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Progress Report on Alloy 709 Base Metal Code Case Testing at ORNL in FY 2023

Yanli Wang
Peijun Hou

July 2023

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Materials Science and Technology Division

PROGRESS REPORT ON ALLOY 709 BASE METAL CODE CASE TESTING AT ORNL IN FY 2023

Yanli Wang
Peijun Hou*

* Imtech Corporation, Knoxville

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ABBREVIATIONS

Argonne	Argonne National Laboratory
ART	Advanced Reactor Technologies
ASME	American Society of Mechanical Engineers
ASTM	American Society for Testing and Materials
ATI	Allegheny Technologies Incorporated
CC	Code Case
CF	Creep-fatigue
DOE	US Department of Energy
ESR	Electroslag Remelting
FR	Fast Reactors
INL	Idaho National Laboratory
LMP	Larson-Miller parameter
ORNL	Oak Ridge National Laboratory
PT	Precipitation Treatment
RT	Room temperature

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ABSTRACT

A collaborative research and development effort in support of the Alloy 709 Code Case qualification in the American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code, Section III, Division 5, High Temperature Reactors is being carried out at Oak Ridge National Laboratory (ORNL), Idaho National Laboratory (INL) and Argonne National Laboratory (Argonne).

In FY 2023, ORNL has continued to conduct a subset of the Code Case testing for tensile, creep rupture, fatigue and creep-fatigue on Alloy 709. This report also updates the key Code Case testing status and results on the first two commercial heats and the preliminary results on the third commercial heat of Alloy 709. The three commercial heats of Alloy 709 are all in plate product form.

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1. INTRODUCTION

Nuclear energy, as one of the leading energy sources reducing carbon emissions, is desirable for applications of producing electricity and facilitating a net-zero electric-industry sector. Sodium Fast Reactor (SFR), with its ability to improve thermal efficiency as compared with light-water reactors, is becoming one of the leading advanced reactor concepts. While SFR technology is relatively mature, there must be improvements in its capital cost and economic return before the private sector invests in large-scale, commercial deployment of SFRs. In particular, advances in the mechanical performance of structural material are critical to realize the improvement in the economics of fast reactors. One of the objectives of the Advanced Materials Development activities of the Advanced Reactor Technologies (ART) Program for the US Department of Energy (DOE), Office of Nuclear Energy (NE) is to provide the technical basis needed to support the regulatory requirements for structural materials for advanced reactors that could be deployed in the near-term to mid-term by the US nuclear industry.

Through a DOE-NE ART material down-selection and intermediate term testing program, Alloy 709, an advanced austenitic stainless steel, was recommended as a Class A construction material for the American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code, Section III, Division 5 (ASME, 2021a) in support of SFR structural applications because of its overall superior structural strength advantage (Sham, et al, 2022). A comprehensive Code qualification plan was developed to generate the data package and to develop material-specific design parameters required for Class A component design in ASME Section III, Division 5. A multi-laboratory advanced materials development effort, involving ORNL, INL and Argonne, was carried out to investigate the mechanical performance of Alloy 709 in support of its codification process.

The data package for the ASME Code Cases requires the evaluation of mechanical properties, including tensile, creep, fatigue, and creep-fatigue from base metal as well as additional tests on its weldments. To this end, this report summarizes the progress on the Code Case testing of the first two commercial heats of Alloy 709 base metal at ORNL in FY 2023. This report also provides the preliminary test results on the third commercial heat of Alloy 709 plates.

2. MATERIAL

The data package for code qualification must contain a minimum of three commercial heats which represent the anticipated compositional ranges. In collaboration with two US steel fabricators, the DOE-NE ART Program has successfully scaled up the production of Alloy 709 in rolled plate form from a laboratory heat of 500 lb to three Alloy 709 commercial heats, with heat sizes of 45,000, 41,000 and 38,000 lb. The commercial Heat 1 plates were fabricated by G.O. Carlson Inc of Pennsylvania, whereas the second and third heats were both fabricated by Allegheny Technologies Incorporated (ATI) Flat Rolled Products.

The plates used in the Code Case testing were produced by argon-oxygen-decarburization (AOD) followed by electroslag remelted (ESR). The plates were hot rolled, and solution annealed at a minimum temperature 1150°C. An additional precipitation heat treatment of the plates was performed to ensure a balanced creep and creep-fatigue (CF) performance (McMurtrey, et al, 2019). The precipitation heat treatment protocol is 775°C for 10 h in air, followed by air cooling.

In this report, the Code Case testing results from commercial Heat 1 and commercial Heat 2 with PT condition are summarized. The preliminary results on commercial Heat 3 plate are also presented. The Code Case testing results from precipitation heat treated specimens are marked with '-PT', whereas those from solution annealed specimens are marked with '-SA'.

Table 1 summarizes the information regarding these three commercial heats of Alloy 709 plates. The specimens used for Code Case testing were machined at the mid-thickness for the commercial Heat 1 plates and at the 1/4-thickness for both commercial Heat 2 and 3 plates. All specimens were along the rolling direction. It is noted that plate CG05368 and CG05453 from commercial Heat 2 was added for use of for a subset of the Code Case testing at ORNL.

Table 1. Three commercial heats of Alloy 709 in plate form for Code Case testing

<table>
<tr><th>Material</th><th>Fabricator</th><th>Master heat number</th><th>Plate IDs for base metal Code Case testing</th><th>Nominal ASTM grain size number</th><th>Plate thickness, (in)</th></tr>
<tr><td>Commercial Heat 1</td><td>G.O. Carlson Inc</td><td>58776</td><td>58776-3RBC1</td><td>7</td><td>1.10</td></tr>
<tr><td rowspan="2">Commercial Heat 2</td><td rowspan="3">Allegheny Technologies Incorporated</td><td rowspan="2">529900</td><td>CG05455; CG05368</td><td>7</td><td>1.81</td></tr>
<tr><td>CG05453</td><td>4</td><td>2.00</td></tr>
<tr><td>Commercial Heat 3</td><td>530843</td><td>CG45192</td><td>4</td><td>1.82</td></tr>
</table>

The chemical compositions of the three heats of Alloy 709 with the heat numbers 58776, 529900 and 530843 are listed in Table 2. For comparison, the specifications for the chemical requirements of Nippon Steel NF709, TP310MoCbN, seamless tubing, with a UNS number of S31025 in ASME SA-213 (ASME, 2021b) are also listed in Table 2. The three commercial heats Alloy 709 plates met the specified NF709 chemical requirements.

Table 2. Chemical compositions of the two commercial heat Alloy 709 plates with heat number 58776, 529900 and 530843 (wt %).

	Commercial Heat 1	**Commercial Heat 2**	**Commercial Heat 3**	**ASME SA-213 UNS-S31025 Specification**
Element	**heat number 58776**	**heat number 529900**	**heat number 530843**	**---**
C	0.066	0.08	0.07	0.10 max
Cr	20.05	19.9	19.8	19.5–23.0
Co	0.02	0.02	0.01	–
Ni	25.14	24.6	25	23.0–26.0
Mn	0.9	0.9	0.9	1.50 max
Mo	1.51	1.5	1.5	1.0–2.0
N	0.152	0.15	0.15	0.10–0.25
Si	0.38	0.39	0.44	1.00 max
P	0.014	0.003	0.008	0.030 max
S	0.001	<0.001	0.001	0.030 max
Ti	0.01	<0.01	< 0.01	0.20 max
Nb	0.26	0.17	0.18	0.10–0.40
Al	0.02	0.02	0.02	–
B	0.003	0.004	0.005	0.002–0.010
Cu	0.06	0.06	0.04	–

3. TENSILE CODE CASE TESTING

3.1 TENSILE CODE CASE TESTING

Figure 1 shows the geometry of tensile specimen tested at ORNL, and the loading direction is along the rolling direction of the Alloy 709 plates. During the FY2023 report period, ORNL performed tensile tests on the commercial Heat 2 & 3 plates in both precipitation treatment (PT) and solution annealing (SA) conditions. The testing temperature of the tensile results was ranging from room temperature (RT) to 950°C. The tensile testing procedure followed ASMT E8/E8M (ASTM, 2022a) at RT and ASTM E21 (ASTM, 2021) at elevated temperatures.

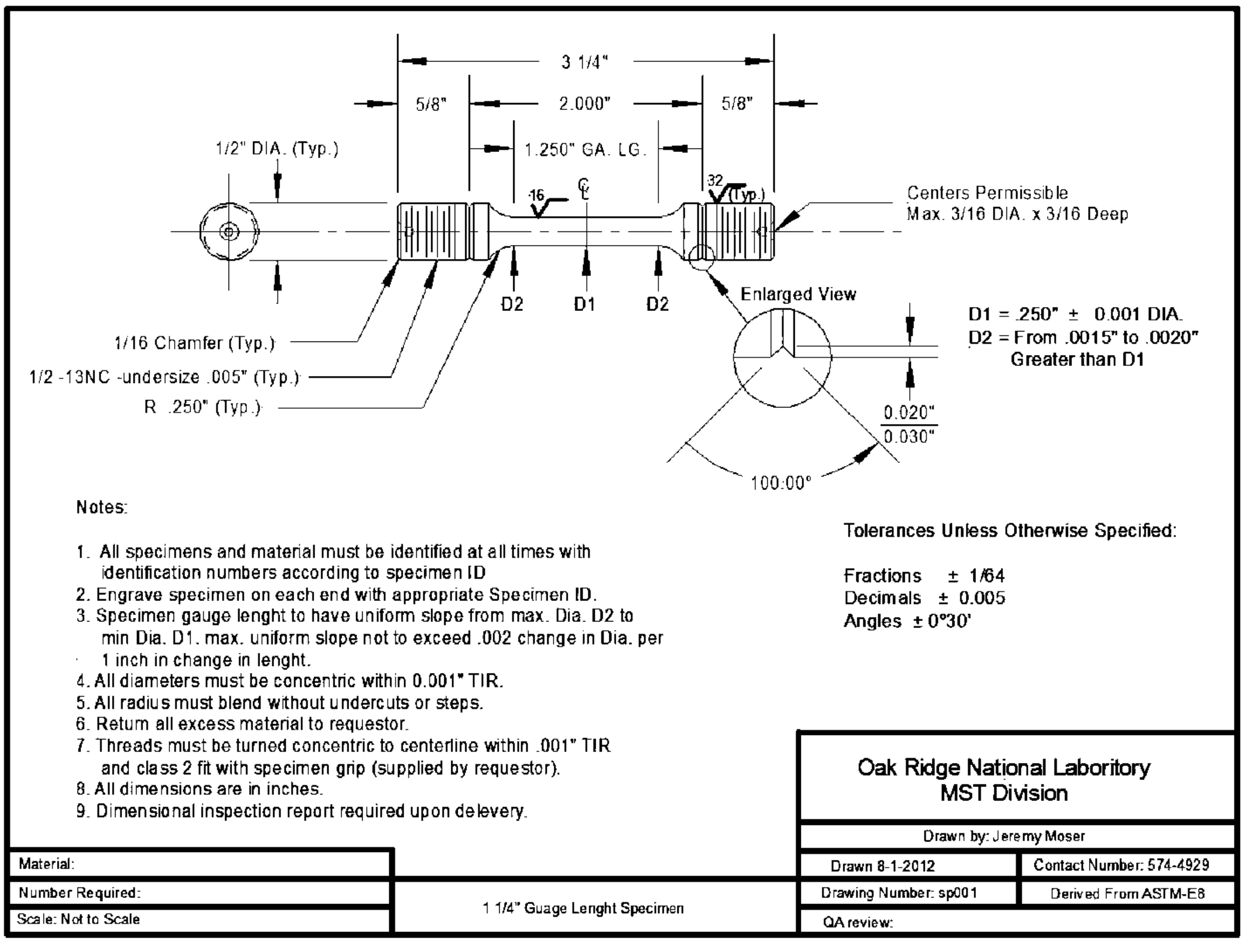

Figure 1. Standard tensile specimen geometry at Oak Ridge National Laboratory (ORNL). Dimensions are in inches.

3.2 TENSILE RESULTS

Table 3 summaries the tensile properties at RT, including the yield strength, σ_Y, taken as the stress at 0.2% strain offset, the ultimate tensile strength (UTS), and the total elongation, e_t. Also, the minimum specified RT tensile properties of Alloy 709 per ASME SA-213 UNS-S31025 specification (ASME, 2021b) are listed in Table 3. It confirms that all the plates from commercial heats of Alloy 709 in PT and SA conditions in this report meet ASME SA-213 requirements.

The tensile testing parameters and results at elevated temperatures for the Alloy 709-PT and -SA conditions are summarized in Table 4 and Table 5, respectively. The tensile properties reported in these tables include yield strength at 0.2% strain offset, σ_Y, the ultimate tensile strength (UTS), the uniform elongation, e_u, total elongation, e_t, and reduction-of-area (ROA).

Table 3. Room temperature tensile properties of Alloy 709 in PT and SA conditions.

Material	Plate ID	Material condition	σ_Y (MPa)	UTS (MPa)	e_t (%)
Commercial Heat 3	CG45192	SA	296	681	49
		PT	305	694	44
			306	691	44
Commercial Heat 2	CG05368	SA	302	685	50
			306	692	49
	CG05453		327	679	48
ASME SA-213 UNS-S31025 specification			**270 minimum**	**640 minimum**	**30 minimum**

Table 4. Tensile properties of commercial heat 3 Alloy 709 plate CG45192 in PT condition.

Number	Temperature (°C)	σ_Y (MPa)	UTS (MPa)	e_u (%)	e_t (%)	ROA (%)
1	100	261	625	37	43	47
2	200	222	578	38	42	50
3	600	187	508	39	45	46
4	600	185	508	39	45	45
5	800	165	300	18	55	75
6	950	97	135	3.5	97	94
7	950	98	135	3.5	97	96

Table 5. Tensile properties of Alloy 709 in SA condition.

Number	Plate ID	Temperature (°C)	σ_Y (MPa)	UTS (MPa)	e_u (%)	e_t (%)	ROA (%)
8	CG45192	600	168	517	44	52	67
9	CG45192	800	159	313	21	54	78
10	CG45192	950	100	137	3.5	88	98
11	CG05368	100	243	623	50	40	70
12	CG05368	100	247	623	40	50	69
13	CG05368	200	215	591	39	47	71
14	CG05368	200	212	585	40	48	65
15	CG05368	300	205	587	41	49	65
16	CG05368	600	167	531	47	57	74
17	CG05368	600	168	532	46	57	64
18	CG05368	800	154	304	21	60	80
19	CG05368	800	161	304	19	61	59
20	CG05368	950	95	132	4.0	107	98
21	CG05368	950	94	131	4.0	108	98
22	CG05453	200	170	302	19	52	77
23	CG05453	600	192	520	42	49	59
24	CG05453	950	104	136	3.5	71	96

The tensile properties as a function of the testing temperature are presented in Figure 2. As a comparison, the previous ORNL tensile Code Case testing results on the commercial Heat 1 and 2 in PT are presented in these plots. Generally speaking, PT slightly increased the yield strength and UTS and reduced the elongation at at testing temperatures of 600 °C and below. The most significant effect of PT is on the decrease of the reduction-of-area at 600 °C and below.

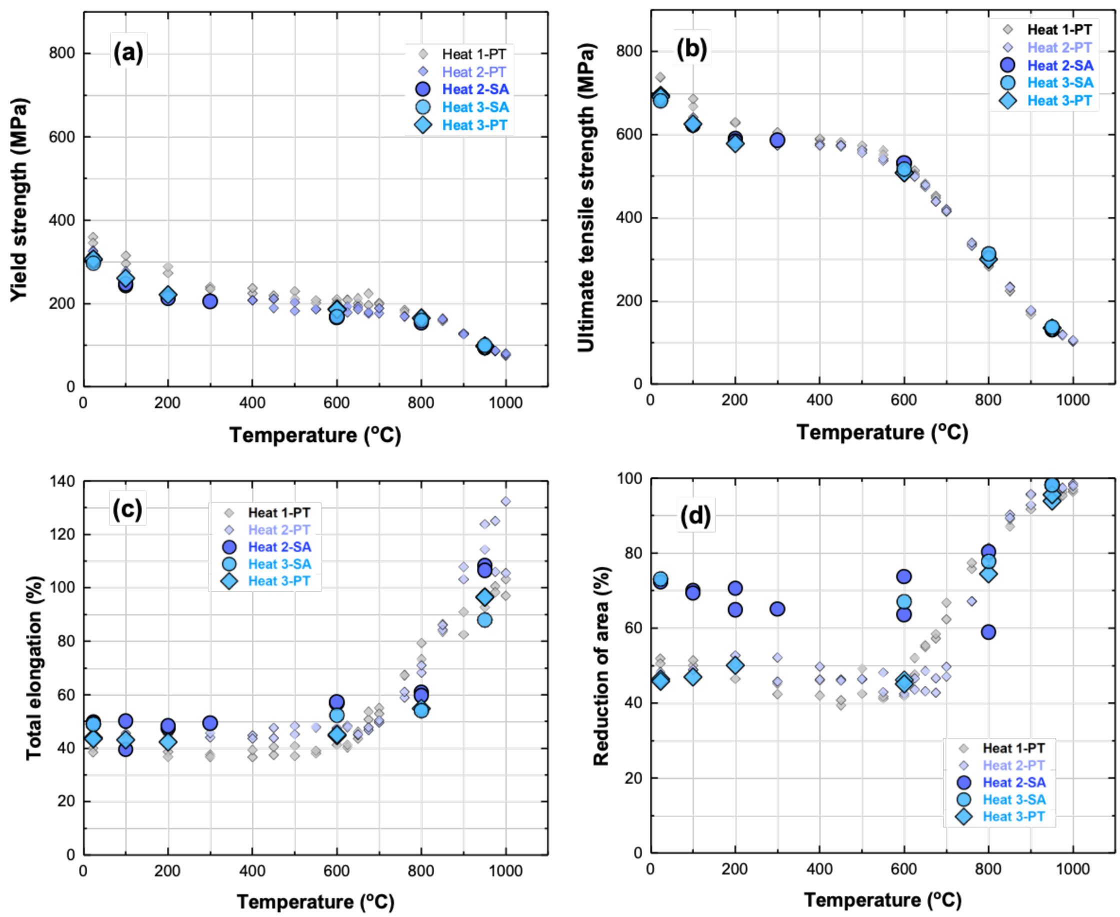

Figure 2. Tensile properties of the three commercial heats of Alloy 709 with (a) Yield strength, (b) UTS, (c) total elongation, and (d) reduction of area.

4. CREEP CODE CASE TESTING

A comprehensive master creep testing matrix for Alloy 709 Code Case testing was developed for commercial Heat 1 and commercial Heat 2 plates in PT condition. The preliminary creep testing matrix for commercial Heat 3 in PT condition was developed and added to the testing plan in FY 2023. The testing activities and research to support the qualification of Alloy 709 are being carried out at Argonne, INL, and ORNL. The creep Code Case testing conditions in terms of stress and temperatures for the three heats of Alloy 709 are presented in Figure 3.

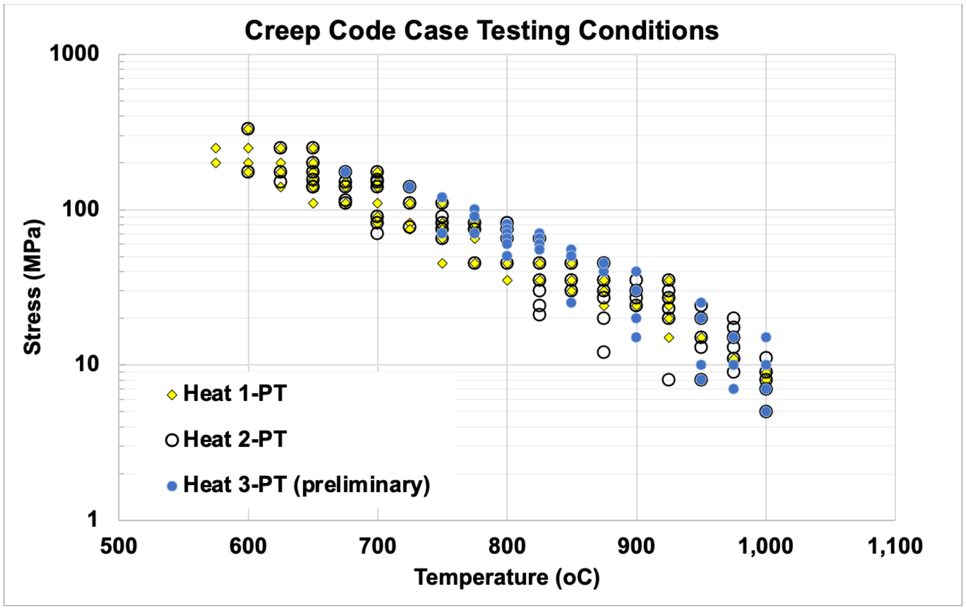

Figure 3. Alloy 709 creep Code Case Testing conditions

The creep testing matrix was used to generate data in support of the development of a series of Code Cases including preliminary CC, 100,000 h Code Case (100K CC), 300,000 h Code Case (300K CC), and 500,000 h Code Case (500K CC). The creep Code Case testing plan, the corresponding supporting creep rupture data and responsible laboratories are summarized in Table 6. ORNL is tasked to carry out all the long-term creep rupture tests in support of the 300K CC and 500K CC in addition to a subset of the tests designed for the development of the preliminary CC and 100K CC.

Table 6. Creep Code Case testing on the two commercial heats of Alloy 709 in PT condition

Target Code Case	Supporting creep rupture data (h)	Labs involved
Preliminary	500–10,000	Argonne/INL/ ORNL
100K CC	15,000–24,000	Argonne/INL/ ORNL
300K CC	25,000–68,000	ORNL
500K CC	91,000–109,000	ORNL

4.1 CREEP RUPTURE CODE CASE TESTING AT ORNL

The specimen geometry for intermediate and long-term Code Case testing at ORNL is shown in Figure 4. The creep specimen was designed to have a 9.53 mm (0.375 in) gage diameter with a nominal gage length of 47.63 mm (1.875 in). Note that the larger-than-normal 6.35 mm (0.25 in) diameter creep specimen geometry was used to reduce the oxidation effect during long-term creep testing. Several short-term creep tests used the standard specimen geometry shown in Figure 1.

The creep Code Case testing procedure followed ASTM E 139 (ASTM, 2018), *Standard Test Methods for Conducting Creep, Creep-Rupture, and Stress-Rupture Tests of Metallic Materials*. The creep test schedule was arranged to best utilize the individual creep machine capacity and estimated testing duration.

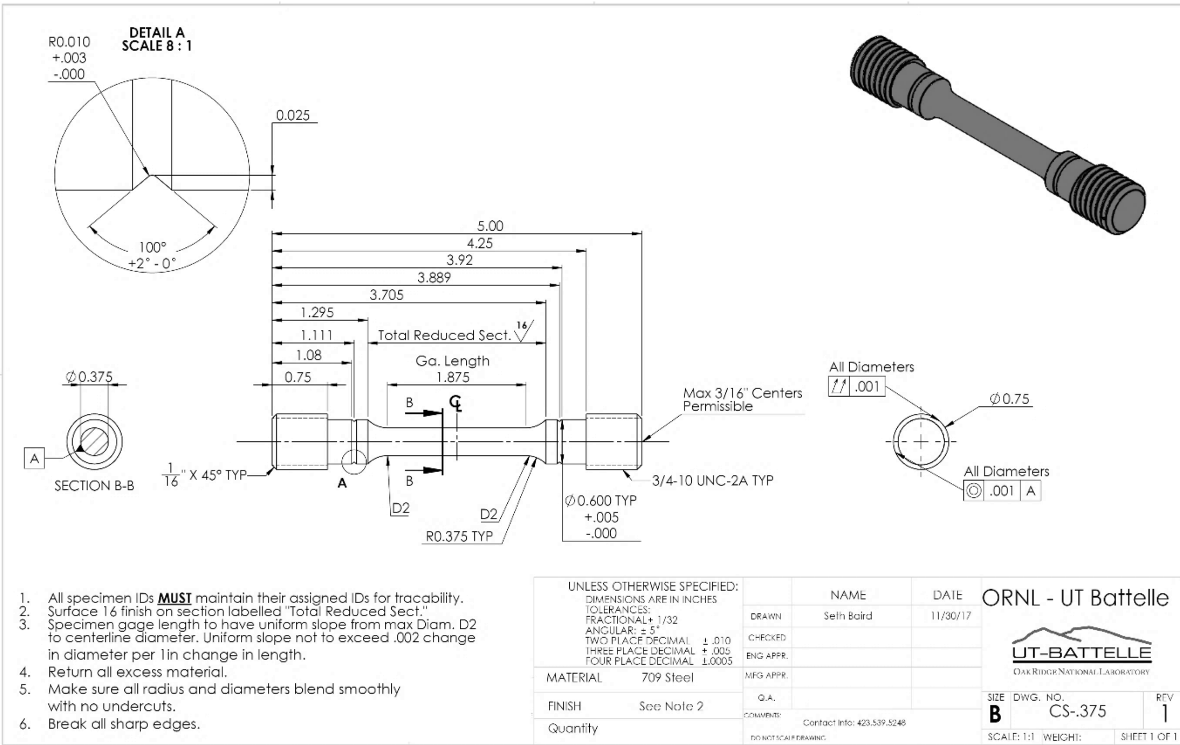

Figure 4. Creep specimen geometry for Alloy 709 Code Case testing at ORNL. Dimensions are in inches.

ORNL, INL and Argonne have collectively generated a total of 95 creep rupture data with 54 on commercial Heat 1-PT condition and 41 on commercial Heat 2-PT condition. To make a preliminary assessment of the creep resistance of the Alloy 709 material, the rupture data generated on the two commercial Heats of Alloy 709 in PT conditions were assembled and the results are presented in the form of Larson-Miller relationship in Figure 5. The 39 rupture data points generated at ORNL are highlighted in green.

The results from the current Larson-Miller analysis are consistent with previous results and the commercial Heat 2-PT condition continues to show slightly stronger in creep strength that the commercial Heat-1-PT condition, especially at lower stress levels.

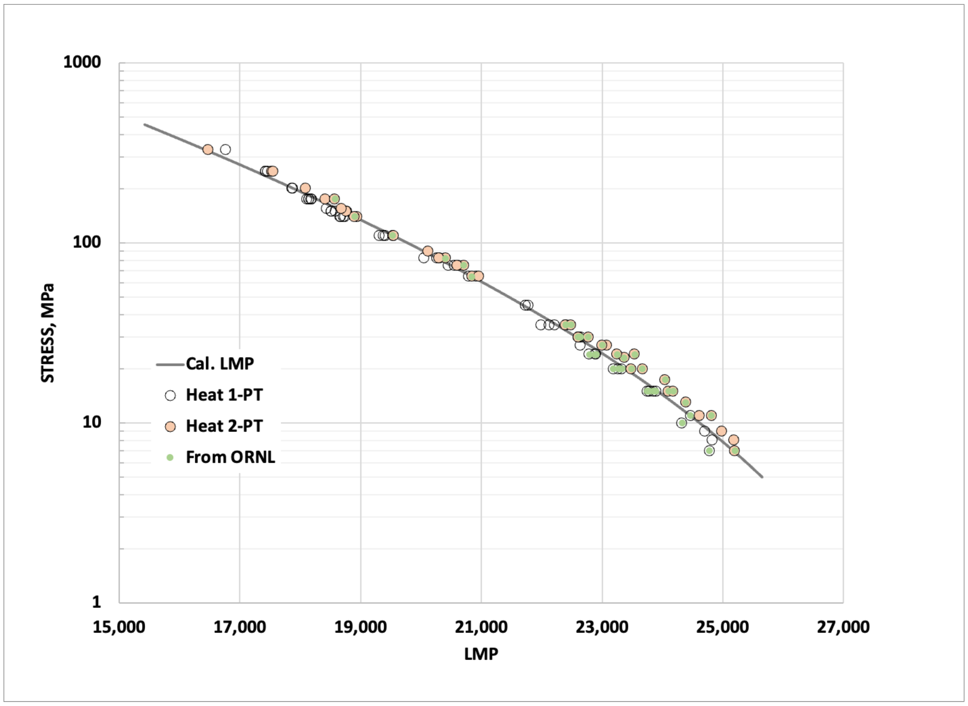

Figure 5. Creep rupture data from the two commercial heats of Alloy 709 in PT condition collected to date on the LMP-stress plot.

5. FATIGUE AND CREEP-FATIGUE CODE CASE TESTING

5.1 FATIGUE AND CREEP-FATIGUE CODE CASE TESTING AT ORNL

In FY 2023, strain-controlled fatigue tests and creep-fatigue tests continued at ORNL in support of the development of the temperature-dependent fatigue design curves and creep-fatigue interaction damage diagram, as a part of the data package in developing the Alloy 709 Code Case.

The geometry of standard fatigue and creep-fatigue specimen tested at ORNL is shown in Figure 6. The specimen has a gauge diameter of 6.35 mm (0.25 in) and a 19.05 mm (0.75 in) gauge length.

The testing procedure for strain-controlled fatigue and creep-fatigue are as follows:

- The fatigue testing followed the ASTM E606 (ASTM, 2021b) standard for conducting strain-controlled fatigue tests. The strain rate was controlled at 1E-3 s^{-1}. A triangular loading waveform with a fully reversed profile, i.e., a loading ratio in strain of $R = -1$, was employed.

- The creep-fatigue testing followed the ASTM E606 (ASTM, 2021b) and/or ASTM E2714-13 (ASTM, 2020) standard for conducting creep-fatigue testing under strain-controlled condition. The loading waveform was fully reversed with various hold-times applied at the peak tensile strain. The loading and unloading strain rate were controlled at 1E-3 s^{-1}.

Additionally, at strain range below 0.3%, one fatigue test was performed under load-controlled mode with the cycling frequency increased to 2.5Hz to generate fatigue failure data. The purpose of this load-controlled test was to assess the fatigue strength at the low strain range and high cycle region within a reasonable amount of the testing duration. Extensometer was used during the load-controlled test to record the strain.

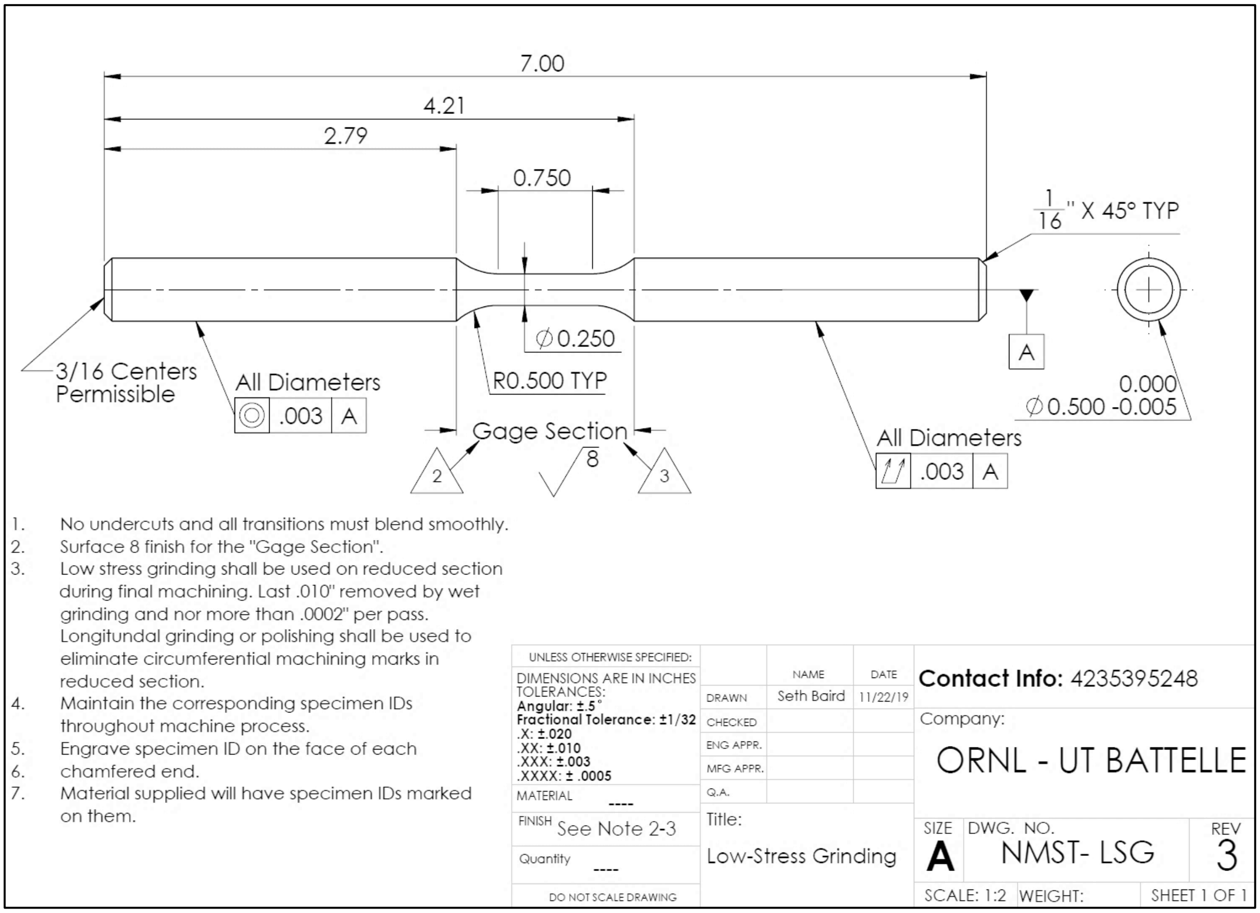

Figure 6. Standard fatigue and CF specimen geometry at Oak Ridge National Laboratory (ORNL). Dimensions are in inches.

To develop the temperature-dependent fatigue design curves and creep-fatigue interaction damage diagram, the cycles to failure are required to be extracted from the test data. In this report, the failure criterion is defined as a 20% drop in the ratio of the maximum stress to the minimum stress as a function of the applied cycles. Figure 7 schematically illustrates the failure criterion and the approach to determine the cycle to failure. There were cases where the specimens failed to meet the 20% drop criterion, and the recorded failure cycle count is reported as a reference. For the final data package in developing the Alloy 709 Code Case, the fatigue and creep-fatigue test results will be further screened to exclude data that are not qualified for ASME code development.

In FY2023, a total of 22 fatigue failure data and 12 creep-fatigue tests to failure data were completed on the three commercial heats of Alloy 709 in PT condition at ORNL. To obtain better assessment of the creep-fatigue properties of Alloy 709 and to support the Code Case data package, a comprehensive creep-fatigue testing plan were developed and updated (Sham et al, 2022). ORNL has adjusted the testing plan accordingly and continued the Code Case testing on the three commercial heats of Alloy 709 in PT condition.

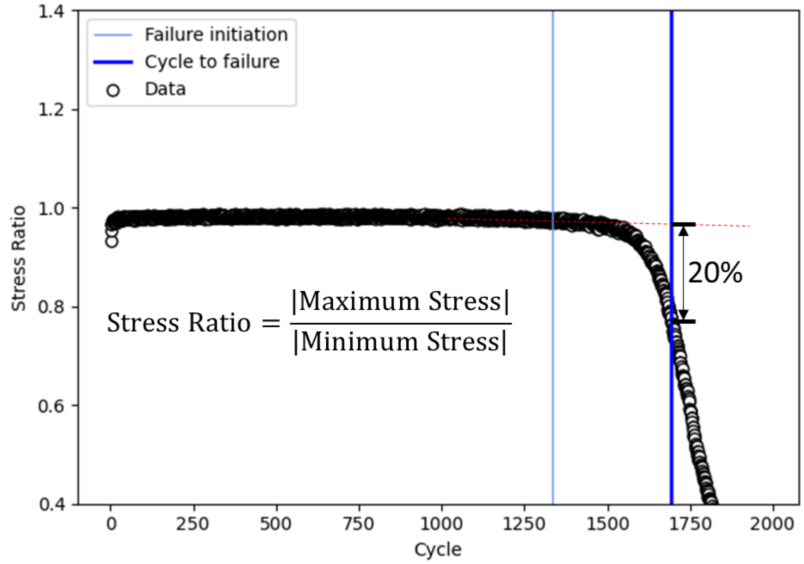

Figure 7. Failure criterion in identifying the cycles to failure for fatigue and creep-fatigue tests.

5.2 FATIGUE AND CREEP-FATIGUE CODE CASES TESTING RESULTS

Fatigue and creep-fatigue Code Case testing are being performed at ORNL and INL on Alloy 709 in PT condition in support of the Code Case development. In FY 2023, the testing matrices have been updated by Sham et al. (2022) based the data generated thus far. ORNL is tasked to continue the fatigue testing effort in support of the development of the full fatigue design curves, and the creep-fatigue testing for generating the interaction damage diagram. The testing and evaluation on the commercial heat 3 plate CG45192 in PT condition have been started at ORNL.

The fatigue testing data generated to date on the three commercial heats of Alloy 709 in PT condition are collected in Figure 8.

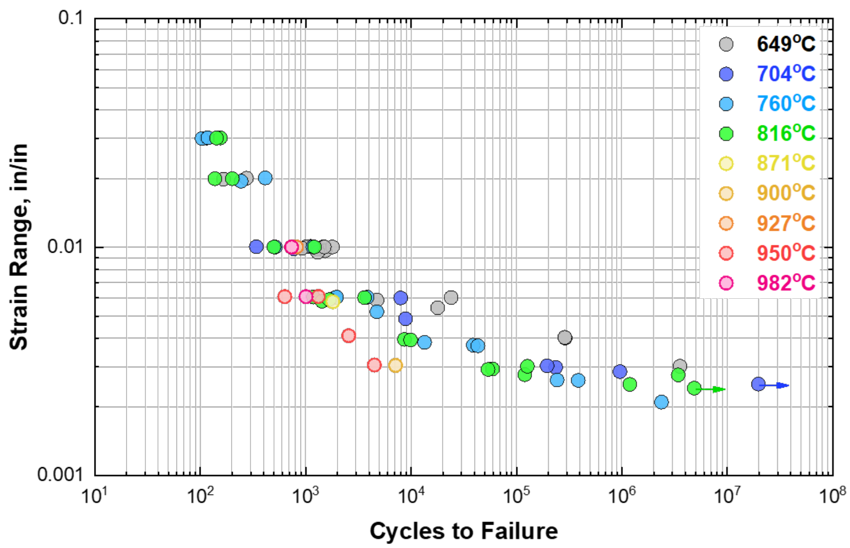

Figure 8. Strain-controlled fatigue data generated on Alloy 709 in PT condition.

To evaluate the effect of hold time on cycle life, Figure 9 presents creep-fatigue results in comparison to pure fatigue results at temperatures of 649°C, 704°C, 760°C, 816°C, and 950°C. When comparing creep-fatigue to pure fatigue, the hold time generally reduces the cycles to failure. However, further increase of the hold time from 1800s to 3600s does not affect cycles to failure significantly, especially for the data collected at larger strain range of 1.0%. The impact of hold time effect on the reduction of cycles to failure is more pronounced at low strain ranges.

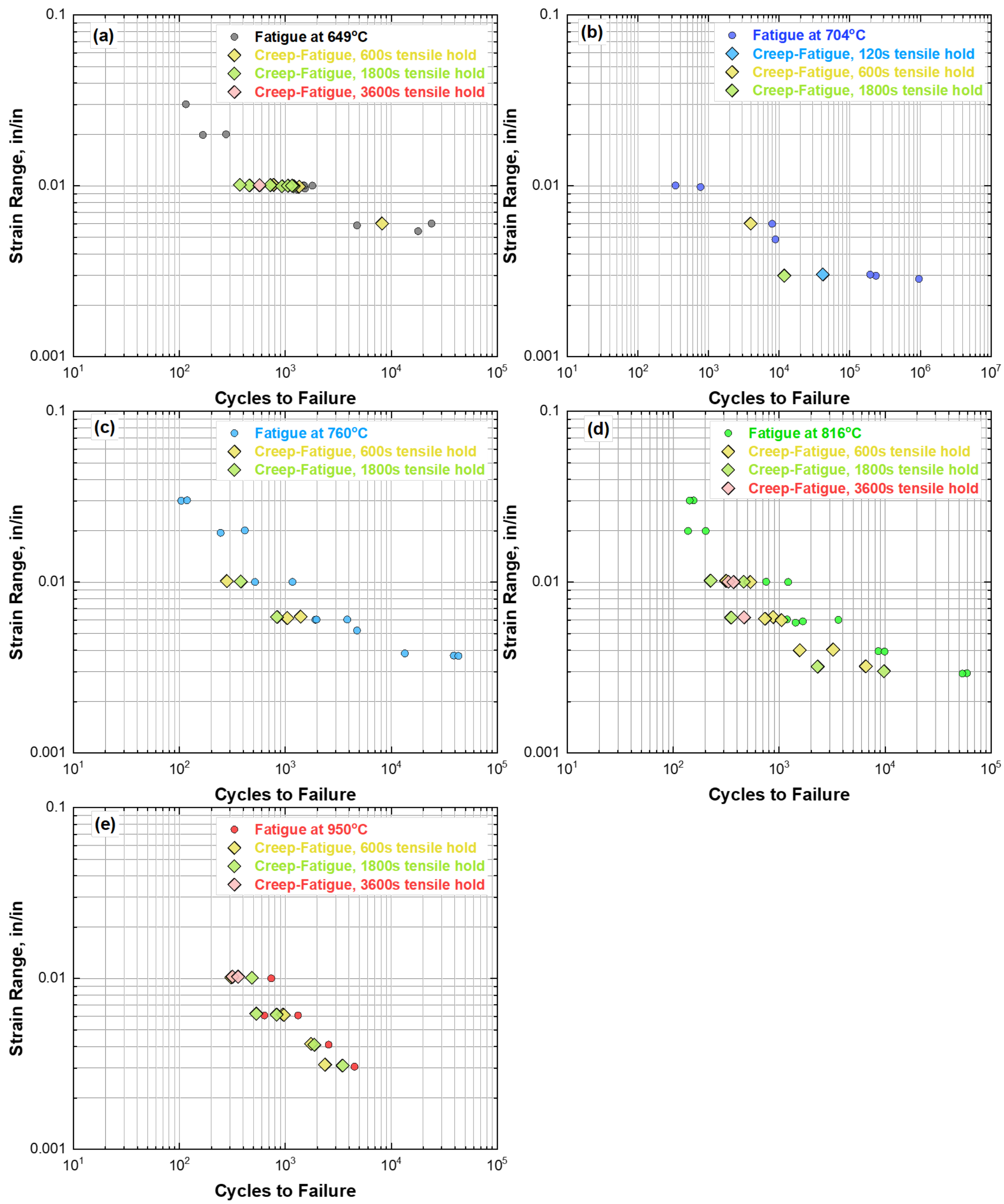

Figure 9. Fatigue and creep-fatigue results on Alloy709 in PT condition at temperatures of 649°C, 704°C, 760°C, 816°C, and 950°C.

The influences of temperature on pure fatigue and temperature and hold time on creep-fatigue life cycles are demonstrated in different type of plots in Figure 10, where the fatigue and creep-fatigue cycles to failure at strain ranges of 1.0%, 0.6%, and 0.3% are presented as a function of testing temperature. An insignificant impact of testing temperature on the fatigue and creep-fatigue failure cycles at 1% strain range was observed in Figure 10a, whereas the increase of temperature significantly reduced the fatigue cycles at 0.6% strain range and more so at the low strain range of 0.3%, in Figure 10b and Figure 10c. At testing temperatures of 760 °C and above, creep-fatigue testing at the same loading conditions does not affect the life cycles significantly based these limited data.

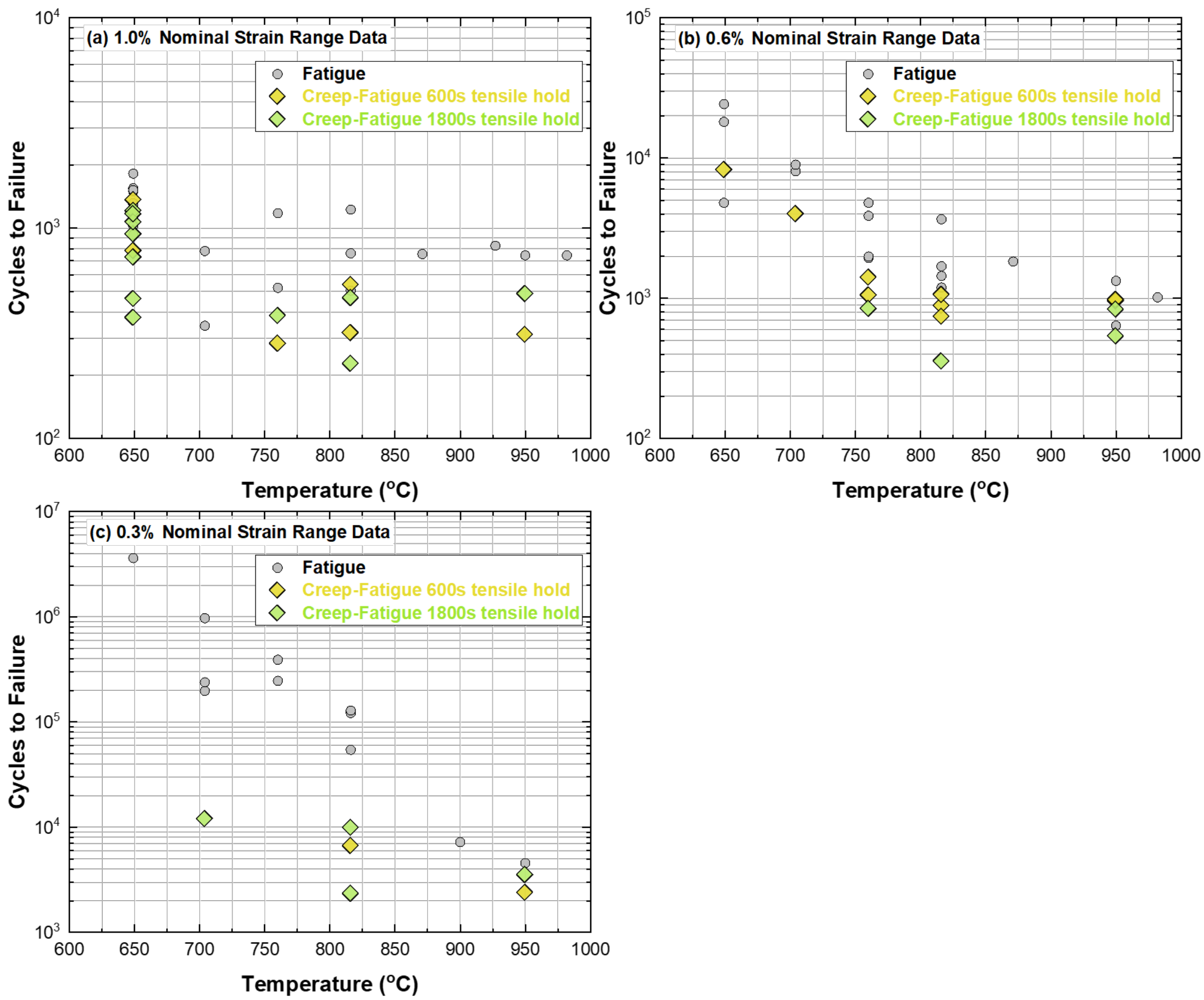

Figure 10. Effect of the testing temperature on fatigue and creep-fatigue failure cycles at strain ranges of 1.0%, 0.6% and 0.3%.

Fatigue and creep-fatigue data collected in FY 2023 and all the previous test results on Alloy 709 in PT condition were analyzed to update the damage diagram, or D-diagram. In this analysis,

- The fatigue-damage fraction for the D-diagram is calculated as the ratio of the cycles to failure of the creep-fatigue tests to those of the pure fatigue tests conducted under the same strain range, strain rate, and temperature. Note that the average cycles to failure from duplicate tests are adopted for the calculation of the fatigue-damage, although there are very limited number duplicates generated so far.

- The creep damage fraction is determined based on the time-fraction method using the stress relaxation curve at each cycle during the tensile hold in the CF tests and the time-to-rupture Larson-Miller correlation from the creep-rupture data, similar to the approach used in Wright, et al (2021). It is noted the all the creep-fatigue cycles, instead of the single mid-life cycle approach, are used for this analysis.

The results are presented in Figure 11 with the damage fractions in logarithm's scale. In this plot, three bi-linear creep-fatigue interaction envelopes are added for reference. It should be noted that the bi-linear envelope does not represent a lower bound of the creep-fatigue interaction data from lab scale test specimens, but as a trend of the creep-fatigue interaction for use in the Division 5 creep-fatigue evaluation procedure. The design margins are embedded in other parts of the creep-fatigue evaluation procedure.

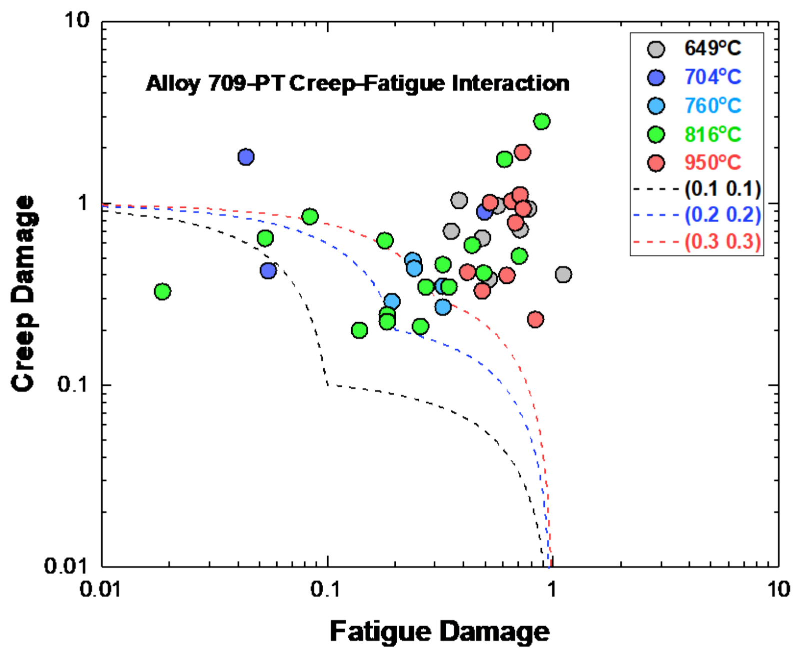

Figure 11. Creep-fatigue interaction damage diagram for Alloy 709 in PT condition.

6. SUMMARY

The Code Case testing effort in support of the qualification of Alloy 709, an advanced austenitic stainless steel in the ASME Boiler and Pressure Vessel Code, Section III, Division 5, High Temperature Reactors continued at ORNL in FY 2023. This report summarized Alloy 709 Code Case testing collected at ORNL, including tensile Code Case testing data of Alloy 709 in PT and SA conditions, creep rupture, fatigue and creep-fatigue Code Case testing data of Alloy 709 in PT condition from three commercial heats of Alloy 709 in plate product form. The data generated to date on Alloy 709 supports the recommendation for its ASME Code qualification in an effort to reduce construction and operating costs for advanced reactor deployment.

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