Natural Resources Canada   Ressources naturelles Canada

Supercritical CO_2 Power Cycles Symposium

The effect of elevated temperature and pressure on five metallic materials in a quasi-static supercritical-CO_2 environment

sCO_2 Power Cycle Symposium

San Antonio, TX, February 21 – February 24, 2022

K. Zanganeh[1], M. Ilinich[1], T. Robertson[3] , H. Dole[1], D. Y. Seo[3] , H. Radfarnia[1], H. Saari[2], A. Beigzadeh[1]

[1] Natural Resources Canada, CanmetENERGY – Ottawa (CE-O)

[2] Carleton University, Department of Aerospace and Mechanical Engineering

[3] National Research Council of Canada, Ottawa

Canada

R&D Activities on sCO_2 Power Cycles at CE-O

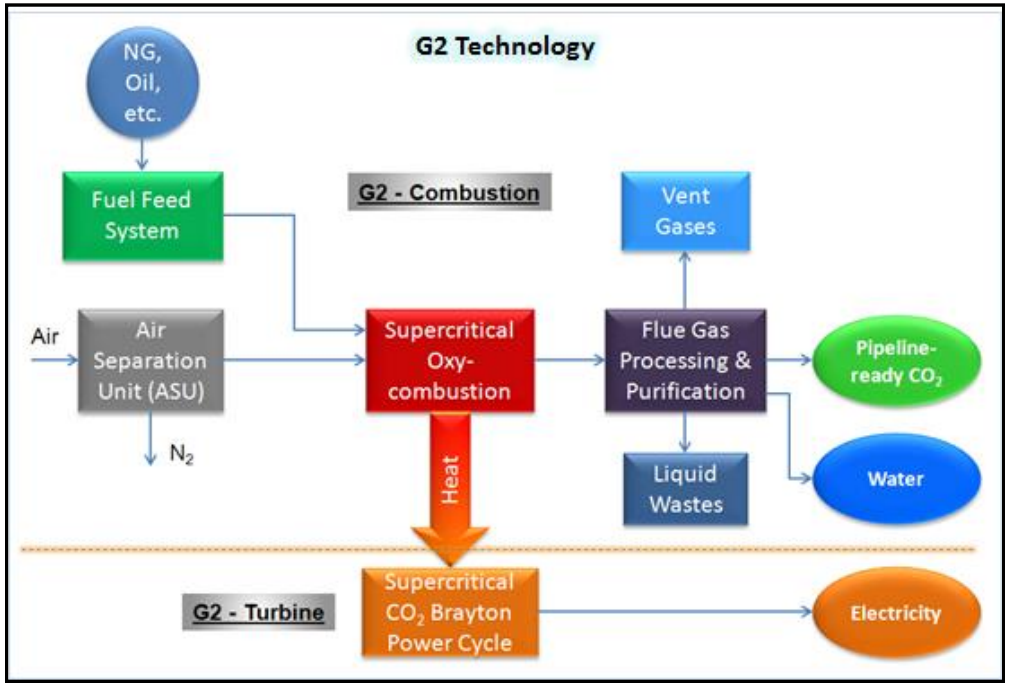
CE-O's R&D on sCO_2 cycles: initiated in 2006 as a cross-cutting advanced power conversion technology for application to zero- or low-emission nuclear, fossil, and renewable energy sources.

CanmetENERGY's G2 Technology:

- Crown patented sCO_2 power cycle technology
- A unique indirectly oxy-fired sCO_2 power cycle (producing electricity, water/steam, and pressurized pipeline-ready CO_2)
- R&D at pilot scale for de-risking the technology and its main components

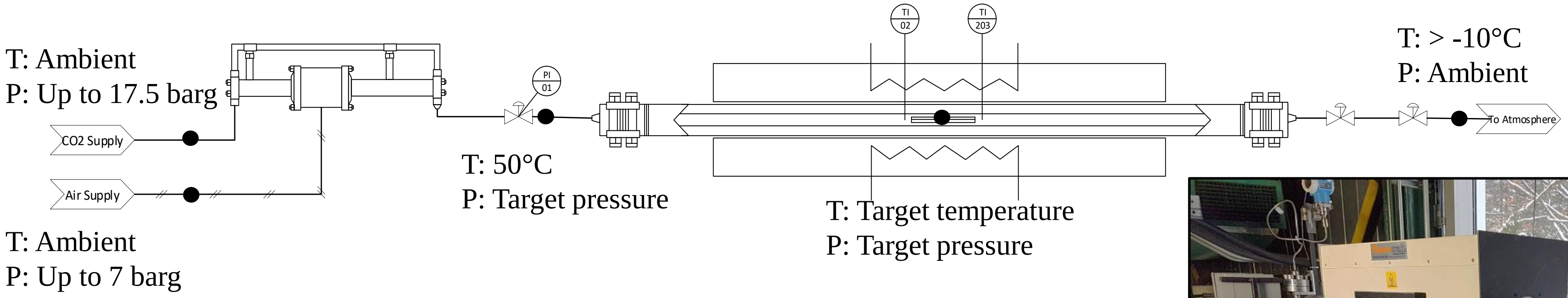
In-kind technical support to "STEP 10 MW_e sCO_2 Pilot Plant Test Facility":

- The project is led by Gas Technology Institute (GTI) and its partners (SwRI, GE-R, DOE-NETL)
- Goal: design, construct, commission, and operate a 10 MW_e sCO_2 pilot plant test facility in Texas, USA
- CE-O's partners: Carleton University, NRC, CMAT

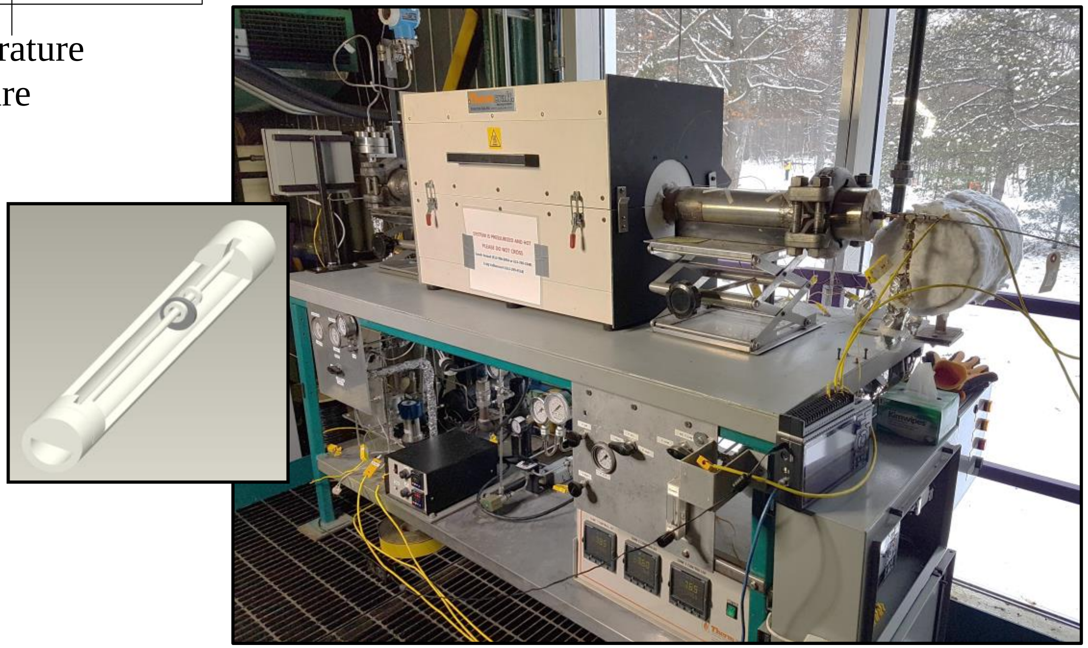


G2 Technology process flow

CE-O/Carleton University sCO_2 Corrosion Test Rig



- Maximum operating conditions: 750°C at 294 bar; 800°C at 176 bar
- Used to generate new data and knowledge on alloys corrosion in sCO_2 environment



sCO_2 Corrosion Test Rig

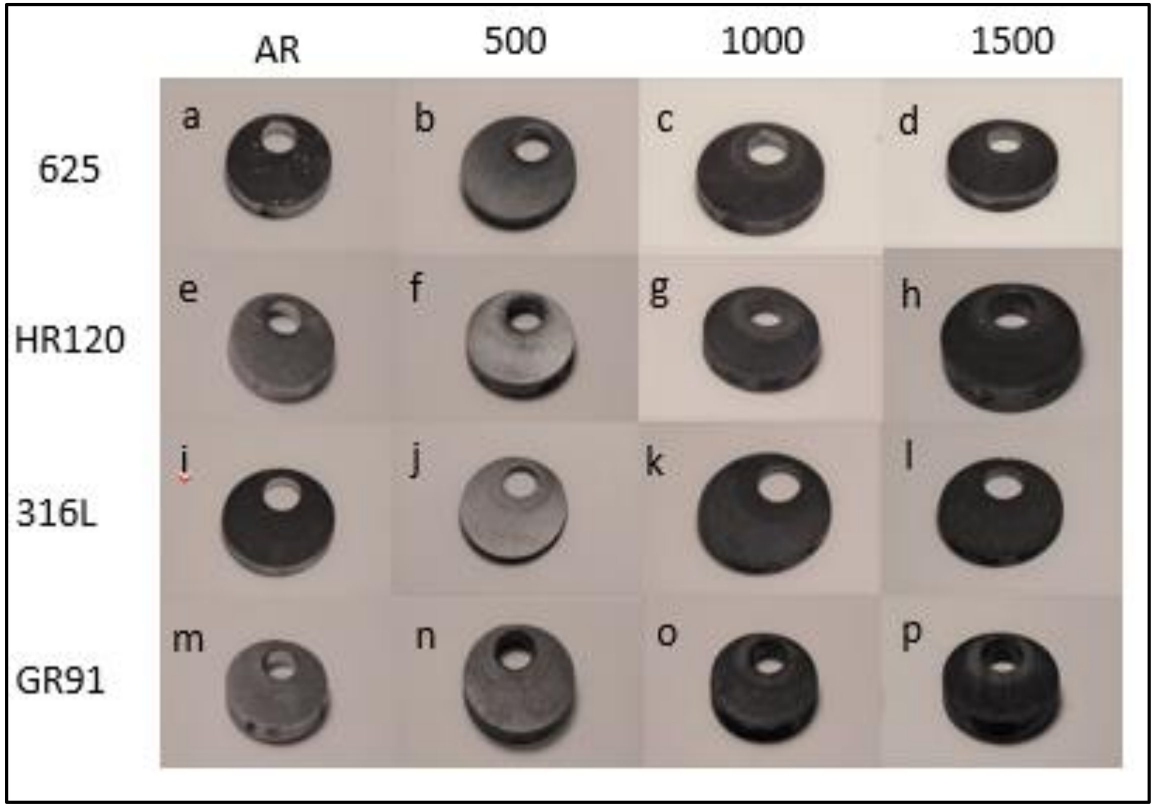
Round Robin Test Campaign

- Led by EPRI; involving 8 research institutions
- CE-O participated through Carleton University, with support from NRC for characterization
- 5 Alloys tested:
  - Inconel 740H, Inconel 625, Haynes HR-120, Stainless Steel 316L, and GR 91 steel
  - Alloy samples exposed to sCO_2 @ 550°C and 700°C, 200 bar for 1500 hrs (in 500-hr intervals)
- Mass gain measured at each 500-hr interval
- Samples characterized using SEM, EDX, and XRD after each 500-hr interval

Alloy samples loaded into specimen boat

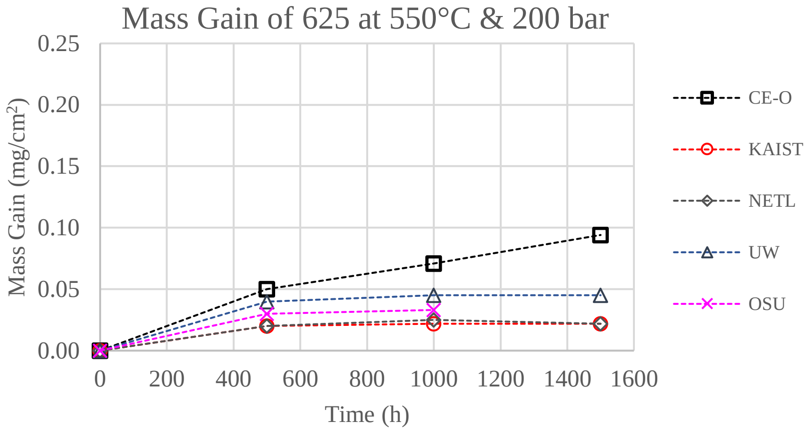
Visual Appearance (550°C)

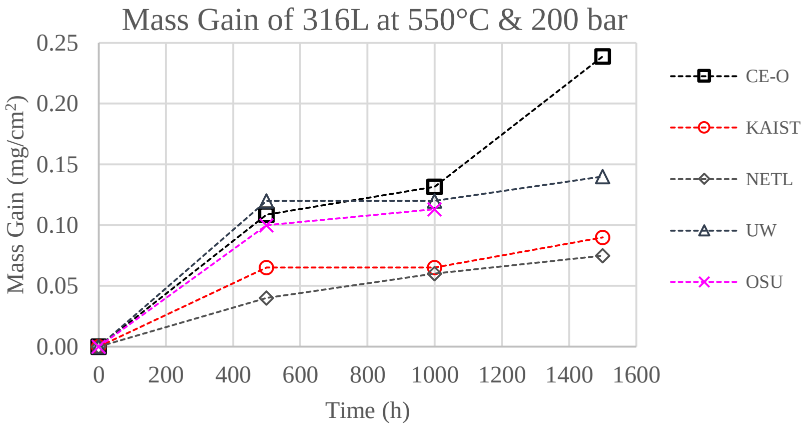
- After 500 hours samples maintained polished appearance
  - 625 displayed slight heat-induced discoloration on both faces
- 1000 hours onward all samples darkened due to thickening of the oxide layer

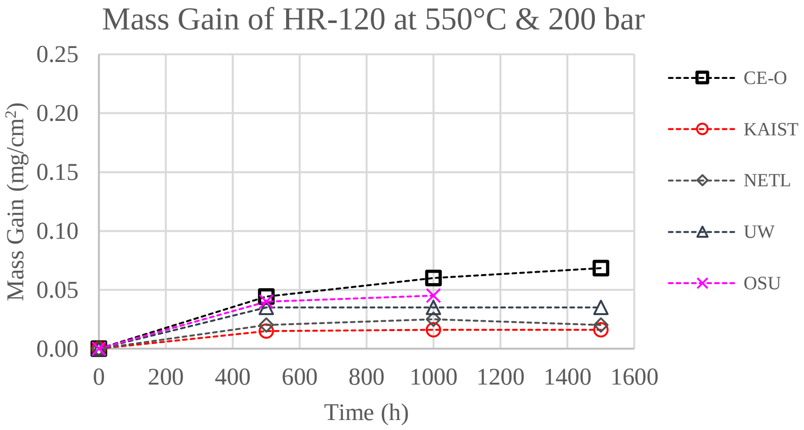


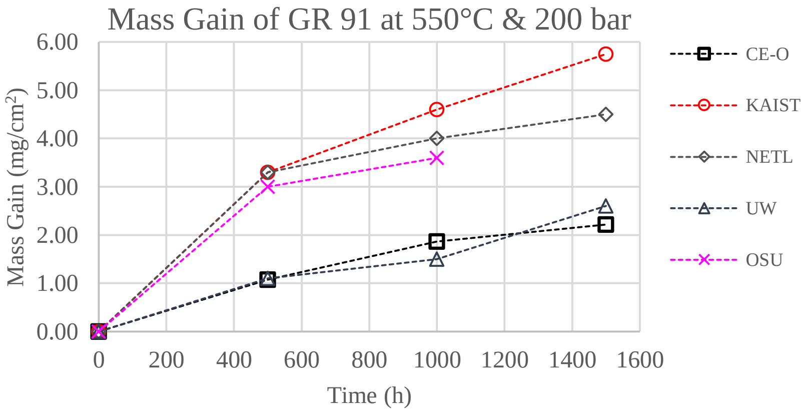
Appearance of alloys during the 1500 hours

Mass Gain and Results Comparison (550°C)



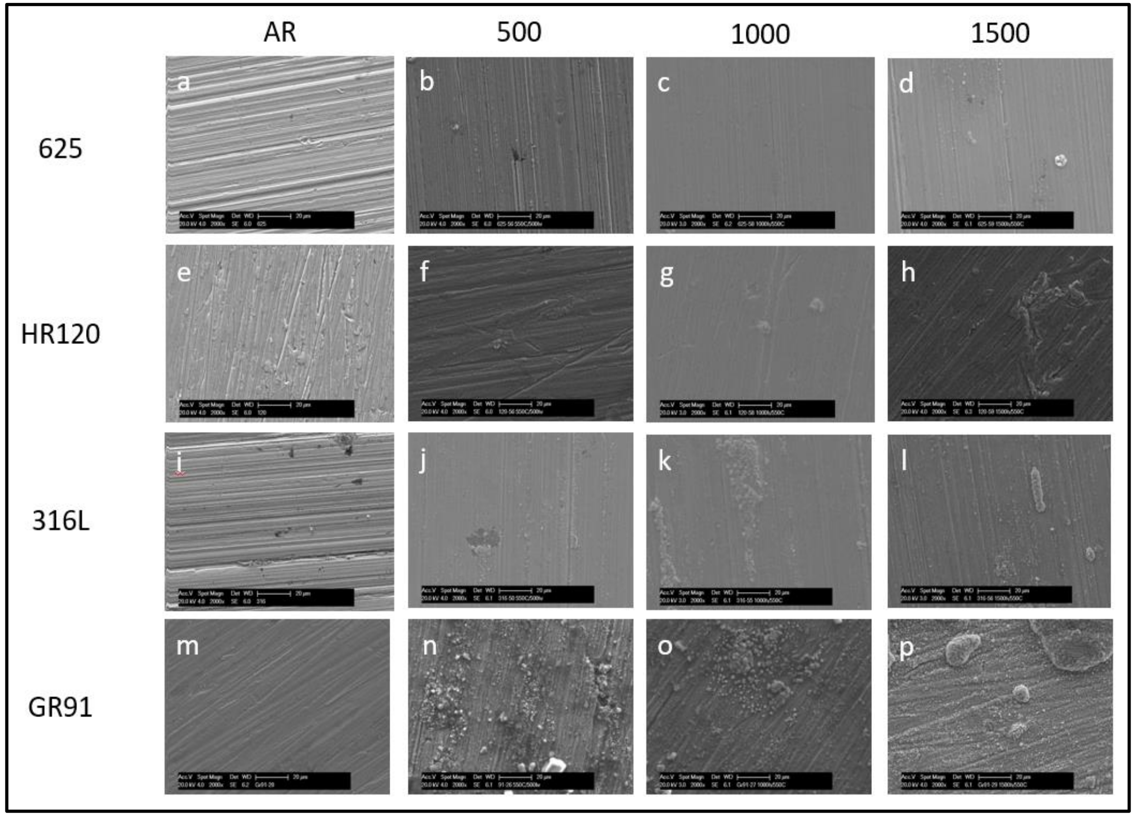






Surface SEM Analysis (550°C)

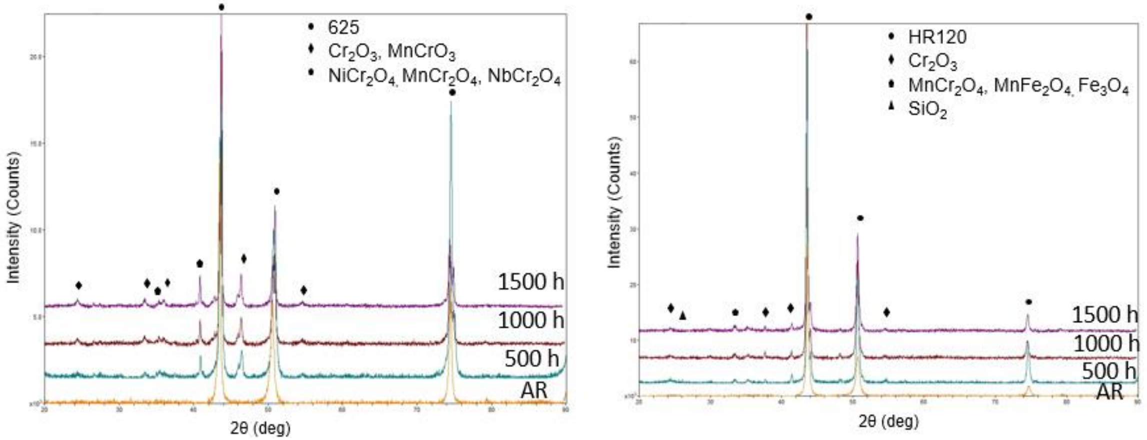
- All alloys developed a thin layer of oxide after 500 hour exposure
- Oversized oxide clusters identified on the surface of GR 91 and 316L steels
  - Clusters grew and combined throughout the exposure
- Some oversized oxide clusters develop on the surface of 625 and HR-120 at the later exposures



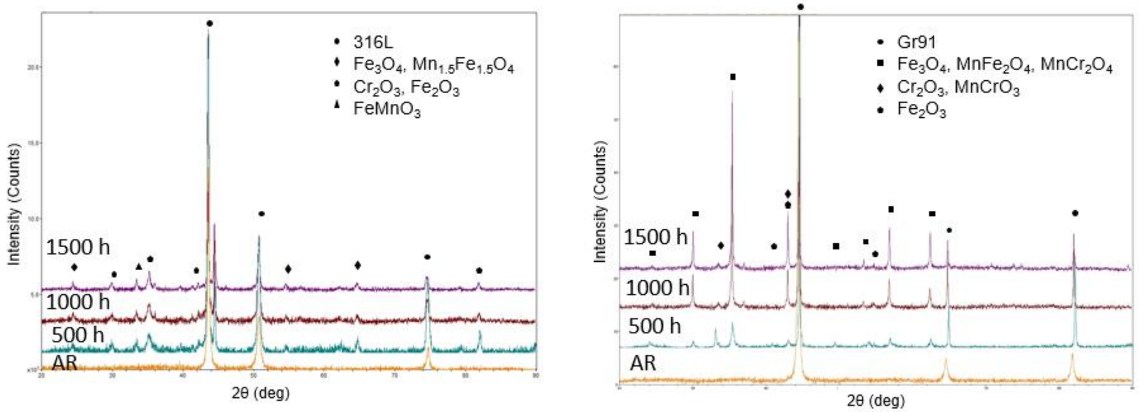
Surface SEM of the samples during the 1500 hours

EDX and XRD Analysis (550°C)

- Scans of 625 and HR-120 exhibit a Cr-based primary oxide layer (Cr_2O_3)
  - XRD scans of 625 and HR-120 indicate low oxidation
  - 625 developed Mn-Ni-Nb spinels
  - HR-120 showed presence of Mn-Fe-Cr spinel structures
- Scans of 316L and GR91 steels indicate formation of Fe-rich primary oxide (Fe_3O_4)
  - GR91 showed significant presence of Fe_3O_4 which is indicative of poor oxidation resistance

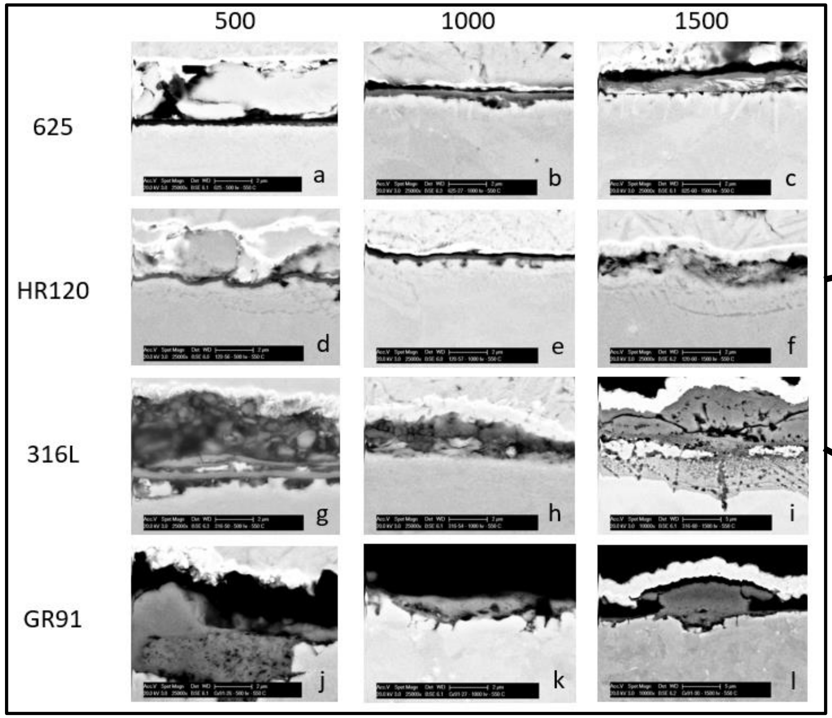


XRD scans of alloys 625 and HR-120 after 1500 hours of exposure

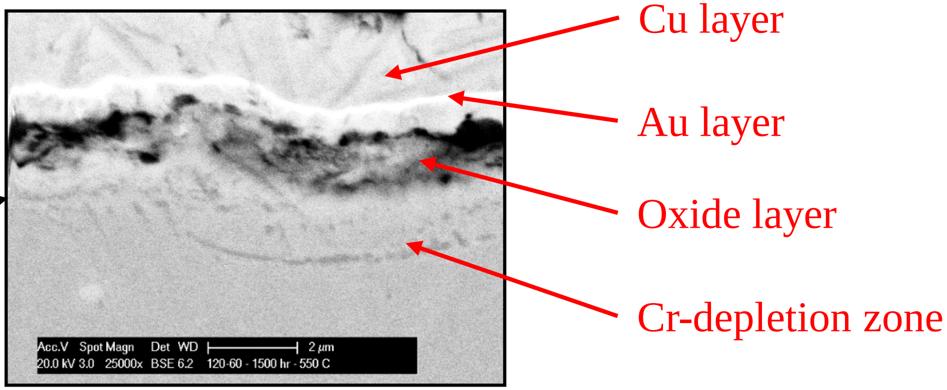


XRD scans of steels 316L and GR91 after 1500 hours of exposure

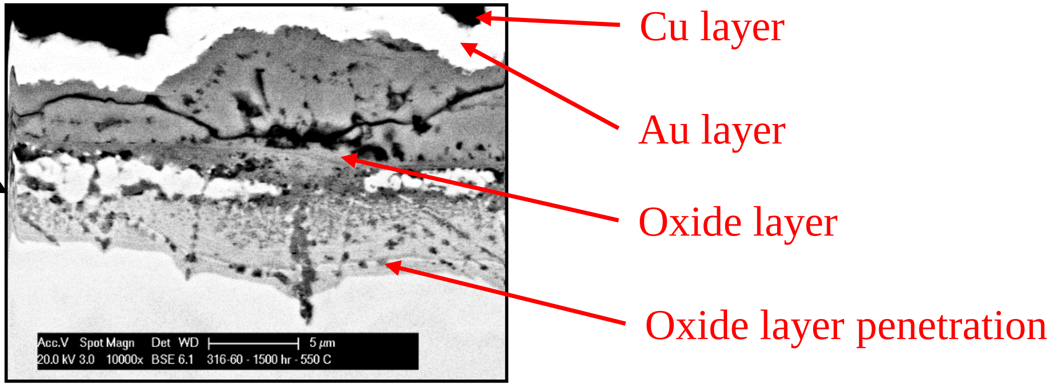
Cross-sectional SEM Analysis (550°C)



Cross-sectional SEM of the four alloys during the 1500 hours



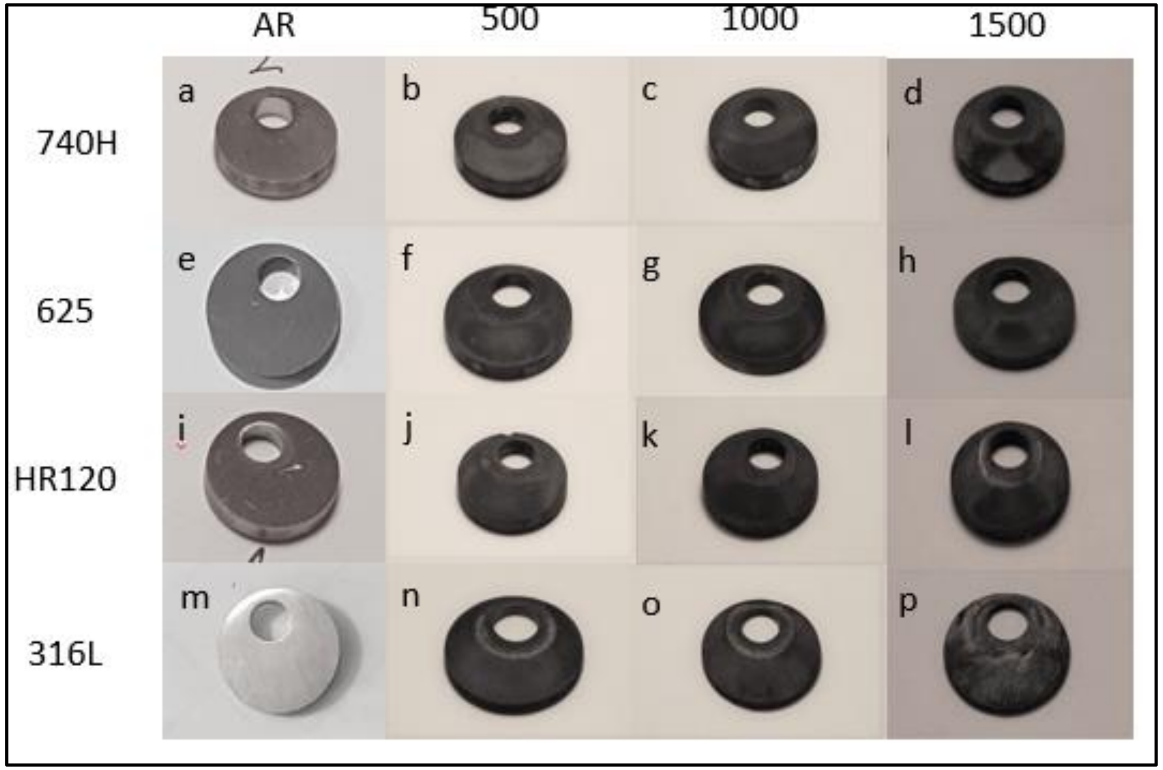
Cross-sectional SEM of HR-120 after 1500 hours



Cross-sectional SEM of 316L after 1500 hours

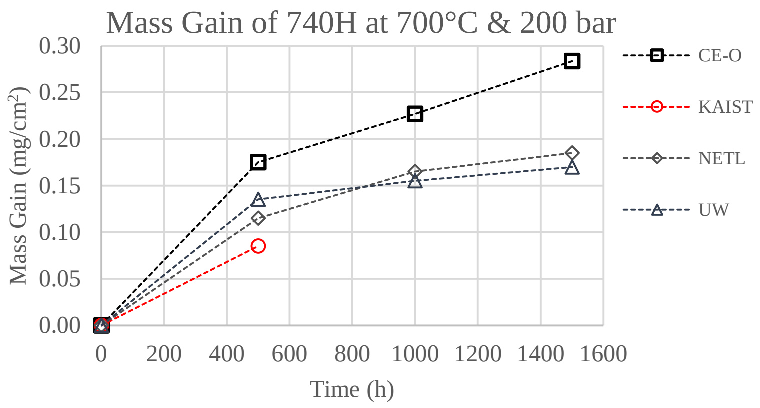
Visual Appearance (700°C)

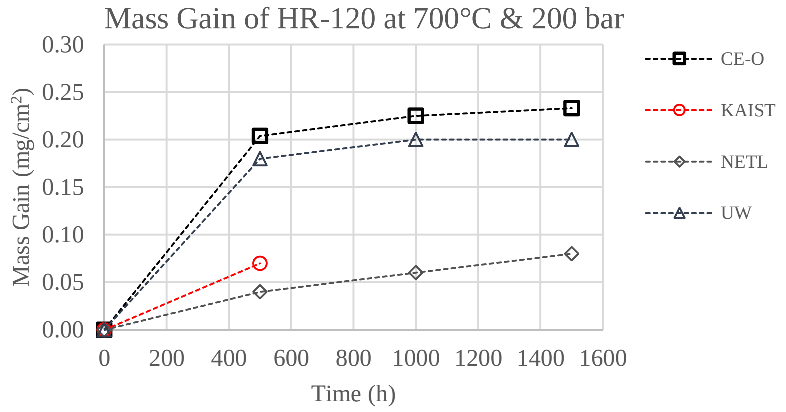
- After 500 hour exposure all samples darkened, indicating oxidation
- By the end of 1500 hours:
  - 625 appeared least discoloured
  - 316L appeared most discoloured
  - All coupons exhibited abrasion caused by flow pattern through the specimen boat

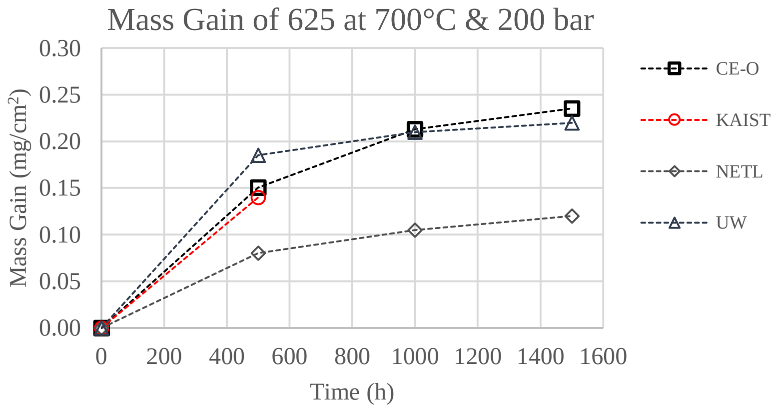


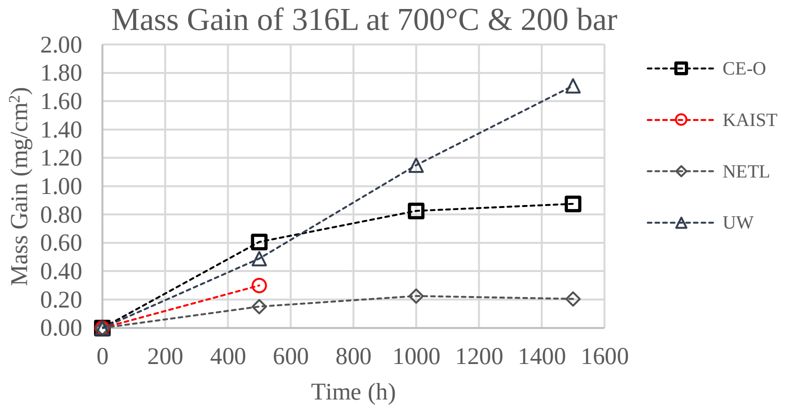
Appearance of alloys during the 1500 hours

Mass Gain and Results Comparison (700°C)



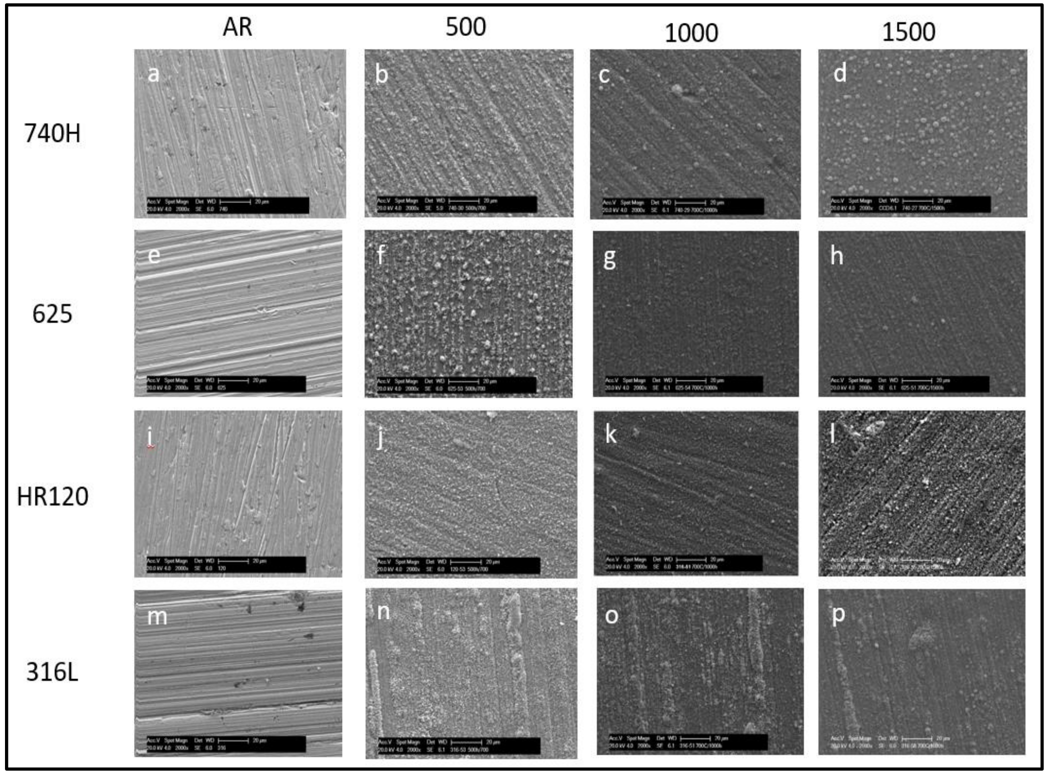






Surface SEM Analysis (700°C)

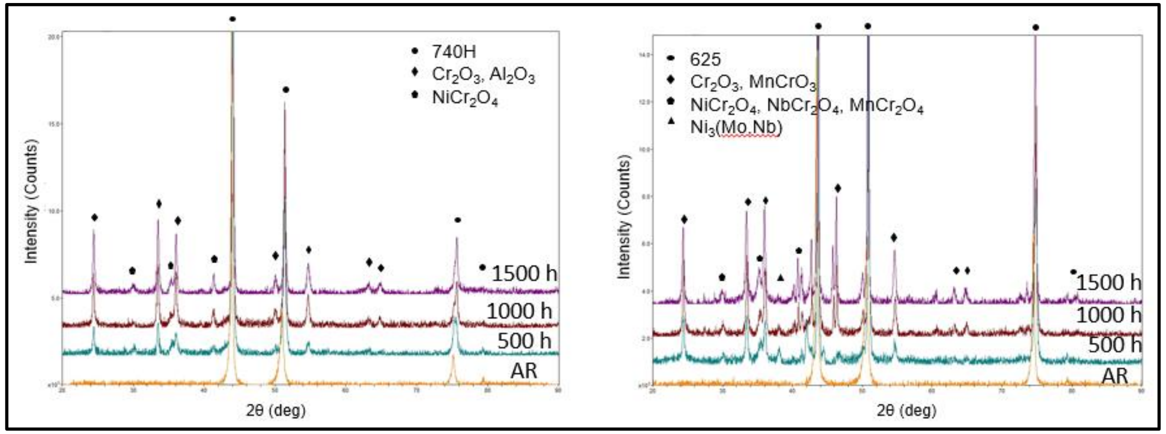
- Substantial layer of oxide was detected on the surface of each alloy
- 316L: uniform base layer with rows of oversized oxide
- HR-120 and 625: uniform layer of oxide after 500 and 1000 hours onward respectively
- 740H: thin base layer with small grains forming after 1000 hours



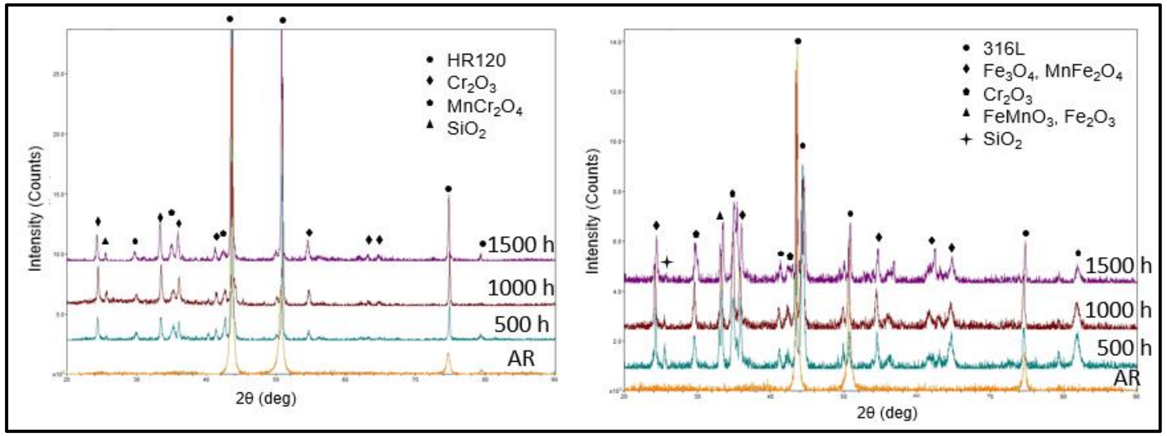
Surface SEM of the samples during the 1500 hours

EDX and XRD Analysis (700°C)

- EDX scans of 740H, 625 and HR-120 exhibit a Cr-based primary oxide layer (Cr_2O_3)
  - 740H displayed an increased content of Nb within the greater oxide regions and Al content near the base of oxide layer
  - 625 exhibited an Nb rich bottom oxide layer
  - Scans for HR-120 indicated that the Cr-rich base oxides also possess a high content of Mn spinels
- 316L surface oxide consisted of Cr prominent base layer with a Fe-based oversized growths
  - Oxide layer is combination of Cr_2O_3 and Fe_3O_4

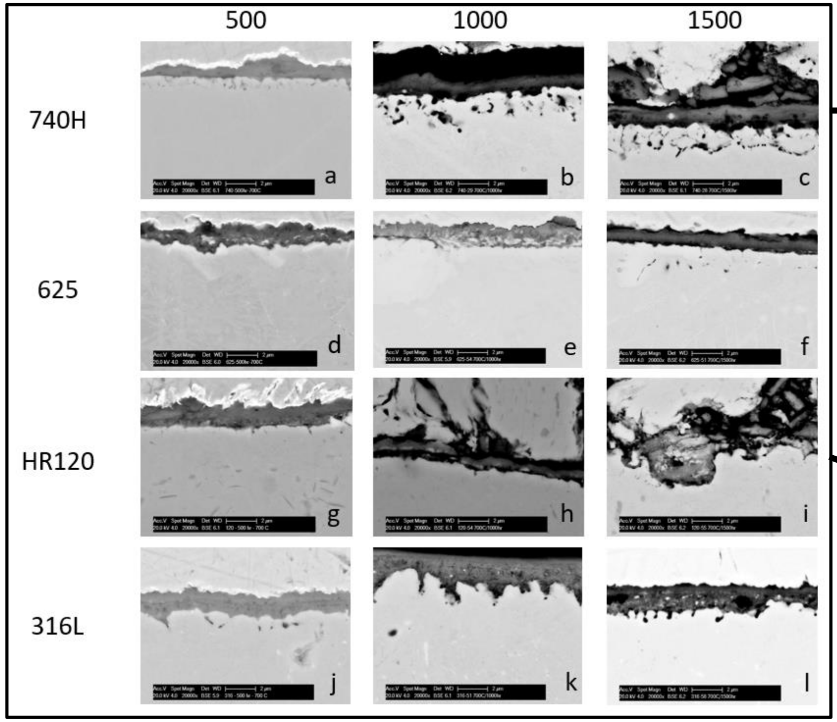


XRD scans of alloys 740H and 625 after 1500 hours of exposure

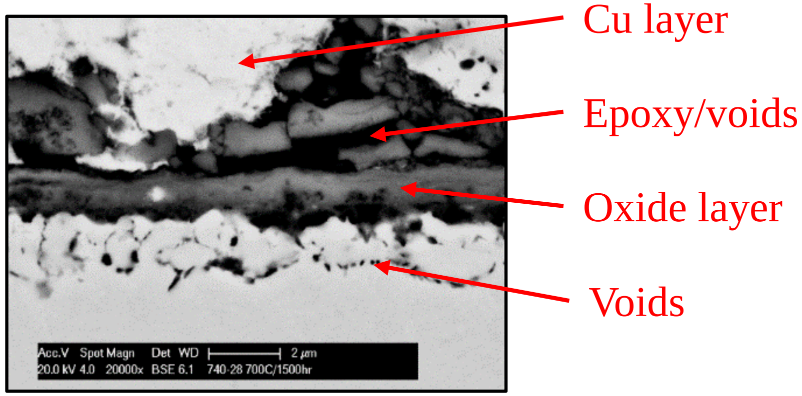


XRD scans of alloys HR-120 and 316L after 1500 hours of exposure

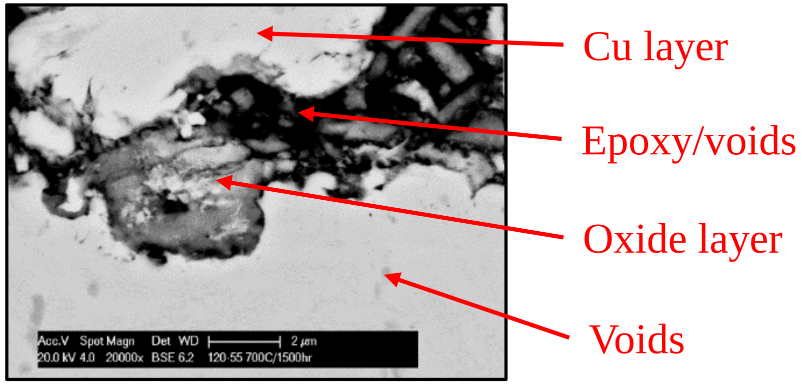
Cross-sectional SEM Analysis (700°C)



Cross-sectional SEM of the four alloys during the 1500 hours



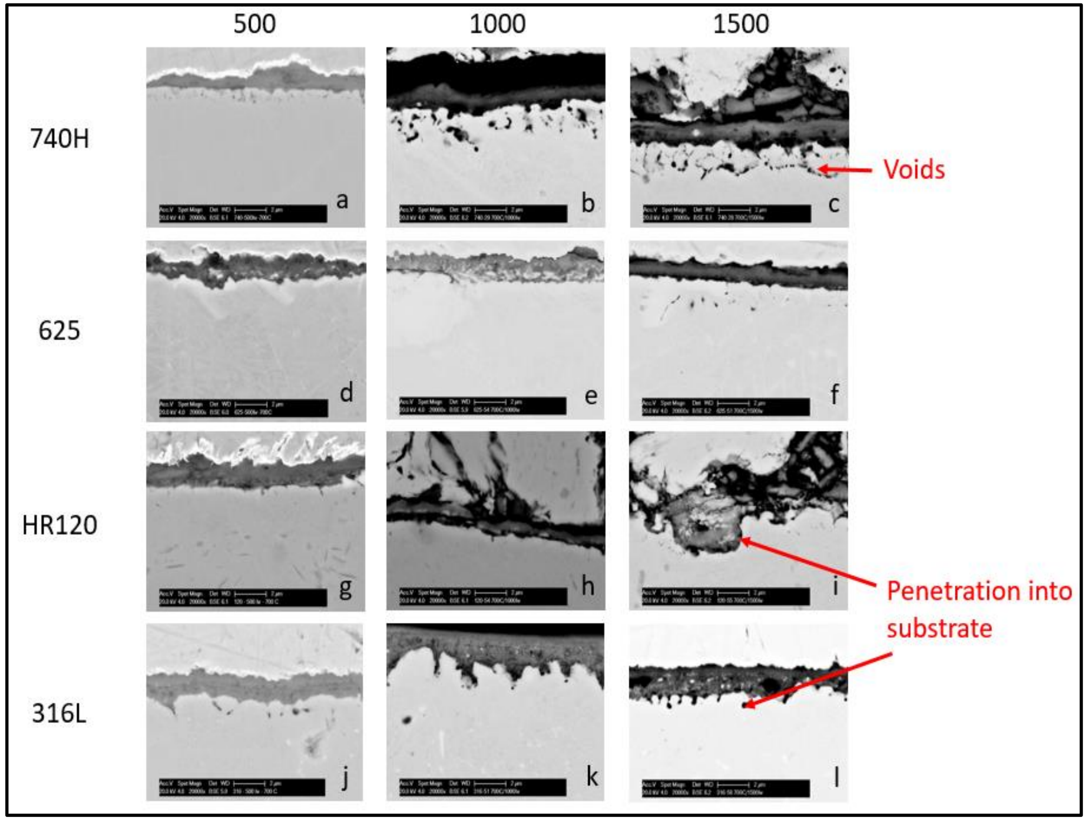
Cross-sectional SEM of 740H after 1500 hours



Cross-sectional SEM of HR-120 after 1500 hours

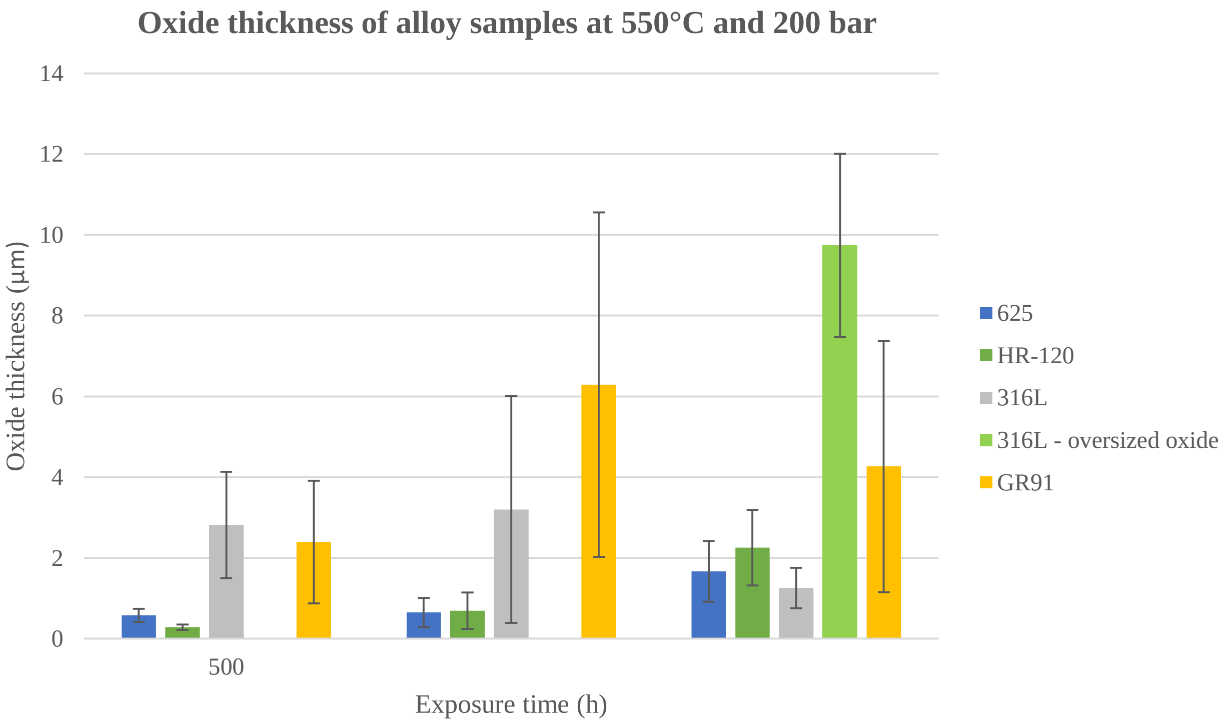
Cross-sectional SEM Analysis (700°C)

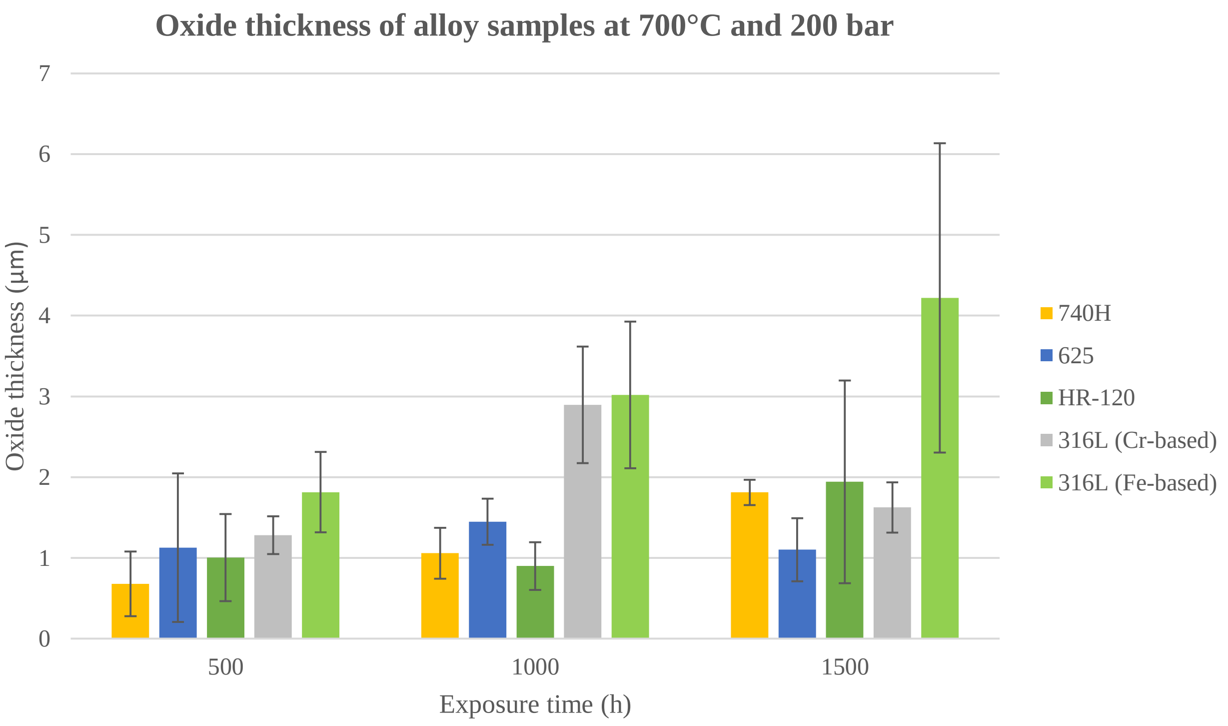
- 740H displayed a uniform oxide layer composed primarily of Cr_2O_3
  - Internal Al_2O_3 oxide layer was detected which forms a barrier to prevent oxygen diffusion
  - Many voids along grain boundaries were found under the oxide layer
- 625 exhibited the thinnest oxide layer composed mainly of Cr_2O_3
- 316L displayed Cr_2O_3 base layer and regions of Fe_3O_4 excessive growth penetrating into substrate through Cr oxide layer
  - This is indicative of low life expectancy of the metal
- HR-120 displayed a discontinuous Cr oxide base layer and a more uniform upper layer
  - Significant oxide penetration was detected and a thick Cr-depletion zone



Cross-sectional SEM of the four alloys during the 1500 hours

Oxides Thickness Comparison (550°C vs 700°C)





Remarks

- Inconel 625 exhibited the best performance at both temperatures
  - 625 had among lowest mass gains and developed the thinnest and most stable oxide layer
- Longer testing needs to be done to determine maximum possible oxide thickness for these materials
  - Long term exposure testing (4,800 hours) at 700°C/200 bar has been completed for 740H, 625, HR-120 & 316L
- Further testing needs to be performed using sCO_2 in the presence of impurities, in particular O_2 and H_2O
  - New sCO_2 corrosion test facility with impurities will be commissioned by the end of this year at CE-O

Acknowledgment

This research work was funded by NRCan's Program of Energy Research and Development (PERD). Alloys samples were supplied by EPRI, through Carleton University. Experiments performed by CanmetENERGY-Ottawa. Characterization of samples was done by National Research Council (NRC).

Contact Information

Dr. Kourosh Zanganeh

Senior Research Scientist and Group Leader

CanmetENERGY-Ottawa

Natural Resources Canada

kourosh.zanganeh@nrcan-rncan.gc.ca

Ms. Margarita Ilinich

Research Engineer

CanmetENERGY-Ottawa

Natural Resources Canada

margarita.ilinich@nrcan-rncan.gc.ca