Corrosion Testing of High Temperature Materials in Supercritical Carbon Dioxide

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Supercritical CO₂ Power Cycles  
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Organisational experience
- Carleton University and Natural Resources Canada
- 100 MW\textsubscript{e} and 10 MW\textsubscript{e} designs, 250 kW\textsubscript{th} test loop

Corrosion test rig
- Design
- Commissioning

Corrosion testing
- Initial testing performed
- Weight change results
- Metallography
Organisational Experience

- Mechanical and Aerospace Engineering, Carleton University
  - Strong history of research and teaching in gas turbine technology

- Natural Resources Canada (NRCan), CanmetENERGY
  - Ottawa Research Centre
    - R&D in clean fossil fuel technologies
    - Pilot-scale research facility

- Design and development of advanced semi-closed and closed gas turbine cycles
Organisational Experience

100 MW_e plant 2006/07 to 2010/11 (full-scale power plant)

10 MW_e turbomachinery 2011/12
Organisational Experience

250 kW\textsubscript{th} pilot-scale Brayton cycle loop 2012/13 to 2015/16
Corrosion Testing

- Material compatibility in S-CO$_2$
- Identified as a technical concern
  - Design
    - Corrosion allowance
    - Geometry and property changes
  - Materials selection
    - Piping, heat exchangers, turbomachinery, valves
- “Minimal” data exist, especially for long term exposure and “real” conditions
Corrosion Test Rig Design Features

- Based on MIT test rig
- 700 °C at 25 MPa or 750 °C at 15 MPa
- Continuous flow (0.5-15 L/hr) and static capability
- 12.7 mm diameter test coupons
- Alumina specimen boat
- Near-autonomous operation
- Passive and active safety systems
- TSSA certification
Corrosion Test Rig
Commissioning and Calibration

- Initial runs at 15 MPa and 2.0 L/hr
- 200 °C/hr to 600 °C
- Metal and S-CO₂ temperatures measured
  - Single-point probe (T-2)
  - Profile probe (T-13 to T-18)
Commissioning and Calibration

- Heat shields added
  - Crimped to thermocouples
  - Perforated for flow
- Increased temperature
- Increased temperature uniformity along heated zone
Commissioning and Calibration

- Furnace set points determined
  - Operation at 15 and 25 MPa
  - 400 to 750 °C in 50 °C increments
  - Maximum temperature variation approximately 10 °C
    - For 550 °C testing: 549-557 °C
    - For 700 °C testing: 696-706 °C
Test Design

- 3 alloys: 316SS, IN718, IN738 (and IN625 riders)
  - 12.7 mm diameter samples
  - High surface area to volume ratio
- 550 and 700 °C, 15 and 25 MPa
  - 100, 250, 500, 1000, 1500 hrs
  - Samples weighed and photographed, one removed
- Analyses
  - Surface analysis
  - Weight change
  - SEM and EDS
## Test Matrix

<table>
<thead>
<tr>
<th>Temp (°C)</th>
<th>316SS</th>
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<th>IN718</th>
<th></th>
<th>IN738</th>
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<tbody>
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</tr>
</tbody>
</table>

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S-CO₂ Flow →

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Test Results

Supercritical CO₂ Corrosion Test Results

- Solid lines: 15 MPa/1500 hrs + 25 MPa/1500 hrs
- Dashed lines: 25 MPa/1500 hrs
- Dotted lines: IN625 riders

- 550 °C
- 316SS and IN718 had similar weight gains
- IN738 had approximately half the weight gain
- IN625 had intermediate weight gains
- Logarithmic rate laws
- Some dependence on pressure

- 700 °C
- IN718 and IN738 had similar weight gains
- 316SS had dramatically higher weight gains
- IN625 had lower weight gain (pre-exposure, relative to 550 °C testing)
- Parabolic rate laws for IN718 and IN738
- Linear rate law for 316SS
- Some dependence on pressure
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Supercritical CO$_2$ Corrosion Test Results

- Solid lines: 15 MPa/1500 hrs+25 MPa/1500 hrs
- Dashed lines: 25 MPa/1500 hrs
- Dotted lines: IN625 riders

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700 °C
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Supercritical CO\text{2} Corrosion Test Results

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- **700 °C**
  - IN718 and IN738 had similar weight gains.
  - 316SS had dramatically higher weight gains.
  - IN625 had lower weight gain (prior exposure, relative to 550 °C testing).
  - Parabolic rate laws for IN718 and IN738.
  - Linear rate law for 316SS.
  - Some dependence on pressure.
Test Results

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  - Linear rate law for 316SS
  - Some dependence on pressure
Test Results

- SEM on samples tested at 700 °C for 1500 hrs
- 316SS: unstable, duplex oxide layer
- IN718 and IN738: thin, stable oxide layer
  - Some evidence of intergranular corrosion
Summary

- 316SS comparable to others at 550 °C
- 316SS at 700 °C
  - linear oxide growth kinetics
  - unprotective duplex oxide layer
- IN718 and IN738 at 700 °C
  - parabolic oxide growth kinetics
  - thin and stable oxide layer
  - some evidence of intergranular corrosion
Questions?

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