

## **WPJET2 – Fuel retention**

Anna Widdowson With many thanks to WPJET2 Team





This work has been carried out within the framework of the EUROfusion Consortium and has received funding from the Euratom research and training programme 2014-2018 and 2019-2020 under grant agreement No 633053. The views and opinions expressed herein do not necessarily reflect those of the European Commission.

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## **Overview of fuel retention studies in JET after 2015-16**



W/CFC Divertor and limiter tiles Beryllium limiter tiles Calibration samples for T measurements



## W/CFC Divertor and limiter tiles from 2016-2017 shutdown

Two successful meetings in 2020 to compare data and identify actions: – (i) IBA labs & (ii) TDS labs Full comparison of fuel retention summary in divertor for ILW1, ILW2, ILW3 by IBA Comparison of cumulative fuel retention – on-going Fuel retention after long term storage – on-going Fuel retention from TDS and remaining inventory using NRA – ongoing OUTLOOK: Work will be completed in 2021

No.	Deliverable description	Organisation	Due date
WC-D-49	IBA of samples from sectioned tiles. Study according to competence & capabilities of laboratories and allocated funding	NCSRD, VR	May-20
WC-D-51	TDS (and full combustion by UoL) of samples from sectioned tiles. Study according to competence & capabilities of laboratories and allocated funding.	CCFE, IAP, UoL	Jun-20
WC-D-54	Shipment of ILW-1 and ILW-2 samples and W/CFC cuts (1/4 samples) after TDS from CCFE to IST for measurements of fuel retention after long-term storage	JOC	Jan-20
WC-D-55	NRA fuel retention measurements after long-term storage	CCFE, IST	Jun-20
WC-D-56	NRA fuel retention measurements in W/CFC samples after TDS	CCFE, IST	May-20

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

- 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**



<ul> <li>More or less constant</li> </ul>		Deposite	d mass (	g)	
deposition rate of D		ILW-1	ILW-2	ILW-3	
	D	0.9	0.7	0.9	
<ul> <li>Slowly varying deposition rate of Be</li> </ul>	Be	53	60	46	
<ul> <li>Decreasing deposition rate of C</li> </ul>	С	13	7	6	
from ILW-1 to ILW-3	De	posited m	ass rate	(µg/s)	
	D	13	10	11	
	Be	771	877	544	
	С	189	102	71	
		Concentra	ation (at.	%)	
	D	6.0	4.6	7.4	
	С	14.6	7.7	8.3	

S. Krat, Phys. Scr. T171 (2020) 014059 M. Mayer, IAEA-FEC 2020



#### Deposition in the tungsten divertor: Cumulative comparison underway



Catarino, Phys. Scr. T171 (2020) 014044

Tile 6

1.W

2.0

a) ILWI

#### Be deposition and D retention

Comparison for cumulative vs summed data i.e., 2011-16 vs 2011-12 + 2013-14 + 2015-16 **Tile 4 and Tile 6 comparison completed** Other tiles to be accessed – fuel retention and deposition





## **Comparison of IBA data**

Overall good agreement of data from IPP and IAP providing IBA data for WPJET2.

14ING1C Tile 1 (in-vessel 2015-16) - full set of data exists from both laboratories.

IPP-4 MeV, SIMNRA

IST – 2.3 MeV, WinNDF

#### Thick films (<300 mm) – good agreement

Thin films (>300 mm) - IPP > IST for D,

- Greater penetration into W coating surface
- Fuel penetrates into W coating

#### Poor agreement with oxygen

O resonance at 2.8 MeV accessed by IPP

Next step: Exchange raw data for analysis





iptn



#### Fuel retention after long term storage



Tiles identified for reanalysis: -

- 2BNG4C Tile 4 divertor (in-vessel 2011-12)
- 2IWG3A Tile 1 (in-vessel 2015-2016)
- 2B4C beryllium dump plate (in-vessel 2013-2014)

Reanalysis on-going, to be followed by comparison of data measured previously.

## Analysis of tritium containing DTE1 samples: SIMS, TDS, pyrolysis, IBA



# MkIIA DTE1 divertor tiles: Tritium analysis and retention after long term storage

Tiles 3 (6IN3) and 4 (4BN4) exposed during DTE1

Samples cored in VTT in December 2019 SIMS, TDS, pyrolysis and IBA SIMS analyses

- Co-deposited layer spalled off in some areas of the tiles due to long storage and handling (~20 years)
- Layered structure on sample from bottom of tile 3, layer thickness ~18µm.
- D retained in co-deposited layer and at interface between co-deposit and CFC tile.
- SIMS profiles for D will be quantified using TDS and ion implanted Be sample





## **TDS comparison of pre-DTE1 and DTE1 samples**



#### Mass 5 and 6 in DTE1 sample attributed to DT and T<sub>2</sub>

Cross calibration with pryolysis of DTE1 sample & TDS of tritiated sample at CCFE NRA for deuterium retention completed at IST

- Tile exposed in 1996 before DTE1 campaign
- no DT signal, T<sub>2</sub> and Be signals very small
- D amount: 2.8E17 cm<sup>-2</sup>



- D amount: 1.1E19 cm<sup>-2</sup>
- T amount: 1.9E16 cm<sup>-2</sup>





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## LID and TDS of JET samples in FZJ: FREDIS status



FREDIS (Fuel <u>Re</u>tention <u>Diagnostic Setup</u>): designed for JET tile analysis Two thermal release methods: laser heating (LID) and oven heating (TDS) Common quantification by Quadrupole Mass Spectrometry (QMS)



2019-2020: upgrade to Be and tritium **completed** 

fully PLCcontrolled tritium cleaning incl. tritium trap **in operation** 

tritium handling limit of lab: 2.5E10 Bq for gaseous T 2.5E13 Bq for bound T





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## Fuel retention analysis: Laser-Induced Desorption (LID)



Sample: Tile 0 (=HFGC 14 N LH) after ILW-3 campaign

Spots 3 mm diameter heated by one short laser pulse (1-5 ms pulse duration)

Desorbed gases quantified by Quadrupole Mass Spectrometry (QMS)

Complete D desorption reached at >1 MJ/m<sup>2</sup> incident laser energy density with 1 laser pulse/position

next samples:

divertor Tile 1 (14 IN G1 C ILW-3), Be limiter (2XR10), W lamellae

This LID-QMS method is in preparation to be applied *in situ* in JET and ITER.

- LID-QMS retention results agree quite well with IBA\*
- IBA values lower probably due to different toroidal position at the end of deposition region (in cores 1b-7b)

\*Ref: S. Krat et al, Phys. Scr. T171 (2020) 014059 doi:10.1088/1402-4896/ab5c11



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## Beryllium limiter tiles from 2016-2017 shutdown



Two successful meetings in 2020 to compare data and identify actions: – (i) IBA labs & (ii) TDS labs Full comparison of fuel retention summary in divertor for ILW1, ILW2, ILW3 by IBA Comparison of cumulative fuel retention – completed Fuel retention after long term storage – on-going Fuel retention from TDS and remaining inventory using NRA – COMPARATIVE STUDY Tritium analysis – TDS, dissolution, others... – COMPARATIVE STUDY

DELAY: Distribution of ILW1-3 Be samples from WPL and DP in November 2020 OUTLOOK: Work will be completed in 2021

No.	Deliverable description	Organisation	Due date
Be-D-73	IBA of Be samples after TDS	IST	May-20
Be-D-65	Shipment of samples to laboratories	JOC, CCFE	Apr-20
Be-D-66	Dissolution for tritium depth profiling	UoL	Jul-20
Be-D-67	TDS and full high temperature outgassing of samples from sectioned tiles. Study according to competence & capabilities of laboratories and allocated funding.	CCFE, IAP, UoL	Jul-20
Be-D-69	Deuterium micro-beam analysis of carbon on plasma-facing surfaces and in the castellation: 3 samples	NCSRD	Aug-20
Be-D-70	Reports	All involved	Feb-20
Be-D-71	Return of all samples to CCFE	All involved	Oct-Nov-20

## Fuel retention in beryllium limiters





Fuel retention in beryllium limiters completed

D retention is concentrated in deposits on the ends of limiter tiles in the mid-plane region

Deuterium retention lowest in high heat load/erosion areas at midplane inner/outer limiters & dump plate ridge

Higher fuel retention in outer limiter compared with inner limiter

Deposits for ILW1-3 tiles thicker than analysis depth therefore total deuterium underestimated

#### **Outer limiter**



Widdowson, (2020). Physica Scripta, T171, 014051. https://doi.org/10.1088/1402-4896/ab5350

## **Global fuel retention in beryllium limiters**



#### Contribution to total fuel retention in surface of beryllium limiter tiles <0.03%

	ILW1	ILW2	ILW3
Total D atom puffed*	2.6x10 <sup>26</sup>	2.6x10 <sup>26</sup>	2.2x10 <sup>26</sup>
Inner limiter	1.4x10 <sup>22</sup>	0.8x10 <sup>22</sup>	1.2x10 <sup>22</sup>
Outer limiter	5.2x10 <sup>22</sup>	0.8x10 <sup>22</sup>	3.2x10 <sup>22</sup>
Dump plate	1.2x10 <sup>22</sup>	0.9x10 <sup>22</sup>	1.6x10 <sup>22</sup>

\* JET gas inventory, GIMs only, 300K

Widdowson, (2020). Physica Scripta, T171, 014051. https://doi.org/10.1088/1402-4896/ab5350



## Tritium in the beryllium limiter tiles - ILW1,2&3





**OF LATVIA** 



Highest T concentrations are in outer wall tile (WPL) – as for deuterium Lowest T concentrations in upper part of the vessel (DP) Anna Widdowson & JET2 Team | JET2 & PFC Annual meeting | Online | 9-10 November 2020 | Page 16

## IFERC Glob

## Global Tritium distribution by radiography (Tritium Imaging Plate)



#### Highest T content is found in the surface region of outer poloidal limiters.

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## **Comparison of tritium measurement methods**



## Three samples cut in 4 pieces to perform 4 different tritium measurements (QMS data is being processed)

#### TDS (scintillation counter), QMS, combustion and dissolution



## 1.2E+14 1F+14 Tritium concentration, atoms/cm<sup>2</sup> 8E+13 6E+13 4E+13 2E+13 598 563 547 Sample No

Dissolution TDS Combustion

Tritium concentration, atoms per cm<sup>2</sup>

Sample	Dissolution	TDS	Combustion
598	3.3·10 <sup>13</sup>	4.1·10 <sup>13</sup>	4.3·10 <sup>13</sup>
563	3.9·10 <sup>13</sup>	7.8·10 <sup>13</sup>	11.3·10 <sup>13</sup>
547	1.2·10 <sup>13</sup>	1.4·10 <sup>13</sup>	1.4·10 <sup>13</sup>



Measured tritium concentration



- Samples mounted in a jig to maintain 2 mm between the sample surface and the detector window
- β radiation emission of T distributed on the sample surface in 2π direction measured;
- Surface activity calculation from reference samples:

#### A=R.2.100/µ

A = activity (Bq), R = CPS in  $2\pi$  of the detector, 2 = conversion factor  $4\pi/2\pi$ ,  $\mu$  = efficiency (%). Tritium surface monitor **LB 1230UMo** with LB 1230 open window gas flow proportional counter detector, Berthold type (USA)





### Surface tritium analysis: beta detector



#### T measurements and determination T content on ILW1-3 Be tiles T activity higher on outer limiter than inner limiter

#### Be IWGL 2XR11 (2010-2016) 0.9 -610 611 Activity (kBq) 0.6 630 631 638 639 640 641 642 643 616 629 644 645 623 624 635 636 633 0.3 666 667 668 677 678 R7 625 626 627 0.0 C4 C1 C2 C3 C1 C2 C3 C4 C5 C6 C7 C8 C9 C10 c11 C1 C3 C1 C2 C3 C4 25 X Coordonate Tritium analysis – WOPL 4D15 – Calibration undergoing 25 Be WPL 4D15 (2010-2016) 20 Activity (kBq) 15 10 Λ 5 10 15 20 25 X Coordinate

#### Tritium analysis – IWGL 2XR11– Calibration undergoing



## **Comparison of TDS results on Be limiter tiles**



- AIM 1 to compare the results of the TDS measurements taken on "identical" samples in different labs.
- AIM 2 to compare the results of the measurements of tritium content between different techniques.
- SAMPLES castellations cut from Be limiter tiles and Be upper dump plate, all removed after ILW3 campaign 2015-2016 → cut into four "quarterpieces".
- LABS:
  - IAP (Romania) sample prep (cutting with a slow dry saw) and TDS.
  - FZJ (Germany) TDS.
  - CCFE (UK) TDS.
  - University of Latvia dissolution (for tritium).
  - IST (Portugal) IBA.

## Analysis plan



- TDS measurements comparison:
  - Compare total deuterium content.
  - > Compare shapes of the spectra and positions of peaks.
- Tritium measurements comparison: **TDS** vs **dissolution** 
  - Compare total tritium content.
- Comparison of TDS and IBA:
   Compare total deuterium content.
- Determination of retained content after TDS with IBA: IST.
  - ➢ IBA before TDS.
  - ➢ IBA after TDS.
  - > Determination of the fraction of deuterium that wasn't released during TDS run.

#### **Castellation samples from ILW3 Be tiles**







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### **Comparison of deuterium content**

D content same order of magnitude Maximum difference factor 5, generally factor 3 - 4





#### **Comparison of deuterium spectra – IWGL**



Variation in heating Variation in total desorption



#### **Comparison of the contribution of HD**



#### Variation of D2 & HD ratios





#### **Observations**



#### Each lab knows which area to work on!

#### **Total inventory**

- Same order of magnitude
- Variations up to factor 5
- Calibration
- ALSO different quarters are not completely identical

#### Variations in desorption spectra

Shapes of the spectra are comparable

- Similar profile changes between labs
- Shift in position of maxima
- Temperature uncertainty particularly at high temperature

## **Comparison with IBA – Fuel inventory**



Overall trends determined by IBA of quarter samples coincide with those determined by TDS.

Mostly total inventories determined by TDS are higher than those determined by IBA.











First few samples, more to be analysed

Significant proportion of deuterium still present after the TDS in these samples

Two samples identified to heat to 1000°C instead of 775 ° C



## Comparison of tritium content in beryllium samples



Tritium measurements comparison: **TDS** (CCFE, IAP, FZJ) vs dissolution (UoL) Only data where comparison between TDS and dissolution is shown Analysis of other samples ongoing

	Sample ID	Black T atoms/cm <sup>2</sup>	Red T atoms/cm <sup>2</sup>	UoL T atoms/cm <sup>2</sup>	Blue T atoms/cm <sup>2</sup>
	460		x	5.9*10 <sup>9</sup>	
DUMP PLATE	463		x	4.9*10 <sup>11</sup>	
	465	х	1.25*10 <sup>13</sup>	5.6*10 <sup>10</sup>	
	483	х	3.71*10 <sup>13</sup>	5.8*10 <sup>11</sup>	
	503	x	3.39*10 <sup>13</sup>	1*10 <sup>12</sup>	
	506	x	4.58*10 <sup>12</sup>	1.2*10 <sup>12</sup>	3.5*10 <sup>14</sup>
	516	x	x	5.8*10 <sup>11</sup>	4.9*10 <sup>14</sup>
	524		3.11*10 <sup>13</sup>	3.5*10 <sup>11</sup>	
	543	х	x	1*10 <sup>13</sup>	
WPL	569	x	x	4.8*10 <sup>13</sup>	
	580	x	x	3.3*10 <sup>13</sup>	

## Conclusions – tritium analysis of beryllium samples



Large variation in tritium data between TDS and dissolution

- Usefulness of TDS for this level of T inventory uncertain.
- Dissolution → T distributions obtained.

Continuation of T reference samples work  $\rightarrow$  better understand the detection limits (see later).



#### **Calibration samples for T measurements**



Design and manufacture mirror-like H, D, He-implanted samples to be used in the calibration of masses 2, 3 and 4 for TDS of PFC with low T contents Design and manufacture H,D, He-containing deposited layers to be used in the calibration of masses 2, 3 and 4 for TDS of PFC with low T contents Manufacture tritiated samples for calibration Determine H,D,He content in implanted and plasma-deposited samples with ToF ERDA Determine T content in the tritiated samples OUTLOOK: Analysis will be completed end 2020/early 2021

No.	Deliverable description	Organisation	Due date
TM-01	Production of 20 Mo mirror-like samples, diameter 10 mm, thickness 3 mm	VR [IAP]	Nov-19
TM-02	Coating of Mo mirrors with 1-2 µm layer of W	VR [IAP]	Dec-19
TM-03	Implantation of mirrors with H, D, He	VR (16),VTT (8)	Dec-19– Jan20
TM-04	Surface analyses with NRA and ToF ERDA: one sample from each production batch	VR, VTT	Feb-20
TM-05	Perform TDS with implanted calibration samples	CCFE, IAP, UoL	Mar-Apr-20
TM-06	Production of magnetron-deposited W layers containing H,D, He (deposition on mirror like surfaces)	IAP	Dec-20
TM-07	Surface analyses with NRA and ToF ERDA: one sample from each production batch	VR [IST]	Feb-20
TM-08	Perform TDS with implanted calibration samples	CCFE, IAP, UoL [FZJ]	Jul-20
TM-09	Manufacture tritiated samples for calibration of T measurements and determine T content	IAP	Jan-20
TM-10	Perform TDS with implanted calibration samples. [T]	CCFE, IAP, UoL [FZJ]	Sep-20
TM-11	Reports	All involved	Oct-20

## Reference sample preparation by HIPIMS at IAP



Production of magnetron-deposited W - IAP Layers containing H, D, He Analysis by GEDOES & TDS



#### Analysis by ERDA, NRA, RBA at VR and IST

ToF ERDA (preliminary): EU2-34: Hydrogen 1.18 at% EU2-35: Helium 13.6 at% EU2-36: Deuterium 1.67 at%, Hydrogen 0.40 at% RBS: 34:  $2.3 \times 10^{19}$ /cm<sup>2</sup> (3.7µm) 35:  $1.1 \times 10^{19}$ /cm<sup>2</sup> (3.7µm) 36:  $1.9 \times 10^{19}$ /cm<sup>2</sup> (3.0 µm) NRA: EU2-36:  $8.8 \times 10^{17}$  D/cm<sup>2</sup> (in 3.0 µm) assuming a density of 19.6 g/cm<sup>3</sup>)

### Reference sample (Mo) preparation by LEIS at VR

#### **Irradiation with Helium**

Analysis - not done yet Typically 10% retention

#### Irradiation with Deuterium

Measured concentration:  $2.5 \times 10^{16}$ /cm<sup>2</sup>

### W-D sample (HIPIMS) TDS comparison – TOTAL D







Facility	TOTAL Inventory: D atoms/cm <sup>2</sup>
RED	8.2*10 <sup>17</sup>
GREEN	<b>1.9*10</b> <sup>18</sup>
BLUE	9.6*10 <sup>17</sup>
BLUE	7.9*10 <sup>17</sup>
IBA (IST)	7.4*10 <sup>17</sup>
IBA (VR)	8.8*10 <sup>17</sup>

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#### W-H sample (HIPIMS) TDS comparison – H signal









Facility	TOTAL Inventory: H atoms/cm <sup>2</sup>
RED	1.1*10 <sup>18</sup>
GREEN	1.45*10 <sup>18</sup>
BLUE	1.11*10 <sup>18</sup>
BLUE	<b>1.01*10</b> <sup>18</sup>
IBA	

### W-He sample (HIPMIS) TDS comparison – He signal







Facility	TOTAL Inventory: He atoms/cm <sup>2</sup>
RED	<b>3.2*10</b> <sup>17</sup>
GREEN	No calibration
BLUE	
BLUE	
IBA	

### **TDS reference sample conclusions**



Inventory values within factor 2.2
 Similar values as compared with IBA data
 Temperature peak positions
 Low temperature peak position is almost identical for all facilities
 2<sup>nd</sup> peak varies – discrepancy at higher temperatures
 Useful comparison for all laboratories to improve measurements
 OUTLOOK: Remaining HIPIMS and implanted reference sample analysed by end 2020/early 2021

## Analysis of tritiated samples



#### Sample preparation at IAP

IAP	2 micron 100% W, Tritiated with Mo desired T content	10 mm x 10 mm surface 1 mm thickness 10 mm x 10 mm 3 mm thickness for LID	4e14 atoms T (250 kBq) 4E17 T atoms (160 MBq)	1. 2. 3.	W coating performed Tritiation calibration performed Tritiation performed	IAP - surface analysis using Tritiated testosterone polystyrene matrix as reference. IAP - Total combustion IAP - Total combusion	CCFE 3 (TDS/Pyrolysis/offgas) IAP 3 (Surface analysis/total combustion/TDS) FZJ 4 (2 LID - 3 mm, 2 TDS) Latvia 3 (TDS/QMS/Dissolution) HR 2 (D beam IBA)
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#### Analysis – December 2020

#### TDS:

10K/min RT to 1000 deg C, holding @1000<sup>0</sup> C for 1 hour, CCFE, FZJ \*, IAP, UoL^ \*FZJ to measure multiple samples due to sensitivity issues ^UoL to start with 5K/min for safety reasons (1/4 sample) – based on first quarter results, Decision to run at 10K/min

#### Full combustion/dissolution:

UoL - technical details tbc

IAP - technical details tbc

**LIDS:** FZJ - technical details tbc



#### Be-coated Inconel tiles from the inner wall cladding

No.	Deliverable description	Organisation	Due date
IN-D-26	Outgassing, Tritium measurements	UoL	Jul-20

#### Tungsten lamellae, Tile 5 after ILW-3

No.	Deliverable description	Organisation	Due date
WL-D-33	IBA: NRA and EPS	IPP, VR	May-20
WL-D-40	TDS	CCFE, IAP	Jun-20

#### Dust from vacuum cleaning and deposited on dust monitors

No.	Deliverable description	Organisation	Due date
D-D-22	Chemical composition (QMS, FTIR) and tritium content	UoL	Jun-20

### Conclusions



#### Majority of deliverable on fuel retention have been completed.

Main fuel retention conclusions from WPJET2 achieved and published

COMPLETED

Analysis of fuel retention in divertor tiles ILW1, ILW2, ILW3 and some cumulative data Analysis of fuel retention in limiter tiles ILW1, ILW2, ILW3 and cumulative data Collaboration through IBA and TDS dedicated workshops to consolidate results

ON-GOING Measurement of fuel retention after long period - IBA Continue comparison of cumulative data for divertor tiles Final TDS analyses Final tritium analyses

OUTLOOK All deliverables will be completed during 2021

#### CHALLENGE

Converge on data to produce JET-ILW overview on material migration, fuel retention and surface modification from plasma interaction