

# Interim Creep, Fatigue and Creep-Fatigue Data from FY 2022 INL Testing of A709 with Precipitation Treatment for ASME Code Case Data Package

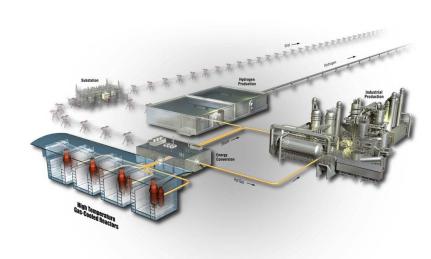
September 2022

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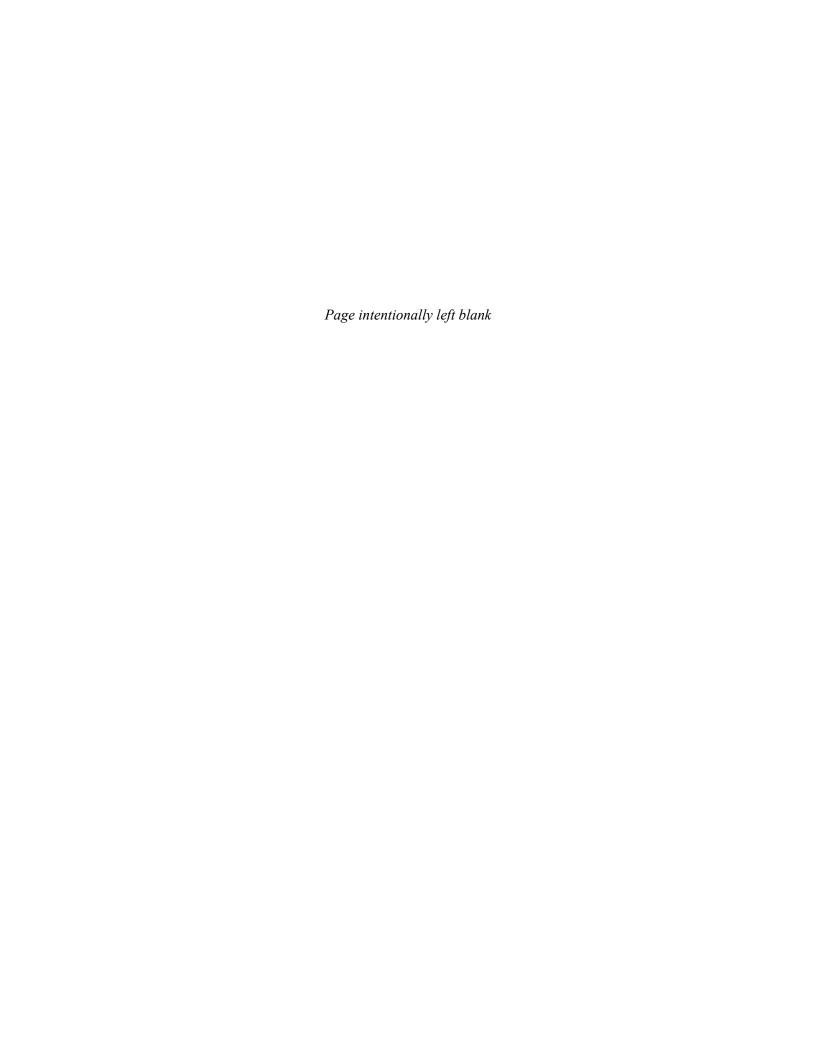
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September 2022

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Prepared for the
U.S. Department of Energy
Office of Nuclear Energy
Under DOE Idaho Operations Office
Contract DE-AC07-05ID14517



#### **INL ART Program**

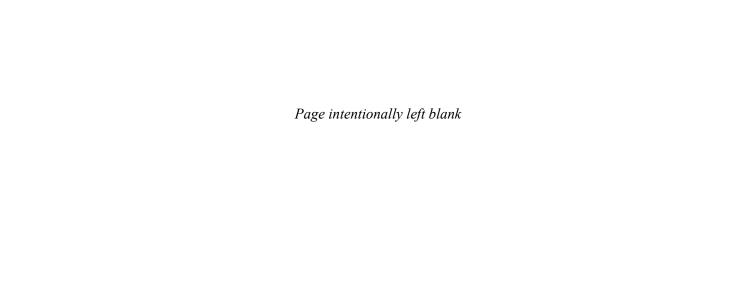
### Interim Creep, Fatigue and Creep-Fatigue Data From FY 2022 INL Testing of A709 with Precipitation

Revision 0 September 2022

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appropriateness of assumptions.)					
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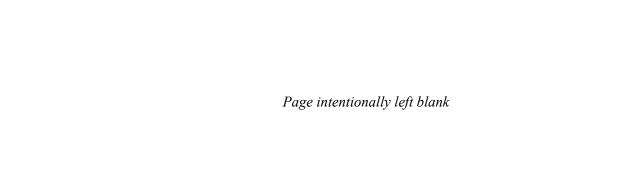
#### **SUMMARY**

This report provides the status of creep, fatigue, and creep-fatigue testing that transpired in fiscal year 2022 at Idaho National Laboratory (INL). This testing is being conducted to develop the data package to qualify A709 in Section III, Division 5 of the American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code (BPVC). This would permit the use of A709 for elevated-temperature nuclear construction. Preliminary results continue to demonstrate the improved creep and fatigue resistance of A709 compared to 316H stainless steel.



#### **ACKNOWLEDGEMENTS**

This research was sponsored by the United States (U.S.) Department of Energy (DOE) under Contract No. DE-AC07-05ID14517 with Idaho National Laboratory (INL), which is managed and operated by Battelle Energy Alliance. Programmatic direction was provided by the Office of Nuclear Reactor Deployment of the DOE Office of Nuclear Energy. The authors gratefully acknowledge the support provided by Sue Lesica, Federal Lead for Advanced Materials, Advanced Reactor Technologies (ART) Program; Brian Robinson, Federal Manager, ART Fast Reactors (FR) Campaign; and Bo Feng, ANL, National Technical Director, ART FR Campaign. The authors also acknowledge helpful discussions with Yanli Wang of Oak Ridge National Laboratory, Xuan Zhang of Argonne National Laboratory, Richard Wright of Structural Alloys, Limited Liability Corporation (LLC), and Michael McMurtrey of INL. Finally, the authors thank their support staff: J.W. Jones, M.P. Heighes, and J.A. Simpson of INL for their technical support.





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#### **ACRONYMS**

ANL Argonne National Laboratory

ASME American Society of Mechanical Engineers
ASTM American Society for Testing and Materials

ATI Allegheny Technologies Incorporated

BPVC Boiler and Pressure Vessel Code

ESR electroslag remelting

FY fiscal year

INL Idaho National Laboratory

NRC U.S. Nuclear Regulatory Commission

ORNL Oak Ridge National Laboratory

SFR sodium-cooled fast reactor

SS stainless steel

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## Interim Creep, Fatigue and Creep-Fatigue Data from FY 2022 INL Testing of A709 with Precipitation Treatment for ASME Code Case Data Package

#### 1. MOTIVATION

President Biden's Executive Order 14008 issued a federal directive for the development of a plan to facilitate a carbon-zero electric-industry sector by 2035 (Exec. Order No. 14008 2021). Nuclear energy is one potential resource to satisfy this executive order. One of the leading advanced nuclear reactor concepts is the sodium-cooled fast reactor (SFR). Other needs not associated with this executive order but important for clean energy—such as the recycling of spent nuclear fuel—can also be attained with SFRs (Sham and Natesan 2017).

SFR commercialization is dependent upon these reactors being economically viable. A crucial aspect to achieving this commercialization is advanced materials, which can reduce capital constructions costs. Although advanced materials are typically more expensive than traditional steels, they offer advantages that may ultimately lead to savings. Advanced materials may enable higher operating temperatures, which improves thermal efficiency and leads to increased power output. Additionally, advanced materials may offer longer design lifetimes, meaning components may not have to be replaced as regularly. Besides economic savings, advanced materials may offer improved safety margins, material reliability, and design flexibility (Sham and Natesan 2017).

A709, an austenitic stainless steel (SS), has been identified as an advanced material that would improve the viability of SFRs. A709 has better creep properties than 316H SS, the reference construction material for SFRs, but is not as expensive as nickel-based alloys usually used at high temperatures. This enables the construction of thinner walled components, reducing construction costs. A709 has an improved resistance to thermal gradients compared to 316H SS. This eliminates the need for expensive add-on hardware required for construction with 316H SS, further reducing construction costs. There are additional benefits beyond the two examples provided for constructing components with A709 as compared to 316H SS. Potential SFR components to be constructed with A709 are the reactor vessel, piping, core supports, intermediate heat exchanger, and compact heat exchanger. The compact heat exchanger could link the SFR to a supercritical carbon dioxide Brayton energy conversion system as one possible application (Sham and Natesan 2017).

In the United States (U.S.), the U.S. Nuclear Regulatory Commission (NRC) licenses and regulates nuclear reactors and nuclear reactor designs. Nuclear plant owners and operators can take advantage of Section III, Division 5 of the American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code (BPVC) to reduce the effort needed to license a nuclear reactor or nuclear reactor design. This section of the Code provides design rules for elevated-temperature nuclear construction (ASME 2021b). The NRC is in the process of evaluating the 2017 version of Section III, Division 5 for potential endorsement (Thomas 2018; Nuclear Regulatory Commission 2022). A709 is currently not qualified in Section III, Division 5 of the ASME BPVC. A program comprised of a collaboration between three U.S. Department of Energy (DOE) national laboratories—Argonne National Laboratory (ANL), Idaho National Laboratory (INL), and Oak Ridge National Laboratory (ORNL)—is in progress to qualify A709 in Section III, Division 5 of the ASME BPVC through multiple Code Cases. These Code Cases require conducting tests to collect A709 data and creating data packages from which the design rules will be established.

The purpose of this report is to provide the status of testing conducted in fiscal year (FY) 2022 at INL to generate the data packages for the A709 Code Cases. Focus will be on creep, fatigue, and creep-fatigue testing. Preliminary results are discussed. To date, Code Case testing has been conducted on Commercial

Heat 1 and Commercial Heat 2. The former was fabricated by Electralloy/G.O. Carlson, heat 58776, and Allegheny Technologies Incorporated (ATI) Flat Rolled Products, heat 529900, respectively.

#### 2. BACKGROUND

A709 is an austenitic SS that originated from NF709, which was developed as a tubing material for ultra-supercritical boiler applications by Nippon Steel. NF709 is strengthened by molybdenum and nitrogen in solution as well as carbonitride precipitates. NF709 was designed so that detrimental intermetallics do not precipitate during long-term, elevated-temperature service (Sham and Natesan 2017). NF709 is included in American Society for Testing and Materials (ASTM) specification A213 and ASME specification SA-213. Both specifications are entitled "Standard Specification for Seamless Ferritic and Austenitic Alloy-Steel Boiler, Superheater, and Heat Exchanger Tubes." NF709 is designated as grade TP310MoCbN and UNS S31025 (ASTM 2021; ASME 2021c). ASME BPVC Case 2581 qualifies Section I construction of NF709 seamless tubes for temperatures up to 815°C (1500°F) (ASME 2021e).

A collaboration between ANL, INL, and ORNL was initiated in 2013 to develop A709 plate and characterize its properties. This work resulted in the recommendation to qualify A709 in Section III, Division 5 of the ASME BPVC for elevated-temperature nuclear construction. This recommendation was based on the following findings: (1) A709 is stable in an elevated-temperature, liquid-sodium environment; (2) A709 can be successfully welded with Alloy 625 or optimized A709 filler metals by automated gas-tungsten arc welding; (3) the production of A709 can be optimized to improve the creep-fatigue properties while maintaining creep strength; and (4) the properties of A709 are improved compared to 316H SS, which is qualified in Section III, Division 5 of the ASME BPVC (ASME 2021b, Sham and Natesan 2017).

A plan for developing the data package needed to qualify A709 in Section III, Division 5 of the ASME BPVC was formulated. Multiple data packages were devised through a staged qualification approach for A709 to support multiple code cases with increasing design lives. This approach entails qualifying a material for a shorter design life initially using data that can be generated in the near term. The qualified design life is then extended at a later date once the longer-term data needed to support this extension, such as creep and thermal aging, becomes available. This enables accelerated qualification of A709. The alternative, non-accelerated approach, is to wait until all necessary data to support a Code Case for the longest design life is generated. This plan took into consideration the needs of both the ASME BPVC and NRC (Sham and Natesan 2017). This includes Nonmandatory Appendix HBB-Y in Section III, Division 5 of the ASME BPVC, which provides guidelines for qualifying new materials, as well as re-occurring issues flagged by the NRC (Sham and Natesan 2017; ASME 2021b; O'Donnell and Griffin 2007). Nonmandatory Appendix HBB-Y specifies the data package for qualifying new materials must be comprised of data from a minimum of three heats, which need to encompass the composition range, product forms, and size of the components to be used in service (ASME 2021b).

The first commercial heat of A709 was procured in FY 2017 from Electralloy/G.O. Carlson, heat 58776. This heat was procured in a manner that enabled the final processing step and the solution annealing temperature to be independently investigated. The final processing step was either argon oxygen decarburization (AOD), electroslag remelting (ESR), or ESR with a homogenization treatment (ESR + HOMO). The plates were solution annealed at 1050°C (1922°F), 1100°C (2012°F), or 1150°C (2102°F). A total of nine unique groups of plates were fabricated, one for each processing combination (Natesan et al. 2017). Creep, fatigue, and creep-fatigue testing were initiated to down-select the final processing step and solution anneal temperature. Plates that were solution annealed at 1050°C (1922°F) were eliminated from consideration because of a too fine grain size and an unacceptably short creep-rupture lifetime. The ESR final processing condition resulted in longer elongations to failure during creep, establishing it as the best method of manufacturing. A tradeoff between creep and creep-fatigue properties was found between the solution annealing temperatures. The plate with the ESR final

processing step, solution annealed at 1150°C (2102°F) had the best creep-rupture properties but inadequate creep-fatigue properties. The processing condition that resulted in the optimal balance in properties was the ESR plate solution annealed at 1100°C (2012°F), although the creep-fatigue properties were worse than desired (McMurtrey 2018; McMurtrey and Rupp 2019; Natesan, Zhang, and Li 2018; Wang et al. 2018). During this time, preliminary results prompted ORNL to start long-term tests of A709 fabricated with AOD and solution annealed at 1100°C (2012°F) (Wang et al. 2018; Wang and Sham 2019; Wang, Hou and Sham 2020; Wang, Hou, and Sham 2021). Later findings resulted in A709 tests with the final processing step of ESR and solution annealed at 1100°C (2012°F) being started at ANL and ORNL (Wang and Sham 2019; Wang, Hou, and Sham 2020; Wang, Hou, and Sham 2021; Zhang and Sham 2019; Zhang and Sham 2020).

A heat treatment referred to as a precipitation treatment for A709 was developed in FY 2019. This precipitation treatment emanated from two questions. The first was concerning whether the beneficial precipitates that form during accelerated testing would form during service at low temperature, low stress conditions. The second was whether the A709 properties, particularly the creep-fatigue properties, could be further improved. Precipitation treating A709 prior to service would ensure the beneficial precipitates would be present during service conditions. The heat treatment needed to precipitate the beneficial MX (Nb, Ti, V: C, N) and Z-phase (CrNbN) without forming the detrimental intermetallics (Fe<sub>2</sub>Mo, Fe<sub>2</sub>Nb). A 10-hour, 775°C (1427°F) heat treatment protocol followed by air cooling was identified (Zhang, Sham, and Young 2019). This heat treatment promoted the precipitation of the beneficial MX and Z-phase and was observed to improve creep-fatigue properties without significantly impacting creep properties (Rupp and McMurtrey 2020). A709 with ESR as the final processing step, solution annealed at 1150°C (2102°F), and subsequently precipitation treated at 775°C (1427°F) for 10 hours followed by air cooling has been identified as having the best properties. Testing of the A709 material with ESR as the final processing step, solution annealed at 1150°C (2102°F), and precipitation treated was initiated to develop the data package to qualify A709 in Section III, Division 5 of the ASME BPVC (Rupp et al. 2021; Wang, Hou, and Sham 2021; Zhang, Sham, and Li 2021).

In FY 2020, INL placed a purchase order with ATI Flat Rolled Products to procure the second commercial heat of A709 (Wright 2020). The A709 plates, heat 529900, were delivered by ATI in March 2021. This heat was comprised of nine plates, all fabricated by ESR. Of these plates, eight were hot rolled and solution annealed; six of the plates were nominally 1.75 inches thick and the remining two were nominally 2.0 inches thick. The solution anneal was conducted at a minimum of 1149°C (2100°F). The remaining plate was nominally 1.75 inches thick and delivered in the hot-rolled condition. Microstructural characterization and preliminary mechanical testing of this heat has been conducted at INL (Bass and Sham 2022; Mohale and Bass 2022). Testing of material from this heat in the precipitation-treated condition to support the generation of the Code Case data packages was initiated in FY 2022.

In FY 2022, the mechanical properties of the first two commercial heats of plate A709 in the precipitation-treated condition were assessed. This assessment evaluated all tensile, creep, fatigue, and creep-fatigue data generated to date. The mechanical properties of precipitation-treated A709 were determined to outperform those of 316H SS. Consequently, the recommendation to qualify A709 in Section III, Division 5 of the ASME BPVC for elevated-temperature nuclear construction was upheld. This recommendation entails the continuation of the collaboration between ANL, INL, and ORNL to continue development of the data packages needed to qualify A709 in Section III, Division 5 of the ASME BPVC (Sham et al. 2022).

#### 3. MATERIAL

#### 3.1 Commercial Heat 1

The first commercial heat of plate A709, heat 58776, was procured from Electralloy/G.O. Carlson in the solution-annealed condition. In FY 2022, the ESR plate solution annealed at 1150°C (2102°F) and precipitation treated for 10 hours at 775°C (1427°F) and subsequently air cooled was investigated. This plate is 1.1 in. thick with the chemical composition shown in Table 1. The chemistry requirements specified in SA-213 for UNS S31025 are also provided in Table 1 for comparison (ASME 2021c). The microstructure, grain size, hardness, and tensile properties of this plate is available in a 2017 report (Natesan et al. 2017). For the remainder of this report, this plate will be referred to as Commercial Heat 1.

#### 3.2 Commercial Heat 2

The second commercial heat of plate A709, heat 529900, was procured from ATI. In FY 2022, Code Case testing was primarily from plate CG05455 and a single fatigue test from plate CG05453 was conducted. These ESR plates were solution annealed at a minimum of 1149°C (2100°F) and precipitation treated for 10 hours at 775°C (1427°F) and subsequently air cooled. CG05455 is 1.806 in. thick while CG05453 is 2.057" thick. Both plates had the same chemical composition which is provided in Table 1. The chemistry requirements specified in SA-213 for UNS S31025 are also provided in Table 1 for comparison (ASME 2021c). The microstructure, grain size, hardness, and tensile properties of these plates are available in a 2022 report (Bass and Sham 2022). For the remainder of this report, both plates will be referred to as Commercial Heat 2.

Table 1. Chemistry composition, in weight percent, of Commercial Heat 1 and Commercial Heat 2 as well the chemistry requirements specified in ASME SA-213 for UNS S31025 (ASME 2021c).

	C	Mn	Si	P	S	Cr	Ni	Mo
Commercial Heat 1	0.066	0.90	0.38	0.014	0.001	20.05	25.14	1.51
Commercial Heat 2	0.08	0.9	0.39	0.003	< 0.001	19.9	24.6	1.5
UNS S31025	0.10 max	1.50 max	1.00 max	0.030 max	0.030 max	19.5 – 23.0	23.0 – 26.0	1.0 – 2.0

	N	Nb	Ti	Cu	Co	Al	В	Fe
Commercial Heat 1	0.152	0.26	0.01	0.06	0.02	0.02	0.0030	Bal.
Commercial Heat 2	0.15	0.17	< 0.01	0.06	0.02	0.02	0.004	Bal.
ASME SA- 213	0.10 – 0.25	0.10 – 0.40	0.20 max	_	-	_	0.002 - 0.010	-

Bal. = Balance

#### 4. FY 2022 CREEP CODE CASE TESTING

In FY 2022, a total of six creep-rupture tests were completed. Another 18 creep-rupture tests are currently in progress at the time of writing this report. These tests can be categorized by the Code Case the test supports; 1) preliminary, 2) 100,000-hour, 3) 300,000-hour, or 4) 500,000-hour. The maximum estimate rupture life for each of these categories is 11,000 hours, 25,000 hours, 68,000 hours, and 110,000 hours, respectively. A breakdown of these tests by category and heat are summarized in Table 2. INL has not been assigned to conduct any creep-ruptures tests to support the 300,000-hour nor the 500,000-hour Code Cases. Consequently, these two categories were not included in Table 2. The test conditions for each of these tests are described in Table 3.

Table 2. Summary of creep-rupture testing during FY 2022.

Category	Commercial Heat	Number of Tests Completed in FY 2022	Number of Tests in Progress at the Time of Writing this Report
Preliminary	1	3	5
	2	3	6
100,000 hours	1	0	3
	2	0	4

Table 3. Creep-rupture tests that transpired during FY 2022.

Commercial Heat	Temperature	spired during FY 2022.  Stress	Status	Sample ID				
Preliminary								
	750°C (1382°F)	65 MPa (9.4 ksi)	In progress	BCHT2-IC-02				
	775°C (1427°F)	65 MPa (9.4 ksi)	In progress	BCHT3-IC-01				
	800°C (1472°F)	45 MPa (6.5 ksi)	In progress	BCHT3-IC-02				
1	825°C (1517°F)	45 MPa (6.5 ksi)	In progress	BCHT3-IC-03				
1	850°C (1562°F)	45 MPa (6.5 ksi)	Complete	BCHT2-IC-03				
	825°C (1517°F)	35 MPa (5.1 ksi)	In progress	BCHT2-IC-06				
	1000°C (1832°F)	9 MPa (1.3 ksi)	Complete	BCHT2-IC-04				
	1000°C (1832°F)	8 MPa (1.2 ksi)	Complete	BCHT2-IC-05				
	650°C (1202°F)	175 MPa (25.4 ksi)	In progress	CG05455-11-AT51				
	650°C (1202°F)	155 MPa (22.5 ksi)	In progress	CG05455-11-AT52				
	800°C (1472°F)	65 MPa (9.4 ksi)	Complete	CG05455-11-AT43				
	825°C (1517°F)	35 MPa (5.1 ksi)	In progress	CG05455-11-AT50				
2	850°C (1562°F)	35 MPa (5.1 ksi)	In progress	CG05455-11-AT44				
	900°C (1652°F)	27 MPa (3.9 ksi)	Complete	GC05455-11-AT42				
	975°C (1787°F)	11 MPa (1.6 ksi)	In progress	CG05455-11-AT53				
	1000°C (1832°F)	9 MPa (1.3 ksi)	Complete	CG05455-11-AT45				
	1000°C (1832°F)	8 MPa (1.2 ksi)	In progress	CG05455-11-AT46				
		100,000 hour						
	675°C (1247°F)	110 MPa (16.0 ksi)	In progress	BCHT-IC-10				
1	700°C (1292°F)	82 MPa (11.9 ksi)	In progress	BCHT-IC-09				
	775°C (1427°F)	45 MPa (6.5 ksi)	In progress	BCHT2-IC-01				
	675°C (1247°F)	110 MPa (16.0 ksi)	In progress	CG05455-11-AT47				
2	700°C (1292°F)	82 MPa (11.9 ksi)	In progress	CG05455-11-AT48				
<i>L</i>	775°C (1427°F)	45 MPa (6.5 ksi)	In progress	CG05455-11-AT49				
	875°C (1607°F)	20 MPa (2.9 ksi)	In progress	CG05455-11-AT55				

Creep-rupture tests were conducted in accordance with ASTM E139, "Standard Test Methods for Conducting Creep, Creep-Rupture, and Stress-Rupture Tests of Metallic Materials" (ASTM 2011). Tests were also in accordance with PLN-3386, "Creep Testing" (Idaho National Laboratory 2016). Cylindrical test specimens were used with the longitudinal direction parallel to the rolling direction of the plate. Specimens were machined from the center of the plate with respect to the plate thickness for Commercial Heat 1. For Commercial Heat 2, specimens were machined from the one-quarter and three-quarters of the plate thickness. The machining of the mechanical test specimens for both commercial heats was in accordance with ASTM guidance (ASTM 2011). Specimens had a 6.35 mm (0.250 in.) minimum diameter and a reduced parallel section that was at minimum 31.75 mm (1.250 in). Test conditions were selected to generate creep-rupture data to cover a wide range of Larson-Miller parameters (Larson, Miller 1952). These conditions also took Section III, Division 5, Nonmandatory Appendix HBB-Y and Section II, Part D, Mandatory Appendix 5 into consideration (ASME 2021b, 2021a). As tests finish and new data becomes available, the growing data package is continuously evaluated for gaps, and if identified, requisite tests are added to the test plan.

#### FY 2022 FATIGUE CODE CASE TESTING

In FY 2022, a total of four fatigue tests were completed. At the time of writing the FY 2021 Code Case testing progress report, two fatigue tests were in progress (Rupp et al. 2021). These two tests finished in FY 2021. A summary of all six of these tests is provided in Table 4. Cycles to failure was determined use N<sub>20</sub>, which indicates a 20% load drop from the crack initiation point, or the point where load vs. cycle deviates from linear behavior.

Table 4. Summary of fatigue testing that transpired since the reporting of the FY 2021 Code Case testing

(Rupp et al. 2021).

Commercial Heat	Temperature	Total Strain Range	Status	Sample ID	Cycles to Failure (N <sub>20</sub> )
1	649°C (1200°F)	0.3%	Complete	BCHT-IF-5	3,614,560
	816°C (1501°F)	0.25%	Complete	BCHT-IF-15	1,206,978
2	650°C (1202°F)	1.0%	Complete	35A7*	1,517
	871°C (1600°F)	1.0%	Complete	CG05455- 13-AF26	755
	927°C (1701°F)	1.0%	Complete	CG05455- 13-AF23	826
	982°C (1800°F)	1.0%	Complete	CG05455- 13-AF22	743

<sup>\*</sup>From plate CG05453

Fatigue tests were conducted in accordance with ASTM E606-12, "Standard Test Method for Strain-Controlled Fatigue Testing" (ASTM 2012). Tests were also in accordance with PLN-3346, "Creep Fatigue Testing" (Idaho National Laboratory 2017). Testing was strain-controlled with a fully reversed (R = -1) triangular strain waveform with a 10<sup>-3</sup> s<sup>-1</sup> strain rate. Long tests were an exception to the 10<sup>-3</sup>s<sup>-1</sup> strain rate, as strain rate was increased when testing exceeded one million cycles. Cylindrical test specimens were used with the longitudinal direction parallel to the rolling direction of the plate.

Specimens were machined from the center of the plate with respect to the plate thickness for Commercial Heat 1. For Commercial Heat 2, specimens were machined from the one-quarter and three-quarters of the plate thickness. Specimens tested at INL had a 7.49 mm (0.29 in.) minimum diameter. The extensometer used at INL had a gauge length of 12 mm (0.47 in.). As fatigue tests finish and new data becomes available, the growing data package is continuously evaluated for gaps, and if identified, requisite tests are added to the test plan.

#### 6. FY 2022 CREEP-FATIGUE CODE CASE TESTING

In FY 2022, one creep-fatigue test was completed. Another test is currently in progress at the time of writing this report. Both tests are from Commercial Heat 1. A summary of these tests is provided in Table 5. Similar to the fatigue tests,  $N_{20}$  was used for cycles to failure.

Table 5. Creep-fatigue tests that transpired during FY 2022. Both specimens are from Commercial Heat 1.

Temperature	Total Strain Range	Hold Time	Status	Sample ID	Cycles to Failure (N <sub>20</sub> )
760°C (1400°F)	0.3%	0.5 hr	In progress	BCHT-IF-17	-
816°C (1501°F)	0.3%	0.5 hr	Complete	BCHT-IF-16	10,492

Creep-fatigue tests were conducted in accordance with ASTM E2714-13, "Standard Test Method for Creep-Fatigue Testing," and ASTM E606-12 (ASTM, 2013; 2012). Tests were also in accordance with PLN-3346 (Idaho National Laboratory, 2017). Creep-fatigue testing uses the same setup and procedures as fatigue testing. The difference between creep-fatigue and fatigue testing is the addition of a hold time to the strain waveform at the maximum tensile strain. Test conditions were selected to cover a spectrum of temperatures, total strain ranges, and hold times. This is necessary to establish the creep-fatigue damage envelope. As creep-fatigue tests finish and new data become available, the growing data package is continuously evaluated for gaps, and if identified, requisite tests are added to the test plan.

#### 7. SUMMARY AND ONGOING WORK

A709 is an advanced material that offers many advantages over 316H SS and would improve the viability of SFRs. Consequently, it has been identified as the next alloy to be qualified in Section III, Division 5 of the ASME BPVC. The A709 Code Case data package is currently being developed through a collaboration between ANL, INL, and ORNL. An update is provided on the status of creep, fatigue, and creep-fatigue testing that transpired during FY 2022 at INL. Preliminary results continue to demonstrate the improved creep and fatigue resistance of A709 compared to 316H SS.

In FY 2021, INL placed a purchase order with ATI to procure the third commercial heat of plate A709. The A709 plates, heat 530843, were delivered by ATI to ORNL in April 2022. This heat is currently being sectioned into smaller pieces by waterjet cutting. The heat will then be distributed among ANL, INL, and ORNL. This heat will be characterized and code case testing will be initiated in FY 2023. Code Case testing will include creep, fatigue, and creep-fatigue testing. This testing will be in conjunction with the creep and cyclic tests that are currently in progress at the time of writing this report.

#### 8. REFERENCES

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