MATERIAL TESTING IN H₂ GAS

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MATERIAL TESTING UNDER HYDROGEN GAS AT CEA/LITEN

OVERVIEW
Uniaxial loading under H$_2$ < 350 bar and < 300°C

Disc/tube test under H$_2$/He < 1000 bar - Impurities

H$_2$/D$_2$ gas permeation tests < 5 bar and < 300°C

Permeation cell
PRESSURE VESSEL
+
SERVO-HYDRAULIC TEST FRAME

• MTS servo-hydraulic test frame (250 kN)
• Custom vessel
• Frontal opening
PRESSURE VESSEL : OVERVIEW

Specific design to allow thermal expansion while remaining aligned

Specific device to screw the door at nominal torque without damaging the load line

Device for pressure vessel alignment
## Pressure vessel

<p>| | |</p>
<table>
<thead>
<tr>
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<tbody>
<tr>
<td>Maximum volume available</td>
<td>5,5L + use of dedicated tool to reduce the gas volume</td>
</tr>
<tr>
<td>Max pressure (MPa)</td>
<td>35</td>
</tr>
<tr>
<td>T (°C)</td>
<td>RT - 300</td>
</tr>
</tbody>
</table>

## Test frame

<p>| | |</p>
<table>
<thead>
<tr>
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<tbody>
<tr>
<td>Device maximum load</td>
<td>50 kN</td>
</tr>
<tr>
<td>Displacement</td>
<td>~100 mm</td>
</tr>
<tr>
<td>Min displacement rate</td>
<td>~1 µm/s</td>
</tr>
<tr>
<td>Fatigue test</td>
<td>R &gt; 0</td>
</tr>
<tr>
<td>Frequency</td>
<td>Tested &lt; 10Hz under H₂</td>
</tr>
</tbody>
</table>
PRESSURE VESSEL : CROSS SECTION

- Material vessel: SS 316L
- Stud
- Front door
- Moving rod
- Water cooling
- Gas-tight ducting for instrumentation
- Seals

Furnace
PRESSURE VESSEL: CROSS SECTION
PRESSURE VESSEL : SEALS

3 O-ring dynamic seals

Monitoring
Pumping
Security alert

Static seals

Kalrez seals
(perfluoroelastomer)

SS S-type seals
(temperature)
Strain gages COD or extensometers

- Signal drift during long term tests under hydrogen pressure (> few hours)
- Not stabilised after 2 days under 30 MPa $H_2$
- Slow drift: not critical for cyclic loadings
- WOL test (with in-situ precracking) not possible
External load cell (100kN) : friction between rod and seals estimated < 300 N
=> detrimental for tests below ~10 kN

Development of an internal 50kN load cell (strain gages protected from H₂ gas)
no drift
very sensitive to alignment and clamping
- H₂, N₂
- Remote control and monitoring
- Pneumatic valves
- H₂O et O₂ sensors: not effective for low ppm (< 10 ppm)
  - Hygrometer: capacitance principle using a gold/alumina oxide probe
  - Trace Oxygen Analyser: Micro fuel cell (electrochemical galvanic device)

⇒ Tests under H₂: N60 purity gas

Always the same procedure including: [Vacuum / N₂] (x 3) before H₂ inlet
Sample preparation:
- traction / LCF: surface roughness
- crack growth / toughness: precrack under air

Gas management procedure to ensure gas purity
- \(N_2\) pressure at testing pressure to check tightness
- \(H_2\) pressure
- Tests using MTS (MPT or specific procedure)
- After testing: \(H_2\) sweeping out, \(N_2\) inlet, vacuum, 1 bar \(N_2\)

External load = \(f(P)\)
Remote control – No access under H₂
Automated gas management procedure
Security camera
Min venting flow rate

Seal tightness control
H₂ and O₂ sensors
2 H₂ thresholds
10 % Inferior flammability limit in air
20 % Inferior flammability limit in air

Automated safety procedure

Shut down all electrical power in test room
→
H₂ sweeping out
N₂ inlet
Max venting flow rate in the room

PRESSURE VESSEL: SAFETY ISSUES

MTS
Gas management
### MAIN ADVANTAGES
- Available volume (+ use of tools to reduce gas volume) / instrumentation
- Remote gas management
  - “Fast” atmosphere switches
- Reliable: 6 years old – No major trouble
- Precise measurement from low to high loads
- Load cell: no drift under H₂
- Alignment: ~ straight crack fronts

### MAIN DISADVANTAGES
- Test in temperature: thermal inertia
- Test preparation: ~3 hours, alignment
- Gas purity analysis
- Pressure below 35 MPa / Low P accuracy below 2 bar

### FUTURE IMPROVEMENTS
- Gas purity analysis
- Extensometer with strain gages: signal drift → LVDT?
- Crack initiation detection from small defect (calibrated hole)
- Towards 100 MPa?
DISK PRESSURE TESTS
DISC PRESSURE TEST : SET-UP

Compressed air
He
H₂

Remote control

H₂O
Remote control

H₂, He: Up to 100MPa, Tmax = 100°C

2 cells

Pressure rate: up to 1000 bar/min above 0.1 bar/min

Controlled pressure rate

Working with impure gas (dedicated cell)
O₂
H₂O (bubbler up to 100°C)
(from few ppm to 5000 ppm)
Including long time steps
DISK PRESSURE TESTS

SEALS

- O-ring (elastomer) at high rates
- Indium at low rates

WATER CONTENT CONTROL

- Dedicated H₂ + H₂O gas bottle
- Using bubbler
- Measuring P and T
- Assuming saturated gas
DEVICE ADAPTED TO TUBE PRESSURE TESTS

Long time range tests
under H$_2$ pressure (few months)
DISC PRESSURE TESTS

Advantages

- Low volume
- Easier to proceed
- Dedicated cell for impure gas
- Fast gas switches (few seconds)
- Closer to service-life loadings

Disadvantages

- Less instrumented
- More difficult to analyze mechanically (use of FEM)
- Boundary conditions
- Small thickness

Improvements

- Measuring in-situ deflection
- Developing tests under fatigue (cycling pressure) with / without defects
**H₂/D₂ GAS PERMEATION DEVICE**

- **Mass spectrometer**

20°C < T < 300°C  
Pabs < 5 bar

- Diffusion coefficient
- Solubility
- Traps (binding energy)
- Oxide layer

**Developments**

Permeation under tensile loading  
Influence of hydrostatic stress gradient
THANK YOU