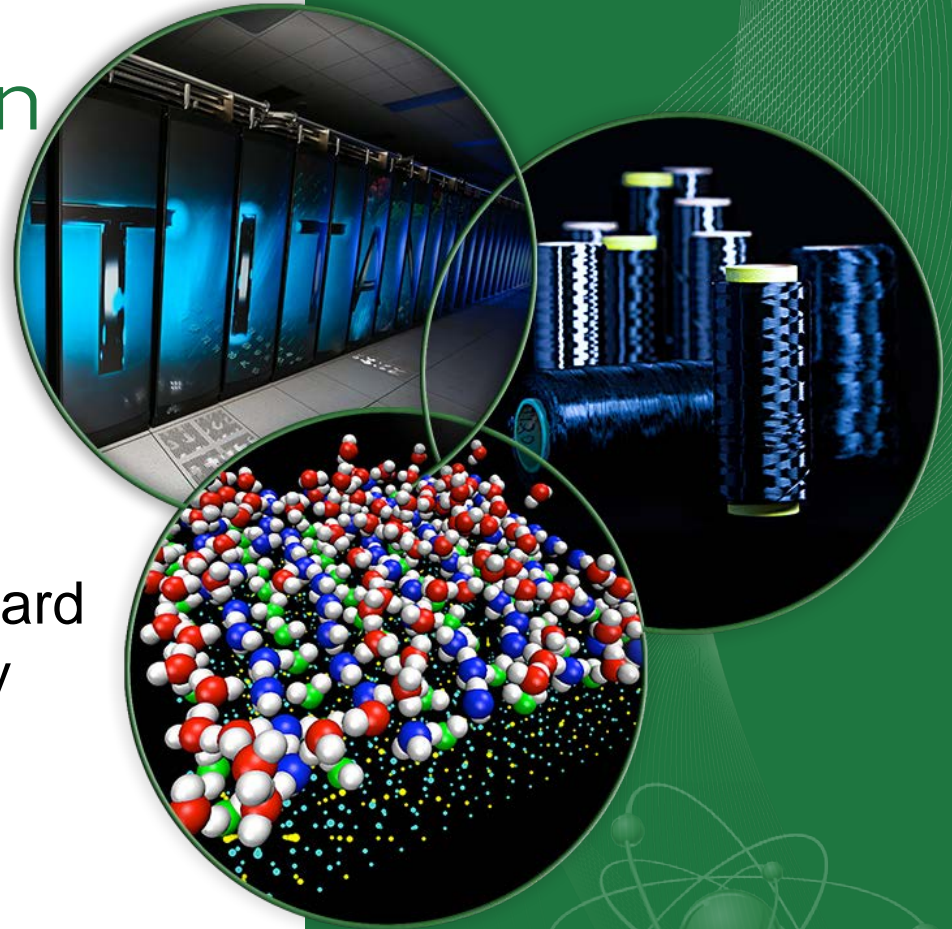


Characterization Of Turbomachinery Materials Exposed In 750°C, 20 MPa sCO₂

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Presentation Outline

- Background and motivation
- Description of ORNL test facilities
- Test plan
- Results of corrosion coupon exposures
 - weight change
 - light microscopy and SEM
- Results of C-ring sample exposures
 - weight change
 - light microscopy, SEM, electron microprobe
- Summary

ORNL Studies Supported Project On Advanced Turbomachinery For Supercritical CO₂ Power Cycles

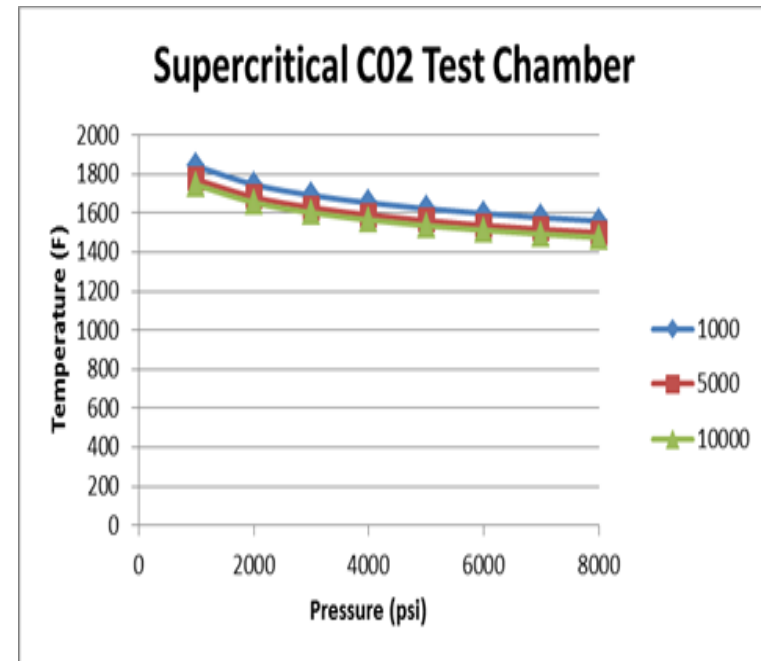
- ORNL is one member of the project team
- Project goal was to define sCO₂ turbomachinery concepts and applicability to indirectly and directly-heated cycles
- One task was to conduct compatibility tests of candidate materials in sCO₂
- ORNL was responsible for conducting exposure tests in sCO₂ and characterizing samples after exposure

Alloys Selected For Testing Represented Several Components In An Advanced Turbine

| Material | Application | Flat Coupons | Stressed C-Rings |
|------------------|-------------|--------------|------------------|
| HK40 | Housing | 3 | 3 |
| HK50 | Housing | 3 | |
| CAFA7 | Housing | | 3 |
| DAFA30 | Housing | | 3 |
| Alloy 282 (cast) | Housing | | 3 |
| Waspaloy | Disk | 3 | 3 |
| Udimet 720 | Disk | 3 | |
| Alloy 718 | Disk | 3 | 3 |
| A-286 | Disk | 3 | |
| René 41 | Disk | 3 | |
| CMSX-8 | Blade | | 3 |
| PWA 1483 | Blade | | 3 |
| René N4 | Blade | | 3 |
| CMSX-4 | Blade | | 3 |

ORNL Autoclaves Used For This Project Were Designed To Achieve Extreme Test Conditions

- Autoclaves are fabricated from Haynes 282 (UNS N07208) alloy which was developed for hot-gas-path components of aircraft engines and land-based gas turbines
- Alloy is a gamma-prime strengthened superalloy with excellent creep strength in the 649-927°C (1200-1700°F) range
- As shown on the chart, the system is capable of pressures as high as 55.2 MPa (8000 psi) at a temperature of about 816°C (1500°F) or 20.0 MPa (2950 psi) at 871°C (1600°F)



ORNL Autoclaves Are Designed For Safe And Versatile Operations

- Autoclave positioned vertically in a 3 zone furnace
- 99.995% purity $s\text{CO}_2$ is fed to the bottom of the autoclave and flows up over the samples
- Fluid is provided by a dual piston pump at a rate up to 24 ml/min; but generally 1-2 ml/min
- Back pressure control valve is used to maintain system pressure
- Rupture disc and a pressure relief valve prevent over pressurization
- Thermowell in center of autoclave permits measurement of sample temperature



ORNL Conducted Three sCO₂ Tests Then Examined Samples After Exposure

- Tests were conducted for 500 hours at 750°C and 20 MPa (2900 psi)
- First test exposed 21 corrosion coupons
- Second test exposed 16 C-rings
- Third test exposed an additional 14 C-rings
- Post exposure characterization included weight change determination as well as light microscopy, scanning electron microscopy and microprobe examinations

Corrosion Coupon Studies

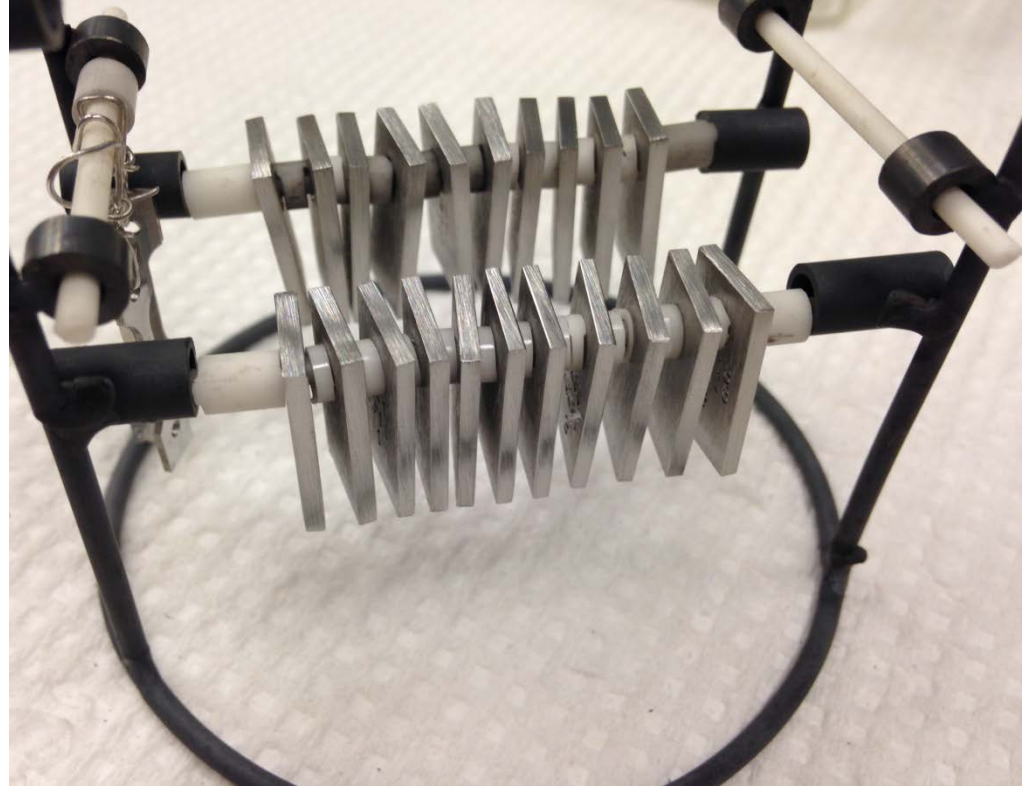
Seven Alloys (3 samples of each) Were Exposed As Corrosion Coupons

| Alloy | Application | Ni | Cr | Co | Mo | Fe | Al | Ti | C | Other |
|------------|-------------|-----|-----|-----|-----|-----|-----|-----|------|--------|
| | | wt% | wt% | wt% | wt% | wt% | wt% | wt% | wt% | wt% |
| Alloy 718 | Disk | 52 | 19 | | 3.0 | 19 | 0.5 | 0.9 | 0.04 | Nb 5.1 |
| HK50 | Housing | 20 | 26 | | 0.5 | Bal | | | 0.5 | |
| Waspaloy | Disk | 58 | 19 | 14 | 4.3 | 2 | 1.5 | 3.0 | 0.08 | |
| HK40 | Housing | 20 | 26 | | 0.5 | Bal | | | 0.4 | |
| Udimet 720 | Disk | 55 | 18 | 15 | 3.0 | | 2.5 | 5.0 | 0.03 | W 1.3 |
| René 41 | Disk | 52 | 19 | 11 | 10 | 5 | 1.5 | 3.1 | 0.09 | |
| A-286 | Disk | 26 | 15 | | 1.3 | 54 | 0.2 | 2.0 | 0.05 | |

Three samples of each alloy were exposed in the first sCO₂ test

The 21 Corrosion Coupons Were Supported On a Rack Near The Bottom Of The Autoclave

- Samples are supported on alumina bars which are held in an alloy 282 metal rack with alumina insulators separating the samples
- Fluid is pumped into the autoclave through a tube that extends to the bottom of the autoclave
- A thermowell in the center of the autoclave permits temperature measurement of the fluid very near the samples

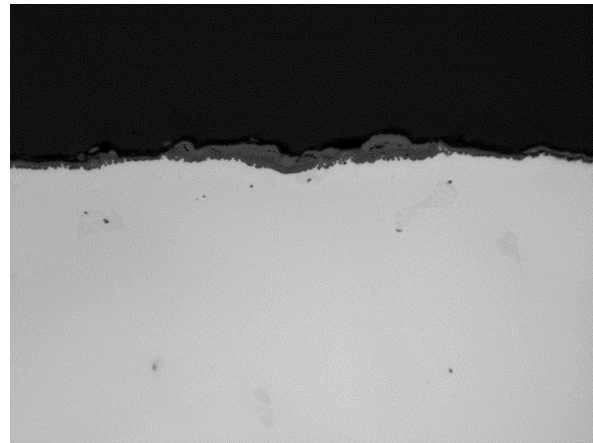


After 500 h Exposure, Weight Change Varied Considerably Between The Alloys

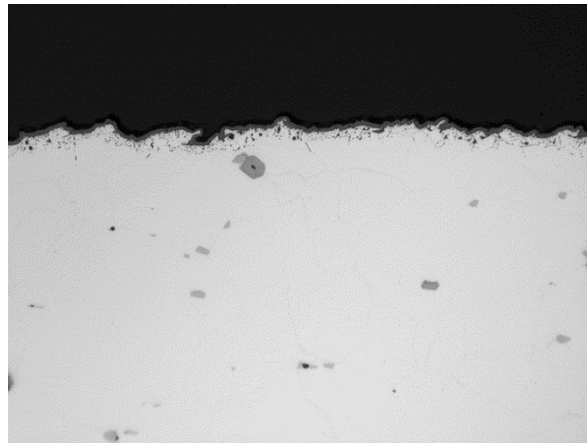
| | Sample #1 | Sample #2 | Sample #3 | Average |
|-------------|--------------------|--------------------|--------------------|--------------------|
| | mg/cm ² | mg/cm ² | mg/cm ² | mg/cm ² |
| A-286 | 17.27 | 17.70 | 17.88 | 17.62 |
| René 41 | 0.55 | 0.54 | 0.56 | 0.55 |
| Udimet 720 | 0.53 | 0.55 | 0.55 | 0.54 |
| HK40 (cast) | 0.49 | 0.49 | 0.56 | 0.51 |
| Waspaloy | 0.40 | 0.34 | 0.37 | 0.37 |
| HK50 (cast) | 0.41 | 0.32 | 0.28 | 0.34 |
| Alloy 718 | 0.28 | 0.27 | 0.34 | 0.30 |

On the basis of these results, the A286 alloy was dropped and three of the others were selected for testing as C-rings

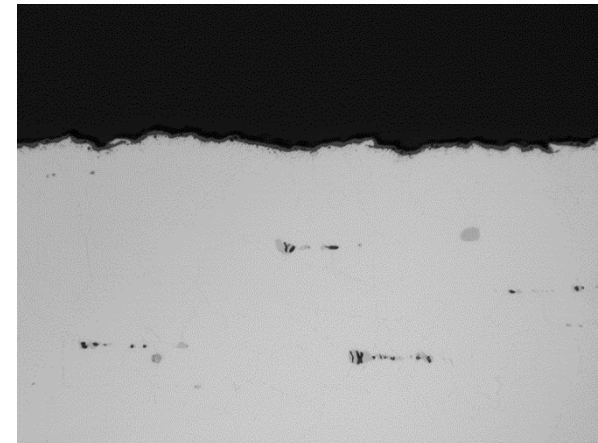
Micrographs Of Exposed Samples Showed Thin, Continuous Oxide Versus Control



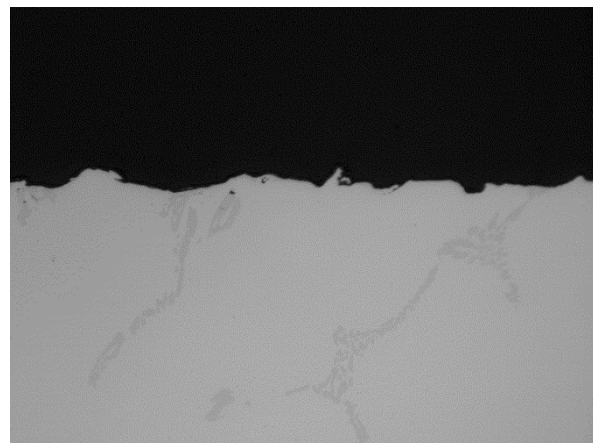
15-0865-01 HK40-02 exp 500X 10µm as polished



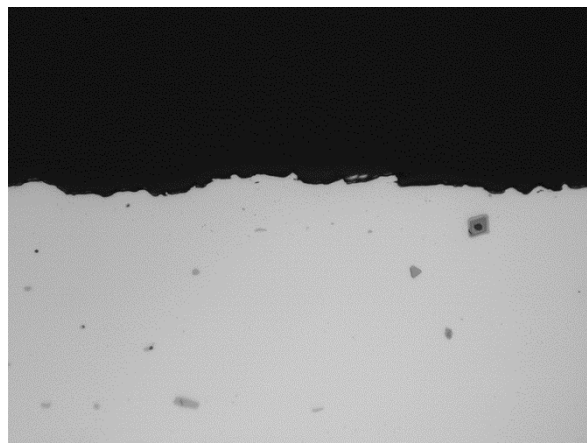
15-0867-01 WASP-01 exp 500X 10µm as polished



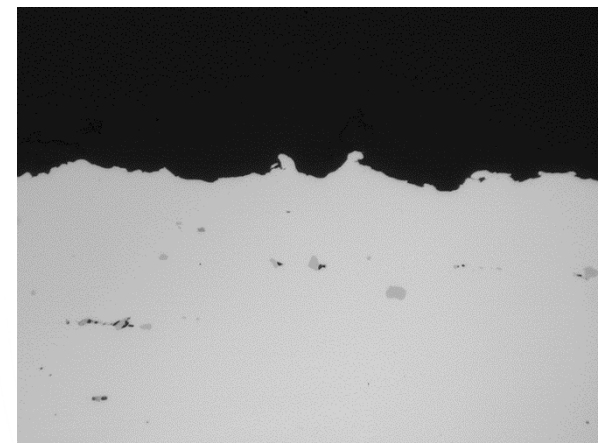
15-0869-01 I718-01 exp 500X 10µm as polished



15-0866-01 HK40-04 control 500X 10µm as polished



15-0868-01 WASP-04 control 500X 10µm as polished



15-0870-01 I718-01 control 500X 10µm as polished

Exposed Waspaloy had shallow subsurface attack

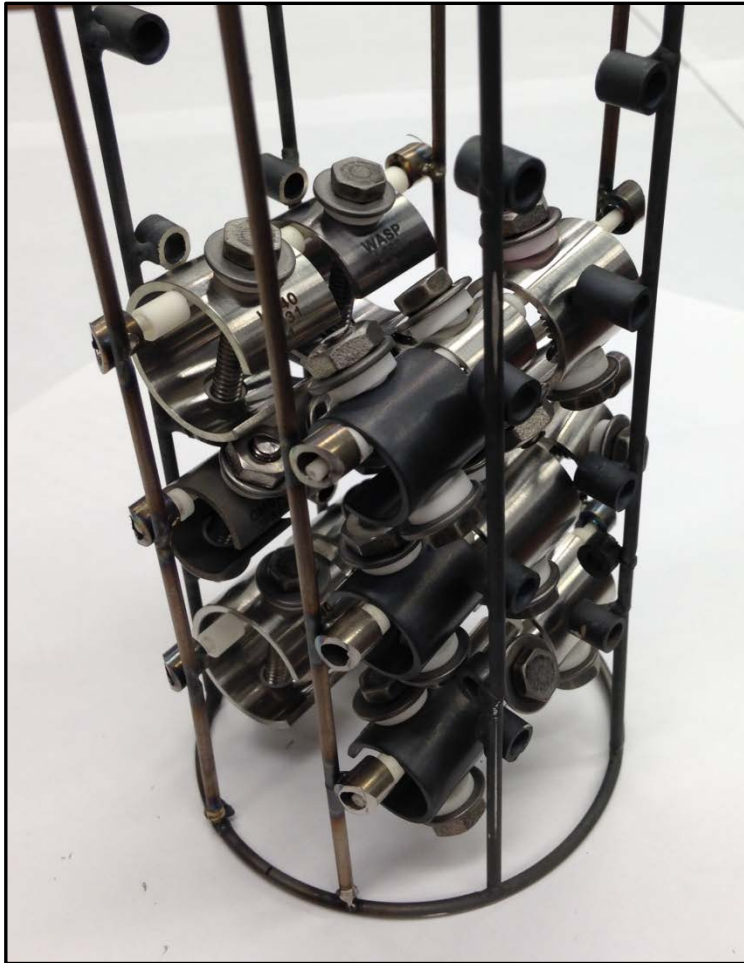
C-Ring Sample Studies

To Further Evaluate The Effect Of sCO₂ On Alloys, Stressed Samples Were Tested

| Alloy | Application | Ni | Cr | Co | Mo | Fe | Al | Ti | C | Other |
|--------------|-------------|-----|------|-----|-----|-----|-----|-----|------|-----------------------|
| | | wt% | wt% | wt% | wt% | wt% | wt% | wt% | wt% | wt% |
| PWA1483 | Blade | Bal | 12.2 | 9.0 | 1.9 | | 3.6 | 4.1 | | W 3.8, Ta 5.0 |
| CAFA7 | Housing | 25 | 15 | | 2.0 | Bal | 3.5 | | 0.4 | W 1.0, Nb 1.0 |
| René N4 | Blade | Bal | 9.0 | 8.0 | 2.0 | | 3.7 | 4.2 | | W 6.0, Ta 4.0, Nb 0.5 |
| Waspaloy | Disk | 58 | 19 | 14 | 4.3 | 2 | 1.5 | 3.0 | 0.08 | |
| HK40 | Housing | 20 | 26 | | 0.5 | Bal | | | 0.5 | |
| CMSX-4 | Blade | Bal | 6.5 | 10 | 0.6 | | 5.6 | 1.0 | | W 6.0, Ta 6.0, Re 3.0 |
| H 282 (cast) | Housing | 57 | 20 | 10 | 8.5 | 1.5 | 1.5 | 2.1 | 0.06 | |
| DAFA30 | Housing | 33 | 14 | | | Bal | 2.7 | 1.6 | 0.1 | Nb 2.8 |
| CMSX-8 | Blade | Bal | 5.4 | 10 | 0.6 | | 5.7 | 0.7 | | W 8.0, Ta 8.0, Re 1.5 |
| Alloy 718 | Disk | 52 | 19 | | 3.0 | 19 | 0.5 | 0.9 | 0.04 | Nb 5.1 |

C-ring samples of these alloys were prepared and stressed to 75%, 85% and 95% of the 500 hour rupture stress

C-Ring Samples Were Also Supported On Alumina Rods On The Metal Rack



- Second sCO₂ test with 16 of the C-ring samples before exposure

First Set Of C-Rings Showed Varying Degrees Of Attack

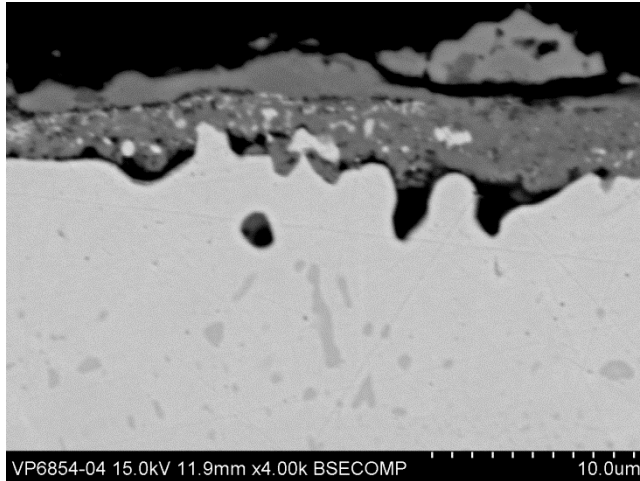


After 500 h, Measured Weight Changes Showed Considerable Differences Between Alloys

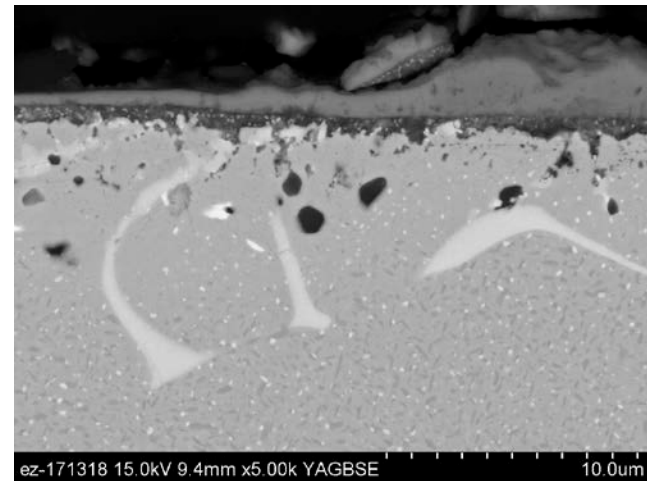
| Alloy | Application | Sample 1 | Sample 2 | Sample 3 | Average |
|-----------|-------------|--------------------|--------------------|--------------------|--------------------|
| | | mg/cm ² | mg/cm ² | mg/cm ² | mg/cm ² |
| DAFA30 | Housing | 0.94 | 1.11 | 1.09 | 1.05 |
| HK40 | Housing | 0.82 | 0.71 | 0.69 | 0.74 |
| CAFA7 | Housing | 0.52 | 0.56 | 0.58 | 0.55 |
| PWA1483 | Blade | 0.43 | 0.50 | 0.52 | 0.48 |
| Waspaloy | Disk | 0.27 | 0.29 | 0.24 | 0.27 |
| Cast H282 | Housing | 0.29 | 0.22 | 0.26 | 0.26 |
| Alloy 718 | Disk | 0.20 | 0.24 | 0.25 | 0.23 |
| CMSX-4 | Blade | 0.20 | 0.19 | 0.21 | 0.20 |
| René N4 | Blade | 0.10 | 0.19 | 0.11 | 0.13 |
| CMSX-8 | Blade | 0.11 | 0.13 | 0.04 | 0.09 |

SEM BSE Micrographs Of Housing Alloys

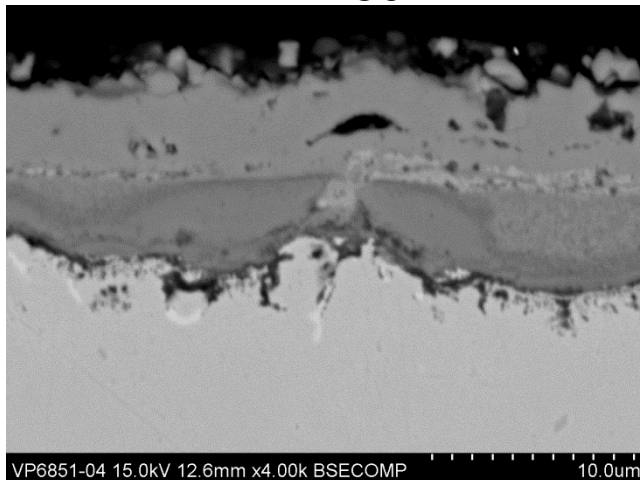
HK40



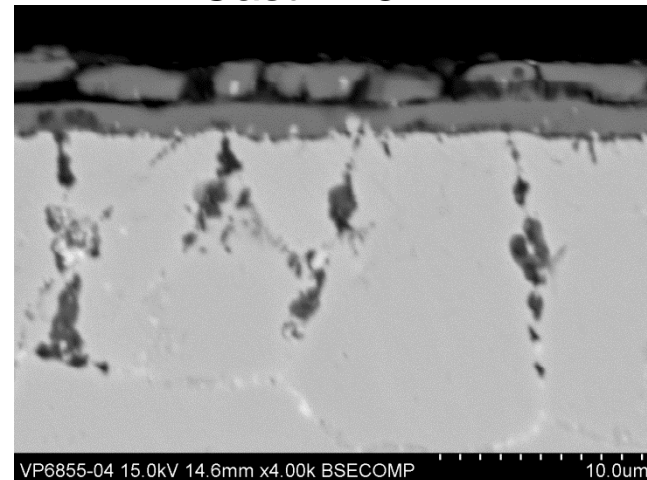
CAFA7



DAFA30



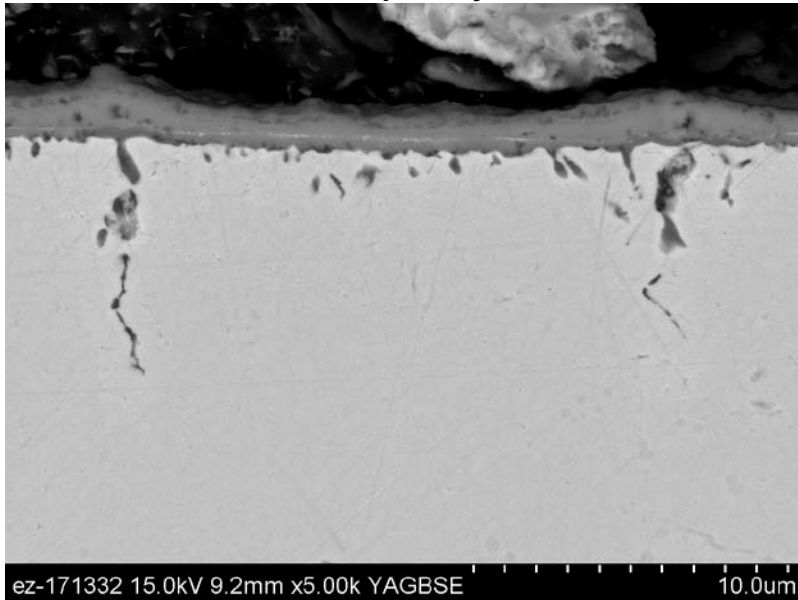
Cast H282



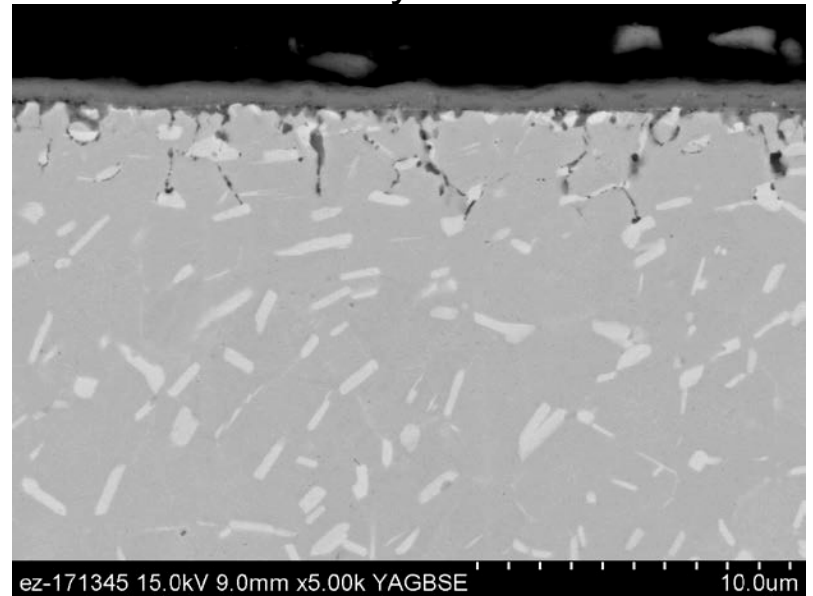
Find continuous oxide and subsurface attack

SEM BSE Micrographs Of Disk Alloys

Waspaloy



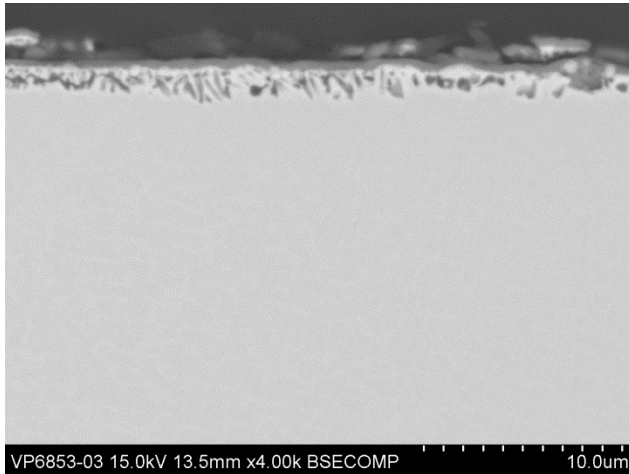
Alloy 718



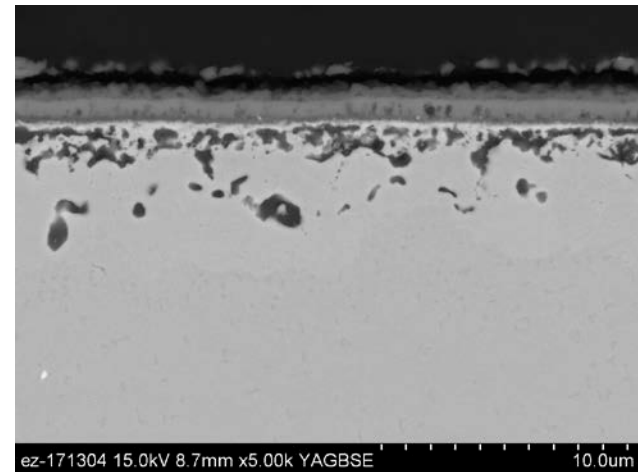
Find continuous oxide and subsurface attack

SEM BSE Micrographs Of Blade Alloys

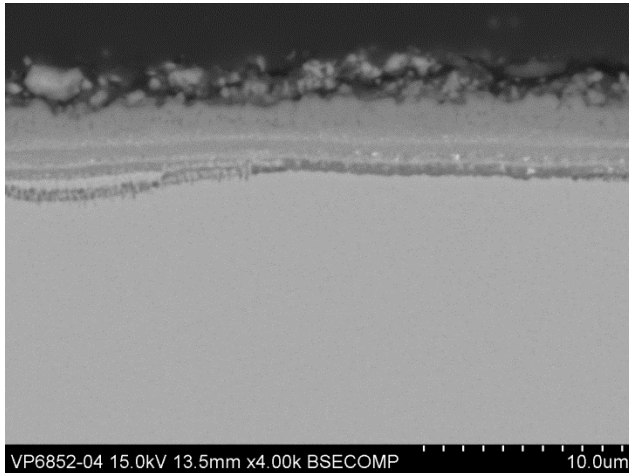
CMSX-8



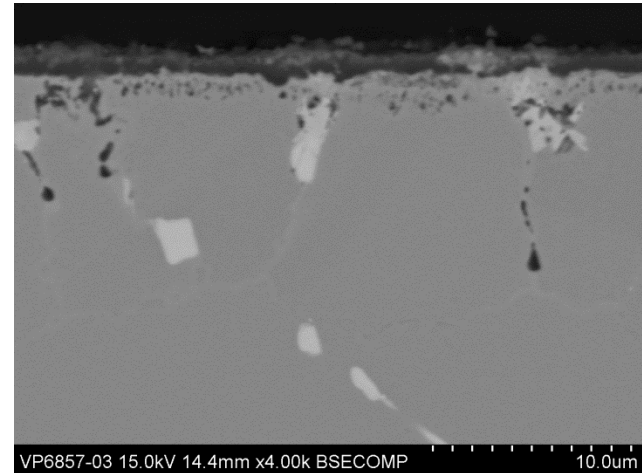
PWA 1483



CMSX-4



René N4



Find continuous oxide and subsurface attack

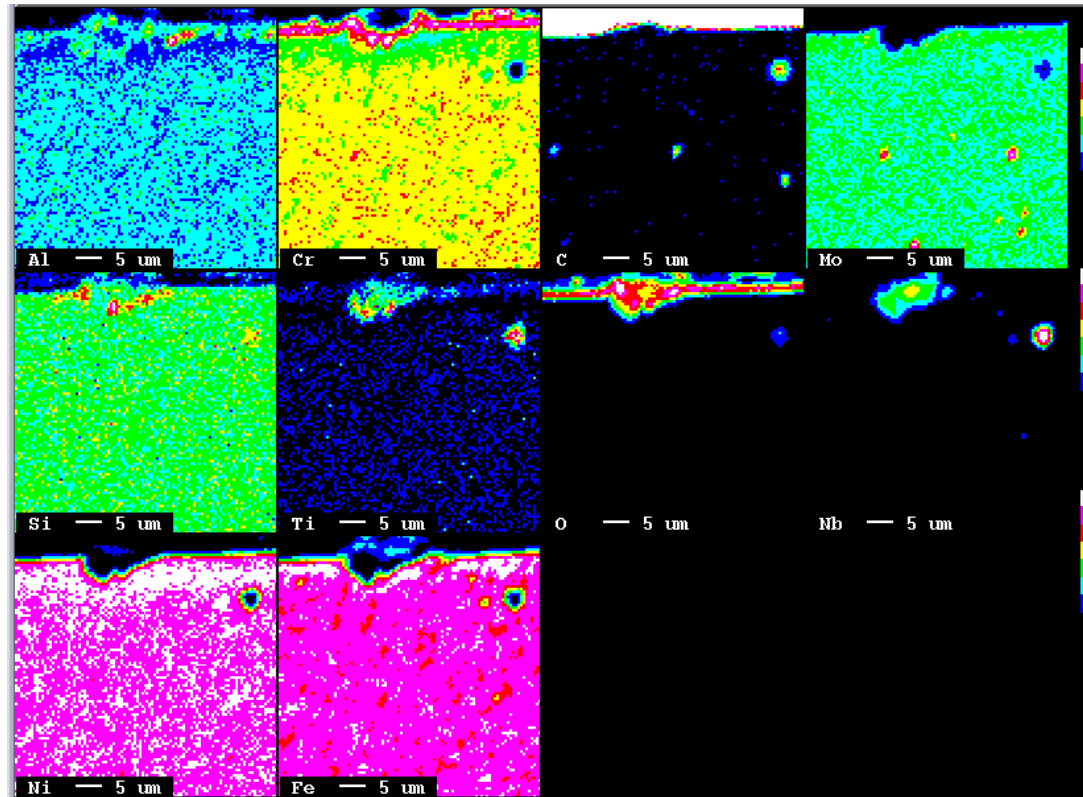
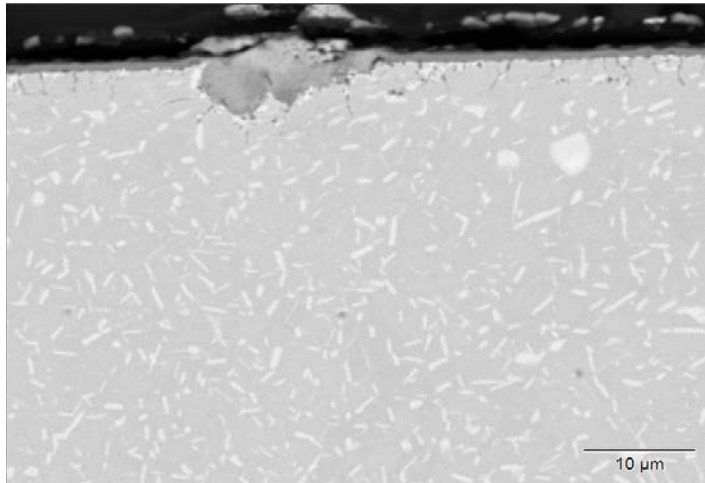
Observations From SEM Examination Of Cross Sections Of C-Ring Samples

- Alloys proposed for the housing showed the greatest weight change while the blade alloys showed the least
- Cross sections of all alloys showed a continuous surface oxide layer
- Nearly all alloys showed subsurface attack
- No evidence of significant cracking was found
- Exposures were only for 500 h, so only preliminary conclusions should be drawn

Four Samples Were Examined With Electron Probe Micro Analyzer

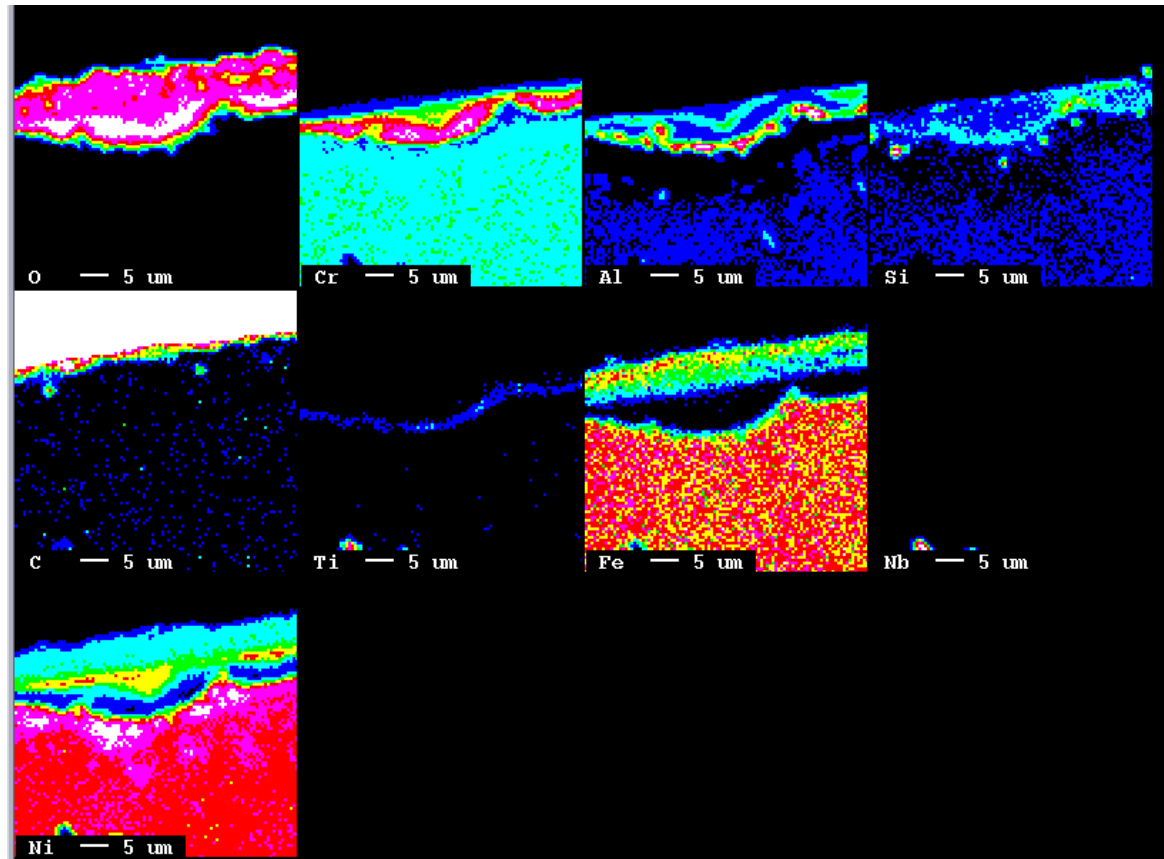
- Provided information on composition of surface layers
- Identified composition of precipitates or second phase particles
- Defined the variation in carbon concentration as a function of distance from surface
 - all four samples showed higher carbon concentration at the surface than in the bulk
 - further studies are in progress

Microprobe Examinations Conducted On Four Samples – Alloy 718 Shown



Microprobe exam of Alloy 718 C-ring cross-section shows Cr rich surface oxide with some near surface Cr depletion and a Ti-Nb rich carbide precipitate

Microprobe Results For DAFA30 Show Several Oxide Layers



The outer oxide is primarily an iron oxide with some nickel content. The inner oxide is primarily chromium oxide with some Al, Si and Ti content.

Summary

- Corrosion coupons and C-ring samples of selected alloys were exposed to $s\text{CO}_2$ at 750°C and 20 MPa
- For the limited exposure time, there were significant differences in the weight changes of alloys for each application
- Electron microprobe provides results on the composition of microstructural features
- Electron microprobe examination revealed possible evidence of carburization
- Longer exposure times along with thorough microprobe examinations will be needed to better define the suitability of each alloy

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- Mike Howell – experiment operation
- Tyson Jordan – light metallography
- Tracie Lowe – SEM examination

THANKS FOR YOUR ATTENTION

I'LL TRY TO ANSWER ANY OF YOUR QUESTIONS