Creep Behavior of Austenitic Steel 316L in Low Oxygen-Containing Pb-Bi Eutectic at 450-550°C

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Austenitic 316L steel in nuclear power plants

- Construct pipes, vessels and in-vessel components in lead-cooled fast reactor, MYRRHA ADS system (heat exchanger, vessel, diafragm, core barrel)

- Corrosion impact the wall thickness in LBE (load-bearing capability) and effect of elevated temperatures on mechanical properties

- No industrial experience of lead alloy-cooled technology (the lead-cooled fast reactor – LFR). But many concepts worldwide. Mechanical properties (incl. creep) were considerably studied in air, while information about effect of HLM (Pb, LBE) on creep is still limited.

- Goal of the current work: To evaluate impact of lead alloys at 450-550°C on mechanical behaviour and to determine links between creep resistance and microstructural evolutions.
Creep-rupture tests in oxygen-controlled heavy liquid metal

Continuous control of oxygen concentration in HLM
- Oxygen activity close to the specimen with air/Pt O₂-sensor
- λ-probes in gas -inlet and -outlet
- Automatic introducing of gas with variable pO₂ (Ar, Ar/H₂ and synthetic air)

Conditions:
- Stagnant Pb or LBE (900 ml)
- T_max = 650°C
- c₀^{max} = c_{HLM}^{saturation}
- c₀^{min} = 10^{-13} mass%

CRISLA Facility:
- 5 capsules for HLM
- 3 capsules for air (gas)
**Tested material: austenitic 316L steel**

<table>
<thead>
<tr>
<th>316L</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe</td>
</tr>
<tr>
<td>bal</td>
</tr>
</tbody>
</table>

Lamellae texture enriched in Cr, slightly in Mo and depleted in Fe, Ni in comparison with the average composition of 316L
## Tests on 316L austenitic steel

<table>
<thead>
<tr>
<th>Experiments</th>
<th>Status</th>
</tr>
</thead>
<tbody>
<tr>
<td>LBE, $c_0 = 10^{-7} - 10^{-10}$ mass%, 550 °C/ 300 MPa</td>
<td>$t_R = 1,060$ h</td>
</tr>
<tr>
<td>LBE, $c_0 = 10^{-6}$ mass%, 550 °C/ 300 MPa</td>
<td>$t_R = 182$ h, 231 h, 365 h</td>
</tr>
<tr>
<td>Air, 550 °C/ 300 MPa</td>
<td>Stopped before rupture, $t_R &gt; 1,150$ h</td>
</tr>
<tr>
<td>LBE, $c_0 = 10^{-8} - 10^{-9}$ mass%, 500 °C/ 325 MPa</td>
<td>$t_R = 5,025$ h</td>
</tr>
<tr>
<td>Air: 500 °C / 325 MPa</td>
<td>Test still running, $t_R &gt; 9,015$ h</td>
</tr>
<tr>
<td>LBE, $c_0 = 10^{-8} - 10^{-9}$ mass%, 450 °C/ 375 MPa</td>
<td>Test still running, $t_R &gt; 3,795$ h</td>
</tr>
<tr>
<td>Air, 450 °C/ 375 MPa</td>
<td>Test still running, $t_R &gt; 2,425$ h</td>
</tr>
</tbody>
</table>
Creep-to-rupture in LBE at 450-550°C

The higher T, the shorter secondary creep zone and $t_R$
Creep-to-rupture at 550°C and 300 MPa

- $\dot{\varepsilon}_s$ up to one order higher in LBE than in air.
- The transition time from secondary to tertiary zone $t_{2,3}$ determines $t_R$: $t_R$ is shorter in LBE than in air even if $\dot{\varepsilon}_s$ in LBE similar to that in air.
- $c_o = 10^{-6}$ mass% shows no improvement in comparison to lower $c_o$. 

$\varepsilon = 3.19 \times 10^{-2} \text{ (%×h}^{-1})$

$\varepsilon = 1.38 \times 10^{-2} \text{ (%×h}^{-1})$

$\varepsilon = 1.64 \times 10^{-3} \text{ (%×h}^{-1})$

$\varepsilon = 1.7 \times 10^{-3} \text{ (%×h}^{-1})$
Creep-to-rupture at 500°C and 325 MPa

- $\dot{\epsilon}_s$ is one order higher in LBE than in air
- $t_{2,3}$ is smaller in LBE than in air → shortage of $t_R$ in LBE in comparison to air

Strain (%) vs. Time (h)

$LBE$: $c_s = 10^4 - 10^5$ mass%

$\dot{\epsilon}_s = 3.1 \times 10^{-4}$ (\%×h$^{-1}$)

$\dot{\epsilon}_s = 2.4 \times 10^{-5}$ (\%×h$^{-1}$)
Creep(-to-rupture) at 450°C and 375 MPa

\[ \varepsilon_s = 1.1 \times 10^{-6} \% \times h^{-1} \]

\[ \varepsilon_s = 3.5 \times 10^{-5} \% \times h^{-1} \]

- \( \varepsilon_s \) is one order higher in LBE than in air
Structural study of 316L tested in LBE

- 316L after LBE for 365h at 550°C, $c_0=10^{-6}$ mass% and 300 MPa as a sample

Typical image of the surface with a ferrite zone

- Steel feature of 316L in LBE under stress: The closer to rupture surface, the thicker the ferrite zone
- (Almost) No ferrite close to screw thread

Corrosion extending inwardly the steel without ferrite zone (exception)
Structural study of 316L tested in LBE

- 316L after LBE for 365h at 550°C, c_o=10^{-6} mass% and 300 MPa as a sample

Typical image of the surface with a ferrite zone

- Oxides can form in LBE close to the steel surface and in the ferrite zone filled with Pb-Bi

Oxides at steel/ferrite interface (exception) and in the ferrite zone

- Steel
- Pb-Bi in Ferrite
- LBE
- Ferrite
- No ferrite

(Fe_{a}Cr_{b})_{2}O_{3}
(Fe_{x}Cr_{y})_{2}O_{3}
Structural study of 316L tested in LBE at 550°C

Stagnant LBE, $c_o=10^{-6}$ mass%, 365 h and 300 MPa

Flowing LBE, $c_o=10^{-7}$ mass%; 2,011 h and 0 MPa (CORRIDA)

Pit-type corrosion damages as a result of selective leaching of the steel elements [V.Tsisar, C.Schroer, KIT]

Diverged cracks propagated in ferrite zone towards the steel and filled with Pb-Bi is a feature of 316L under stress in LBE

Cracks in ferrite zone filled with Pb-Bi
Structural study of 316L tested in LBE

- Ductile rupture mode
- Transformation of austenite into ferrite due to selective-leaching steel elements into LBE at 500-550°C independently on $c_0$ ($\leq 10^{-6}$ mass%)
- The austenite-to-ferrite transformation is characterised with a shortage of $t_R$ in comparison to air.
- Cracks found not only in ferrite but also in the steel. Therefore rupture topography can contain from one up to multiple rupture planes.

Ductile-dimple rupture mode at RT in air [Structural Alloys for Power Plants, ed. by A. Shirzadi & S. Jackson, 2014]
## Structural study of 316L tested in LBE

<table>
<thead>
<tr>
<th>T / °C</th>
<th>Enviroment</th>
<th>Co, mass% / tR, h</th>
<th>Strain / %</th>
<th>Reduction of area / %</th>
<th>Thickness of ferrite / μm</th>
</tr>
</thead>
<tbody>
<tr>
<td>550</td>
<td>LBE</td>
<td>10^{-6} / 231</td>
<td>14-22</td>
<td>32</td>
<td>0-86</td>
</tr>
<tr>
<td></td>
<td></td>
<td>10^{-6} / 365</td>
<td></td>
<td></td>
<td>0-37</td>
</tr>
<tr>
<td></td>
<td></td>
<td>10^{-7-10^{-10}} / 1,059</td>
<td></td>
<td></td>
<td>0-326</td>
</tr>
<tr>
<td></td>
<td>air¹</td>
<td>1,025</td>
<td>3</td>
<td>10</td>
<td>-</td>
</tr>
<tr>
<td>500</td>
<td>LBE</td>
<td>10^{-8-10^{-9}} / 5,025</td>
<td>12</td>
<td>43</td>
<td>93-200</td>
</tr>
<tr>
<td></td>
<td>air²</td>
<td>after 7,729</td>
<td>0.4</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>RT</td>
<td>air</td>
<td>50-70</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

¹ - stopped; ² - running

- Strain at rupture at 500-550°C in LBE is significantly less than at RT. Reduction of area increases in LBE with lowering of T. But ductile-dimple rupture mode of austenitic 316L steel is remains in LBE at 500-550°C.
- Ferrite zone at lower T, c₀ and longer tᵣ is more regular distributed and it is thicker than at c₀=10^{-6} mass% (at 550°C)
316L tested in LBE

**Conclusion:**

- 316L austenitic steel degrades stronger in LBE than in air at 500 and 550°C due to selective leaching of the steel elements leading to:
  
  (i) austenite-to-ferrite transformation at steel/liquid metal interface and
  
  (ii) penetration of liquid metal into ferrite and cracks formed into the ferrite and propagated further into the steel

- $c_o=10^{-6}$ mass% (at 550°C) does not decrease LBE effect in comparison to lower $c_o$.

**Acknowledgment:**

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Functionality check of Pt/O-sensor

- Pt/O-Sensor after creep-to-rupture test with 316L in LBE at 550°C, \( c_o = 10^{-6} \text{ mass\%} \), \( t_R = 365 \text{ h} \)

- Oxygen sensor output (V)

- Volume flow (Nccm/min)

- Concentration of solved oxygen (mass\%)

- Time (h)

- Temperature (°C)

- Activation stage

- \( \lambda \) probe

- Liquid metal