INL/MIS-21-65266-Revision-0

# Alloy 709 Advanced Austenitic Stainless Steel

November 2021

Changing the World's Energy Future

Ting-Leung Sham, Michael D McMurtrey

Idaho National Laboratory

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**November 2021**

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**Idaho Falls, Idaho 83415**

**http://www.inl.gov**

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# Alloy 709 Advanced Austenitic Stainless Steel

# History of 20Cr-25Ni Austenitic Alloys

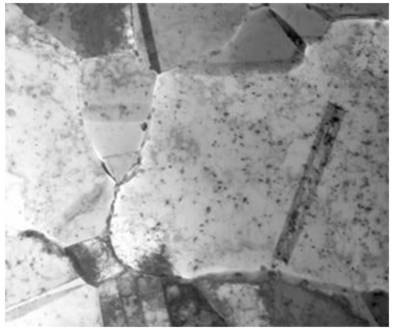
- 20Cr25Ni/Nb stainless steel (1950s)
- Used as fuel cladding in British AGR fleet (since 1962)
- Temperature 550ºC to 850ºC
- Neutron exposure ~ 6 yrs
- 0.38 mm wall thickness, 14.5 mm diameter, 1 m long
- Approximately 90,000 fuel pins per AGR
- 14 AGRs built

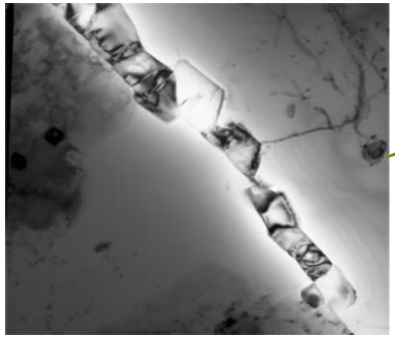
- Nippon Steel NF709 seamless tubing (1980s)
- Superheater and reheater applications in power boilers (’95 to ’05, ~500 metric tons used in Japan power plants)
- Based on modified 20Cr25Ni composition
- UNS No. S31025
- ASTM A213 TP310MoCbN (for seamless tubes)
- ASME SA-213 Code Case 2581 for Section I power boilers

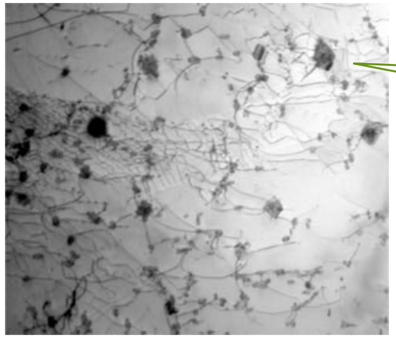
| | C | Mn | Si | P | S | Cr | Ni | Nb | B | Mo | Ti | N |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 20Cr25Ni/Nb | 0.015 | 0.5 | 0.6 | 0.01 | 0.02 | 20 | 25 | 0.6 | 0.0001 | | | <0.004 |
| NF 709 Spec | 0.10 max | 1.50 max | 1.00 max | 0.03 max | 0.01 max | 19.0-23.0 | 22.0-28.0 | 0.10-0.40 | 0.002-0.010 | 1.0-2.0 | 0.02-0.20 | 0.10-0.25 |
| Typical NF 709 | 0.07 | 0.97 | 0.4 | 0.001 | 0.007 | 20.68 | 24.7 | 0.24 | 0.005 | 1.45 | 0.04 | 0.17 |

# Alloy 709 Is Nitrogen-stabilized, Niobium-Strengthened Austenitic Alloy

- Based on the Fe-20Cr-25Ni composition for power boiler applications
- Excellent oxidation resistance at high temperatures
- Niobium and nitrogen additions increase the tensile and creep strength



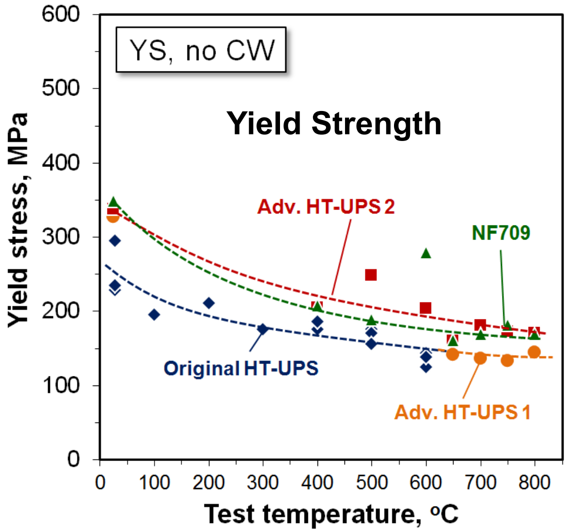



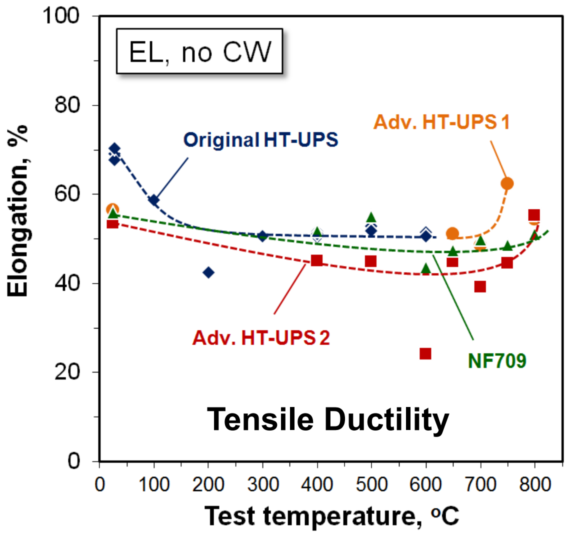



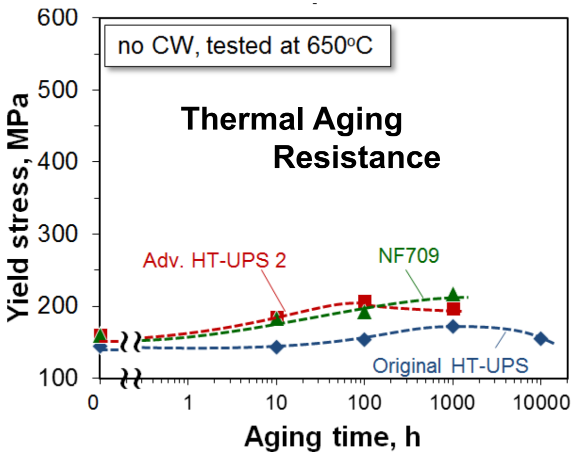

Microstructure after creep test for 5015h @ 750C, 100MPa (TEM-BF images)

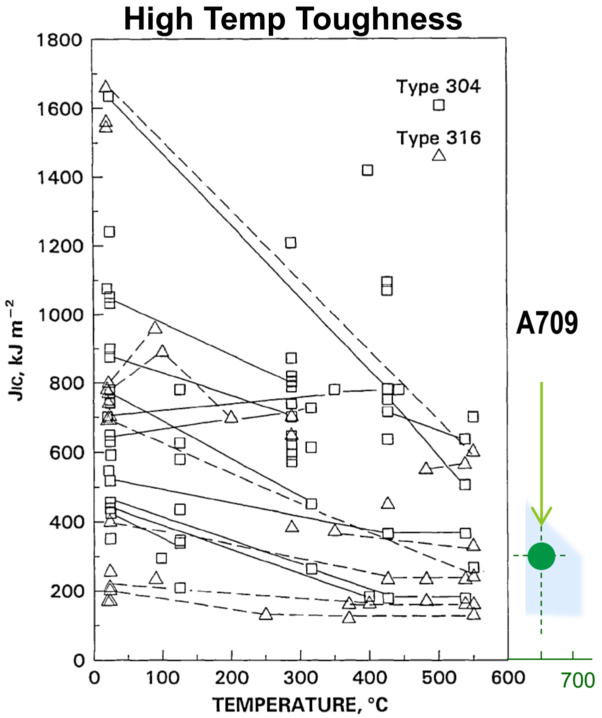
- Most of the grain boundary (GB) is fully covered by precipitates
- Two different sizes of precipitates in the grain interior (GI)

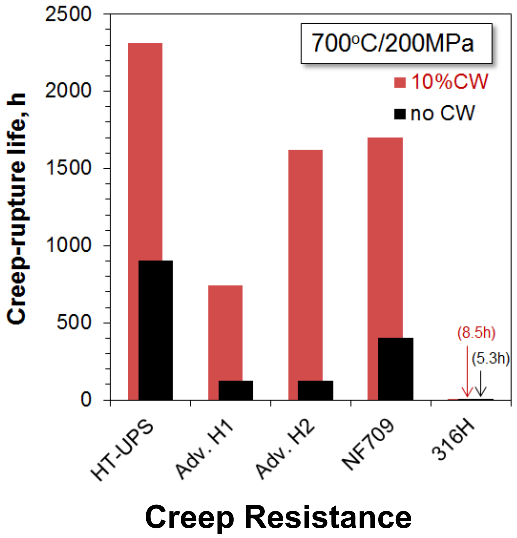
# Austenitic Alloys Down-Selection – Want Balanced Properties

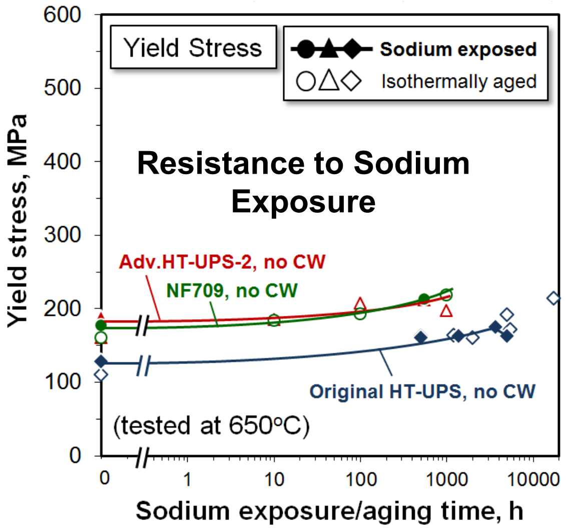


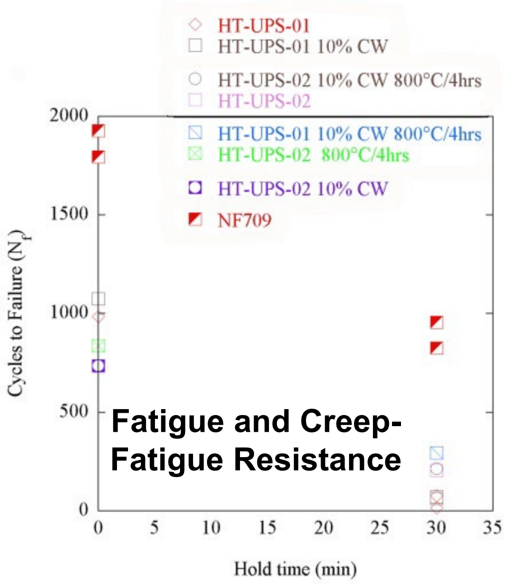



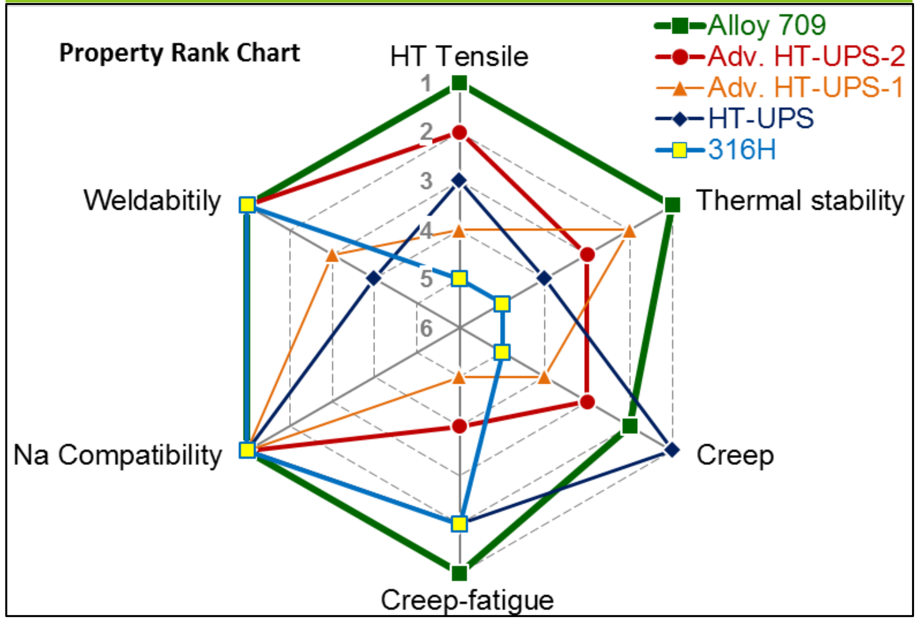




Weldability

# Alloy 709 for ASME Code Qualification

- Alloy 709 offers improved safety margins and design flexibility as well as reduced commodity requirements compared to the austenitic stainless steels qualified for high-temperature nuclear construction
- Alloy 709 ranked as #1 in 5 different properties comparing to other austenitic alloys under consideration in previous analysis
- Alloy 709 has the following improved properties compared to 316H
  - Higher creep strength
  - Better performance in sodium and supercritical $CO_2$ according to short-term test data
  - Higher swelling resistance based on ion irradiation

**Property rank chart for austenitic alloys**



# Comparison Between Alloy 709 and 316H Stainless Steel

| | **316H Stainless Steel** | **Alloy 709** |
|---|---|---|
| Composition | **18Cr-12Ni-**2Mn-**2.5Mo-**0.07C | **22Cr-25Ni**-2Mn-**1.5Mo-**V-Nb-C-B-N |
| Strengthening mechanism | Solution hardening (Mo) + Precipitate hardening ($M_{23}C_6$) | Precipitate hardening {M(C,N), $M_{23}C_{6,}$ CrNbN} |
| YS/UTS/EL (RT) | 205MPa/ 515MPa/ 35% | 270MPa/ 640MPa/ 30% |
| at 650$^{o}$C | 156MPa/ 349MPa/ 24% | 200MPa/ 450MPa/ 45% |
| Advantage | • Lower material cost<br>• Good oxidation resistance<br>• Good weldability<br>• Good sodium compatibility<br>• Widely used, large experience base | • Excellent creep properties<br>• Better oxidation resistance<br>• Good sodium compatibility<br>• Better compatibility with sCO2<br>• Higher creep strength expands design envelope |
| Disadvantage | • Lower creep strength restricts design envelope | • Higher commodity cost due to high Ni<br>• Need improvement on welding |

# Alloy 709 Fabrication Scale-up

400-lb heats

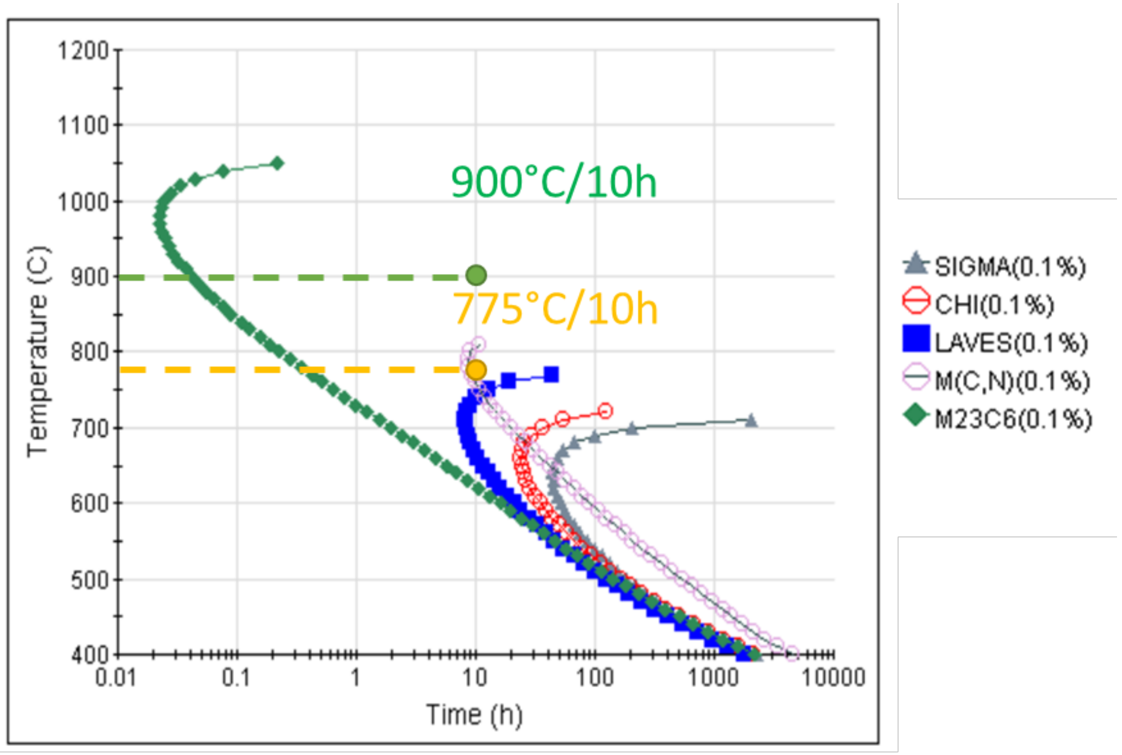
1st 45,000-lb commercial heat, from Carlson

2nd 40,000-lb commercial heat, from ATI

3rd 40,000-lb commercial heat, from ATI, to be delivered in March 2022

# Precipitation Treatment (PT) of Alloy 709

- **Objective**
  - Develop heat treatment plan to age Alloy 709
  - To bring out the desired nano-sized precipitates, such as MX (Nb, Ti, V:C, N) or Z-phase (CrNbN) on dislocations
  - To result in optimized and balanced mechanical properties (creep versus creep-fatigue)
- **Modeling**
  - Conducted thermodynamic and kinetics modeling
  - Selected precipitation treatment (PT) of 775C for 10 hours (775C/10hrs) to:
    - Avoid the formation of the intermetallic laves phase ($Fe_2Mo$, $Fe_2Nb$), and
    - Promote the formation of $M_{23}C_6$ (Cr, Fe, Mo, Ni:C)



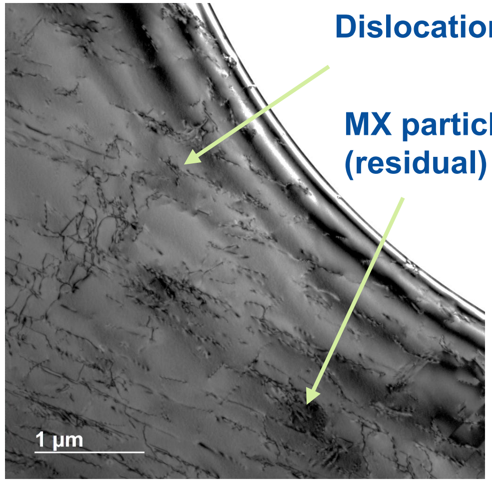

Quench temperature: 1150 °C

# Characterization of Precipitation Treated Alloy 709

## ESR Solution Anneal @ 1100C

Dislocations

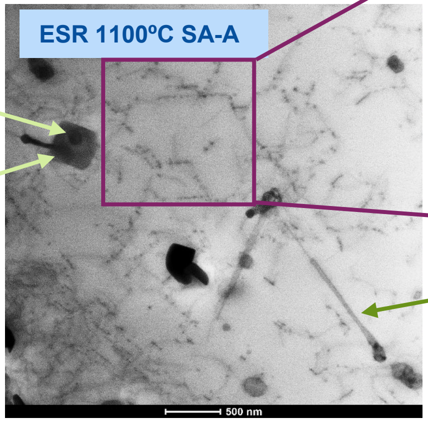
MX particles (residual)



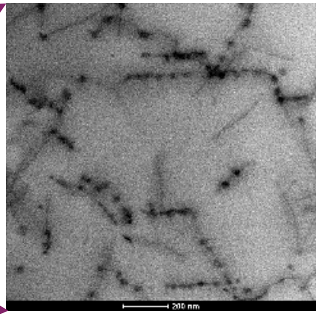

## After Precipitation Treatment (PT) of 775C for 10 hrs

Core: MX particles (residual)

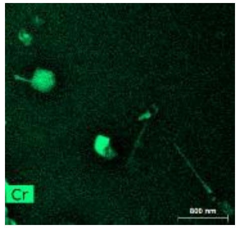
Shell: $M_{23}C_6$

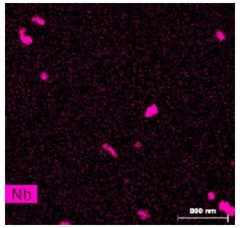


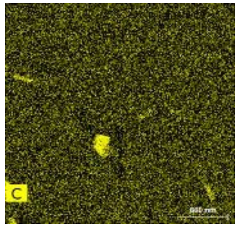

Nb-rich particles on dislocations

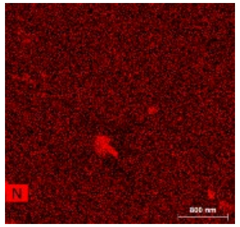


Tail: Cr-rich phase



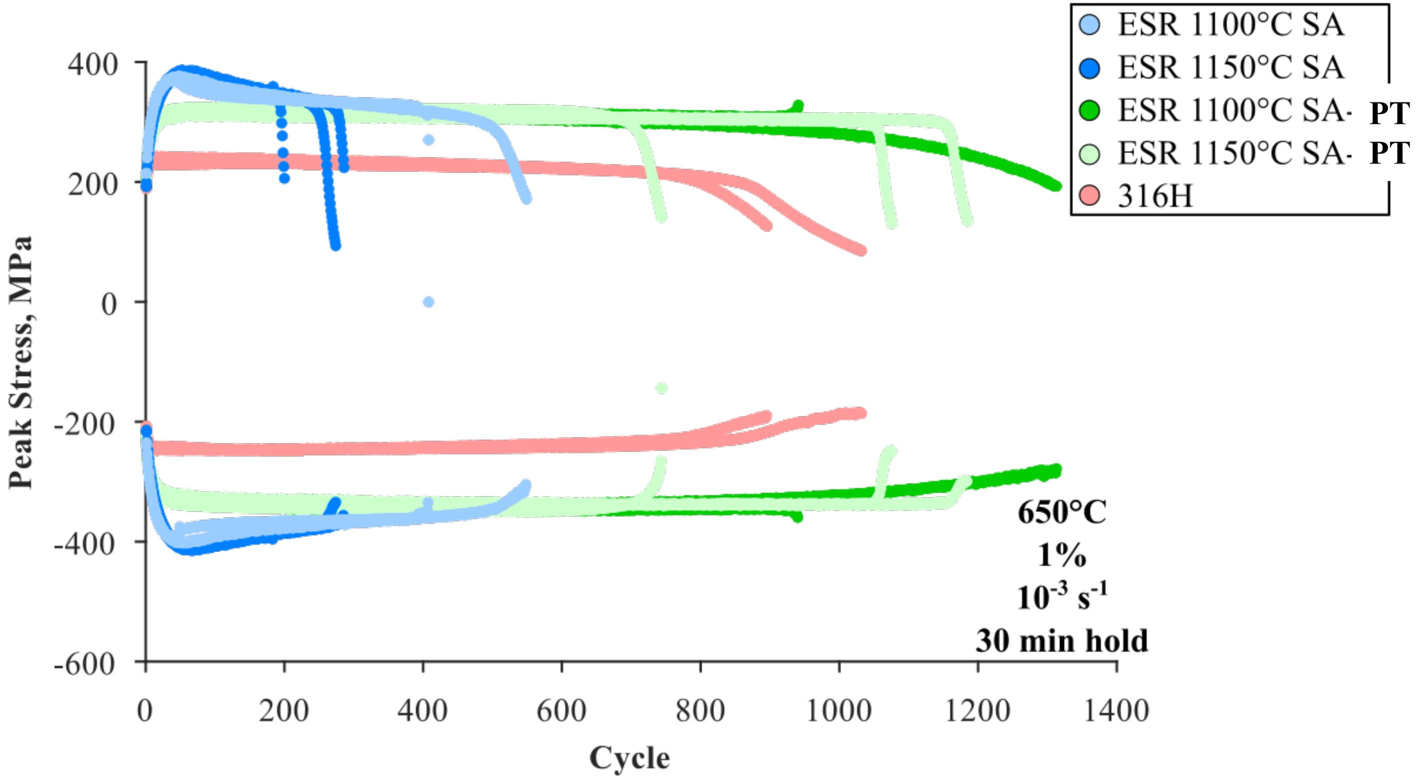




Chemical composition of melt of A709 heat # 58776 (in wt%)

| A709 | C | Cr | Ni | Mn | Mo | N | Si | P | Ti | Nb | B | Fe |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Actual** | 0.066 | 19.93 | 24.98 | 0.91 | 1.51 | 0.148 | 0.44 | 0.014 | 0.04 | 0.26 | 0.0045 | Bal |

# Creep-Fatigue Performance of Alloy 709 (PT and Non-PT) and 316H

- ESR A709 Solution Anneal (SA) plus Precipitation Treatment (PT) has the greatest number of cycles to failure
- Followed by 316H and finally ESR A709-SA
- The ESR A709-SA heat exhibits a different creep-fatigue behavior than ESR A709-A and 316H
- The overall peak stress of the ESR A709-SA and ESR A709-PT is greater than 316H



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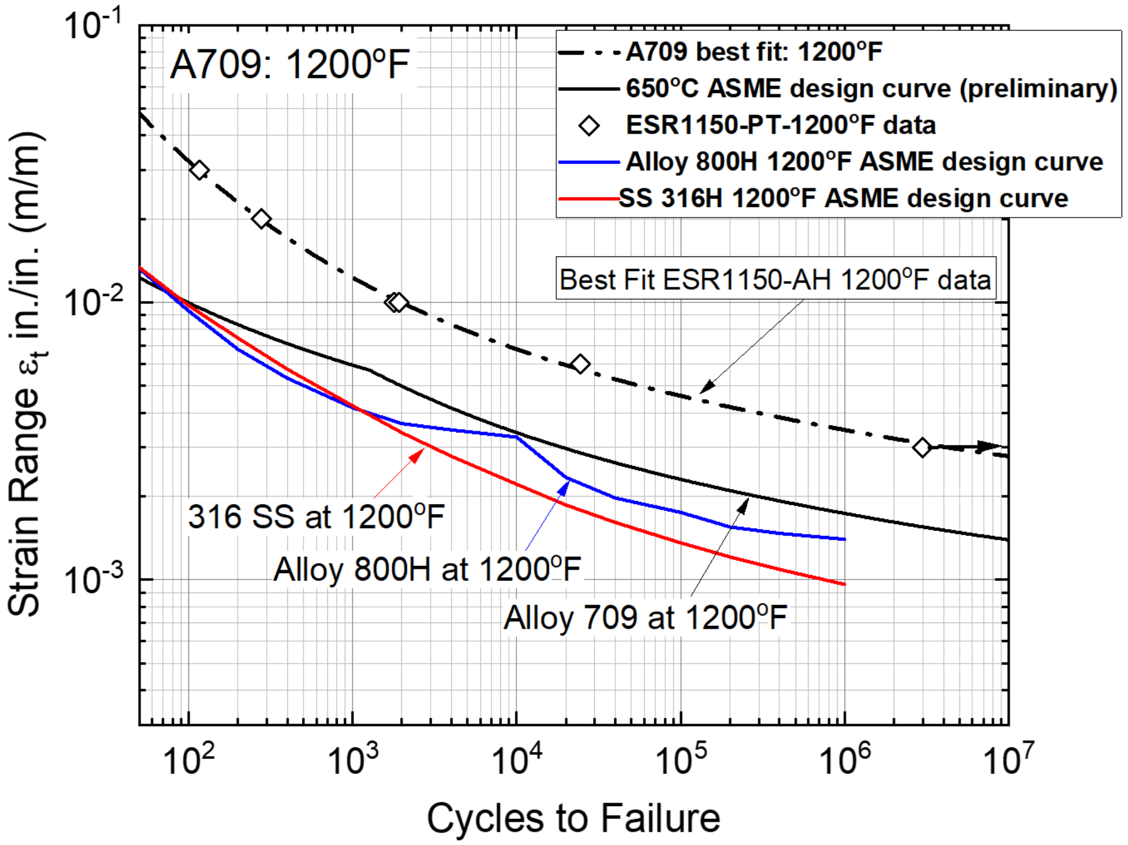
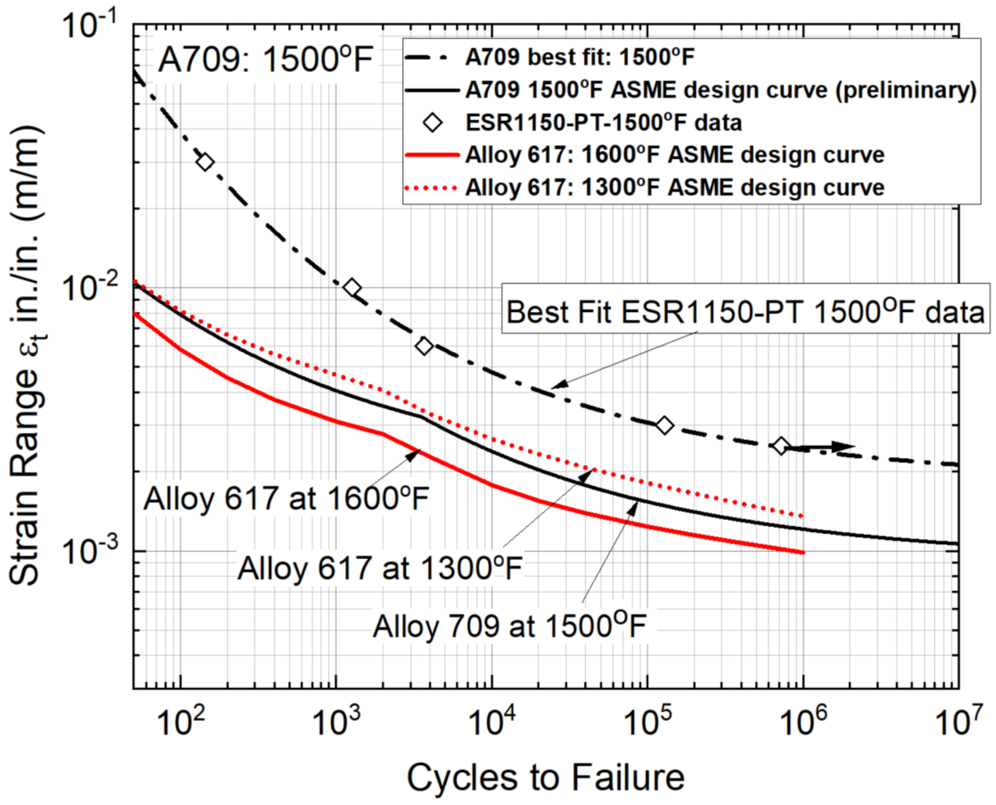
# Alloy 709-PT Has Enhanced Fatigue Properties

### Alloy 709-PT better than Alloy 800H and 316H at 1200F (650C)

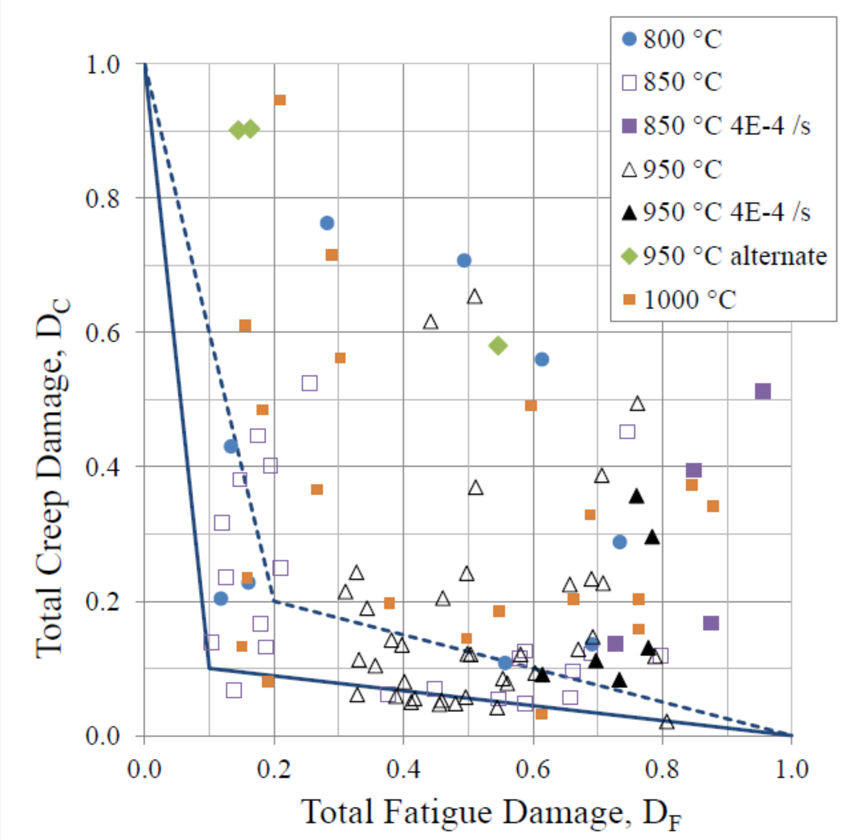



### Alloy 709-PT comparable to Alloy 617 at 1500F (816C)

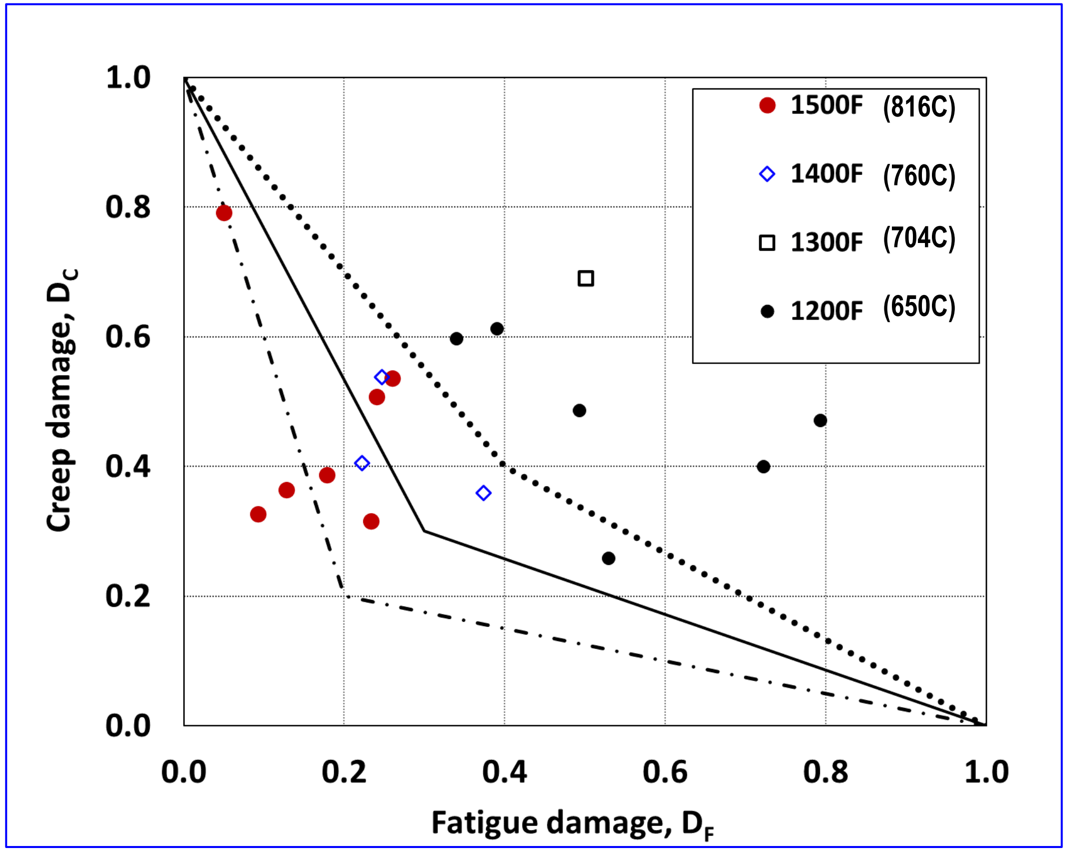


# Alloy 709-PT Has Very Good Resistance to Creep-Fatigue Interaction

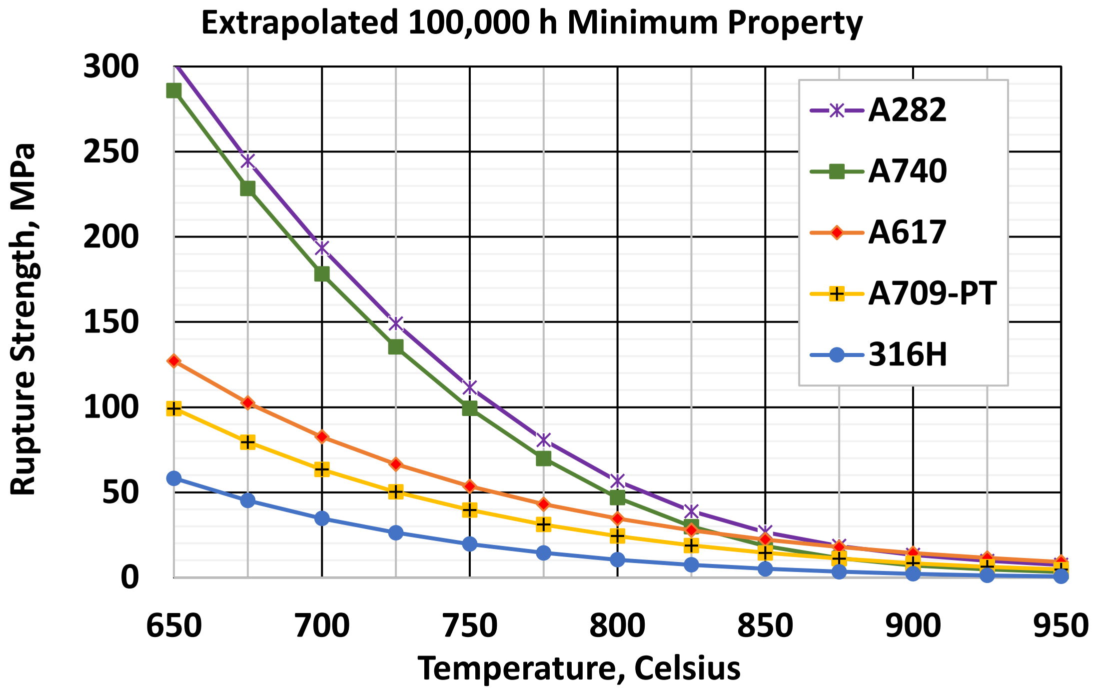
Alloy 617 Creep-Fatigue Interaction

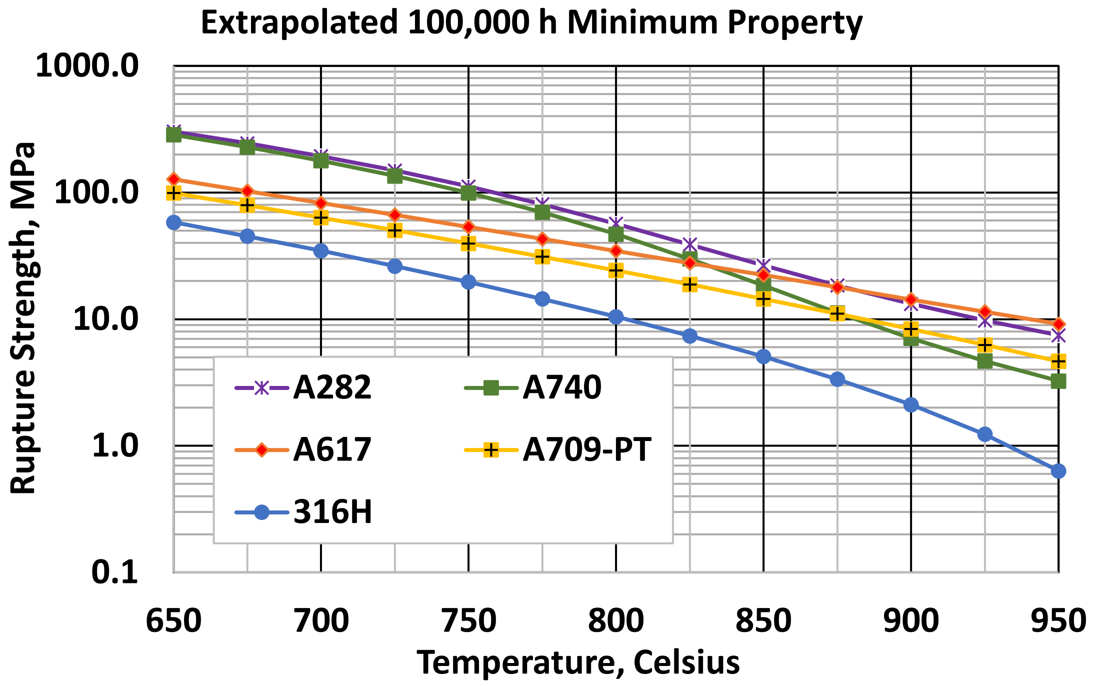



Alloy 709-PT Creep-Fatigue Interaction



# Extrapolated 100,000-Hour Minimum Rupture Stress: Alloys 282, 740, 617, 709-PT & 316H Stainless



# Extrapolated 100,000-Hour Minimum Rupture Stress

| Temp, C | Minimum Rupture Stress Values | | | | |
|---|---|---|---|---|---|
| | Nickel Alloys | | | Stainless Steels | |
| | A282 | A740 | A617 | A709-PT | 316H |
| 650 | 302.7 | 286.0 | 127.1 | 99.2 | 58.2 |
| 675 | 244.7 | 228.4 | 102.5 | 79.5 | 45.1 |
| 700 | 193.6 | 178.3 | 82.6 | 63.4 | 34.6 |
| 725 | 149.2 | 135.4 | 66.5 | 50.3 | 26.3 |
| 750 | 111.6 | 99.3 | 53.5 | 39.7 | 19.6 |
| 775 | 80.8 | 69.9 | 43.0 | 31.1 | 14.5 |
| 800 | 56.7 | 46.9 | 34.6 | 24.3 | 10.4 |
| 825 | 38.9 | 29.9 | 27.7 | 18.8 | 7.4 |
| 850 | 26.6 | 18.4 | 22.3 | 14.5 | 5.1 |
| 875 | 18.5 | 11.2 | 17.8 | 11.0 | 3.4 |
| 900 | 13.2 | 7.1 | 14.3 | 8.4 | 2.1 |
| 925 | 9.8 | 4.7 | 11.4 | 6.3 | 1.2 |
| 950 | 7.5 | 3.3 | 9.1 | 4.6 | 0.6 |

| Temp, C | Minimum Rupture Stress Ratios (/w A282) | | | | |
|---|---|---|---|---|---|
| | Nickel Alloys | | | Stainless Steels | |
| | A282 | A740 | A617 | A709-PT | 316H |
| 650 | 1.0 | 0.95 | 0.42 | 0.33 | 0.19 |
| 675 | 1.0 | 0.93 | 0.42 | 0.32 | 0.18 |
| 700 | 1.0 | 0.92 | 0.43 | 0.33 | 0.18 |
| 725 | 1.0 | 0.91 | 0.45 | 0.34 | 0.18 |
| 750 | 1.0 | 0.89 | 0.48 | 0.36 | 0.18 |
| 775 | 1.0 | 0.87 | 0.53 | 0.39 | 0.18 |
| 800 | 1.0 | 0.83 | 0.61 | 0.43 | 0.18 |
| 825 | 1.0 | 0.77 | 0.71 | 0.48 | 0.19 |
| 850 | 1.0 | 0.69 | 0.84 | 0.54 | 0.19 |
| 875 | 1.0 | 0.61 | 0.97 | 0.60 | 0.18 |
| 900 | 1.0 | 0.54 | 1.08 | 0.63 | 0.16 |
| 925 | 1.0 | 0.48 | 1.17 | 0.64 | 0.13 |
| 950 | 1.0 | 0.44 | 1.23 | 0.62 | 0.09 |

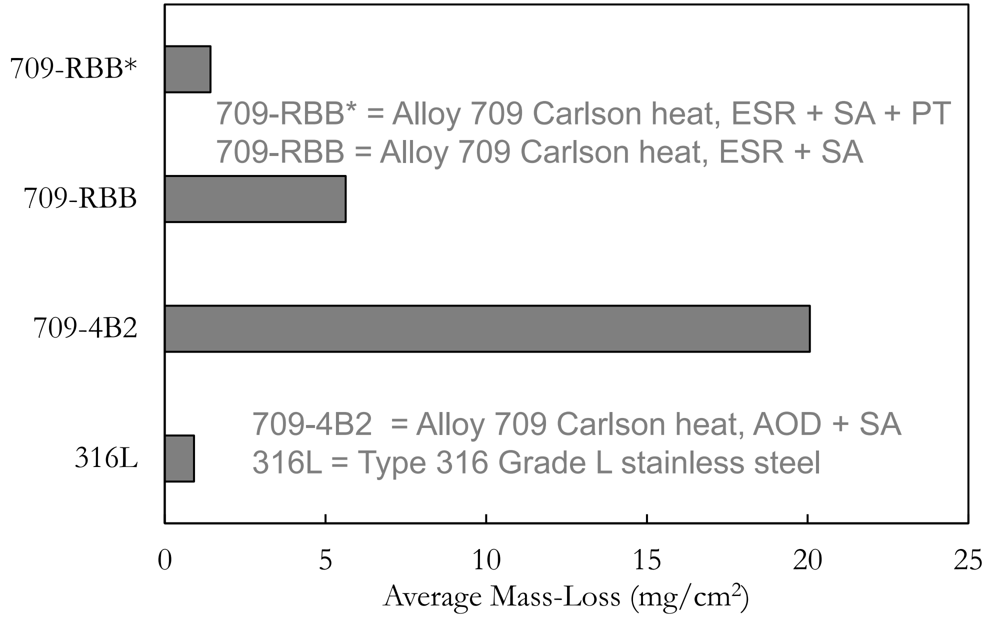
# Estimates of Commodity Prices for Elements of Alloy 282, Alloy 740 & Alloy 709-PT

| | USD/MT | Alloy Composition Cost, USD/MT | | | Composition, Wt % | | |
|---|---|---|---|---|---|---|---|
| | | A282 | A740 | A709-PT | A282 | A740 | A709-PT |
| Ni [1] | $19,376 | $10,828 | $9,353 | $4,840 | 55.9 | 48.3 | 24.98 |
| Cr [2] | $9,400 | $1,880 | $2,350 | $1,873 | 20 | 25 | 19.93 |
| Co [1] | $80,490 | $8,049 | $16,098 | $0 | 10 | 20 | 0 |
| Mo [1] | $26,000 | $2,210 | $130 | $393 | 8.5 | 0.5 | 1.51 |
| Ti [1] | $4,800 | $101 | $86 | $2 | 2.1 | 1.8 | 0.04 |
| Al [1] | $2,834 | $43 | $26 | $0 | 1.5 | 0.9 | 0 |
| Fe (iron ore) [1] | $114 | $2 | $1 | $59 | 1.5 | 0.7 | 51.70 |
| Mn [1] | $2,060 | $6 | $6 | $19 | 0.3 | 0.3 | 0.91 |
| Si [2] | $1,700 | $3 | $9 | $7 | 0.15 | 0.5 | 0.44 |
| C [2] | $122 | $0 | $0 | $0 | 0.06 | 0.03 | 0.066 |
| B [2] | $3,680 | $0 | $0 | $0 | 0.005 | 0 | 0.0045 |
| Nb [1] | $42,280 | $0 | $846 | $110 | 0 | 2 | 0.26 |
| N [2] | $140 | $0 | $0 | $0 | 0 | 0 | 0.148 |
| P [2] | $2,690 | $0 | $0 | $0 | 0 | 0 | 0.014 |
| | | $23,121 | $28,904 | $7,304 | 100.0 | 100.0 | 100.0 |

[1] https://www.metalary.com/
[2] https://en.wikipedia.org/wiki/Prices_of_chemical_elements

# Alloy 709 Chloride Salt Compatibility Scoping Study [1]

- Chloride salt: Purified $MgCl_2$-NaCl-KCl (45-53-2 wt.%)
- Uncoated materials
  - Corrosion exposure at 800C for 100 hours in static capsule
  - Average corrosion rate (slowest to fastest)
    1. 316L (0.91 mg/cm$^2$)
    2. 709-RBB* (1.42 mg/cm$^2$)
    3. 709-RBB (5.63 mg/cm$^2$)
    4. 709-4B2 (20.07 mg/cm$^2$)




- Selected Alloy 709 material with processing condition that gave the worst corrosion performance, 709-4B2 (AOD + SA), for coating
  - Chloride salt exposure at 700C for 120 hours in static capsule
  - Preliminary examination of pre-exposure and post-exposure coated sample
    - Coating is effective in preventing chloride salt corrosion for the worst performing uncoated Alloy 709 processing condition (709-4B2)

[1] Results courtesy of J. Zhang, Virginia Tech

# Summary

- Alloy 709 is an advanced austenitic alloy that has very high creep rupture strength among austenitic stainless steels
- Alloy 709-PT (Precipitation Treatment) has balanced creep vs. creep-fatigue performance
- Creep strength of Alloy 709-PT is lower than nickel alloys such Alloy 282, Alloy 740 and Alloy 617, particularly for lower temperature applications
- At higher temperatures, creep strength of Alloy 709-PT drops more gradually as temperature is increased, even up to 950C
- Fatigue and creep-fatigue performance of Alloy 709-PT is excellent
- Data package generation sponsored by DOE-NE is ongoing to codify Alloy 709-PT in ASME Boiler and Pressure Vessel Code, Section III, Division 5 for high temperature reactor applications
- Limited short-term chloride salt static exposure data showed coating as an effective strategy to prevent molten chloride salt corrosion of Alloy 709-PT
  - Longer term exposure and high temperature mechanical properties testing are required to confirm the beneficial effect of coating for Alloy 709-PT in molten chloride salt

# Backup Slides

# Extrapolated 100,000-Hour Minimum Rupture Stress: Alloys 740, 617, 709-PT & 316H Stainless