Low Cost High Performance Austenitic Stainless Steels for A-USC (FWP-FEAA133)

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ORNL is managed by UT-Battelle, LLC for the US Department of Energy

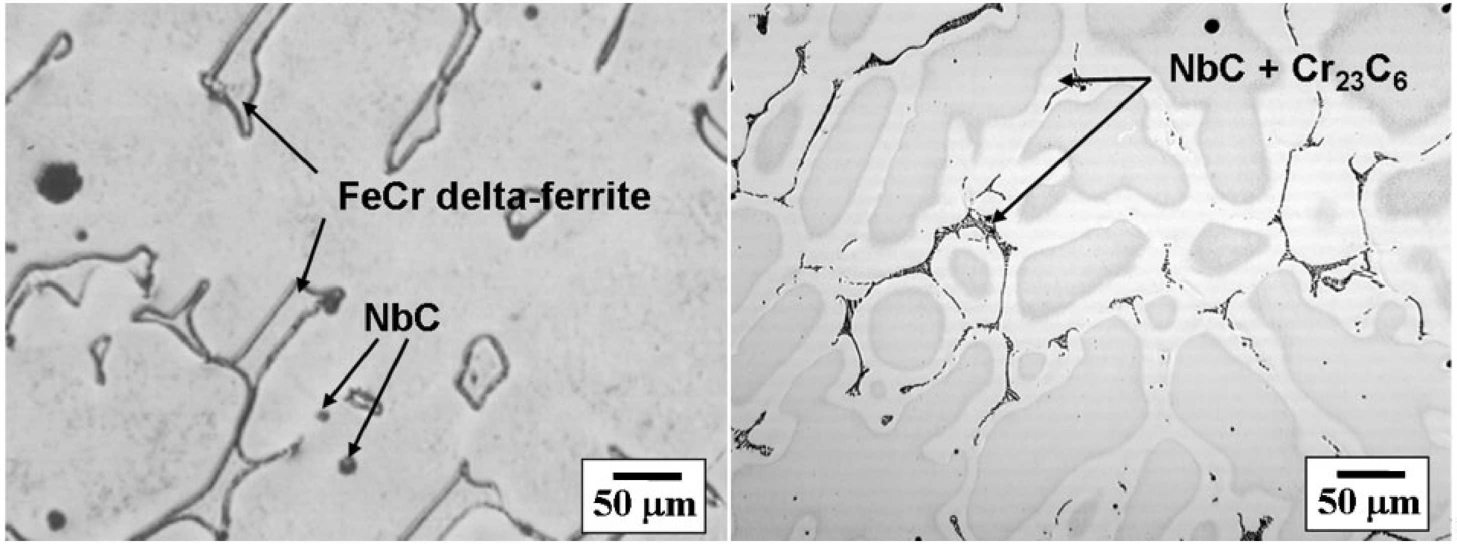
2024 FECM/NETL Spring R&D Project Review Meeting
April 23 – 25, 2024
Pittsburgh, PA

Background (1/2)

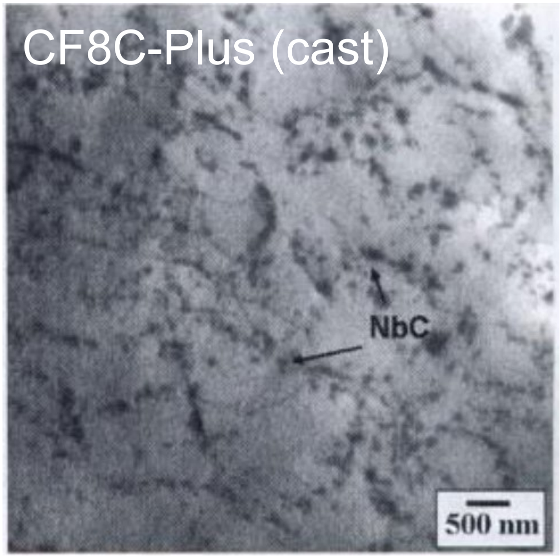
CF8C-Plus is a heat- and corrosion-resistant cast austenitic stainless steel developed by the Oak Ridge National Laboratory and the Caterpillar Technical Center

Composition (wt%)

	C	Si	Mn	Cr	Mo	Ni	Nb	N	Fe
CF8C-Plus	**0.08**	**0.5**	**4.0**	**19.0**	**0.3**	**12.5**	**0.80**	**0.25**	**Bal**
CF8C	0.1	1.0	1.0 max	19.0	0.3	10	0.80	-	Bal



As-cast microstructure: CF8C (left) & CF8C-Plus (right)



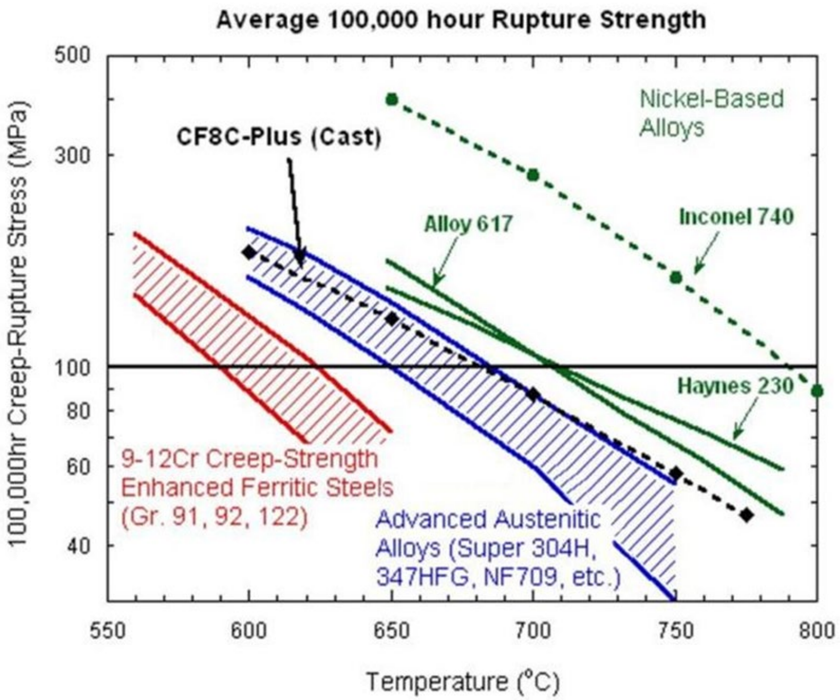
Nanoscale NbC precipitates in CF8C-Plus (courtesy of EPRI)

Shingledecker et al., Energy Materials 2006

Background (2/2)

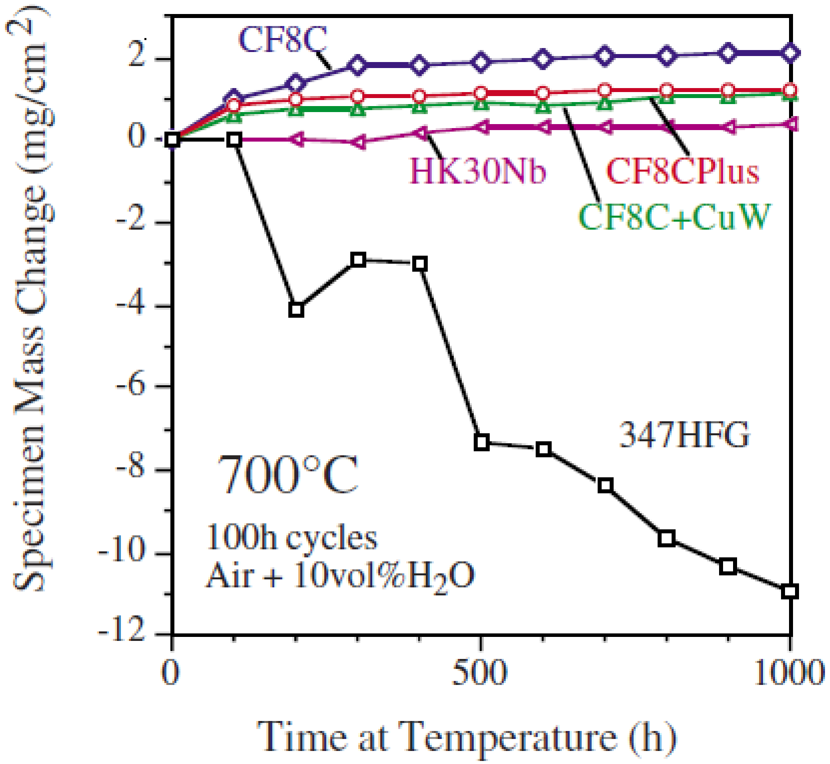
High temperature strength

Bridging between 9-12Cr CSEF steels and nickel-based alloys (courtesy of EPRI)



Corrosion resistance

Better corrosion resistance in 700°C humid air than 347HFG



Castability

CF8C-Plus fluidity spiral

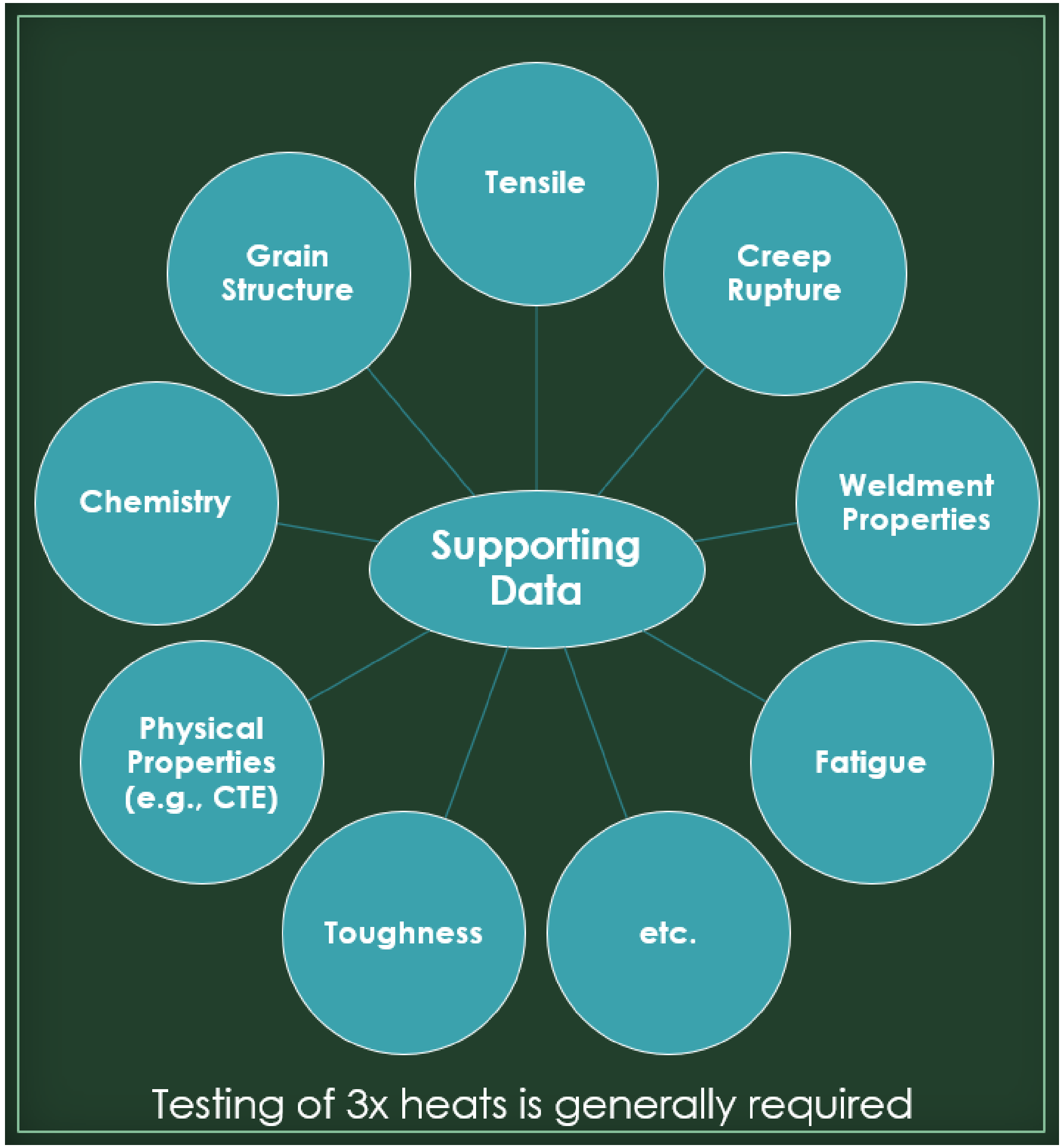
Weldability

Cross-section view of SMAW of CF8C-Plus

The strength, corrosion resistance, and weldability are found in the **as-cast condition** without additional heat-treatment

Maziasz and Pint, J ENG GAS TURB POWER, 2011

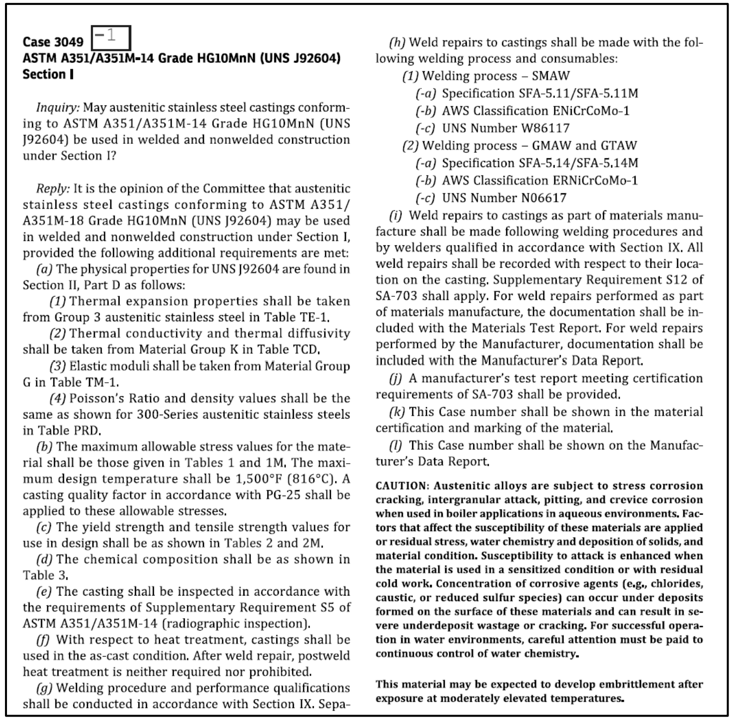
Project Objective: create **cast (ORNL lead)** and **wrought (EPRI lead)** CF8C-Plus data packages and pursue ASME Code Case approvals



Testing of 3x heats is generally required

Cast CF8C-Plus Code Case Status

ASME BPVC Sec I code case (CC) #3049-1 and ASME B31.1 CC #199-2 approved



Case 3049 -1
ASTM A351/A351M-14 Grade HG10MnN (UNS J92604)
Section I

Inquiry: May austenitic stainless steel castings conforming to ASTM A351/A351M-14 Grade HG10MnN (UNS J92604) be used in welded and nonwelded construction under Section I?

Reply: It is the opinion of the Committee that austenitic stainless steel castings conforming to ASTM A351/A351M-18 Grade HG10MnN (UNS J92604) may be used in welded and nonwelded construction under Section I, provided the following additional requirements are met:

(a) The physical properties for UNS J92604 are found in Section II, Part D as follows:

(1) Thermal expansion properties shall be taken from Group 3 austenitic stainless steel in Table TE-1.

(2) Thermal conductivity and thermal diffusivity shall be taken from Material Group K in Table TCD.

(3) Elastic moduli shall be taken from Material Group G in Table TM-1.

(4) Poisson's Ratio and density values shall be the same as shown for 300-Series austenitic stainless steels in Table PRD.

(b) The maximum allowable stress values for the material shall be those given in Tables 1 and 1M. The maximum design temperature shall be 1,500°F (816°C). A casting quality factor in accordance with PG-25 shall be applied to these allowable stresses.

(c) The yield strength and tensile strength values for use in design shall be as shown in Tables 2 and 2M.

(d) The chemical composition shall be as shown in Table 3.

(e) The casting shall be inspected in accordance with the requirements of Supplementary Requirement S5 of ASTM A351/A351M-14 (radiographic inspection).

(f) With respect to heat treatment, castings shall be used in the as-cast condition. After weld repair, postweld heat treatment is neither required nor prohibited.

(g) Welding procedure and performance qualifications shall be conducted in accordance with Section IX. Sepa-

(h) Weld repairs to castings shall be made with the following welding process and consumables:

- *(1)* Welding process – SMAW
  - *(-a)* Specification SFA-5.11/SFA-5.11M
  - *(-b)* AWS Classification ENiCrCoMo-1
  - *(-c)* UNS Number W86117
- *(2)* Welding process – GMAW and GTAW
  - *(-a)* Specification SFA-5.14/SFA-5.14M
  - *(-b)* AWS Classification ERNiCrCoMo-1
  - *(-c)* UNS Number N06617

(i) Weld repairs to castings as part of materials manufacture shall be made following welding procedures and by welders qualified in accordance with Section IX. All weld repairs shall be recorded with respect to their location on the casting. Supplementary Requirement S12 of SA-703 shall apply. For weld repairs performed as part of materials manufacture, the documentation shall be included with the Materials Test Report. For weld repairs performed by the Manufacturer, documentation shall be included with the Manufacturer's Data Report.

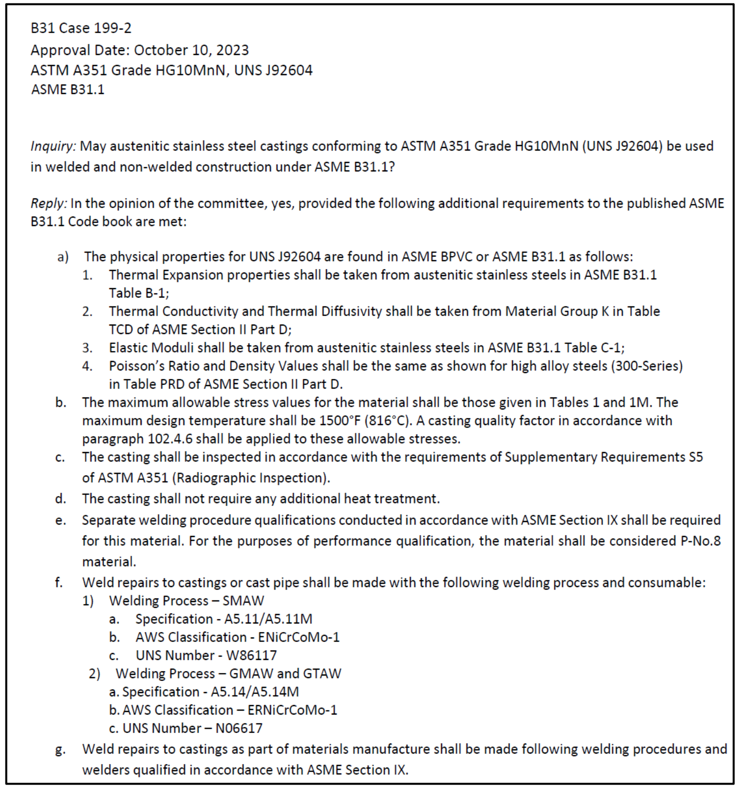
(j) A manufacturer's test report meeting certification requirements of SA-703 shall be provided.

(k) This Case number shall be shown in the material certification and marking of the material.

(l) This Case number shall be shown on the Manufacturer's Data Report.

CAUTION: Austenitic alloys are subject to stress corrosion cracking, intergranular attack, pitting, and crevice corrosion when used in boiler applications in aqueous environments. Factors that affect the susceptibility of these materials are applied or residual stress, water chemistry and deposition of solids, and material condition. Susceptibility to attack is enhanced when the material is used in a sensitized condition or with residual cold work. Concentration of corrosive agents (e.g., chlorides, caustic, or reduced sulfur species) can occur under deposits formed on the surface of these materials and can result in severe underdeposit wastage or cracking. For successful operation in water environments, careful attention must be paid to continuous control of water chemistry.

This material may be expected to develop embrittlement after exposure at moderately elevated temperatures.



B31 Case 199-2
Approval Date: October 10, 2023
ASTM A351 Grade HG10MnN, UNS J92604
ASME B31.1

Inquiry: May austenitic stainless steel castings conforming to ASTM A351 Grade HG10MnN (UNS J92604) be used in welded and non-welded construction under ASME B31.1?

Reply: In the opinion of the committee, yes, provided the following additional requirements to the published ASME B31.1 Code book are met:

a) The physical properties for UNS J92604 are found in ASME BPVC or ASME B31.1 as follows:
   1. Thermal Expansion properties shall be taken from austenitic stainless steels in ASME B31.1 Table B-1;
   2. Thermal Conductivity and Thermal Diffusivity shall be taken from Material Group K in Table TCD of ASME Section II Part D;
   3. Elastic Moduli shall be taken from austenitic stainless steels in ASME B31.1 Table C-1;
   4. Poisson's Ratio and Density Values shall be the same as shown for high alloy steels (300-Series) in Table PRD of ASME Section II Part D.

b. The maximum allowable stress values for the material shall be those given in Tables 1 and 1M. The maximum design temperature shall be 1500°F (816°C). A casting quality factor in accordance with paragraph 102.4.6 shall be applied to these allowable stresses.

c. The casting shall be inspected in accordance with the requirements of Supplementary Requirements S5 of ASTM A351 (Radiographic Inspection).

d. The casting shall not require any additional heat treatment.

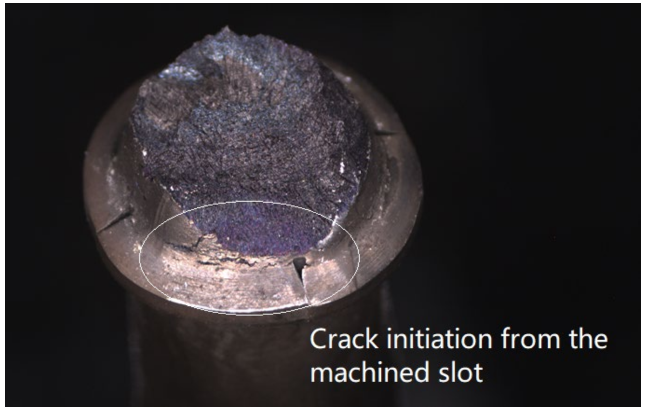
e. Separate welding procedure qualifications conducted in accordance with ASME Section IX shall be required for this material. For the purposes of performance qualification, the material shall be considered P-No.8 material.

f. Weld repairs to castings or cast pipe shall be made with the following welding process and consumable:
   1) Welding Process – SMAW
      a. Specification - A5.11/A5.11M
      b. AWS Classification - ENiCrCoMo-1
      c. UNS Number - W86117
   2) Welding Process – GMAW and GTAW
      a. Specification - A5.14/A5.14M
      b. AWS Classification – ERNiCrCoMo-1
      c. UNS Number – N06617

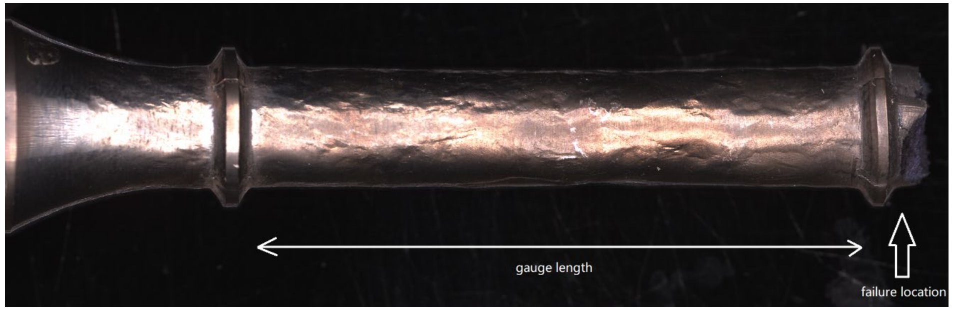
g. Weld repairs to castings as part of materials manufacture shall be made following welding procedures and welders qualified in accordance with ASME Section IX.

CCs 3049-1 (left) and 199-2 (right)

Future work: Both CCs are being revised due to an accidental inclusion of one problematic creep test result



Crack initiation from the machined slot



Material heat	Temperature (°C)	Stress (MPa)	Rupture time (hrs)
257R	538	344.8	273
257R	538	344.8	2040

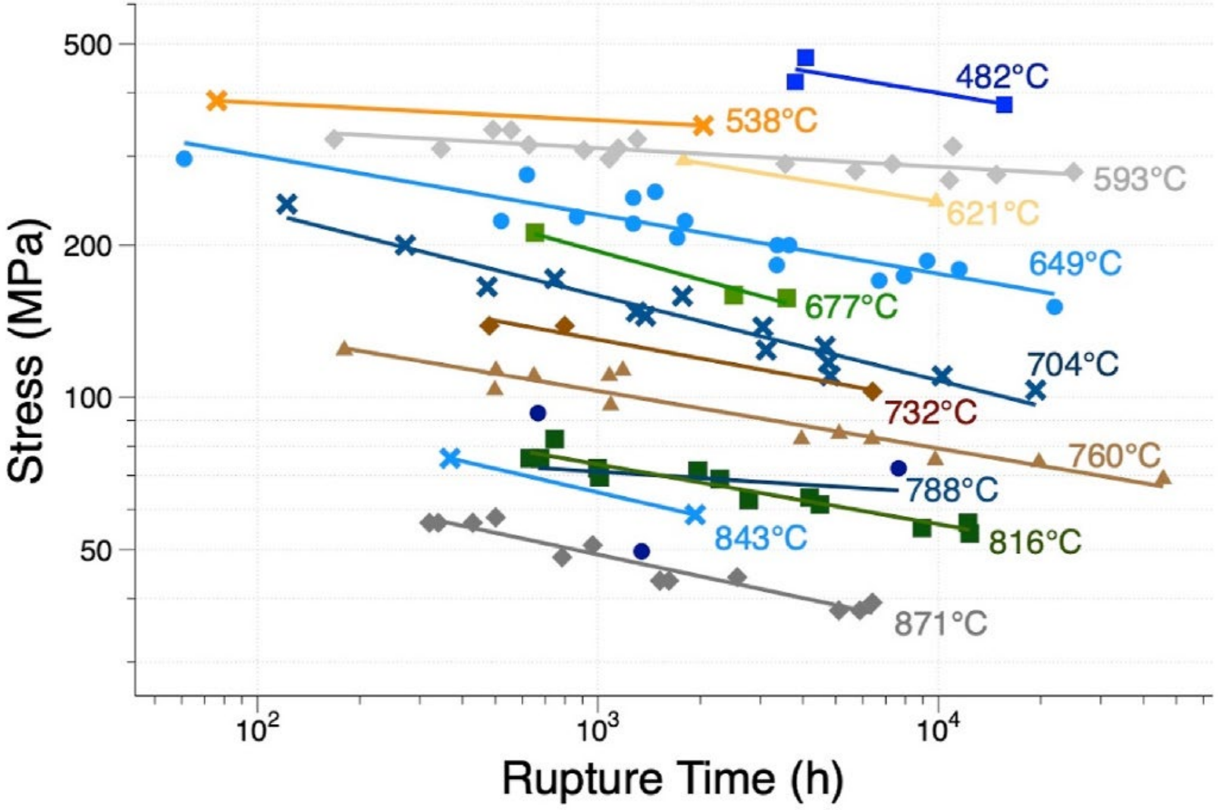
Cast CF8C-Plus Creep Life Modeling

M.L. Santella, P.F. Tortorelli, M. Render, H. Wang, T. Lach, B.A. Pint, P.J. Maziasz, V. Cedro III, X. Chen

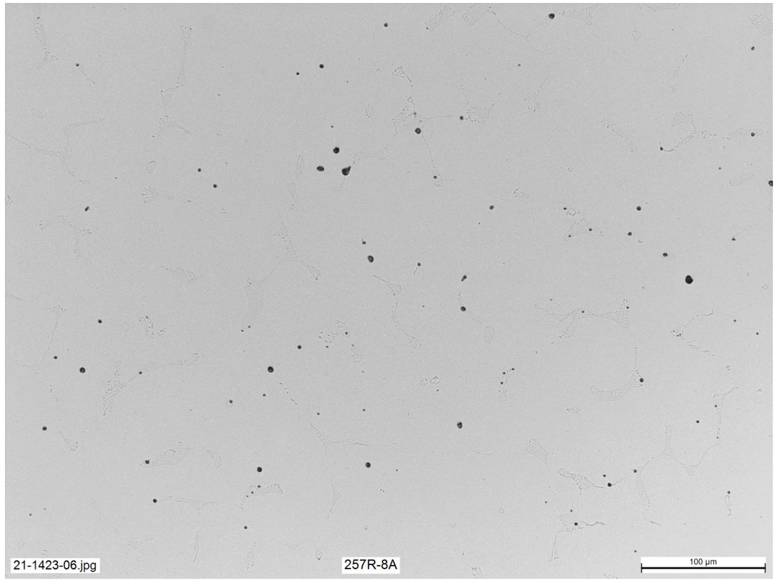
International Journal of Pressure Vessels and Piping 205 (2023) 105006

Motivation

- The availability of the relatively large creep-rupture dataset for cast CF8C-Plus
- Impact of larger scatter in creep rupture data for cast CF8C-Plus
- Effect of starting microstructures, temperature, and applied stress



Applied stress versus rupture time of CF8C-Plus



Optical image showing porosity in cast CF8C-Plus

Average % porosity, pore diameter, number of pores (N), and grain size for cast CF8C-Plus

Heat	% Porosity	Pore Diam. µm	N	Grain Size (±1σ) µm
257 R	0.14 ± 0.02	2.2 ± 1.8	795	481 (±211)
DA20	0.15 ± 0.05	2.7 ± 2.2	553	746 (±380)
T038	0.09 ± 0.03	2.2 ± 1.8	471	591 (±358)

Methodology

Input:

Stress and temperature

Creep life modeling →

Output:

Creep rupture time

1. Larson-Miller Parameter (LMP)

$$LMP = T(\log t_f + C)$$

T: *temperature in K*, t_f: *time to rupture in hours*, C: *constant*

$$LMP = f(\sigma) = B_0 + \Sigma B_n (\log \sigma_a)^n$$

$B_0, B_1, \ldots, B_n$ *are constants*

$$\log t_f = \frac{B_0 + \Sigma B_n (\log \sigma_a)^n}{T} - C$$

2. Wilshire

$$f(\sigma) = -\frac{\ln k}{u} + \frac{1}{u}\left\{\ln\left[-\ln\left(\frac{\sigma_a}{\sigma_{TS}}\right)\right]\right\}$$

k *and* u *are constants*

$$f(\sigma) = \ln t_f - \left(\frac{Q_c^*}{RT}\right)$$

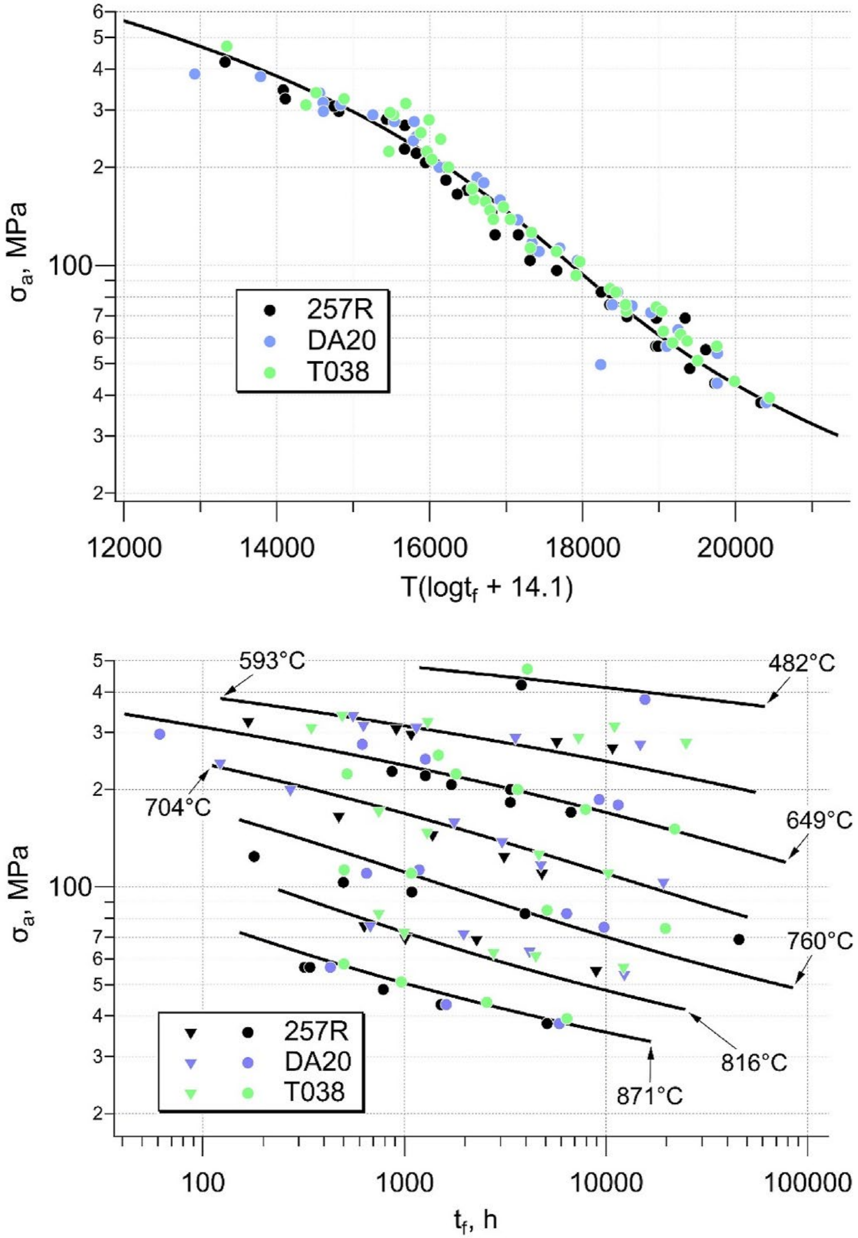
R: *gas constants*

Q_c^*: *activation energy*

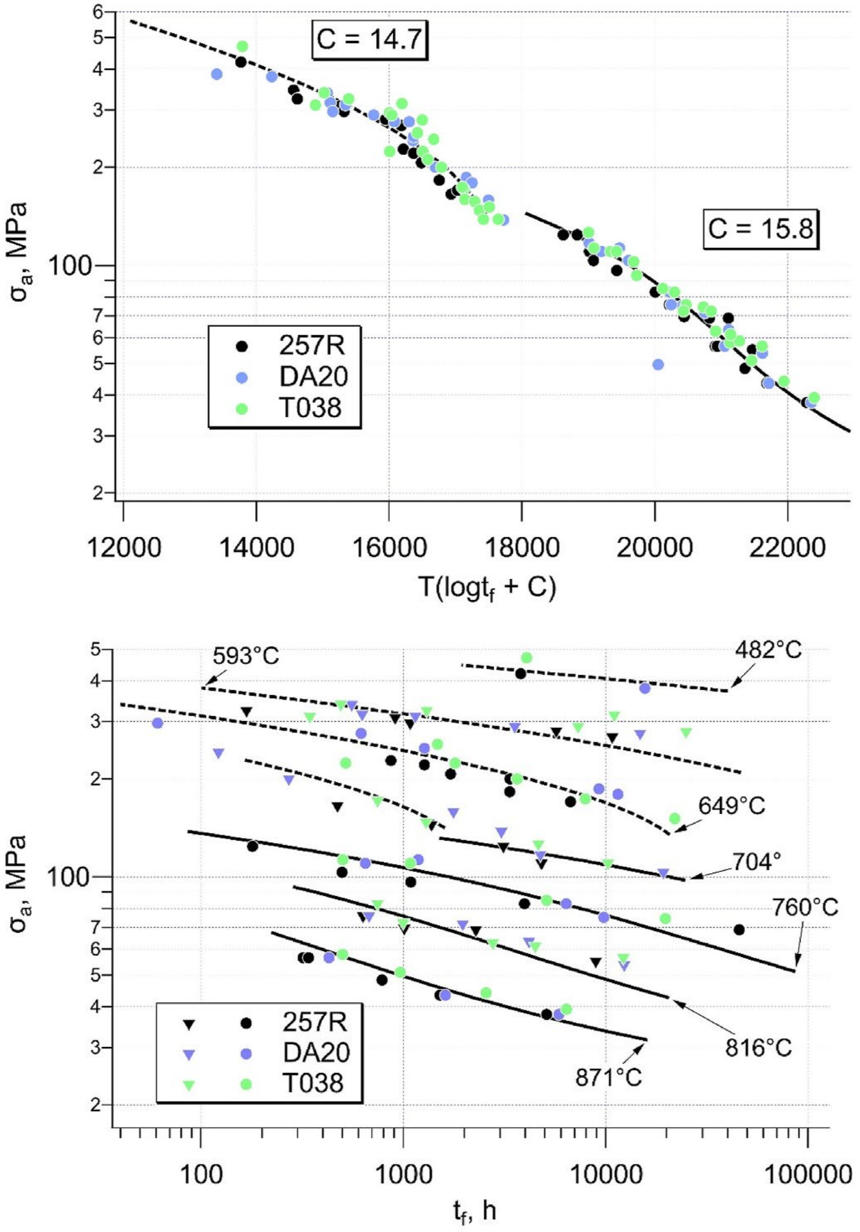
$$\frac{\sigma_a}{\sigma_{TS}} = \exp\left\{-k\left[t_f \exp\left(-\frac{Q_c^*}{RT}\right)\right]^u\right\}$$

LMP Results

- Third-order polynomial stress function (n = 3) produced the best overall fit to the data
- Evaluated single-region fit for all data and split-region fit for stress above YS and below YS
- R^2:
  - Single-region: 0.65
  - Split-region: 0.69
- No significant difference between two analyses

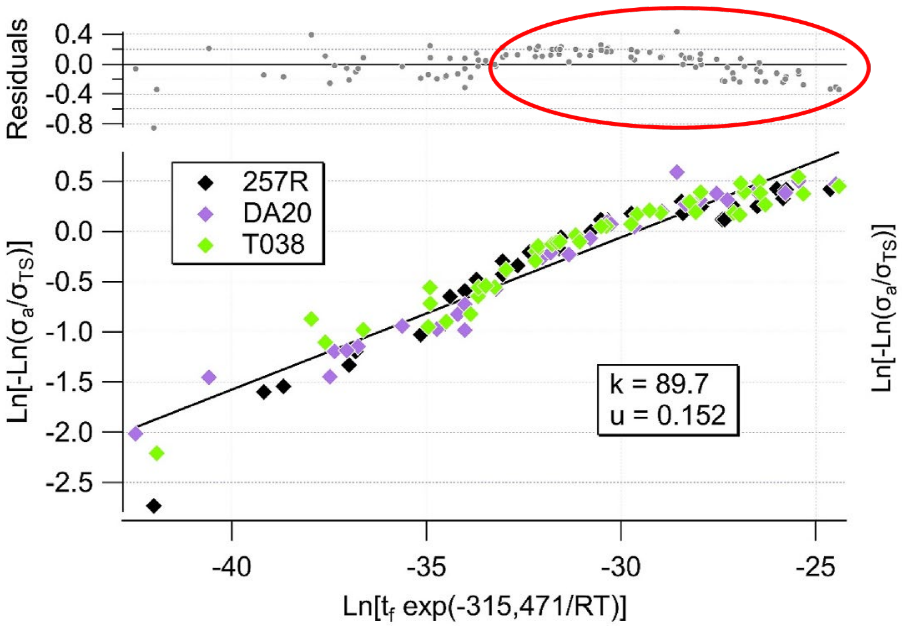


Single-region fit

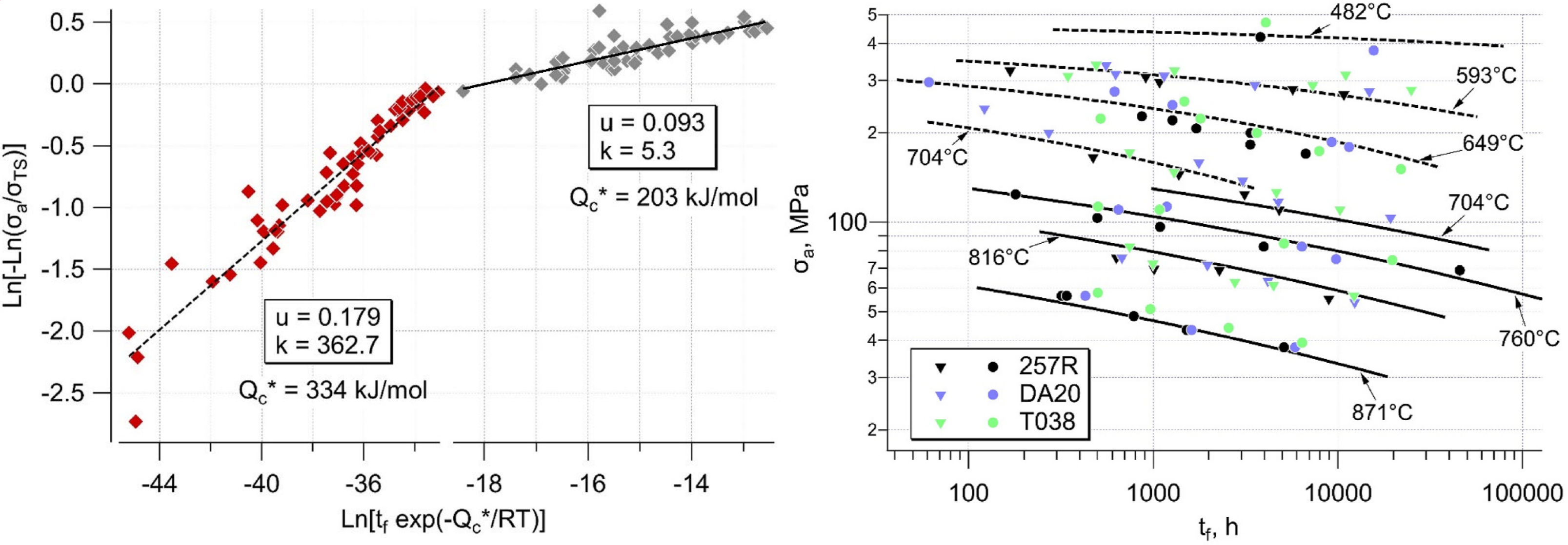


Split-region fit (above YS: dashed line, below YS: solid line)

Wilshire Results



Single-region fit

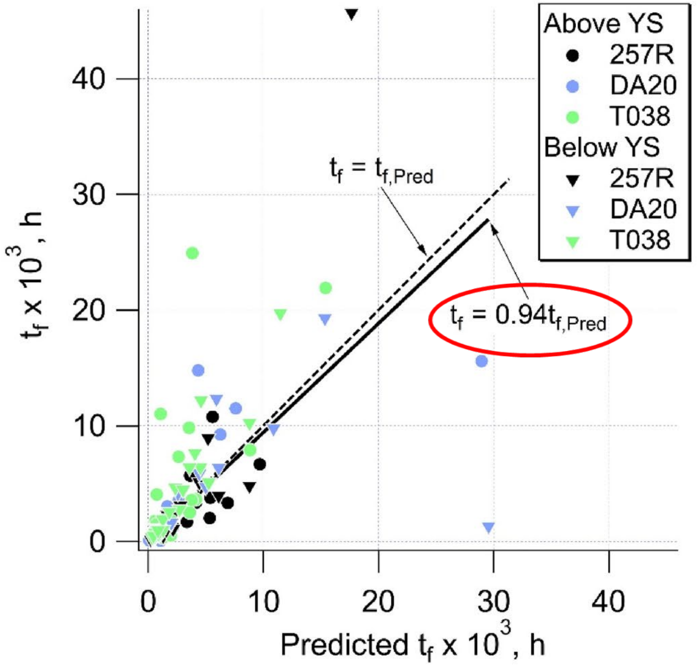


Split-region fit (above YS: dashed line, below YS: solid line)

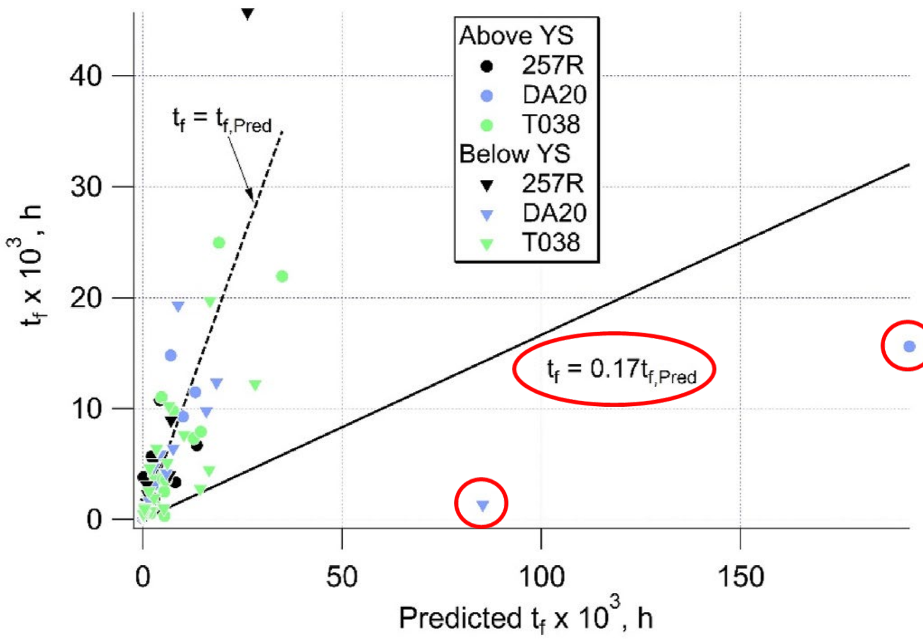
- Single-region analysis applied to all data was not suitable in Wilshire analysis
- Split-region analysis for stress above YS and below YS yielded better fitting of experimental data in Wilshire analysis
- Q_c^* in split-region analysis corresponded to reasonable creep mechanism
  - Above YS: Q_c^*=334 kJ/mol, hot deformation
  - Below YS: Q_c^*=203 kJ/mol, lattice diffusion and grain boundary diffusion

Effect of Experimental Data Scatter on Creep Model Accuracy

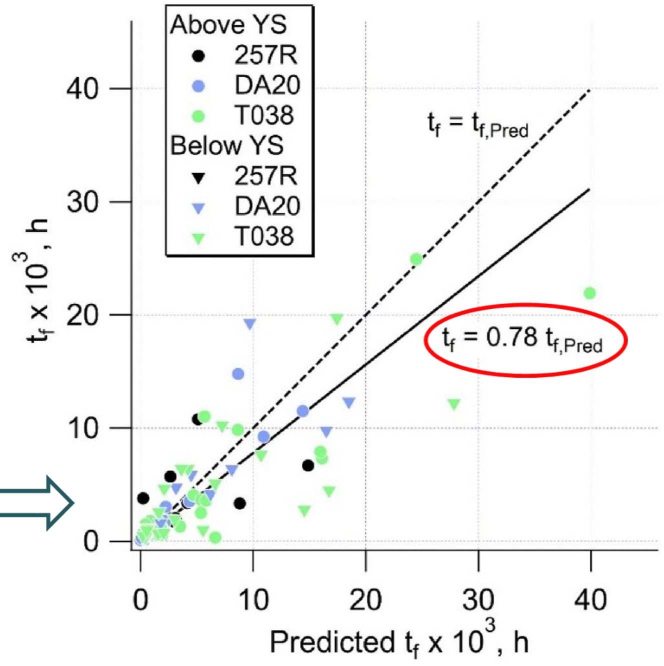
- Larger data scatter in cast CF8C-Plus compared with wrought Inconel 740H [1] and Haynes 282 [2]
  - Due to greater heterogeneity of cast microstructures, including the presence of casting defects.
- The LMP model appears more robust than the Wilshire model for cases when there is greater scatter of the experimental creep lifetime



Experimental versus predicted rupture time for split region LMP analysis of all data



Experimental versus predicted rupture time for split region Wilshire analysis of all data



Experimental versus predicted rupture time for split region Wilshire analysis of all data without two outline data

1. M. Render et al., Mater. Trans. 52 (6) (2021) 2601-2612
2. M.L. Santella et al., Mater. Sci. Eng. A 838 (2022) 142785

Oak Ridge National Laboratory

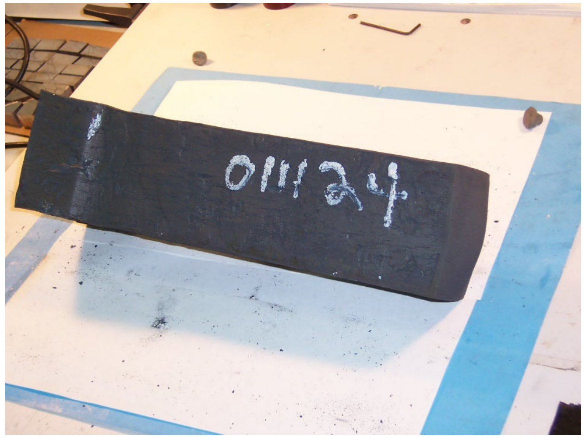
EPRI
ELECTRIC POWER
RESEARCH INSTITUTE

Wrought CF8C-Plus Development and ASME Code Case Application

EPRI and ORNL are leading product development and commercial-scale demonstration of wrought CF8C-Plus

2009

Carpenter produced
188 kg VIM heat
Open-die forged 5:1 and 12:1



2011

Special Metals produced
two 472 kg VIM heats
Extruded to pipes >130 mmØ

2016

Carpenter produced 4th 2800 kg
powder metallurgy heat
Wyman-Gordon extruded 400 mmØ pipe

2017

Detailed SEM and TEM
microscopy of precipitates
following mechanical testing

2020

This project with ORNL
kicked off to produce
5th heat and ASME code case

2021

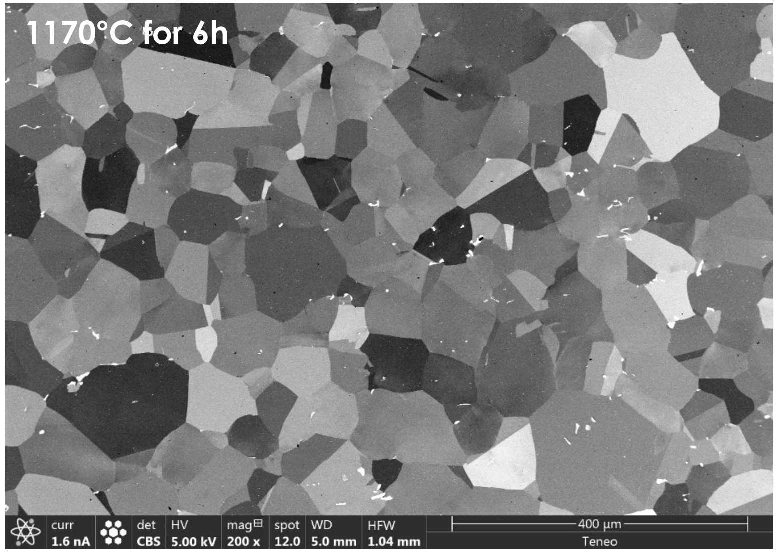
Produced large ESR heat
using optimized chemistry
process mapping and production

Manufacturing Studies of a High-Temperature Stainless Steel (2017)
EPRI Report 3002009212

Development of 5th Heat (589832)

wt%	Cr	Ni	Mn	Nb	C	N	Cu	W	Si
Min	19.5	12.5	3.7	0.6	0.05	0.23			0.5
Max	20.5	13.5	4.5	0.8	0.1	0.28	<0.3	<0.01	1
589832	19.9	12.8	4.0	0.7	0.08	0.26	0.05	0.02	0.9

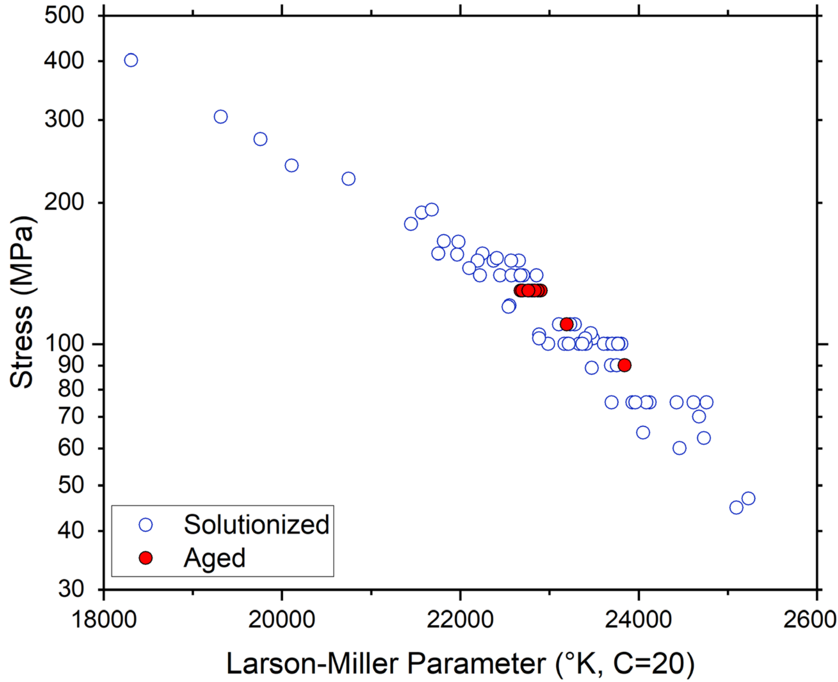
1. Alloy design and chemistry targets
    - Computational thermodynamic assessment of carbide and nitride stability
    - TEM/STEM work on precipitates from several heats
    - Optimized chemistry targets from cast formulation
2. Ingot production at Carpenter
    - EAF+AOD, ESR, 2 ingots ≈ 12,600kg
3. Gleeble-based study for evaluating optimal solutionizing heat treatment cycle and modeling high temperature extrusion/strain
4. Extrusion at Wyman Gordon
    - 3,500 kg segment to produce **a pipe with 900 mm length X 400 mm OD X 44 mm wall thickness**
5. Microstructure evaluation and heat treatment optimization…



Heat Treatment Optimization on 5th Wrought Heat

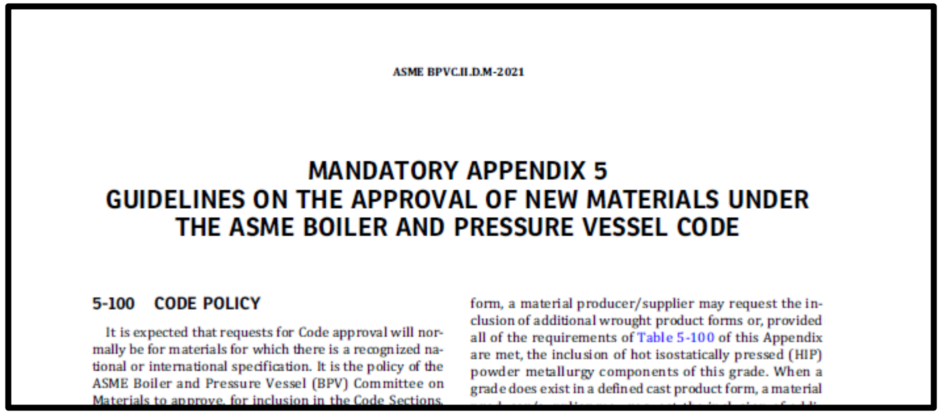
- Hardness measured for different heat treatment conditions
  - Average **solutionized** condition: 181 HV
  - Average **aged** condition: 191 HV
- Short-term creep behavior
  - Tested at 750°C, rupture lives ~200-2000hr
  - Aged conditions fell within scatter of solutionized data
- **Relatively insignificant influence of heat treatment on final properties across tested conditions**
- Final heat treatment for the remainder of 5th heat: solutionized at 1170°C for 2hr performed at Wyman-Gordon

ID	Average (HV)	Solution Temp (°C)	Solution Time (h)	Ageing Temp (°C)	Ageing Time (h)
AR0	176	As-Received			
AR1	182	As-Received		750	8
A1	177	1220	2		
A2	190	1220	2	750	8
A3	183	1220	6		
A4	192	1220	6	750	8
B0	175	1170	2	Air cool	
B1	184	1170	2		
B2	188	1170	2	750	8
B3	185	1170	6		
C1	177	1120	2		
C2	193	1120	2	750	8
C3	182	1120	6		
C4	191	1120	6	750	8



Development of Data Package for ASME Code Case

- A large amount of mechanical property data is required for ASME to qualify a new material
- Requirements outlined in ASME Section II Mandatory Appendix 5 for a Section I Code Case
- Creep rupture testing is by far the most burdensome:
  - **Multiple heats** (a minimum of 3 heats)
  - **Multiple temperatures**
    - Spanning time-dependent regime up to max. use temperature +50°C
    - 25-50°C temperature intervals
  - **4x tests per heat at each temperature with rupture lives spanning 500h to 10kh+**



ASME BPVC.II.D.M-2021

MANDATORY APPENDIX 5
GUIDELINES ON THE APPROVAL OF NEW MATERIALS UNDER THE ASME BOILER AND PRESSURE VESSEL CODE

5-100 CODE POLICY

It is expected that requests for Code approval will normally be for materials for which there is a recognized national or international specification. It is the policy of the ASME Boiler and Pressure Vessel (BPV) Committee on Materials to approve, for inclusion in the Code Sections,

form, a material producer/supplier may request the inclusion of additional wrought product forms or, provided all of the requirements of Table 5-100 of this Appendix are met, the inclusion of hot isostatically pressed (HIP) powder metallurgy components of this grade. When a grade does exist in a defined cast product form, a material

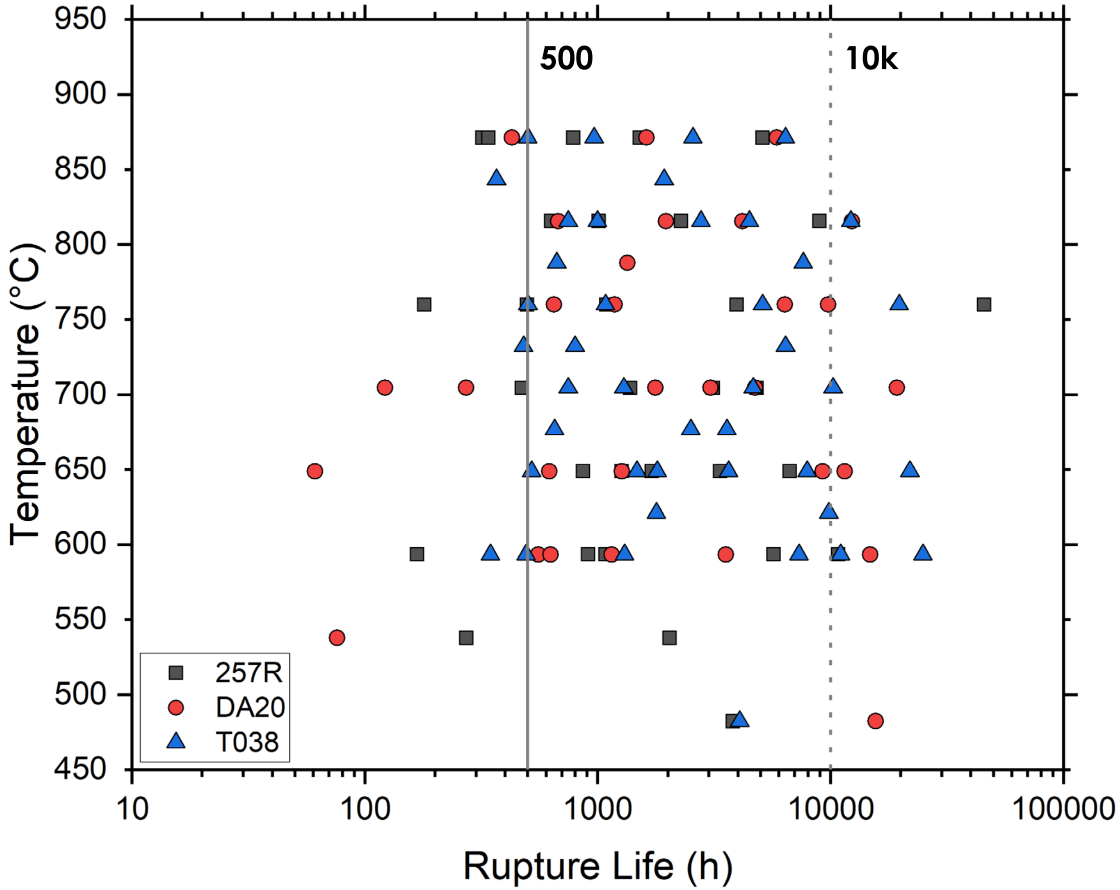
Summary of Wrought CF8C-Plus Heats for ASME Qualification

Form	Heat#	Producer	Reduction Ratio	Heat Treatment Condition	Final Dimensions	ASTM Grain Size
Forging	011124*	Carpenter	5:1	Solutionize 2200°F / WQ	5:1, 3.5" x 2.75" x 10" (~28lb slab)	6
Forging	011124*	Carpenter	12:1	Solutionize 2200°F / WQ	12:1. 3.5" x 1.25" x 20" (~28lb slab)	7
Extrusion	HF8726C	PCC Energy Group	5.3:1	Solutionize 2200°F / WQ	5.3:1. 6" OD, 0.75" WT (~1000lb smls pipe)	7
Extrusion	HF8728	PCC Energy Group	9.4:1	Solutionize 2200°F / WQ	9.4:1. 5.25" OD, 0.5" WT (~1000lb smls pipe)	7
Extrusion	589832	Carpenter + Wyman Gordon	9:1	Solutionize at 2138°F / WQ	9:1. 16" OD, 1.5" WT (~7700lb smls pipe)	~5 (pending further analysis)

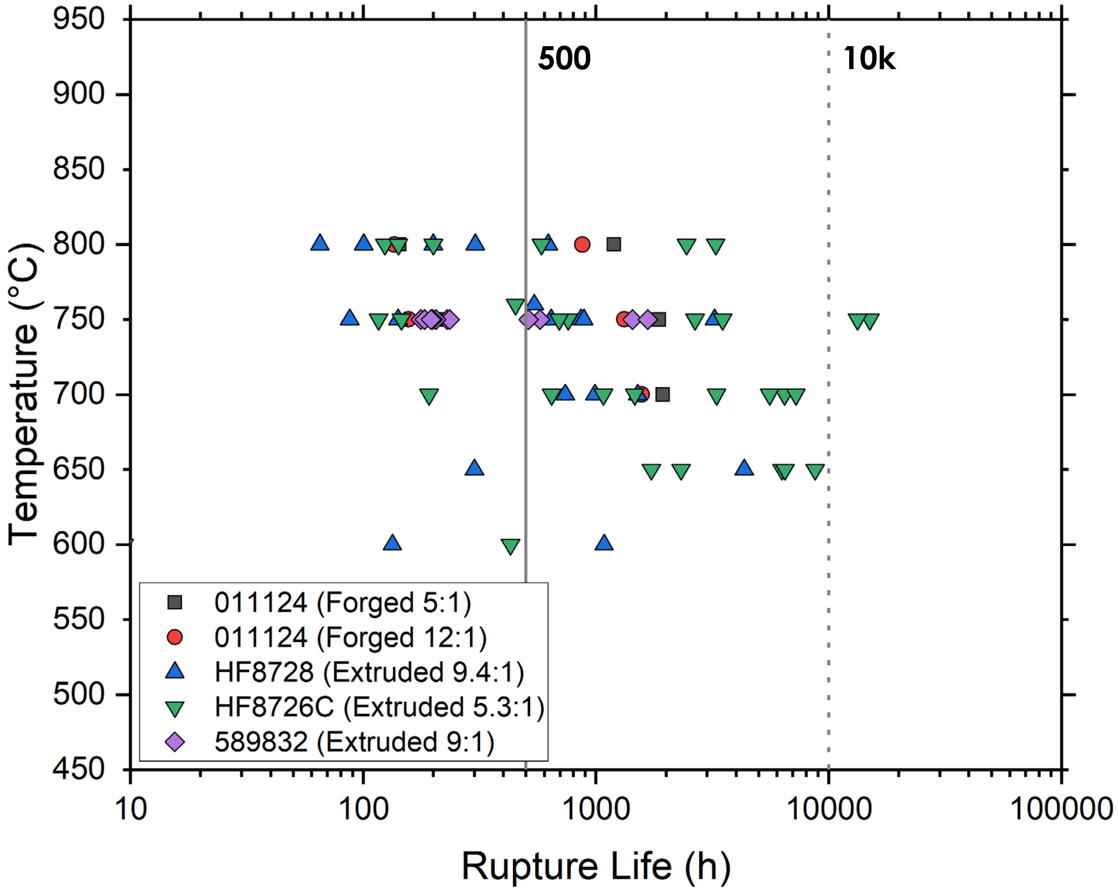
*Used for supplemental data only

Comparison of CF8C-Plus Creep Rupture Databases (Cast vs. Wrought)

Creep rupture data (ASME cast CC)



Creep rupture data (wrought) as of Dec. 2022



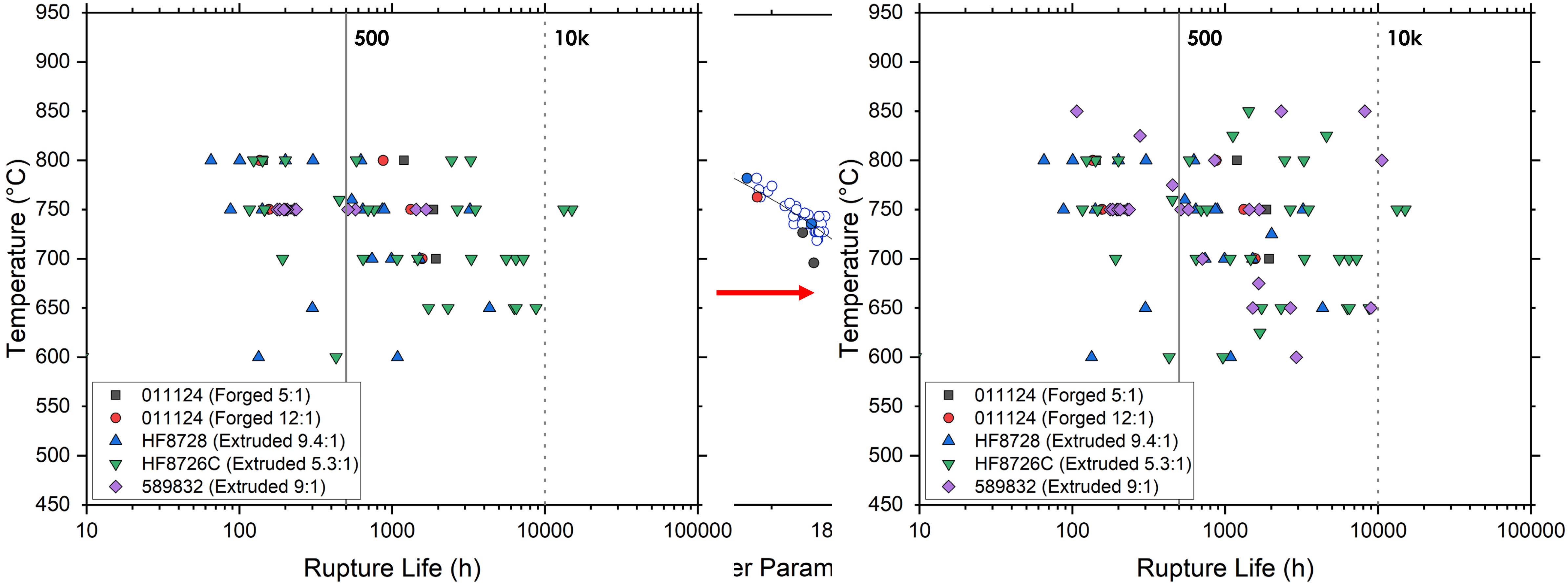
Data need for wrought CF8C-Plus: wider temperature range and longer times at all temperatures

Current Wrought CF8C-Plus Creep Rupture Database

Creep rupture data (wrought) as of Dec. 2022

Ruptured and active creep tests for wrought CF8C-Plus

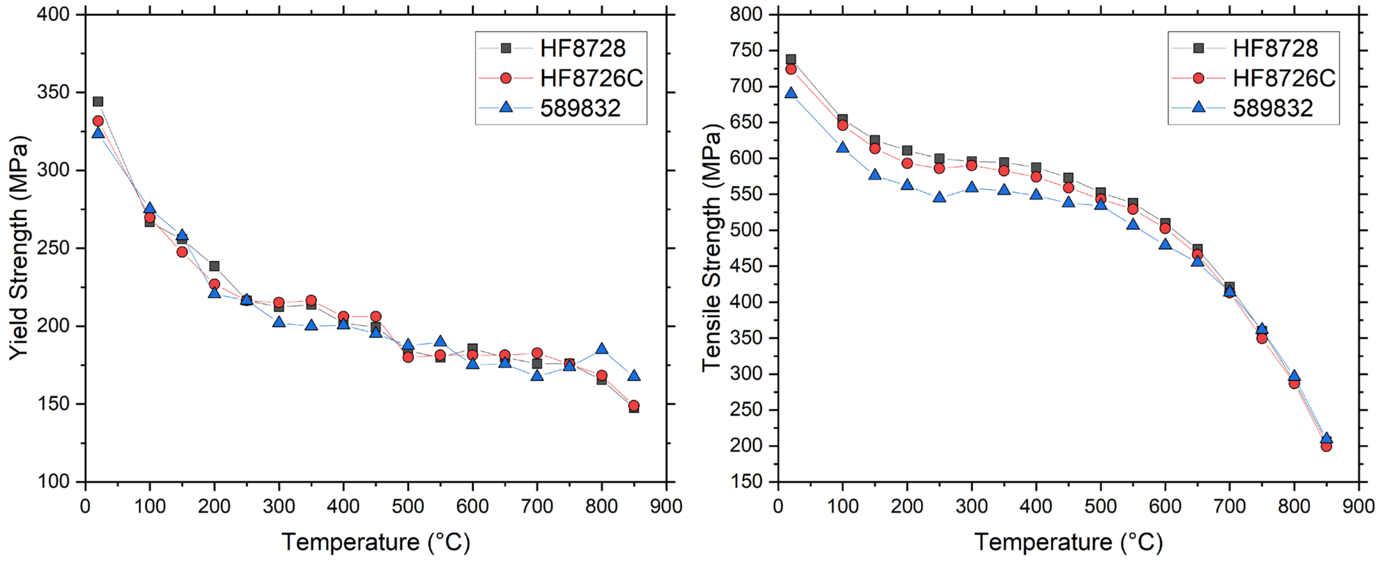
Creep rupture data (wrought) as of Apr. 2024



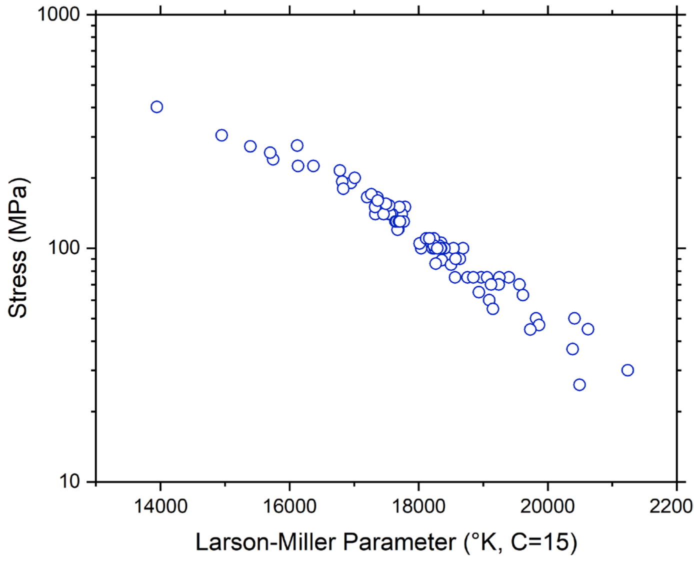
Approximately 115,000 hours of creep data collected under this program since December 2022

Preliminary ASME Section II Allowable Stress Analysis for Wrought CF8C-Plus

Time-independent (tensile) data



Time-dependent (creep) data



Existing test data can be used to develop early estimates for ASME allowable stresses using criteria from ASME Section II as a function of temperature

Yield Strength	Tensile Strength	Stress Rupture
$\frac{2 * S_Y}{3}$	$\frac{S_T}{3.5}$	$F_{avg} * S_{Ravg}$
$\frac{2 * S_Y * R_Y}{3}$	$\frac{1.1 * S_T * R_T}{3.5}$	$0.8 * S_{Rmin}$
$0.9 * S_Y * R_Y$		

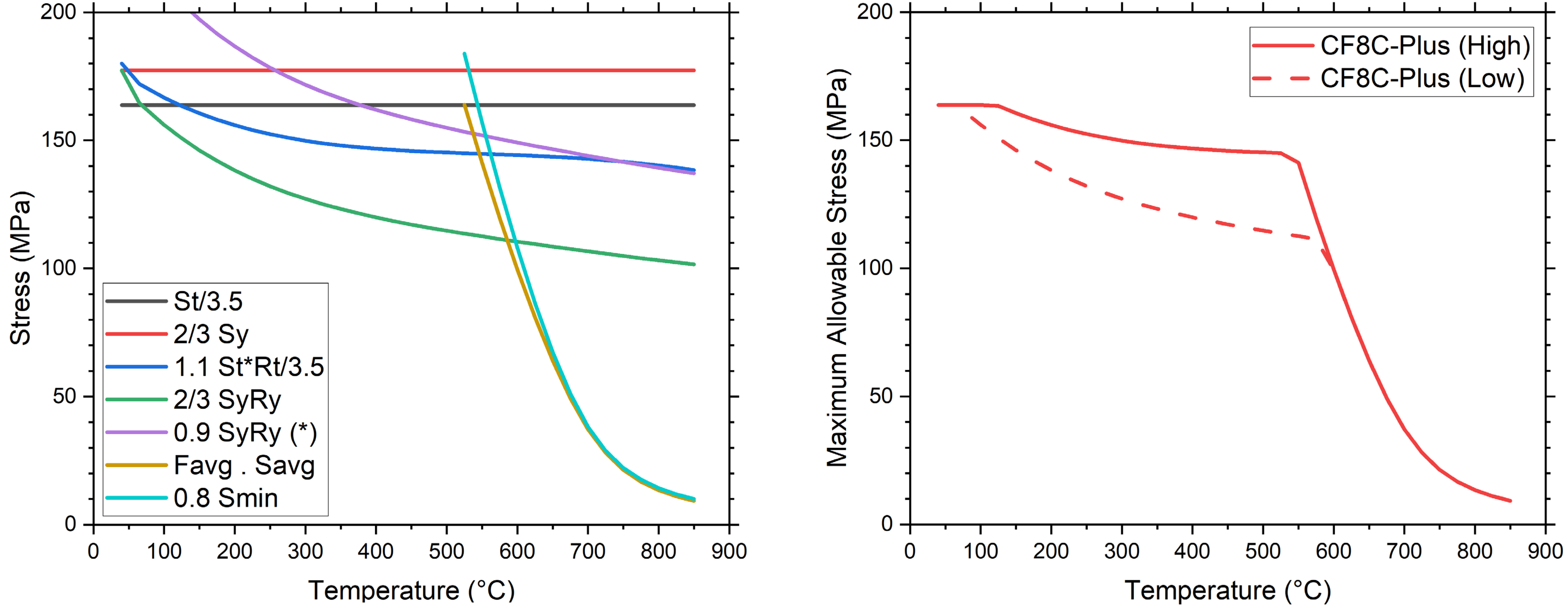
Assumptions:
S_Y = 0.8*AVG RT YS = 266 MPa
S_T = 0.8*AVG RT TS = 573 MPa

Preliminary ASME Section II Allowable Stress Analysis for Wrought CF8C-Plus

Wrought CF8C-Plus Calculated Allowable Stress Criteria

Minimum across temperature range

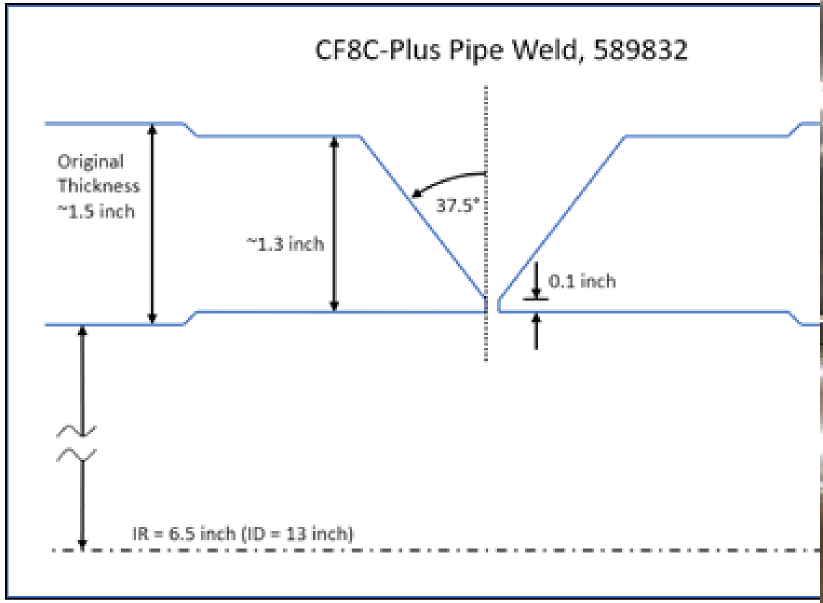
Preliminary Allowable Stress Values

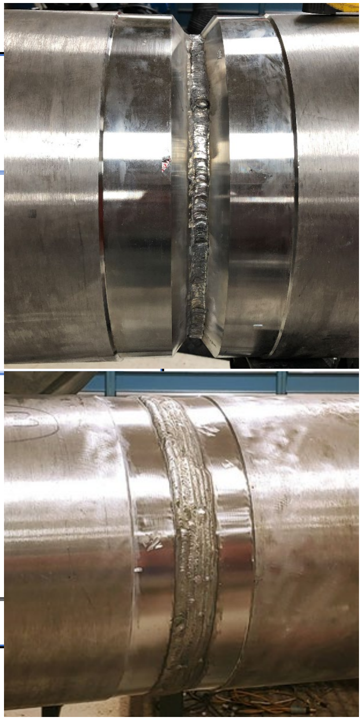


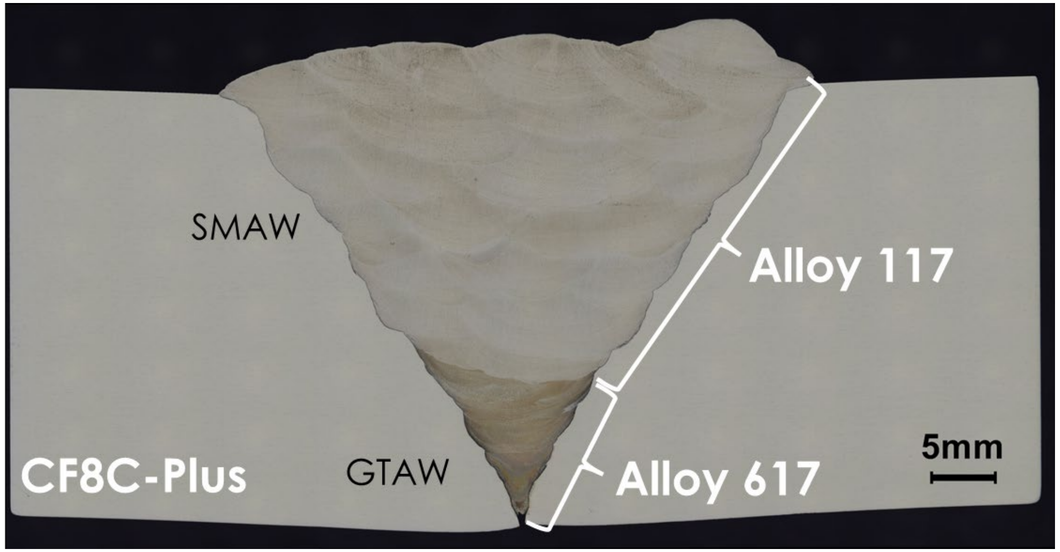
Note: these results are preliminary and the allowable stress calculations are subject to change as additional creep data becomes available

Wrought CF8C-Plus Pipe Weldment

- Pipe weld produced to support ASME code case development
- Base metal: 5th Heat (589832)
- Followed identical strategy and materials that were used for Cast CF8C-Plus code case development
- Partial GTAW / SMAW - common practice for qualifying multiple processes
- GTAW (617, ERNiCrCoMo-1)
- SMAW (117, ENiCrCoMo-1)







CF8C-Plus Pipe Weld Testing for ASME Data Package

- Cross-weld tensile testing
- Side bend testing
- Cross-weld creep testing

Establish that a quality weld was produced (ASME Section IX)

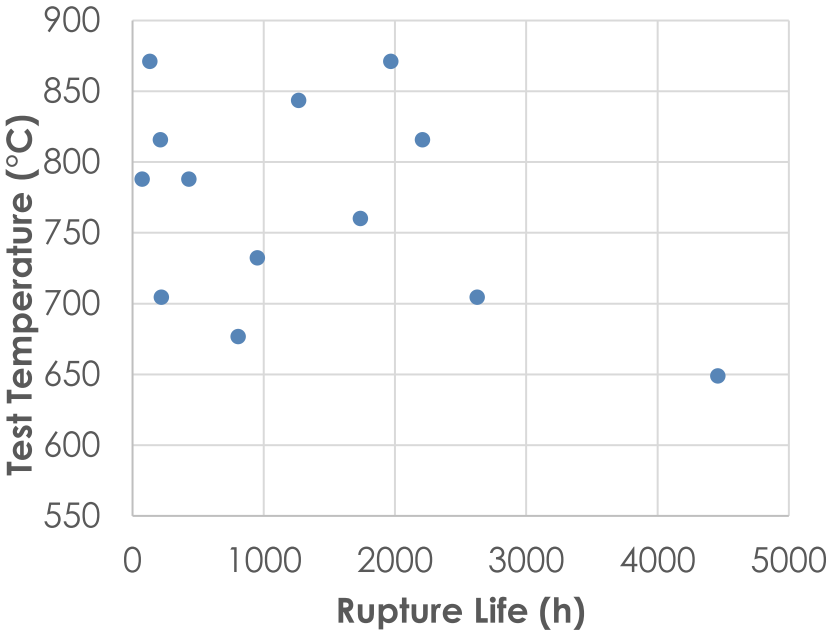
Determine weld strength reduction factors

Cross-weld tensile

Sample	Tensile Strength (MPa)	Failure Mode	Failure Location
GTAW-1	668	Ductile	Base Metal
GTAW-2	668	Ductile	Base Metal
SMAW-1	672	Ductile	Weld Metal
SMAW-2	689	Ductile	Base Metal

Side bend testing

Cross-weld creep testing matrix follows cast code case



Characterization underway to understand unexpected side bend failure

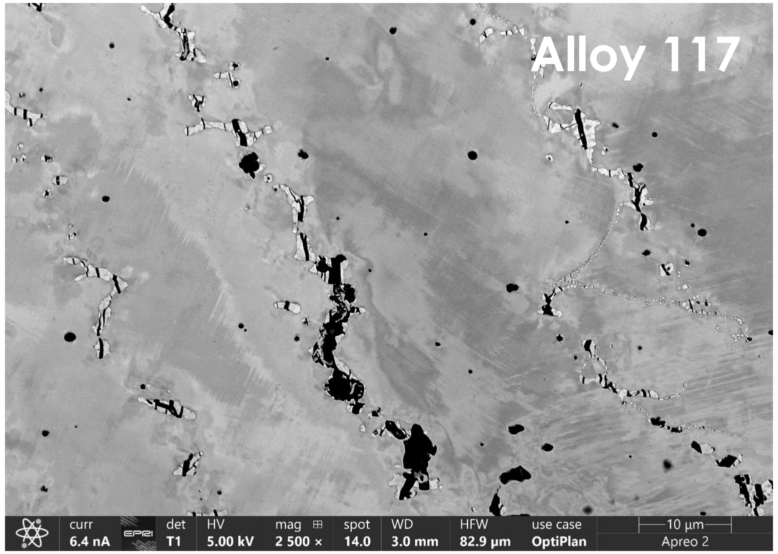
CF8C-Plus Pipe Weld Characterization

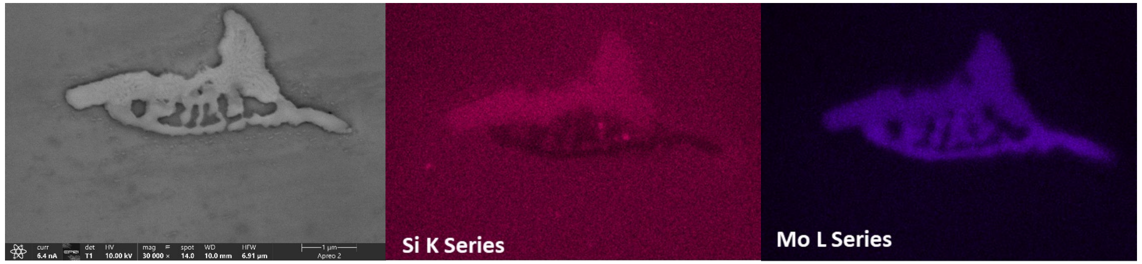
- Two primary contributors to failed weld bend specimens:
  - Lack-of-fusion at the weld root
  - Limited ductility of Alloy 117 shielded metal arc weld
- Lack-of-fusion associated with improper fit-up, but does not limit value of weld for cross-weld mechanical testing
- Alloy 117 ductility issue associated with eutectic films along solidification grain (and subgrain) boundaries (Si-rich and Mo-rich)
  - Unclear if this can be mitigated by selecting a different heat of Alloy 117 or if only Alloy 617 should be used moving forward

Failed weld bend specimens

Lack-of-fusion

Mechanical failure in A117





Summary and Plans for FY24/FY25

Completed to-date

- Material development and production of multiple heats
- Room temperature and elevated temperature tensile testing
- Preliminary ASME allowable stress analysis
- Macrohardness
- Chemical analysis
- Weld production for qualification
- Cross-weld tensile testing

Future Plans

- Continue base metal creep testing for ASME data package
- Weld characterization to understand source of bend failures
- Cross-weld creep testing to establish strength reduction factors
- Physical property measurements
- Draft ASME Section I CC

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