



# **WPJET2: Material erosion, migration and dust**

**Marek Rubel , Per Petersson and JET2 Team**



This work has been carried out within the framework of the EUROfusion Consortium and has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement number 633053. The views and opinions expressed herein do not necessarily reflect those of the European Commission.



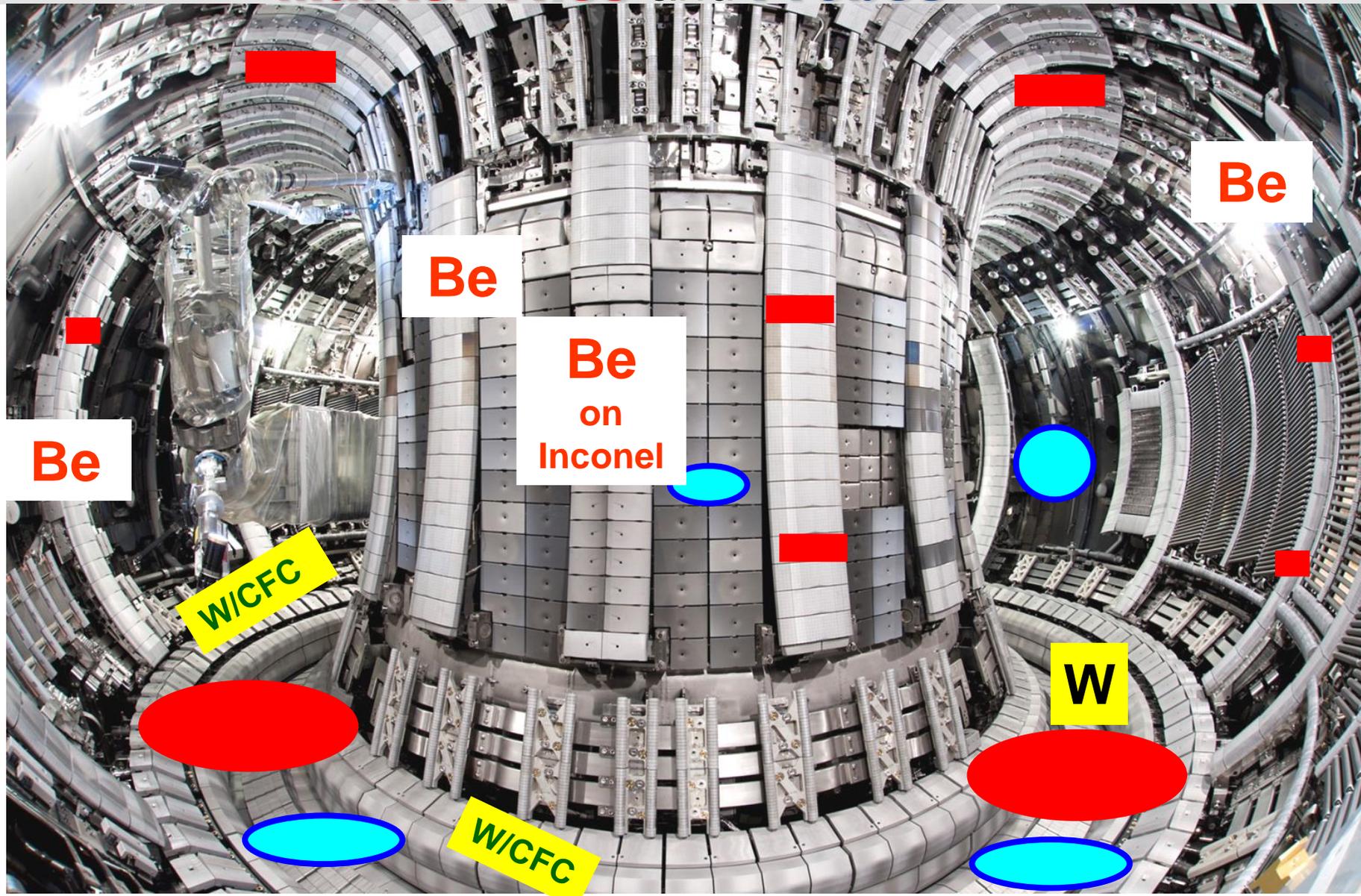
## ***Many thanks to the Team for excellent contributions.***

**In three JET2 overview talks we shall try to present highlights.**

- We have received many slides for the overview talks.
- All contributions will be addressed.
- Sorry if not all slides will be shown (*time constraints*).



## Marker Tiles and Probes



The aim is to have a complete overview of material migration and material damage, not just a number (even large) of analysis points and isolated findings.

# WPJET2: 7 years



**We work with PFCs and tokamakium containing beryllium and tritium.**

- *Laboratories: 12 in EU and cooperation with IFERC within Broader Approach*
- *Great human capital: experience and expertise*
- *Special equipment and many dedicated developments.*
- **Over 40 different material characterisation techniques from nano to macro:**
  - *Imaging*
  - *Composition from the first nm to depth (cross-sectioning)*
  - *Mechanical properties*
  - *Optical properties*
  - *9 techniques for deuterium and hydrogen studies.*
  - *9 (10) techniques/approaches in tritium assessment and analyses.*

Original scope of WPJET2 (2014) has been vastly broadened by new projects originating from:

- *new findings, own ideas, needs for clarification;*
- *ITER needs (requests).*



- ***Limiter tiles***
- ***Divertor tiles***
- ***Wall Probes***
- ***Dust and metal splashes***

## Comparison of campaigns

<b><i>ILW-1: 19.3 h</i></b>	<b><i>150 GJ</i></b>
<b><i>ILW-2: 19.1 h</i></b>	<b><i>201 GJ</i></b>
<b><i>ILW -3: 23.6 h</i></b>	<b><i>245 GJ</i></b>

- Exposure during single campaigns.
- Exposure during two or all three campaigns.

***Are material migration effects additive (retention, deposition)?***

# Beryllium Handling Facility (BeHF) at JET

## Handling of beryllium and tritiated components



All materials retrieved from JET go through that place and get their "passports" before shipment to other laboratories for detailed analyses or sectioning to smaller specimens.



# Sectioning of JET tiles:

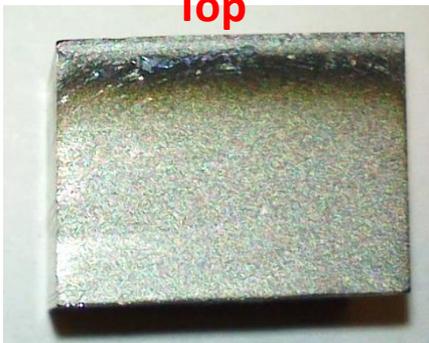


pre-requisite for definition of tasks and detailed studies

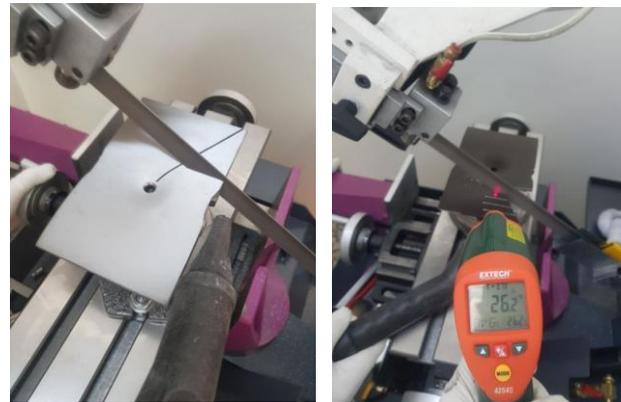
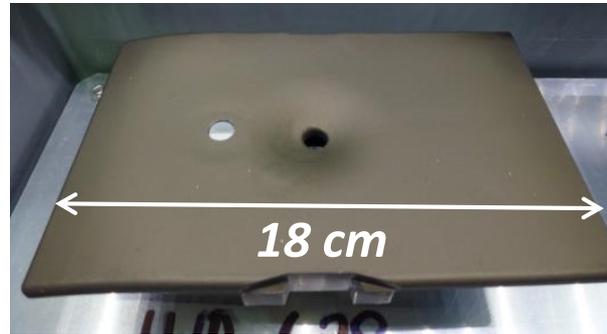
## Segmented Be limiters



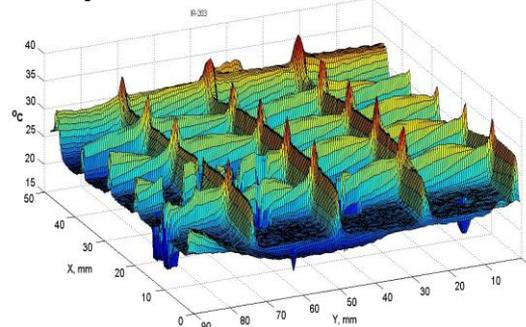
Top



## Be-coated Inconel

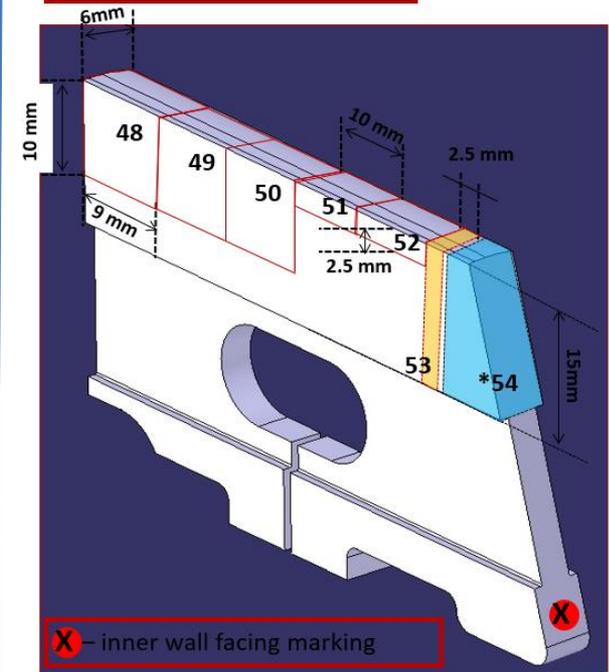


Temperature control:  $< 50\text{ }^{\circ}\text{C}$



## Bulk W lamella, Tile 5

A23 – Standard lamella

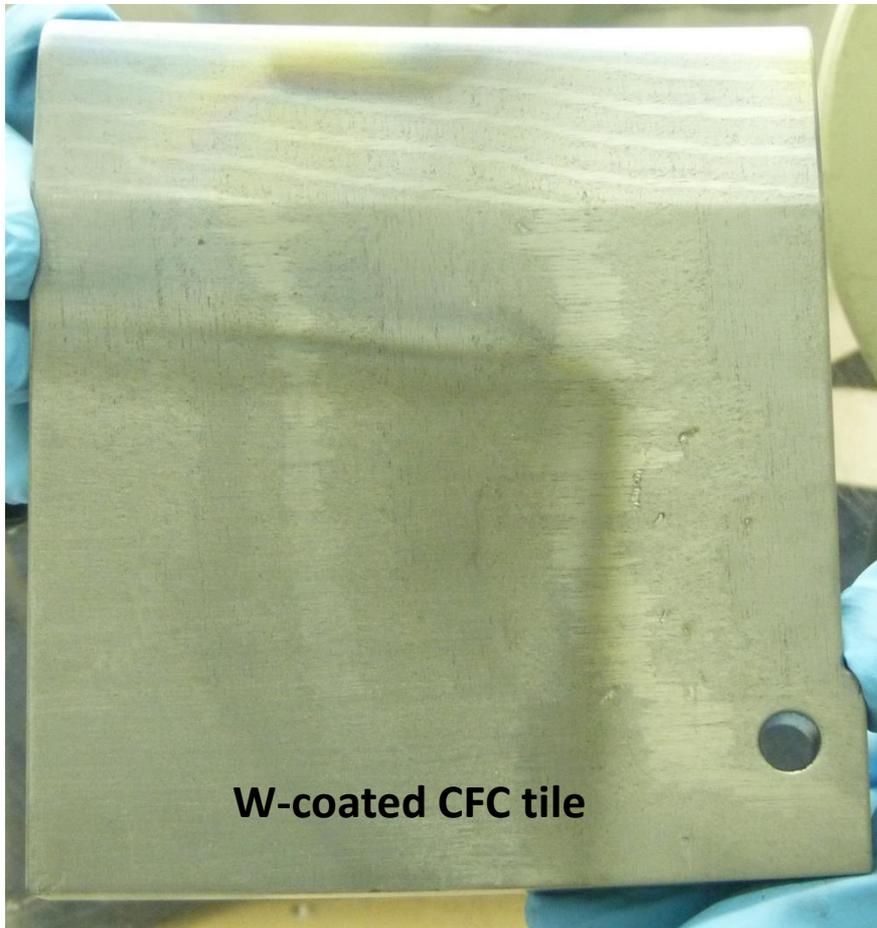


- For microscopy
- Ion beam analyses
- Thermal desorption
- Tritium measurements
- Emissivity
- Metallography

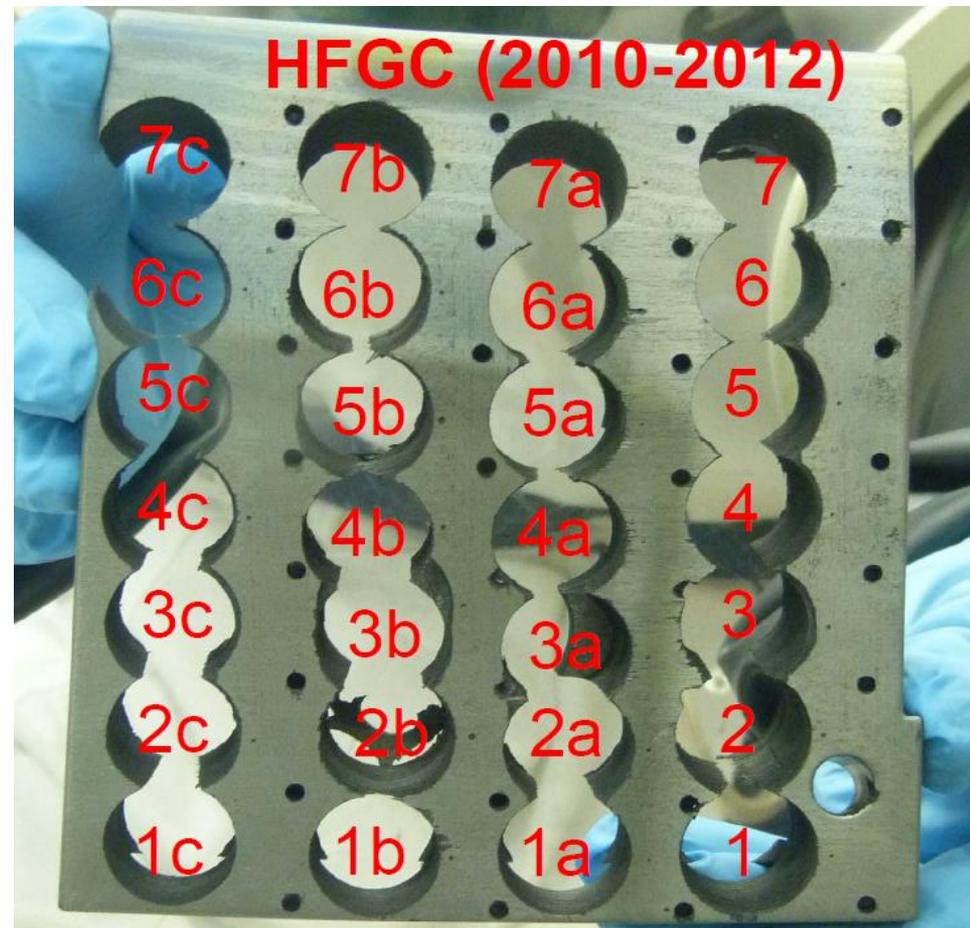
# Sampling/cutting & traceability/labeling



**Before**



**... and after coring**



Sampling for:

*SIMS, metallography, SEM, FIB/TEM, EDX, m-beam IBA, TDS, etc.*



# **Erosion - Deposition pattern in three campaigns**

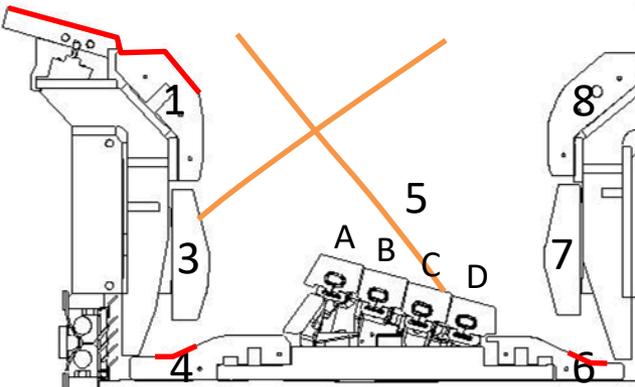
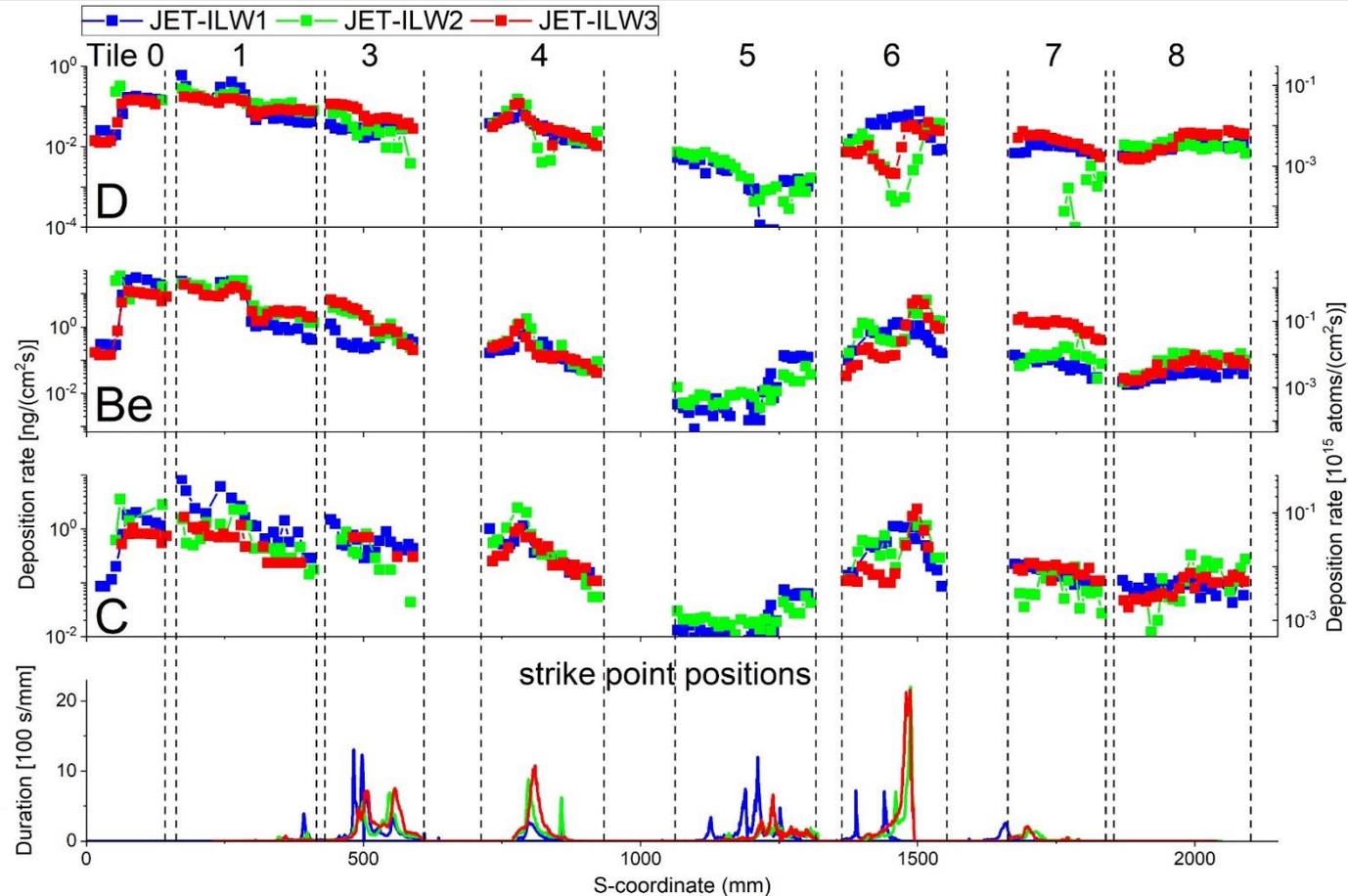
## **Divertor**

# Divertor: deposition from ILW-1 to ILW-3



## Messages:

- Qualitatively similar distributions in all campaigns.
- Maximum deposition on Tiles 0, 1
- Higher deposition on Tiles 4, 6 in ILW-2 and ILW-3



# Divertor: deposition from ILW-1 to ILW-3



## Deposited mass (g)

	ILW-1	ILW-2	ILW-3
<b>D</b>	0.9	0.7	0.9
<b>Be</b>	53	60	46
<b>C</b>	13	7	6

## Deposited mass rate ( $\mu\text{g/s}$ )

	ILW-1	ILW-2	ILW-3
<b>D</b>	13	10	11
<b>Be</b>	771	877	544
<b>C</b>	189	102	71

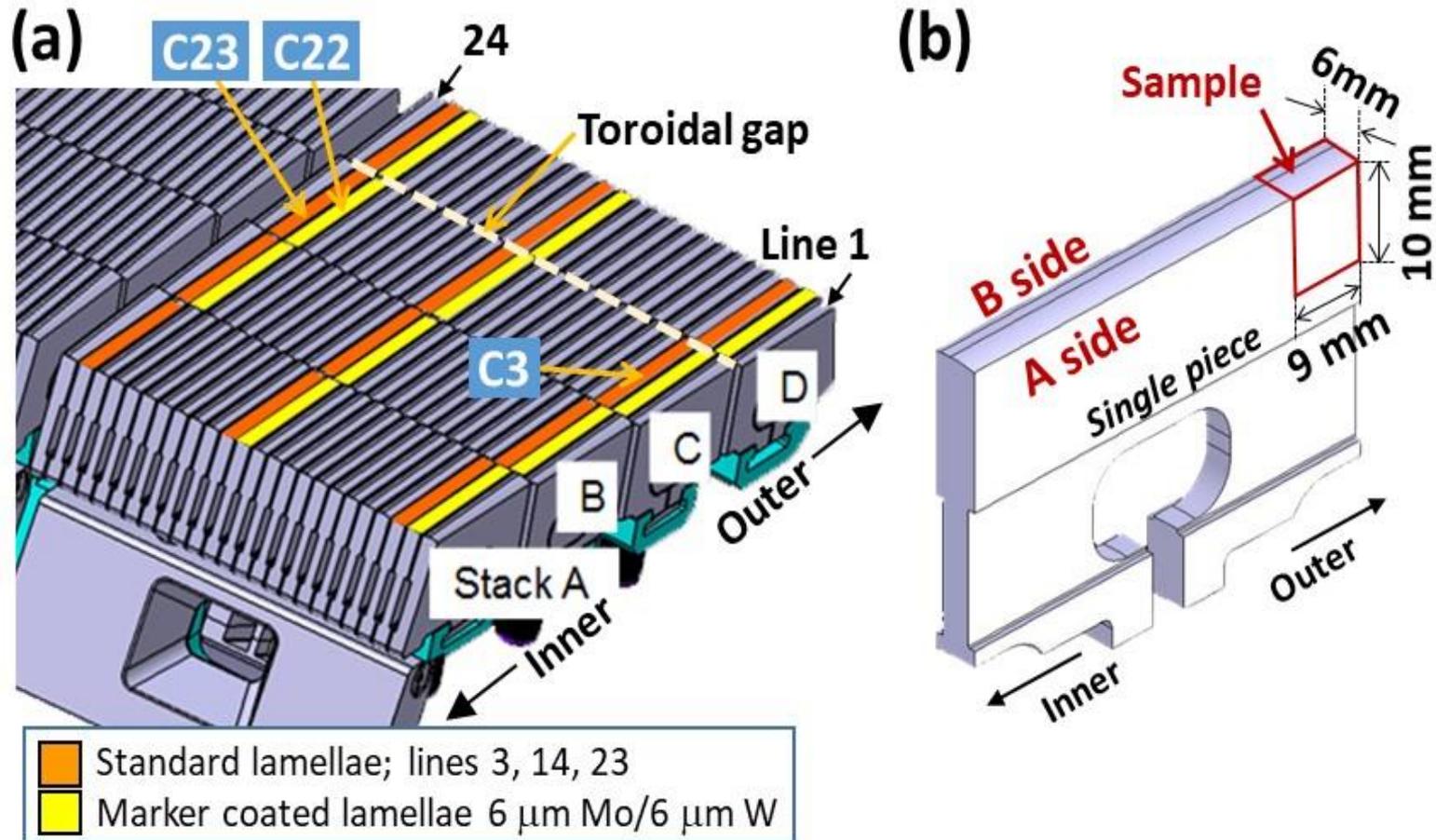
## Concentration (at.%)

<b>D</b>	6.0	4.6	7.4
<b>C</b>	14.6	7.7	8.3

## Messages:

- *More or less constant deposition rate of D.*
- *Slowly varying deposition rate of Be.*
- *Decreasing deposition rate of C from ILW-1 to ILW-3.*

# Divertor: Tile 5, Bulk W lamellae



*Plasma operation mainly on Stack C and D.*

*Lamellae 1-3: in the shadow of the adjacent tile*

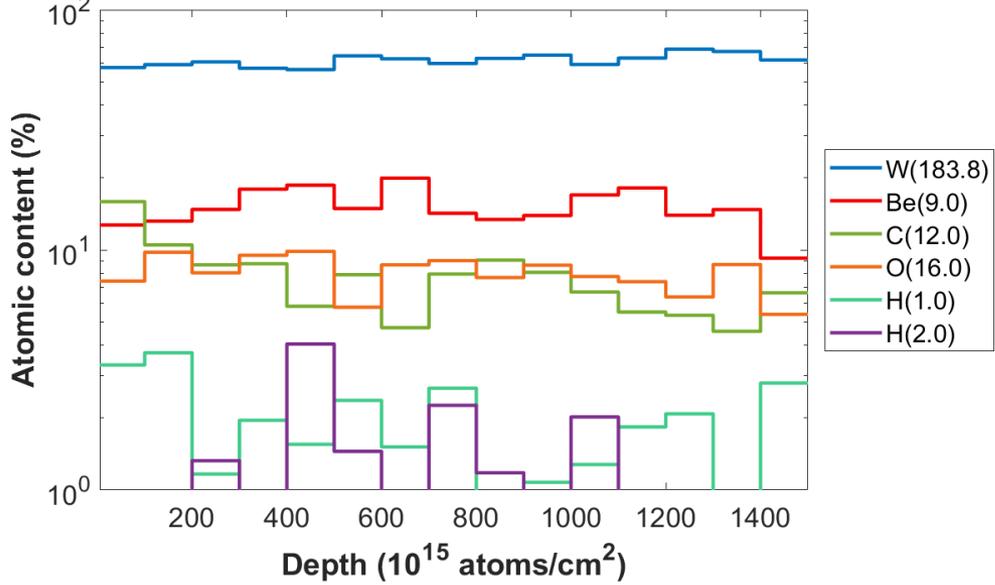
*Lamellae 12-14: central part of the assembly*

*Lamellae 20-24: most exposed*

# Divertor, W bulk lamellae: Depth profiling with ERDA



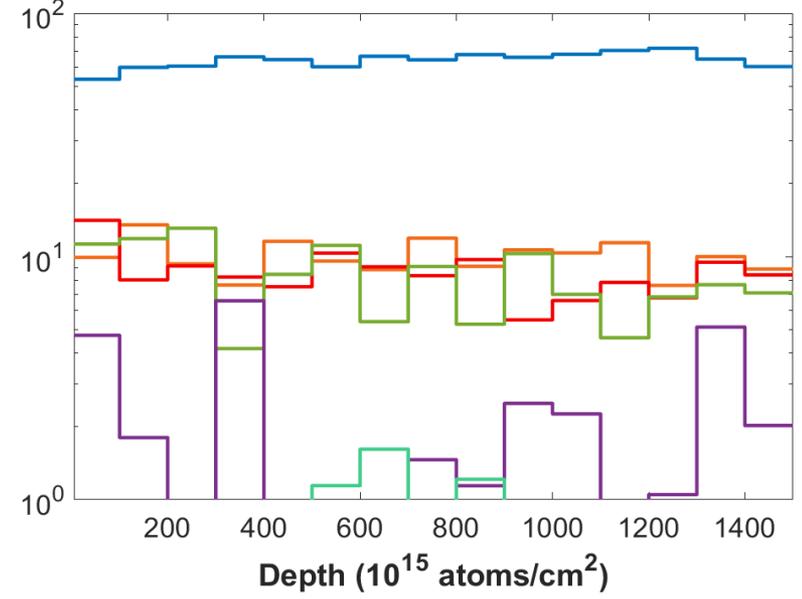
## ILW-2, lamella C3 (sample 55)



Deuterium content:  $1.7 \times 10^{16} \frac{\text{at.}}{\text{cm}^2}$   
 Hydrogen content:  $3.2 \times 10^{16} \frac{\text{at.}}{\text{cm}^2}$

**More Hydrogen than Deuterium**  
*(End of ILW-2 campaign in hydrogen.)*

## ILW 1+3, Lamella C2 (sample 104)



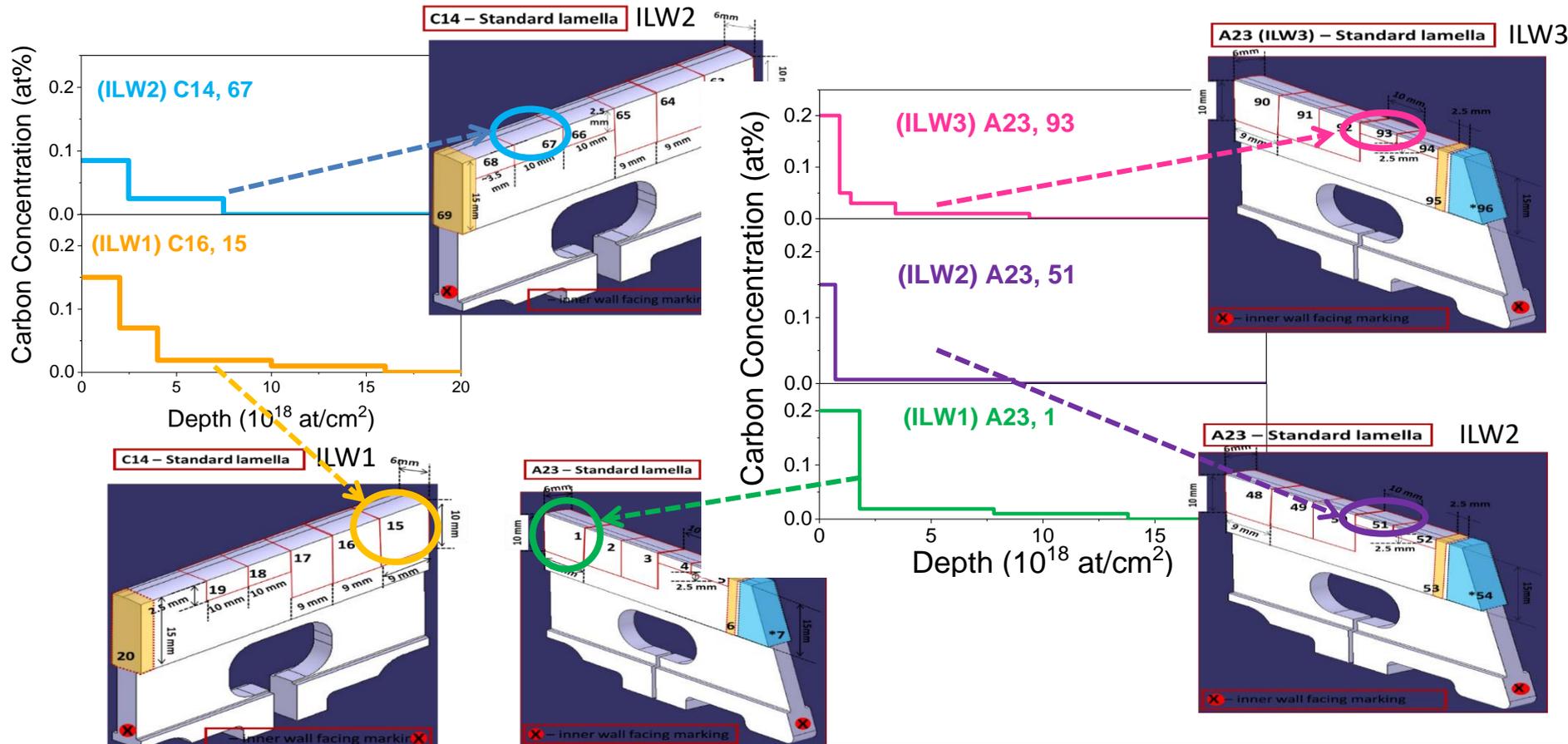
Deuterium:  $3.1 \times 10^{16} \frac{\text{at.}}{\text{cm}^2}$   
 Hydrogen:  $1.1 \times 10^{16} \frac{\text{at.}}{\text{cm}^2}$

**More Deuterium than Hydrogen**

### Other species, contents in $10^{16} \text{ cm}^{-2}$ :

	ILW 2	ILW 1+3
<b>Be</b>	24	14
<b>C</b>	14	13
<b>N</b>	6.5	3.4
<b>O</b>	13	17
<b>Ni</b>	--	4

# Carbon on W lamellae: Deuterium NRA on top surfaces

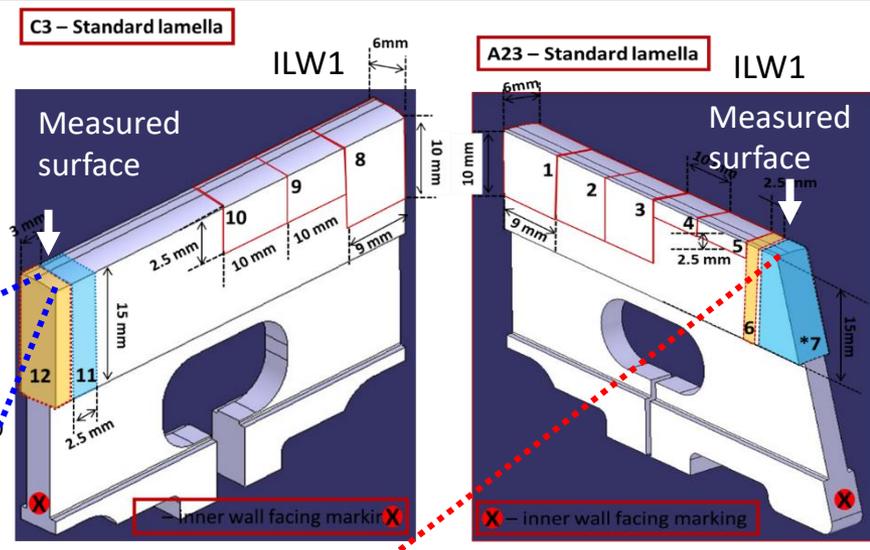
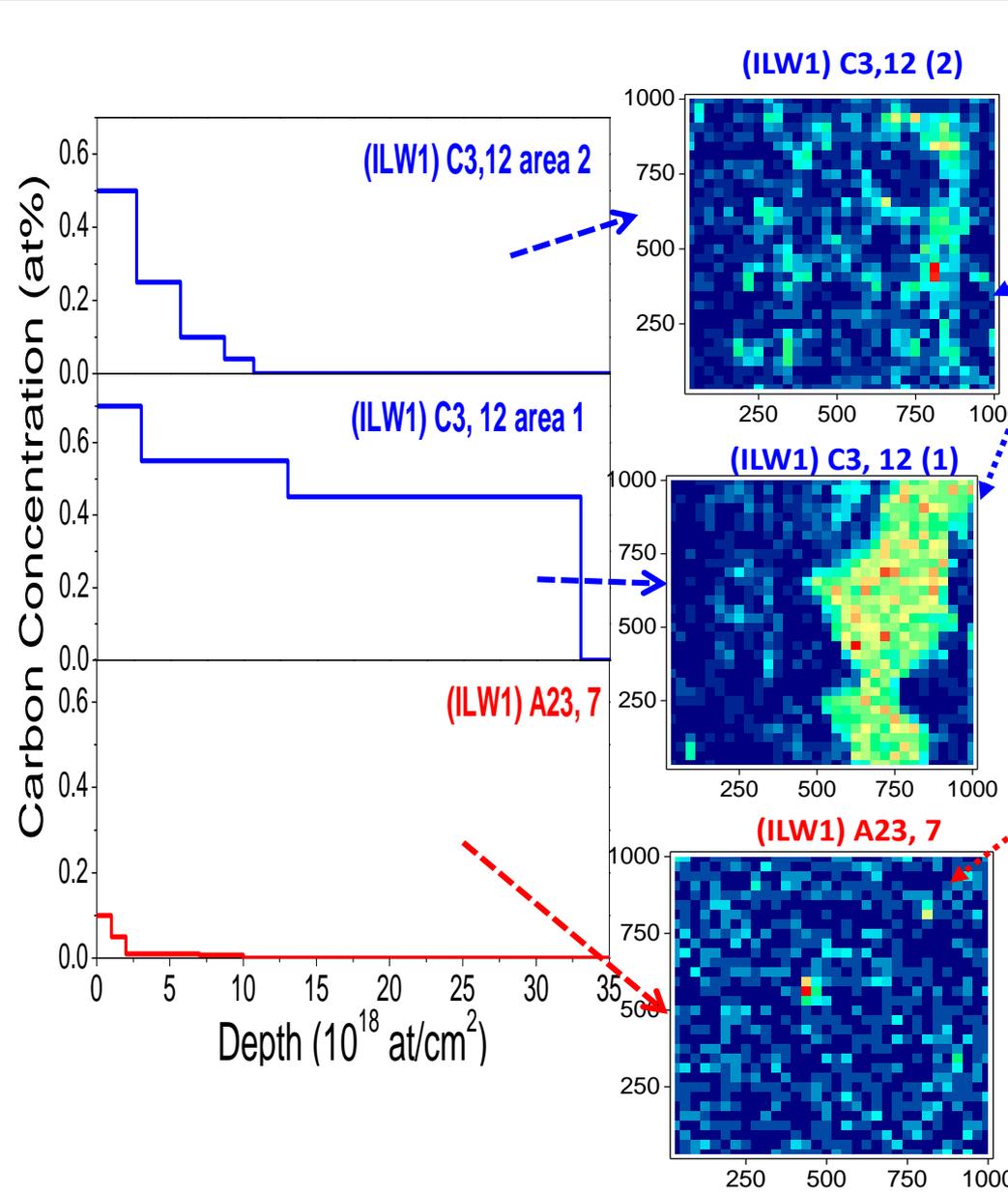


Lamella	Experiment Period	Sample No	Carbon ( $10^{17}$ at/cm <sup>2</sup> )	Deposition thickness ( $10^{18}$ at/cm <sup>2</sup> )
A23	ILW 3	93	3.25	9.4
A23	ILW 2	51	1.53	8.7
A23	ILW 1	1	5.34	13.8
C14	ILW 2	67	3.38	7.5
C14	ILW1	15	6.14	16

## Messages

- The highest **carbon** deposition with the largest thickness is observed in ILW-1 samples.
- Central lamella in ILW1 presents the highest **carbon** amount and larger thickness overall

# Carbon on W lamellae: D micro-beam NRA on top surfaces



Lamella	Exp. Period	Sample	C Amount ( $10^{17}$ at/cm <sup>2</sup> )	Deposition Thickness ( $10^{18}$ at/cm <sup>2</sup> )
C3	ILW-1	12 (2)	24.8	10.7
C3	ILW-1	12 (1)	166	33
A23	ILW-1	7	2.21	10

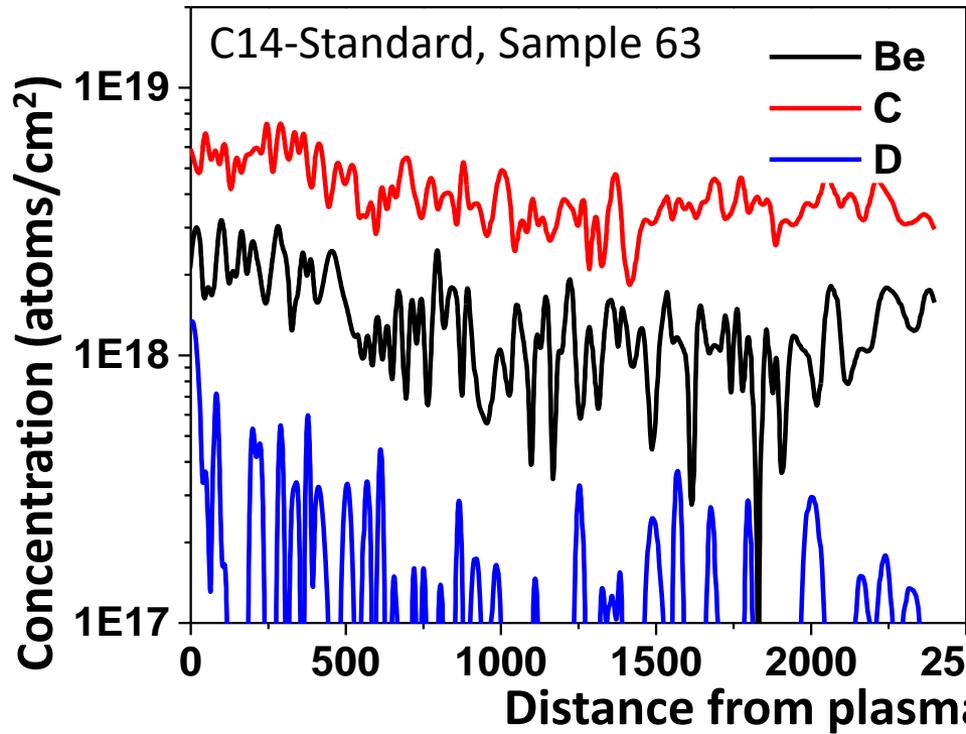
## Messages

- C3,12 during ILW1 presents stripe-like C deposition having a width varying between  $\sim 100 \mu\text{m}$  (area 2) and  $\sim 250 \mu\text{m}$  (area 1) and 2 to 3 orders higher C amount than other W lamella.
- Carbon agglomerates with diameter of  $\sim 50 \mu\text{m}$  in A23,7

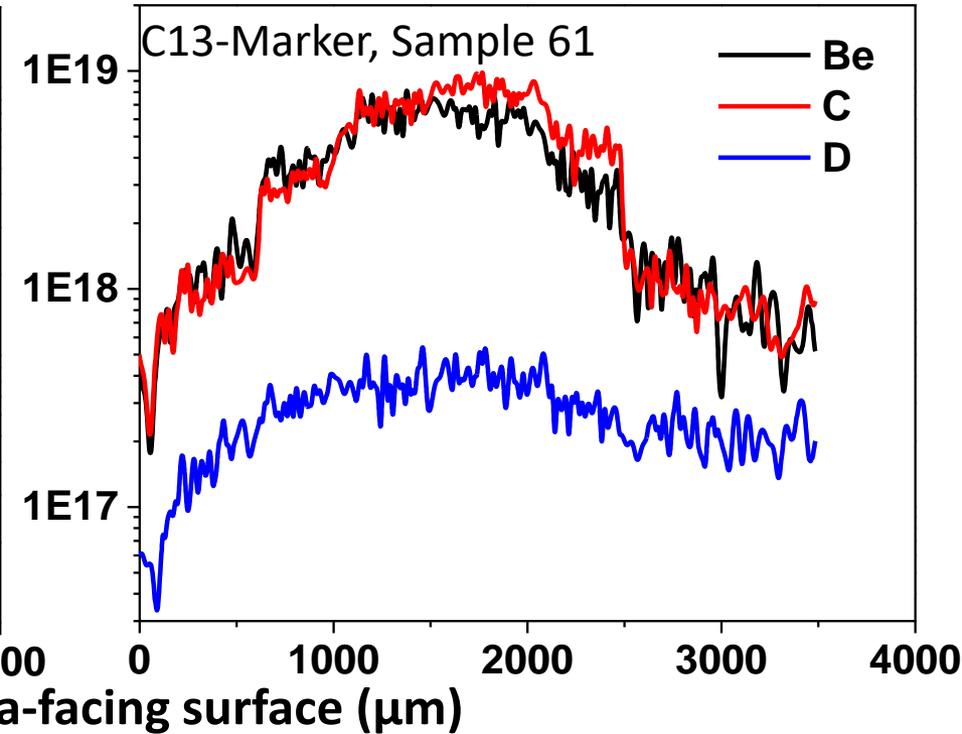
# W lamellae ILW-2: Deposition in gaps, $\mu$ -NRA



0.4 mm wide gap to another lamella

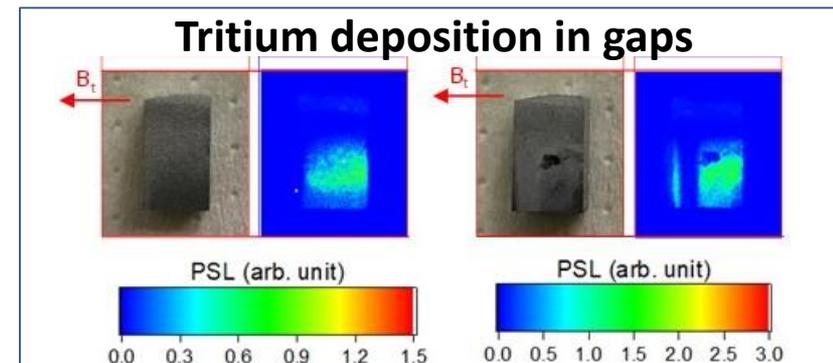


1.5 mm wide gap to Stack D

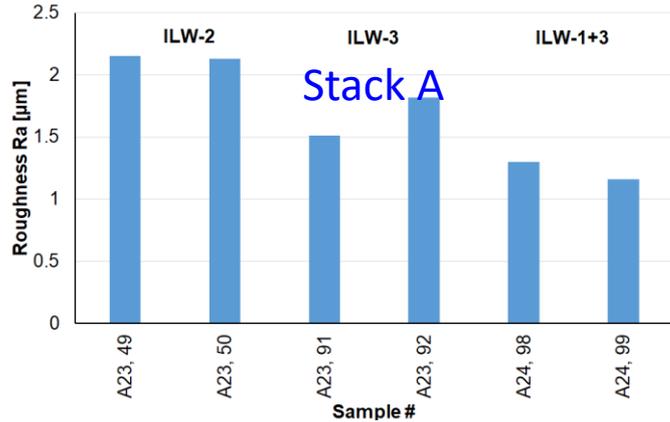


## Messages:

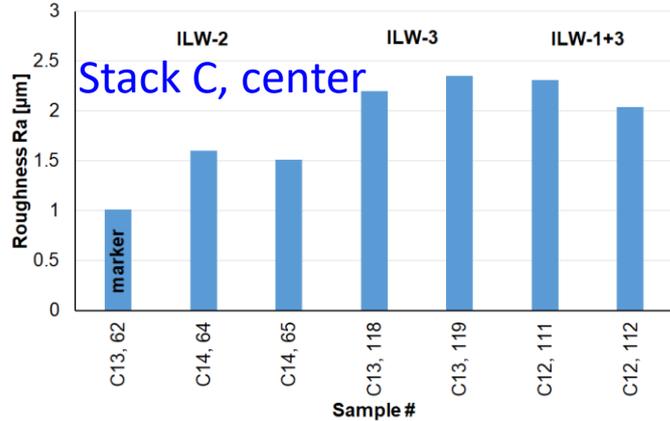
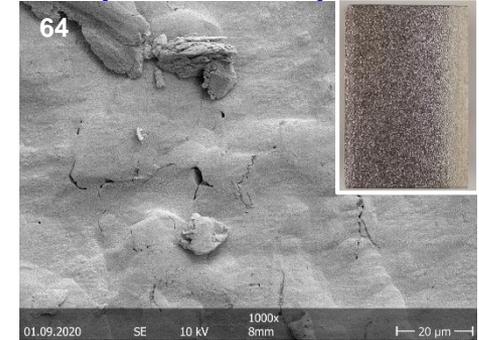
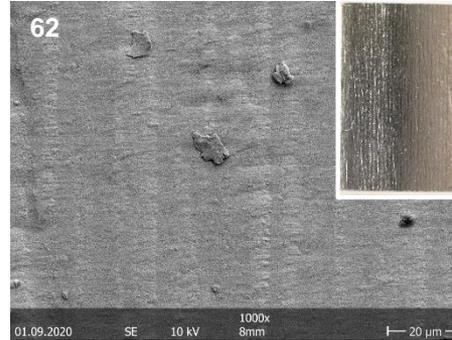
- Deposition increases with the gap width.
- Relatively high C content,  $C/Be > 1$ .
- Coherence with T deposition pattern in gaps between stacks.



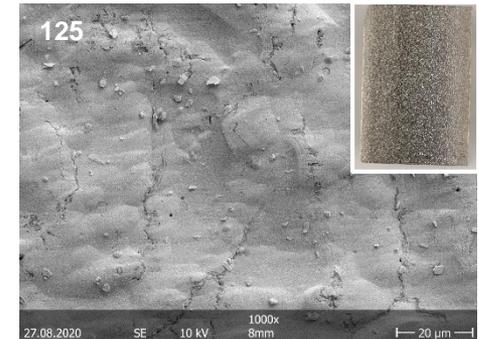
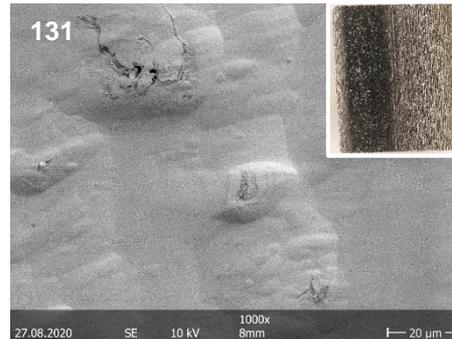
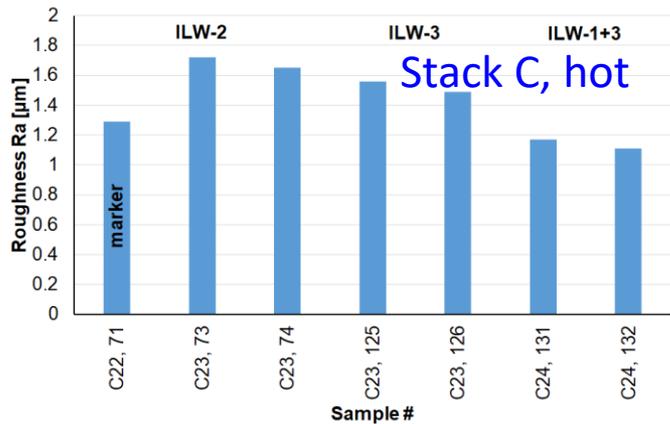
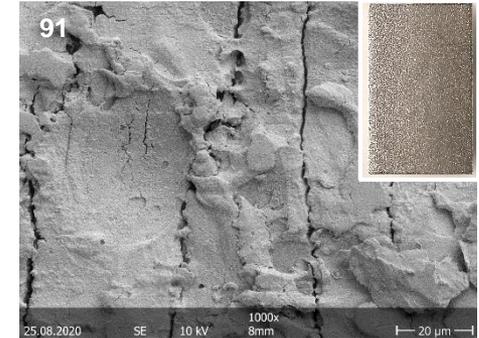
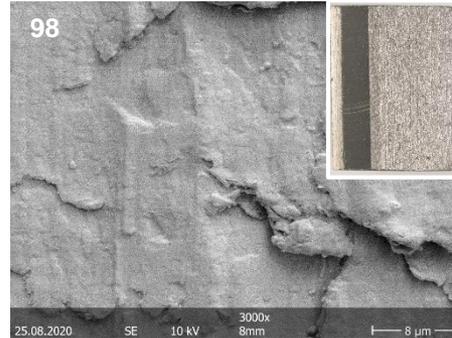
# Tungsten lamellae: ILW-2, ILW-3, ILW-1+3



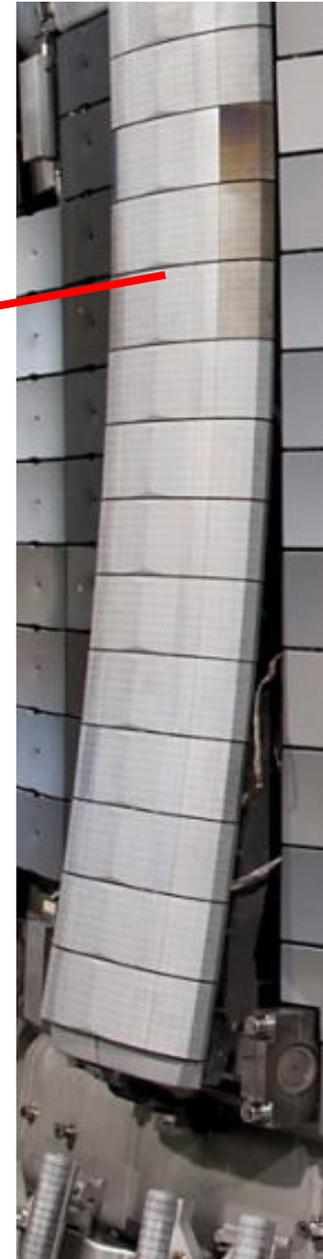
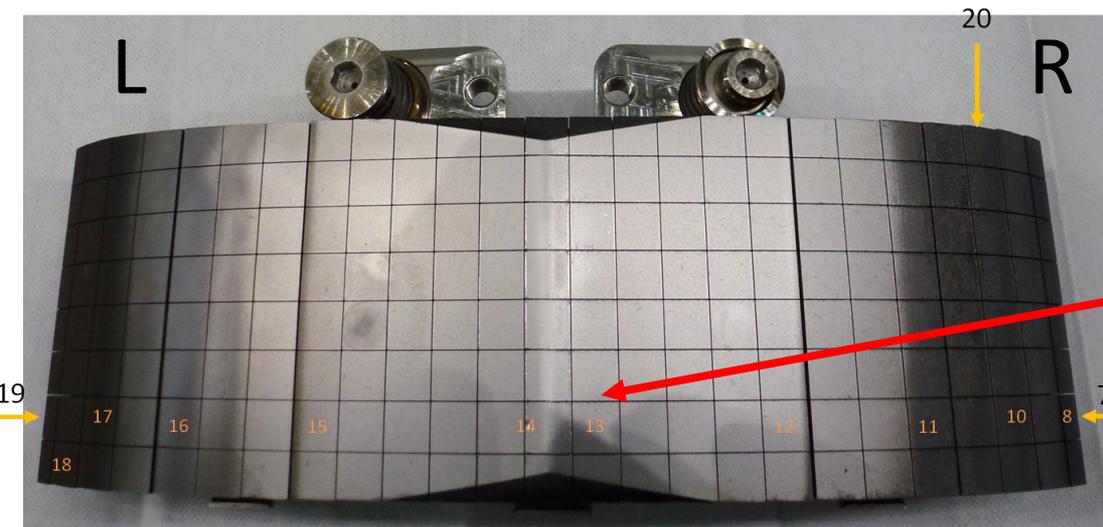
- ❖ Reduced surface roughness for marker lamellae.
- ❖ In some locations marker layer was fully eroded.



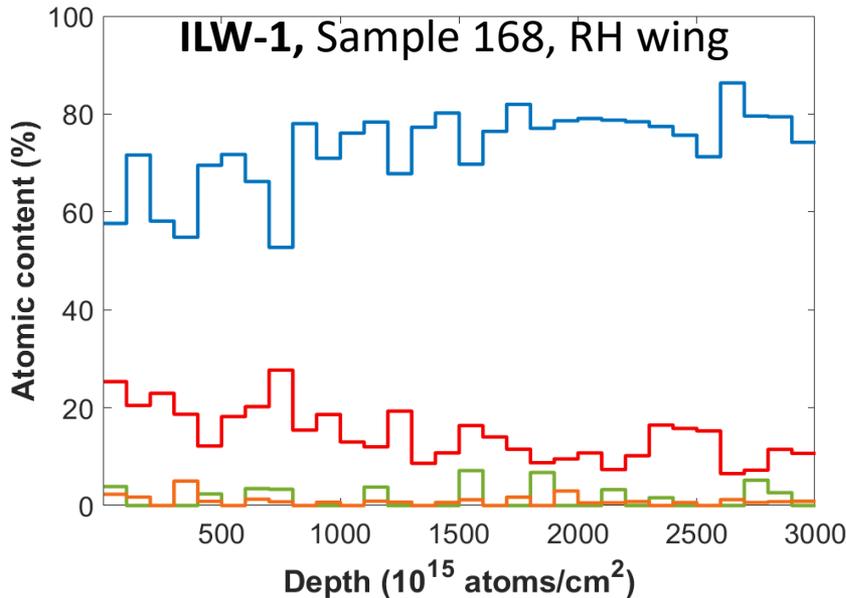
- ❖ Reduced surface roughness for ILW-1+3



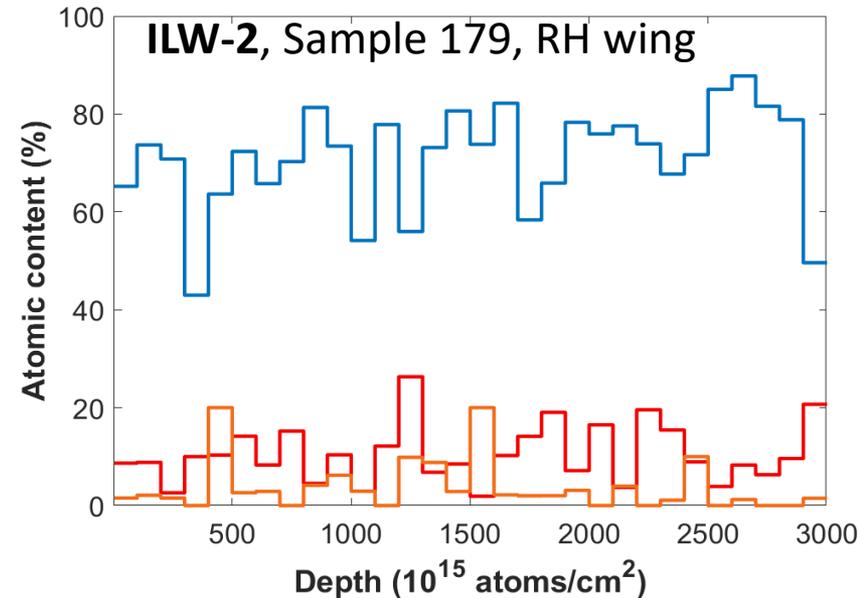
# Beryllium limiters



# Beryllium Inner Wall Guard Limiters: ILW-1 and ILW-2



Deuterium content:  $4.8 \times 10^{16} \frac{\text{at.}}{\text{cm}^2}$   
 Hydrogen content:  $2.7 \times 10^{16} \frac{\text{at.}}{\text{cm}^2}$



Hydrogen content:  $1.2 \times 10^{17} \frac{\text{at.}}{\text{cm}^2}$

**No Deuterium on ILW-2**  
*(The result of ILW-2 campaign finish in H)*

**Other species, contents in  $10^{16} \text{ cm}^{-2}$ :**

	ILW-1	ILW-2
<b>Be</b>	226	220
<b>C</b>	14	14
<b>N</b>	5	11
<b>O</b>	45	32
<b>Ni</b>	10	3
<b>W</b>	traces at the detection limit.	

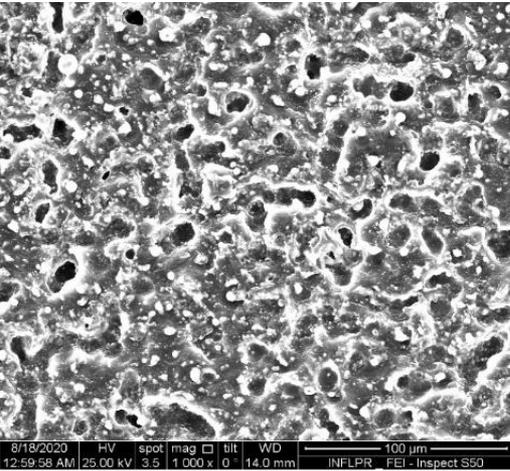
# Beryllium limiters: SEM/EDX



IWGL 2XR11 – sample 674

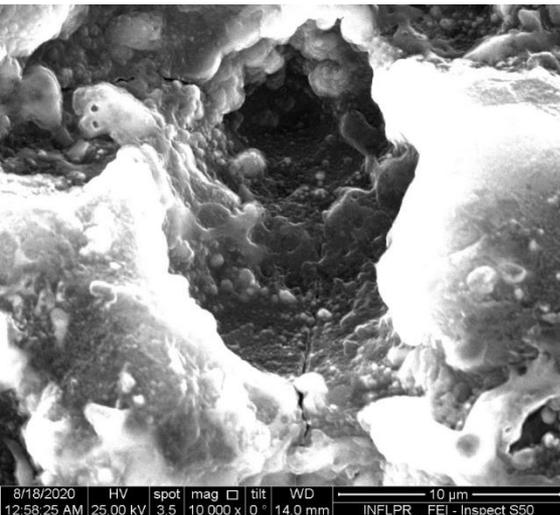
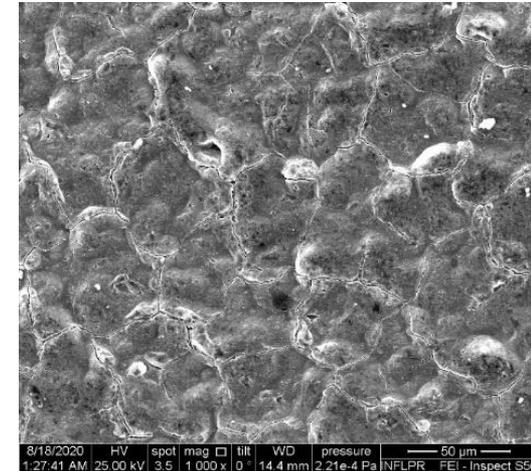
WOPL 4D15 – sample 754

Impurities from EDX measurements:

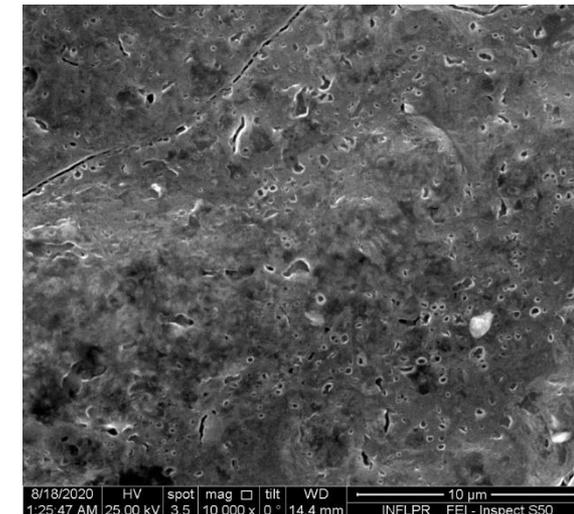


Element	At. %
O	55.34
Cr	3.17
Fe	4.84
Ni	12.73
Cu	5.71
W	3.18

Element	At. %
O	68.39
Cr	2.81
Fe	4.57
Ni	11.16
Cu	5.16
W	3.72



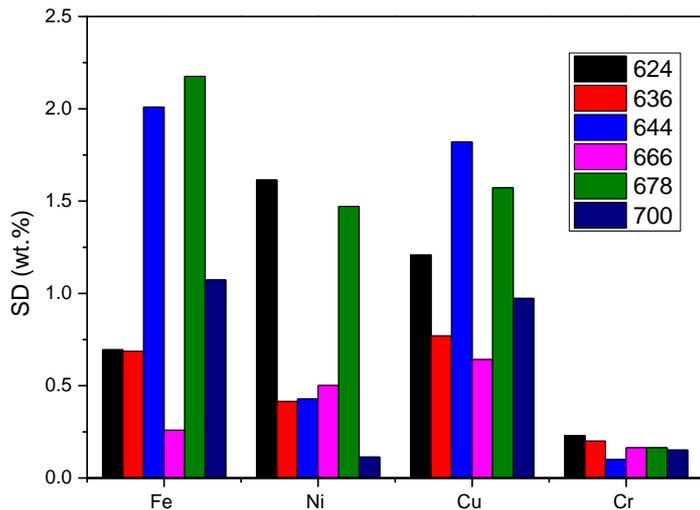
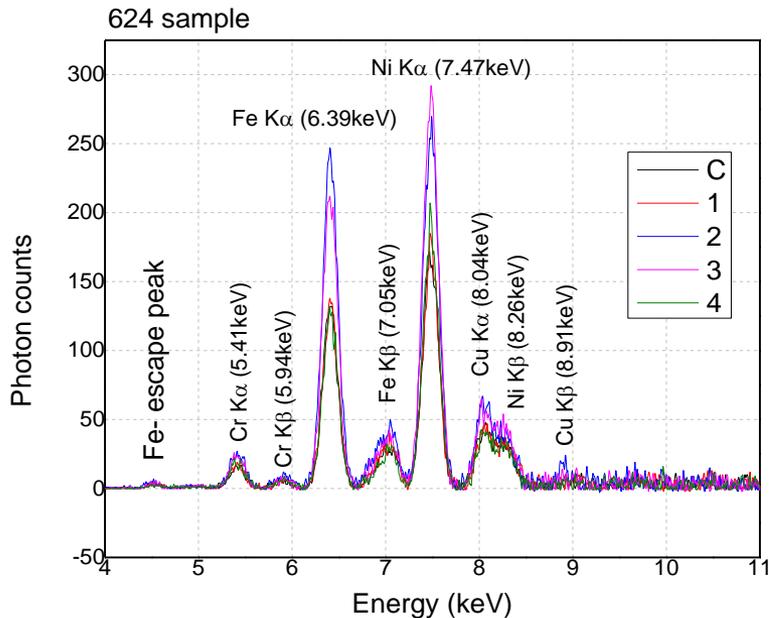
C. Lungu, C. Porosnicu, IAP, Romania



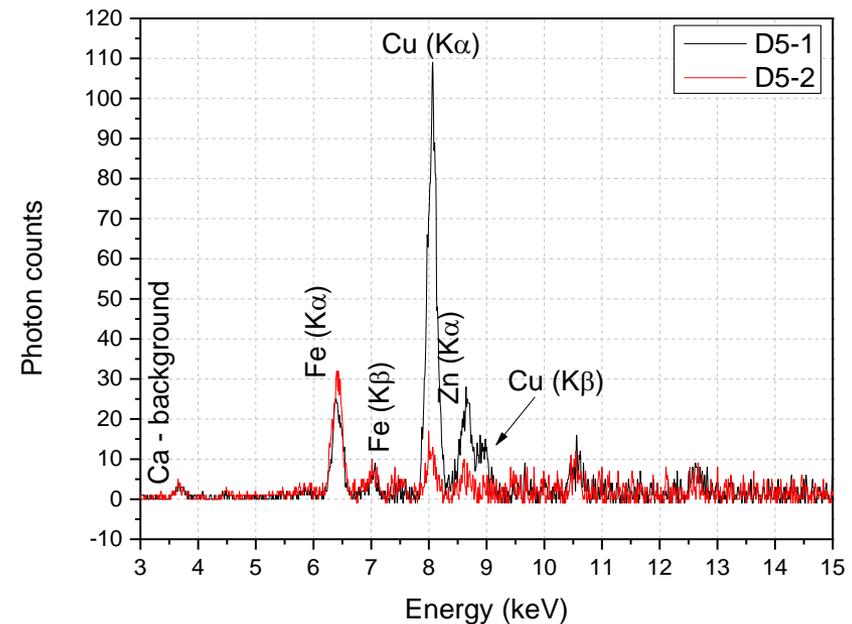
# $\mu$ XRF in Be limiter castellations and on dust



## $\mu$ XRF on Be limiters



## $\mu$ XRF/XCT on dust



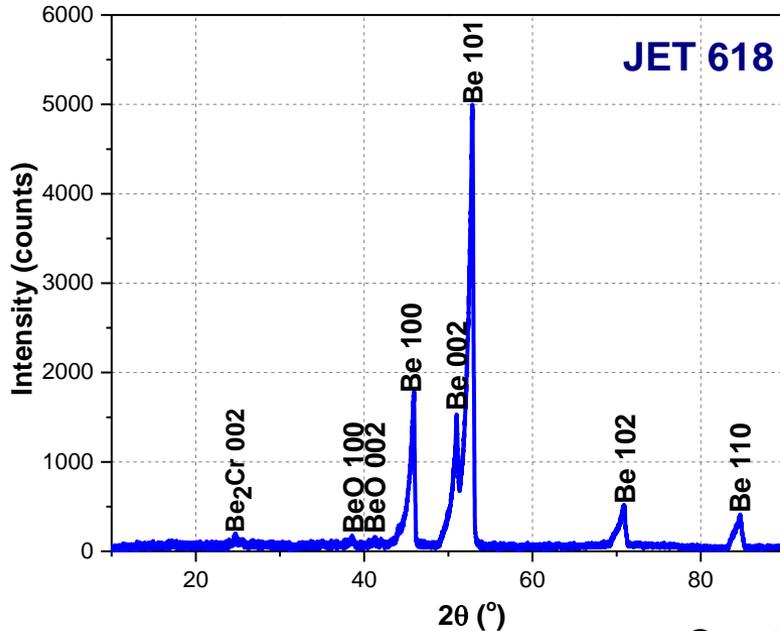
### Composition of inclusions in dust particles (D5)

Element	D5-1 (Wt.%)	D5-2 (Wt.%)
Fe	13.8+1.2	15.5+1.3
Cu	70.4+3.1	69.4+3.1
Zn	15.7+1.6	15.1+1.6

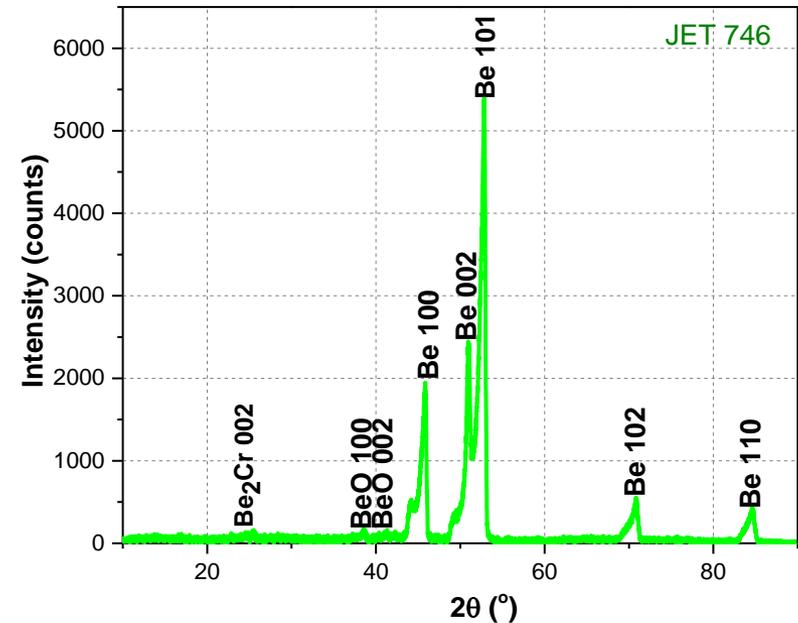
# Be limiters after ILW-3: X-ray diffraction



## Inner Wall Guard: 2XR11



## Outer Poloidal: 4D15



### Crystalline Phases:

➤ Be; BeO (bromellite)

### Grain size measured on Be 101 peak:

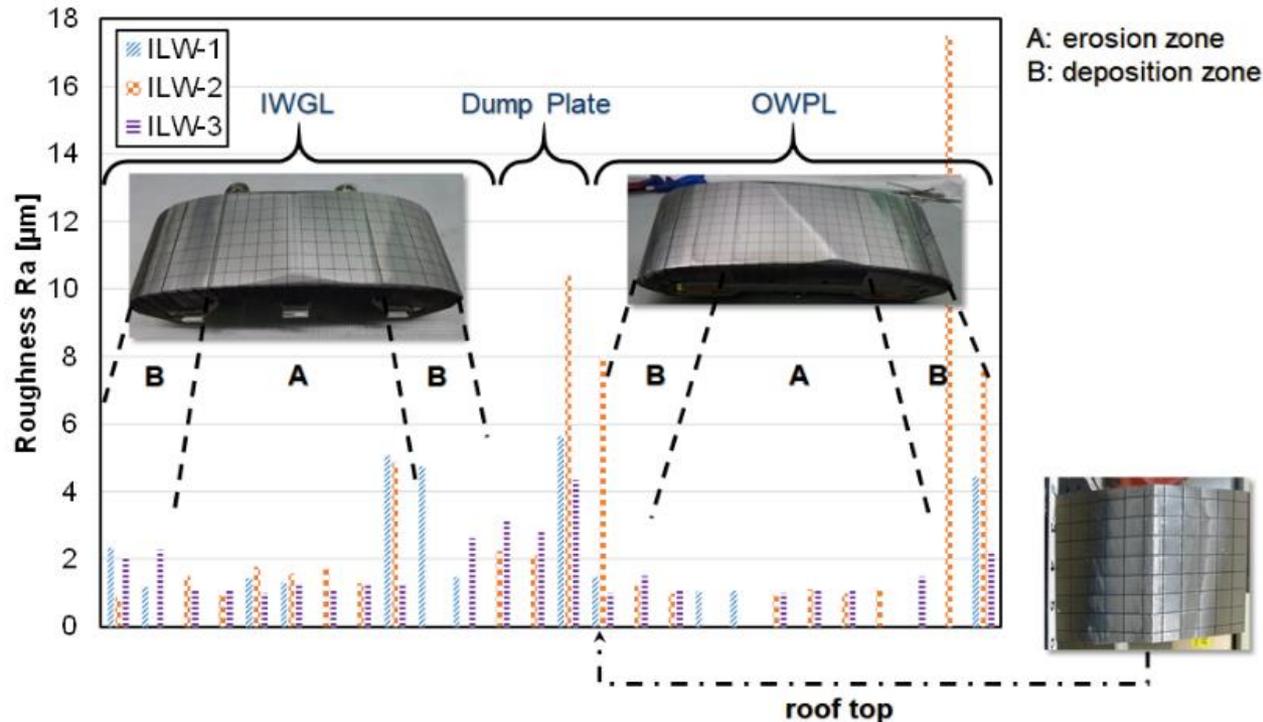
➤ 13.48 nm (618)

➤ 11,83 nm (746)

## Messages:

- No significant changes in crystalline phases observed on all the samples.
- No other peaks corresponding to other elements (impurities) observed.

# Be limiters ILW-1 to ILW-3: Surface Roughness



## Roughness:

- ❖ Enhanced either by melting (dump plate) or in the deposition zones.
- ❖ Hardly any effect for the erosion zones.
- ❖ For ILW-1 and ILW-3 almost identical results.
- ❖ Significantly increased roughness on OWPL, ILW-2.

## Messages

- ❖ In ILW-3 areas with full or partial erosion of the Be top layer.
- ❖ ILW-3: In contrast to ILW-1 and ILW-2 no areas with melting.
- ❖ For the dump plate no relevant erosion nor melting of the roof top was found (specimen 462, 2B2C); 2B4C (not part of this investigation) shows heavy melting.
- ❖ Metallographic results showed more or less full erosion of the marker layer in all the locations, at least in the cross sectional micrographs no Ni-remains were found as it was the case for ILW-1 and ILW-2.

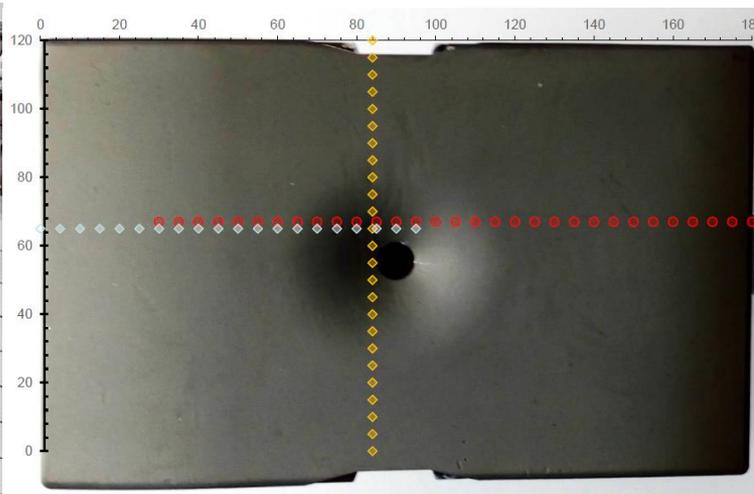


# **Inner Wall Cladding**

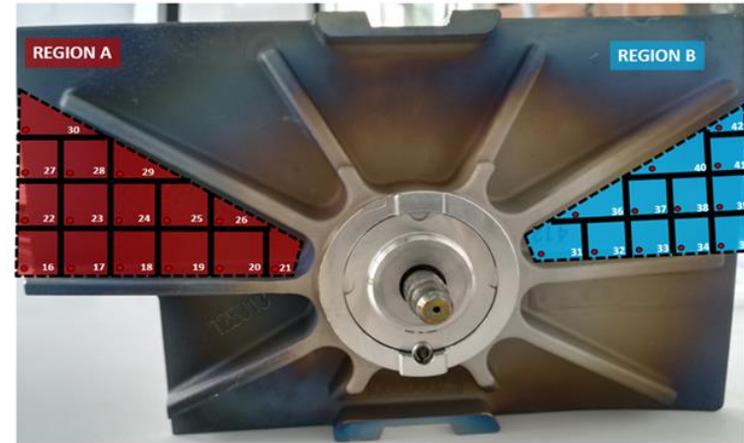
# Inner Wall Cladding



Be  
on  
Inconel



IWC – 412(2011-16) cutting plan



Total IWC surface area:  $\sim 9 \text{ m}^2$

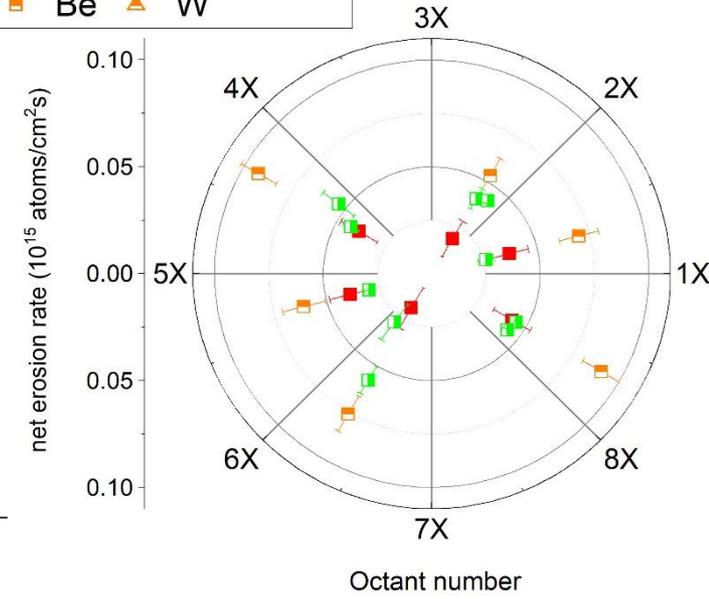
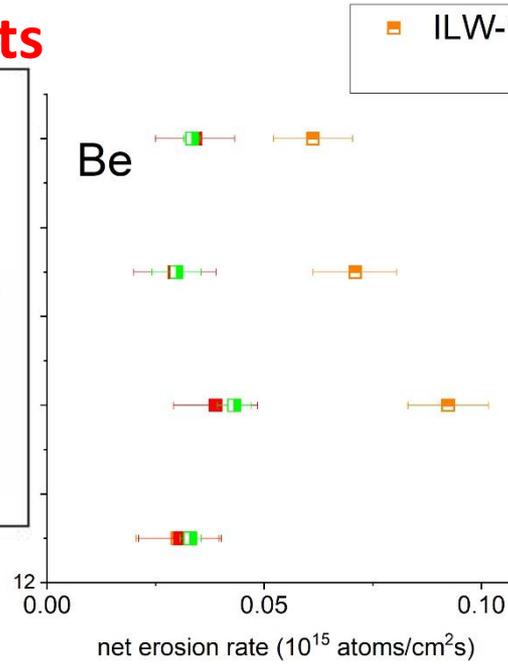
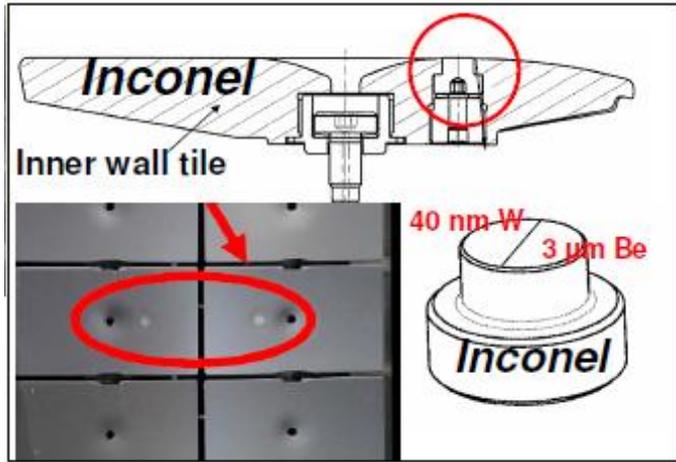
## Main questions:

- *Erosion rate*
- *Fuel content*
- *Impurity content*

# Inner wall erosion: from ILW-1 to ILW-3

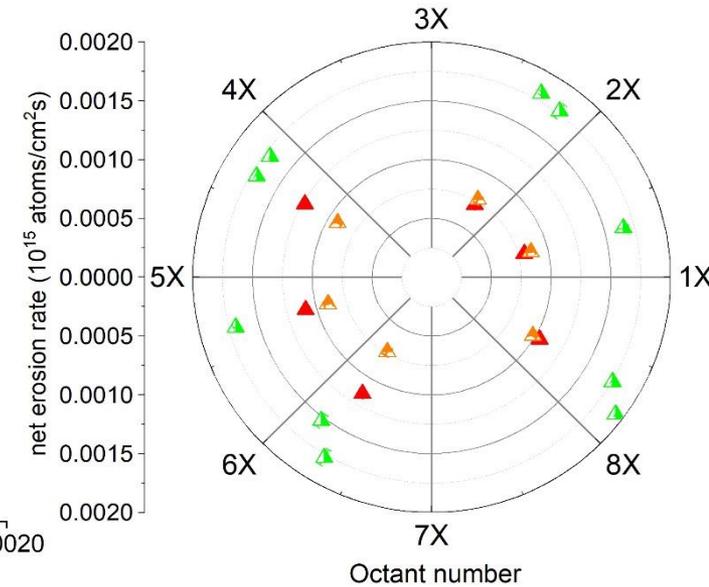
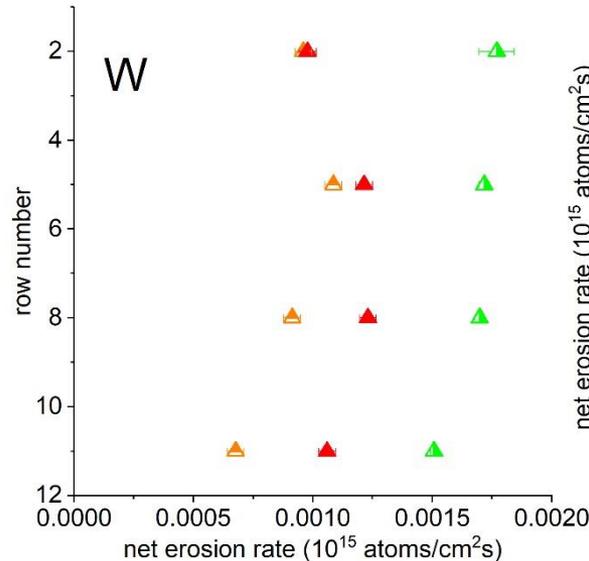


## Data for Be/W Wall Inserts

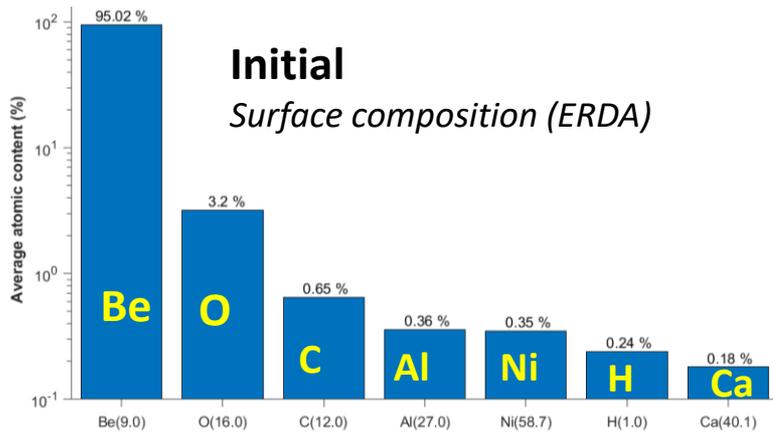


### Messages:

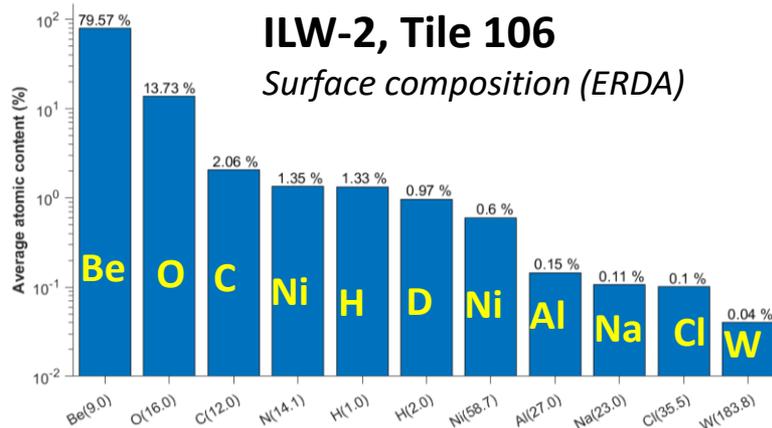
- Fairly homogeneous erosion rate in toroidal and poloidal directions, both for Be & W.
- Variation of erosion rate within factor 2 for all 3 ILW campaigns
- Very small erosion rate for W < 0.3 pm/s



# Inner Wall Cladding: ERDA and <sup>3</sup>He-NRA



The average composition of the sample to a depth of  $6000 \times 10^{15}$  atoms/cm<sup>2</sup>



The average composition of the sample to a depth of  $6000 \times 10^{15}$  atoms/cm<sup>2</sup>

## NRA – whole layer analysis

Sample	Thickness 10 <sup>18</sup> cm <sup>-2</sup> (μm)	Impurity %	D <sub>surface</sub> 10 <sup>17</sup> cm <sup>-2</sup>	D content %
<b>Initial</b>	<b>112 (9.4 μm)</b>	<b>4 – 5</b>	<b>0</b>	<b>0</b>
<b>ILW-2 (106)</b>	<b>82-110 (6.7 – 9.3)</b>	<b>7 - 9</b>	<b>0.9 - 1.7</b>	<b>0.13 – 0.20</b>
<b>ILW 1-3 (412)</b>	<b>125 -149 (10.1 – 12.0 )</b>	<b>8 - 15</b>	<b>3 - 6</b>	<b>0.20 – 0.44</b>

### Messages:

- *Low or no layer erosion.*
- *Probably Be deposition from eroded limiters*
- *Low fuel retention, both at the surface and in the bulk.*
- **Total D inventory: 0.8 - 5.4 x10<sup>21</sup> (3 – 21 mg)**
- *Microscopy is needed to check topography.*

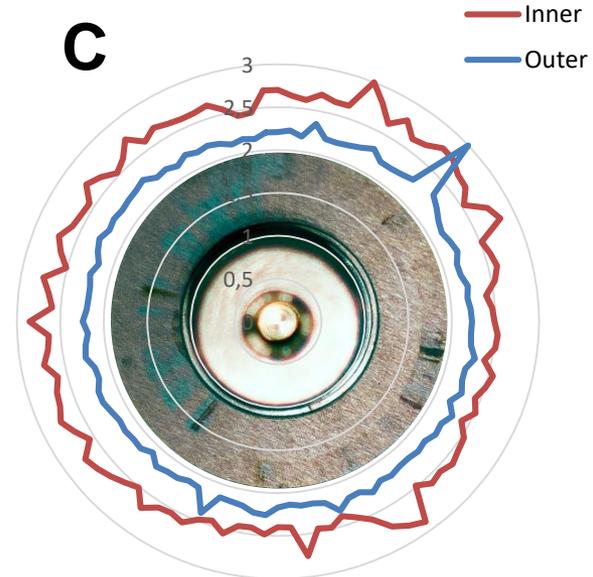
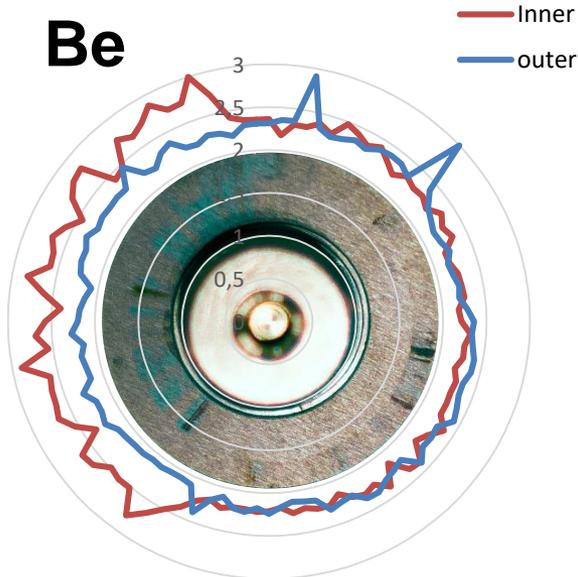
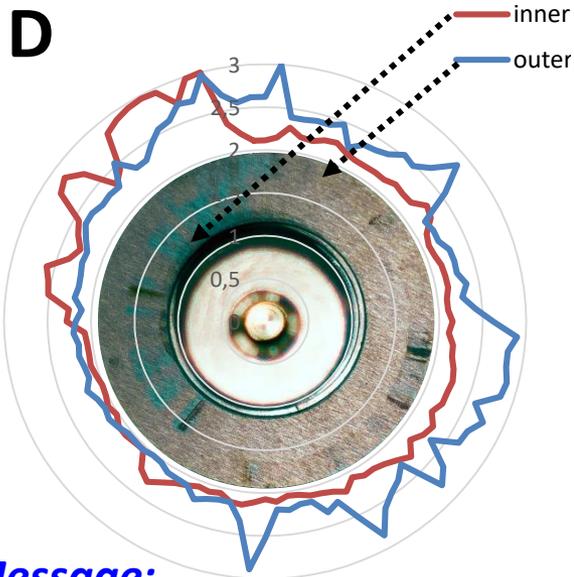
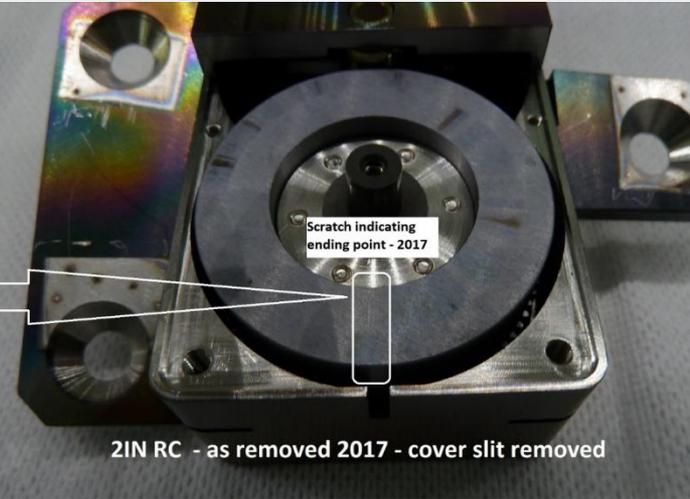
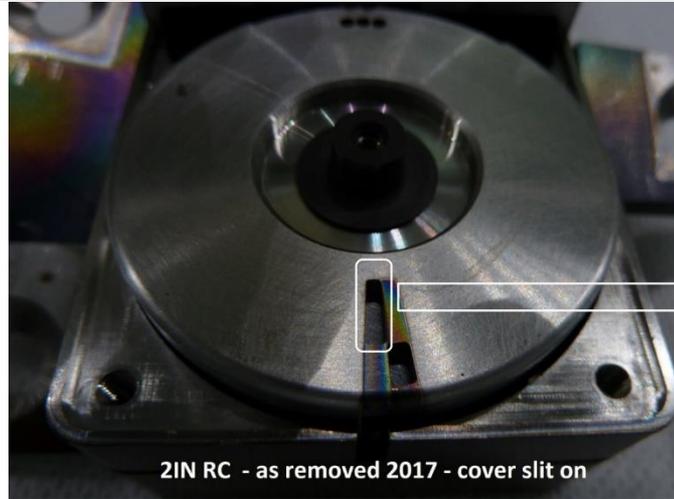


# Wall Probes

# Rotating Collectors



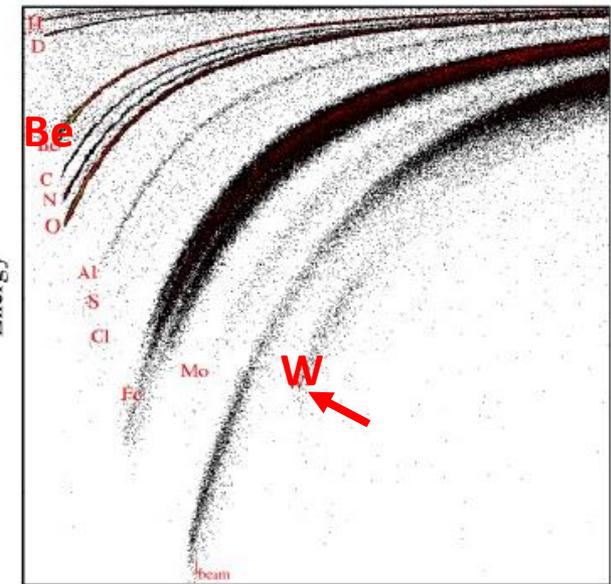
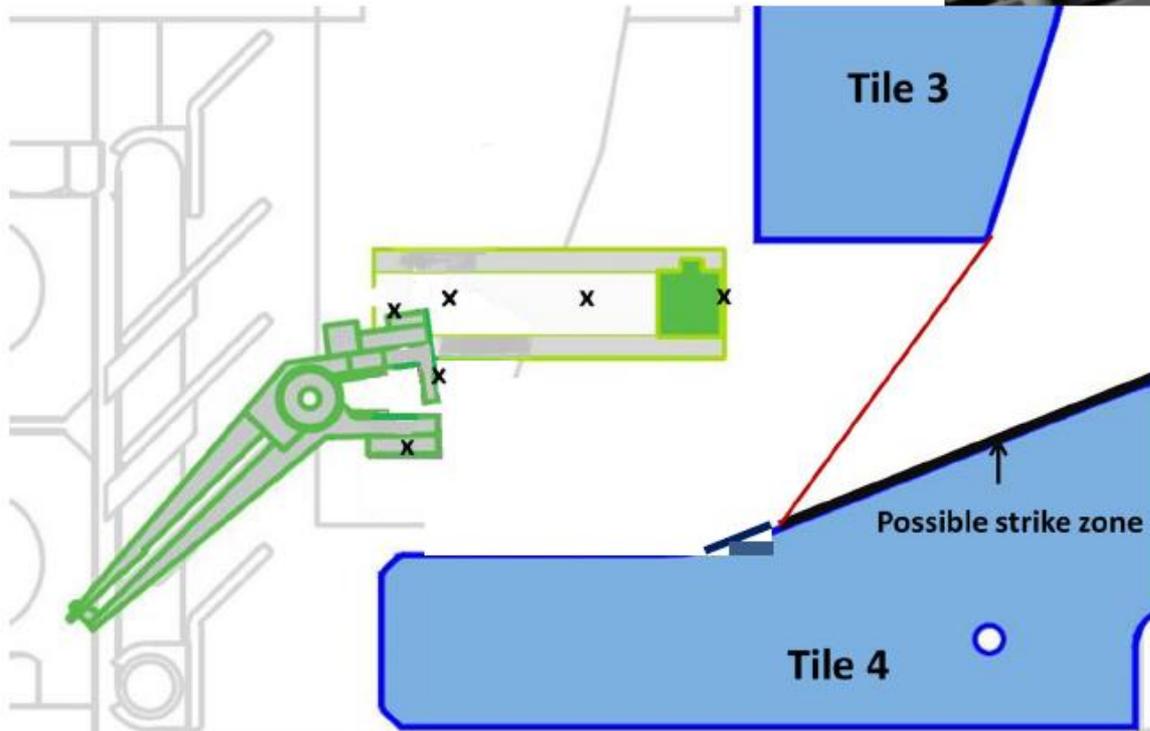
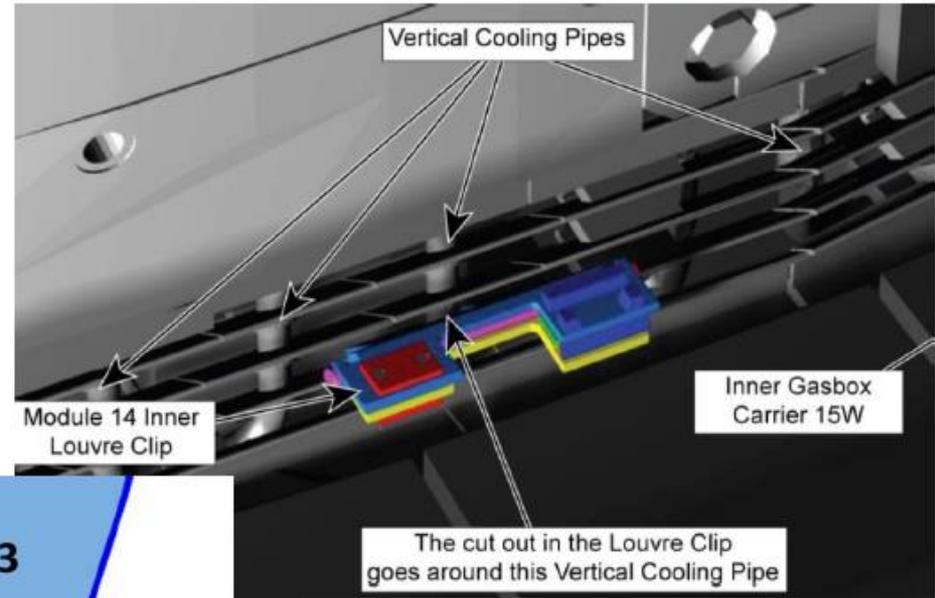
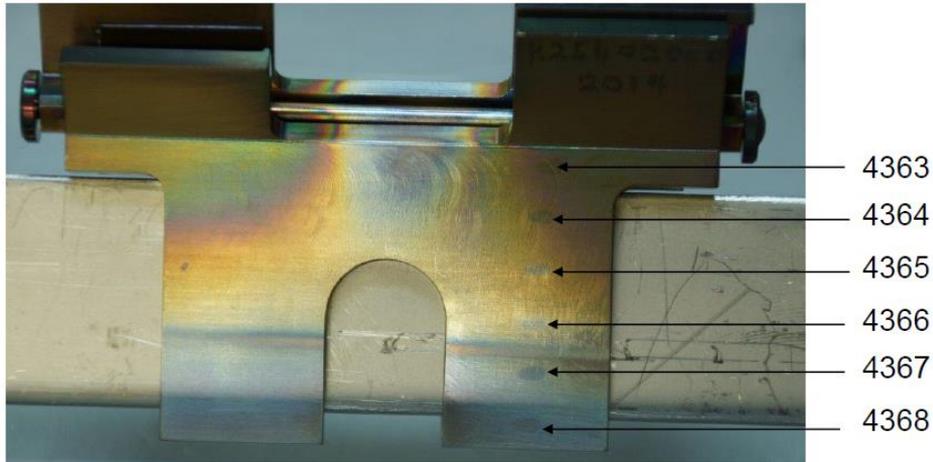
- 2 cycles of exposure.
- Each up to 3000 shots.



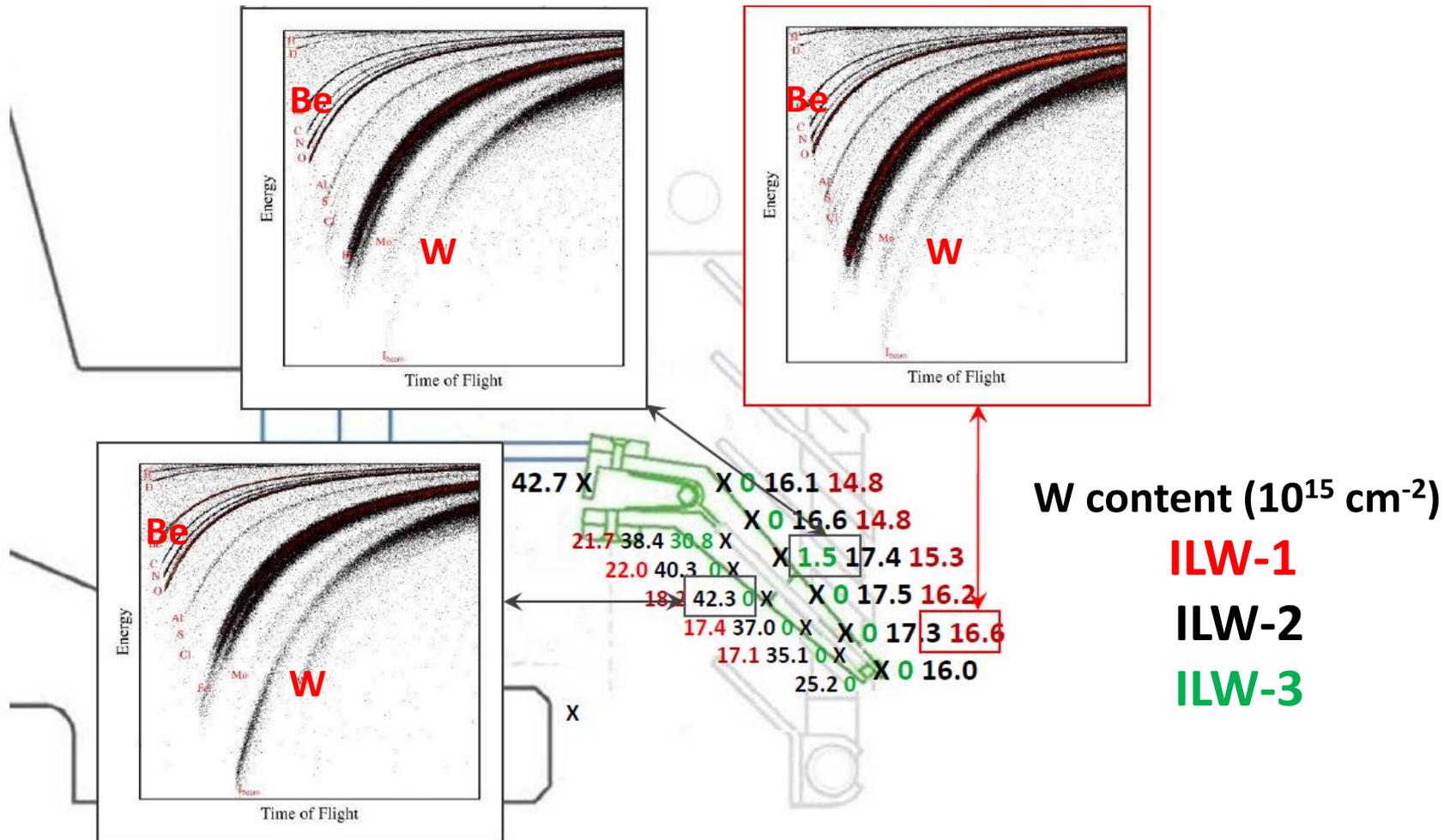
## Message:

- Analyses completed, data ready for modelling/correlation with operation modes.
- Contents of metals are very on the  $10^{15}/\text{cm}^2$  level.

# Louvre clips ILW-1 to ILW-3



# Louvre clips ILW-1 to ILW-3: Transport of Be and W to remote areas



## Message and main question (for modellers)

*How do metals (Be, W) travel to such remote areas, to all surfaces of the clips?*

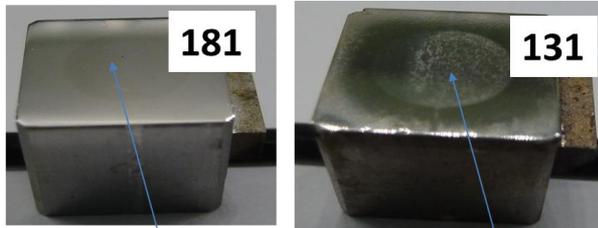


# **Mirrors**

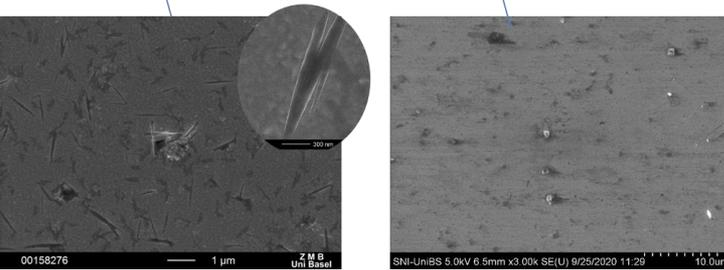
## **Plasma-assisted cleaning**



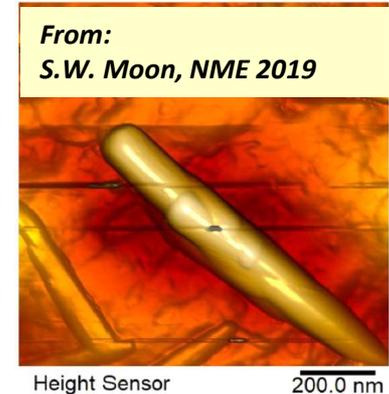
# Plasma-assisted mirror cleaning



SEM before cleaning revealed needle-like structures on JET-ILW mirror exposed in cassette with fins. No needle without fins.

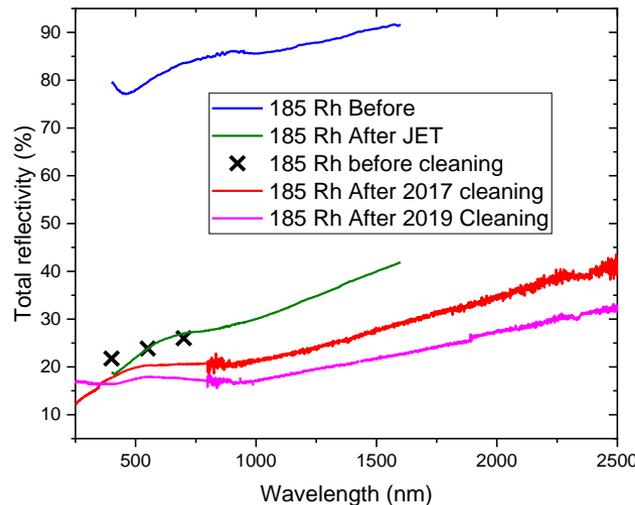


**Aim:**  
To demonstrate cleaning on mirror with needles.



From:  
S.W. Moon, NME 2019

Height Sensor 200.0 nm



220 eV He ions are reducing the amount of Be (393 nm to 37 nm) but not removing the final layer.

XPS shows an enrichment of W oxide that stopped the cleaning.

Adding 3 h in 2019 (in comparison to 23 h for 2017) is not reducing the Be amount as measured by HIERDA.

Reflectivity not recovered for these steps, Ar gas is required.

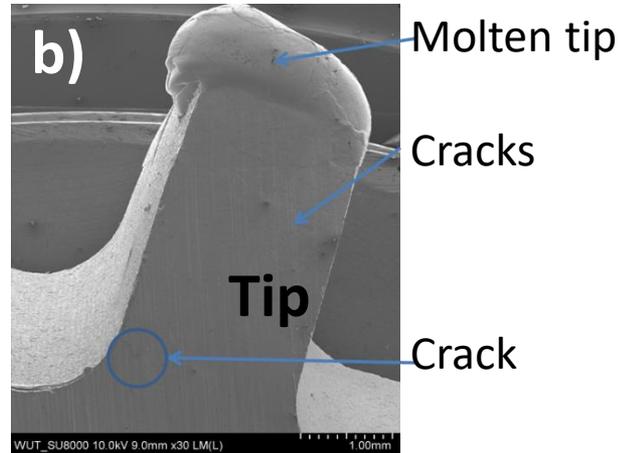
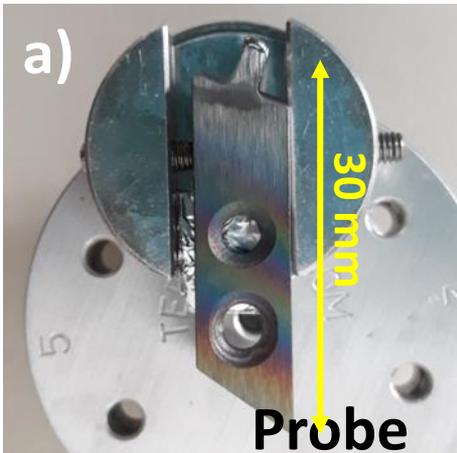
- Further cleaning experiments are delayed due to the COVID situation.
- Next experiments will be performed at the beginning of 2021.



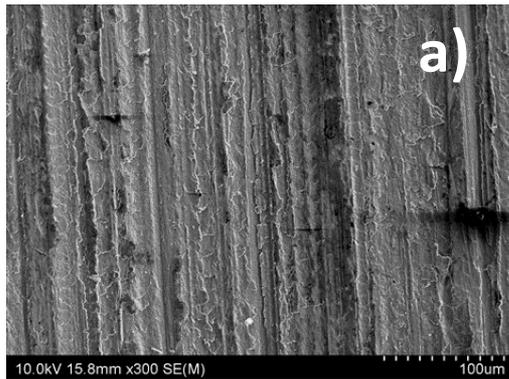
# **Mechanical properties of bulk W**

## **Nanoindentation**

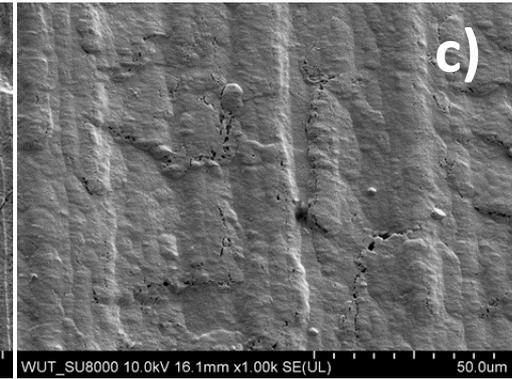
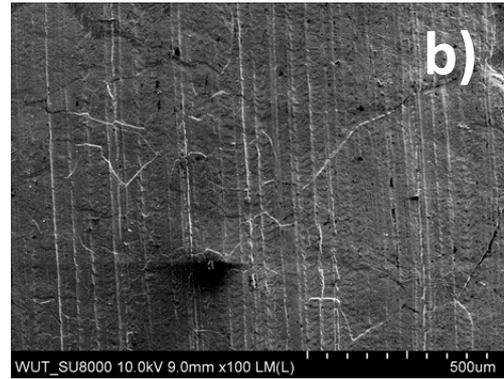
# Langmuir Probe (No. 5, module 16IN, Tile 3, ILW-2)



Areas of measurements



(a) Zone III



(b-c) Tip

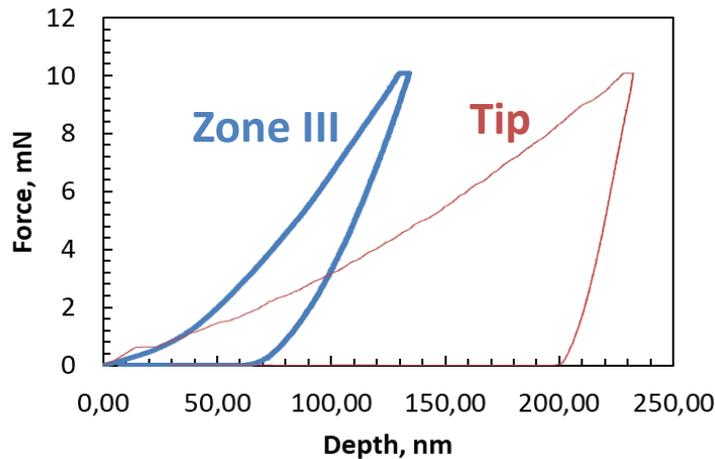
# Langmuir Probe: Nano-indentation



	Young modulus, E, GPa			Hardness, H, GPa		
	Average(E)	SD*(E)	Error (E)	Average(H)	SD(H)	Error (H)
ZONE III	257	42	12	15.37	2.68	0.74
TIP	261	25	6	5.18	0.72	0.19

SD – standard deviation

## Comparison of force-depth curves for both areas.



- *Hysitron Ti-800 triboindenter.*
- *Force 10 mN.*
- *15 measurements in each area.*

- *Due to the large development of the surface, the surface was imaged in the SPM (scanning probe microscope) mode before each measurement.*
- *Large differences in the hardness between the tip and Zone III.*



**Dust**

# Dust

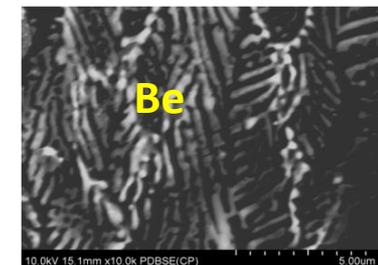
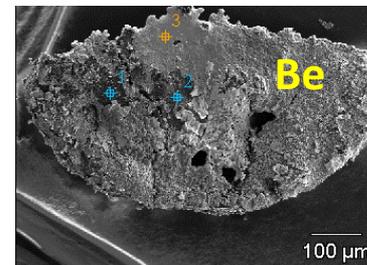
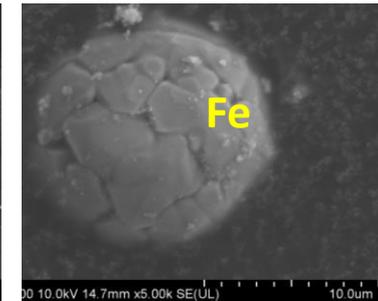
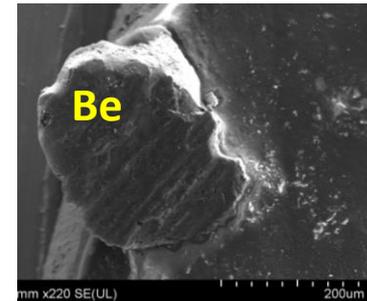
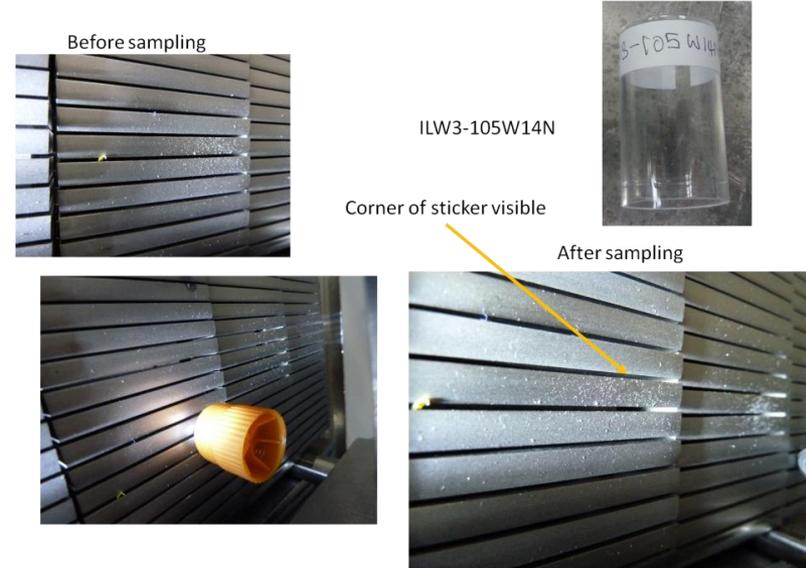


Types/Locations	Methods
Vacuum cleaning ILW - all	Optical microscopy
Sticky pads: Tiles 0, 1	SEM
Sticky pads: Tile 5	FIB
Sticky pads: Tile 8	TEM, STEM
Sticky pads: Tiles B, C	EDX, WDX, EPMA
Sticky pads: Be IWGL	$\mu$ XRF/XCT
Dust monitors: ILW-2 and 3	$\mu$ -NRA
Mirrors (FMT)	$\mu$ -PIXE
KY-6 Mirror ILW-1 and ILW-2	$\mu$ -RBS
Spatial blocks	HIERDA (dust monitors)
QMB crystals	ICP-OES
	TDS: H, D, T
	Full combustion: T
	Radiography: T
	Cameras (in discharges)

## Messages:

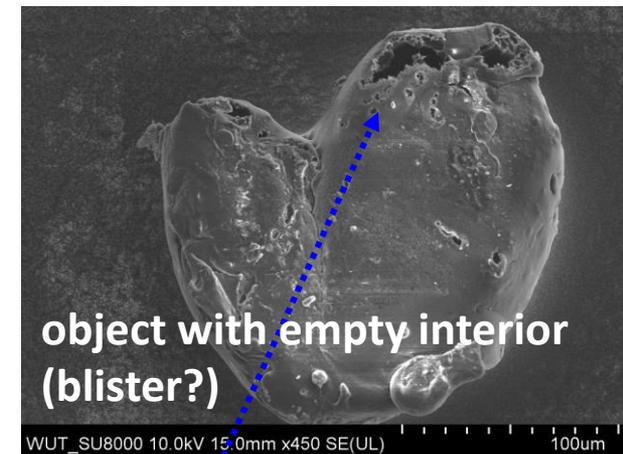
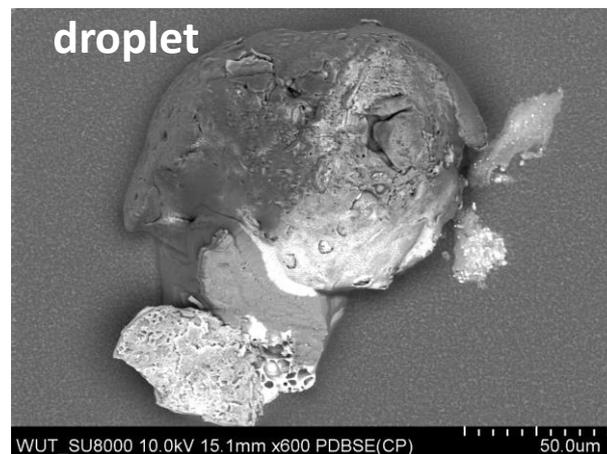
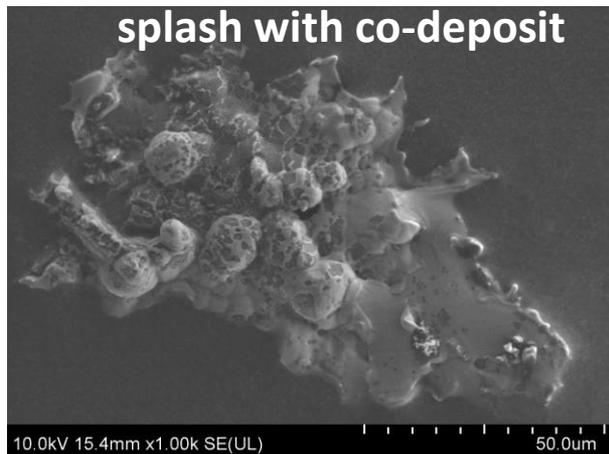
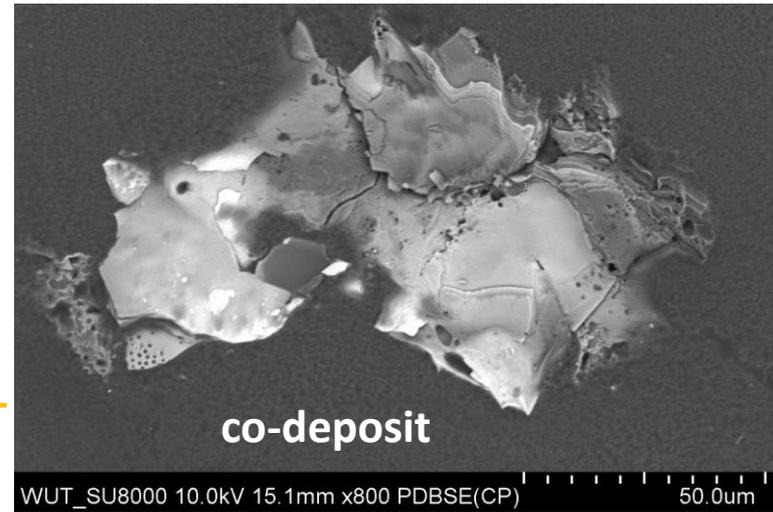
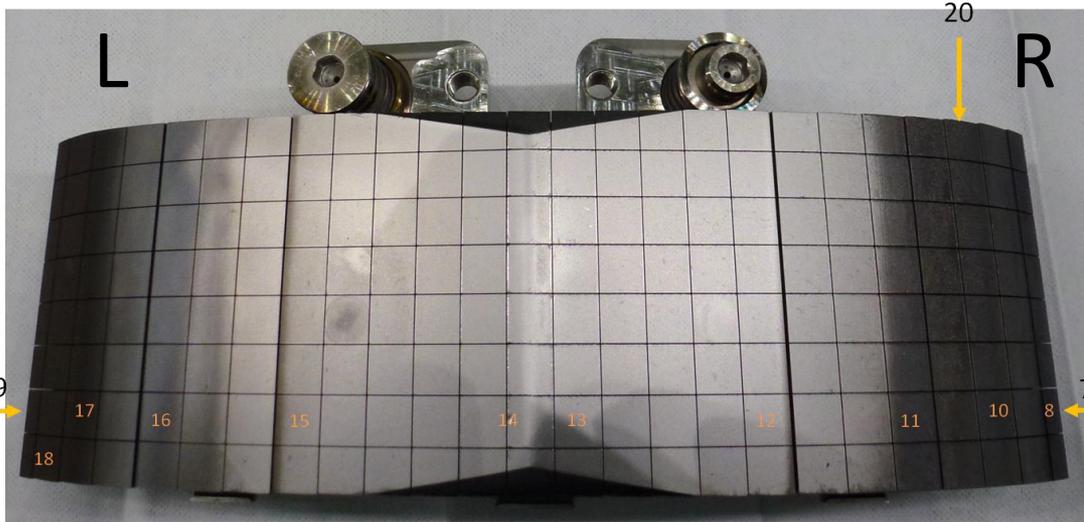
- *Very small quantity of loose matter in the divertor: 1 g/campaign.*
- *Very strong adhesion of dust to the substrate.*
- *No tungsten droplets.*

## Tile 5, bulk W lamellae, IWL-1 + ILW-3



E. Fortuna, IPPLM-WUT

# Dust: Inner Wall Guard Limiter 2XR11 ILW1-3



## Messages:

- Numerous splashes, co-deposits and irregular objects with empty interior.
- Strong adherence of particles to the substrate.

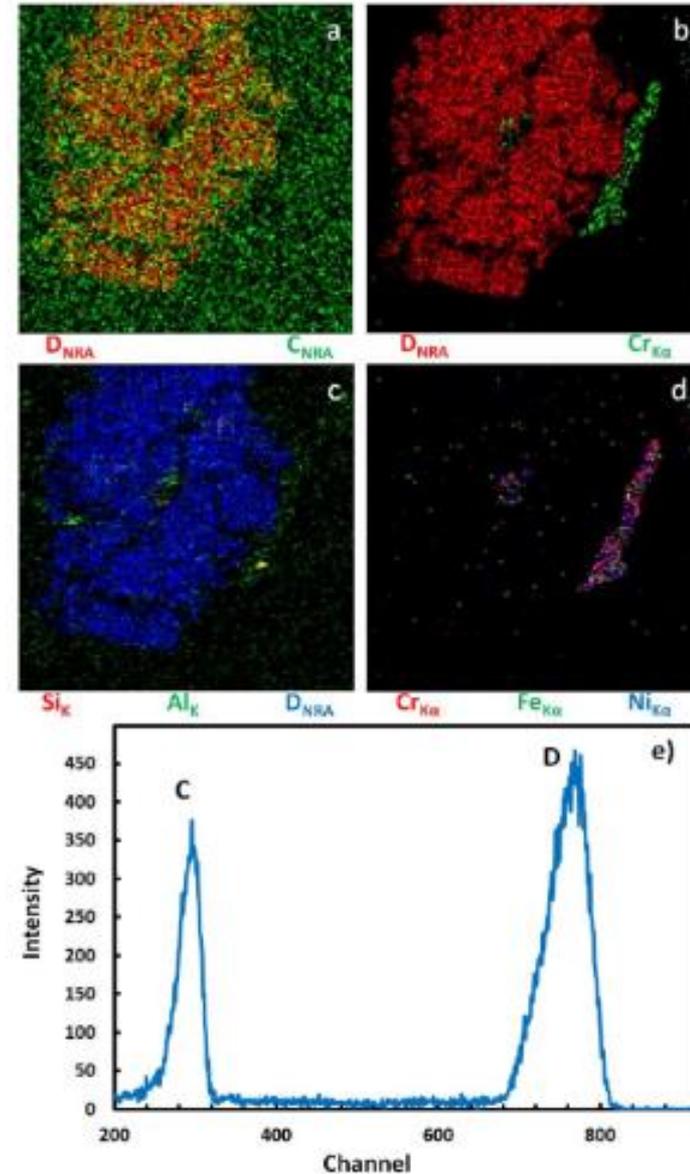
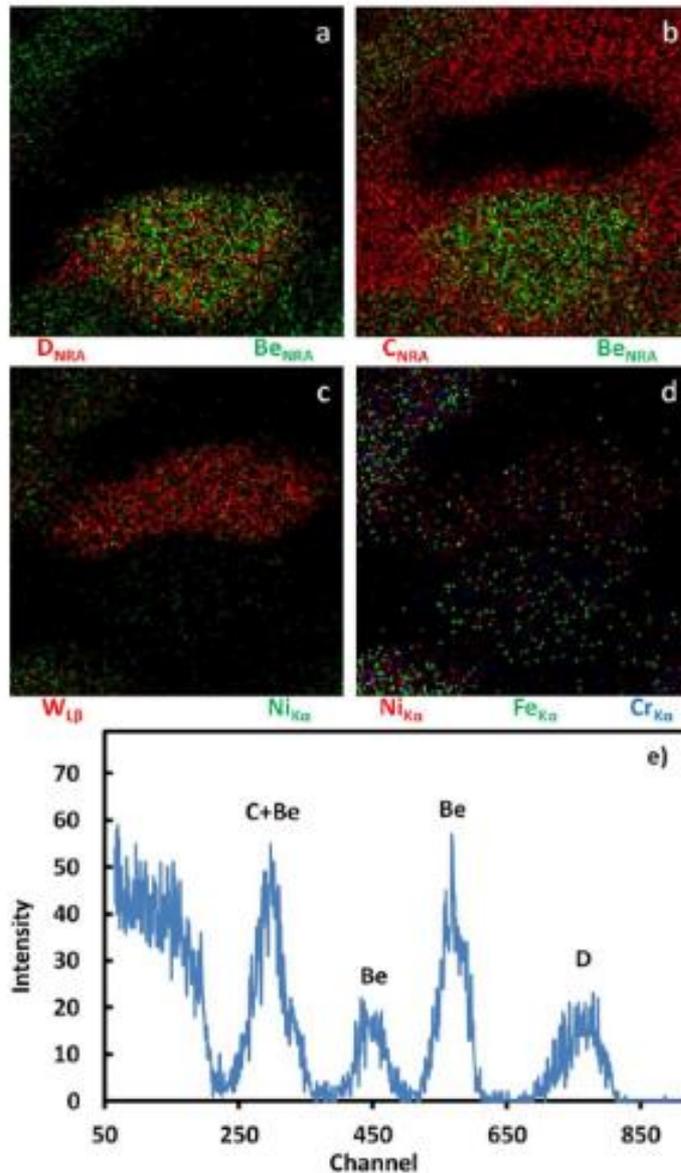
# Dust collected by vacuum cleaning after ILW-3: Ion Micro-beam Analysis



**Main message:**

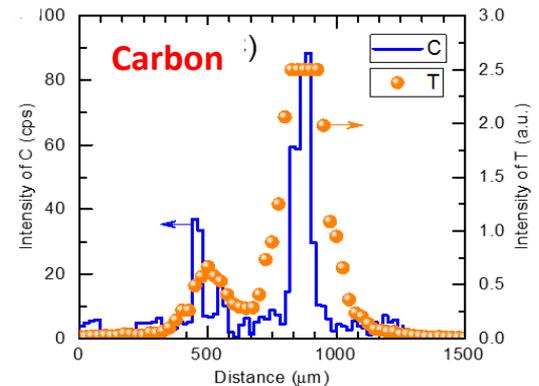
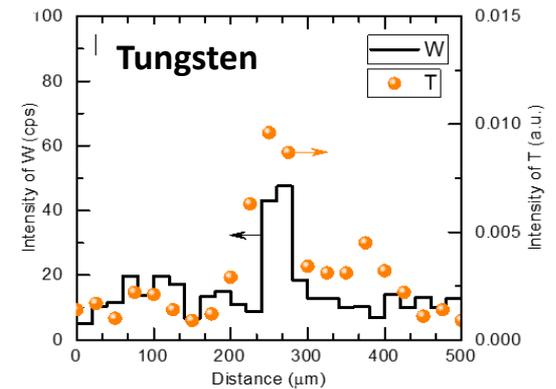
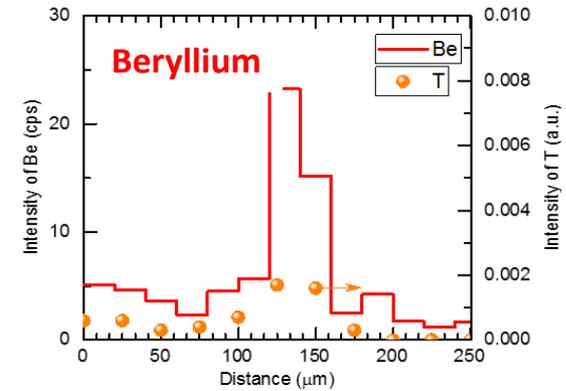
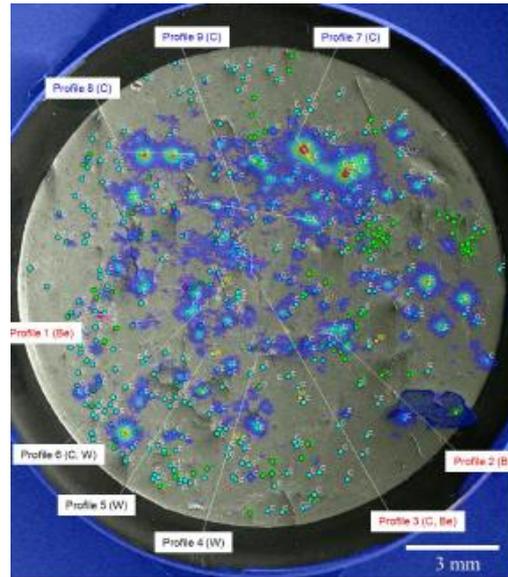
**D retention is associated mainly with C:**

***~ 10–20 times greater than in metal-based particles.***



# DUST:

## Radiography of T in different particles (ILW-1)



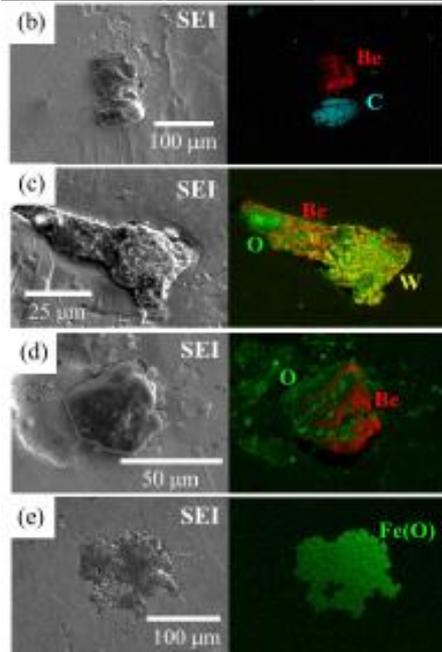
- *Dust particles are placed on In disk.*
- *Composition of individual particles is identified with EPMA (WDS).*
- *$\beta^-$  (T) recorded by radiography.*
- *Superposition of profiles.*

**Message:**

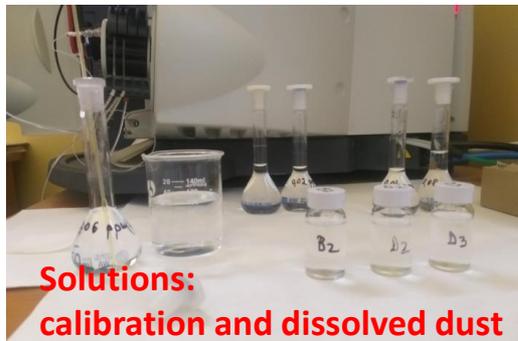
**Tritium presence associated with C.**

**T. Otsuka, Osaka-Kindai U. / IFERC**

**Note the scale**

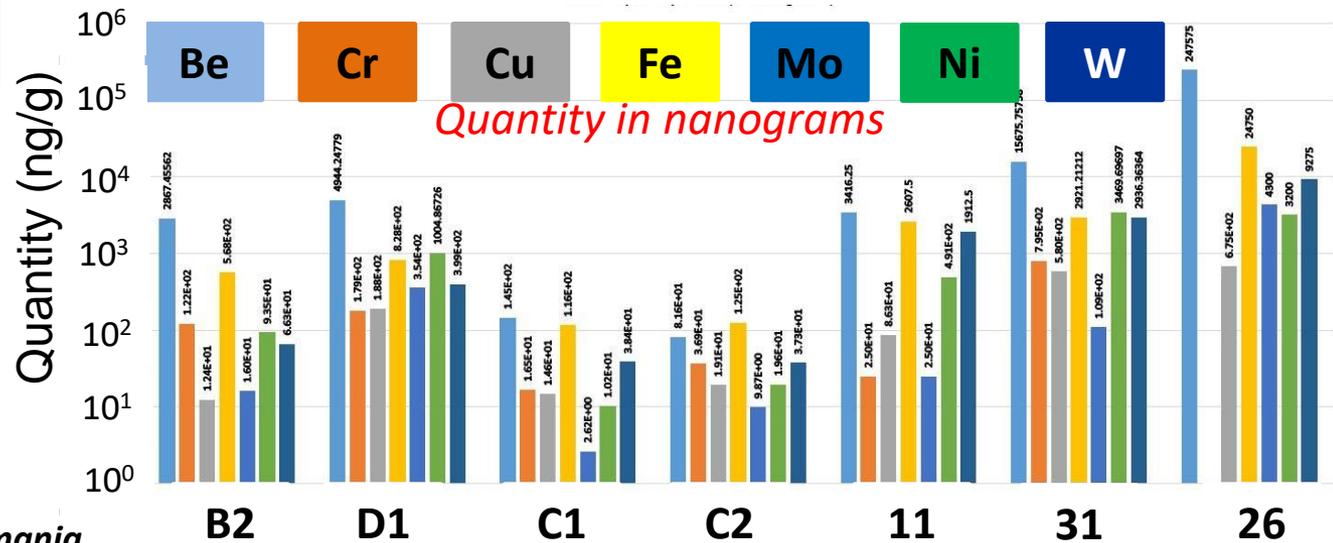
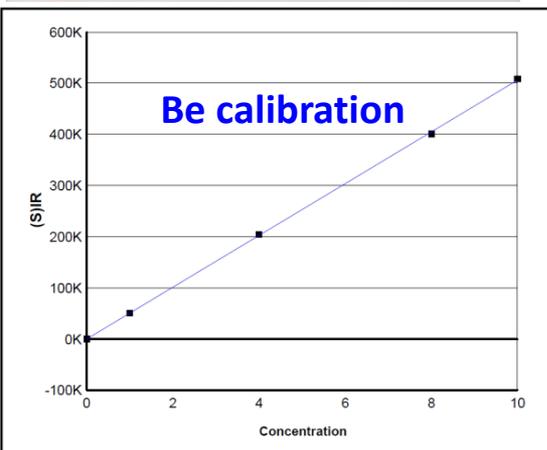


# Dust: Analyses of metals\* in dust by ICP-OES



Sample	B2	D1	31	C1	C2	11	26
Be Wt ratio	0.766	0.626	0.592	0.422	0.248	0.399	0.854
Cr Wt ratio	0.032	0.023	0.030	0.048	0.112	0.003	0.000
Cu Wt ratio	0.003	0.024	0.022	0.043	0.058	0.010	0.002
Fe Wt ratio	0.152	0.105	0.110	0.338	0.379	0.304	0.085
Mo Wt ratio	0.004	0.045	0.004	0.008	0.030	0.003	0.015
Ni Wt ratio	0.025	0.127	0.131	0.030	0.059	0.057	0.011
W Wt ratio	0.018	0.051	0.111	0.112	0.113	0.223	0.032

- Carbon and other non-metals cannot be analysed by ICP-OES.
- Be is the major metal constituent of dust.

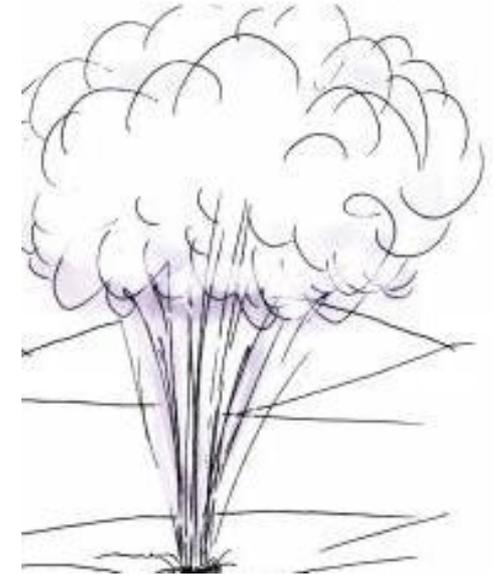


- B2** – swab (with vacuumed dust)
- D1** - divertor 2014-11
- 31** - HFGC tile (2014)
- C1** - 15BNG6D -2010-2014
- C2** – 14BWG6B –2010-2014
- 11** - SEM stubs
- 26** - SEM stubs



**ITER wants to know:**

***What happens to beryllium-rich deposits when hot water/steam enters the vessel?***



# Impact of moisture on dust generation: Experiment



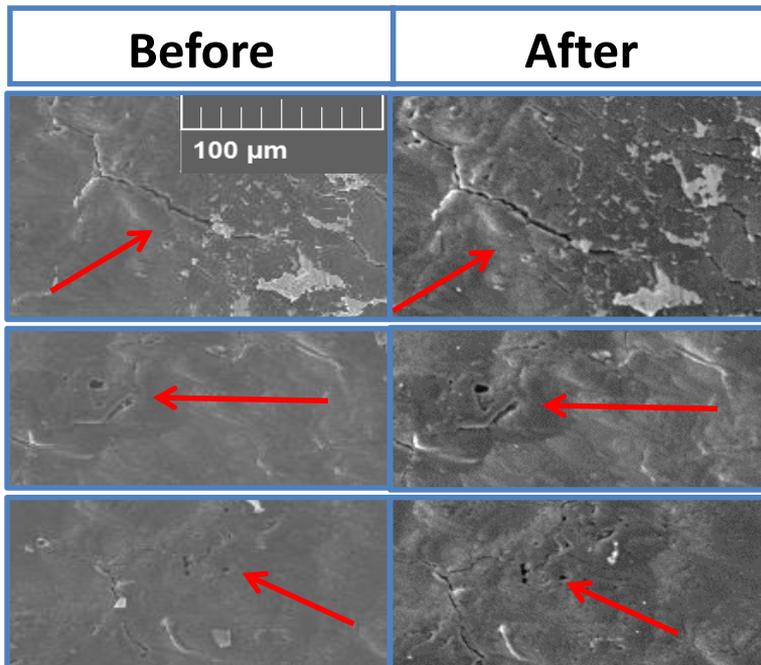
## Task requested by ITER:

*Determine dust generation under off-normal events like water and air leaks.*

## Experiment:

- “Cook” Be limiter samples with/without deposits for some hours.
- Prevent water evaporation (use of cooler)
- Determine T content in H<sub>2</sub>O (tritium transfer).
- Determine surface properties before and after exposure to hot water.
- Investigate a solid residue to check for the released dust.

Current status: One sample with deposit (cut from 2XR10 tile IWGL) “cooked” for ~4 h.



## Main messages:

- Exposure of Be with deposit doesn't lead to “mechanical” damage to the layer: no cracking or large-scale flaking.*
- No flakes/dust found in the flask → mechanical stability of the deposit exposed to boiling water.*
- Agreement (a & b) with data obtained in earlier experiments. (P. Petersson, E. Fortuna, 2019)*
- Tritium detected in water:  $\sim 6 \times 10^{11}$  atoms ( $\sim 10$  % of total T inventory within the sample)*

# Where are we NOW?



- **The original scope of WPJET2 (2014) has been vastly broadened by new projects:**  
*(i) identified research needs, (ii) own ideas; (iii) ITER needs/requests.*
- **The majority of work has already been done or it is a very advanced stage.**
- **Most results have already been published.**

**Samples to be distributed** *(delay caused by Covid):*

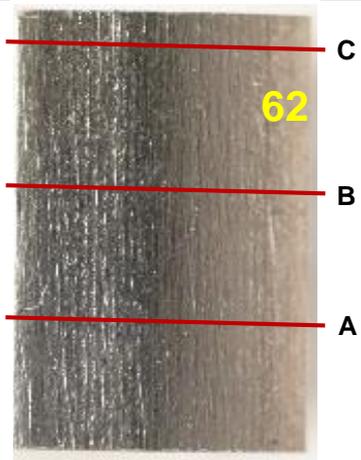
- **Be castellation samples from tiles exposed to ILW 1-3**
- **Sample of filter in JET exhaust ventilation system**
- **Langmuir probe – non-exposed**

**Anna with tell more tomorrow.**

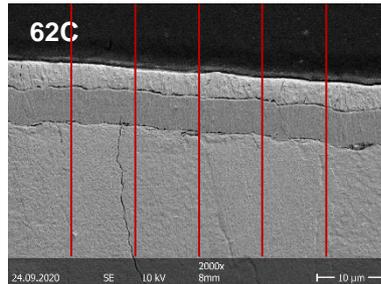


**Extra slides with  
detailed information**

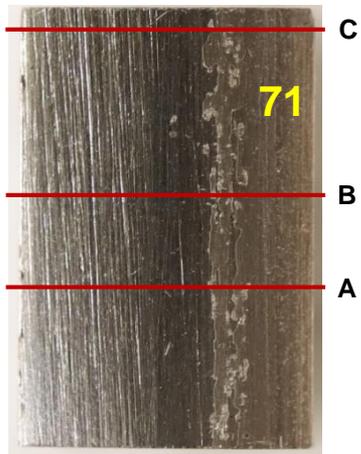
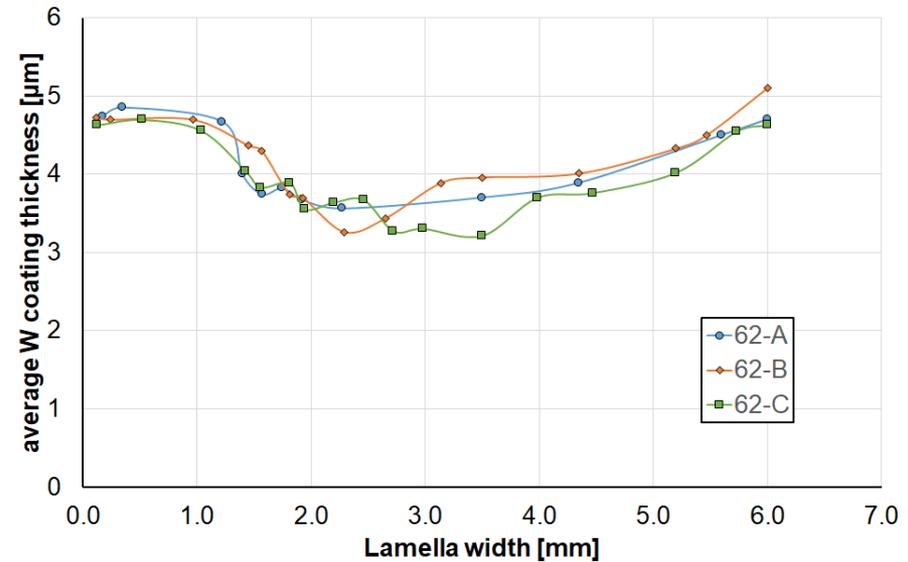
# Tungsten lamellae with marker (ILW-2): W-Mo coating thickness



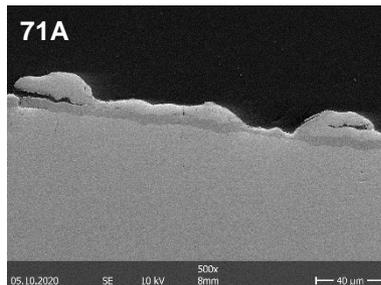
X



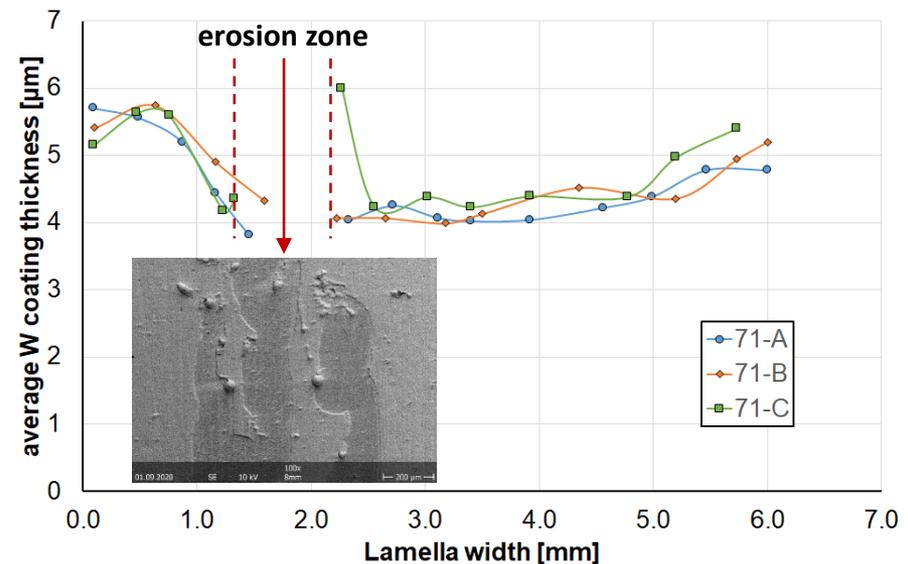
measurement @  $\geq 5$  positions



X



Measurement not always possible due to ablation/melting



X ... inner wall facing marking