Carleton UNIVERSITY

Corrosion Testing of High Temperature Materials in Supercritical Carbon Dioxide

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Supercritical CO_2 Power Cycles
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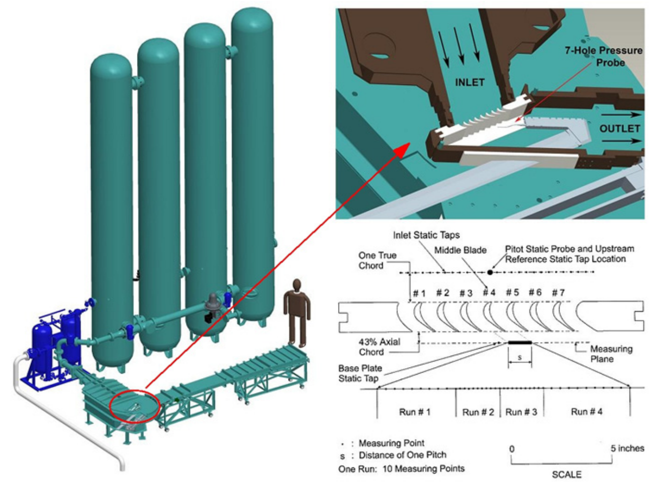
Outline

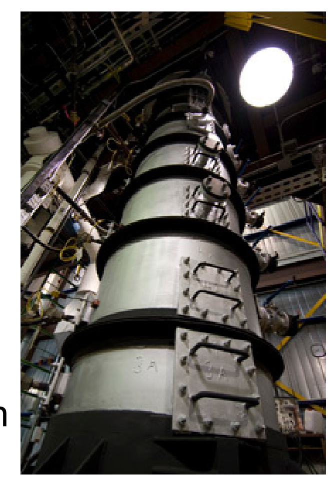
- Organisational experience
  - Carleton University and Natural Resources Canada
  - 100 MW_e and 10 MW_e designs, 250 kW_{th} test loop
- Corrosion test rig
  - Design
  - Commissioning
- Corrosion testing
  - Initial testing performed
  - Weight change results
  - Metallography

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

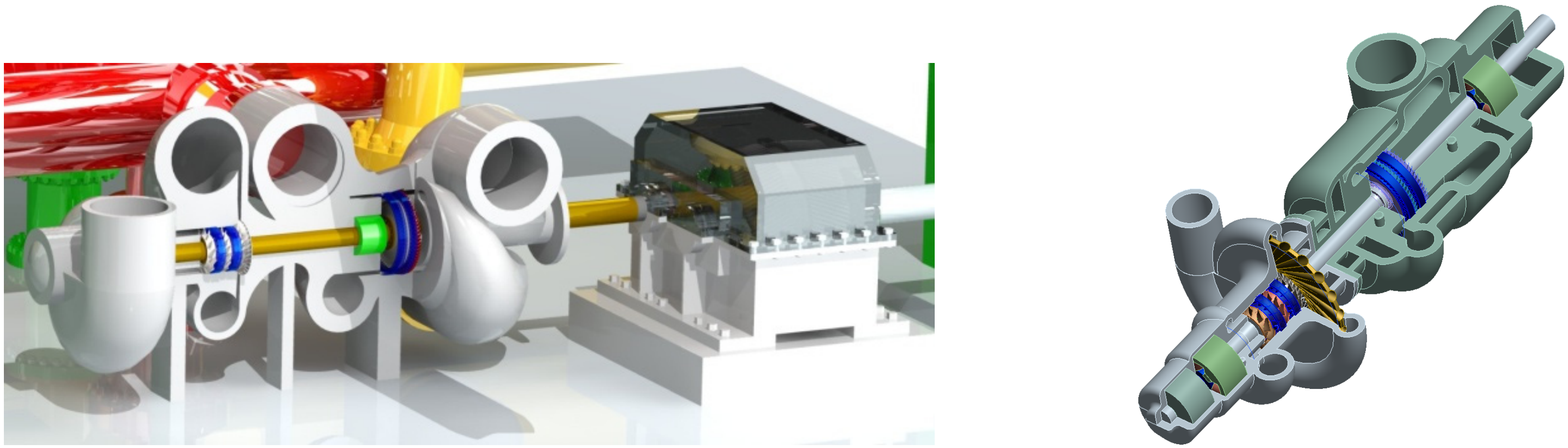


Pratt & Whitney Canada High-Speed Wind Tunnel

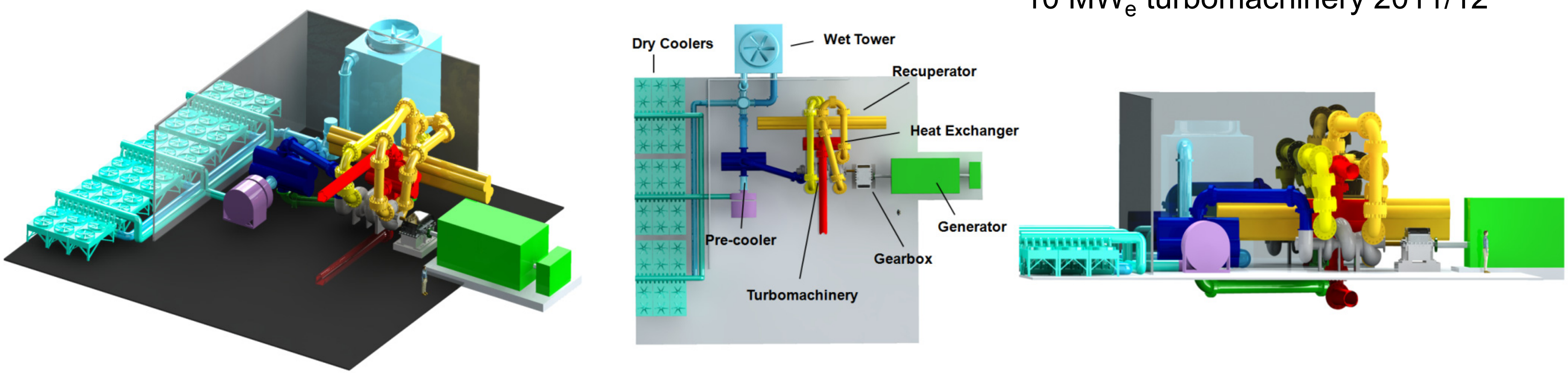


NRCan's Vertical Combustor Research Facility (VCRF)

Organisational Experience

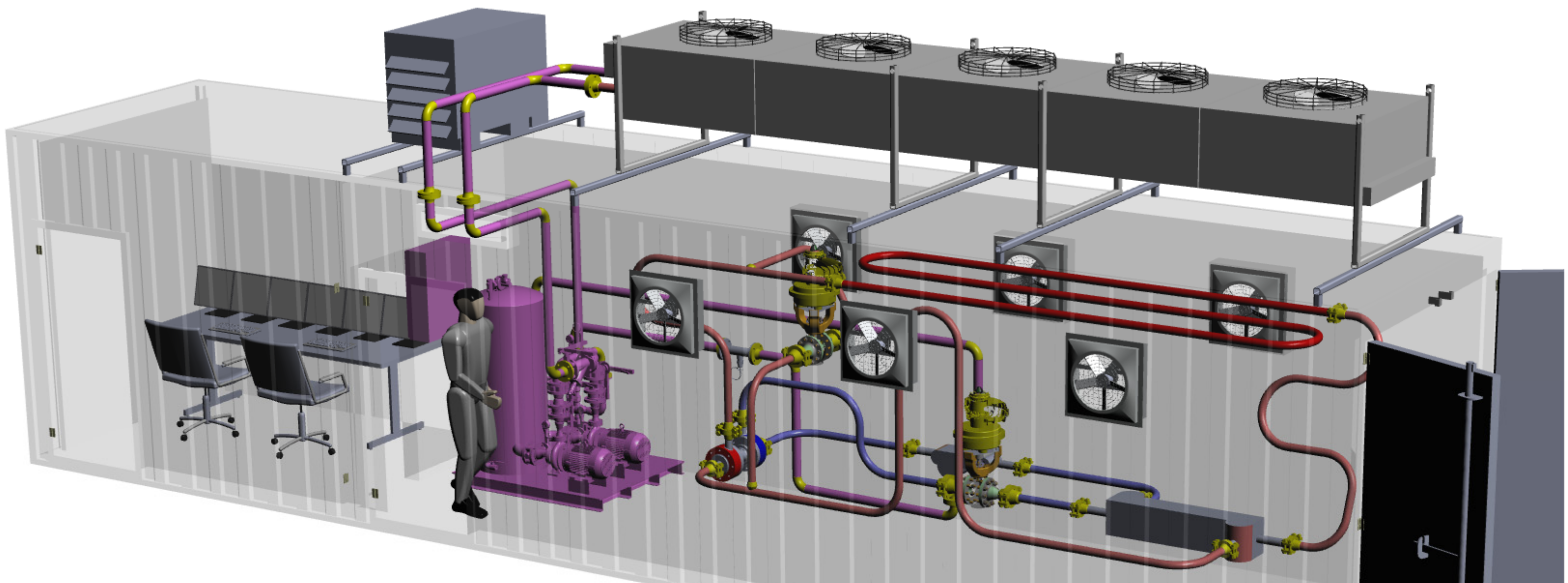


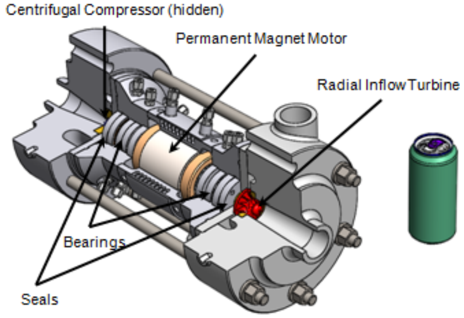
10 MW$_e$ turbomachinery 2011/12



100 MW$_e$ plant 2006/07 to 2010/11 (full-scale power plant)

Organisational Experience





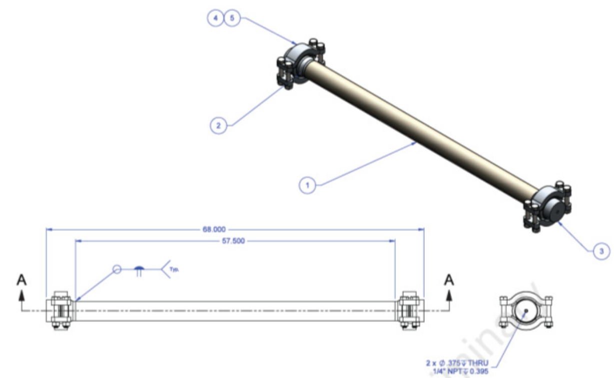
250 kW$_{th}$ pilot-scale Brayton cycle loop 2012/13 to 2015/16

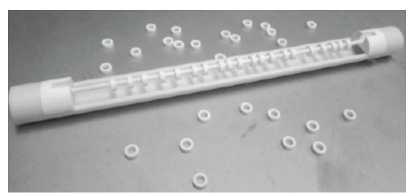
Corrosion Testing

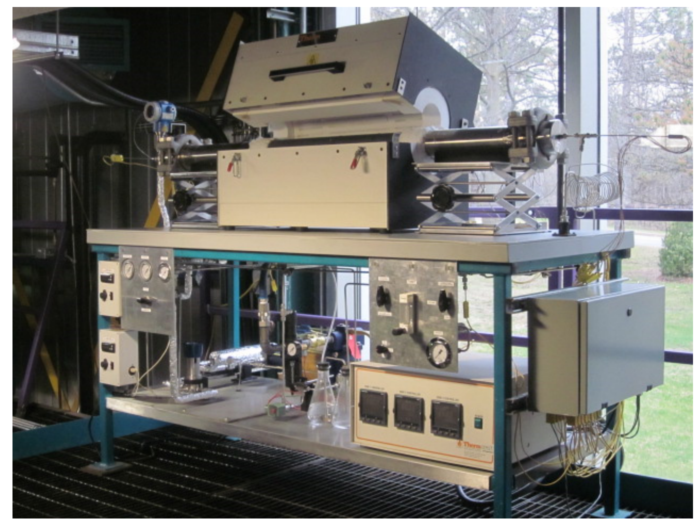
- Material compatibility in S-CO_2
- Identified as a technical concern
  - Design
    - Corrosion allowance
    - Geometry and property changes
  - Materials selection
    - Piping, heat exchangers, turbomachinery, valves
- "Minimal" data exist, especially for long term exposure and "real" conditions

Corrosion Test Rig Design Features

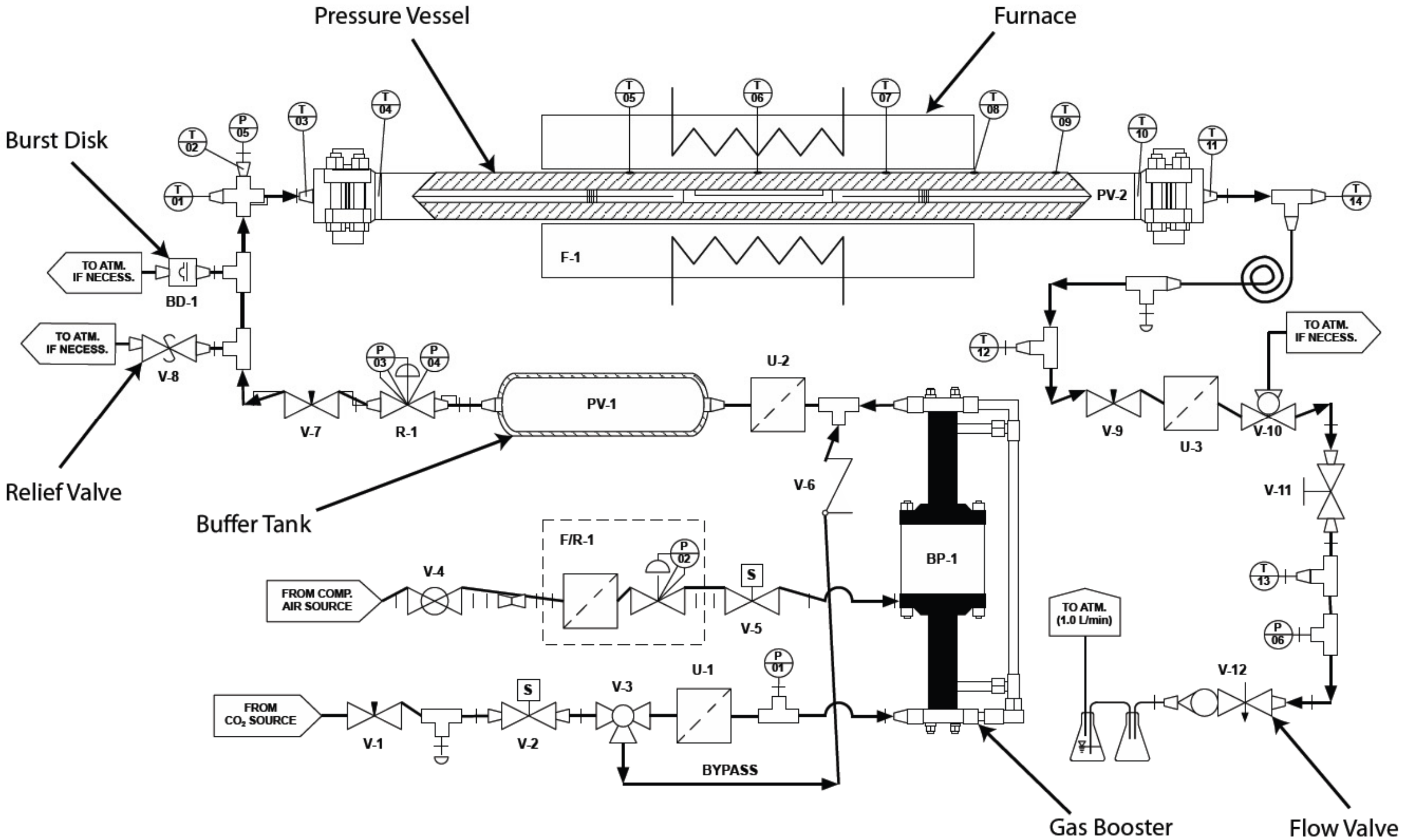
- Based on MIT test rig
- 700 °C at 25 MPa or 750 °C at 15 MPa
- Continuous flow (0.5-15 L/hr) and static capability
- 12.7 mm diameter test coupons
- Alumina specimen boat
- Near-autonomous operation
- Passive and active safety systems
- TSSA certification



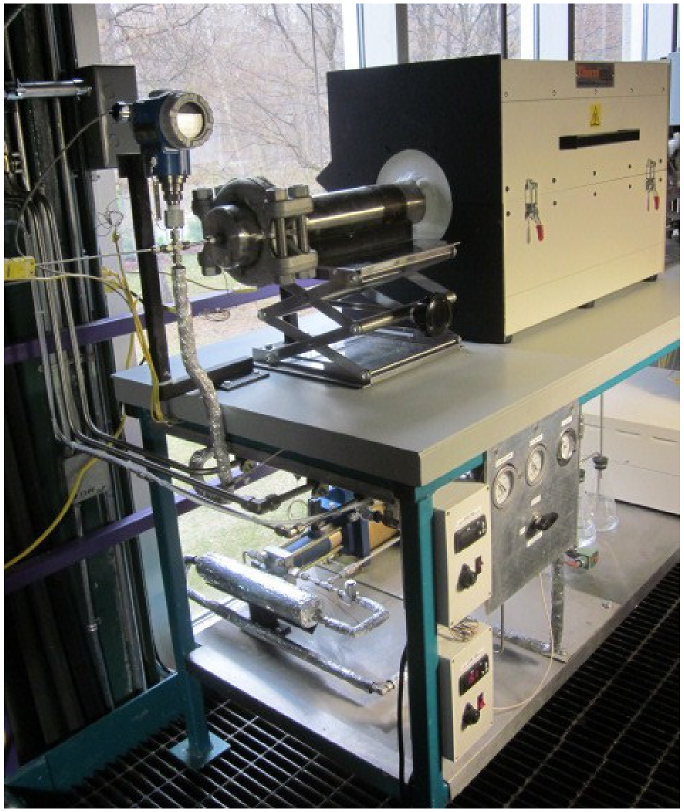


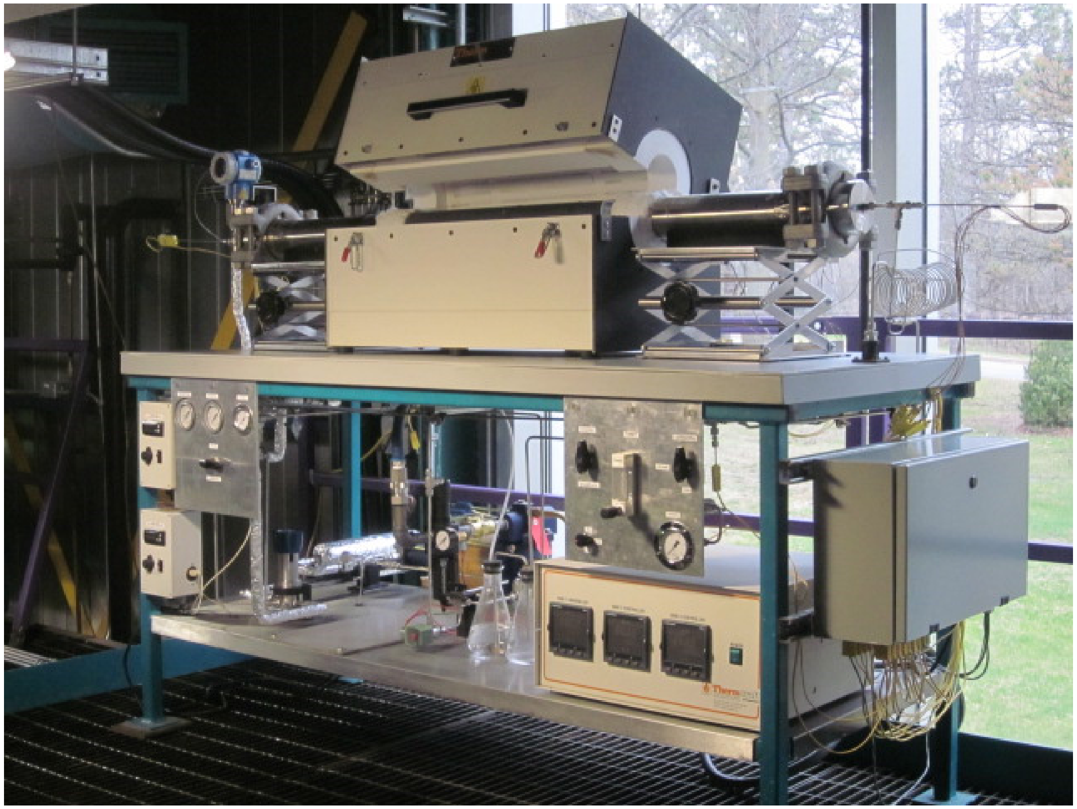


Corrosion Test Rig P&ID

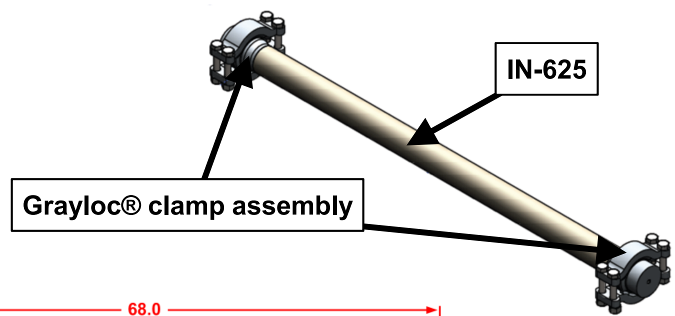


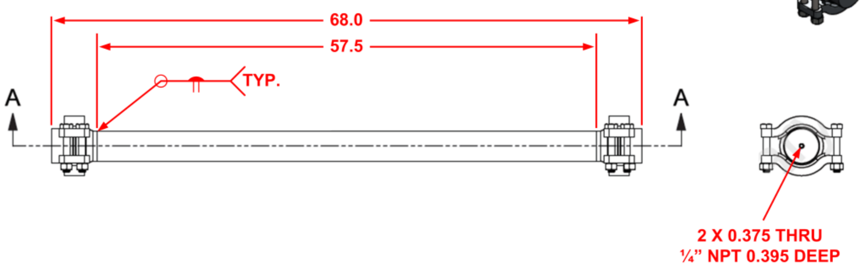
Corrosion Test Rig

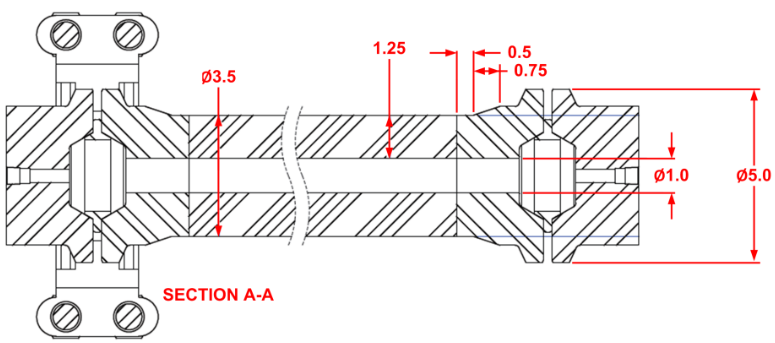


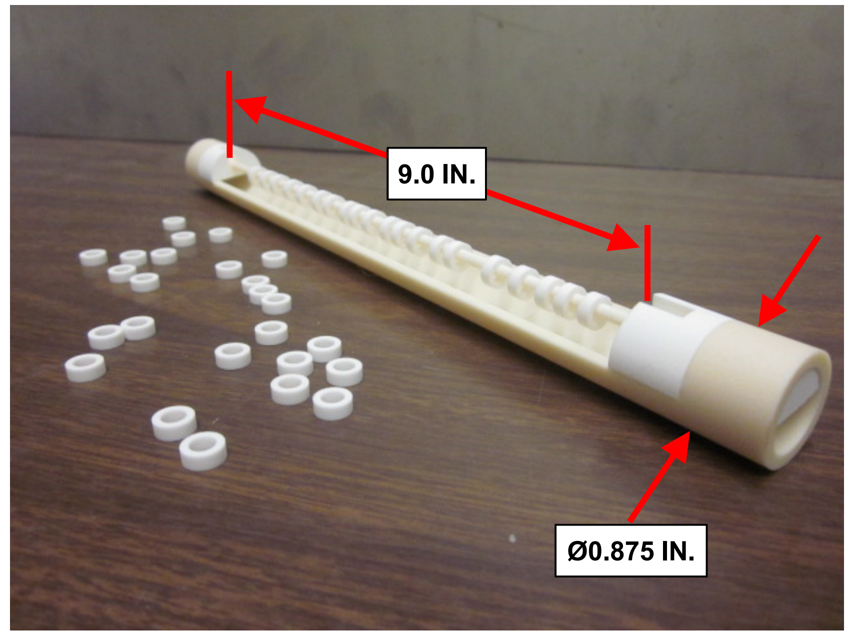


Pressure Vessel and Specimen Boat



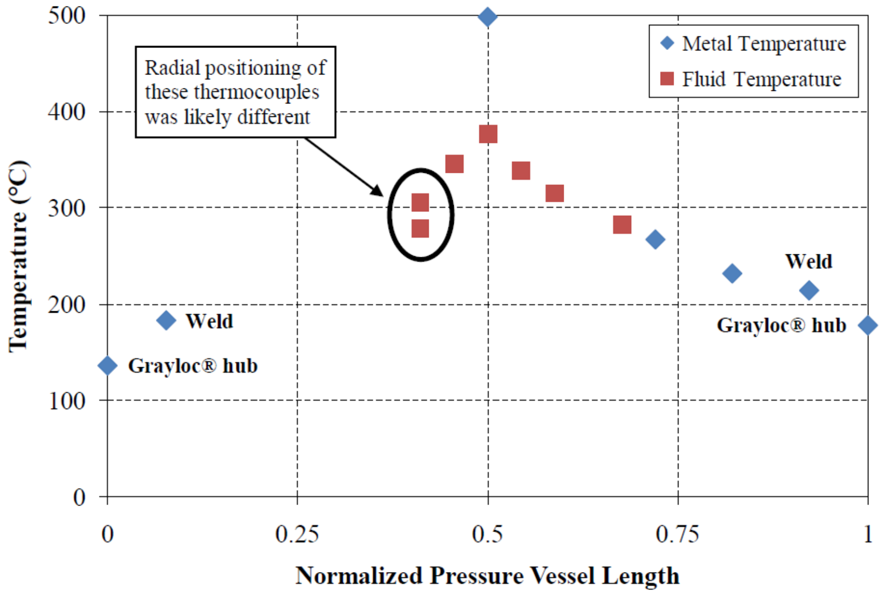


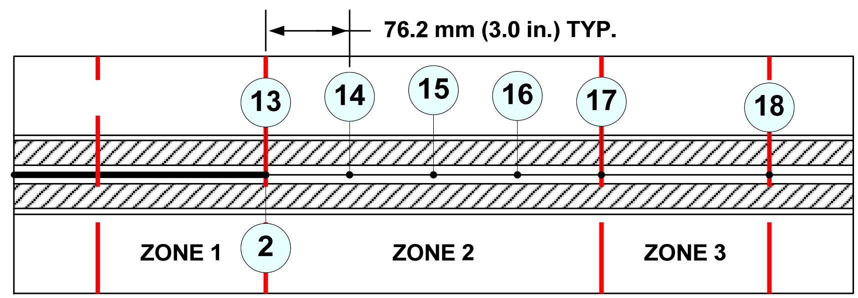




Commissioning and Calibration

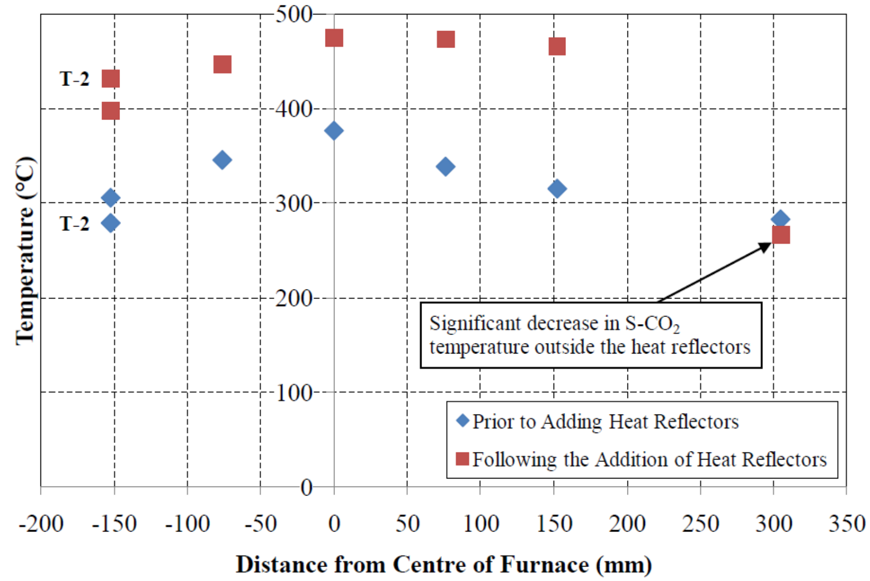
- Initial runs at 15 MPa and 2.0 L/hr
- 200 °C/hr to 600 °C
- Metal and S-CO_2 temperatures measured
  - Single-point probe (T-2)
  - Profile probe (T-13 to T-18)

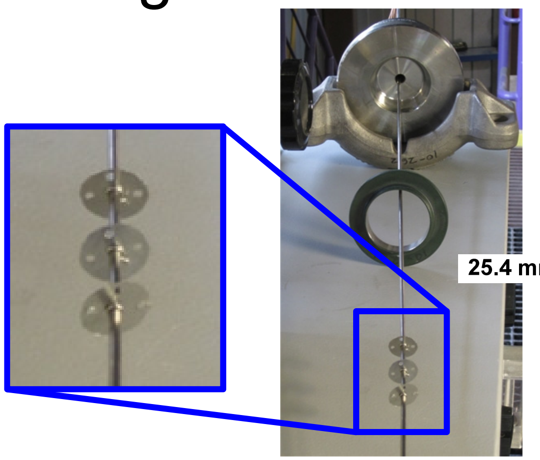


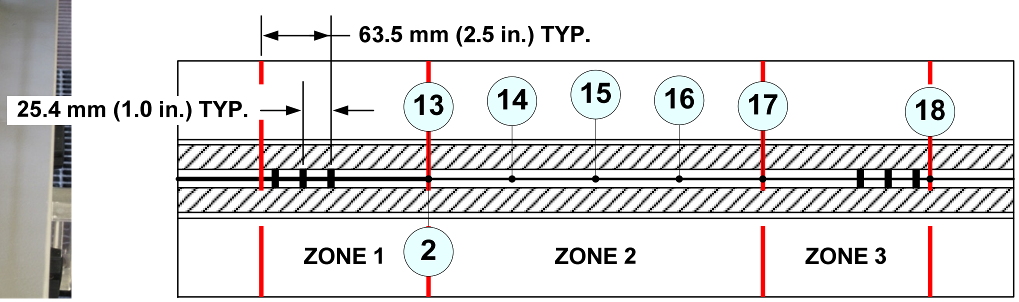


Commissioning and Calibration

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

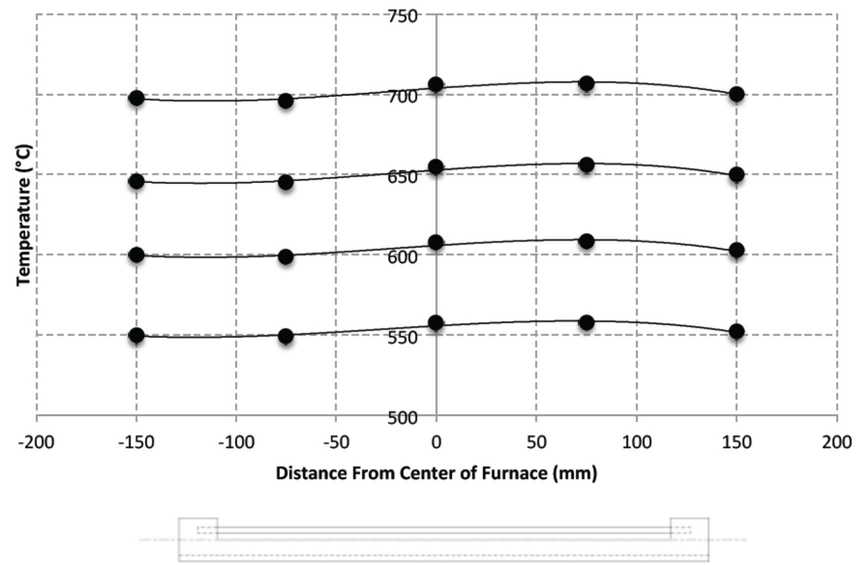


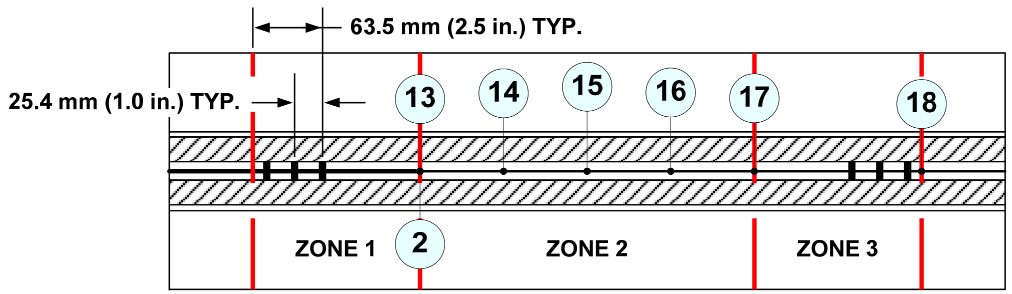




Commissioning and Calibration

- 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

Temp (°C)	Exposure Time (hours)					
	316SS		IN718		IN738	
	15 MPa	25 MPa	15 MPa	25 MPa	15 MPa	25 MPa
550	1500		1500		1500	
550	1500 + 1500		1500 + 1500		1500 + 1500	
550		1500		1500		1500
700	1500		1500		1500	
700	1500 + 1500		1500 + 1500		1500 + 1500	
700		1500		1500		1500

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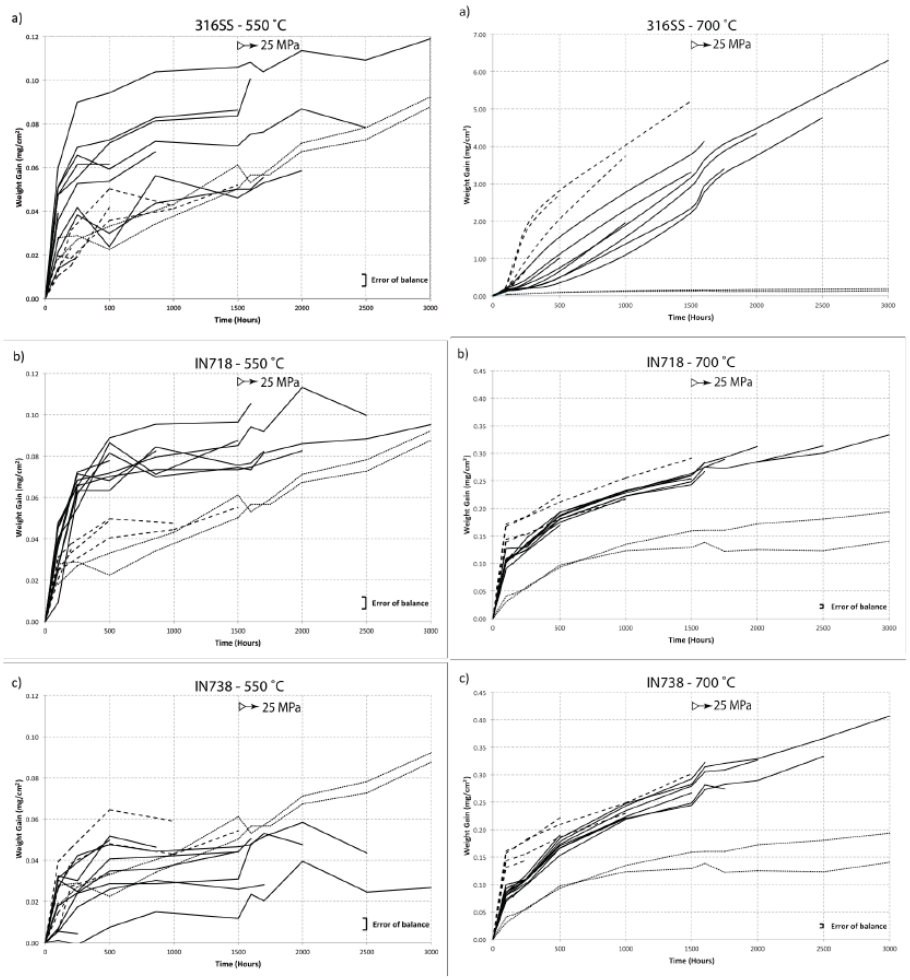
$S\text{-}CO_2$ Flow →

Test Results



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

- 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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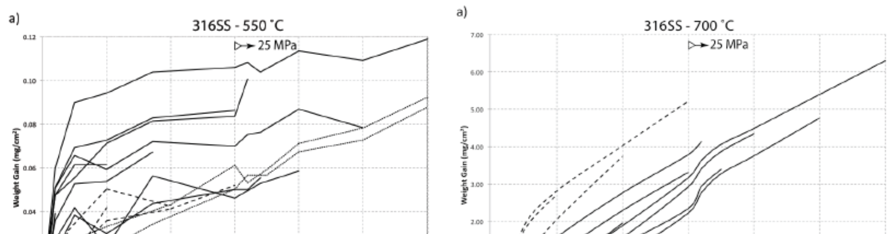

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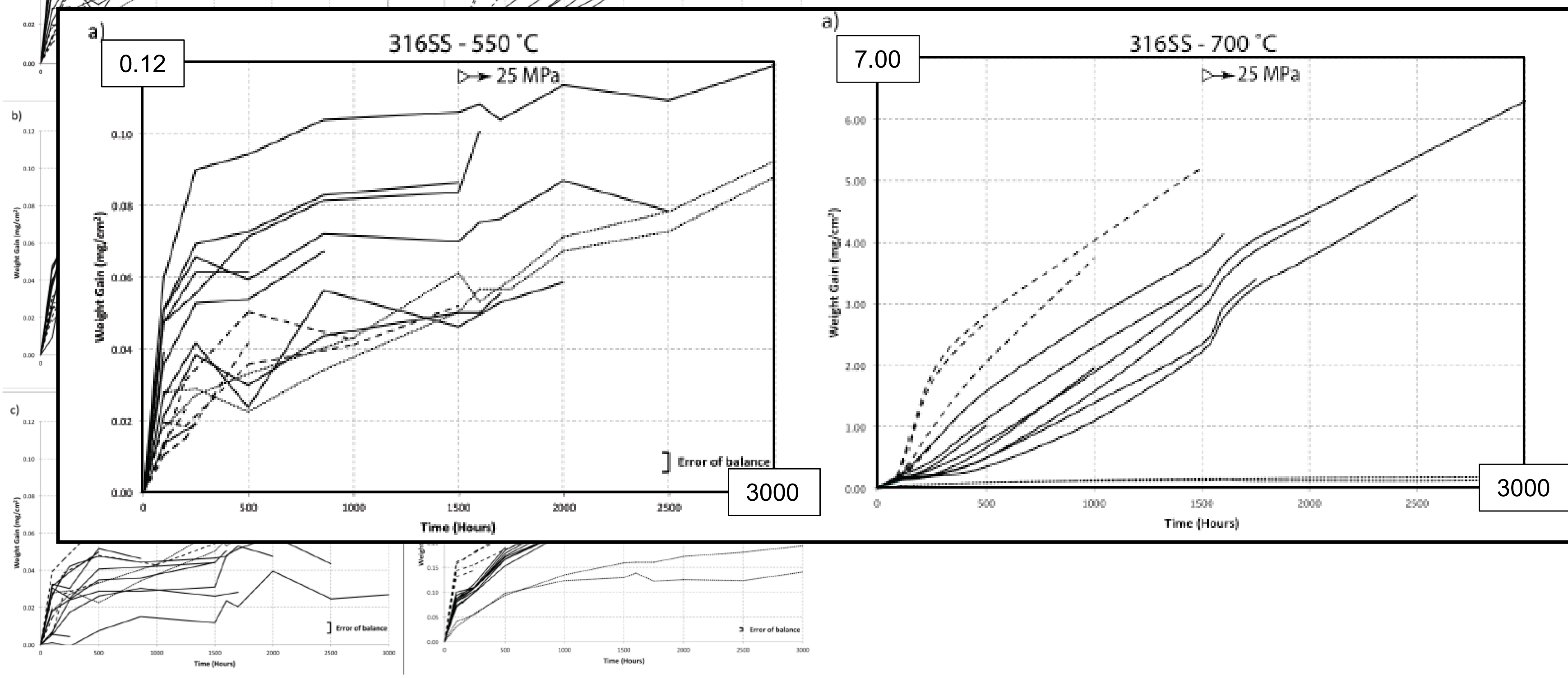

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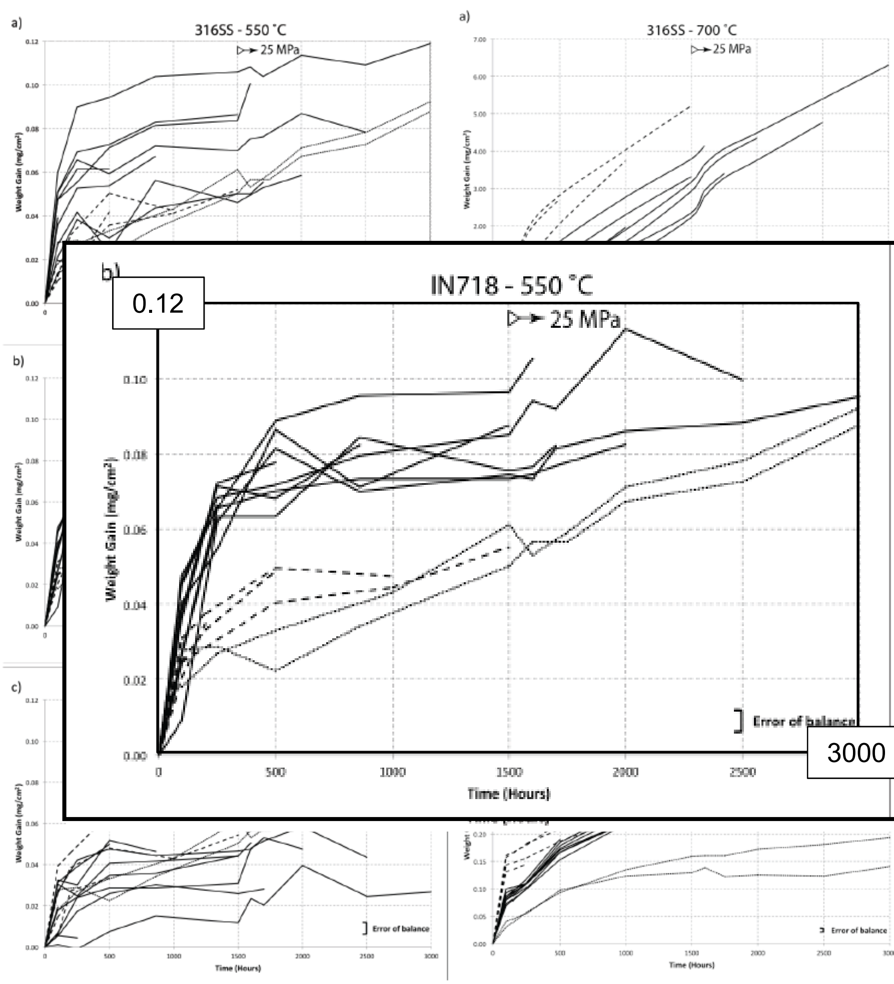

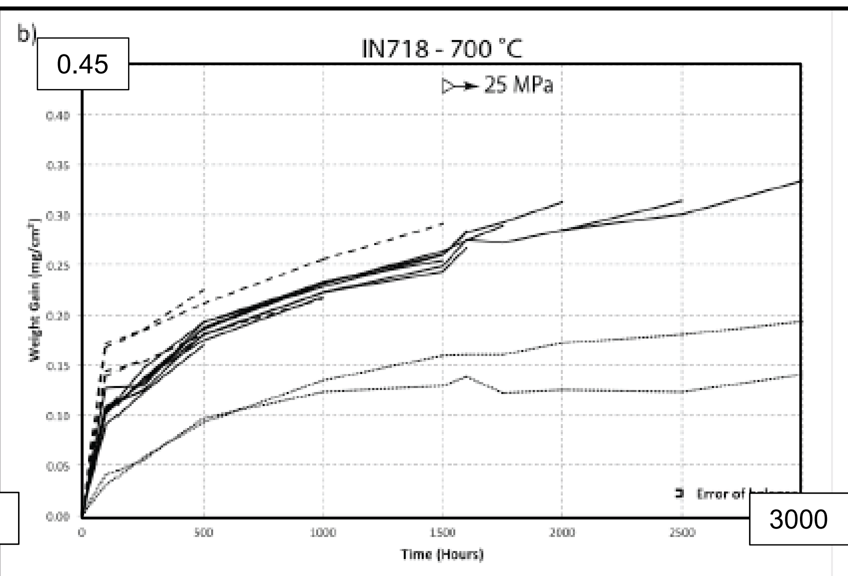

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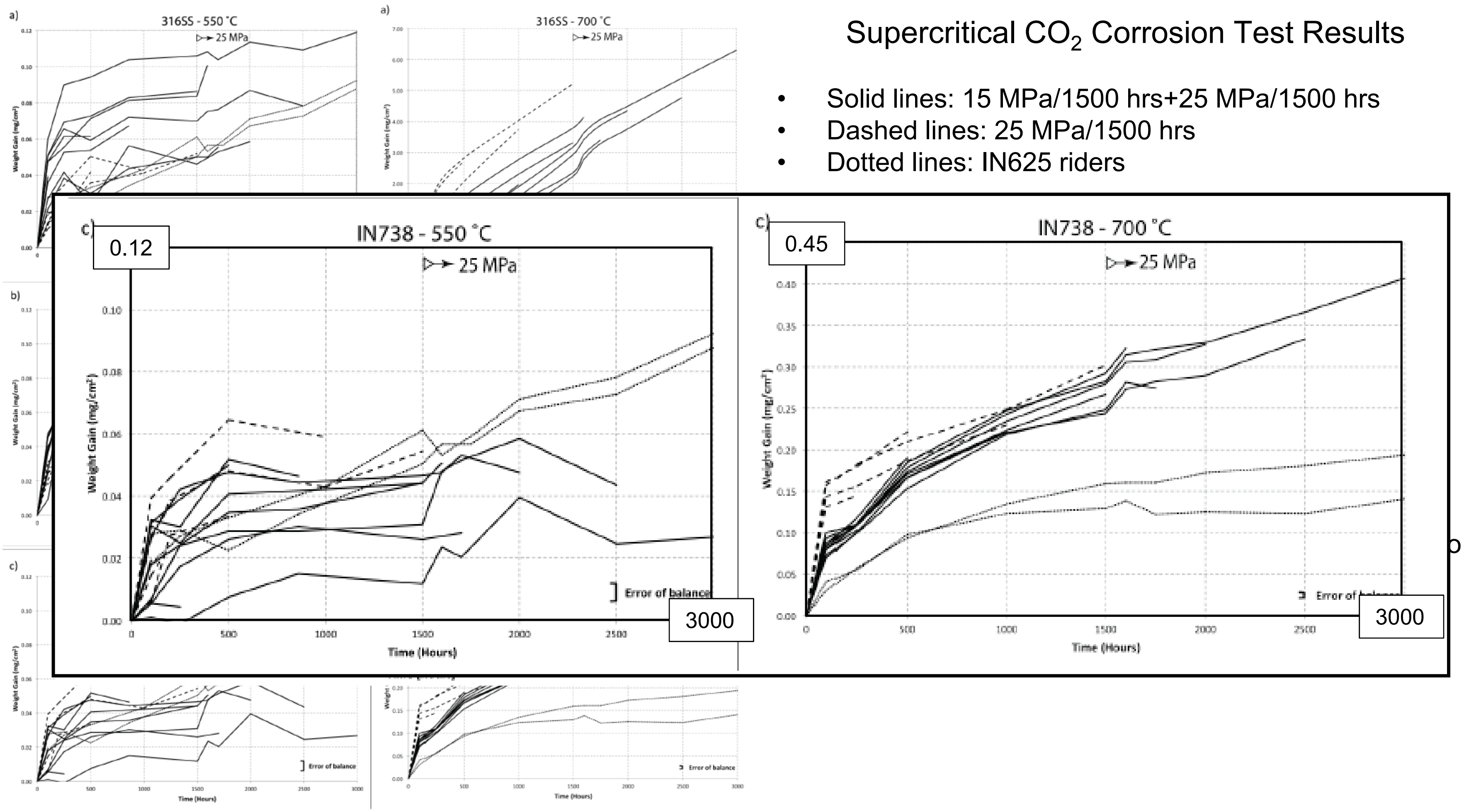

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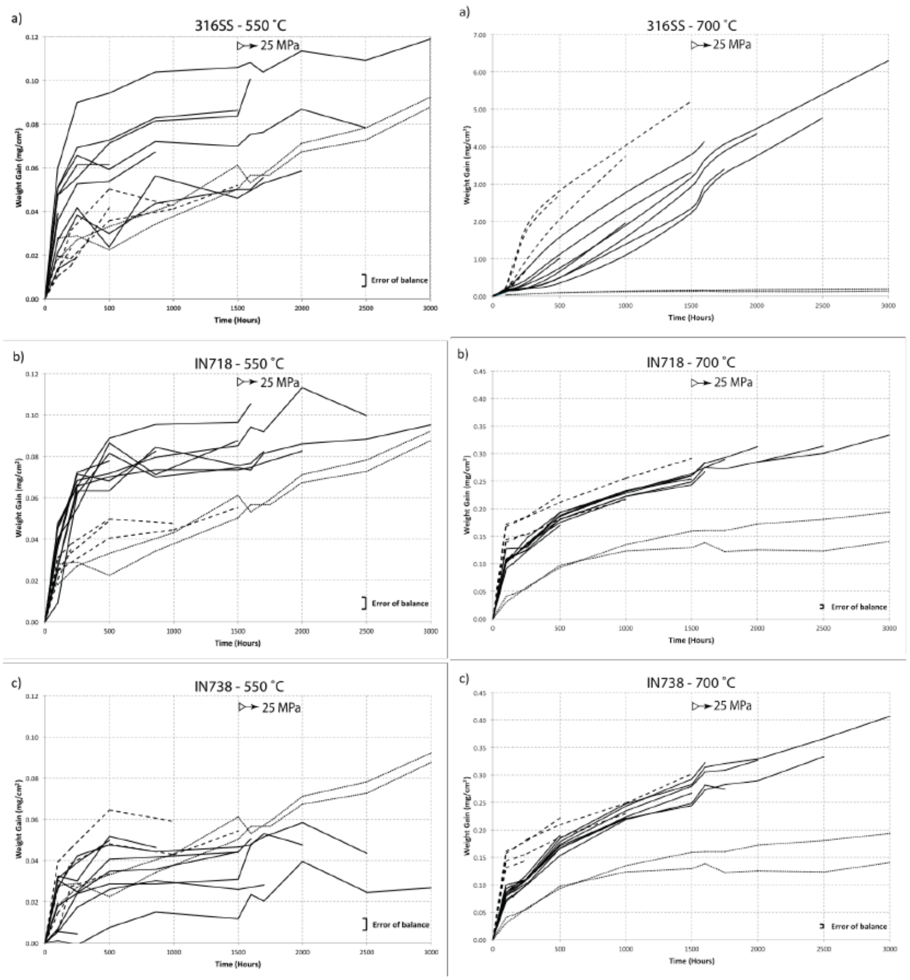

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- Some dependence on pressure

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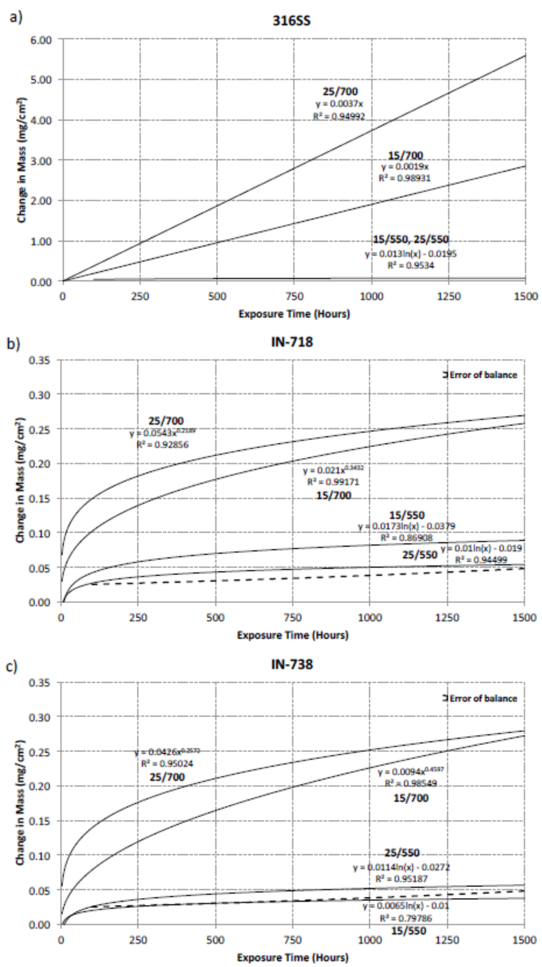

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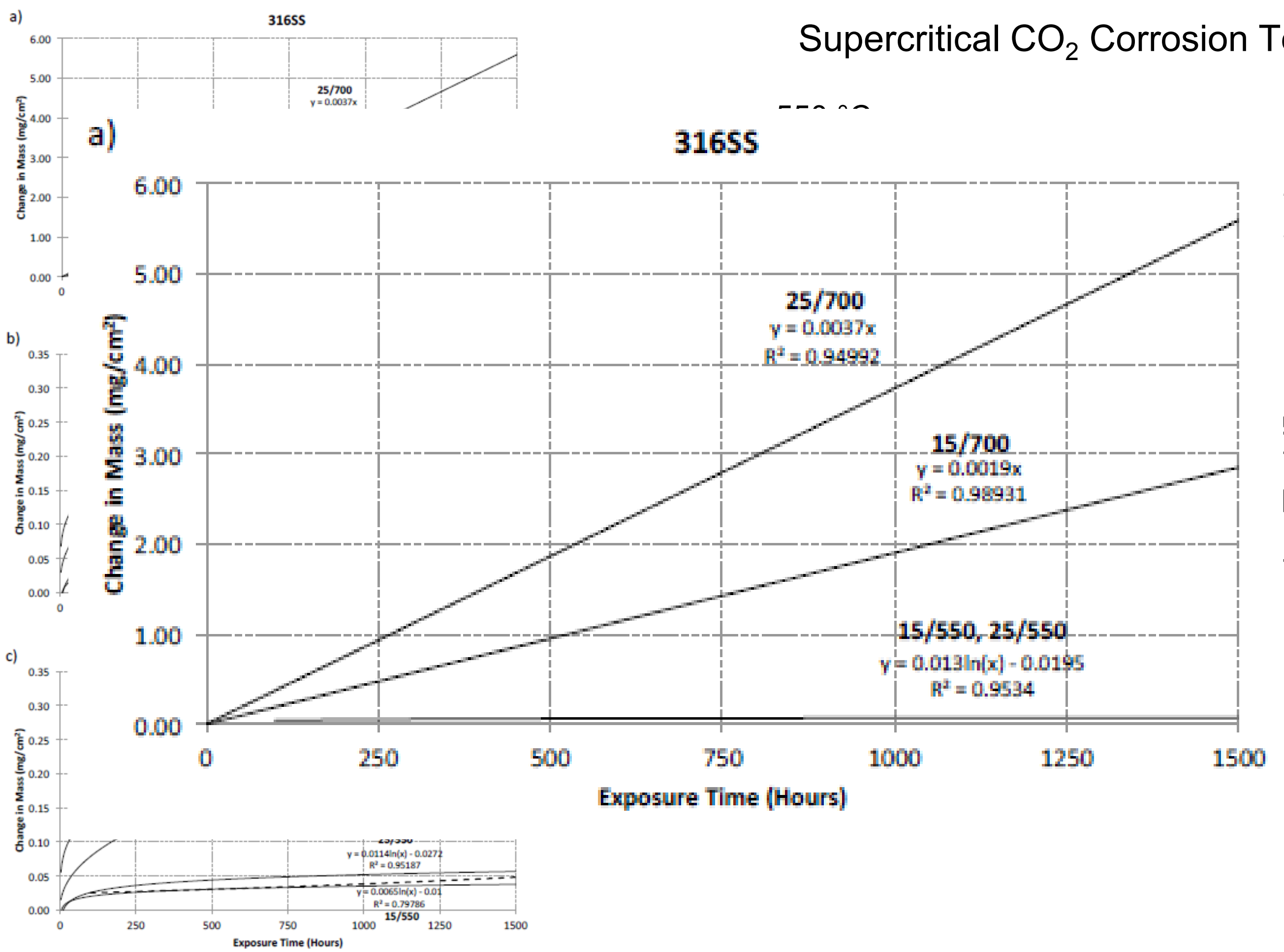

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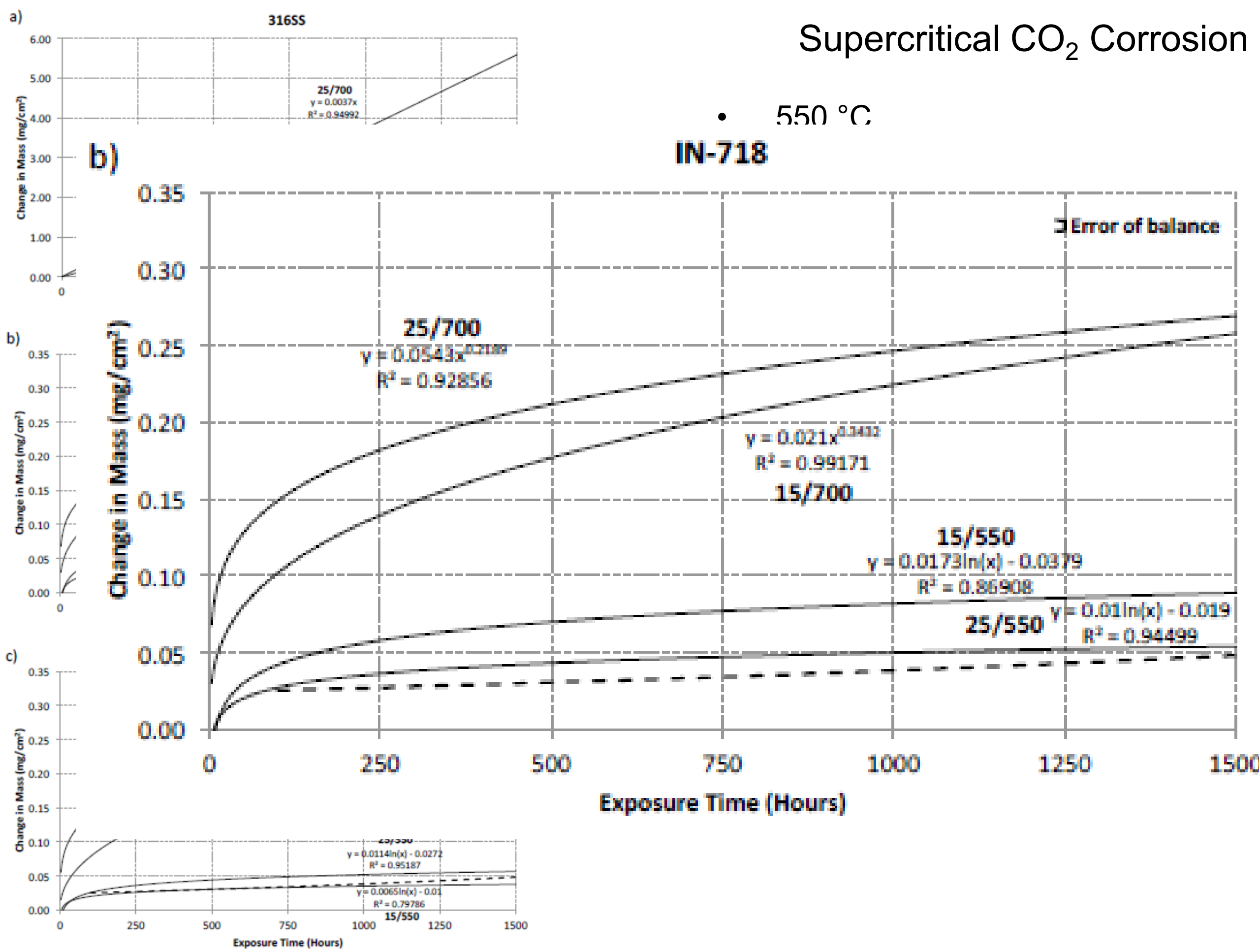
Test Results



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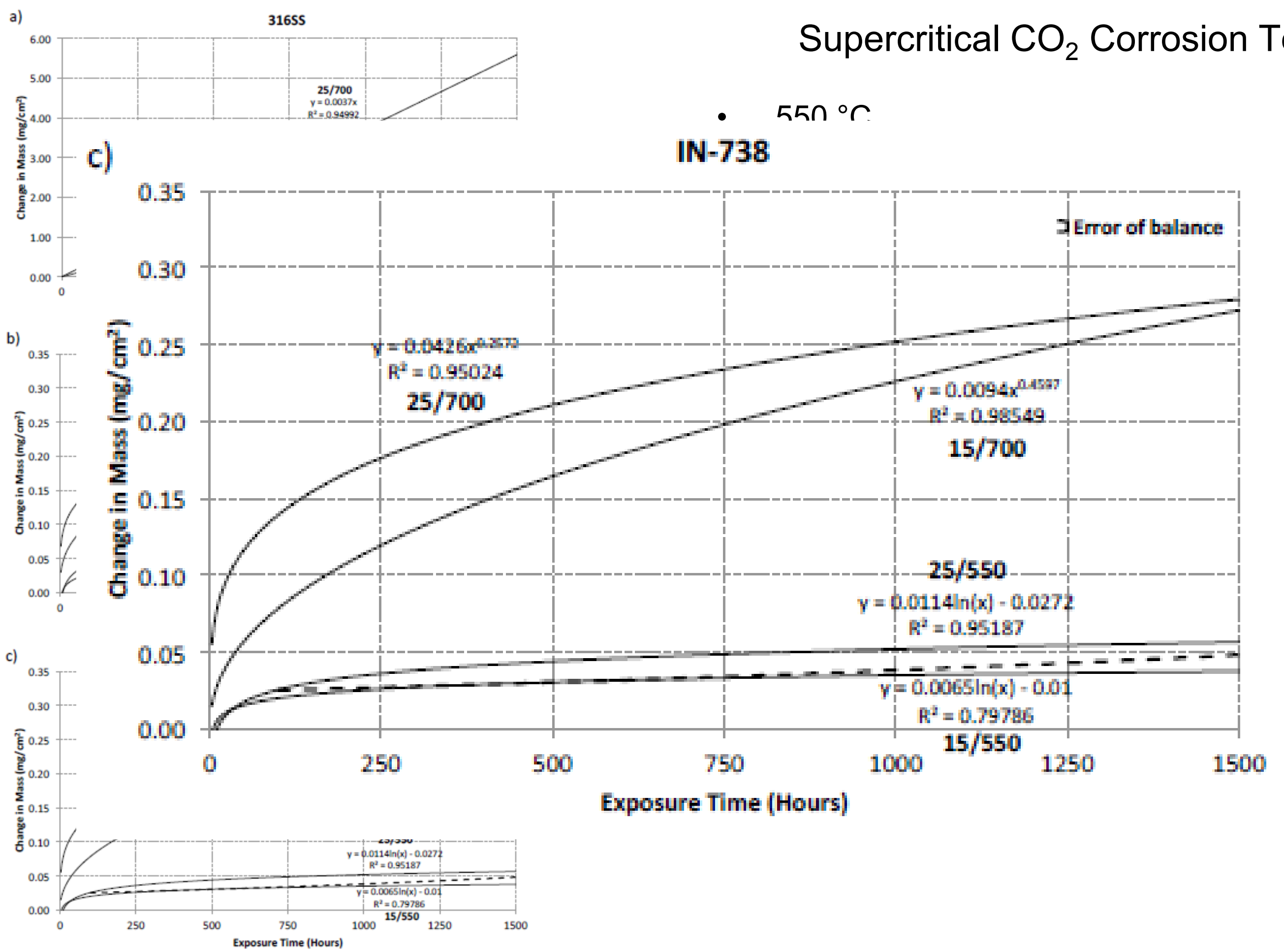

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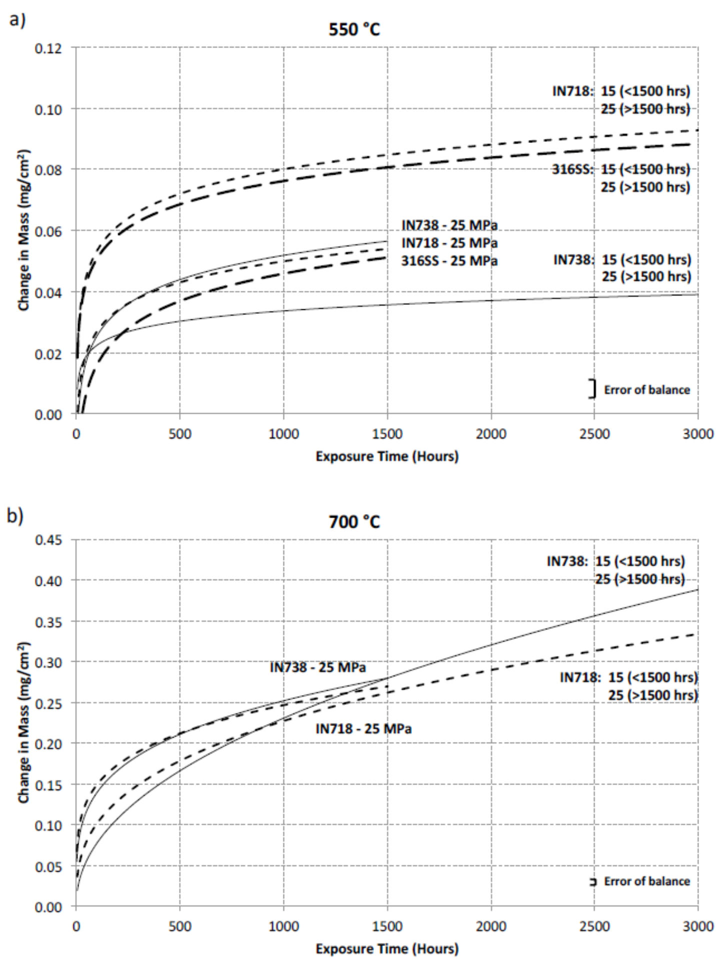

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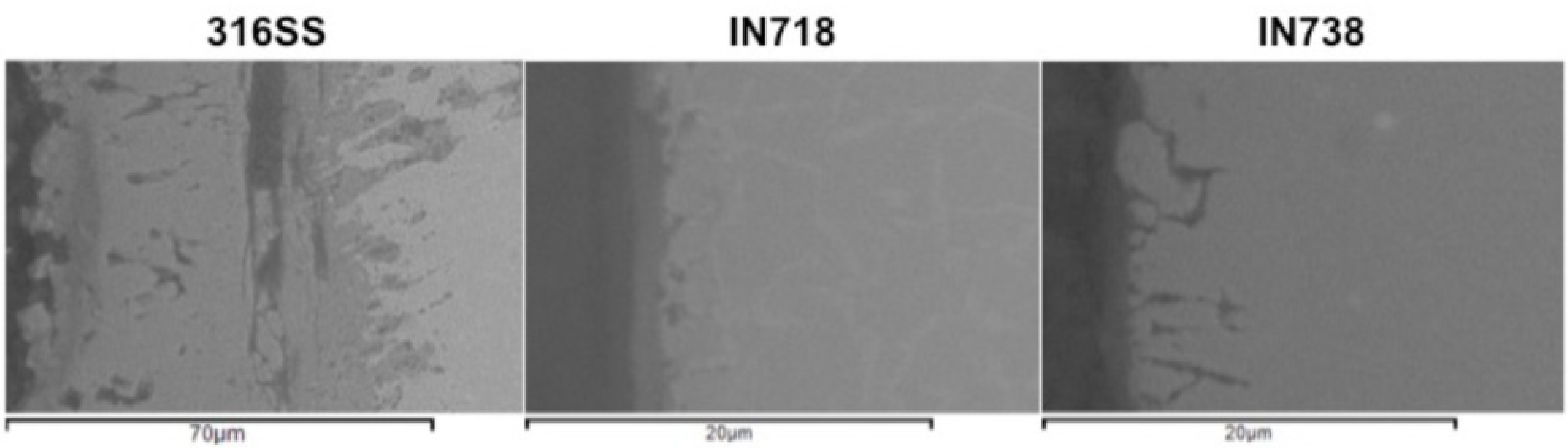
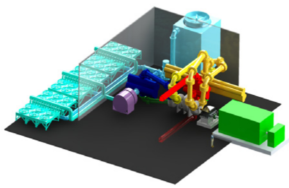


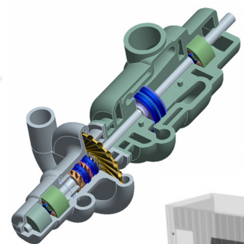
- SEM on samples tested at 700 °C for 1500 hrs
- 316SS: unstable, duplex oxide layer
- IN718 and IN738: thin, stable oxide layer
  - Some evidence of intergranular corrosion

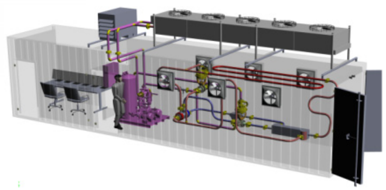
Summary

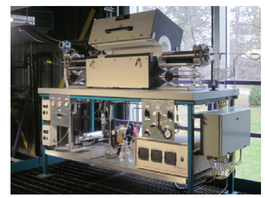
- 316SS comparable to others at 550 °C
- 316SS at 700 °C
  - linear oxide growth kinetics
  - unprotective duplex oxide layer
- IN718 and IN738 at 700 °C
  - parabolic oxide growth kinetics
  - thin and stable oxide layer
  - some evidence of intergranular corrosion

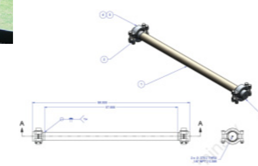
Questions?

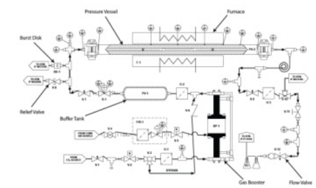


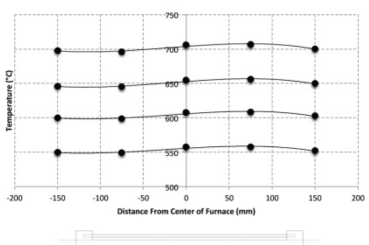












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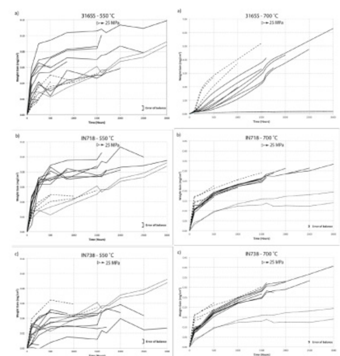
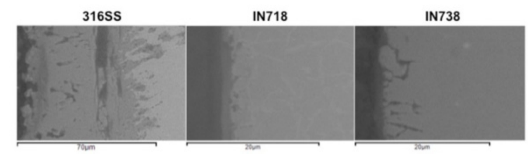
S-CO$_2$ Flow





Acknowledgements

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