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Supercritical CO_2 Round Robin Test Program

Julie D. Tucker and Many More

March 29, 2018

Nuclear Materials & Metallurgy
NMM
Oregon State University

School of Mechanical, Industrial, and Manufacturing Engineering
COLLEGE OF ENGINEERING

Overview

- **DOE NEUP**
  - *Advancement of Supercritical Carbon Dioxide Technology through Round Robin Testing and Fundamental Modeling*
  - 2015-2018
  - Nuclear Reactor Technology Program

- **Co-PIs & Collaborators**:
  - Julie Tucker (PI) & Líney Árnadóttir Oregon State University
  - Mark Anderson - University of Wisconsin, Madison
  - Bruce Pint - ORNL
  - Ömer Doğan - NETL
  - Henry Saari - Carleton University, Canada
  - Changheui Jang - KAIST
  - John Shingledecker - EPRI

NEUP Project Objectives

- Develop a sCO_2 materials working group
- Perform round robin test program
- Comparison of sCO_2 to SCW data
- Joint testing in sCO_2
- Empirical modeling
- Atomistic modeling of fundamental mechanisms

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Round Robin Team Members

Round Robin Testing
- Oregon State University: Julie Tucker, Lucas Teeter, Benjamin Adam
- University of Wisconsin-Madison: Mark Anderson, Jacob Mahaffey*
- ORNL: Bruce Pint
- NETL: Omer Dogan, Gordon Holcomb, Casey Carney
- Carleton University: Henry Saari
- KAIST: Changheui Jang

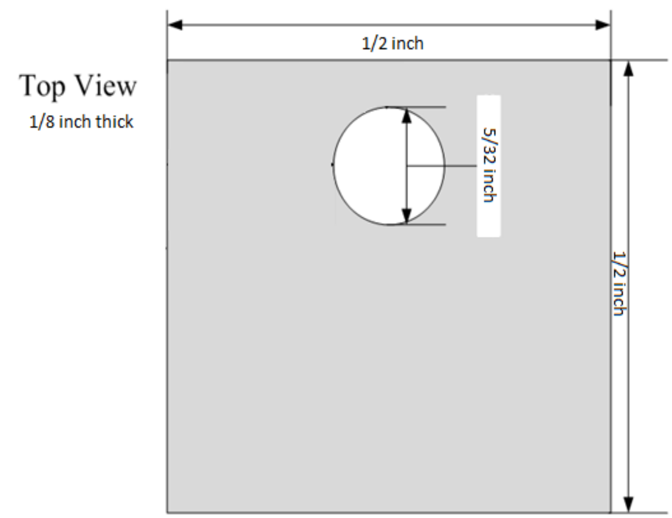
Alternative Testing
- CSIRO Energy Center: Rene Olivares

Additional Support
- EPRI: Steven Kung & John Shingledecker

*Sandia National Lab

Round Robin Goals & Parameters

- Demonstrate comparable and reproducible results across organizations performing sCO_2 corrosion testing
- Environment: Research grade CO_2 (99.999% pure)
- Time of Exposure: 1500h in 500h increments
- Temperatures: 550°C and 700°C
- Pressure: 20 MPa
- Testing 5 alloys
    - 740H (700°C only)
    - 625
    - 316L
    - HR120
    - Grade 91 (550°C only)



Characterization Plan

Characterization	500h	1000h	1500h
Weight Change	6	5	4
SEM-EDS	1	1	1

- OSU performs AR characterization on all alloys
- Testing on 6 sample of each alloy at each temperature
- Weight change and SEM oxide thickness at each checkpoint
- Selected XRD, TEM and XPS as needed

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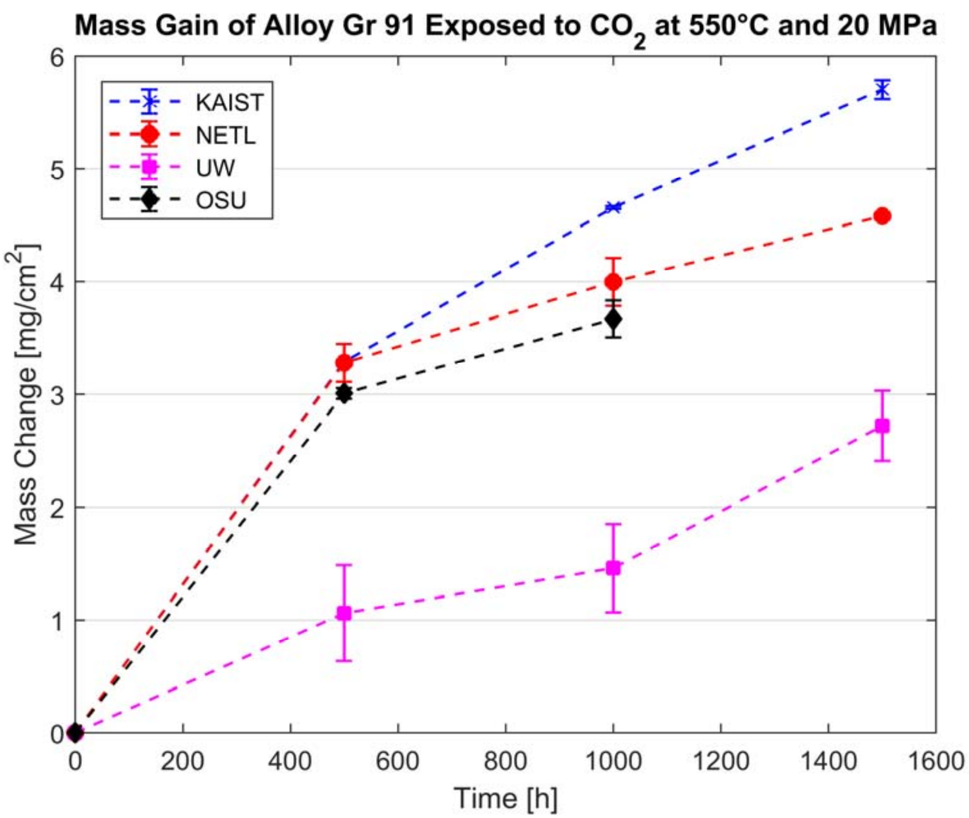
Mass Change Results – Grade 91 @ 550°C



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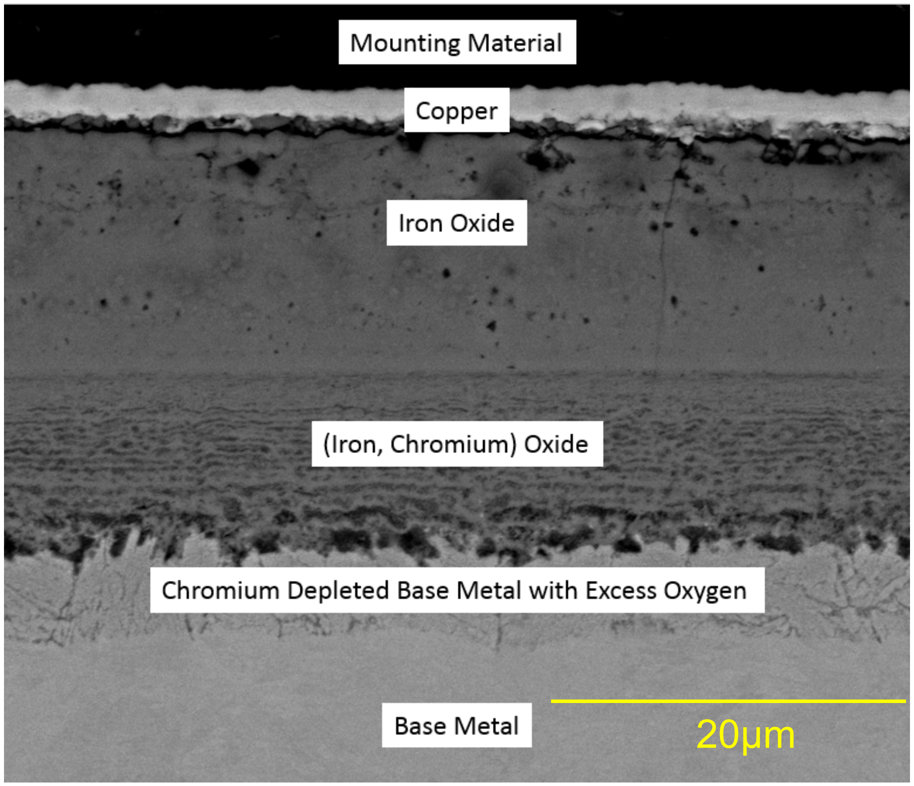
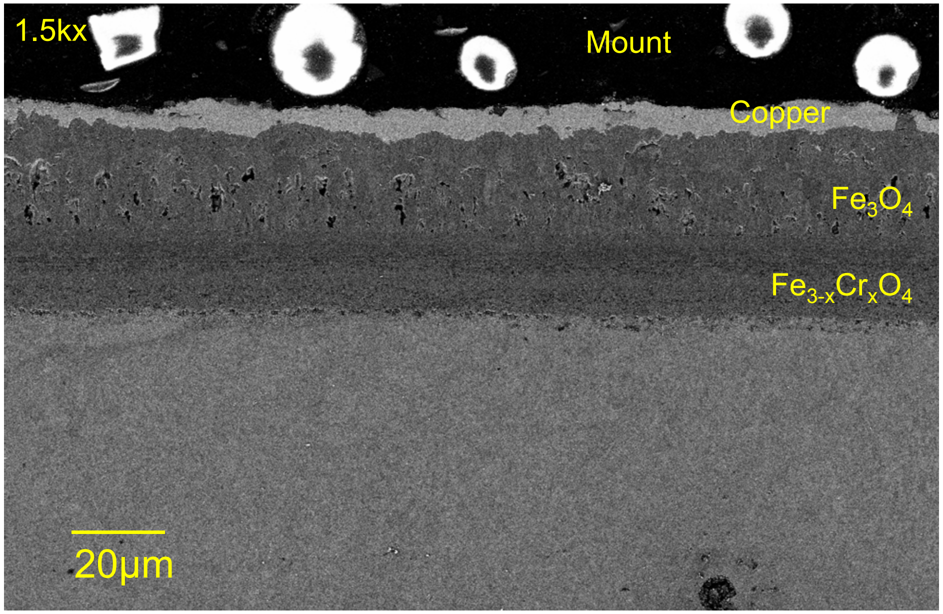
Oxide Characterization – Grade 91

OSU exposure 1000h

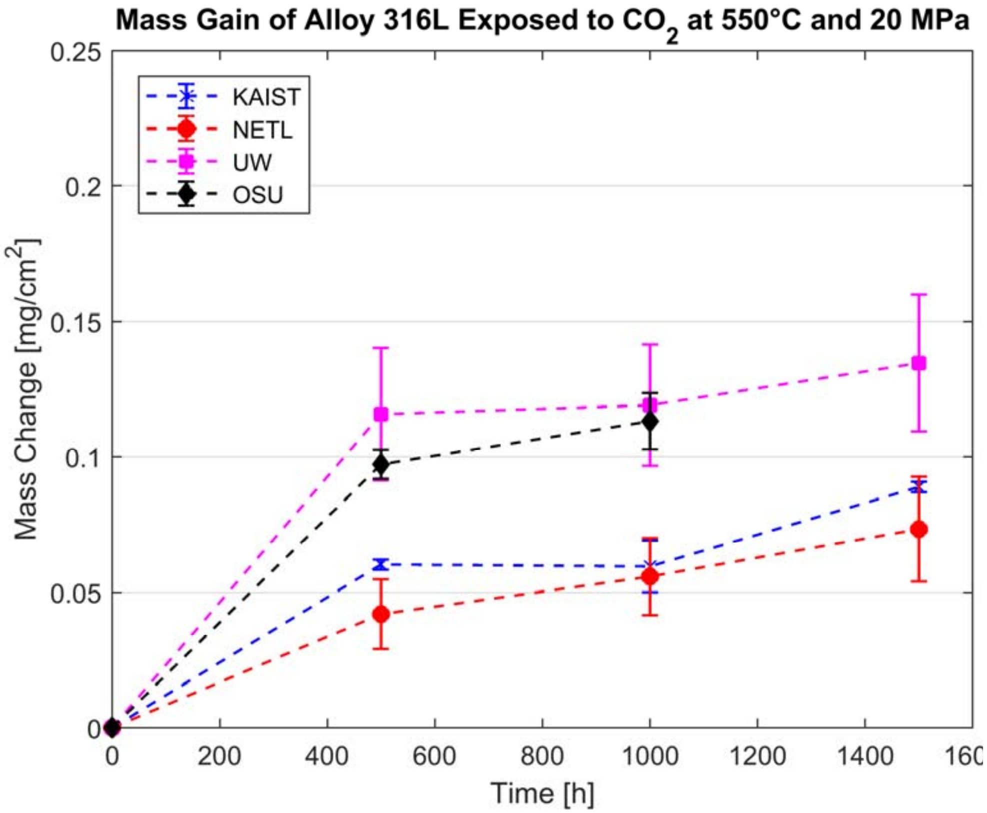


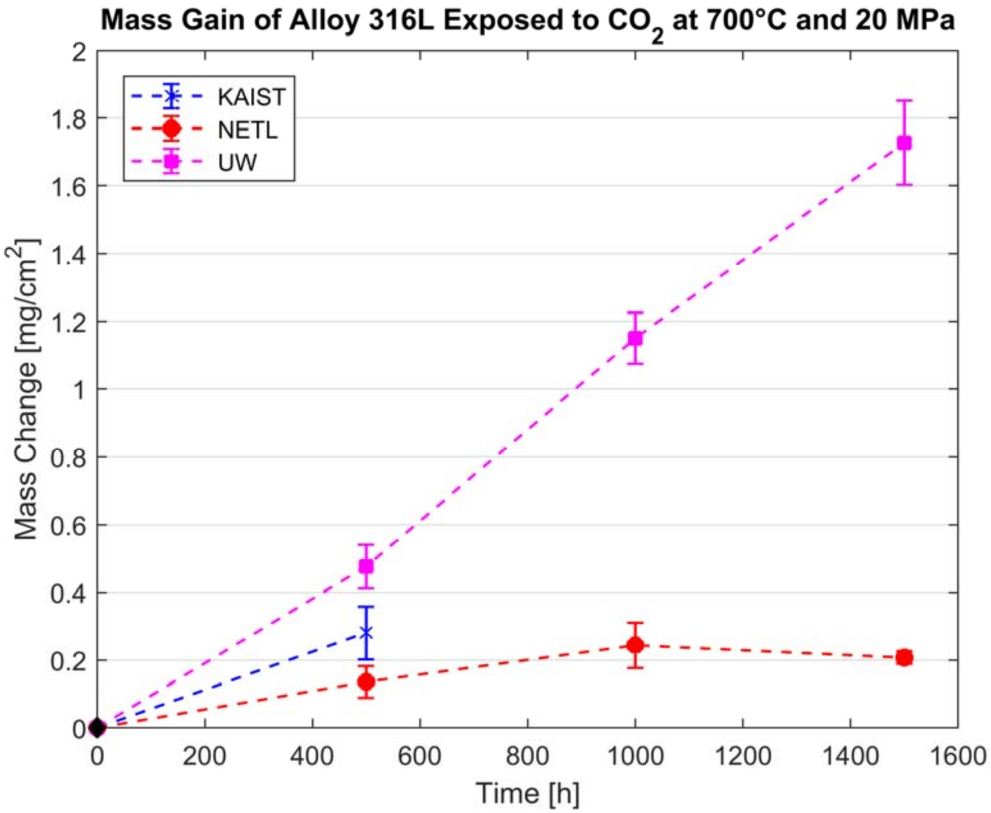
UW exposure 1500h



Oxide Thickness
OSU 1000h ~23μm
UW 1500h ~40μm
KAIST 1500h ~39μm

Mass Change Results





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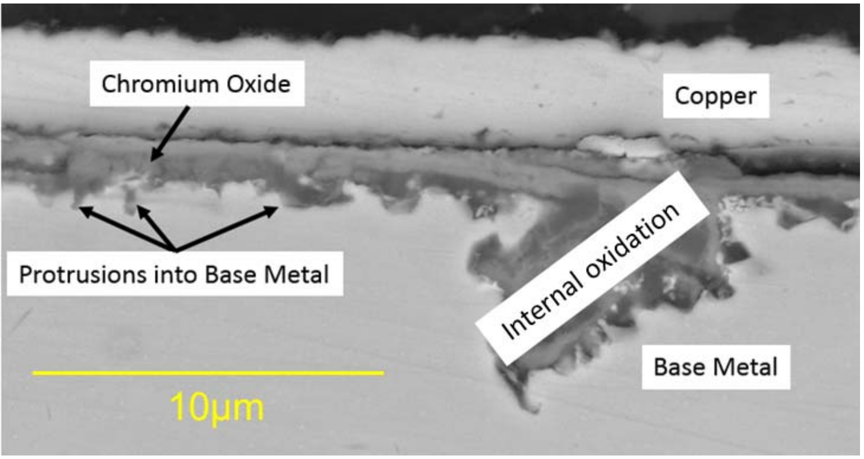
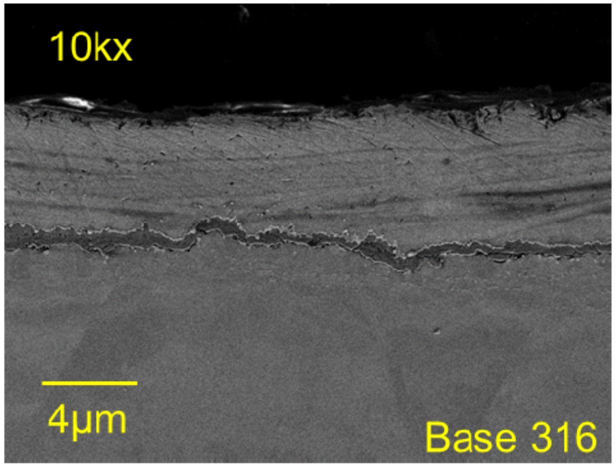
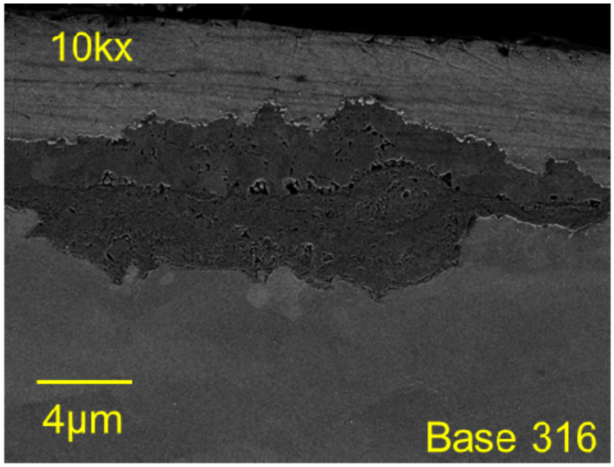
Oxide Characterization – 316L @ 550°C

OSU exposure 1000h



UW exposure 1500h





Oxide Thickness
OSU 1000h ~1.5µm
UW 1500h ~0.5µm continuous/ ~6µm (nodules)
KAIST 1500h ~6µm

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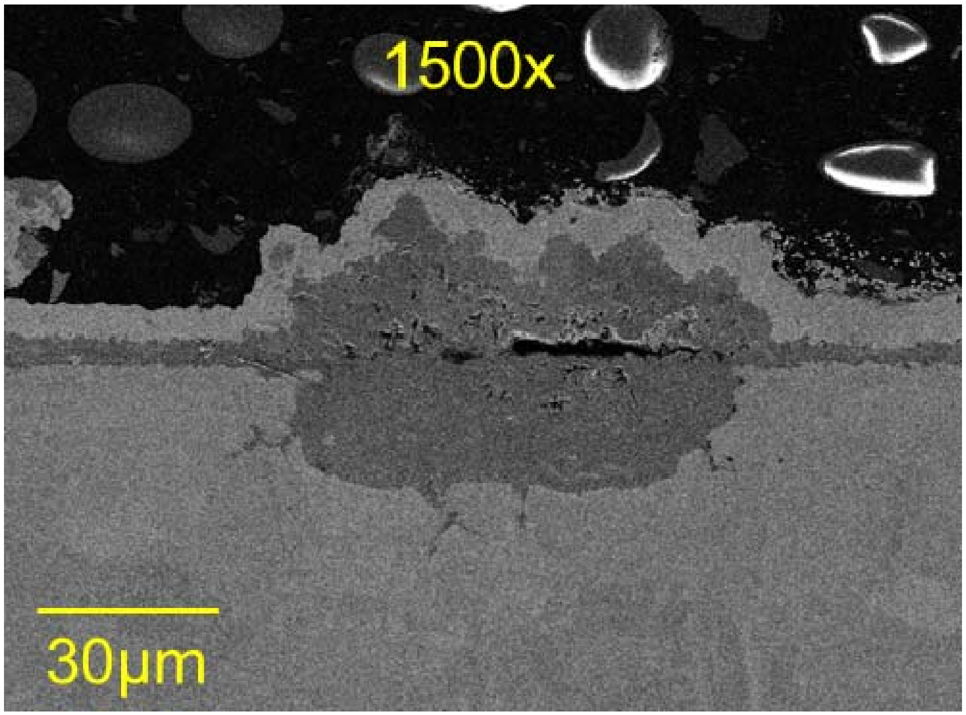
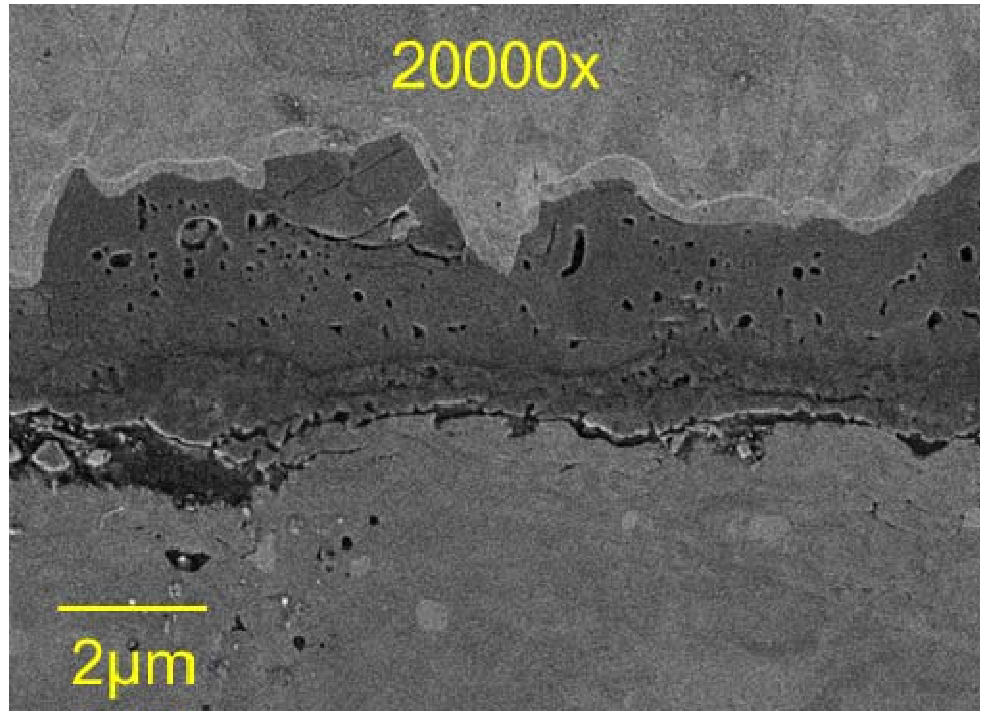
Oxide Characterization – 316L @ 700°C

UW exposure 1500h





Oxide Thickness at 1500h
UW ~3μm continuous/ ~36μm (nodules)

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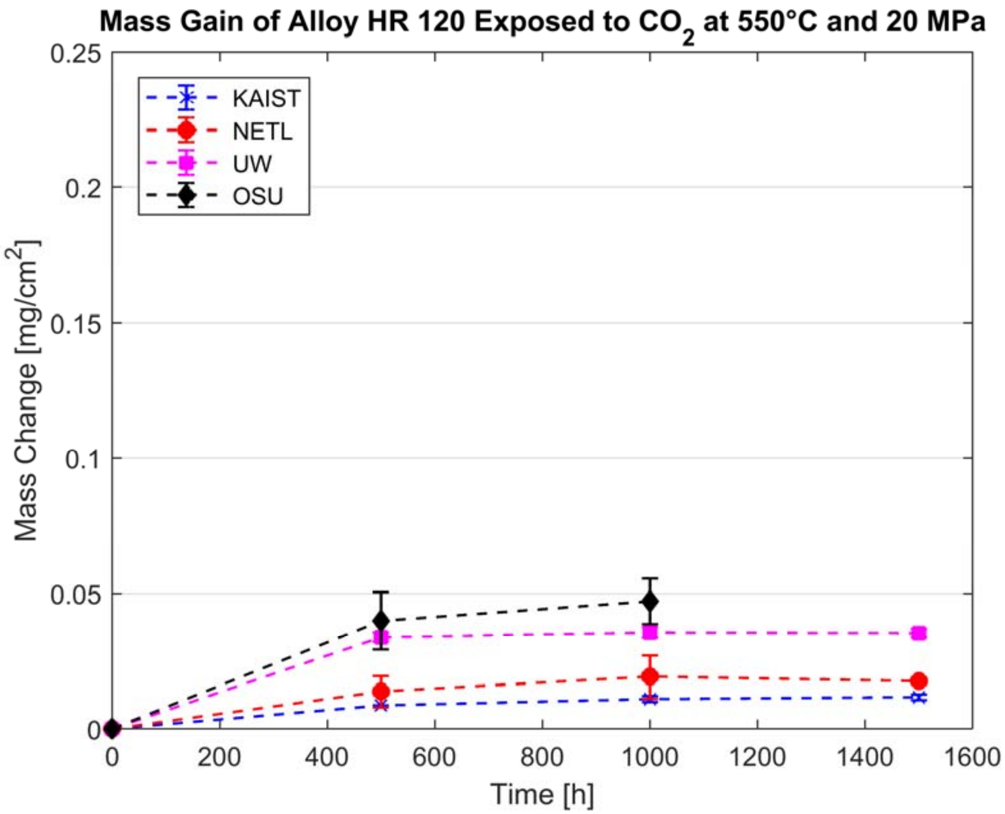
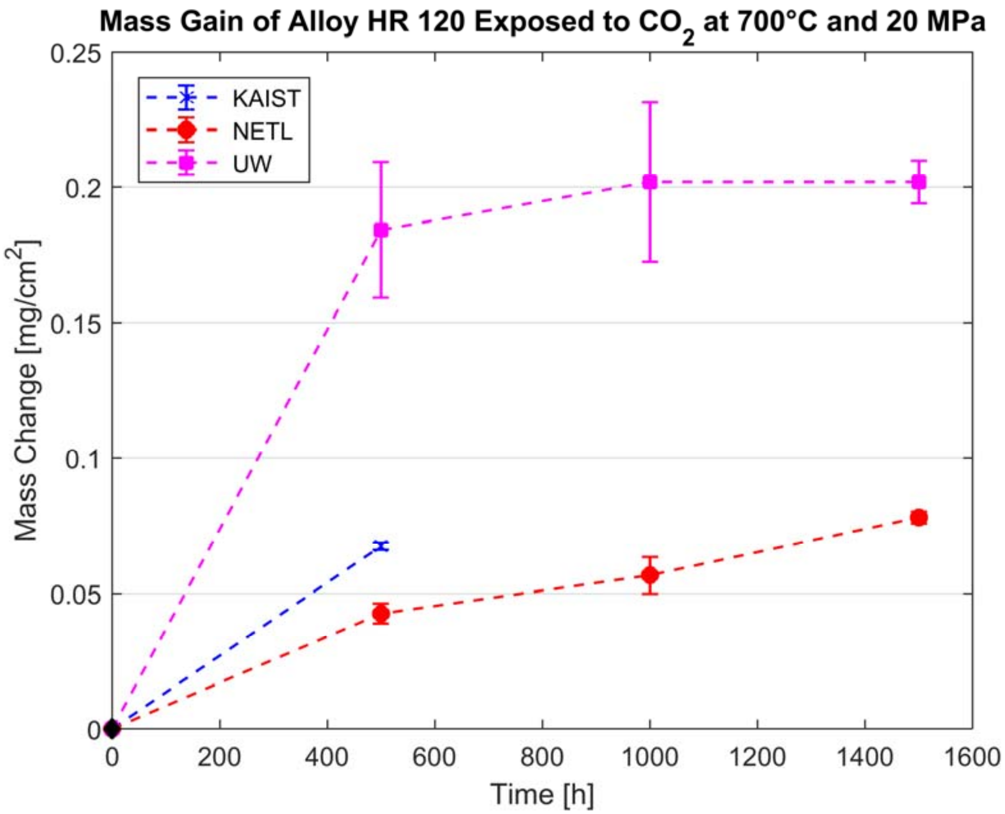
Mass Change Results – HR 120





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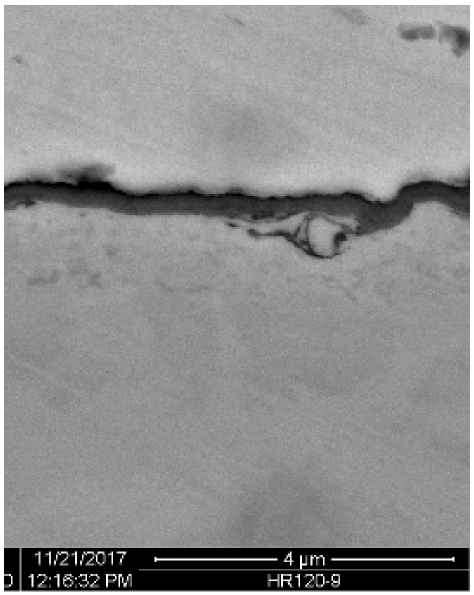
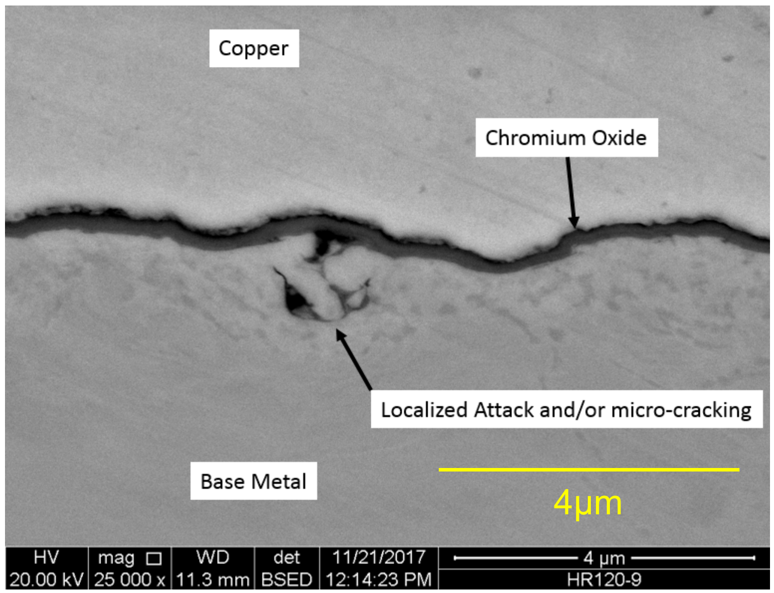
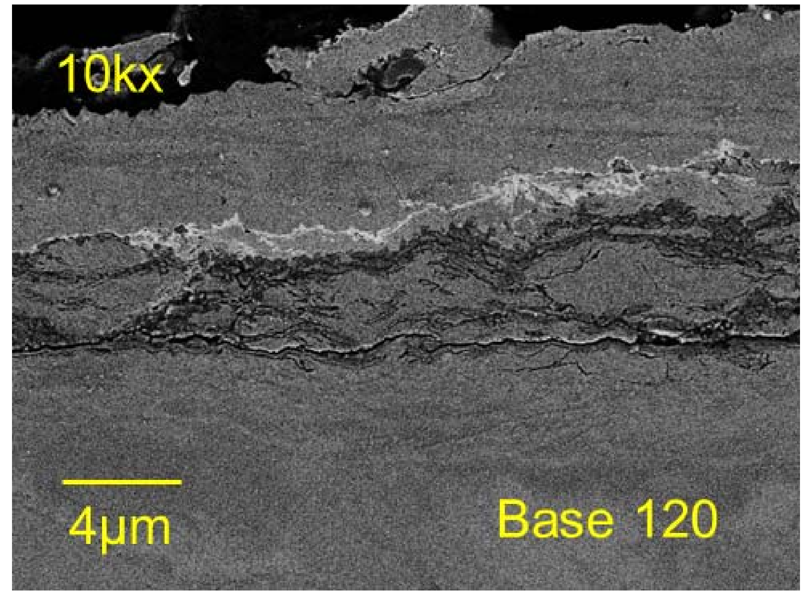
Oxide Characterization – HR120 @ 550°C

OSU exposure 1000h





UW exposure 1500h



Oxide Thickness

OSU 1000h ~0.4µm

UW 1500h ~3µm continuous

KAIST 1500h ~0.1µm

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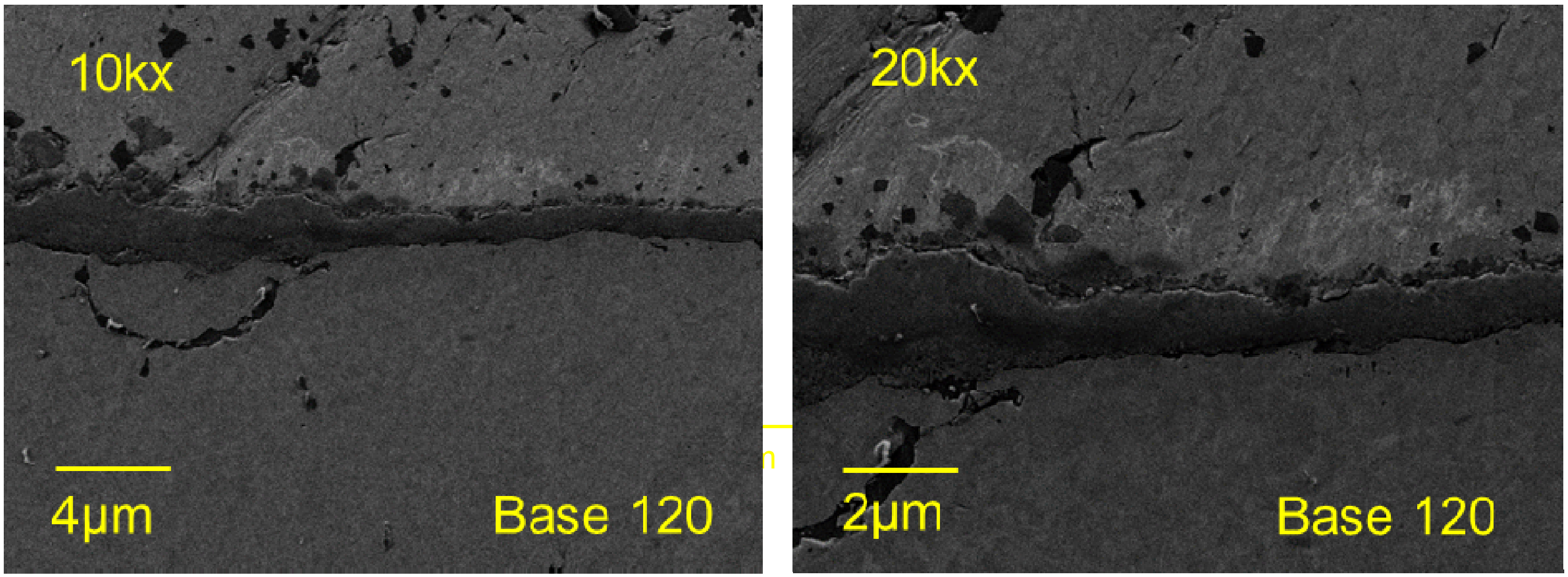
Oxide Characterization – HR120 @ 700°C

UW exposure 1500h



Oxide Thickness

UW 1500h ~1.3μm continuous/3μm nodules

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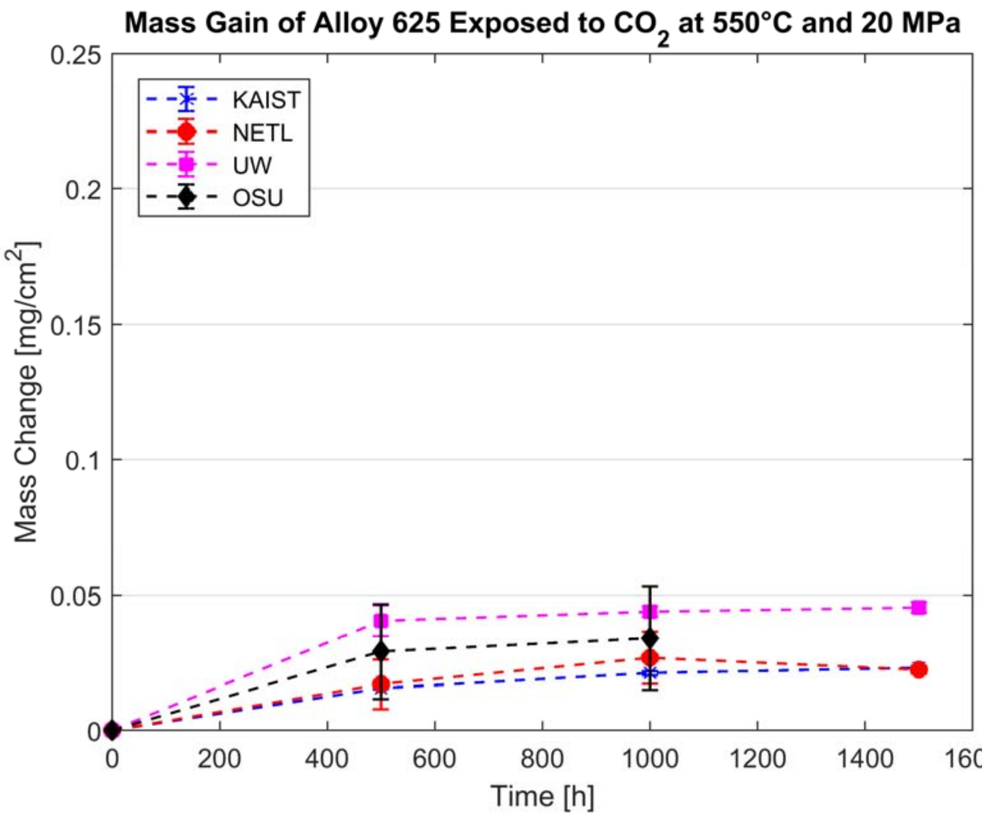
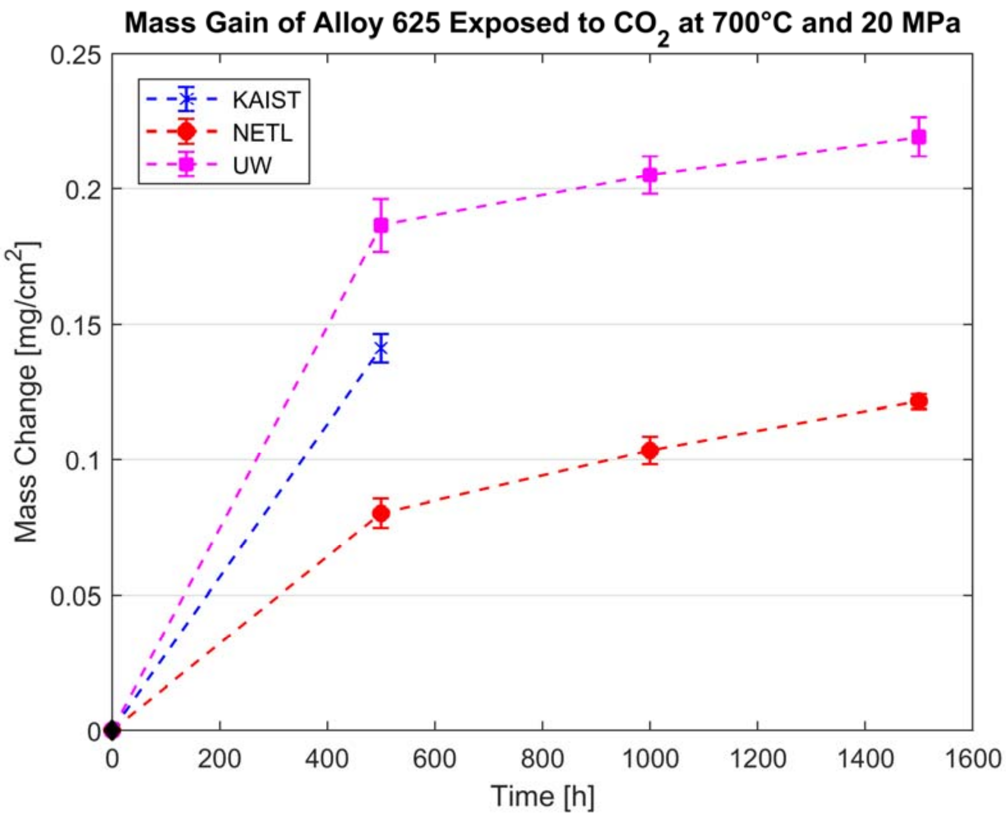
Mass Change Results - 625





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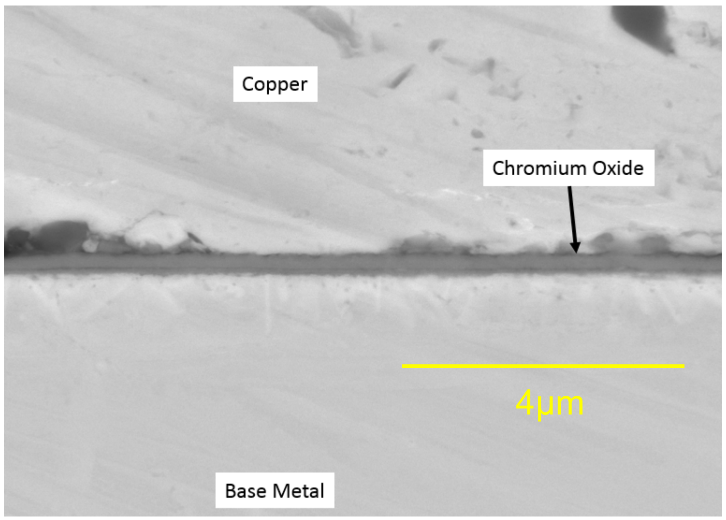
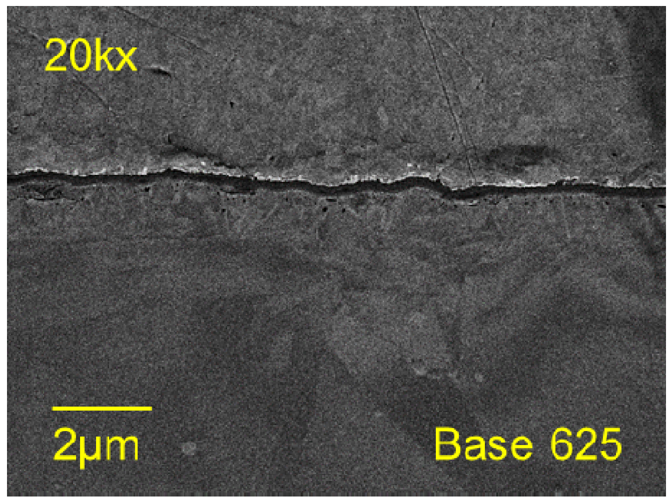
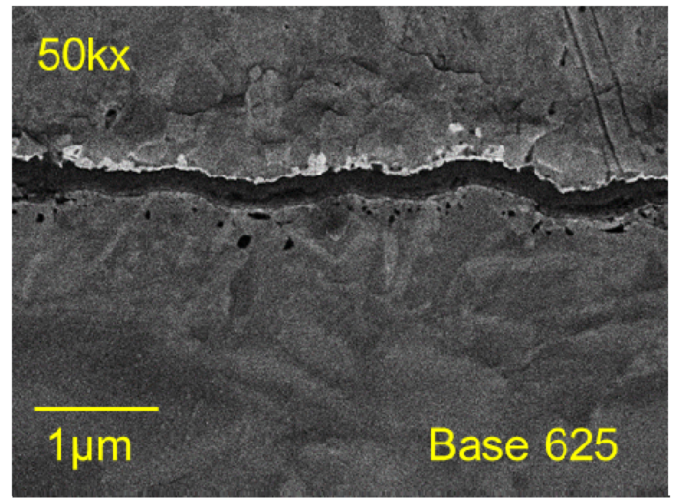
Oxide Characterization – 625 @ 550°C

OSU exposure 1000h



UW exposure 1500h





Oxide Thickness
OSU 1000h ~0.2µm
UW 1500h ~0.2µm continuous
KAIST 1500h ~0.2µm

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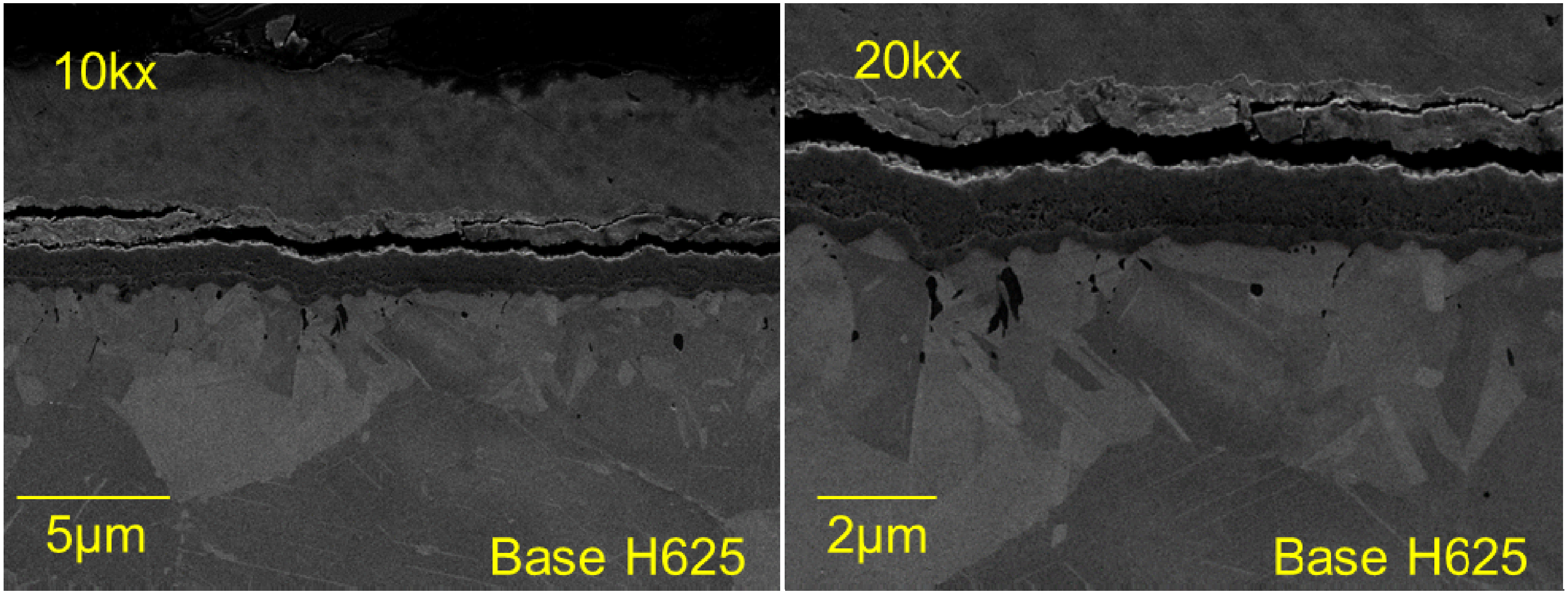
Oxide Characterization – 625 @ 700°C

UW exposure 1500h



Oxide Thickness at 1500h
UW ~1.3µm

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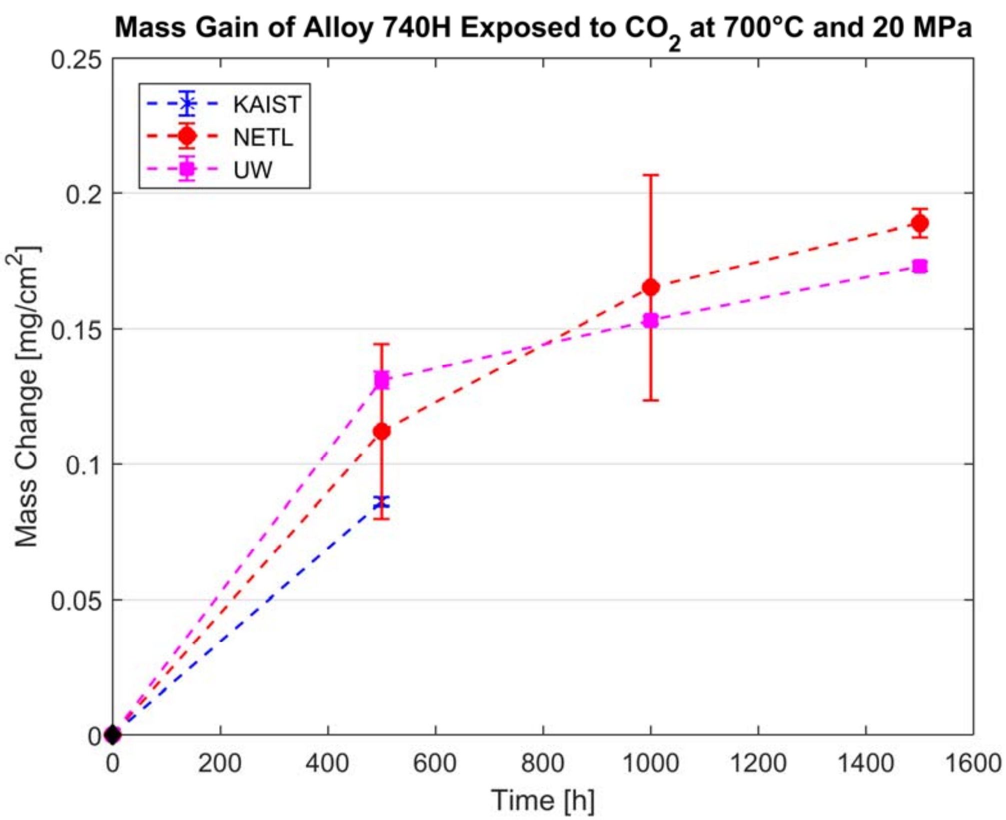
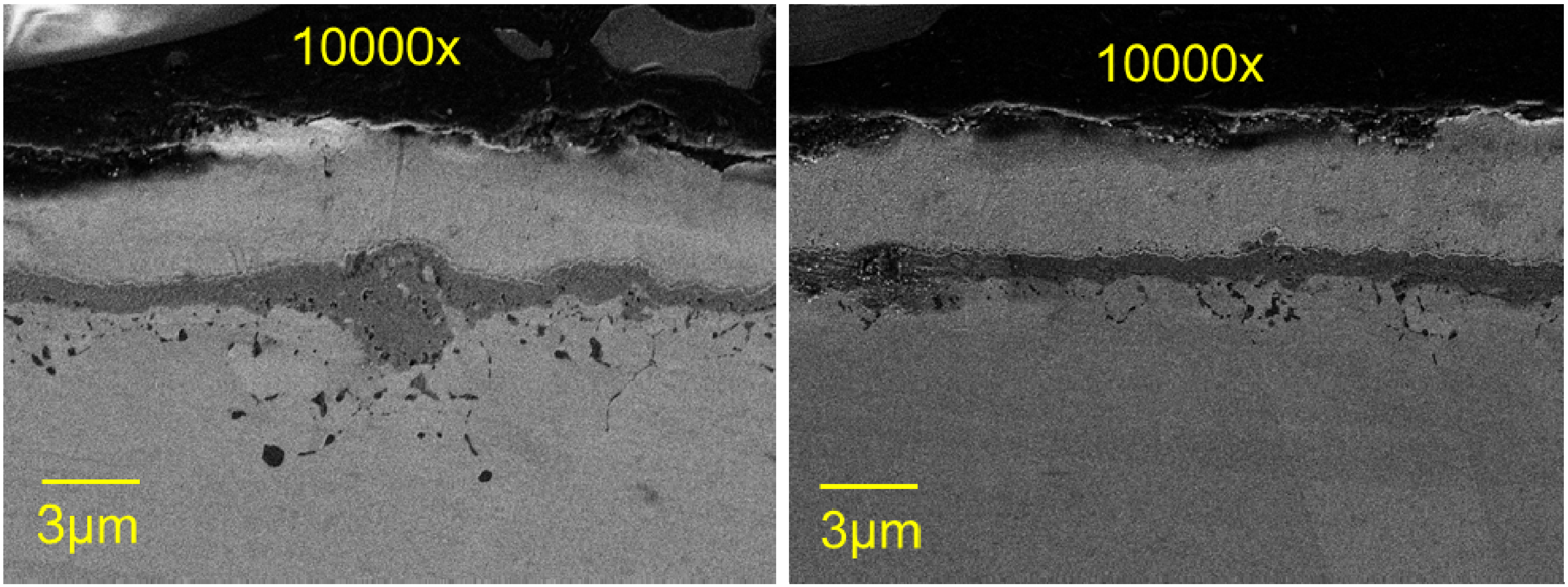
Mass Change Results – 740H



Oxide Characterization – 740H @ 700°C

UW exposure 1500h



Oxide Thickness at 1500h
UW ~1µm

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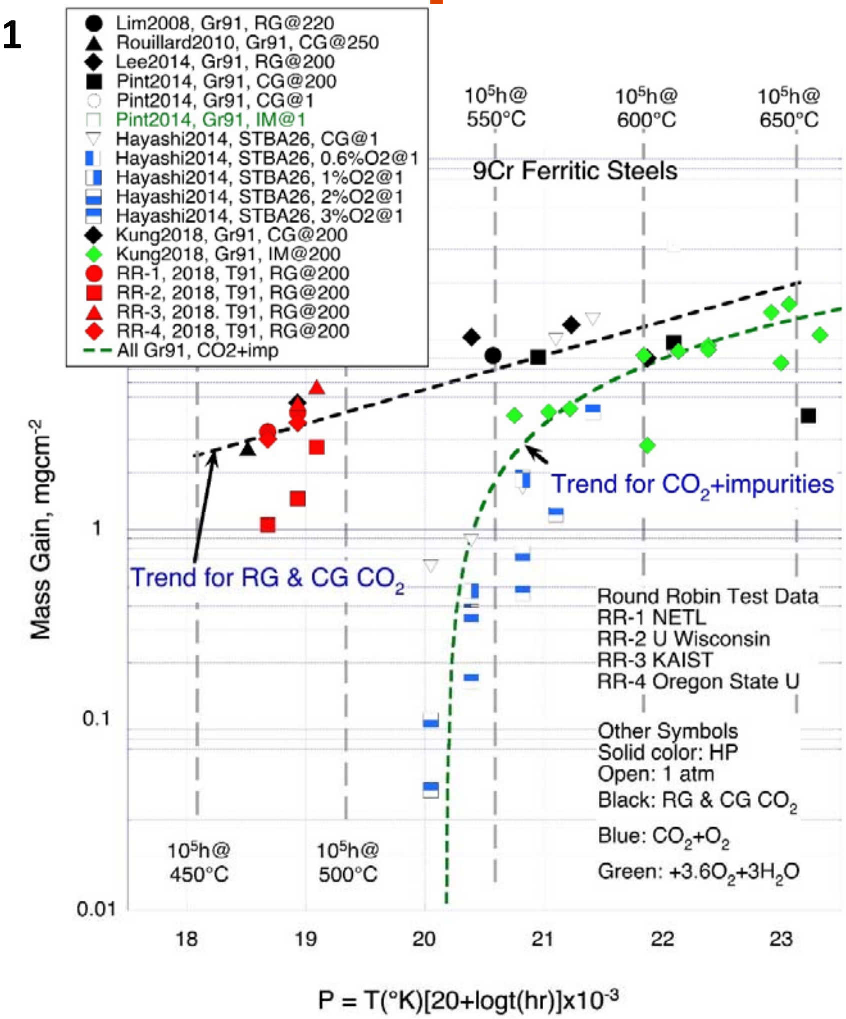
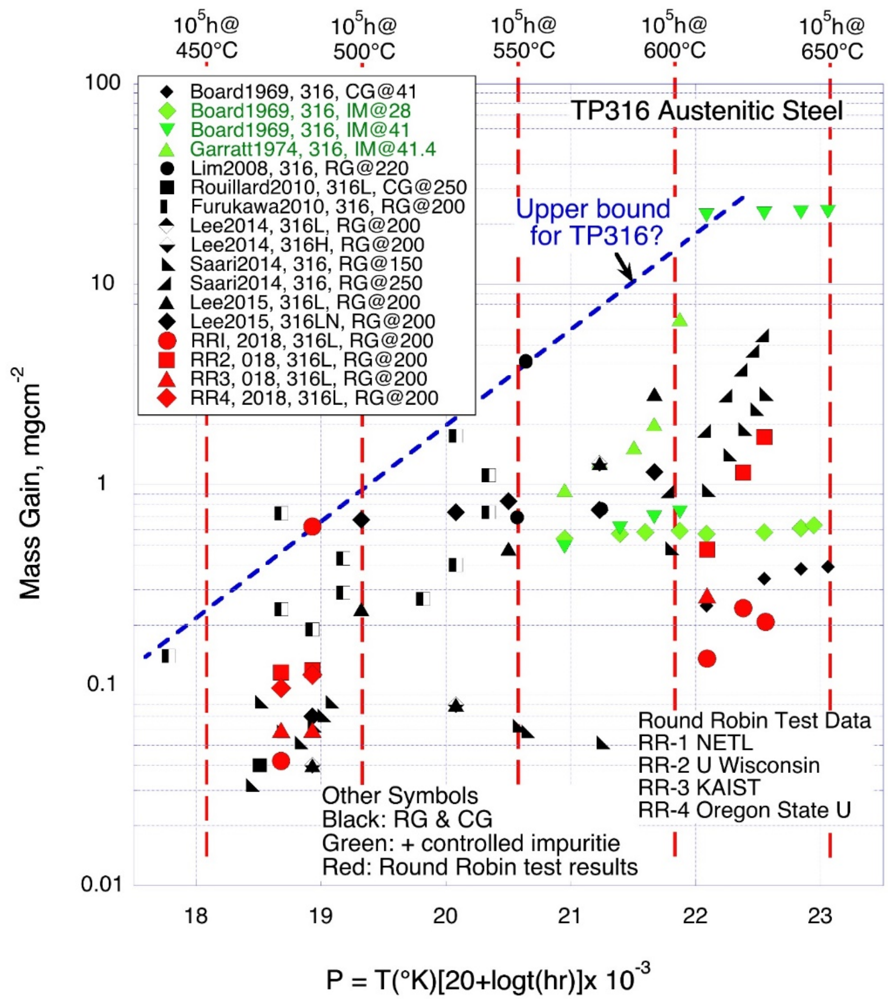
Comparison to Literature Results

Gr91

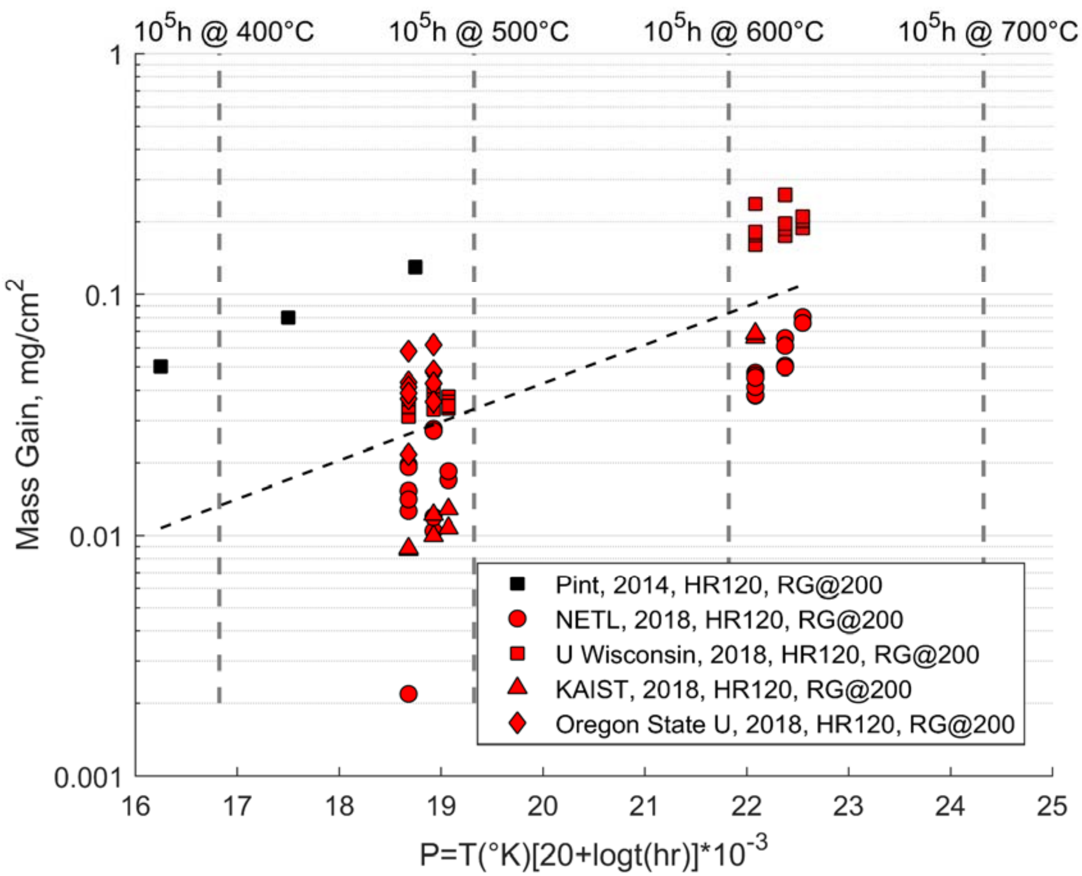


316SS



Comparison to Literature Results

HR120



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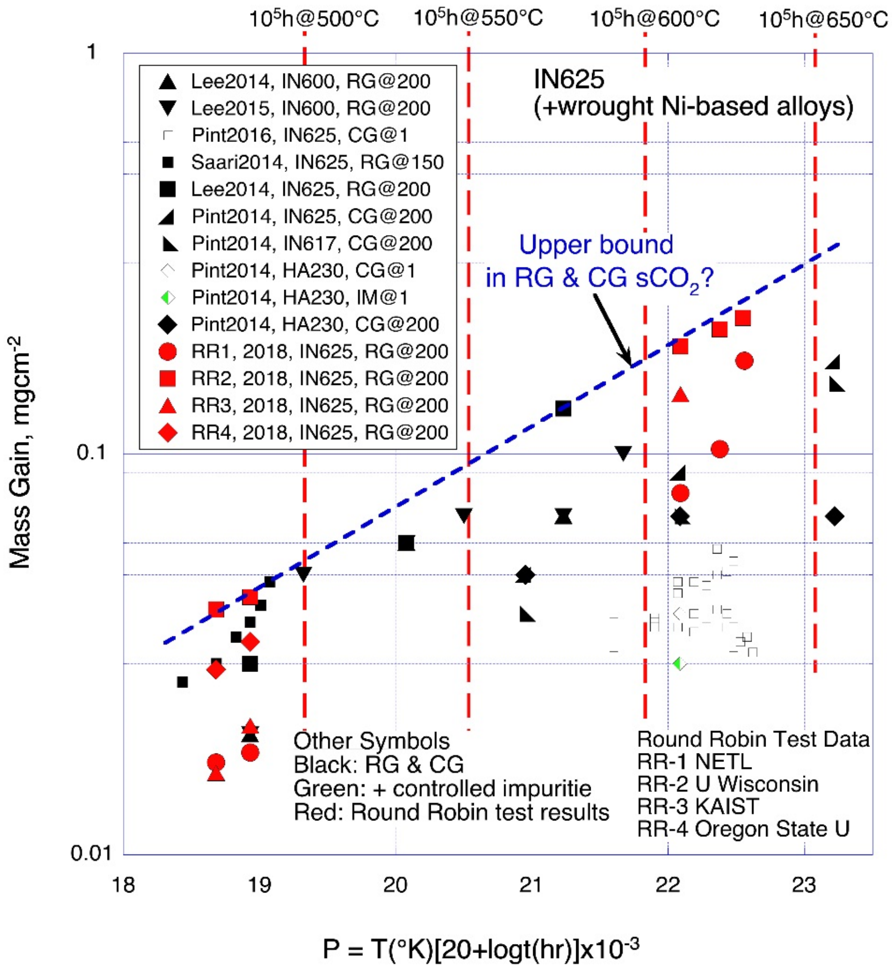
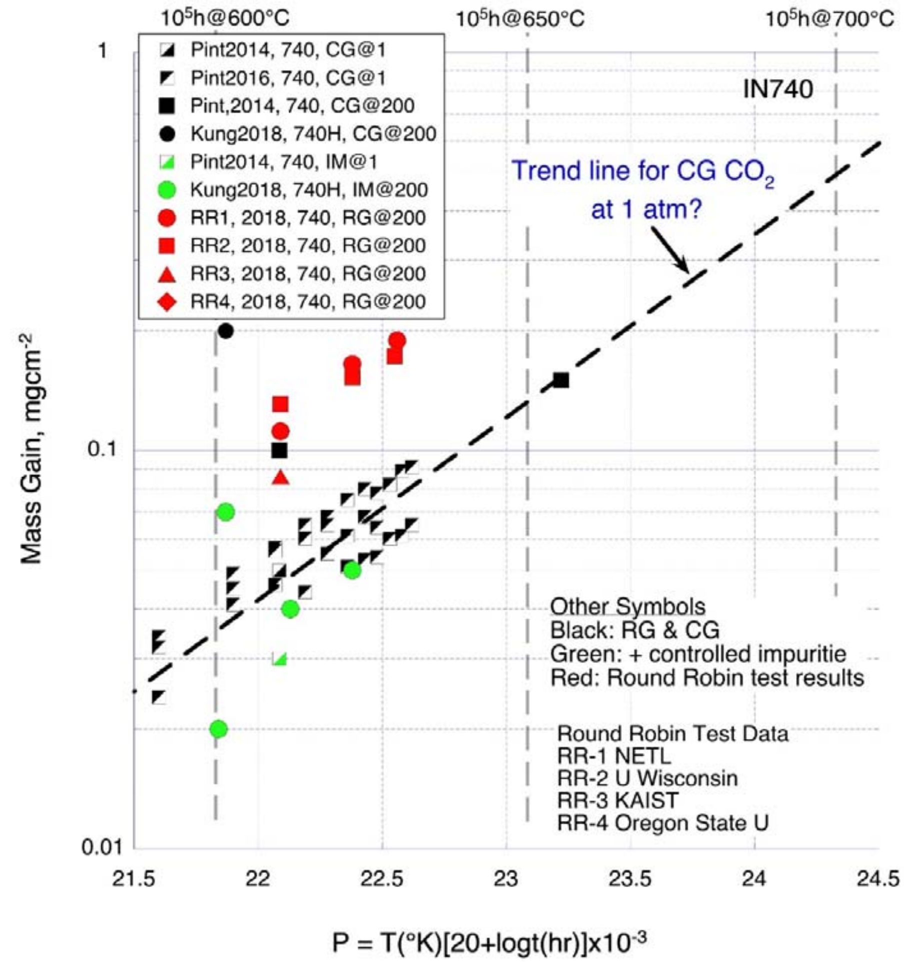
Comparison to Literature Results

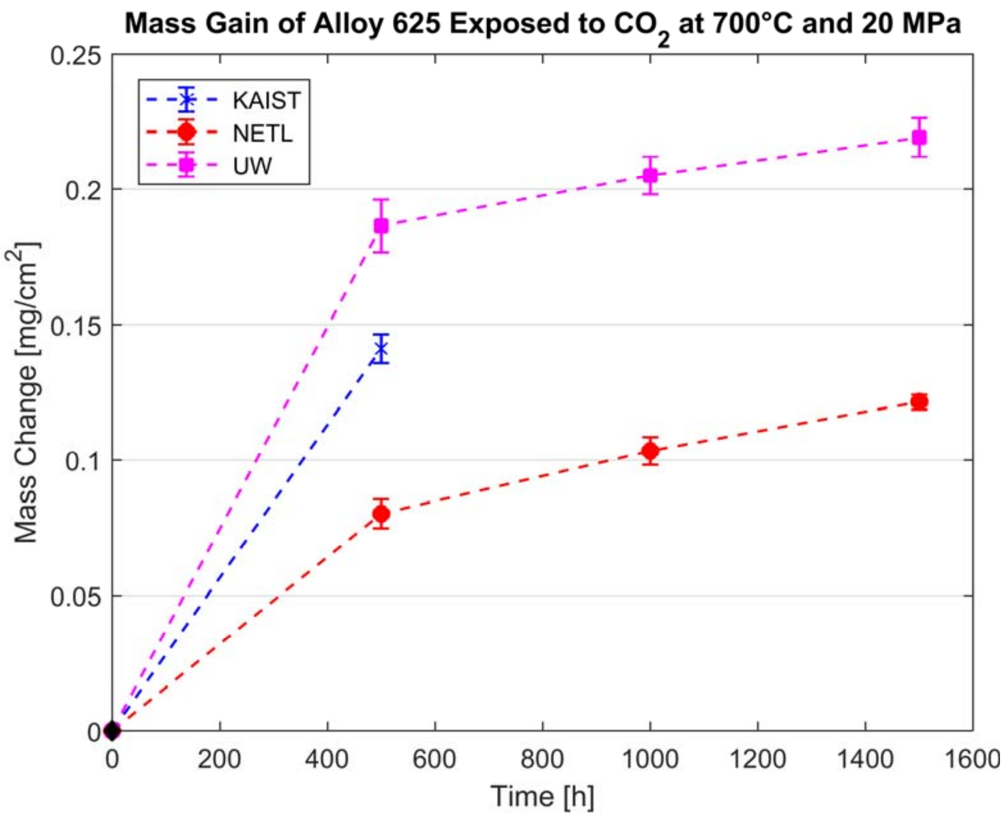
625

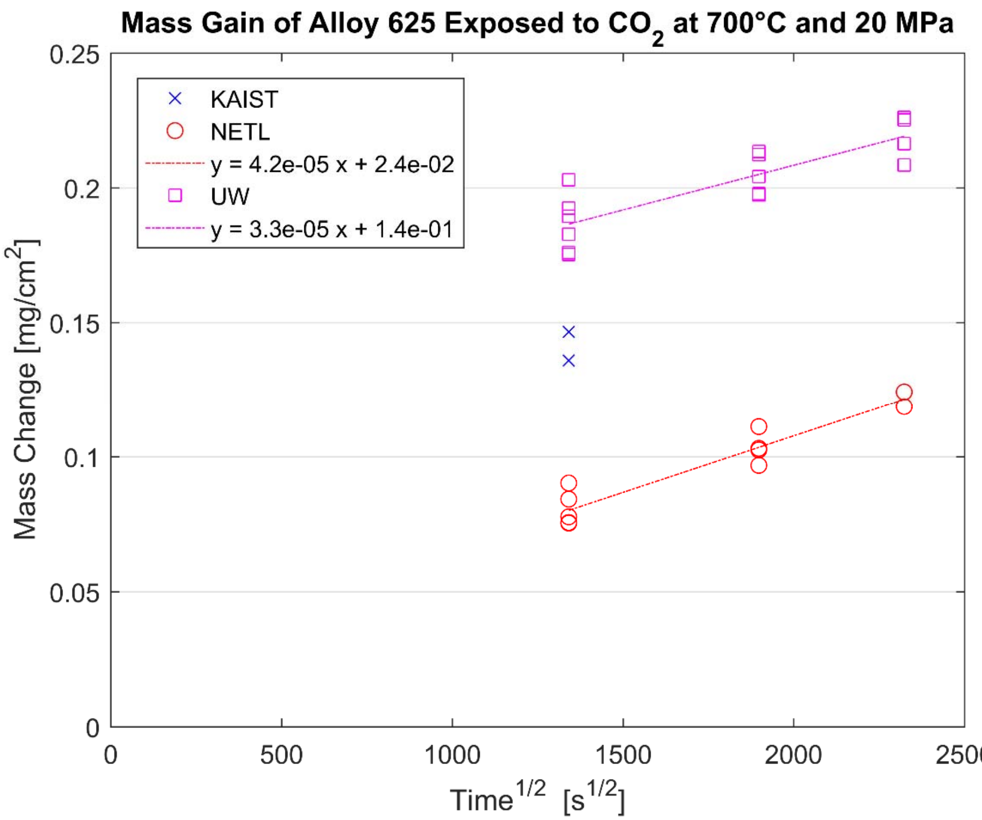


740



Calculation of Rate Constants





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Parabolic rate constant K_p (mg^2/cm^4-s)

	550°C				700°C			
Institution	G91	316L	HR 120	625	316L	HR 120	625	740H
KAIST	6.3E-06	7.8E-10	9.6E-12	5.9E-11	-	-	-	-
NETL	1.7E-06	9.0E-10	3.0E-11	7.6E-11	1.2E-08	1.1E-09	1.8E-09	6.9E-09
UW	2.6E-06	2.9E-10	2.9E-12	2.6E-11	1.7E-06	4.0E-10	1.1E-09	1.8E-09
OSU	1.4E-06	8.4E-10	1.7E-10	7.9E-11	-	-	-	-
Average	**3.0E-06**	**7.0E-10**	**6.1E-11**	**6.0E-11**	**8.5E-07**	**7.4E-10**	**1.4E-09**	**4.3E-09**
Std. Dev.	2.2E-06	2.8E-10	9.4E-11	2.4E-11	1.2E-06	4.9E-10	4.8E-10	3.6E-09

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Summary

- Mass change data available to date has shown reasonable agreement among teams and with existing literature data.
  - some discrepancies between data sets on alloys with higher corrosion rates
    - G91 at 550°C and 316L at 700°C
- Oxide thickness similar for G91
- Higher mass change shown for 316L is more consistent with data generated under similar conditions
- Additional data from more round robin teams and future oxide analysis will help to clarify sources of discrepancies

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Acknowledgements

- Materials donated by Special Metals, Haynes & EPRI
- Machined, polished and shipped by EPRI
- Ian Wright and the EPRI-DOE University Turbine Systems Research (*UTSR*) *Project for assistance in gathering data for the Larson-Miller plots.*
- Financial support from DOE NEUP DE-NE0008424 (Project 15-8495) *“Advancement of Supercritical Carbon Dioxide Technology through Round Robin Testing and Fundamental Modeling”*

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Materials Degradation in CO_2 Environments

Symposium at the…

Technical Meeting and Exhibition

MS&T18

MATERIALS SCIENCE & TECHNOLOGY

Abstracts due this
Saturday, March 31

OCTOBER 14 – 18, 2018
GREATER COLUMBUS CONVENTION CENTER
COLUMBUS, OHIO USA

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Questions?

Thank you!

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Round Robin Test Samples

- Materials donated by Special Metals, Haynes & EPRI
- Independent chemical analysis performed
- Machined, polished and shipped by EPRI

wt.%	Fe	Ni	Cr	Co	Nb	Mn	Mo	Al	Si	Ti	Cu
Gr 91	89.27	0.13	8.234	0.018	0.063	0.45	0.93	0.010	0.279	0.003	0.091
316L	68.29	9.93	16.844	0.214	0.009	1.58	1.98	<0.002	0.360	0.010	0.492
HR120	34.48	37.44	24.936	0.248	0.561	0.80	0.47	0.069	0.483	0.015	0.065
625	3.66	61.65	21.168	0.178	3.422	0.28	8.70	0.204	0.168	0.210	0.159
740H	0.11	50.42	24.144	20.421	1.559	0.23	0.31	1.312	0.153	1.374	0.002

Sample Summary

Alloy	Supplier	Material Supplied	Material Area	# Samples Total	# Samples per Lab	Material Need (Sheet)
740H	Special Metals	4.5”x11.5”sheet 1/8” thick	51.75 in^2	42	6	26 in^2
625	Haynes	2*(8”x8” sheet)	96 in^2	84	12	52 in^2
316L	Rolled Alloys	12x12 sheet 1/16” thick	144 in^2	84	12	52 in^2
HR 120	Haynes	2*(8”x8” sheet)	96 in^2	84	12	52 in^2
Grade 91	EPRI	Stock		42	6	26 in^2

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Round Robin Capabilities

Organization	Maximum Temperature	Maximum Pressure	Chamber Volume	Flow rate (mL/min)	Autoclave Material
OSU	800°C	26 MPa	1235 cm^3	0-24	Haynes 230
UW (2 systems)	750°C 760°C	25 MPa 38 MPa	900 cm^3 (combined)	0-24 0-24	Inconel 625 Haynes 282
ORNL	850°C	30 MPa	1400 cm^3	0-24	Haynes 282
NETL	800°C	28 MPa	1040 cm^3	0-24	Haynes 230
Carleton	750°C	25 MPa	1150 cm^3	0-250	Inconel 625
KAIST (2 systems)	700°C	25 MPa	1077 cm^3 (each)	0-24	Inconel 625
CSIRO	1000°C	25 MPa	68 cm^3		Variable Tube

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Pre-Exposure Sample Preparation

- Cut to size with EDM, water jet cutting or other low heat process
- Drill sample holes
- Polish the larger area surfaces to a 30 micron grit finish, which is equivalent to a 600-grit finish or 20-40 Mesh
- Hand polish the sides up to 600-grit.
- Rinse, Ultrasonic bath with Ammonium Hydroxide based solution, Rinse
- Ultrasonic bath with propanol remove oils and surface particles.
- Store in a desiccator until testing

Post-Exposure Sample Preparation

- 10-15nm gold sputter deposition layer
- Copper electroplating
- Sample mounting
- Polishing