Behavior of tungsten under thermal and plasma exposure and development of advanced tungsten materials


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3rd RCM on PWI with irradiated Tungsten and Tungsten Alloys in Fusion Devices, 27 - 30 June 2017, Vienna, Austria
Outline of FZJ contributions

- Thermal shock behavior of irradiated and un-irradiated W grades
- Change of W micro-structure under simultaneous heat and particle loads and impact on W erosion and fuel retention in W
- Development of advanced tungsten materials with improved micro-structure
- Characterization of commercially available tungsten grades
Environmental conditions - test facilities
Environmental conditions

- very high thermal loads
- plasma exposure
- neutrons
Environmental conditions

- very high thermal loads
- plasma exposure
- neutrons
Environmental conditions

**Steady state heat loads:**
- up to 20 MWm\(^{-2}\) in ITER (lower loads in DEMO)
  - recrystallization
  - failure of joints

**Transient thermal loads:**
- up to 60 MJm\(^{-2}\) (disrupt., ELMs, VDEs)
  - crackings
  - melting
  - dust formation

**Plasma loads:**
- sputtering
- hydrogen
- helium

**Neutrons:**
- up to 14 MeV
- defects
- transmutation

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Facilities at FZJ

**Electron beam facility JUDITH 1**
- max. power 60 kW
- acceleration voltage < 150 kV
- EB diameter ~1 mm (FWHM)

**Linear plasma device PSI-2**
- plasma diameter 60 mm
- particle flux ≤ $10^{23}$ m$^{-2}$s$^{-1}$
- incident ion energy (bias) 10 – 300 eV
- Nd:YAG laser 1064 nm
- laser energy 32 J
Low and high pulse number test
Environmental conditions

Expected heat loads in ITER divertor:

- **Disruptions**: $10^2$ W/m², 100 ms, $n \sim 10^6$
- **MGI**: $0.3$ GW/m², 3 ms, $n \sim 3000$
- **ELMs**: $1$ GW/m², 0.5 ms, $n >> 10^6$
- **Divertor**: $5$-20 MW/m², 450 s, $n \sim 30000$

*Ref.*
- J. Linke, Transactions of fusion science and technology 49 (2006) 455-464
Mechanical properties

Tensile tests
- deformation speed: 0.2 mm/min
- deformation rate: $10^{-4}$ s$^{-1}$
Thermal shock - damage mapping

- **Damage threshold**
- **Fracture strain**
- **Yield strength (0.2%)**
- **Base temperature [°C]**
- **Power density [GW/m²]**

1. Fracture strain ≈ 17 %
   - Yield strength (0.2%) ≈ 448 MPa
   - At 500 °C

2. Fracture strain ≈ 34 %
   - Yield strength (0.2%) ≈ 455 MPa
   - At 500 °C

3. Fracture strain ≈ 71 %
   - Yield strength (0.2%) ≈ 150 MPa
   - At 500 °C

- **Surfaces**
  - Transversal
  - Longitudinal

- **Damage states**
  - No damage
  - Surface modification
  - Small cracks
  - Crack network

- **Pulse duration** 1 ms
- **Absorption coefficient**: 0.55
- **100/1000 pulses**
High pulse number tests

Tungsten at high pulse numbers

Surface condition after testing pure W at $T_{\text{surf}} \approx 700 \, ^\circ\text{C}$ (10 MW/m\textsuperscript{2} SSHL)

- **no damage**
- **roughening**
- **small cr**
- **cr network**
- **cr+melting**

**Damage threshold**

- $\Delta t = 0.48 \, \text{ms}$
- $f_{\text{ELM}} = 25 \, \text{Hz}$
- abs. coeff.: 0.55

Th. Loewenhoff et al., Physica Scripta T145 (2011) 014057

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No. 12
High pulse number tests

Tungsten at high pulse numbers

Surface condition after testing pure W at $T_{surf} \approx 700$ °C (10 MW/m² SSHL)

Th. Loewenhoff et al., Physica Scripta T145 (2011) 014057
Institut für Energie- und Klimaforschung, Forschungszentrum Jülich
Investigation of fatigue effects

recrystallization

melting

recrystallization around crack edges

original grain structure

Th. Loewenhoff et al., Fusion Engineering and Design 87 (2012), 1201-1205

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Influence of microstructure

Tungsten at high pulse numbers

Surface condition after testing pure W at $T_{\text{surf}} \approx 700 \, ^\circ\text{C}$ (10 MW/m$^2$ SSHL)

- $\Delta t = 0.48 \, \text{ms}$
- $f_{\text{ELM}} = 25 \, \text{Hz}$
- abs. coeff.: 0.55

![Graph showing power density vs. number of pulses with damage threshold and small cr, cr network markers.](image-url)
Influence of microstructure

Tungsten at high pulse numbers

Surface condition after testing pure W at $T_{surf} \approx 700 \, ^\circ C$ (10 MW/m$^2$ SSHL)

- Damage threshold lower for recrystallized and transversal material

$\Delta t = 0.48 \, ms$

$\lambda_{ELM} = 25 \, Hz$

$\lambda_{abs. \, coeff.} : 0.55$

$F_{HF} [\text{MW/m}^2 \cdot \text{sqrt(s)}]$ vs. number of pulses

$0 \rightarrow 10^7$

Power density [GW/m$^2$]
Combined particle and heat flux exposure of tungsten
Combined tests in PSI-2

- commercially available sintered tungsten product
- representative example:

<table>
<thead>
<tr>
<th>Material Type</th>
<th>Description</th>
<th>Microstructure</th>
<th>Characterization at 1000 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Transversal</td>
<td>50 µm polarized light</td>
<td></td>
<td>fracture strain ≈ 22 %</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>yield strength (0.2%) ≈ 370 MPa</td>
</tr>
<tr>
<td>Longitudinal</td>
<td>50 µm polarized light</td>
<td></td>
<td>fracture strain ≈ 17 %</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>yield strength (0.2%) ≈ 340 MPa</td>
</tr>
<tr>
<td>Recrystallized</td>
<td>50 µm polarized light</td>
<td></td>
<td>fracture strain ≈ 68 %</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>yield strength (0.2%) ≈ 100 MPa</td>
</tr>
</tbody>
</table>

- characterization of the high pulse number thermal shock performance (fatigue) with steady state particle background
High pulse number tests in PSI-2

Laser beam
ELM-like heat loads at 730 °C
absorbed power density: 0.38 GW/m²
pulse duration: 0.5 ms (f = 10 Hz)

H/He (6 %) - Plasma
particle energy ≈ 35 eV
plasma flux ≈ 6.0 \times 10^{21} \text{ m}^{-2}\text{s}^{-1}
fluence ≈ 9.0 \times 10^{24} \text{ m}^{-2} / 6.0 \times 10^{25} \text{ m}^{-2}
High pulse number tests in PSI-2

10,000 pulses

100,000 pulses
Surface roughness and structure

Arithmetic mean roughness ($R_a$)
- significant increase for higher number of pulses (accumulation of plastic deformation)
- high strength/low ductility of the transversal and longitudinal grain orientation leads to severe damage
- lower strength/higher ductility of the recrystallized materials leads to a faster damage evolution but lower $R_a$ values after high pulse numbers

Hill and valley structure after 100,000 pulses
- severe hill and valley structure
- height differences up to 425 µm
- could be an indication for erosion of large parts of the surface (dust formation, plasma contamination)
- enhanced risk of overheating/melting, especially for low angle of incident
Comparison PSI-2 and JUDITH 2

PSI-2 (laser + plasma)  
JUDITH 2 (pure thermal)

100,000 pulses
Comparison PSI-2 (laser + plasma) and JUDITH 2 (pure thermal)

- combination of steady state particle background with transient thermal loads leads to a much faster damage evolution (fatigue) compared to pure thermal ($\Rightarrow$ H/He embrittlement, degradation of mechanical strength)
- effect of lower strength/higher ductility of the recrystallized materials also reflected in the pure thermal results

Th. Loewenhoff et al., Fusion Engineering and Design 87 (2012), 1201-1205
Microstructural changes

10,000 pulses

100,000 pulses

- near surface microstructural changes occur already after 10,000 pulses
- region increases for higher pulse numbers
- sub-grains/grain nucleation can be observed

EBSD Band Contrast Image + Grain Boundaries (≥ 5°)
Microstructural changes

- 10,000 pulses
- 100,000 pulses

EBSD Band Contrast Image + Grain Boundaries (red: 3.5° up to 10°, black: > 10°)

- Microstructural changes also visible for recrystallized material (1600 °C, 1 h)
- Formation of small angle grain boundaries, grain refinement, dynamic recrystallization
- Increase of the effected zone with higher number of pulses
- significant increase of the depth from 10,000 to 100,000 pulses
- depth of the zone depends on the time (number of pulses) and temperature gradient ⇒ saturation for higher number of pulses?
- change of the mechanical properties in a near surface region ⇒ reduced strength/higher ductility like for the recrystallized material?
- impact on the diffusion/retention of H/He not clear ⇒ possibly higher retention as reported in: A. Huber et al. Physica Scripta T167, art. no. 014046 (2016)
Microstructural changes

100,000 pulses

PSI-2 (laser + plasma)

JUDITH 2 (pure thermal)
FIB cuts and He bubbles/layer

10,000 pulses

- He bubbles/layer only visible (in SEM) in the **laser + plasma** exposed area after 10,000 pulses

100,000 pulses

- He bubbles/layer become visible (in SEM) in the **laser + plasma** and **only plasma** exposed area after 100,000 pulses
Size of the visible He bubbles

- size of the visible bubbles increases for higher number of pulses/fluence
- additional transient heat loads accelerate this effect
- impact on the He bubble density not clear

Size of the He effected layer

- depth of the He affected layer increases with number of pulses/fluence
- additional transient heat loads result in an extension of the He affected layer
- higher thermal gradients could lead to a deeper diffusion into the bulk material
D

Tungsten characterization
W monoblock after HHF testing

### SMALL SCALE MOCK-UPS

<table>
<thead>
<tr>
<th>A) 1000 cycles at 10 MW/m²</th>
<th>C) 1000 cycles at 10 MW/m² + 500 cycles at 20 MW/m²</th>
<th>D) 1000 cycles at 10 MW/m² + 1000 cycles at 20 MW/m²</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image1.png" alt="Image" /></td>
<td><img src="image2.png" alt="Image" /></td>
<td><img src="image3.png" alt="Image" /></td>
</tr>
</tbody>
</table>

- no visible defects in tungsten
- small cracks in copper
- recrystallization → enhanced for HRP (≤ 2 mm)
- surface roughening / erosion → enhanced for HIP
- cracking → enhanced for W-sheet / HRP
- recrystallization → HRP (2-4 mm)
- surface roughening / melting → peak/valley of ≤ 500 μm

### VERTICAL TARGET PROTOTYPICAL COMPONENTS (VTPCs)

<table>
<thead>
<tr>
<th>A) 1000 cycles at 10 MW/m²</th>
<th>B) 1000 cycles at 10 MW/m² + 1000 cycles at 15 MW/m²</th>
<th>E) 1000 cycles at 10 MW/m² + 1000 cycles at 15 MW/m² + 300 cycles at 20 MW/m²</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image4.png" alt="Image" /></td>
<td><img src="image5.png" alt="Image" /></td>
<td><img src="image6.png" alt="Image" /></td>
</tr>
</tbody>
</table>

- cracking → W-sheet / HRP® (initially existing damage?)
- cracking → W-rod / HIP®
- recrystallization (2-4 mm)
- surface roughening / erosion → enhanced for W-rod / HIP®
- cracking


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Difference in W Materials

- **Observation**
  - Self-castellation often appeared in W monoblocks used by EU industry

- **Conformity of W material with ITER material specification**
  - Chemical composition: similar
  - Hardness: similar
  - Density: similar

<table>
<thead>
<tr>
<th></th>
<th>W-Plansee</th>
<th>W-Polema</th>
<th>W-ALMT</th>
<th>W-AT&amp;M</th>
</tr>
</thead>
<tbody>
<tr>
<td>HV30</td>
<td>441</td>
<td>443</td>
<td>461</td>
<td>448</td>
</tr>
<tr>
<td>density [g/cm³]</td>
<td>19.25</td>
<td>19.12</td>
<td>19.17</td>
<td>19.25</td>
</tr>
</tbody>
</table>

- Microstructure: different

  N.B. production routes are different (e.g. forged bar vs rolled plates)

- **Microstructure**
  - Strength in y-direction would be different

See supporting data in M. Wirtz, et al. presented at SOFT2012 http://dx.doi.org/10.1016/j.fusengdes.2013.05.07
Recrystallization Sensitivity Tests

- Heat treatment at 1300 °C, 1500 °C, 1800 °C for 1 hour in vacuum
- Test surface yz-plane
- Vickers hardness HV30, microstructure and grain size

Temperature profiles up to 1300, 1500 and 1800 °C for the annealing treatment of the tungsten products

High temperature furnace with the position of the thermocouples.
Vickers Hardness HV30
tested surface xy-plane
temperature treatment for 1 h

xy-plane
Vickers Hardness HV30
tested surface yz-plane
temperature treatment for 1 h

yz-plane

Hardness Measurements

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No. 35
Hardness difference of the xy and yz-plane temperature treatment for 1 h

\[ \Delta(xy/yz)\text{-plane} \]
## Investigated surface xy-plane

<table>
<thead>
<tr>
<th>Temperature</th>
<th>RT</th>
<th>1300 °C</th>
<th>1500 °C</th>
<th>1800 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>ALMT</td>
<td>0.63</td>
<td>0.53</td>
<td>0.62</td>
<td>0.55</td>
</tr>
<tr>
<td>Ansaldo Polema</td>
<td>0.59</td>
<td>0.53</td>
<td>0.59</td>
<td>0.61</td>
</tr>
<tr>
<td>AT&amp;M</td>
<td>0.61</td>
<td>0.62</td>
<td>0.59</td>
<td>0.60</td>
</tr>
<tr>
<td>Plansee (M213)</td>
<td>-</td>
<td>0.59</td>
<td>0.63</td>
<td>0.63</td>
</tr>
<tr>
<td>MMC NSMC</td>
<td>0.62</td>
<td>0.63</td>
<td>0.63</td>
<td>0.65</td>
</tr>
<tr>
<td>STARCK</td>
<td>0.53</td>
<td>0.67</td>
<td>0.62</td>
<td>0.65</td>
</tr>
</tbody>
</table>

### Grain size xy-plane

- **P1**: Blue
- **P2**: Red
- **P3**: Green
- **P4**: Purple
- **P5**: Cyan
- **P6**: Orange

### Microstructural observation

- **RT**: 1800 °C
- **1300 °C**: 1500 °C
- **1500 °C**: 1800 °C

<table>
<thead>
<tr>
<th>Aspect Ratio</th>
<th>ALMT</th>
<th>Ansaldo Polema</th>
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<td>0.55</td>
<td>0.59</td>
<td>0.63</td>
<td>0.62</td>
</tr>
<tr>
<td>1800 °C</td>
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<td>0.66</td>
<td>0.61</td>
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Investigated surface yz-plane

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<tr>
<td>RT</td>
<td>0.50</td>
<td>0.47</td>
<td>0.48</td>
<td>0.50</td>
<td>0.36</td>
<td>0.27</td>
</tr>
<tr>
<td>1300 °C</td>
<td>0.50</td>
<td>0.43</td>
<td>0.53</td>
<td>0.65</td>
<td>0.49</td>
<td>0.61</td>
</tr>
<tr>
<td>1500 °C</td>
<td>0.56</td>
<td>0.69</td>
<td>0.60</td>
<td>0.59</td>
<td>0.57</td>
<td>0.60</td>
</tr>
<tr>
<td>1800 °C</td>
<td>0.54</td>
<td>0.70</td>
<td>0.53</td>
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Summary & Outlook

- Extensive characterization of the thermal shock behavior of W (interaction between material properties and damage behavior)
- Synergistic effects of particle and transient heat loads on thermal shock performance of W (H/He embrittlement, microstructural changes)
- Development of advanced tungsten materials with improved micro-structure
- Characterization of commercially available and new developed W grades

- Selection of W reference materials/samples for n-irradiation
- Thermal shock exposure of W reference materials after n-irradiation/comparison with un-irradiated damage response
- Thermal shock exposure of new developed W grades (e.g. PIM, Wf/W)
- Synergistic effects of particle and transient heat loads on thermal shock performance of reference W and new developed W grades after n-irradiation
- Characterization of the thermal and mechanical properties after n-irradiation