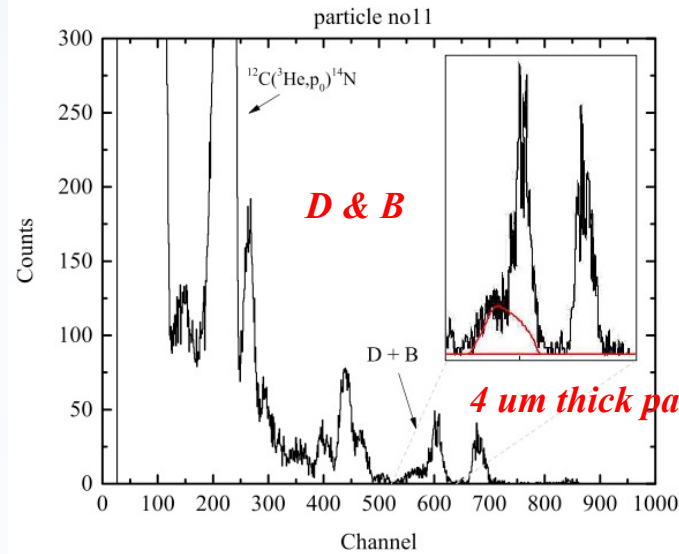


In AUG first wall is Be-free, however, Boron could be present due to boronization, that creates a thin B film on the walls to reduce the levels of impurity contamination in plasma

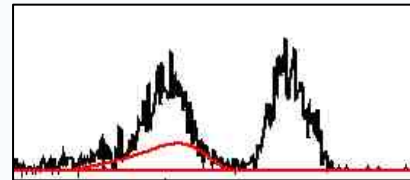


NRA analysis for D quantification can be challenging due to signal overlap.

# Micro-analyses of particles from AUG @ RBI



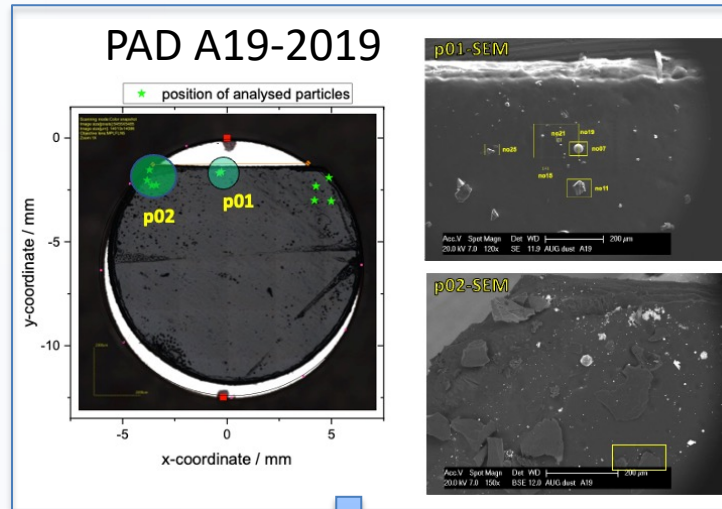
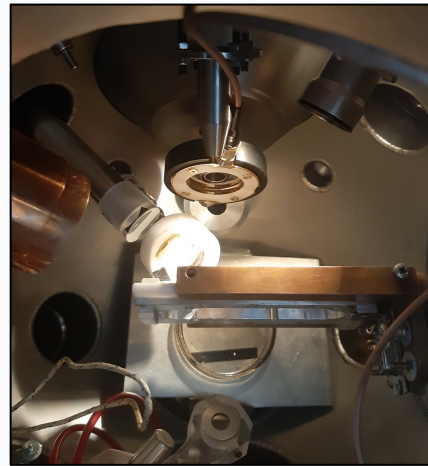
***8 um thick particle***



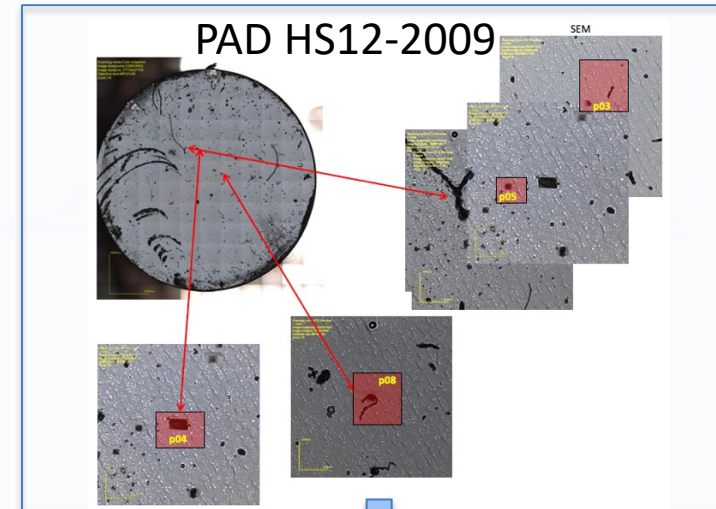
NRA analysis for D content carried out using SIMNRA, taking into account the angular spread of the detector.

# Micro-analyses of particles from AUG @ RBI

The two pads were inserted in the microprobe and with the use of a microscope ROIs were identified



Deuterium with Boron



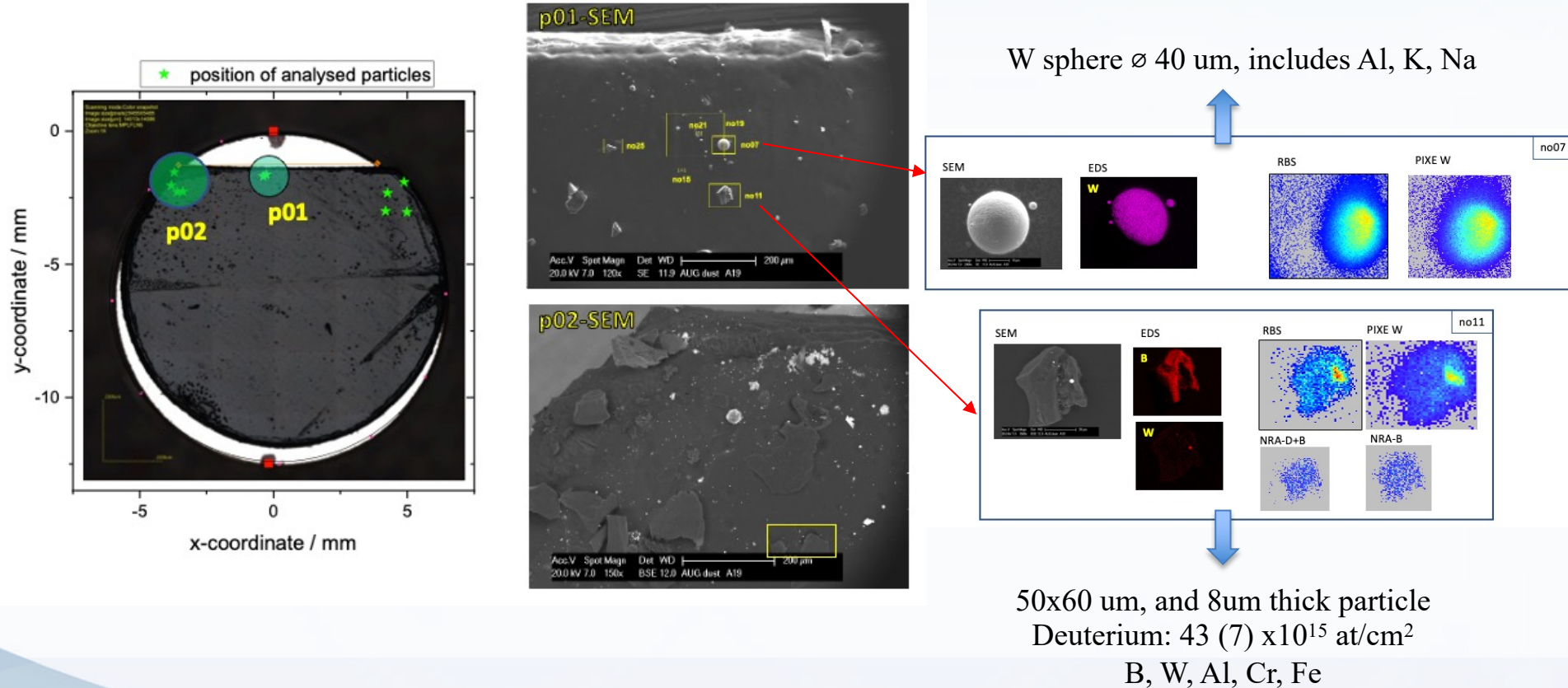
Boron free = Clear isolated D peak for analysis



# Micro-analyses of particles from AUG @ RBI

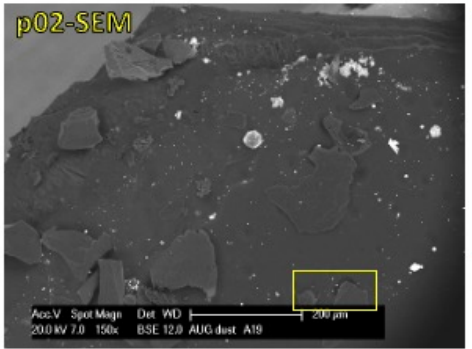
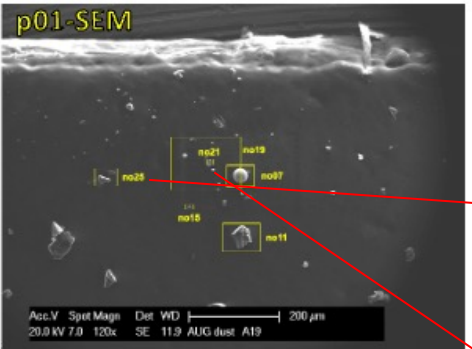
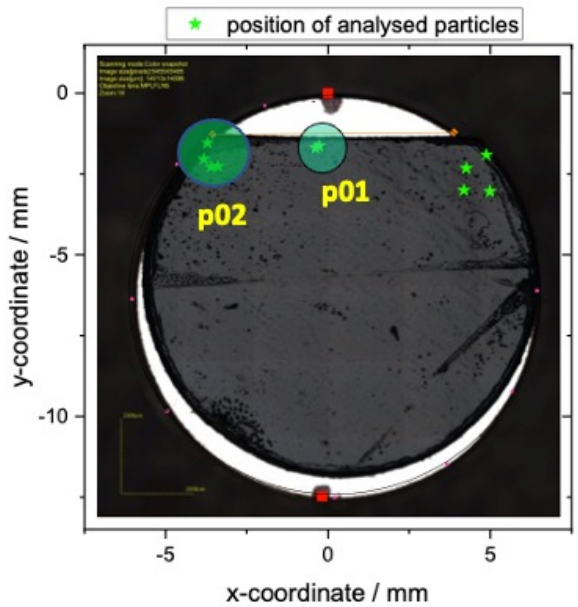
PAD A19-2019

Most of the collected particles in the first pad are located near the top edge

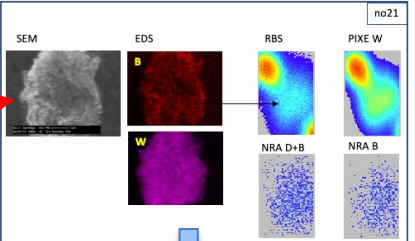
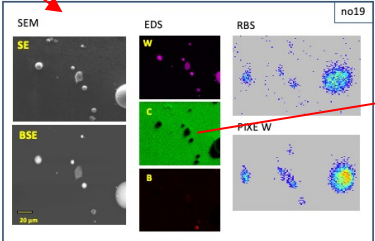
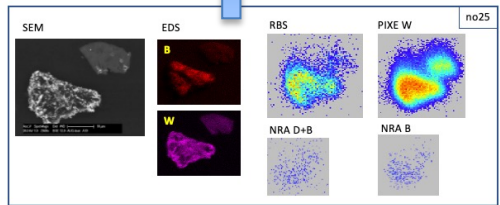


# Micro-analyses of particles from AUG @ RBI

PAD A19-2019



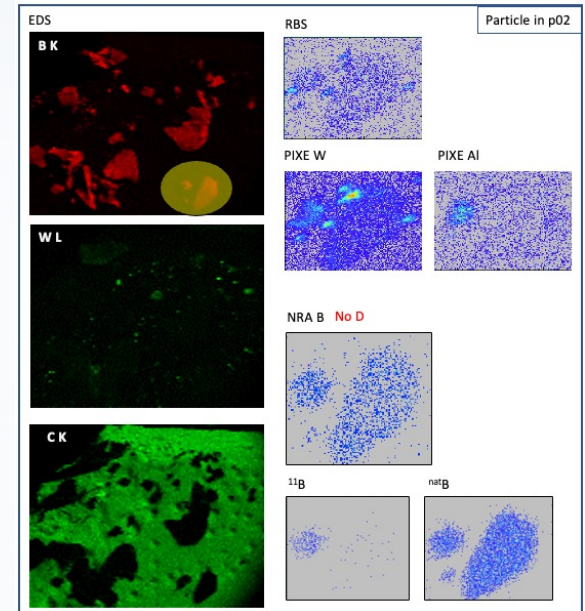
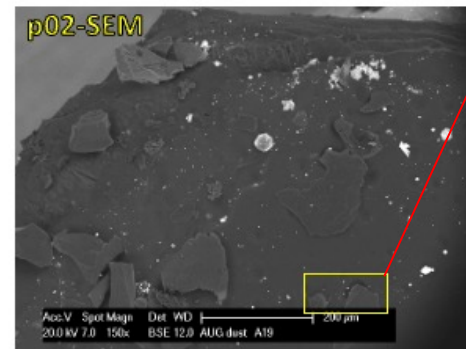
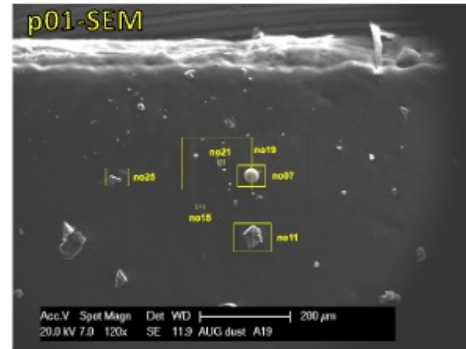
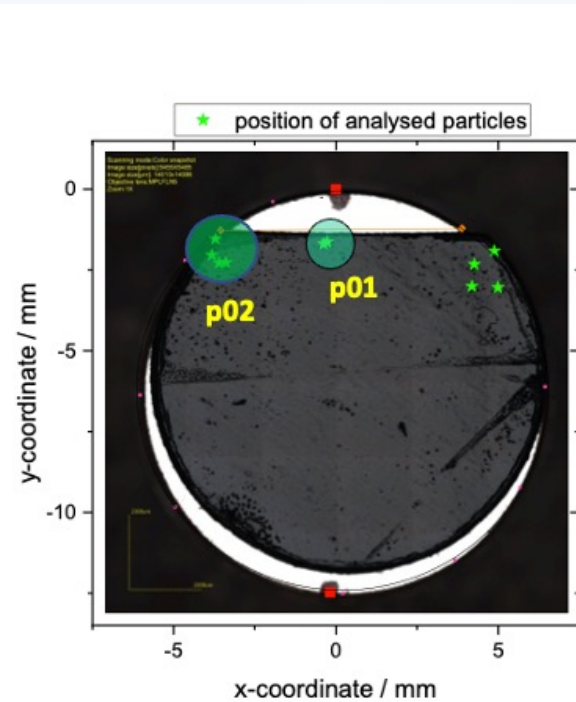
2 particles  
 20x20, 30x40 um and 8 um thick  
 Deuterium:  $18 (3) \times 10^{15}$  at/cm<sup>2</sup>  
 B, W, Al, Cr, Fe



Middle particle of 3 particles in map  
 15x20 um and 4 um thick particle  
 Deuterium:  $12 (2) \times 10^{15}$  at/cm<sup>2</sup>  
 B, W, Al, Cr, Fe

# Micro-analyses of particles from AUG @ RBI

PAD A19-2019



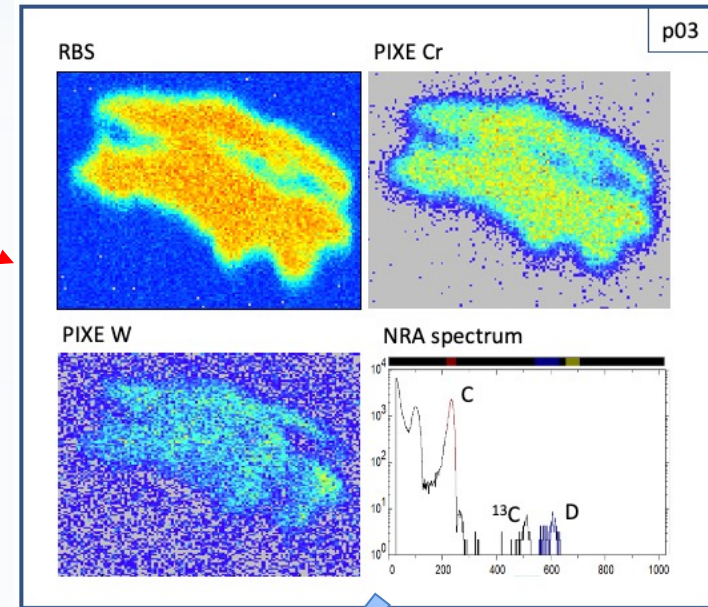
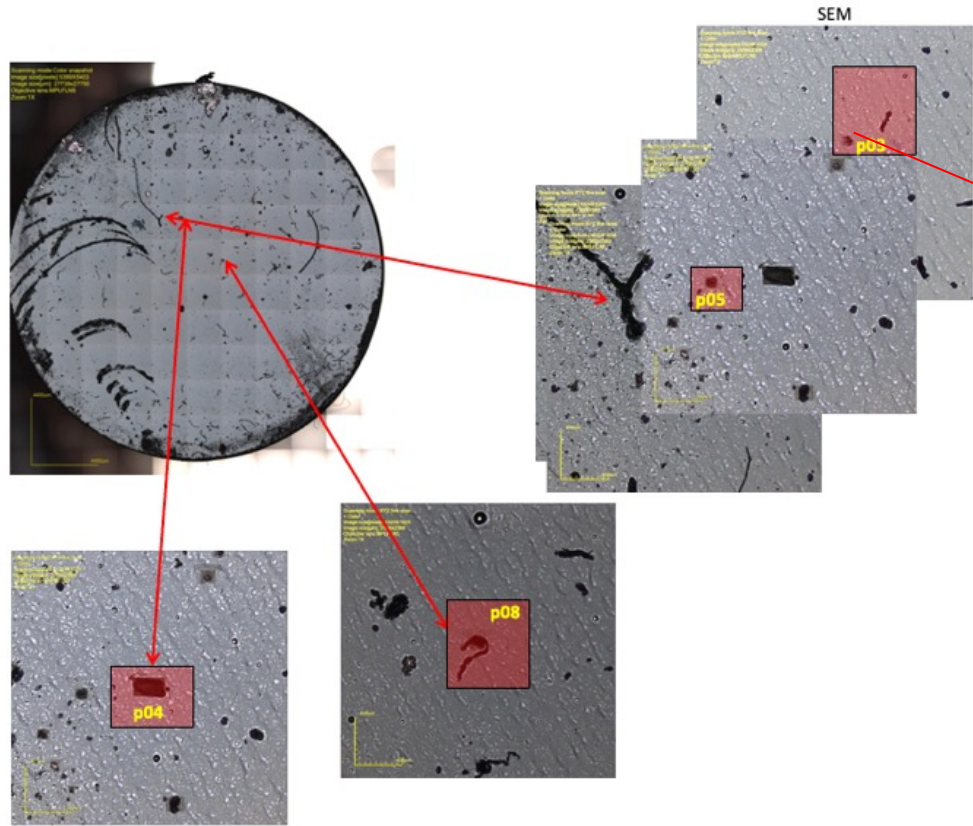
Strange? area of two Boron particles 50x60 μm and 20x20 μm  
 No Deuterium  
 One particle enriched in <sup>11</sup>B the other natB,  
 Includes W, Al.



# Micro-analyses of particles from AUG @ RBI

PAD HS12-2009

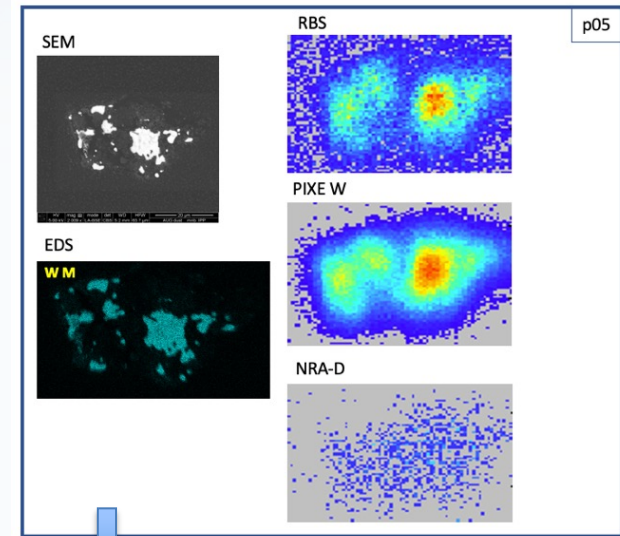
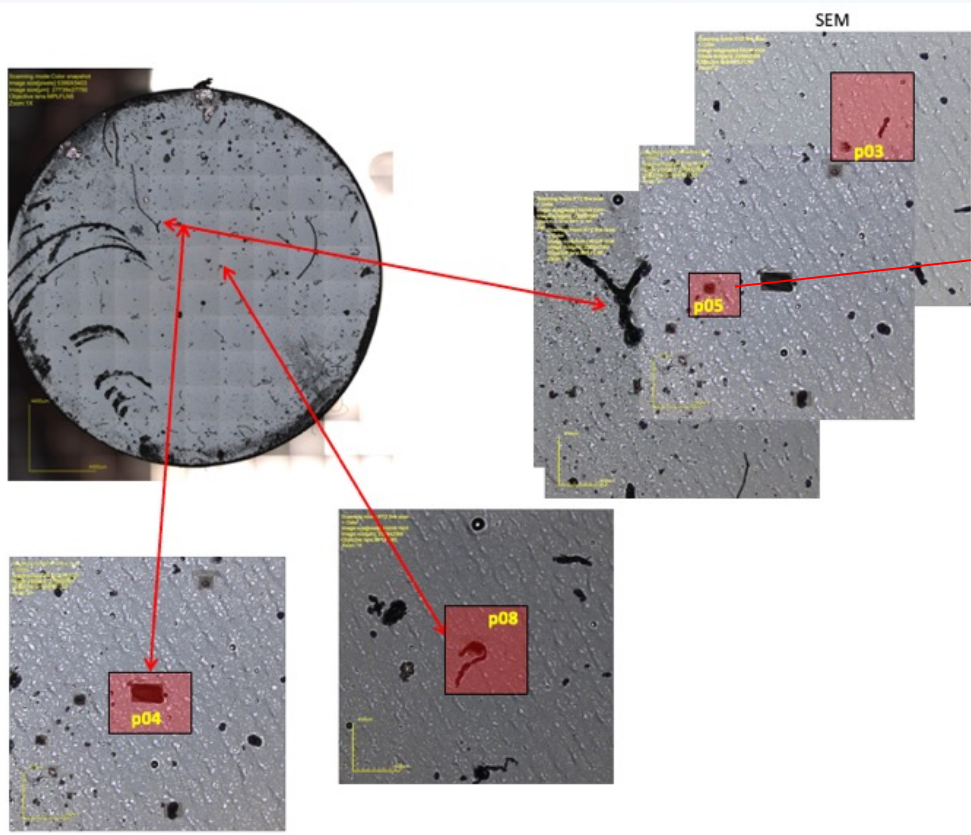
Absence of Boron. Areas of analysis near the middle of the pad



Big particle, 100x50 um and 10 um thick  
 Deuterium:  $5 (1) 10^{15}$  at/cm<sup>2</sup>  
 Cr, W, Fe present

# Micro-analyses of particles from AUG @ RBI

PAD HS12-2009



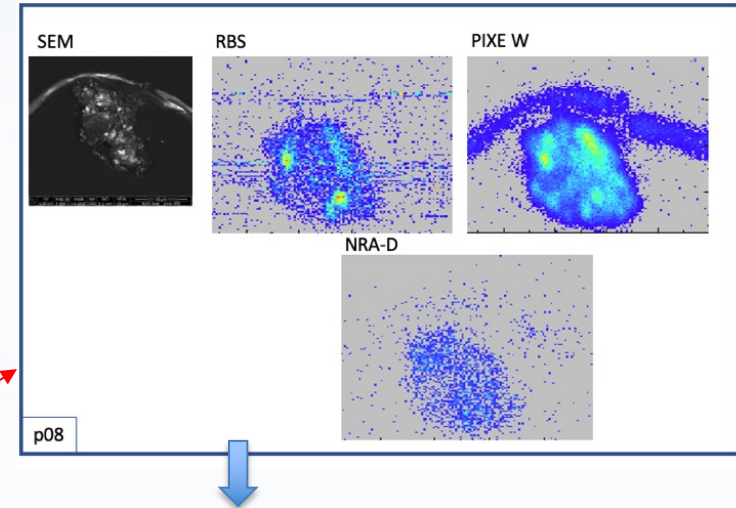
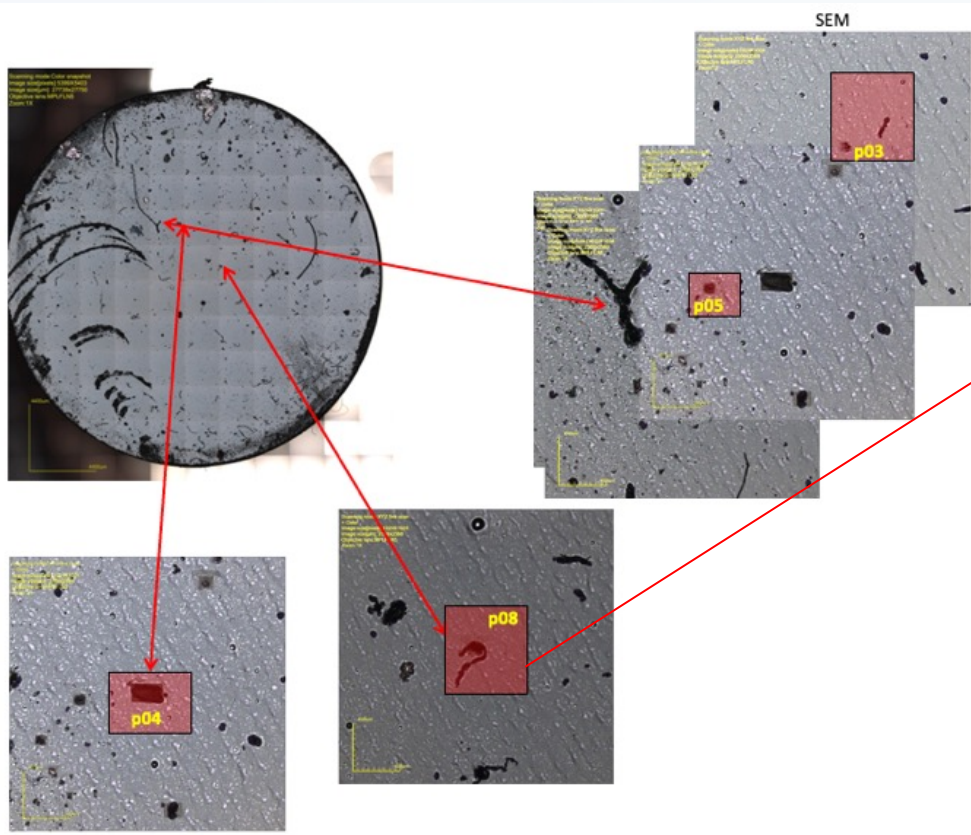
70x50 um area with a cluster of particles. Average 3 um thick.

Deuterium:  $105 (16) 10^{15}$  at/cm<sup>2</sup>  
W, Al, K.



# Micro-analyses of particles from AUG @ RBI

PAD HS12-2009

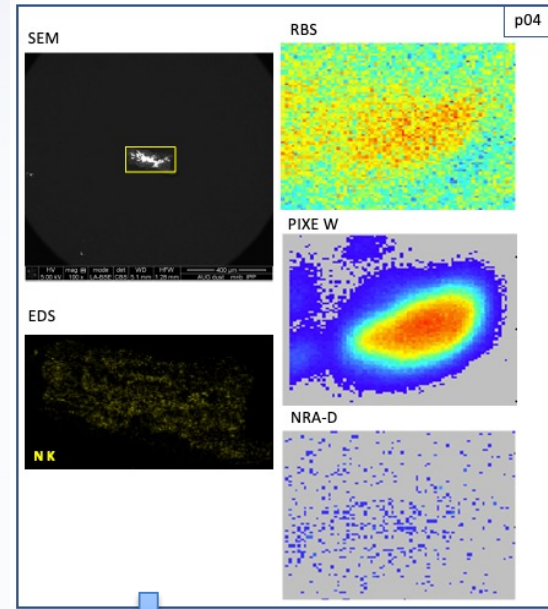
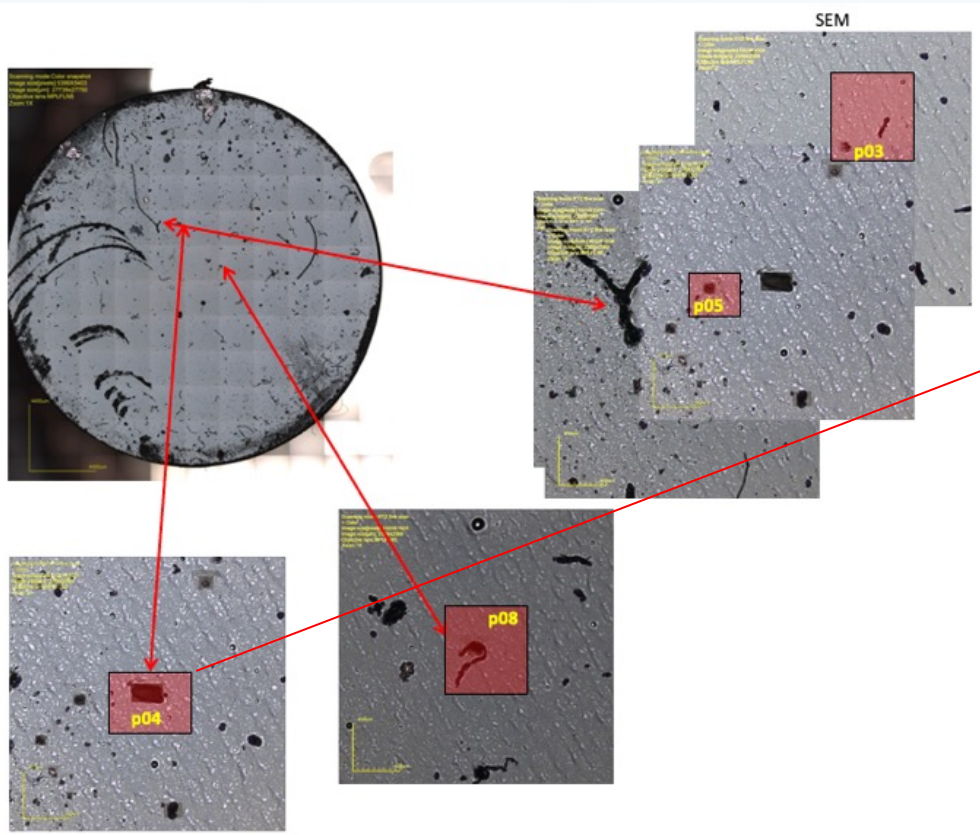


70x40  $\mu\text{m}$  area with 2 particles, 10  $\mu\text{m}$ . On of them contains D 10  $\mu\text{m}$  thick  
 Deuterium:  $487 (40) 10^{15} \text{ at/cm}^2$   
 W, Al, Cr, Fe, K, Na.



# Micro-analyses of particles from AUG @ RBI

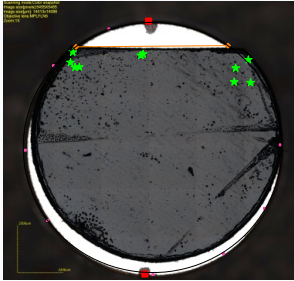
PAD HS12-2009



A big, 200x200 um area with few big structures. In average 10 um thick  
 Deuterium:  $13 (2) 10^{15}$  at/cm<sup>2</sup>  
 W, Al, Cr, Fe.

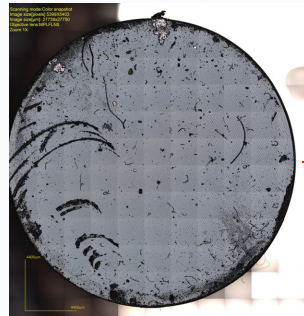
## Micro-analyses of particles from AUG @ RBI

### PAD A19-2019



Particles or groups of particles with sizes up to 50x60  $\mu\text{m}$   
Particles with Deuterium up to  $43 \times 10^{15} \text{ at/cm}^2$   
Containing B, W, Al, Cr, Fe

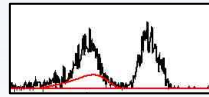
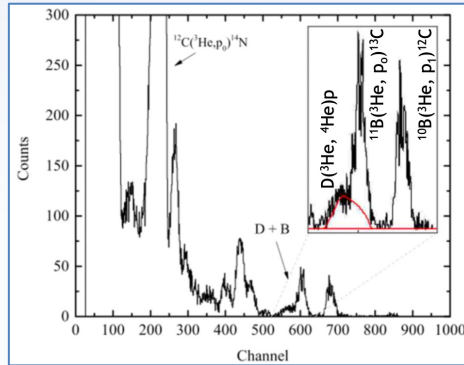
### PAD HS12-2009



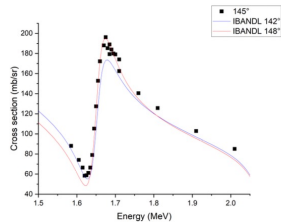
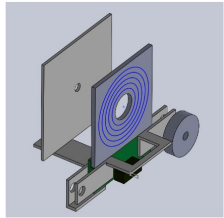
No Boron

Larger particles or groups of particles with sizes up to 200x200  $\mu\text{m}$   
Two types of particles with Deuterium:  
Areal density up to  $13 \times 10^{15} \text{ at/cm}^2$   
Areal density up to  $487 \times 10^{15} \text{ at/cm}^2$   
Containing W, Al, Cr, Fe,

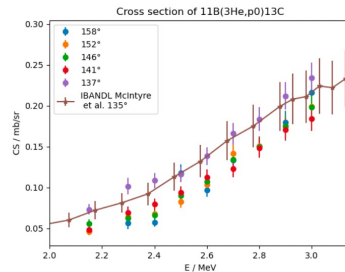
# On the road to accurate NRA analyses



Need for reliable  $^{11}\text{B}(^3\text{He},p_0)^{13}\text{C}$  and  $^{10}\text{B}(^3\text{He},p_1)^{12}\text{C}$  reactions cross sections



Cross sections measured for 5 different angles in the  $^3\text{He}$  energy range of 2.15 - 3 MeV



- Novel setup with annular detector used for cross section measurements
- Thin B targets prepared and characterized at RBL

- Setup calibrated with known Si resonance

Within the IAEA CRP F11023, contract no. 24308 We collaborate with RUBION Bochum accelerator laboratory in the study of:

- $^9\text{Be}(^3\text{He},p_i)^{11}\text{B}$  up to 6 MeV
- $^{12}\text{C}(^3\text{He},p_i)^{14}\text{N}$  up to 6 MeV
- $^{11}\text{B}(^3\text{He},p_i)^{13}\text{C}$  up to 6 MeV
- $^{10}\text{B}(^3\text{He},p_i)^{12}\text{C}$  up to 6 MeV



*Thank you!*