



Target materials R&D & procurement

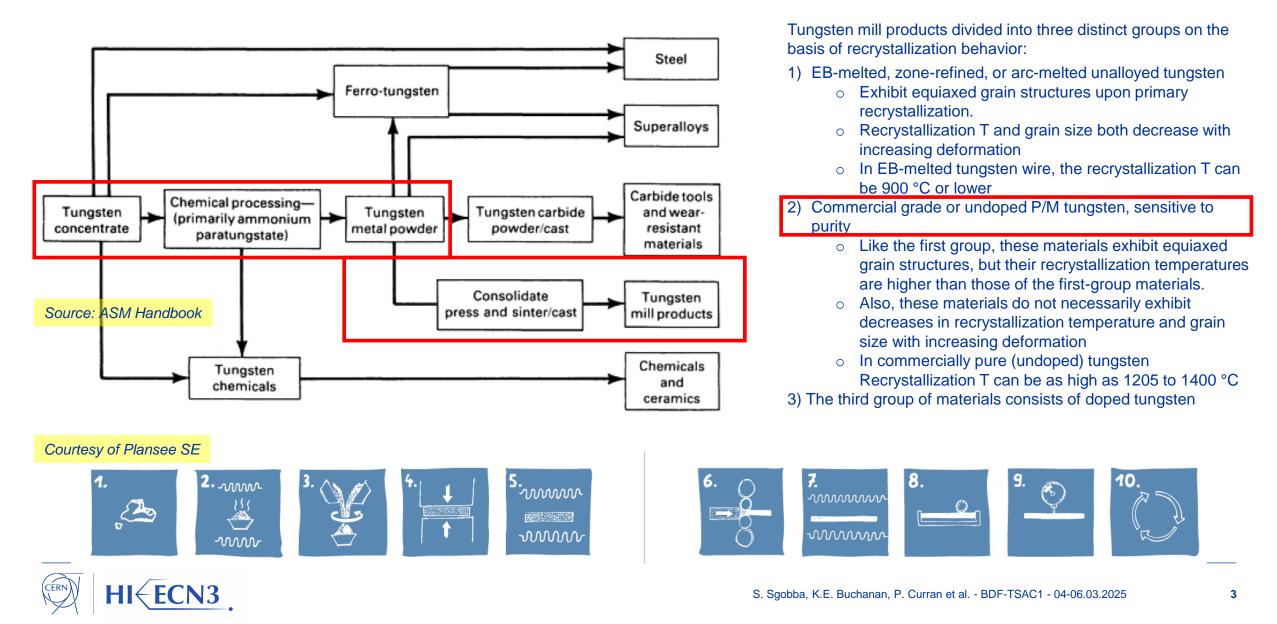
- Ongoing and required material R&D to meet the project requirements
- Palette of tests for the material target and why
- Explain the choice of hot rolled W as baseline, procurement considerations
- Radiation damage considerations on materials do we care?

Stefano Sgobba, Katie Elizabeth Buchanan, Patrick Michael Curran, Enrique Rodriguez Castro, Ignacio Aviles Santillana, Ana Teresa Perez Fontenla, Marlini Simoes

04/03/2025



Pure W - Metallurgy and processing background



Pure W - Metallurgy and processing background

Table 22 Typical purity of the three commen

Impurity element	Concentration, ppm, in tungsten							
	Electron beam zone refined	Undoped	Doped					
Iron	1	10	11					
Nickel	2	5	5					
Silicon	5	21	47					
Aluminum	<2	<5	15					
Potassium	<1	12	91					
Oxygen	10	27	36					
Carbon	20	31	24					

PSE-605-PS-016	

S – PRODUCTSPECIFICATI	ION		PLANSEE 4/6			
is document is subject to electronic ve		status before using.		470		
Chemical c	omposition					
		ansee	Standard	EU-Directive		
Main and Minor Components		ontent	AOTH D 700	B-110-0		
W	99.99 % ^{b)}	Min. 99,97 % ^{b)}	Balance	•		
	Max. Va	lues [µg/g]	Max. Values	Max. Values		
Impurities	Typical	Guaranteed	[µg/g]	[µg/g]		
AI	1	- 15	-	-		
Cr	3	20	-	-		
Cu	1	10	-	-		
Fe	8	30	100	-		
к	1	10	-	-		
Mo	12	100	-	-		
Ni	2	20	100	-		
Si	1	20	100	-		
С	6	30	100	-		
Н	-	5	-	-		
N	1	5	100	-		
0	2	20	100	-		
Cd	1	5	-	100		
Hg	-	1	-	1000		
Pb	1	5	-	1000		
Cr (VI)			-	1000		
				1		

a) EU-directives 2015/863/EU, 2011/65/EU and 2000/53/EC.

b) Metallic purity without Mo

**) The presence of Cr (VI) and organic impurities can definitely be excluded because of the production process (multiple heat treatments at temperatures above 1000 °C in H₂-atmosphere.

The chemical composition is checked by means of random sampling. The sampling inspection plan, analysis and evaluation methods are determined in the internal instruction PSE-020-WI-003. The application of the measured values for the chemical analysis is defined in PSE-680-WI-001.

Remarks: The specified physical and chemical characteristics are disclosed not regarding measurement accuracy.

				PLAN:	SEE
lansee SE, Metallwerk F Shipping address	iansee-Straße 71, 6600 Reutte, Austr	a	TEST-REPC acc. to EN 10204	RT 820532318	000010
ERN			Date	Oct 15, 2024	
Réception PRE Site de Prevess			Order number	49202395 / 10	Sep 10, 2024
01631 CERN C			Delivery	820532318 / 10	
FRANCE			Total quantity	1 PCE	
			Total weight	13.211 KG	
Customer info	ermation		Internal informat	tion	
Customer num	ber 2011844		Material	15028974	
PO number	CA1221931	Sep 9, 2024	Basic material	W	
			Description	W Sheet 5 x 370 x	370 mm
			Batch	0095159413	
			Specification	PSE-605-PS-016/	100/04

Hardr	GUARANTEED REQUIREMENTS: Hardness HV EN ISO 6507-1; Density EN ISO 3369:		=>450 >19,20 g/cm	3					
	GUARANTEED CHEMICAL COMPOSITION: (d					PSE-020-	WI-003)		
W Al Fe Si H Cd	min. max. max. max. max. max.	99,97 %** 15 µg/g 30 µg/g 20 µg/g 5 µg/g 5 µg/g	Cr K Mo N Hg	max. max. max. max. max.	20 μg/g 10 μg/g 100 μg/g 5 μg/g 1 μg/g	Cu Ni C Pb	max. max. max. max. max.	10 µg/g 20 µg/g 30 µg/g 20 µg/g 5 µg/g	
Cŕ(VI) The ro hazar *) The	Cd max. 5 μg/g Hg max. 1 μg/g Pb max. 5 μg/g **) Metallic purity without Mo Cr(VI) + Organic impurities (e.g. PBB, PBDE, PFOS, PFOA)* The requirements of the EU-directives 2015/863/EU, 2011/65/EU and 2000/53/EC for the restriction of hazardous substances (RoHS) are fulfilled. *) The presence of Cr(VI) and organic impurities can be excluded definitely because of the production process (multiple heat treatment at temperatures above 1000°C in H2-atmosphere).								

- HR product ordered in 5 mm and 17 mm thickness
- Limited availability in heavy gauges with guaranteed properties - due to a minimum reduction to be provided to achieve final properties

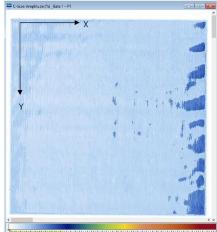


Testing results

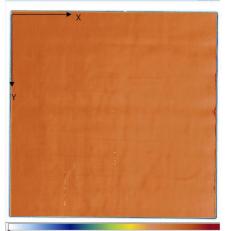
No indications above TFP 1.2mm detected.

All pieces are acceptable to EN 4050-4 Class 5

Amplitude of defect echoes



ຸຣ ທີ່ 15 20 25 20 25 40 45 90 95 90 570 75 10 16 90 95 100 105 Amplitude of backwall echo



5 10 15 20 25 30 35 40 45 50 55 60 65 70 75 80 85 90 95 100 1

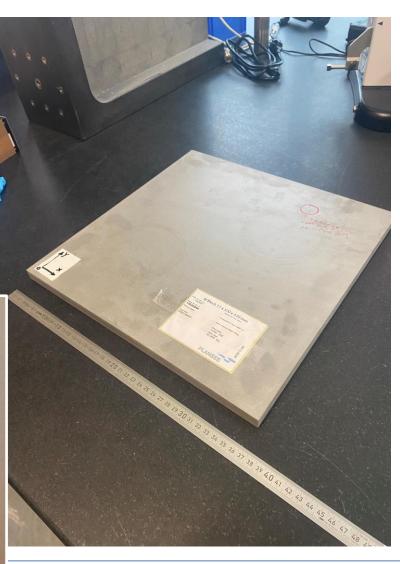
↑ UT results on the 5 mm plate: excellent homogeneity_ and high acceptance level (to 1.2 mm FBH equivalent), EDMS 3190589

Pure W - Metallurgy and processing background

To the extent of the inspections performed, dimensions and tolerances conforming to ASTM B760, see EDMS 3180443

HR 5 mm ↓ and 17 mm thick → plates ordered and received from Plansee





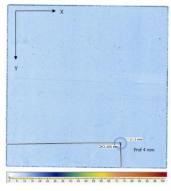
No indications above FBH 1.2mm detected.

The piece is acceptable to EN 4050-4 Class 5

Several reflectors with equivalent reflectivity up to FBH #0.7 mm are observed at depth ~4 mm from the label face

C-SCAN RESULTS - amplitude part N°1

Amplitude of defect echoes





0 5 10 15 20 22 30 35 40 45 50 55 50 55 10 75 80 45 50 55 10

Pure W - Metallurgy and processing background

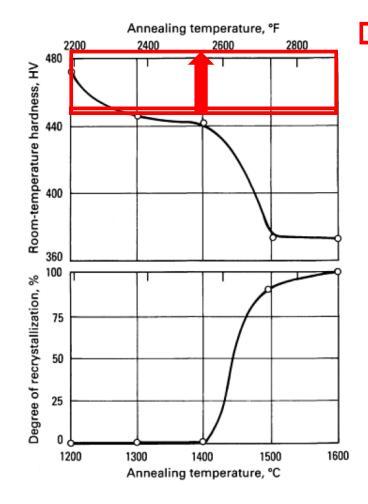


Fig. 26 Recrystallization behavior of undoped tungsten bar

	A DECEMPT	DEOLUDENE	NTO						
Hardne	ss HV	EN IS	SO 6507-	-1:	=>450				
Density		LIVIC	0.0000		- 18,20 g/cm				
GUARA W	NTEE	CHEMICAL C 99,97 %**	OMPOS	SITION: (determined in P	E-020	-WI-003)		
Al Fe Si	max. max. max.	15 μg/g 30 μg/g 20 μg/g	Cr K Mo	max. max. max.	20 µg/g 10 µg/g 100 µg/g	Cu Ni C	max. max. max.	10 µg/g 20 µg/g 30 µg/g	
H Cd **) Meta	max. max. allic pur	5 μg/g 5 μg/g ity without Mo	N Hg	max. max.	5 μg/g 1 μg/g	O Pb	max. max.	20 μg/g 5 μg/g	
The rec hazardo	quireme ous sub	stances (RoHS	irectives) are fulf	2015/86 illed.	3/EU, 2011/65/E			C for the restriction of	
					s can be exclude 1000°C in H2-a			use of the production process	
									Refined, oriented,
									directional grain structure
									\searrow /
							$\boldsymbol{\wedge}$	Normo	
								100 µm	
								A REAL PROPERTY OF THE REAL	and the second se

Benjamin Corbett, https://indico.cern.ch/event/1480601/

	HV1	S)	Grain Size Number
Transverse	490.6		6.6	10
Normal	485.3		8.4	9.5
Rolling	500.9		17.8	10.5

ransvers

	HV1	SD	Grain Size Number
Longitudinal	350.4	8.7	7
Transverse	350.6	5.2	7
Short-transverse	353.14	9.5	7

ongitudir

HI ECN3

5mm rolled Hardness and Grain Size

Forged Hardness and Grain Size

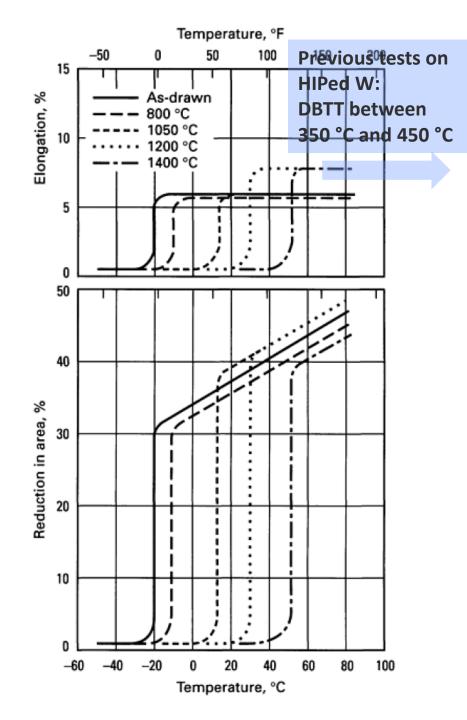
Short-steres

ansvers

Pure W - Metallurgy and processi

Remarks: Upon request, the tungsten sheets are delivered stress-relieved annealed. We point out that the stress-relieved-annealing may lead to a material specific embrittlement

- Recrystallized tungsten undergoes a ductile-to-brittle transition above 205 °C .
- Only by heavy warm or cold working is the DBTT lowered to below room temperature
- Annealing raises the DBTT of cold-worked tungsten until it approaches that of recrystallized material
- The exact ductile-to-brittle transition temperature is influenced by many factors, including grain size, strain rate, and impurity levels
- The DBTT decreases with grain size unless the grains are larger than 1 mm in diameter.
- The DBTT also drops with increases in strain rate, but it climbs rapidly as impurity levels increase.
- Like all brittle metals, tungsten is very notch sensitive. Therefore, removal of even minute surface flaws by grinding, oxidizing, or electrolytic polishing prior to service improves ductility and lowers the DBTT





Pure W flat products – Standard framework

This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.



Designation: B760 – 07 (Reapproved 2019)

Standard Specification for Tungsten Plate, Sheet, and Foil¹

This standard is issued under the fixed designation B760; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

3.2 Product Forms:

3.2.1 *foil*, *n*—a flat product less than 0.005 in. (0.13 mm) in thickness.

3.2.2 *plate*, *n*—a flat product 0.188 in. (4.75 mm) or more in thickness.

3.2.3 sheet, n—a flat product from .005 in. (0.13 mm) to 0.187 in. (4.75 mm) in thickness.

TABLE 1 Chemical Composition/Check Analysis

Element	Composition, max, %	Permissible Variations in Check Analysis, %
С	0.010	±0.002
OA	0.010	+ 10 % relative
N	0.010	+ 0.0005
Fe	0.010	+ 0.001
Ni	0.010	+ 0.001
Si	0.010	+ 0.001

^{*A*} If chemical analysis is performed on a sample from the powder blend used to make the finished product, oxygen will be reported for information only.

7. Metallurgical Condition

7.1 Plate, sheet and foil shall be furnished in one of the following conditions as designated on the purchase order:

Form	Metallurgical Condition		
Plate	hot-rolled		
	hot-rolled, stress-relieved		
Sheet	hot-rolled		
	hot-rolled, stress-relieved		
	cold-rolled		
	cold-rolled, stress-relieved		
Foil	cold-rolled		
	cold-rolled, stress-relieved		

7.2 Other conditions can be specified as agreed upon between the purchaser and the manufacturer at the time of purchase.

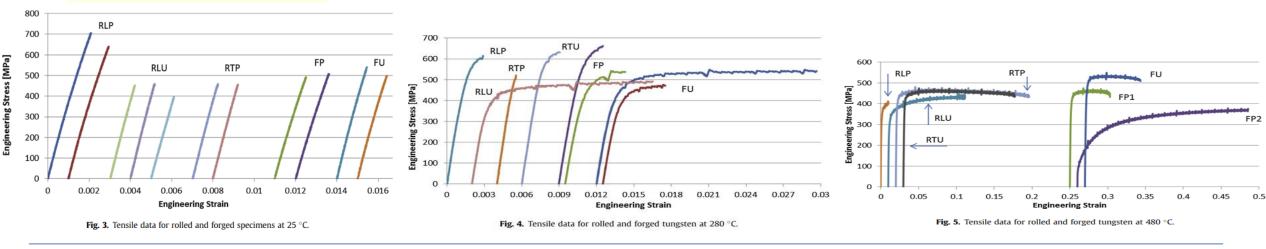
10.4 Material may be supplied with as-rolled, as-cleaned, as-machined, or as-ground finish.



Ongoing and required material R&D to meet the project requirements

Operating conditions:

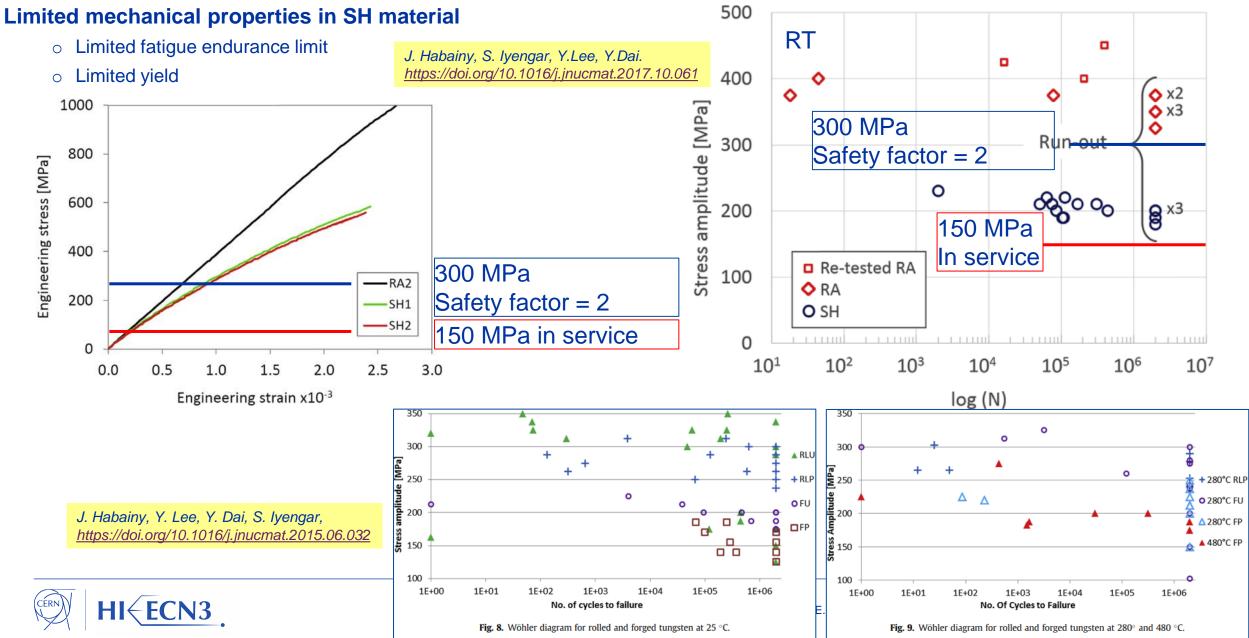
- RT to 400°C
- o Stresses: -100 MPa to 150 MPa
- Fatigue life of 10⁷ cycles
- Irradiation: approximately 1.2 1.6 dpa (< 2 dpa)
- o Individual block height: 17 mm (as HR?) to 350 mm (as HIPed) thick and 250 mm diameter (present baseline)
- o Chemical additions from He: 143-220 ppm(at)
- o Beam Parameters: sigma 8 mm (possibly 16 mm)
- As HR and HR + HIPed properties are relevant
- o HR product form looks a reasonable choice availability in heavy gauges with guaranteed properties limited



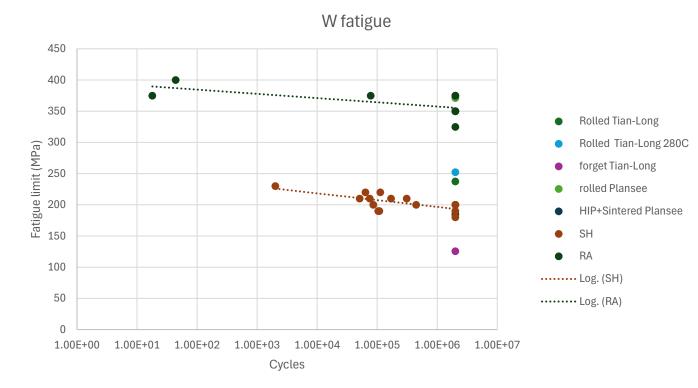
J. Habainy et al. / Journal of Nuclear Materials 465 (2015) 438-447



Ongoing and required material R&D to meet the project requirements



Ongoing and required material R&D to meet the project requirements



Axial forging is possible but there is a limitation:

- Maximum of 110 mm heigth with 260 mm diameter
- From this dimensions reduced mechanical properties as no more axial nor radial deformation can be done.

Rolled tungsten:

- Shows superior fatigue properties than forged and PM HIPed tungsten
- However, it has nearly zero ductility at room temperature and is highly anisotropic
- The orientation of the rolled tungsten bricks with respect to the proton beam needs to be carefully considered.
- The post-pulse maximum stress should be below 100 MPa, and the post-pulse peak stress amplitude lower than 50 MPa (see J. Habainy, Y. Lee, Y. Dai, S. lyengar, <u>https://doi.org/10.7566/JPSCP.28.031004</u>)

Mechanical testing:

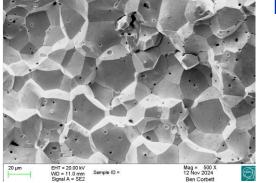
- $\circ~$ Charpy impact toughness testing of the base material, from RT to 600 ^{o}C
- RT & high T tensile testing with possibility of an inert or vacuum atmosphere
- $\circ~$ As above, fatigue testing
- Tensile testing of the adhesion strength of the HIP bonded plates W-W & W-Ta-W.
- Material hardness (as a function of T?)

Ongoing in house on HR plates:

- o A complete grain size analysis
- o Grain orientation analysis
- Recast layer analysis & post cut processing
- o Density evaluation
- Microstructural changes of the base material during the heating exposure/HIPing process

Oxidation studies:

- \circ in relation to NDT
- $\circ~$ and operating environment



• Immersion UT of all incoming

Flatness evaluation ASTM

plates EN 4050-4

Roughness evaluation

Metrology & NDT

B760-07

Material characterization





Palette of tests

Oxidation studies vs. operating environment

- o similar behaviour expected as reported for Mo ⇒
- cleaning of W is desirable to remove compounds that could cause carbon contamination during exposure to a heat cycle
- a variety of cleaning agents may be used to remove oils and hydrocarbons (vapor degreasing and hand or automatic washing with detergent solutions)
- chemical cleaning only needed for heavily oxidised surfaces

See <u>J. Habainy et al., Formation of oxide layers on tungsten</u> at low oxygen partial pressures:

- In Ar gas with a maximum oxygen partial pressure of 5 ppm, a thin and adherent oxide film was observed to form on the surface of tungsten specimens even at 500 °C
- For the operation of the ESS spallation facility, it is important to note that tungsten can be oxidized in environments with low oxygen partial pressures at T as low as 400 °C
- Below 500 °C it is expected that the oxide formed will be of the protective type
- However, considering that the tungsten will be cooled by a massive helium flow with a flux of 3 kg/s at 1.1 MPa, surface erosion of the oxide scale was a concern
- Erosion experiments were performed to study the adhesion of the oxide formed on tungsten at 500 °C
- The sample, pre-oxidized for 1 h in a He-0.5%O2 gas mixture, showed no signs of erosion caused by the helium jet
- Still, the spalling of the oxide layer due to beam pulse and beam trip induced thermal cycling, and fatigue, during five years of target lifetime, remains an issue
- Also of concern is the possibility of tungsten oxide sublimation in accidental cases such as over-focused beam combined with loss of helium confinement.
- To minimize such risks, the target primary cooling loop will be purified and the impurity levels will be monitored during operation.

Atmosphere								
Oxygen	At higher pressures, fo volatile oxides resultin oxide scales; evaporati	g in weight losses. At on of volatile oxides, 1	face tes	Complex oxidation behavior showing weight gains and weight losses				
	with temperature- and Mo is degassed by CO	pressure-dependent weight losses. C-containing						
Water vapor	O-H compounds. At lo	At high pressures, oxidation and evaporation of volatile oxides and Mo- O-H compounds. At low pressures, formation of gaseous H ₂ and evaporation of volatile oxides; no surface scales, steady states with						
	20							
	g/cm ²	♦ He-0.5%O2			♦			
	່ຜີ 15 ອີມ 10	XHe-Ar-H2O			×			
	ass char		*	×				
	Final mass change [mg/cm ²] 0 5 01 5 10	*	× ¢					
	ц 4(00 60	8 00	00	10	000		
Temperature [°C]								

Fig. 16. Final mass change for the unalloyed tungsten discs as a function of temperature.

Hydrocarbons	Carbon solution and carbide formation with H_2 desorption	Carbon solution and external and/or internal carbide formation
Inert gas	Reduction of metal evaporation; in case of oxygen-containing impurities, formation of volatile oxides resulting in additional metal losses	Reduction of evaporation of base or alloying metals; in case of oxygen- containing impurities, oxidation processes
Vacuum	Degassing of H and N via H2 and N2 desorption, degassing of C and O via CO formation, degassing of O via oxide evaporation; at high residual pressures, contamination possible	Degassing processes, contamination



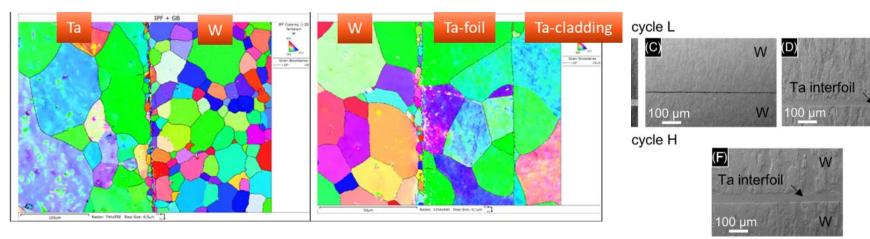
	Tensile testing					
	Fatigue testing				Charpy testing	
	CERN	NLR	Lucideon	SincoTec	Amentum	Nordmetall
Country	Switzerland	Netherlands	United States	Germany	United Kingdom	Germany
Test types	Tensile test & Fatigue test	Tensile, Fatigue & Density	Fatigue & Tensile	Fatigue	Fatigue	Charpy impact
Samples	Open	<mark>6 Samples</mark> 2x Tensile 2x Fatigue 2x Density	<mark>6 Samples</mark> 2x Tensile (RT &HT), 6 x Fatigue (3x air 3x argon)	<mark>30 samples</mark> RT & HT, spread TBD by CERN	20 samples	<mark>46 Samples</mark> 6x Reference 40x Room –> high temperature (~600 ° C)
Sample geometry	Fatigue- L- 135 mm, W 100 mm	Tensile - L100 mm, G 25 mm, W 6 mm Fatigue - L 134 mm, Density- 10 mm x 10 mm x 5 mm (L x W x T)	TBD	L 160 mm, W 30 mm 16 per plate	L- 134 mm, W30 mm	10 mm x 10 mm x 55 mm
Machining	EDM-CERN	EDM-CERN	EDM-CERN	Machined-SincoTec	EDM-CERN	EDM-CERN
Machine parameters	Testing up to <mark>20 Hz</mark>	Testing up to <mark>100 Hz</mark>	Frequency to be determined	Testing up to <mark>80Hz</mark>	Testing up to <mark>20 Hz</mark>	Testing in increasing increments from room temperature to 600 °C
Heating & atmosphere	Room temperature only in atmosphere	Furnace heated in argon oven (Experienced in this testing)	Information not given	Induction heating in argon test chamber. (Experience in this testing)	Furnace heated (including grips) Static argon during test (no experience in this testing)	Information not given
Duration	6 days per sample fatigue	12 days	11-12 weeks	~ 6-7 weeks	<mark>17 weeks</mark>	4 weeks
Price	EDM cost only	<mark>€40, 400.00</mark>	<mark>£23,280.00</mark>	<mark>€51,786.00</mark>	<mark>£104,681.11</mark>	<mark>€4,600.00</mark>

Previous work: Cladding

 HIP has been proven as a valid technique to bond W to Ta and W to W (through a Ta interfoil): erosion-corrosion resistant claddings in representative geometries of the final target

Homogeneous bonding

- With stronger interfaces and theoretical thermal conductivities were achieved in Ta-TZM, Ta-W, and Ta2.5W-TZM. Ta2.5W-W showed 70% of the cladding material's strength [1] [2]
- Checked by NDT, microstructural and mechanical characterization on unirradiated and irradiated components





Ta-cladding by HIPing exhibited reliable and good bonding quality (Ta to W with and without foils and W/Ta/W)

Robust heat transfer from core to cooling water

More studies are needed to complete assessment mechanical strength

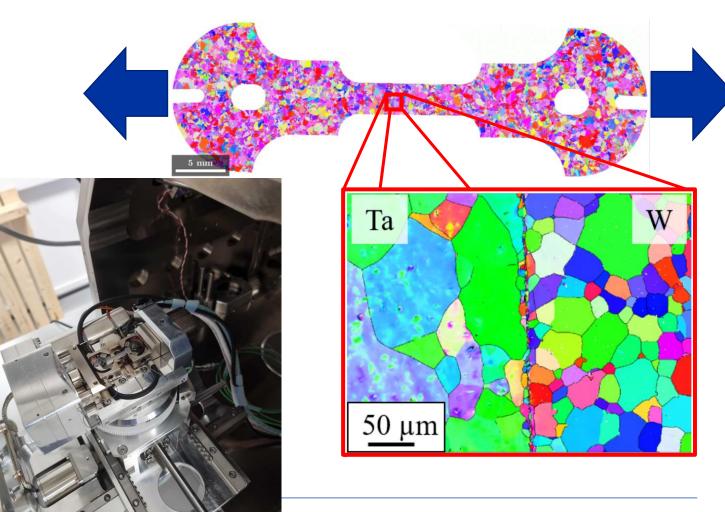
[1] 2018 \rightarrow Recent developments on the application of Hot Isostatic Pressing (HIP) technologies for proton beam targets at CERN with Fraunhofer <u>J. Busom et al.</u> [2] 2024 \rightarrow PIE of a prototype tantalum-clad target in collaboration with Framatome <u>T. Griesemer et al.</u>

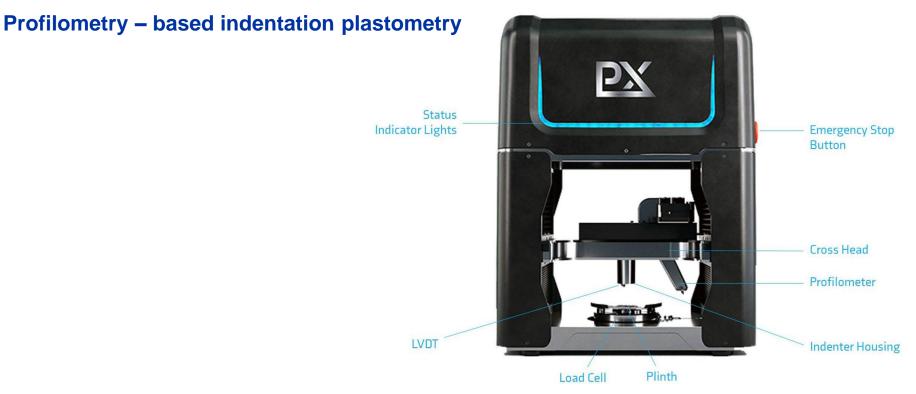


In-situ proposal

- o Collaboration with the University of Manchester, experts in this field
- o Mechanical testing inside SEM
- Well adapted to test interfaces (W/Ta/W)
- **RT and high temperature in-situ**
- **o SEM vacuum environment**
- \circ Cost: ≈ 6000 CHF per test







- The system allows obtaining stress-strain curves (up to uniform elongation) based on indentation + profilometry data
- We (EN-MME) have benchmarked it with a 'blind test' on a variety of materials (stainless steel P506, Nb RRR 300, electroformed Cu) with very consistent results (difference in Rp_{0.2} and Rm < 10%)
- The HT option has been validated for pure tungsten up to 800°C
- Estimated 131 kCHF
- In-SEM also being evaluated for specific tests (collaboration Prof. Kermouche / Ecole des Mines de Saint Etienne) and own equipment (200 kCHF)



17

High T (1200°C) + vacuum Universal Testing System

- Scarce availability
- High (and highly scattered) cost of high cycle fatigue testing at high temperature under vacuum or protective atmosphere
- Equip ourselves with state-of-the-art HT fatigue testing device?
- Two potential suppliers: Instron /UK & MTS/USA
- Both suppliers work with a French partner which is a reference of vacuum furnaces: AET
- Seamless integration with the unit, vacuum environment to avoid oxidation, and the whole specimen is at HT
- The system could eventually be installed in an extension of our current mechanical testing laboratory
- Both machine and vacuum furnace would be in the range of 550 – 600 kCHF, including grips and extensometers





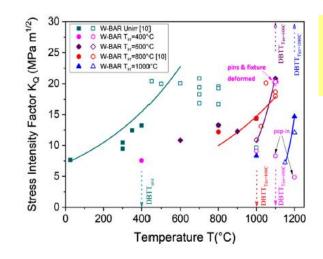
Radiation damage considerations on materials – do we care?

General outcome: increase in the DBTT of irradiated samples

- The increase of the DBTT depends on fluence and irradiation temperature & it is pronounced even for doses as low as ~1 dpa
- Early works report DBTT of sintered unirradiated W ~ 400°C and of irradiated W ~ 600°C (ITER Materials Assessment Report (MAR), ITER Doc. G 74 MA 10 01-07-11 W0.3)
- o Recent works confirm that irradiation strongly impacts the fracture-mechanical behaviour of wrought W irradiated to 1 dpa
- Depending on the irradiation temperature, the DBTT increases by about 650 °C or even higher

DBTT and \triangle DBTT of IGP-W bar. The scattering of unirradiated DBTT values reflects the difference in the DBTTs determined on KLST (10) and on DCT (13) specimens, see text. The results for the unirradiated state and 1dpa@800°C are from (10).

Condition	DBTT (°C)	$\Delta DBTT$ (°C)
Unirradiated	400-450	-
1dpa@400°C	1100	650
1dpa@600°C	≥1100	≥650
1dpa@800°C	1000-1025	600-625
1dpa@1000°C	≥1200	≥750



Gaganidze E., et al (2021). Effect of irradiation temperature on the fracture-mechanical behaviour of tungsten irradiated to 1 dpa, Journal of Nuclear Materials, 556 <u>https://doi.org/10.1016/j.jnucmat.2021.153200</u>

 Tungsten wires irradiated at 0.2, 1, and 10 dpa and tested at RT. No significant change in the ductility of both irradiated and nonirradiated wires was found - Lürbke, R. et al. (2025), <u>https://doi.org/10.1016/j.nme.2024.101858</u>



Conclusions

- The selected metallurgical route, form of product (HR plates), HIP diffusion bonding techniques are promising, based on previous assessments and ongoing tests
- However, an extensive material characterisation and assessment of mechanical properties at intermediate temperature is still needed to support this material choice
- \circ Testing campaigns are extensive, complex and costly \rightarrow consider developing in-house test facilities
- Critical points are the limitation of the thickness: a HIP cycle required, equivalent to an annealing, that might raise the DBTT
- Irradiation hardening and embrittlement are potential concerns (primary degradation phenomena of W):
 - hardness and yield strength increase, ductility loss at high doses, shift of DBTT
 - Large DBTT shift expected even at a low dose of 1 dpa
 - Brittle regime in all temperature range eventually expected, no self-recovery
- Thermomechanical processing techniques, such as rolling and forging and/or additives/dopants can reduce the DBTT of W, even down to RT. Nevertheless, both approaches have proven to be less effective when W is subjected to high-temperature annealing or irradiation



20



home.cern

