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Advanced Reactor Technologies

FY-24 Progress Report on A709 Mechanical Properties Data Development and A709 Thermal Aging Status

SEPTEMBER 2024

Heramb Mahajan

Idaho National Laboratory

Battelle Energy Alliance manages INL for the U.S. Department of Energy's Office of Nuclear Energy

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September 2024

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INL ART Program

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Technical Reviewer: (Confirmation of mathematical accuracy, and correctness of data and appropriateness of assumptions.)

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SUMMARY

The report provides the status of the creep, fatigue, and creep-fatigue testing to date conducted at Idaho National Laboratory to generate a data package. This data package evaluates the material performance from three commercial heats to support the Alloy 709 qualification in American Society of Mechanical Engineers, Boiler and Pressure Vessel Code, Section III, Division 5. First, procured heat was manufactured by G.O. Carlson with heat number 58776. Second and third heats were fabricated by ATI Specialty Rolled Products, with heat numbers 529900 and 530843, respectively. A series of creep and cyclic specimens were fabricated from these three heats, and tests were performed. A list of finished, ongoing, and planned test are presented for creep, fatigue, and creep-fatigue tests. Cyclic properties of three commercial heats are compared.

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ACRONYMS

ANL	Argonne National Laboratory
AOD	Argon-oxygen decarburization
ART	Advanced Reactor Technologies
ASME	American Society of Mechanical Engineers
ASTM	American Society for Testing and Materials
BPVC	Boiler and Pressure Vessel Code
DOE	Department of Energy
ESR	Electroslag remelting
FRP	Fast Reactor Program
INL	Idaho National Laboratory
NE	Nuclear Energy
ORNL	Oak Ridge National Laboratory
PT	Precipitation treatment
SA	Solution Annealing
SFR	Sodium-cooled Fast Reactors

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1. INTRODUCTION

Sodium-cooled fast reactor (SFR) deployment is a key step towards a zero-carbon emission goal. The SFR construction and deployment is dependent on the development of advanced material development. The goal of the materials development of the Fast Reactor Campaign of the Advanced Reactor Technologies (ART) Program within the Office of Nuclear Energy of the Department of Energy (DOE) is to develop and qualify advanced structural materials to enable improved reactor performance. Higher reactor efficiency is expected at elevated temperatures. Thus, materials with higher performance at elevated temperature can reduce the SFR component construction costs, enabling the economic viability of SFR and enable higher safety margins over longer operating time.

A709 is an austenitic stainless steel with a better performance characteristic at elevated temperatures than 316H stainless steel. A significant effort to optimize and evaluate the material properties by three laboratories—Argonne National Laboratory (ANL), Idaho National Laboratory (INL), and Oak Ridge National Laboratory (ORNL)—led to the selection of A709 as a candidate material for American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code (BPVC), Section III, Division 5 [1]. This code provides component construction rules operating in the creep regime under cyclic loads. Developing design material properties, Section III Division 5 requires material property data from a minimum of three commercial heats. The staged code qualification plan and a test plan to support the code case were developed [2].

Significant work was done to develop the precipitation treatment for A709, evaluate its mechanical properties, and develop a data package for A709 over last few years [3–11]. These studies explored the microstructure and mechanical properties and evaluated the influence of different heat treatments on microstructure and mechanical properties. The solution annealing (SA) temperature of 1150°C or higher yielded the best creep properties, but with creep-fatigue properties that were less than optimal. Plates in SA states have been characterized and documented in reports [3,8,9]. Additional heat treatment of 775°C for 10 hours, referred as precipitation treatment (PT), achieved a balance in creep and creep-fatigue properties through precipitation of beneficial Nb-rich nano precipitates [6,7,11]. A709 material with SA followed by PT condition, referred as A709-PT in this report, is selected to support the code case.

This report presents test efforts to date at INL on the A709-PT to quality the A709 material code case. A list of finished, ongoing, and planned creep, fatigue, and creep-fatigue tests conducted in air environment are presented. Material properties from three commercial heats are compared for fatigue and creep-fatigue load conditions. Test efforts to understand the material properties degradation due to aging are discussed, and ongoing aging tests are documented.

2. MATERIAL

This report uses three different commercial heats in a plate product form from fabricators G.O. Carlson and ATI Specialty Rolled Products. Two different heats from ATI were procured, with heat numbers 529900 and 530843, which are referred in this report as ATI-1 and ATI-2, respectively. An argon-oxygen decarburization (AOD) followed by electroslag remelting (ESR) technique produced ingots. These ingots were hot rolled into the plate product form. The plates were SA at a minimum temperature of 1150°C. All plates used for code case testing were precipitation treated at 775°C for 10 hours in air, followed by air-cooling. Table 1 summarizes the detailed information of three commercial heats.

Table 1. Material information of three A709 commercial heats used for code case testing.

Heat number	Material details			Plate IDs used for Code Case testing	Nominal Grain size number	Nominal plate thickness (in.)
	Fabricator	Master heat number	Reference name used in this report			
1	G. O. Carlson	58776	Carlson	58776-3RBC1	7	1.1
2	ATI Specialty Rolled Products	529900	ATI-1	CG05455	7	1.75
3		530843	ATI-2	CG45192	4	1.75

The nominal grain size of Carlson heat and ATI-1 were Number 7, and the grain size of ATI-2 was Number 4. The parent plate identifiers (IDs) and nominal plate thickness of procured plates from three commercial heats are documented in Table 1. All specimens were machined along the rolling direction of the plates. Carlson heat specimens were machined from the mid-thickness of plates. For the other two commercial heats, two rows of specimens were machined from the plate thickness centered at one-quarter of thickness from plate surfaces. The chemical composition of the three commercial heats, as reported by fabricators in the vendor material certification, are listed in Table 2. The ASME BPVC SA213/SA213M-23 specification [12] provides the chemistry specification for A709 and is listed in Table 2.

Table 2. Chemical composition of three commercial heats of A709 in weight (%); G.O. Carlson, ATI-1 and ATI-2.

Fabricator	Heat Number	C	Cr	Co	Ni	Mn	Mo	N	Si
Carlson	58776	0.066	20.05	0.02	25.14	0.9	1.51	0.152	0.38
ATI-1	529900	0.08	19.9	0.02	24.6	0.9	1.5	0.15	0.39
ATI-2	530843	0.07	19.8	0.01	25	0.9	1.5	0.15	0.44
ASME SA-213	—	0.1 max	19.5–23.0	—	23.0–26.0	1.50 max	1.0–2.0	0.10–0.25	1.00 max
Fabricator	Heat Number	P	S	Ti	Nb	Al	B	Cu	Fe
Carlson	58776	0.014	0.001	0.01	0.26	0.02	0.003	0.06	Bal.
ATI-1	529900	0.003	<0.001	< 0.01	0.17	0.02	0.004	0.06	Bal.
ATI-2	530843	0.008	0.001	< 0.01	0.18	0.02	0.005	0.04	Bal.
ASME SA-213	—	0.030 max	0.030 max	0.20 max	0.10–0.40	—	0.002–0.010	—	—

3. CREEP-RUPTURE TESTS

All creep tests were conducted in an air environment. Creep specimens were fabricated and tested per ASTM, International Standard E139 [13] and PLN-3386, “Creep Testing” [14]. The nominal dimensions of creep specimens were 6.4 mm diameter and 32 mm reduced length. All test frames were manufactured by Applied Test System. Except for low-stress tests, typical load mechanisms consisted of a 20:1 lever arm. Creep test below 25 MPa stress were directly loaded. Two thermocouples monitored temperatures at the top and bottom of the specimen gauge. Generally, for creep-rupture tests at temperatures above 750°C, R-type thermocouples were used, and for other creep tests, K-type thermocouples were used. An extensometer attached to the specimen measured strain values at a resolution greater than 0.01%.

Pre- and post-test dimensions of specimens were measured using an optical comparator and calipers, respectively. These measurements were conducted at room temperature and were used to calculate the percentage area reduction and elongation of specimen. Table 3 lists the tests conducted to date and ongoing tests on three different heats.

Table 3. Creep test matrix of ongoing and finished tests on three different A709-PT heats.

Heat name, heat no., plate no.	Specimen ID	T (°C)	Stress (MPa)	Heat name, heat no., plate no.	Specimen ID	T (°C)	Stress (MPa)
Carlson-PT, 58776, 58776-3RBC1	BCHT-IC-1	600	330	ATI-1-PT, 529900, CG05455	CG05455-11-AT52	650	155
	BCHT-IC-6	650	155		CG05455-11-AT51	650	175
	BCHT-IC-5	650	175		CG05455-11-AT47	675	110
	BCHT-IC-10	675	110		CG05455-11-AT48	700	82
	BCHT-IC-9	700	82		CG05455-11-AT49	775	45
	BCHT-IC-3	700	175		CG05455-11-AT57	800	45
	BCHT2-IC-02	750	65		CG05455-11-AT57	800	45
	BCHT-IC-8	750	82		CG05455-11-AT43	800	65
	BCHT2-IC-01	775	45		CG05455-11-AT50	825	35
	BCHT3-IC-01	775	65		CG05455-11-AT44	850	35
	BCHT3-IC-02	800	45		CG05455-11-AT55	875	20
	BCHT-IC-12	800	65		CG05455-11-AT42	900	27
	BCHT2-IC-06	825	35		CG05455-11-AT56	950	8
	BCHT3-IC-03	825	45		CG05455-11-AT53	975	11
	BCHT-IC-7	850	35		CG05455-11-AT46	1000	8
	BCHT2-IC-03	850	45		CG05455-11-AT45	1000	9
	BCHT-IC-11	875	35	ATI-2-PT, 530843, CG45192	32A8-1-2	650	210
	BCHT-IC-4	925	27		32A8-1-3	675	195
	BCHT-IC-2	925	35		32A8-1-4	750	65
	BCHT2-IC-05	1000	8		Planned	825	50
	BCHT2-IC-04	1000	9		Planned	825	55
	—	—	—		Planned	850	40
	—	—	—		32A8-1-1	950	20

4. FATIGUE AND CREEP-FATIGUE TESTING

This subsection discusses both pure fatigue and creep-fatigue tests. Button-head cyclic specimens were fabricated with a nominal diameter of 7.49 mm following specifications of ASTM Standards E606 and E2714 [15, 16]. The detailed cyclic test procedure per plan PLN-3346 [17] was adopted. Both tests were executed in an air environment and at a constant temperature, using the strain-controlled loading waveforms as shown in Figure 1, where ε is the strain range, and t_h is the dwell time during which strain is held constant for stress relaxation. These tests were performed in servo-hydraulic frames with a mounted three-zone furnace manufactured by Materials Test Systems. Two R-type thermocouples were spot welded on top and bottom shoulders of the specimen to monitor temperatures. A direct-mounted extensometer, manufactured by Epsilon Technology, was used to measure strain in the specimen-gauge section. This extensometer gauge was 12 mm long with a total travel range of 5 mm. The optical comparator measured the critical dimensions of specimen prior to testing.

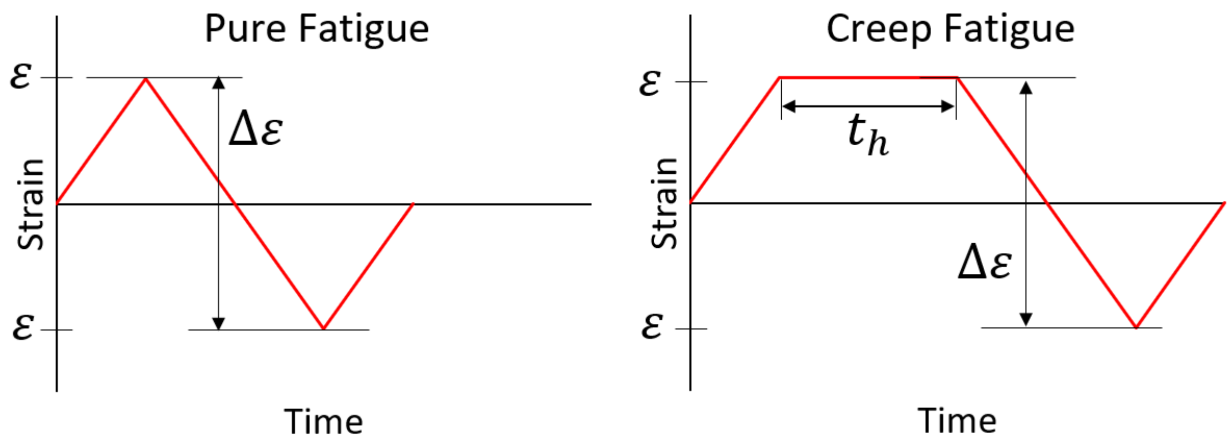

Figure 1. Strain profiles for pure fatigue and creep-fatigue tests.

Table 4 and Table 5 list the pure-fatigue and creep-fatigue tests, respectively. These tables present tests finished to date and ongoing/planned test on all three different heats to support the A709 code-case development. These tables show specimen ID, parent-plate ID, and heat number with the test parameters. A wide set of strain ranges is selected from 0.3 to 3%. Most of the fatigue and creep-fatigue tests were performed at 649°C and 954°C. A few fatigue tests were performed at other intermediate temperatures.

Table 4. Low-cycle fatigue-test matrix of tests finished on different A709-PT heats.

Heat name, heat no., plate no.	Specimen ID	T (°C)	Strain range (%)	Heat name, heat no., plate no.	Specimen ID	T (°C)	Strain range (%)
ATI-2-PT, 530843, CG45192	32A4-3-5	649	0.3	Carlson-PT, 58776, 58776-3RBC1	Planned	649	0.3
	32A4-8-5	649	0.3		BCHT-IF-5	649	0.3
	32A4-2-2	649	0.6		11-1-4	649	0.6
	32A4-2-5	649	0.6		BCHT-IF-6	649	0.6
	32A4-2-4	649	1		11-4-2	649	2
	32A4-5-4	649	2		BCHT-IF-7	649	2
	32A4-5-6	649	3		BCHT-IF-8	649	3
	32A4-1-2	950	0.4		BCHT-IF-1	650	1
	32A4-1-1	954	0.25		BCHT-IF-2	650	1

Heat name, heat no., plate no.	Specimen ID	T (°C)	Strain range (%)	Heat name, heat no., plate no.	Specimen ID	T (°C)	Strain range (%)
	32A4-2-6	954	0.25		BCHT-IF-15	816	0.25
	32A4-3-6	954	0.3		BCHT-IF-14	816	0.3
	32A4-8-1	954	0.3		BCHT-IF-13	816	0.6
	32A4-1-6	954	0.4		BCHT-IF-12	816	1.0
	32A4-3-2	954	0.4		BCHT-IF-11	816	3.0
	32A4-1-4	954	0.6		11-1-3	954	0.25
	32A4-8-6	954	0.6		Planned	954	0.25
	32A4-2-1	954	1		11-1-7	954	0.3
	32A4-5-2	954	1		Planned	954	0.3
	32A4-3-3	954	2		11-1-2	954	0.4
	32A4-8-3	954	2		Planned	954	0.4
	32A4-5-5	954	3		11-1-5	954	0.6
ATI-1-PT, 529900, CG05455	CG05455-13-AF26	871	1		11-1-1	954	1
	CG05455-13-AF23	927	1		Planned	954	1
	CG05455-13-AF33	954	0.25		11-1-6	954	2
	CG05455-13-AF30	954	0.4		Planned	954	2
	CG05455-13-AF31	954	1		—	—	—
	CG05455-13-AF34	954	2		—	—	—
	CG05455-13-AF35	954	3		—	—	—
	CG05455-13-AF22	982	1		—	—	—

Table 5. Test matrix of creep-fatigue tests finished on different A709-PT heats.

Heat name, heat no., plate no.	Specimen ID	Temperature (°C)	Strain range(%)	Hold time (min)
ATI-2-PT, 530843, CG45192	32A4-1-3	954	0.3	10
	32A4-8-4	954	0.4	10
	32A4-3-4	954	0.4	30
	32A4-5-1	954	0.4	30
	32A4-8-2	954	0.6	10
	32A4-1-5	954	0.6	30
	32A4-2-3	954	0.6	60
	32A4-5-3	954	1	60
ATI-1-PT, 529900, CG05455	CG05455-13-AF29	954	0.3	10
	CG05455-13-AF25	954	0.4	10
	CG05455-13-AF28	954	0.4	30
	CG05455-13-AF32	954	0.4	30

Heat name, heat no., plate no.	Specimen ID	Temperature (°C)	Strain range(%)	Hold time (min)
	CG05455-13-AF21	954	0.6	10
	CG05455-13-AF24	954	0.6	10
	CG05455-13-AF36	954	0.6	60
	CG05455-13-AF27	954	1	30
Carlson-PT, 58776, 58776-3RBC1	Planned	649	0.3	10
	Planned	649	0.3	10
	Planned	649	0.6	10
	BCHT-IF-9	649	0.6	10
	Planned	649	1	10
	Planned	649	1	60
	BCHT-IF-3	650	1	30
	BCHT-IF-4	650	1	30
	BCHT-IF-10	704	0.6	10
	Planned	954	0.3	10
	Planned	954	0.3	10
	Planned	954	0.4	10
	Planned	954	0.4	10
	Planned	954	0.4	30
	Planned	954	0.4	30
	Planned	954	0.6	10
	Planned	954	0.6	10
	Planned	954	0.6	30
	Planned	954	0.6	60
	Planned	954	0.6	60
	Planned	954	1	10
	Planned	954	1	10
	Planned	954	1	30
	Planned	954	1	30
	Planned	954	1	60

The absolute ratio of maximum to minimum stress is calculated and defined as r. This ratio is plotted against the number of cycles and used to calculate the failure cycle. A line is drawn using the steady state r values with an offset of 20% drop. The cycle at which this line intersects with the r is defined as cycle to failure N_{f20}. Figure 2 illustrates the schematics of failure-cycle determination for cases with either cyclic hardening or cyclic softening. This approach was used to calculate failure-cycle determination for fatigue and creep-fatigue tests. Figure 3 shows the failure cycles of fatigue tests at 649, 650, 950, and 954°C.

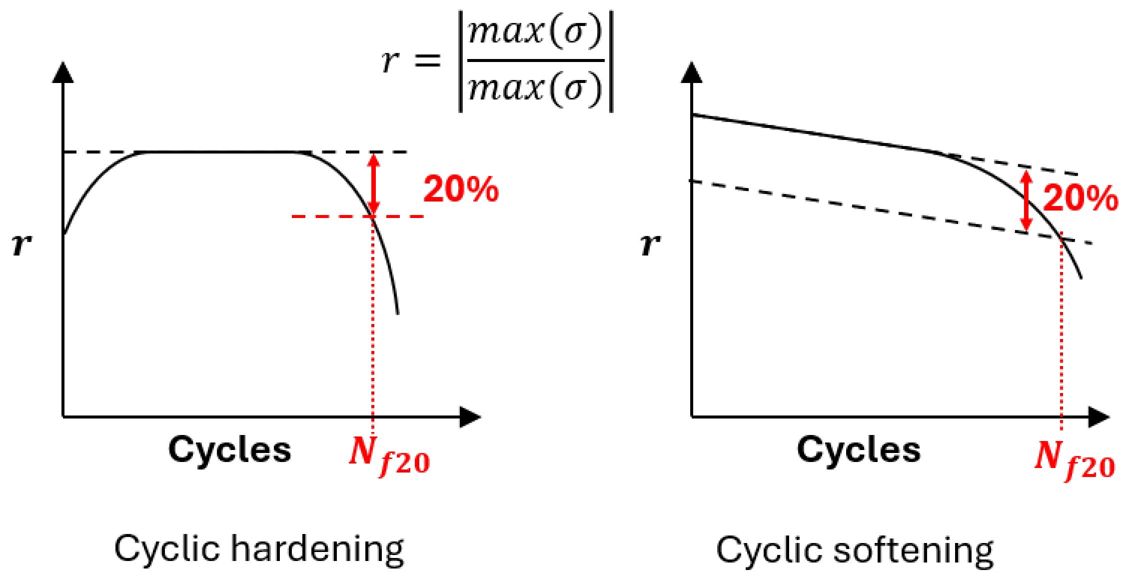

Figure 2. Schematic figures illustrating the approach to calculate cycles to failure in fatigue and creep-fatigue tests for cyclic hardening and cyclic softening cases.

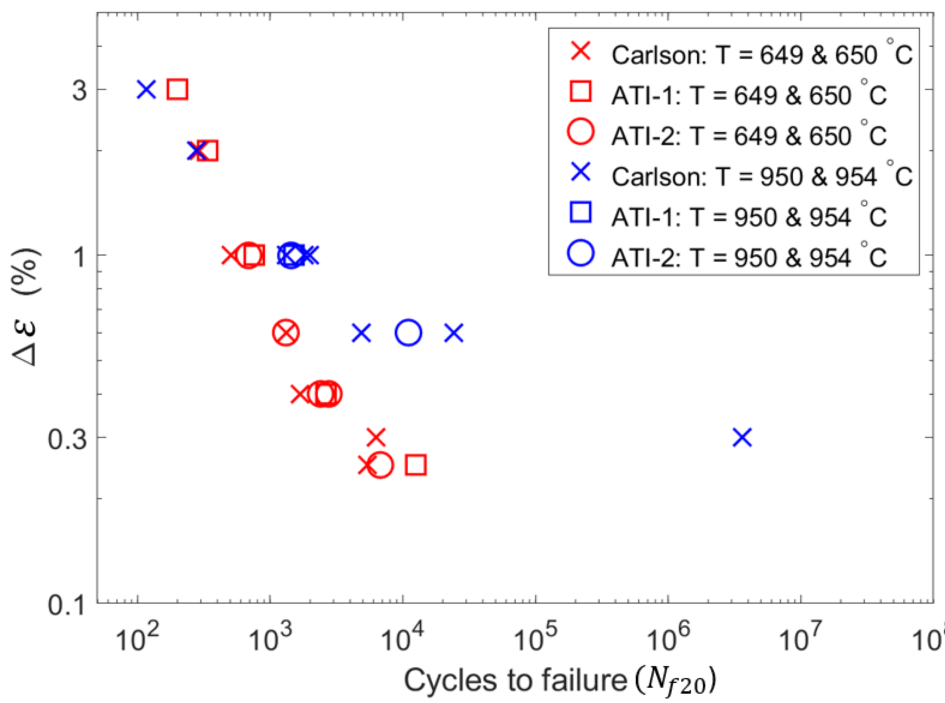

Figure 3. Number of cycles to failure (N_{f20}) against the applied strain range from fatigue tests on three commercial heats of A709-PT at 649, 650, 950, and 954°C.

The fatigue tests conducted on three commercial heats were compared. Figure 4 shows the hysteresis stress-strain loop at the half-life cycle ($N_{f20}/2$), and it displays peak and valley stresses over a number of cycles at 954°C and a strain range of 1%. The hysteresis loops from the three commercial heats were reasonably identical. The peak and valley stresses show cyclic softening for all three heats. Although, ATI-2 heat had small nominal grain size, the failure life variation was marginal.

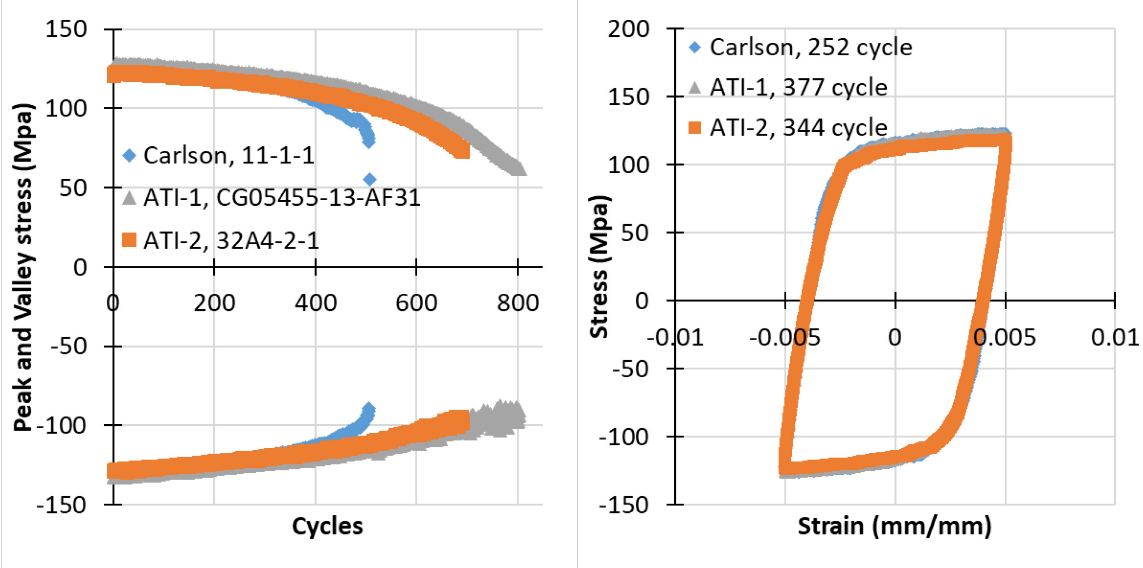

Figure 4. Comparison of three different heats under pure fatigue test at 954°C and 1% strain range showing peak and valley stresses against cycles with heat and specimen ID, and half-life hysteresis stress strain response with half life cycle.

Comparisons are presented in Figure 5, where data of creep-fatigue test at 954°C, strain range of 0.6% and dwell time of 10 minutes at peak strain from the two heats are presented. Cyclic softening was observed, and cycles to failure for ATI-1 and ATI-2 were similar. The half-life ($N_{f20}/2$) hysteresis loops show comparable material response. The stress-relaxation time histories during the strain dwell of the half-life loop are compared in Figure 6. Stress-relaxation rates were consistent for ATI-1 and ATI-2 heats, and no significant difference in material response were observed. Creep-fatigue test at 954°C, strain range of 0.6% and dwell time of 10 minutes on the Carlson heat is in progress. These data will be used to calculate the creep- and fatigue-damage fractions per Section III, Division 5, Class A, to establish the creep-fatigue damage envelop of A709.

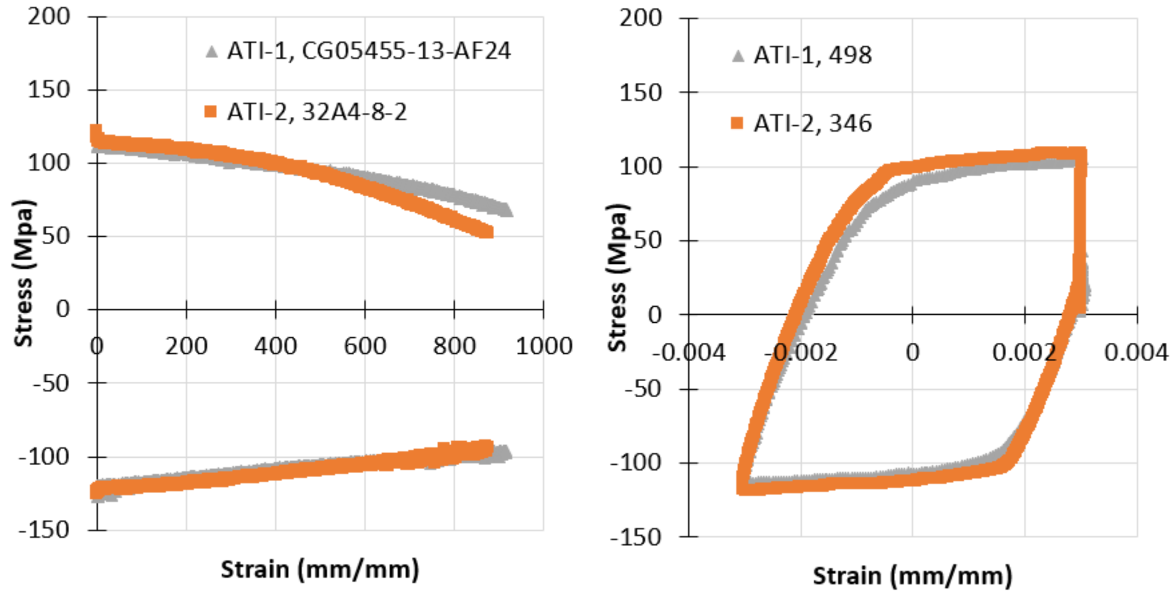

Figure 5. Comparison of two different heats under creep-fatigue test at 954°C, 0.6% strain range and 10 minutes of dwell time at peak strain showing peak and valley stresses against cycles with heat and specimen ID, and half-life hysteresis stress-strain response within the half-life cycle

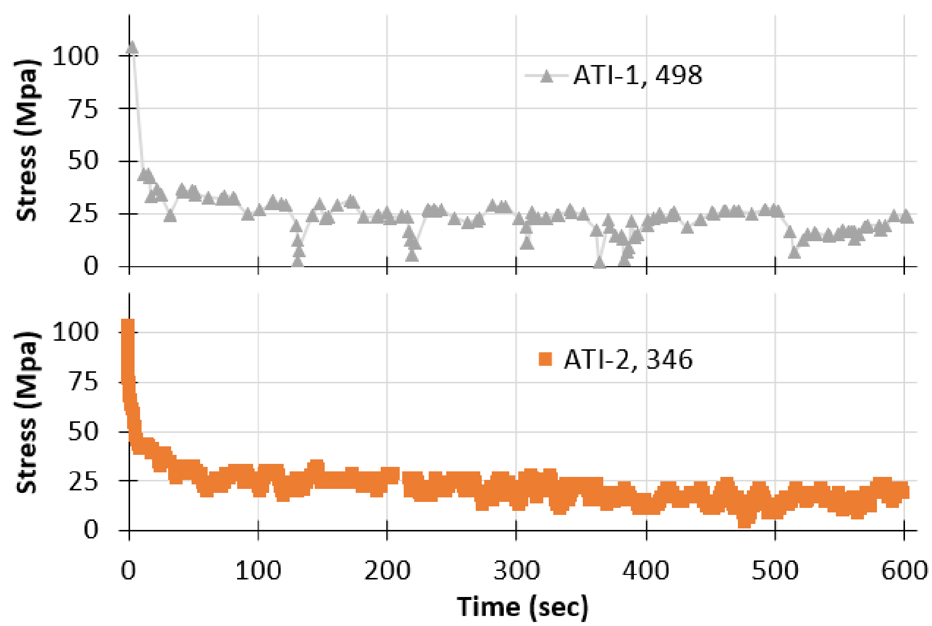

Figure 6. Stress-relaxation profiles from two different heats under creep-fatigue test at 954°C, 0.6% strain range, and 10 minutes of dwell time at peak strain for a half-life cycle.

5. THERMAL AGING

Sustained exposure of materials to elevated temperature leads to precipitate accumulation. These precipitate depositions within grains and along grain boundaries lead to mechanical-property changes. Such prolonged exposures are anticipated in SFR, which makes understanding the influence of aging on material properties an essential step towards code qualification of A709. Section III, Division 5, requires material-degradation information due to aging on tensile properties and subsequent allowable stresses.

The thermal aging study is intended to support the data package of A709 for code qualification through exposing material at elevated temperatures for desired time periods. Table 6 lists aging parameters of ongoing aging tests on A709 at INL. Plates from three commercial heats discussed in the materials section were selected. All plates were SA at a minimum temperature of 1150°C, followed by PT at 775°C for 10 hours in air with subsequent air-cooling. Post-aged plates will be used to study the microstructural and mechanical changes on A709-PT material. A set of metallographic, tensile, and cyclic specimens will be machined from three commercial heats per plan in Table 6. From the Carlson and ATI-1 heats, a few compact-tension specimens will be machined to study the influence of aging on fracture toughness. These tests will establish the degradation factors due to aging for A709-PT material.

Table 6. Thermal aging on three commercial heats and planned tests at INL

Commercial heat name	Aging parameters		Planned post-aging tests
	Temperature (℃)	Time (hr)	
Carlson	538	10000, 30000, 60000, 100000	Tension tests, cyclic tests, Compact Tension test, metallographic study
	649	3000, 10000, 30000, 60000, 100000	
ATI-1	538	10000, 30000, 60000, 100000	
	649	3000, 10000, 30000, 60000, 100000	
Carlson	538	15000, 20000	Tension tests, cyclic tests, metallographic study
	649	15000, 20000	
ATI-1	538	15000, 20000	
	649	15000, 20000	
ATI-2	538	10000, 30000, 60000, 100000	
	649	3000, 10000, 30000, 60000, 100000	

Four electric resistive-heating furnaces are used for the aging study. Two sets of furnaces, with two furnaces in each set, were dedicated to aging temperatures of 538 and 649°C. One furnace has plates identified for aging time up to 30,000 hours, and the other furnace has plates with an aging time of 60,000 and 100,000 hours. The aging program adopts the standard per PLN-6871, "Thermal Aging of Alloy 709" [18]. Temperature is monitored through K-type thermocouple attached to the A709 temperature monitoring block. Free spaces of 2 in. were provided on all furnace walls, and 1/2 in. of space was provided between plate stacks. In the next fiscal year, aging of three commercial heats—A709-PT at 538 and 649°C for 3,000 hours—will be finished. Initial results will be compared against the A709-PT data to explore the need of aging factors for short-duration exposures. The remainder of the aging study will continue during the next fiscal year.

6. SUMMARY

This report summarizes the test status of creep, fatigue, and creep-fatigue testing to date, conducted at INL on A709-PT on three commercial heats to support the A709 code-case development. The test data are used in the development of such material properties as allowable stresses, minimum stress-to-rupture, fatigue design curves, and creep-fatigue damage envelop. A list of finished and ongoing creep, fatigue, and creep-fatigue tests are presented and documented data are summarized. Planned tests will be finished in the next FY and will be used to develop the A709 data package. A list of ongoing aging tests on A709-PT plates from three commercial heats is presented. Aging temperatures of 538 and 649°C were selected, and aging times range from 3,000 to 100,000 hours. A plan of post-aging tests is presented to study and characterize the material-properties degradation due to aging at elevated temperatures. These tests will support the development of reduction factors for A709 due to aging.

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