U.S. DEPARTMENT OF ENERGY | Office of NUCLEAR ENERGY

A709 Procurement and ASTM Specification Status

Joint ART Materials/AMMT Program Review

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The Alloy 709 Team

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INL Work Package

- **RD-23IN040402**
  - A709 Development - INL

Introduction

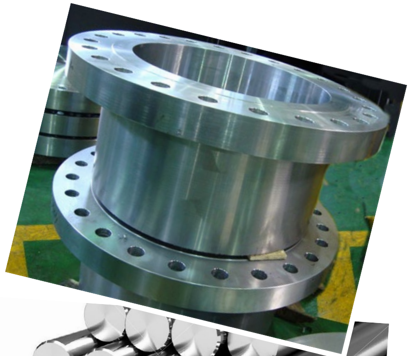
- Alloy 709 ASME Code qualification requires property characterization for three commercial scale heats
- One heat was obtained from G. O. Carlson in FY 17
- Two additional heats were obtained from ATI Flat Rolled Products in FY 21 and FY 22 respectively
- A pre-requisite for ASME qualification is to have ASTM standards in place for the desired product form

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/

- Available industrial fabrication processes and infrastructure are important factors in deploying Alloy 709 as construction material for SFRs



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

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

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

ATI Flat Rolled Product Heats

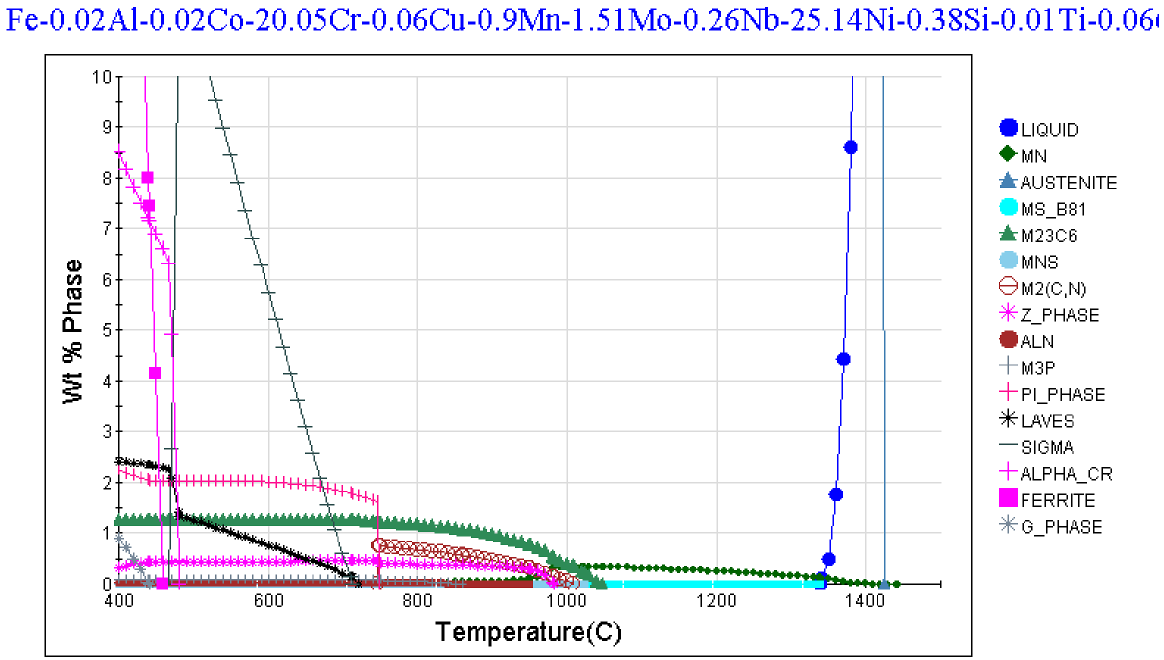
- Two heats approximately 40,000 pounds each melted using electro-slag remelt process
- Plates rolled to 1.75- and 2.0-inch thickness
- Solution annealed above 1150˚C – specific temperature determined based on experience of vendor
- Grain size between ASTM grain sizes of 4-7

Development of a Precipitation Treatment Protocol

- Alloy 709 derived its enhanced creep strength from the precipitation of nano-sized 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 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
  - 900C/10 hr and 775C/10 hr
- Microstructure analyses of PT samples showed the 775C/10 hr PT gives the desirable microstructure
- The PT condition of 775C/10 hr was selected

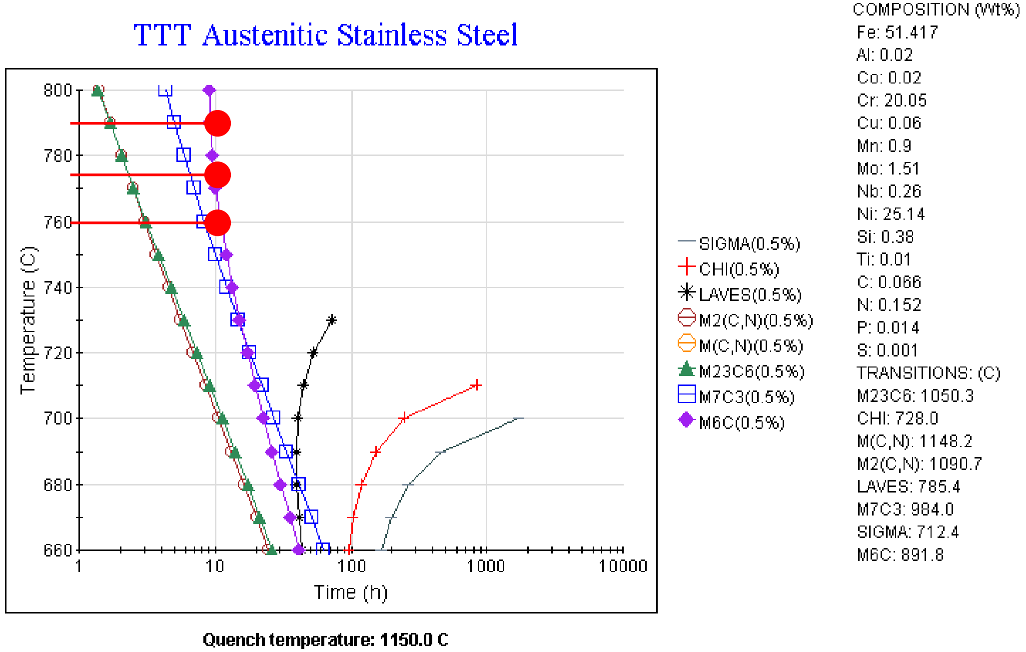
Computational Materials Simulations

Carlson Heat



Equilibrium Phase Diagram

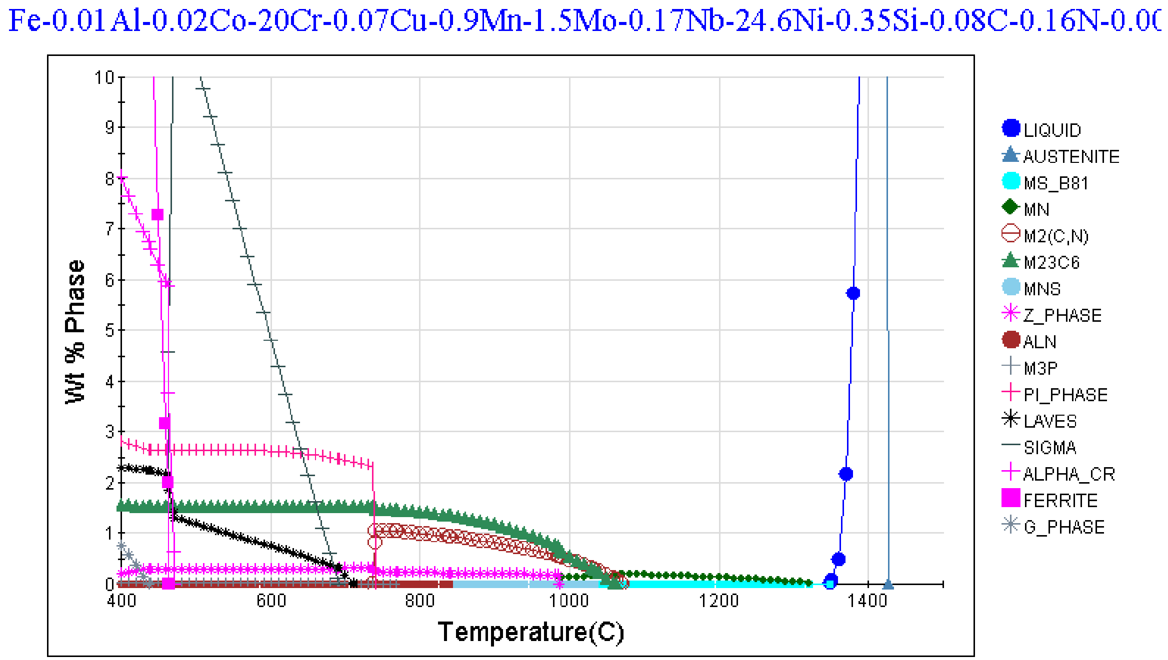
PT: 775 ± 15C for 10 hours



Results from multi-component phase equilibria and phase transformation simulations using JMATPRO provided by George Young Jr of Kairos Power

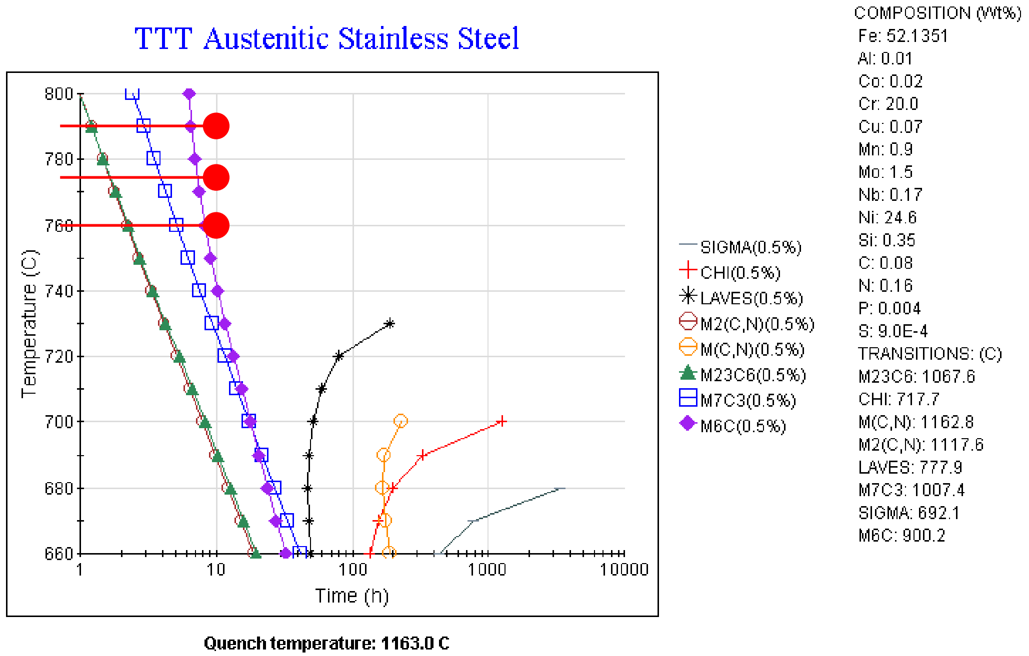
Computational Materials Simulations

ATI-1 Heat



Equilibrium Phase Diagram

PT: 775 ± 15C for 10 hours

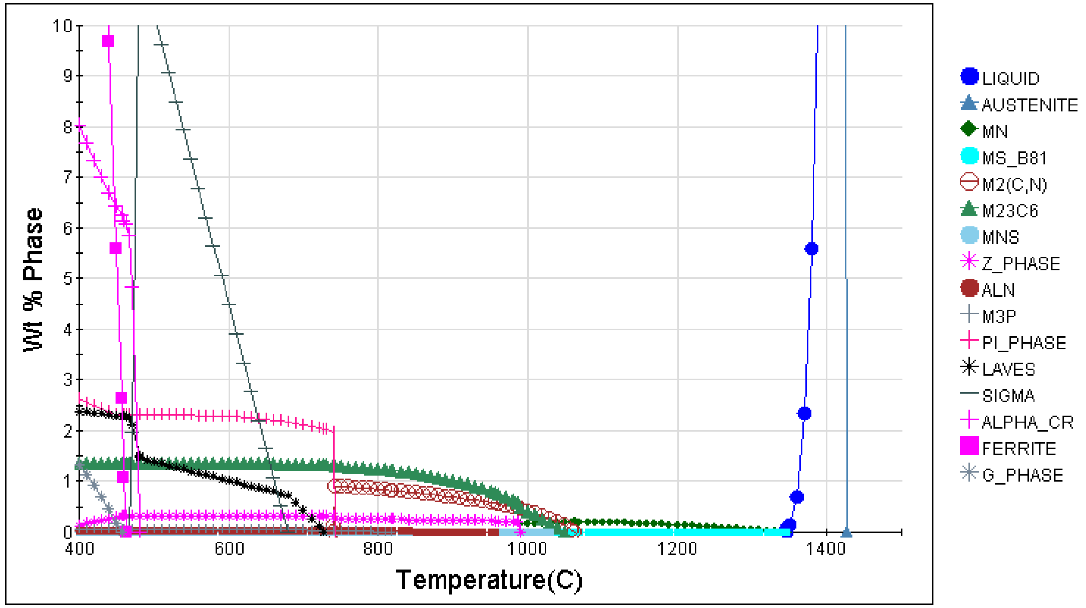


Results from multi-component phase equilibria and phase transformation simulations using JMATPRO provided by George Young Jr of Kairos Power

Computational Materials Simulations

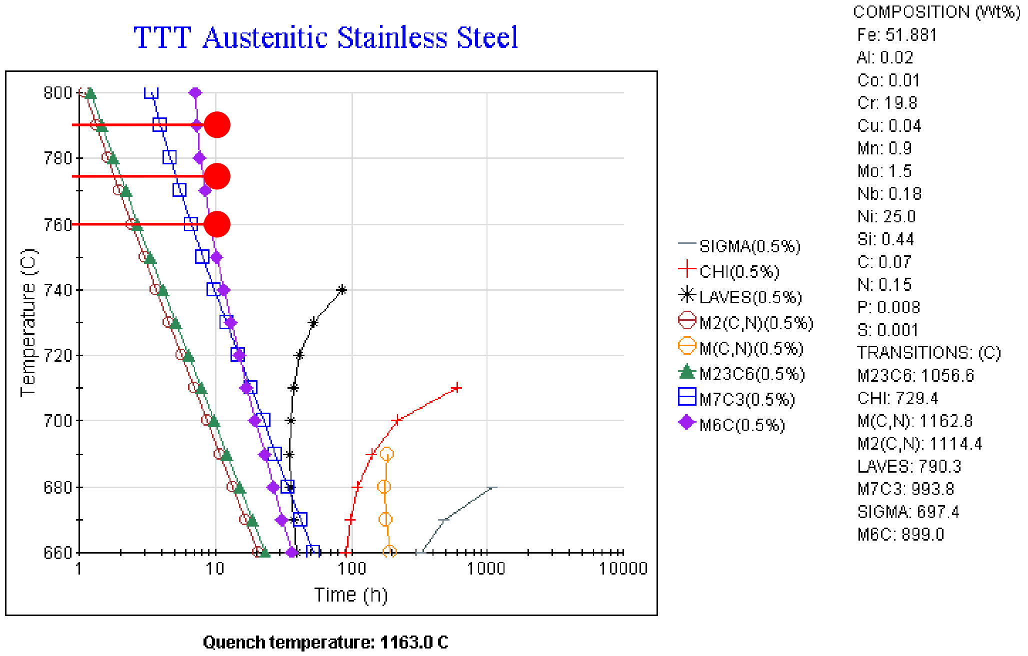
ATI-2 Heat

Fe-0.02Al-0.01Co-19.8Cr-0.04Cu-0.9Mn-1.5Mo-0.18Nb-25Ni-0.44Si-0.07C-0.15N-0.0(



Equilibrium Phase Diagram

PT: 775 ± 15C for 10 hours



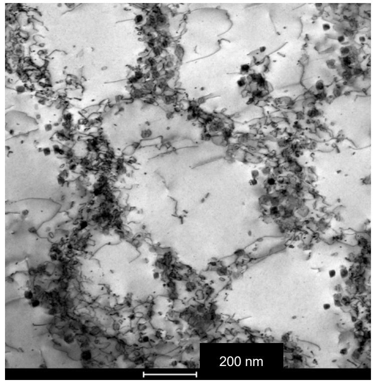
Results from multi-component phase equilibria and phase transformation simulations using JMATPRO provided by George Young Jr of Kairos Power

Role of Solutes in Mechanical Behavior of Alloy 709

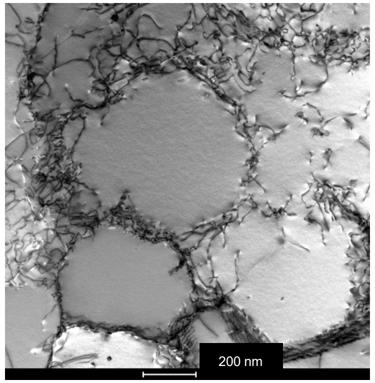
- Compared to solution annealing, aging reduces the amount of nitride or carbo-nitride forming elements in solution through precipitation of MX or Z phase prior to deformation
- The precipitation treatment (PT) of 775C/10 hours has resulted in reduced precipitation of MX or Z phases on dislocation substructures during subsequent cyclic deformation
- 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 precipitation 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

Microstructure of Cyclic-Tested Materials

Fatigue at 650°C

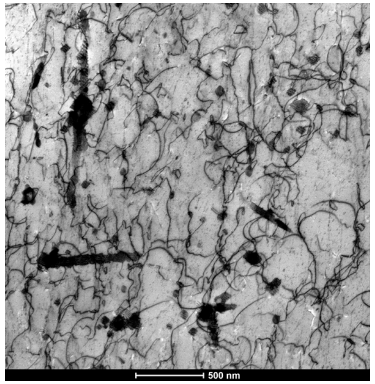


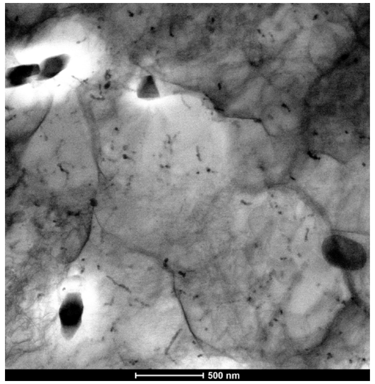
A709-SA: Cuboidal $M_{23}C_6$ distributed on dislocation cell walls



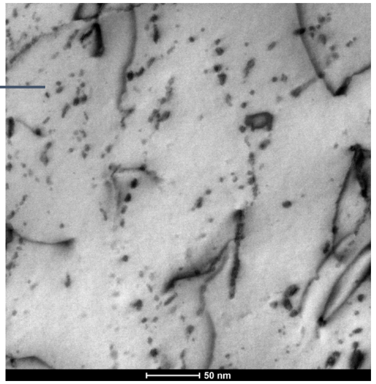
A709-PT (775°C for 10 hours): no precipitation on dislocation cell walls

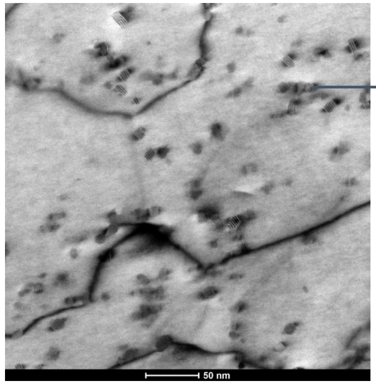
Creep-fatigue at 650°C





Average 6 nm dia.



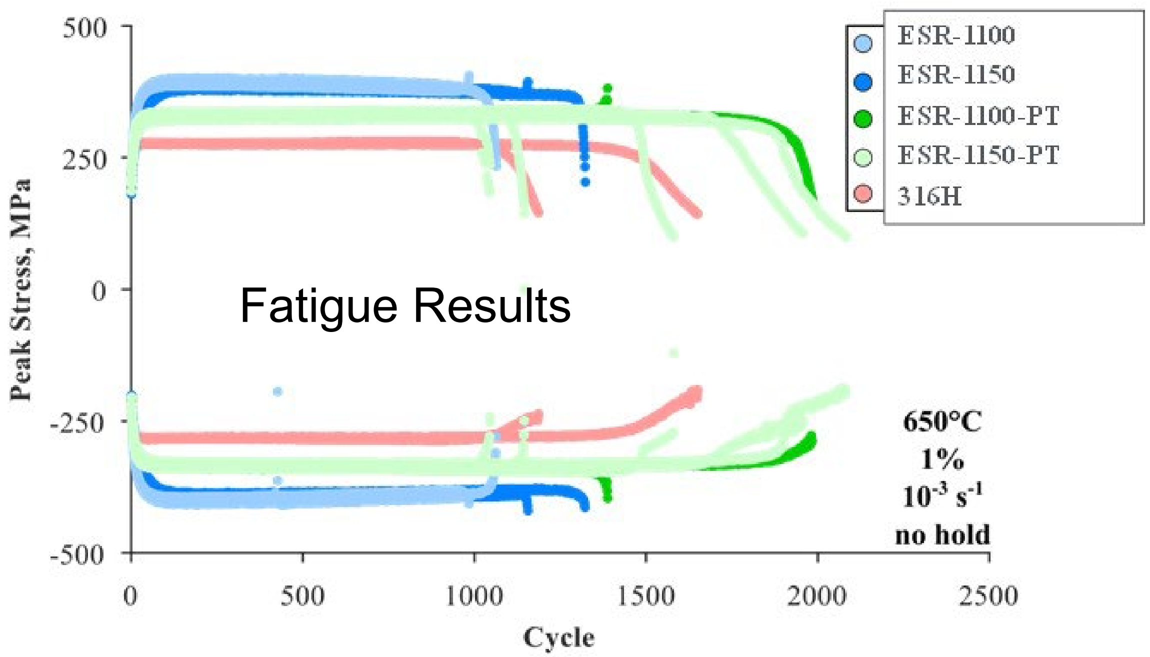


Average 10 nm dia.

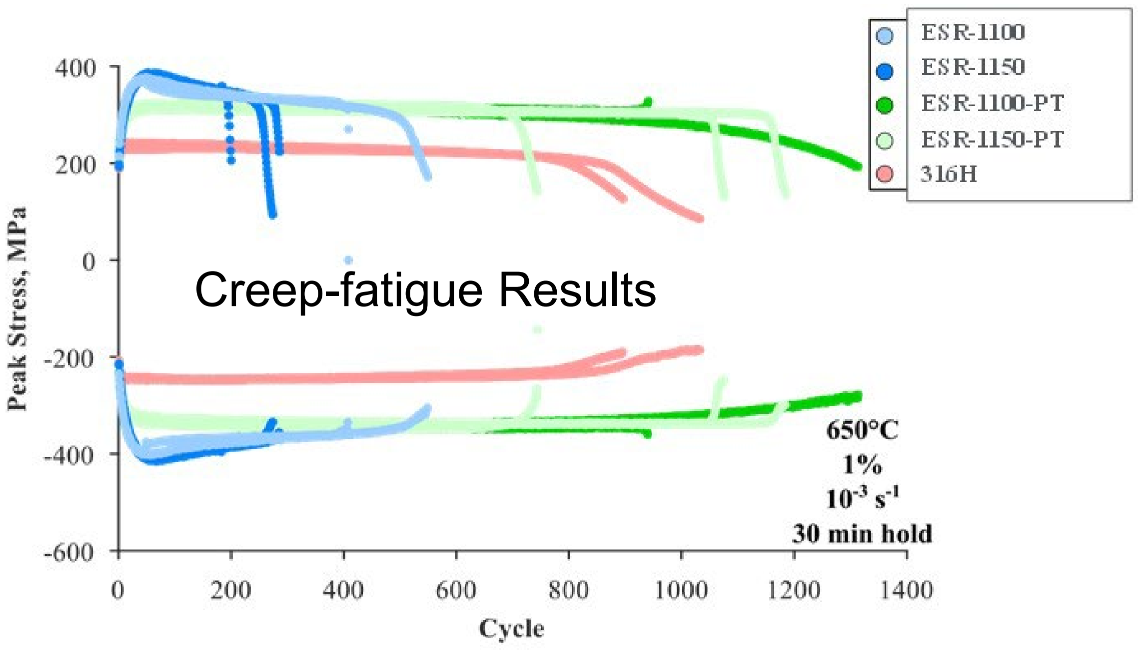
A709-SA: coarse $M_{23}C_6$ and fine Z-phase

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

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

ASTM Standards

- ASTM Standards define the requirements for alloy chemistry, specified mechanical properties, heat treatment and other characteristics such as melt practice, grain size and limits on product variability
- The specification allows a user to purchase material to the specification and provides acceptance criteria for the user’s quality organization to certify the material meets the acceptance criteria
- ASTM has standards for many materials
- A fraction of those specifications have been adopted by ASME to allow use of those adopted materials for constructions Codes

ASTM Standard Specification Development for Alloy 709

- ASTM has approved opening a ballot to incorporate Alloy 709 in ASTM Standard A 240 for solution annealed plate
- The Precipitation Treatment (PT) will be a supplementary requirement to ASTM Standard A 240, and not a mandatory requirement
- The supplementary requirement will say: "S3.1 If specified on the purchase order; solution annealed material, up to 2 inches (50 mm) thick, shall be heat treated at 1425F (775C) (±25 F / 15C) for 10 hr (±1 hr). For thickness greater than 2 inches (50 mm) heat treatment time shall be increased by 1 hour per inch (2.4 minutes per mm)"

ASME, Section II Specification Development for Alloy 709

- Will incorporate ASTM Standard A 240 directly into ASME, Section II, Part A, Ferrous Material Specification, SA 240, which will allow use of the alloy in Code constructions
- Will invoke the supplementary requirement of PT from SA 240 to improve creep-fatigue performance as a footnote to Section III, Division 5, Table HBB-I-14.1(a)
  - With this approach only Section III, Division 5 will call out the PT as mandatory requirement, and everywhere else (ASTM, A 240 and ASME, SA 240) it is a supplementary requirement
  - This will facilitate other applications, e.g., Section I or Section VIII, to promote supply chain development
- There is an additional requirement for a creep-fatigue acceptance test for austenitic steels used at high temperature in Section III, Division 5
  - That requirement is in Division 5, Subarticle HBB-2800, and not in the standard(s) and will be invoked in the Alloy 709 Code Case

Section III Division 5 Permissible Base Materials

MANDATORY APPENDIX HBB-I-14
TABLES AND FIGURES

Table HBB-I-14.1(a)
Permissible Base Materials for Structures Other Than Bolting

Base Material	Spec. No.	Product Form	Types, Grades, or Classes
Types 304 SS and 316 SS [Note (1)], [Note (2)], [Note (3)]	SA-182	Fittings & Forgings	F 304, F 304H, F 316, F 316H
	SA-213	Smls. Tube	TP 304, TP 304H, TP 316, TP 316H
	SA-240	Plate	304, 316, 304H, 316H
	SA-249	Welded Tube	TP 304, TP 304H, TP 316, TP 316H
	SA-312	Welded & Smls. Pipe	TP 304, TP 304H, TP 316, TP 316H
	SA-358	Welded Pipe	304, 316, 304H, 316H
	SA-376	Smls. Pipe	TP 304, TP 304H, TP 316, TP 3
	SA-403	Fittings	WP 304, WP 304H, WP 316, WP 316H, WP 304W, WP 304HW, WP 316W, WP 316HW
	SA-479	Bar	304, 304H, 316, 316H
	SA-965	Forgings	F 304, F 304H, F 316, F 316H
	SA-430	Forged & Bored Pipe	FP 304, FP 304H, FP 316, FP 316H
Ni-Fe-Cr (Alloy 800H) [Note (4)]	SB-163	Smls. Tubes	UNS N08810
	SB-407	Smls. Pipe & Tube	UNS N08810
	SB-408	Rod & Bar	UNS N08810
	SB-409	Plate, Sheet, & Strip	UNS N08810
	SB-564	Forgings	UNS N08810
2¹/₄Cr-1Mo [Note (5)]	SA-182	Forgings	F 22, Class 1
	SA-213	Smls. Tube	T 22
	SA-234	Piping Fittings	WP 22, WP 22W [Note (6)]
	SA-335	Forg. Pipe	P 22
	SA-336	Fittings, Forgings	F 22a
	SA-369	Forg. Pipe	FP 22
	SA-387	Plate	Gr 22, Class 1
	SA-691	Welded Pipe	Pipe 2¹/₄ CR (SA-387, Gr. 22, Cl. 1)
9Cr-1Mo-V	SA-182	Forgings	F91
	SA-213	Smls. Tube	T91
	SA-335	Smls. Pipe	P91
	SA-387	Plate	91

NOTES:
(1) These materials shall have a minimum specified room temperature yield strength of 30,000 psi (207 MPa) and a minim spe-cified carbon content of 0.04%.
(2) For use at temperatures above 1,000°F (540°C), these materials may be used only if the material is heat treated by heating to a minimum temperature of 1,900°F (1 040°C) and quenching in water or rapidly cooling by other mea
(3) Nonmandatory Appendix HBB-U provides nonmandatory guidelines on additional specification restrictions to improve performance in certain service applications.
(4) These materials shall have a total aluminum-plus-titanium content of at least 0.50% and shall have been heat treated at a temperature of 2,050°F (1 120°C) or higher.
(5) This material shall have a minimum specified room temperature yield strength of 30,000 psi (207 MPa), a minimum specified room temperature ultimate strength of 60,000 psi (414 MPa), a maximum specified room temperature ultimate strength of 85,000 psi (586 MPa), and a minimum specified carbon content of 0.07%.
(6) The material allowed under SA-234 shall correspond to one of:
  (a) SA-335, Grade P 22
  (b) SA-387, Grade 22, Class 1
  (c) SA-182, Grade F 22, Class 1 in compliance with Note (4).

Alloy 709 with specification number, product form and grade will be added to the Permissible Base Materials table

Footnote on the requirement of precipitation treatment (PT) for Alloy 709 will be added to the Notes Section of the Permissible Base Materials table

Summary

- Incorporation of Alloy 709 into ASTM Standard A 240 for plate material in the solution annealed condition with a precipitation treatment as a supplemental requirement is underway
- Incorporation of Alloy 709 into ASME, Section II, Specification SA 240 will be made upon the completion of the ASTM task
- The required commercial scale heats of Alloy 709 for property characterization to provide the technical basis for Code qualification have been procured
- Laboratory testing of properties from these three heats is underway as will be discussed separately at this review meeting

Thank you

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