Mechanical Degradation of Tungsten Alloys at Extreme Temperatures in Vacuum and Oxidation Atmospheres

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Extreme conditions

- High ion/neutron flux
- High heat load (~ 10 MW/m²)
- High temperature
- Thermal stresses & cycling
- Off normal events (e.g. plasma disruption)
why tungsten as PFM?

features an **unique property combination**

- the highest melting point of all metals
- good thermal conductivity
- low tritium retention
- low physical sputtering yield
- high thermal resistance
- high temperature strength

at the divertor perform also a structural application, so ductility is needed and W has a **brittleness** problem.

oxidation resistance

\[ + \text{Y}_2\text{O}_3 \]
\[ + \text{V} \]
\[ + \text{Ti} \]
\[ + \text{La}_2\text{O}_3 \]

Operation temperature range

- Lower limit: ductile-brittle transition temperature (DBTT) 400-650 °C
- Upper limit: rxx temperature (RT) 1300 °C

rxx processes and irradiation effects during operation can induce embrittlement

\[
\begin{align*}
W &- 1 \text{ wt}\% \text{ Y}_2\text{O}_3 \quad (W-1Y) \\
W &- 2 \text{ wt}\% \text{ Ti} - 1 \text{ wt}\% \text{ La}_2\text{O}_3 \\
&\quad (W-2T1L)
\end{align*}
\]
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<table>
<thead>
<tr>
<th>Material</th>
<th>Purity</th>
<th>Particle Size</th>
</tr>
</thead>
<tbody>
<tr>
<td>W</td>
<td>99.9%</td>
<td>&lt; 5 µm</td>
</tr>
<tr>
<td>Ti</td>
<td>99.9%</td>
<td>&lt; 110 µm</td>
</tr>
<tr>
<td>La₂O₃</td>
<td>99.5%</td>
<td>0.01-0.05 µm</td>
</tr>
<tr>
<td>Y₂O₃</td>
<td>99.5%</td>
<td>0.01-0.05 µm</td>
</tr>
</tbody>
</table>

Material processed in UC3M, Spain

MA 20h

Canned degassed
400 °C, 24 h

HIP
1300 °C, 2 h, 195 MPa
produced in UC3M, Spain

Ø = 30 mm
L = 50 mm

1.6 x 1.6 x 25 mm³

TPB tests samples

L = 25 mm
B = 1.6 mm
D = 1.6 mm
Lₛ = 16 mm
L = 50 mm

Smooth bend bars

Notched bend bars

HOW?
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- notched samples
- diamond disc: \( \sim 200 \mu m \)
- diamond wire: \( \sim 75 \mu m \)
- fast, plastic damage, big notch root radius
- metal blade: \( \sim 5-7 \mu m \)
- no plastic damage
- slow complex process
notched samples

laser
1-50 nm

- no plastic damage
- no rxx of the tip grains
- similar to a crack

Profilometer image
TPB tests were performed in notched samples of the 4 methods to check the notch root radius effect.

- dispersion results
- 25-30% of the value
W-2T1L:
- with the addition of the alloying elements the grain size become nanometric
- change from polyedral coarse grains (pure-W) to coarse W grains and Ti pools surrounded by a W-Ti-La solid solution

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Y₂O₃ nanoparticles:
- spheroidal dispersoids with variable size
- located in the grain boundaries
- sometimes agglomeration

C. Ballesteros, UC3M

edx spectra from black contrast areas
FESEM image of W-2Ti-1La$_2$O$_3$ alloy with mapping (W), mapping (Ti) and mapping (La).

microstructure (W-2T1L)

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**Mechanical properties**

**Hardness**

- Small influence of the load
- Comparable results with instrumented method
- W-2T1L ↑ hardness

**Young modulus**

- Comparable values
- W-1Y ↓ E
- W-2T-1L Small increase of the values

*IET= Impulse Excitation Technique
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TPB tests

- **displacement rate:** 100 µm/min
- **heating rate:** 50 °C/min

- **inert atmosphere (vacuum)**
  - T ≤ 1200 °C; 10⁻⁶ mbar

- **oxidation atmosphere (air)**
  - T ≤ 1000 °C

- **liquid N immersion**
  - T = -196 °C
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TPB tests – flexural strength

linear elastic until failure (except 1200 °C)
slightly influence of the atmosphere
↑ flexural strength values (W-2T1L)
thermal degradation – W-2T1L T>1000 °C
TPB tests – flexural strength

Sample tested a 1200 °C (not break) intergranular progress of the crack
TPB tests - fracture toughness

Representative curves for some W-2T1L tensile tests

linear elastic until failure (all the T range)
improve of the behavior: ↑T (W-1Y), all T (W-2T1L)
Fracture surfaces of the tested samples at low (up) and high (down) temperature

- Pure-W
- W-1Y
- W-2T1L

flat, fracture decohesion by grain boundary
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W-1Y 1000 °C, air

Bimodal morphology of grains:
- polyhedral coarse grains of W (surrounded by oxide needles)
- rounded smaller grains with dispersion of Yttria nanoparticles
• Powder route was used to process W-alloys with additions of $Y_2O_3$, Ti and $La_2O_3$.

• TPB tests were perform to obtain flexural strength, yield strength and fracture toughness (-196 to 1200 °C, air & vacuum). Moreover nanoindentation, Vickers and IET tests (H, E).

• Laser notching of the samples show a notch like crack with a root radio between 1-50 nm and less dispersion of the results.

• W-1$Y_2O_3$:
  • Slightly enhancement of properties at high T, but increased porosity.
  • Microstructure - Yttria nanoparticles located in the grain boundaries and bimodal morphology of grains.
  • Fracture analysis - intergranular breakage between grain boundaries (support the brittle behavior of the performed tests up to high T).

• W-2Ti-1$La_2O_3$:
  • The porosity slightly decreases and the grain size became nanostructured.
  • Mechanical properties according to TPB tests increase. The values remain stables in all the temperature range (exc. 1200 °C, ductile).
  • Fracture surfaces remain flat and show intergranular breakage between grain boundaries. Supported by brittle behavior of the performed TPB tests.
Thank you for your attention!
Pure-W is highly reactive with oxygen above 400 °C, but with the addition of the alloying elements (Ti, La$_2$O$_3$), the oxidation processes decreased (W-1Y, W-2T1L).

- The outer yellow scale remain thinner and the mass gain is lower in all the temperature range.
- At 1000 °C some cracks appears in the scale as a consequence of the stresses produced during oxide growing.