Mechanical and Corrosion Performance of the Weld of 740H and 282

Andrew Brittan

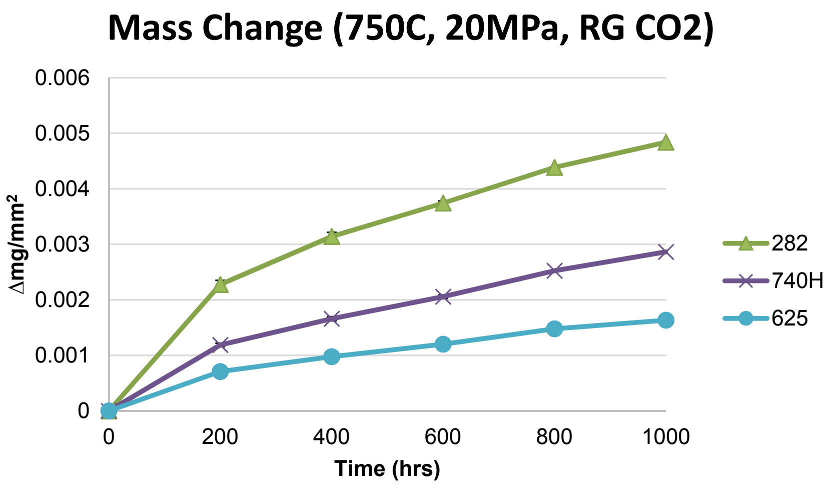
Acknowledgements: DOE-EE0007120
Special Metals
Haynes International

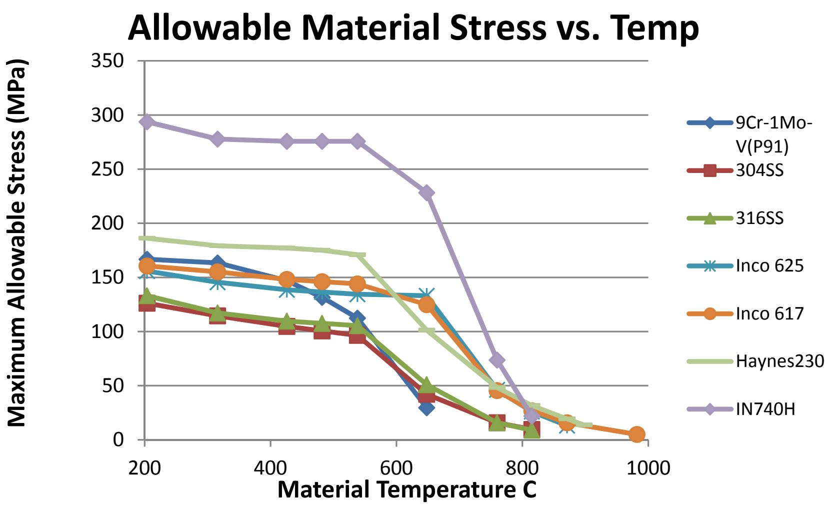
Outline

- Background
- $S\text{-}CO_2$ Testing Facility
- Sample Design & Procurement
- 740H Results
- 282 Results
- Other Exposure Work
- Overview & Planned Work

Background

- Ni-based superalloys are being considered for high temperature components due to **good corrosion resistance** and **good mechanical properties** at high temperatures.
- 740H and 282 have shown similar mass gain to other superalloys
- 740H is currently code case certified with high allowable stress at high temperatures



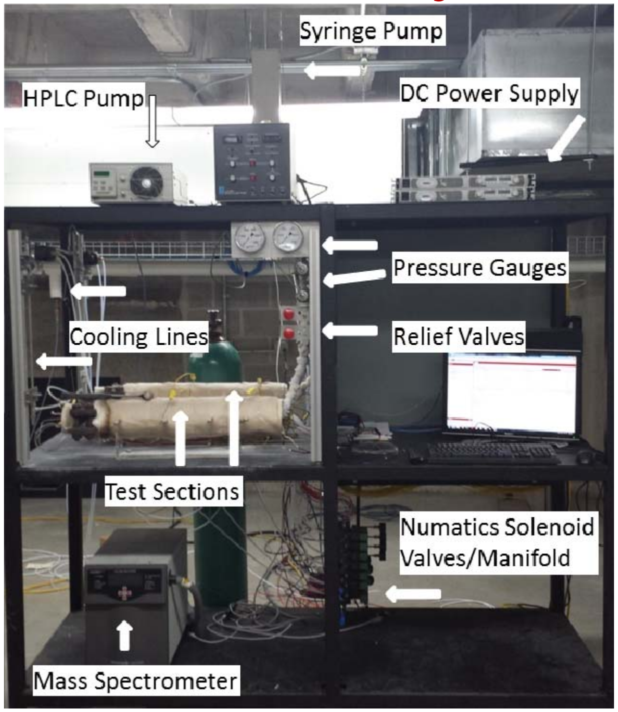


Source: Boiler, A. S. M. E. "Pressure Vessel Code Section II Part D, 2013,"Material properties (metric),"." *American Society of Mechanical Engineers*.

Background

- Failure points of pressure vessels are at the weakest points –welds are common weak points.
  - Important to understand the effect of the CO_2 environment on the mechanical properties of welds.
- Heat Treatment: prolonged exposure of alloys to elevated temperatures causes diffusion and coarsening/depletion of phases.
  - Ni-based superalloys of interest (740H and 282) both utilize γ' phase, $Ni_3(Al,Ti,Nb)$, for high temperature strength
  - Post-annealing or post-weld heat treatments are commonly at 800°C
  - Temperatures of interest for these alloys in my study: 750°C
- Carburization: dissociation of CO_2 into C during oxidation process liberates carbon
  - Typical to find carbides near grain boundaries from simply heat treating these alloys due to compositional carbon
  - Analyze the effect of increased carbon in the system
- **Large Concern**: Chromium-carbide formation causes Cr-depletion in the matrix – increases γ' solubility (they then dissolve into the base matrix)
  - Result = lower strength near carbides.

High Temperature SC-CO_2 Autoclave System at UW-Madison

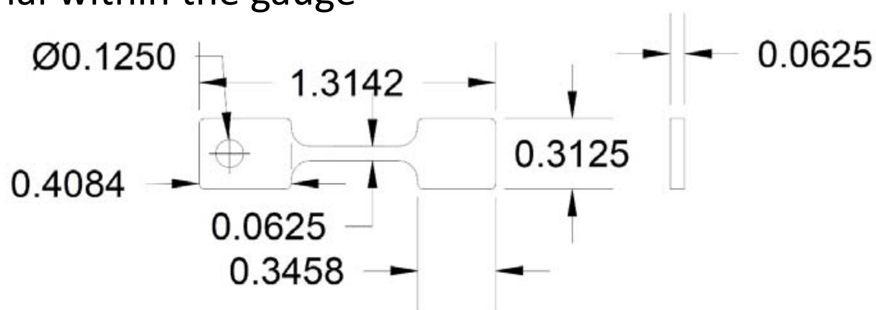


Current setup of test facility

- **Temperature control allows system to operate within ±1.5ºC**
- **Testing temperatures range up to 850°C**
- **Pressure can be held at up to 25±0.02MPa (temperature dependent)**
- **System operates at an average flow rate of 0.11kg/hr, which causes a CO_2 refresh rate every two hours**
- **Real-time gas composition analysis (GCMS, Mass Spectrometer)**

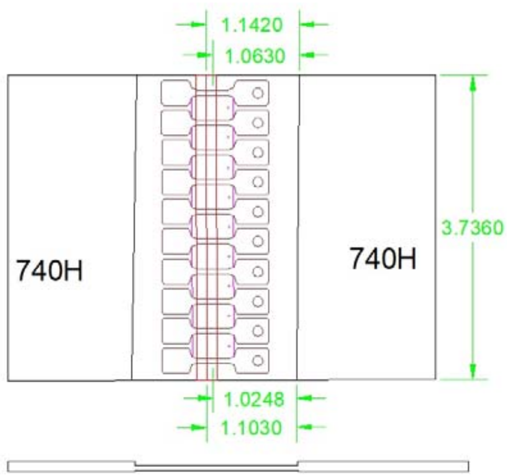
Sample Design

- Samples designed to fit ASTM standard while fitting inside autoclaves to be aged in s-CO_2 (ID ≈ 1.5”)
- From there, optimized gauge length and tab size according to:
  - Maximum gauge length to incorporate weld/HAZ
  - Large enough tabs to ensure tensile test can be conducted without slippage
  - Hole in one tab in order to suspend during exposure to CO_2 (tab slightly elongated to maintain surface area for gripping during tensile testing)
- Weld placed slightly off-center so as to incorporate all three features of [1)]fusion zone, [2)]heat affected zone, and [3)]base material within the gauge



Sample Procurement

- Superalloy was procured from and welded by the manufacturers in the form of 0.125” plates:
  - 740H – Special Metals
  - 282 – Haynes
- Plates were butt-welded using a GTAW process with similar filler metal and given the prescribed PWHT.
- Surface features – bead, oxidation, any warping – were avoided by machining down from both sides on the plate to ~0.0625” thickness.
- Samples were cut by EDM, transverse to the weld, with additional weld samples for corrosion analysis cut in the space between the gauges.



Welded 740H sample - etched

Tensile Results – Unexposed

- Prior to exposure, samples were tensile tested in order to ensure weld was of high enough quality for an accurate comparison.
- All testing performed at room temperature.
- Special Metals weld of 740H performed up to their standard.
- Haynes weld was found to have slightly lower properties than found in literature.
  - Haynes confirmed results were within expected tolerances.

	UTS (MPa)	0.2% YS (MPa)	Elongation (%)	Failure Location
740H Unexposed	1,111 ± 6	679 ± 2	38.1 ± 1.3	N/A
740H-740H Unexposed	1,068 ± 22	759 ± 16	26.3 ± 2.6	Fusion Zone
282 Unexposed	1,105 ± 15	728 ± 7	39.9 ± 0.4	N/A
282-282 Unexposed	867 ± 25	637 ± 11	10.1 ± 1.0	FZ & FZ/HAZ Interface

Testing Conditions

- 1,000 hours of exposure performed continuously
- Environment: research grade CO_2 (99.999% pure)
- Temperature: 750°C
- Pressure: 20MPa

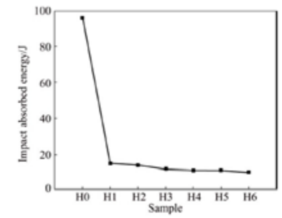
Tensile Results – 740H

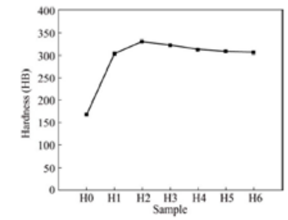
- Base Material Samples:
  - UTS (tensile strength) and YS (yield strength) increased, with only a moderate drop in elongation
  - Effect is consistent with continued aging of material – coarsening of γ’ precipitates & intergranular carbides
- Welded Samples:
  - Significant drop in UTS and YS, with a large drop in elongation.

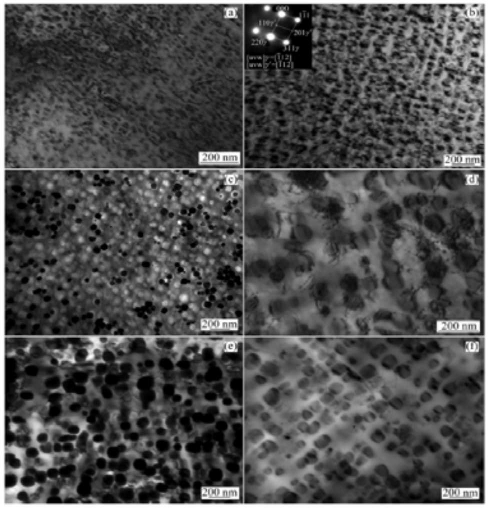
	UTS (MPa)	0.2% YS (MPa)	Elongation (%)	Failure Location
740H: Unexposed	**1,111 ± 6**	**681 ± 3**	**38 ± 1.3**	**N/A**
740H: 750°C CO_2	1,187 ± 2	713 ± 3	26 ± 0.5	N/A
Welded 740H: Unexposed	**1,068 ± 22**	**759 ± 16**	**26 ± 2.6**	**Fusion Zone**
Welded 740H: 750°C CO_2	940 ± 10	638 ± 7	12 ± 2.0	Fusion Zone

740H – Exposure Analysis

- 740H has been shown to have precipitate free zones (PFZ) along grain boundaries after initial heat treatment (due to formation of $M_{23}C_6$ carbides)[1]
- Heat treatment for 1,000 hours at 750°C has been shown to result in:
  - Coarsened grains[2]
  - Coarsening of γ’ precipitates to an average diameter of ~65nm[2]
  - Precipitation and coarsening of $M_{23}C_6$ carbides along grain boundaries[3]
- Need to confirm size of γ’ precipitates in both the base material and fusion zone, as well as for any γ’-denuded regions
  - Surface dissolution of γ’?
  - Grain boundary PFZ’s?





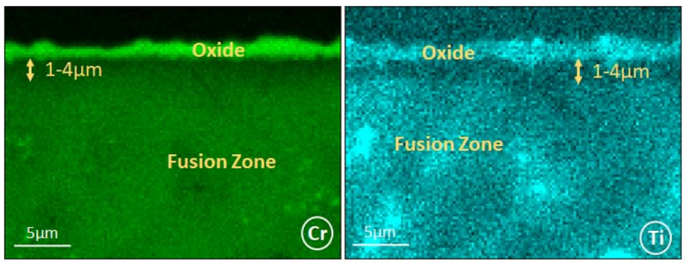


TEM images of 740H after standard post-annealing aging and then aged in 750°C for (a) 300h, (b) 1000h, (c) 3000h, (d) 5000h, (e) 10000h[1]

[1] Yan, G. U. O., et al. "Microstructure and phase precipitate behavior of Inconel 740H during aging." *Transactions of Nonferrous Metals Society of China* 26.6 (2016): 1598-1606
[2] Yan, Chong, et al. "Microstructure evolution and mechanical properties of Inconel 740H during aging at 750 C." *Materials Science and Engineering: A* 589 (2014): 153-164.
[3] DuPont, John N. "Welding of Nickel-based alloys for energy applications." *Welding Journal* 93 (2013): 31-45.

740H – Cross-section Analysis

- Surface Analysis: PFZ with a depth of 1-4µm from oxide. PFZ a result of Cr and Ti-depletion.

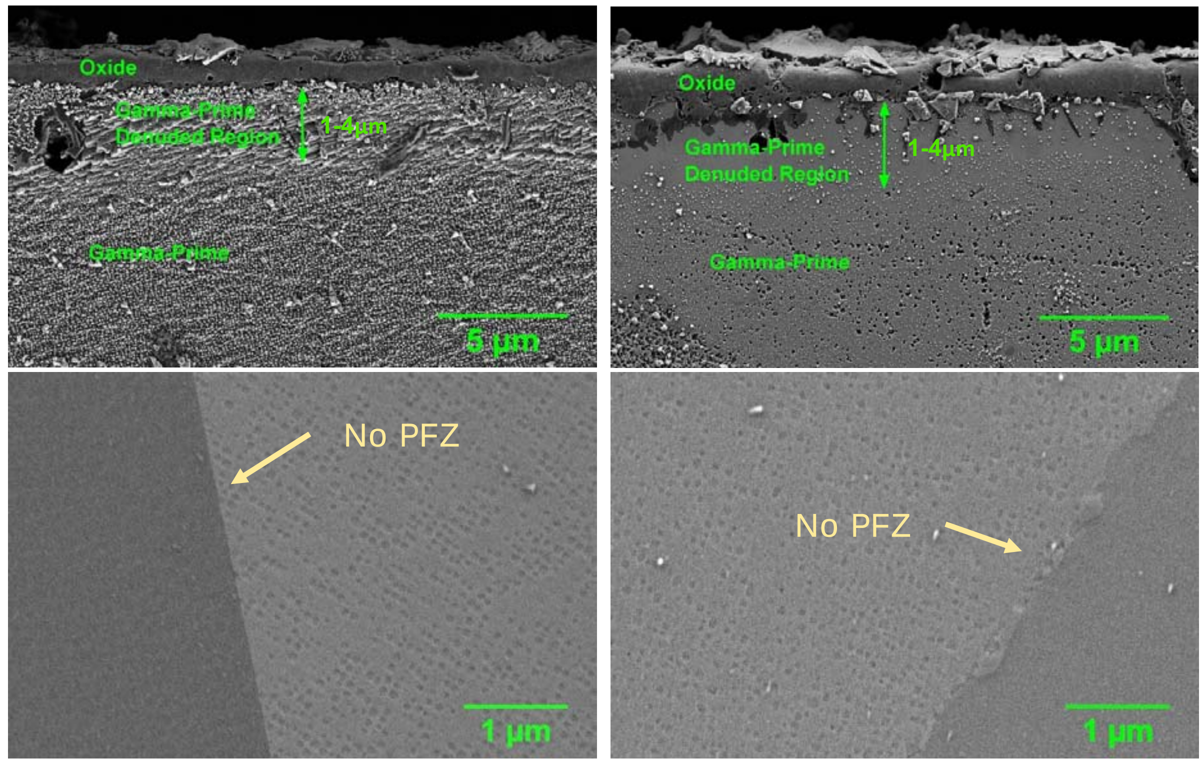


- Bulk Analysis: **no PFZ observed** (using SEM) despite presence of carbides
    - Base Material: continual Cr-enrichment along grain boundaries (assumed to be carbides)
    - Fusion Zone: intermittent Cr-enrichment along grain boundaries

Base Material | Fusion Zone

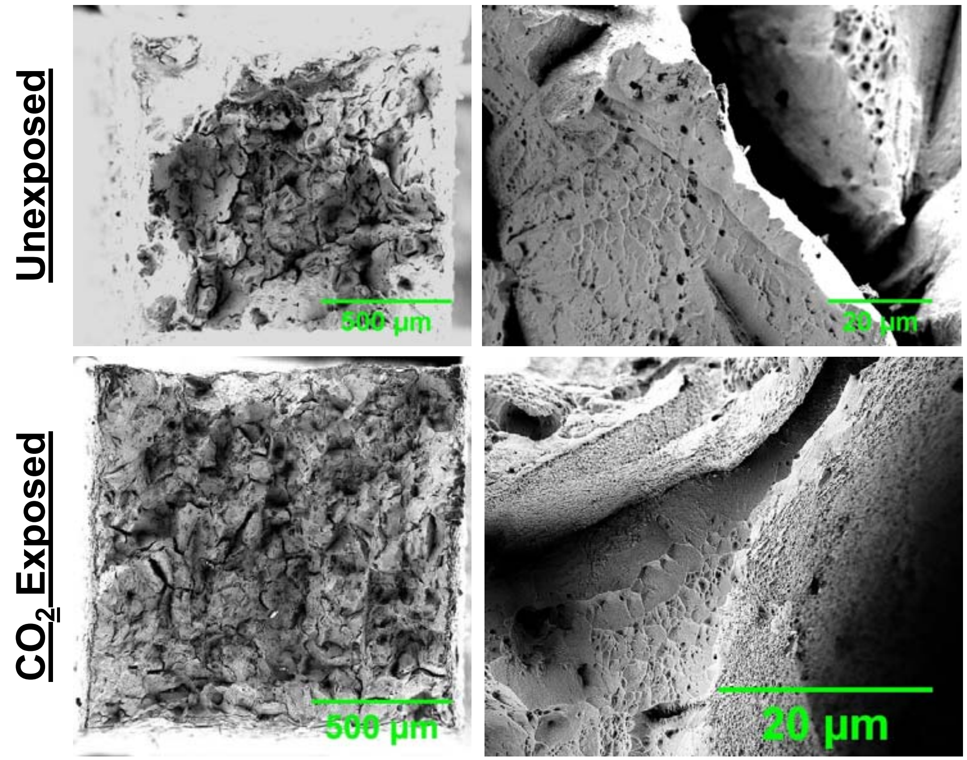
Surface

Bulk



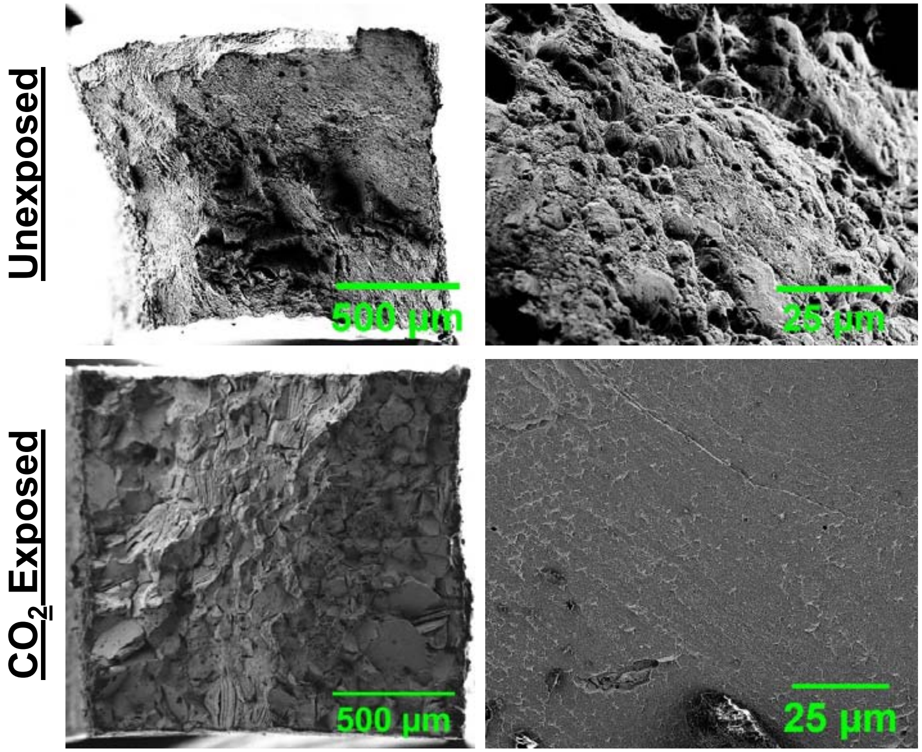
740H Base Material – Post-Fracture Analysis

- Unexposed:
    - Intergranular fracture combined with micro-void coalescence (MVC).
    - Carbides and γ’ precipitated during standard heat treatment, but still relatively small – results in larger dimples indicating better ductility.
- Exposed:
    - Intergranular fracture combined with MVC – decrease in dimple size indicating slight embrittlement.
    - Carbides and γ’ precipitates coarsened during exposure from prolonged diffusion of alloying elements.



740H Weld – Post-Fracture Analysis

- Unexposed:
  - Intergranular fracture combined with MVC.
  - Carbides and γ’ precipitated during post-weld heat treatment, but still relatively small – dimples of size similar to that of base material.
- Exposed:
  - Intergranular fracture with combination of micron-scale cleavage & grain boundary cracking (revealing dendritic structure – see right).
  - Carbides and γ’ precipitates coarsened during exposure and effect is enhanced by larger grains.



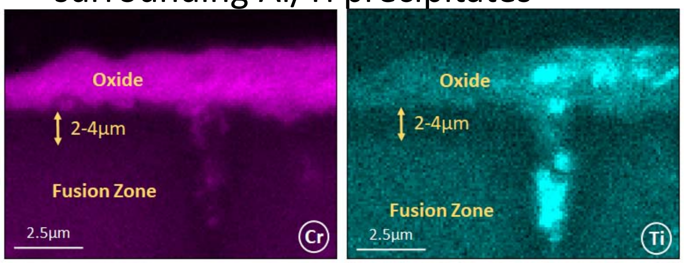
Tensile Results – 282

- Base Material Samples:
  - UTS remained relatively constant with a slight increase in YS. 50%, 67% decrease in elongation in CO_2/Ar.
  - Properties largely unchanged other than a drop in ductility.
- Welded Samples:
  - Significant increase in both UTS and YS, while elongation was unaffected by the exposure.
  - Unexposed results were on the lower end of acceptable for Haynes, so perhaps aging effects are present, just not (yet) the dominant contribution to fracture.

	UTS (MPa)	0.2% YS (MPa)	Elongation (%)	Failure Location
282: Unexposed	**1,105 ± 15**	**728 ± 7**	**39.9 ± 0.4**	**N/A**
282: 750°C CO_2	1,105 ± 9	741 ± 1	20.5 ± 1.0	N/A
Welded 282: Unexposed	**867** ± 25	**637** ± 11	**10.1** ± 1.0	**Fusion Zone**
Welded 282: 750°C CO_2	966 ± 5	684 ± 2	9.6 ± 0.5	Fusion Zone

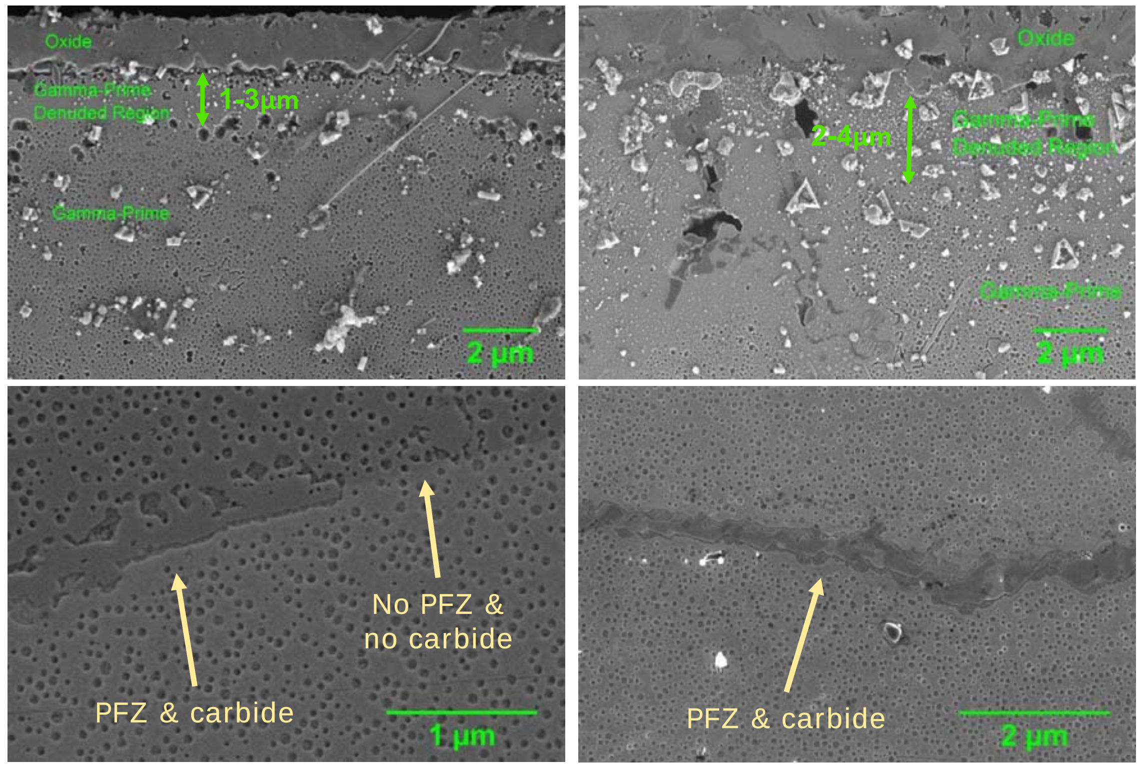
282 – Cross-section Analysis

- <u>Surface Analysis</u>: PFZ with a depth of 1-3µm (base) or 2-4µm (fusion)
  - PFZ continued deeper into material surrounding Al/Ti precipitates



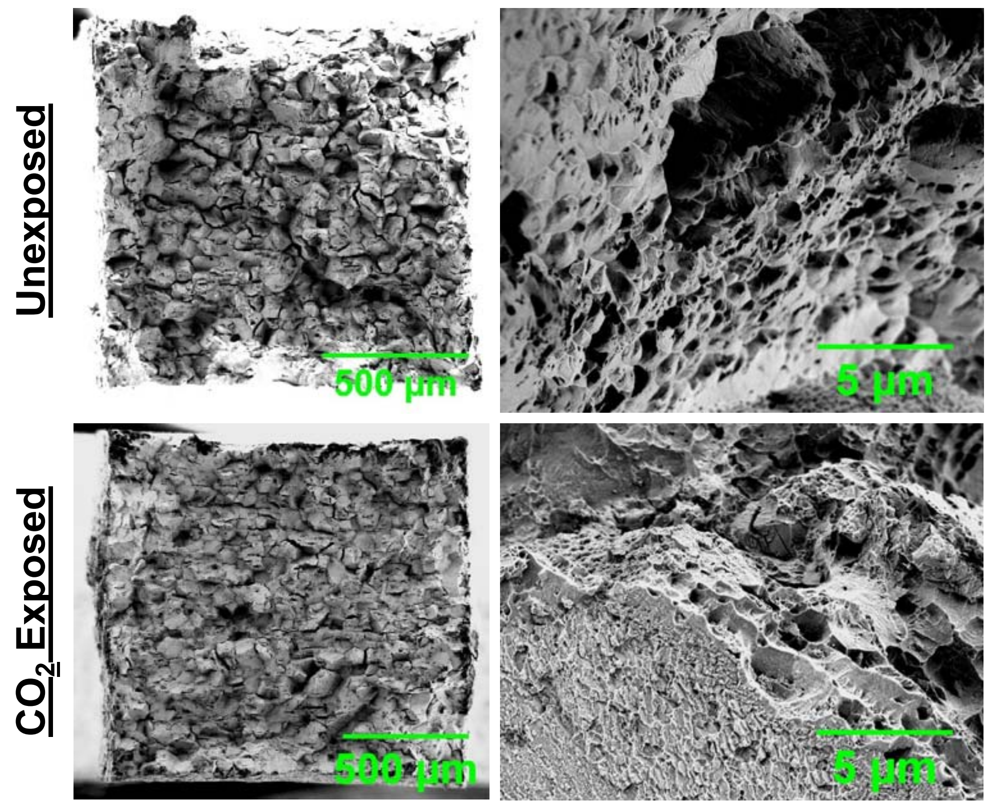
- <u>Bulk Analysis</u>: **100nm thick PFZ observed** corresponding to grain boundary carbide presence:
  - <u>Base Material</u>: intermittent carbides along grain boundaries
  - <u>Fusion Zone</u>: continual grain boundary carbides and PFZ

<u>Base Material</u> **<u>Fusion Zone</u>**



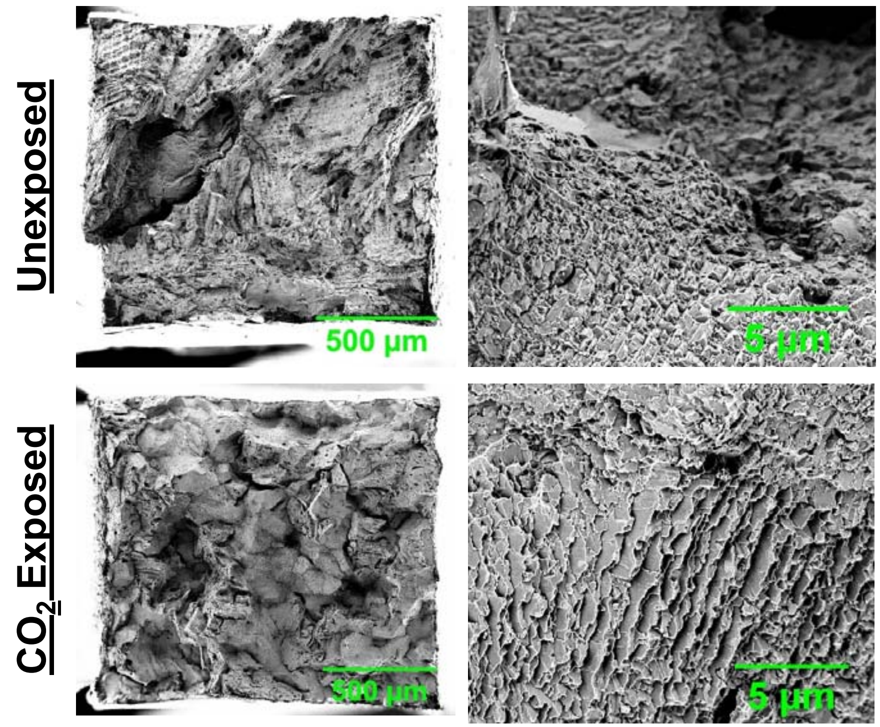
282 Base Material – Post-Fracture Analysis

- Unexposed:
    - Intergranular fracture with MVC – micron-scale dimples.
    - Carbides and γ’ precipitated during standard heat treatment, but still relatively small – results in larger dimples indicating better ductility.
- Exposed:
    - Intergranular fracture with a combination of MVC (similar-size dimples) and sub-micron-scale cleavage.
    - Carbides and γ’ precipitates coarsened during exposure from prolonged diffusion of alloying elements.



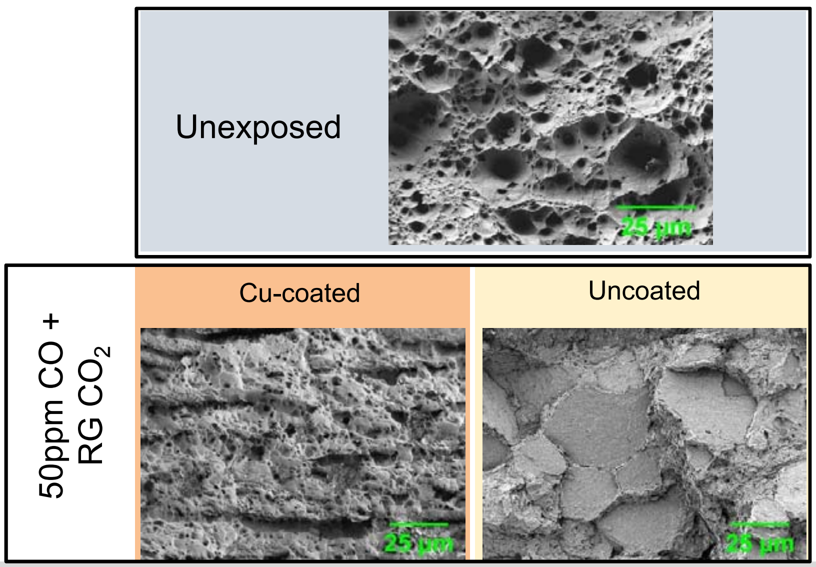
282 Weld – Post-Fracture Analysis

- Unexposed:
  - Intergranular fracture with combination of MVC – micron-scale dimples – and sub-micron-scale cleavage.
  - Carbides and γ’ precipitated during post-weld heat treatment, but still relatively small – dimples of size similar to that of base material.
- Exposed:
  - Intergranular fracture with micron-scale cleavage – facets noticeably increased in size.
  - Lack of MVC (compared to base material) indicates the fracture mechanism is altered due to the grain boundary system of continuous, coarsened carbides and associated PFZ.



Other Tensile Results – Cu-Coating with Impurities

- Calculations show that at least ~10ppm CO in pure CO_2 lowers the pO_2 to a point that renders Cu inert at 750°C
- Tensile results showed exposure at 750°C for 1,000 hours had a strong detrimental effect on mechanical properties after exposure at 750°C every case except Cu-coated, 50ppm CO.
- Cu-coated 316L exposed to 50ppm CO in RG CO_2 increases UTS, slightly decreases YS and has the effect on elongation:
  - Base Material: **slight decrease**
  - Welded: **no change**
- 316L exposed bare or Cu-coated without CO-doping exhibited severely decreased elongation and deteriorated UTS and YS.



	UTS (MPa)	0.2% YS (MPa)	Elongation (%)
316L: Unexposed	**605 ± 1**	**297 ± 3**	**81.2 ± 1.2**
316L: 750°C CO_2	598 ± 15	220 ± 1	26.8 ± 4.7
316L: 750°C CO_2 + 50ppm CO	498 ± 14	291 ± 34	7.5 ± 1.7
316L Cu: 750°C CO_2	590 ± 18	233 ± 12	29.1 ± 11.0
316L Cu: 750°C CO_2 + 50ppm CO	641 ± 4	263 ± 4	68.7 ± 4.2
316L-316L: Unexposed	**541 ± 5**	**278 ± 2**	**41.2 ± 0.8**
316L-316L: 750°C CO_2	586 ± 11	240 ± 13	19.2 ± 0.4
316L-316L: 750°C CO_2 + 50ppm CO	538 ± 2	252 ± 1	14.3 ± 0.5
316L-316L Cu: 750°C CO_2	581 ± 21	237 ± 6	21.9 ± 5.4
316L-316L Cu: 750°C CO_2 + 50ppm CO	651 ± 15	256 ± 4.9	40.1 ± 4.3

Overview

- Summary:
  - Base material and transverse weld samples of 282 and 740H were exposed in RG CO_2 for 1,000 hours at 750°C. Tensile testing was performed at RT.
  - 740H: base material exhibited only a moderate decrease in mechanical properties; welded samples experienced a large decline in strength and elongation. No PFZ's were observed in either base material or fusion zone.
  - 282: base material exhibited a decrease in mechanical properties while welded samples showed an increase in strength with no loss in elongation. PFZ's were observed intermittently in base material and continuously in the fusion zone.
  - Cu-coating has been shown to be protective against carburization of 316SS in CO-doped CO_2, maintaining high elongation in both base material and welds after 1,000 hours at 750°C.
- Future Work:
  - Currently investigating performance of 316SS and grade 92 F-M steel (and their welds) in CO_2 at lower temperatures, as well as dissimilar welds between them and superalloys 740H and 282.
  - Beginning the evaluation of diffusion bonds after exposure in CO_2.

Thank you

Questions?

Backup Slides

Tensile Results – 740H (additional: heat treatment)

- Base Material Samples:
  - UTS (tensile strength) and YS (yield strength) increased, with only a moderate drop in elongation
  - Effect is consistent with continued aging of material – coarsening of γ' precipitates & intergranular carbides
- Welded Samples:
  - Significant drop in UTS and YS, with a large drop in elongation.
  - Effect is magnified by CO_2 exposure

	UTS (MPa)	0.2% YS (MPa)	Elongation (%)	Failure Location
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740H: 750°C HT	1,119 ± 22	673 ± 4	19 ± 2.0	N/A
Welded 740H: Unexposed	**1,068 ± 22**	**759 ± 16**	**26 ± 2.6**	**Fusion Zone**
Welded 740H: 750°C CO_2	940 ± 10	638 ± 7	12 ± 2.0	Fusion Zone
Welded 740H: 750°C HT	1,039 ± 4	710 ± 4	16 ± 1.0	Fusion Zone

Tensile Results – 282 (additional: heat treatment)

- Base Material Samples:
  - UTS remained relatively constant with a slight increase in YS. 50%, 67% decrease in elongation in CO_2/Ar.
  - Properties largely unchanged other than a drop in ductility.
- Welded Samples:
  - Significant increase in both UTS and YS, while elongation was unaffected by the exposure.
  - Unexposed results were on the lower end of acceptable for Haynes, so perhaps aging effects are present, just not (yet) the dominant contribution to fracture.
  - HT not performed because samples not procured at time of exposure.

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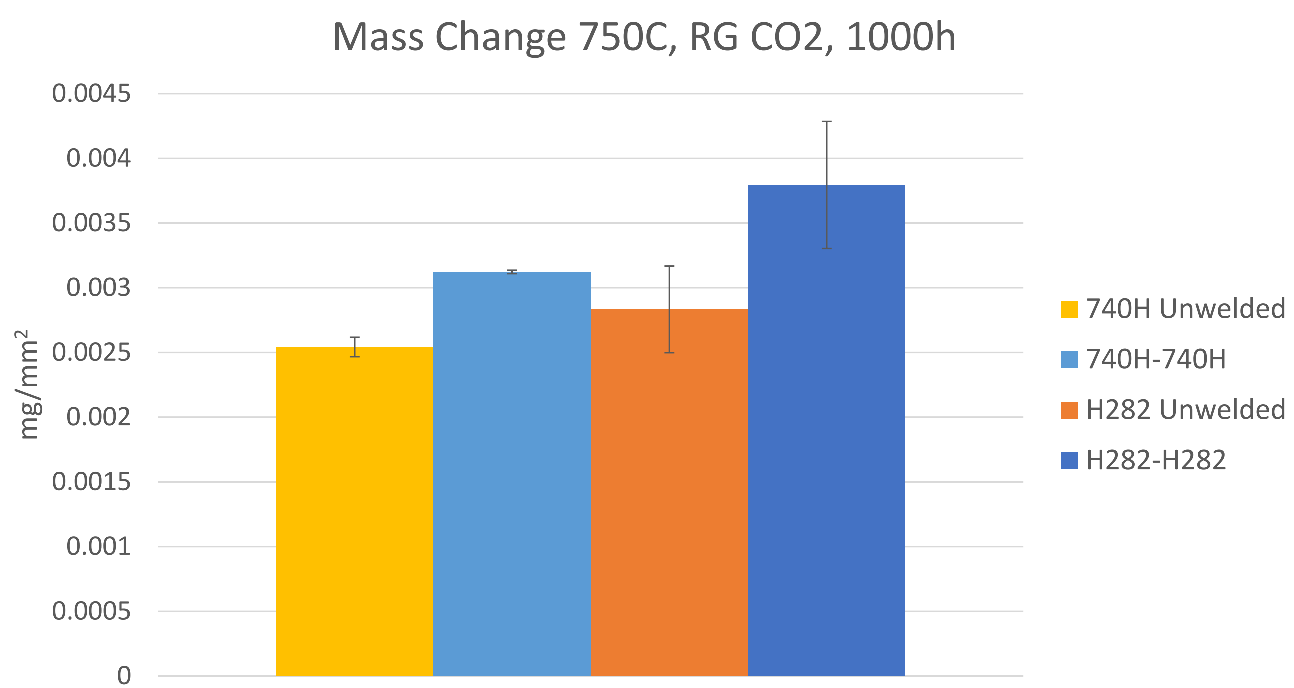
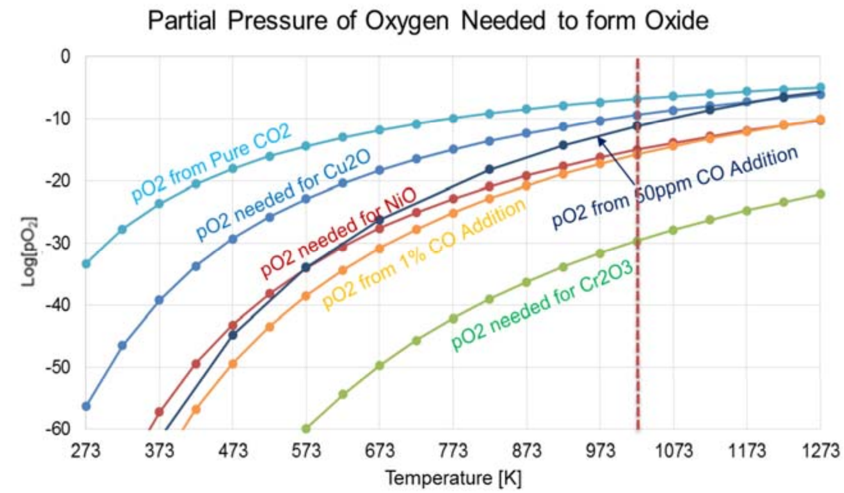
282: Unexposed	**1,105 ± 15**	**728 ± 7**	**39.9 ± 0.4**	**N/A**
282: 750°C CO_2	1,105 ± 9	741 ± 1	20.5 ± 1.0	N/A
282: 750°C HT	1,052 ± 84	746 ± 18	13.0 ± 3.1	N/A
Welded 282: Unexposed	**867** ± 25	**637** ± 11	**10.1** ± 1.0	**Fusion Zone**
Welded 282: 750°C CO_2	966 ± 5	684 ± 2	9.6 ± 0.5	Fusion Zone

Mass Change – 750C, 20MPa, 1,000 hours



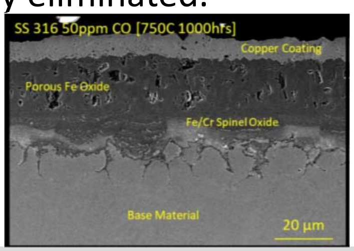
Other Tensile Results – Cu-Coating with Impurities

- Calculations show that at least ~10ppm CO in pure CO_2 lowers the pO_2 to a point that renders Cu inert at 750°C
- Coefficients of thermal expansion is very similar to that of 316SS (Cu – ~18.5μm/m-K; 316 – 17.5μm/m-K)
  - **Coating 316 in Cu is a viable option**
- Experimentally shown that Cu-coating is very protective of 316L and 316L welds in CO_2 doped with 50ppm CO.
  - Acts primarily as a **carbon barrier**, causing bulk 316 to only experience heat treatment during exposure in CO_2 environment.
  - Some oxidation still occurs, but environmental carburization is effectively eliminated.



Source: *Mahaffey, Jacob Thomas, author. Effect Of Partial Pressure of Oxygen and Activity of Carbon on the Corrosion of High Temperature Alloys in Supercritical Carbon Dioxide Environments. [Madison, Wis.] :[University of Wisconsin--Madison], 2017. Print*

No Cu-coating:



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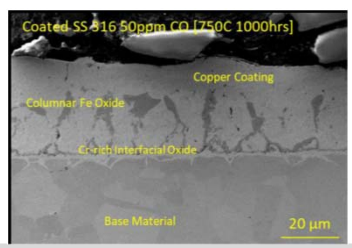

Source: *Schroeder, Anthony, author. Copper as a Corrosion Barrier in Supercritical CO_2 at 750C. [Madison, Wis.] :[University of Wisconsin--Madison], 2017. Print*