

Code Qualification of Alloy 709 for High Temperature Reactor Structural Applications

September 2022

Ting-Leung Sham





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Ting-Leung Sham

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Idaho National Laboratory Idaho Falls, Idaho 83415

http://www.inl.gov

Prepared for the U.S. Department of Energy Under DOE Idaho Operations Office Contract DE-AC07-05ID14517 Ting-Leung (Sam) Sham TingLeung.Sham@inl.gov

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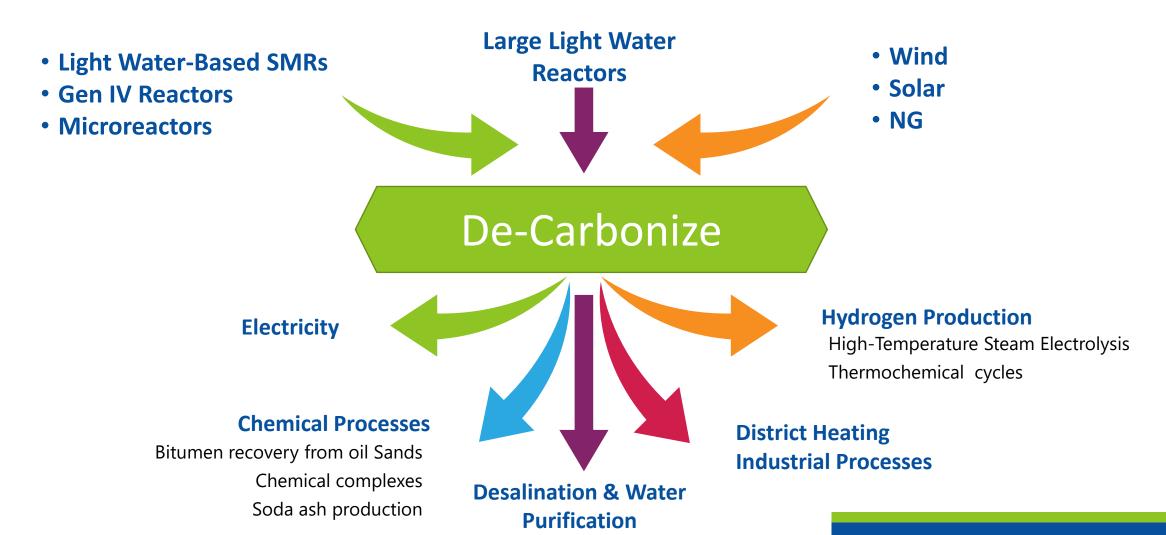
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Alloy 709 Development Team

- DOE-NE Advanced Reactor Technologies (ART) Program, Advanced Materials, current and former Lab staff
 - Argonne National Laboratory
 - Ken Natesan, Meimei Li, Xuan Zhang, Yiren Chen, Mark Messner
 - Idaho National Laboratory
 - Richard Wright, Laura Carroll, Tom Lillo, Mike McMurtrey, Ryann Bass, Sam Sham
 - Oak Ridge National Laboratory
 - Yanli Wang, Zhili Feng, Yuki Yamamoto, Grace Burke

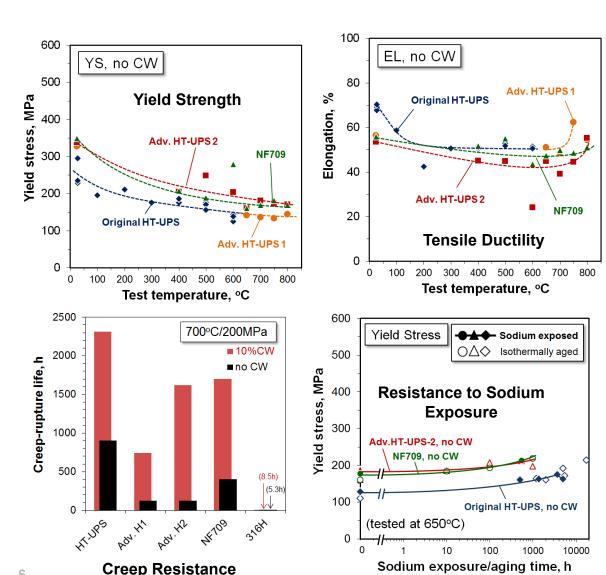
Nuclear Beyond Electricity – Advanced Reactors THE FUTURE

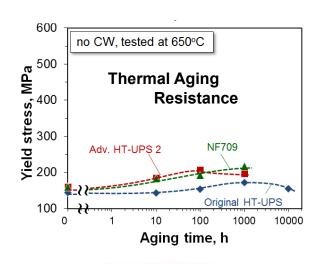


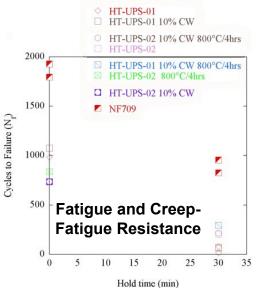
Advanced Materials Development & Qualification

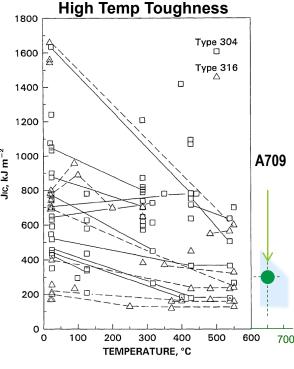
2008 Established Alloy Development Priority List			2016 - Present Code qualify Alloy 709		
 Considered a large class of structural materials for further development Involved 5 U.S. national Laboratories and 5 U.S. universities Considered experience from Fusion, Gen IV, Space Reactor, and development activities in Fossil Energy Established alloy development priority list: Ferritic-Martensitic steels Grade 92 (NF616) Grade 92 with thermomechanical treatment (TMT) Austenitic stainless steels HT-UPS NF-709 	 Established comprehensive downselection metrics Considered tensile properties, creep, creep-fatigue, toughness, weldability, thermal aging, sodium compatibility, mechanical and TMT processes Integrated R&D activities by DOE Labs Oak Ridge National Laboratory Argonne National Laboratory Idaho National Laboratory Materials considered include Optimized-Gr92, Ta/Ti/V-modified 9Cr, Gr92, Gr91 (baseline material) HT-UPS (Fe-14Cr-16Ni), Modified HT-UPS, A709 (Fe-22Cr-25Ni), 316H (baseline material) Based on overall performance w/ comprehensive metrics (and accelerated test data), Optimized-Gr92 with TMT and A709 were downselected for further assessment	 Further optimize mechanical and TMT processes Procure larger heats Validate performance gains Longer-term testing of base metals and weldments Recommended Alloy 709 for ASME Code qualification Development of roadmap for staged qualification of Alloy 709 in a series of ASME nuclear code cases 	 Established mechanical properties testing capabilities at the Labs Fabrication scale up from lab to commercial scale Procured commercial heat per industry melt practice Initiated code case testing at ANL, INL, ORNL collaboratively 		

Austenitic Alloy Down-Selection (HT-UPS, A709)





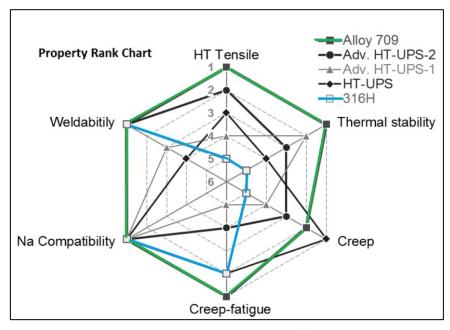






Alloy 709 for ASME Code Qualification

- 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
- 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
- Higher strengths permit structural components to withstand higher cyclic and sustained loading, leading to higher safety margins, and the prospect of eliminating costly add-on hardware instituted in past designs, and other design innovations and simplifications (Sham and Jetter, PVP2017-65102)



Property rank chart for austenitic alloys

Codes and Standards for High Temperature Reactor Structural Design

- ASME, Section III, Division 5 is a component code
- It is organized by Code Classes:
 - Class A and Class B for metallic coolant boundary components
 - Class SM for metallic core support structures
 - Class SN for nonmetallic components
- The Code Classes allow a choice of rules that provide a reasonable assurance of structural integrity and quality commensurate with the relative importance assigned to the individual components of the advanced reactor plant

Materials Data Requirements for Section III, Division 5 Components

Component Class (A or B)

Structural Failure Modes

Design Parameters

Required Test Data

Design Parameters Required to Address Failure Modes for Class A Components

Design Parameters	Required Test Data			
Allowable Stresses				
• S_m : based on yield and ultimate strengths at temperature	Tensile data at temperature (time-independent)			
 <i>S_t</i>: based on time to 1% total strain, time to onset of tertiary creep, time to rupture <i>S_r</i>: based on stress to rupture 	Creep rupture data with full creep curves (time-dependent)			
 S_{mt}: lesser of (S_m, S_t) S₀: lesser of (S, S_{mt}@300,000h) 	Derived design parameters			
R: Stress rupture factor - based on rupture strengths of base metal and weldment	Stress rupture data from base metal and weldment (time dependent)			
Thermal aging factors on yield and ultimate	Tensile data of aged material (time-dependent)			
Isochronous stress-strain curves constructed based on creep tests	Tensile stress-strain curves (time-independent), and creep strain data up to 3% (timedependent)			

Design Parameters	Required Test Data				
Fatigue design curves	Strain-controlled continuous cycling tests				
Creep-fatigue interaction diagram	Strain-controlled cyclic tests with hold times				
EPP design parameters	Two-bar and SMT tests; cyclic stress-strain curves				
Inelastic material model parameters	Test data for other design parameters; and strain rate change and thermomechanical cycling				
Huddleston effective stress parameters	Multiaxial creep rupture data				
External pressure charts	Tensile stress-strain curves (time-independent)				
Time-temperature limits for external pressure charts	Isochronous strain-strain curves				

Some design parameters are for setting design limits; some are for providing behavioral trends to support design evaluations

Structural Materials Qualified for ASME Section III, Division 5 Class A Components

- Type 304 and 316 stainless steels
- Alloy 800H
- 2.25Cr-1Mo (Solution Annealed, Grade 22)
- 9Cr-1Mo-V (Grade 91)
- Alloy 617
- SA 508 Grade 3, Class 1 forgings and SA 533 Type B, Class 1 plates qualified for short-term elevated temperature excursions

Required Testing to Support Design Parameters Development for Class A Components

- Refer to Section II Materials and Section III, Division 5
 "Nonmandatory Appendix HBB-Y, Guidelines for
 design data needs for new materials"
- Required Tests
 - Tensile, creep rupture, fatigue, creep-fatigue, constitutive, multiaxial creep rupture, EPP
- Time dependent data (creep rupture) dominates the test times for data generation
 - Allow limited extrapolation of time for creep properties
 - Well-behaved alloys such as stainless steels and nickel alloys may extrapolate in time of no more that a factor of 5 to reach intended life
 - Metastable alloys such as the creep strength enhanced ferritic/martensitic steels may extrapolate with a factor of 3
 - Require metallurgical justification for 3 < extrapolation factor ≤ 5

Design Life (hours)	Minimum Time to Complete Creep Rupture Testing (years)						
	Solid Solution Alloys	Ferritic- Martensitic Steels					
100,000	2.3	3.8					
300,000	6.8	11.4					
500,000	11.4	19.0					

A long and arduous process!

A "Staged" Qualification Approach for Alloy 709

Time from initiation of long-term testing (years)

1.0 7.0 8.0 10.5 11.5 0.5 1.5 2.0 3.0 4.0 9.0 10 Class B CC 100,000 hr CC Concept Design Creep tests Guide Creep tests for 300,000 hr CC

Creep tests for 500,000 hr CC

(Determination of mechanisms giving rise to time dependent properties through simulation validated by experiment could allow larger extrapolation factors)

Other mechanical properties testing common to all CCs

A four-year testing program, without resource constraints, would generate data package to support:

- Conceptual design
 - Conceptual Design Guide for 500,000-hour lifetime
- Preliminary design
 - 100,000-hour Class A code case
 - Class B material code case

Additional creep data at 7-year mark from start:

- Final design
 - 300,000-hour Class A code case

Additional creep data at 12-year mark from start:

- Nth-of-a-kind
 - 500,000-hour Class A code case

Tests initiated at the

History of 20Cr-25Ni Austenitic Alloys





- 20Cr25Ni/Nb stainless steel (1950s)
- Used as fuel cladding in British AGR fleet (since 1962)
- Temperature 550°C to 850°C
- 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

	С	Mn	Si	Р	S	Cr	Ni	Nb	В	Мо	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 Fabrication Scale-up

• Small heats (~ 400 lb) from specialty vendors were procured to support intermediate term testing



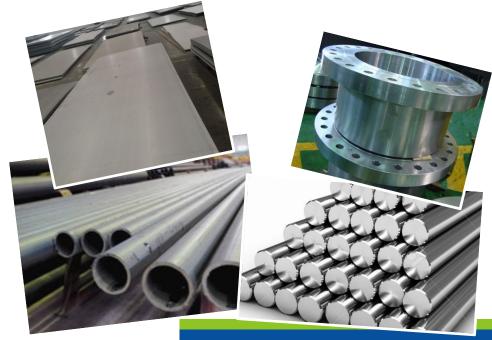


Images from http://www.electralloy.com/

are important factors in deploying Alloy 709 as construction material for SFRs

Available industrial fabrication processes and infrastructure

• Different product forms are required for component constructions (plate, pipe, bar, tubing, forging)



Code Case Requires a Minimum of Three Commercial Heats of Alloy 709 for Qualification

- First commercial heat received in FY17
 - Totaling about 45,000 lbs
 - Nine process conditions
 - ASTM grain size range 5-8
 - Down-selected ESR-1150SA melt practice

Photograph of first commercial heat A709 plates in as-rolled condition at vendor location

- Second commercial heat received in FY21
 - Totaling about 40,000 lbs
 - ESR melt practice
 - Hot rolled into 1.75" and 2" plate product form
 - ASTM grain size range 4-7

Photograph of second commercial heat in solution-annealed condition at INL

- Third commercial heat received in FY22
 - Totaling about 38,000 lbs
 - ESR melt practice
 - Hot rolled into plate product form
 - Characterization in progress

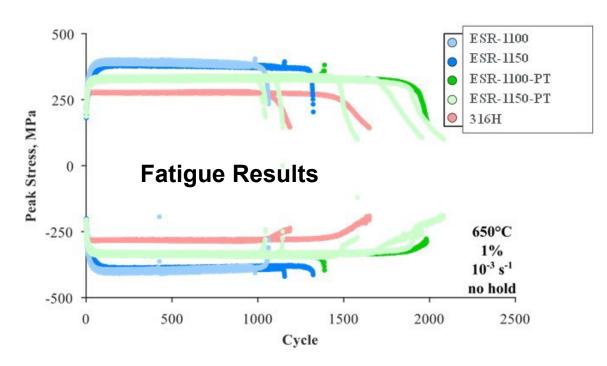


Photograph of third commercial heat in solution-annealed condition at ORNL

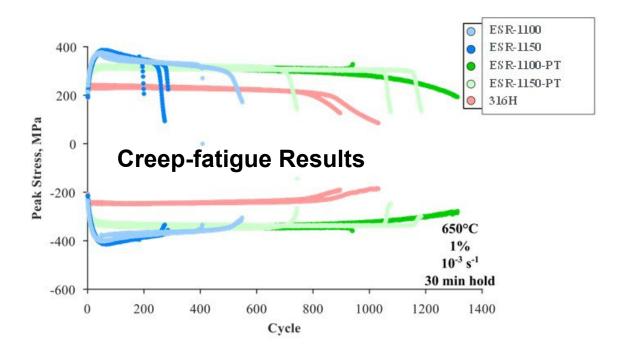
Development of a Precipitation Treatment Protocol

- Alloy 709 derived its enhanced creep strength from the precipitation of nanosized carbonitride M(CN) particles on dislocations in a stable, fine dispersion as promoted by the time-at-temperature and the stress conditions during reactor plant operations
- But there is concern that formation of these precipitates are not favored for lower temperature (less than 525C) reactor operations due to slow kinetics, and other detrimental phases may form instead
- Based on computational materials simulations, two precipitation treatments (PTs) were developed
- Microstructure analyses of PT samples showed the 775C/10 hr precipitation treatment gives the desirable microstructure

Alloy 709 Cyclic Data at 650C

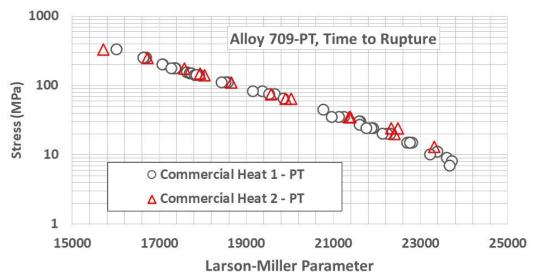


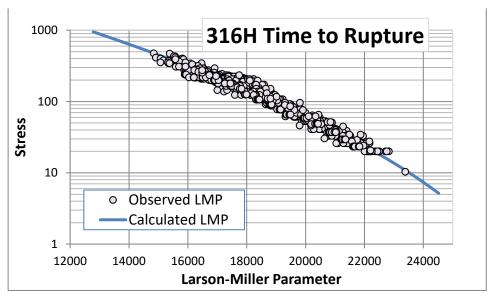
650C, Fully-Reversed 1% Strain Range, No Hold Time

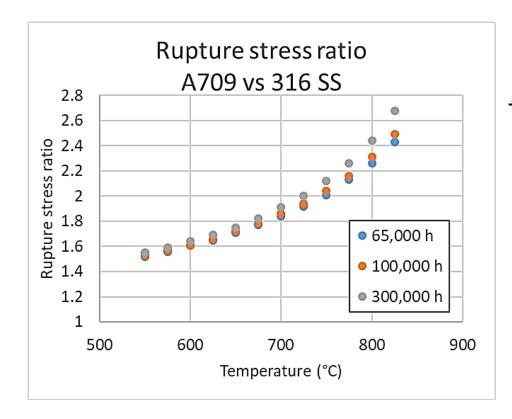


650C, Fully-Reversed 1% Strain Range, 30-Minute Hold Time

Creep Rupture Data To Date



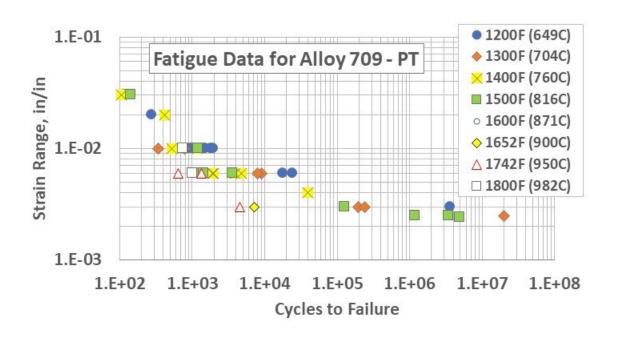


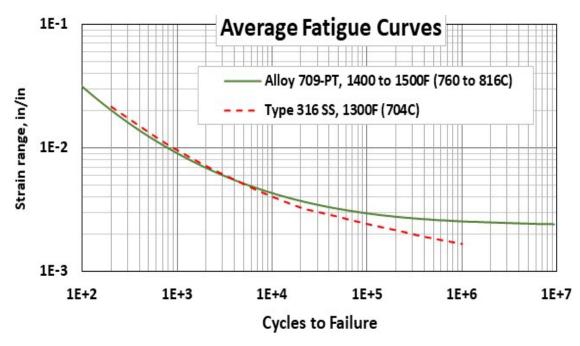


A709-PT 316SS

The trends show that the creep rupture strengths of precipitation-treated Alloy 709 are significantly higher than those of Type 316 stainless steel

Fatigue Data To Date





- The initial set of fatigue data were generated to establish the general trends of the fatigue resistance of Alloy 709-PT
- The fatigue curve generated using 1400-1500F data for Alloy 709-PT lies above the 1300F curve for Type 316 stainless steel, showing that Alloy 709-PT has a higher fatigue resistance over Type 316 stainless steel

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 as Alloy 617; but has significant advantage over Type 316 stainless steel
- Fatigue and creep-fatigue performance of Alloy 709-PT is excellent
- Ongoing data package generation effort sponsored by DOE-NE to codify Alloy 709-PT in ASME Boiler and Pressure Vessel Code, Section III, Division 5 for high temperature reactor applications
- Plan to complete data package for first Division 5 Code Case by end of 2024

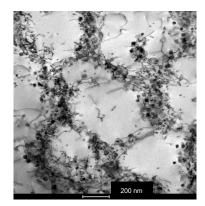
Thank You!

Backup Slide

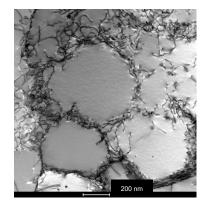
Role of Solutes in Mechanical Behavior of Alloy 709

- Compared to solution annealing, aging reduces the amount of nitride or carbo-nitride forming elements in solution through precipitation of MX or Z phase prior to deformation
- This aging treatment has been shown to restrict the precipitation of fine MX or Z phases on dislocation substructures during subsequent cyclic testing
- The result is that there is less cyclic hardening compared to testing solution annealed material and the cycles to failure in creep-fatigue is significantly increased
- The aging treatment does lead to a modest reduction in the creep rupture strength, but it gives a much more well-balanced set of design properties for advanced reactor structural applications

Microstruture After Fatigue at 650°C

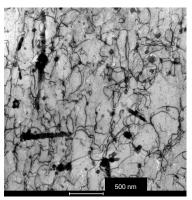


A709-SA: Cuboidal M₂₃C₆ distributed on dislocation cell walls

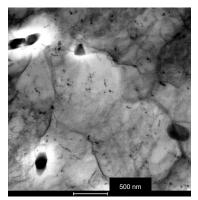


A709-PT (775°C for 10 hours): no precipitation on dislocation cell walls

Microstruture After Creep-fatigue at 650°C



A709-SA: coarse $M_{23}C_6$ and fine Z-phase



A709-PT (775°C for 10 hours): coarse complex precipitates and fine Z-phase