A709 Qualification Plan Update and Mechanical Properties Data Assessment

May 2022

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

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SUMMARY

This report provides a summary of the development effort for the qualification of Alloy 709, an advanced austenitic stainless steel in the American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code, Section III, Division 5, High Temperature Reactors. It provides an assessment of the mechanical properties data generated to date from the tensile, creep, fatigue, and creep-fatigue tests of the precipitation-treated Alloy 709 from two commercial heats in plate product form.

It was concluded that the mechanical properties of Alloy 709 with the precipitation treatment continued to outperform those of Type 316 stainless steel. This affirms the recommendation for its Code qualification to support the effort to reduce construction and operating costs for advanced reactor deployment.

The data also demonstrated that the precipitation treatment is effective in enhancing the creep-fatigue resistance of Alloy 709 while maintaining a significant creep strength advantage over Type 316 stainless steel.

This report also provides an update to the test conditions for the creep, fatigue, and creep-fatigue test matrices in order to cover the Code Case data package more effectively.

Finally, it is recommended to continue the Alloy 709 Code Case Testing Program to develop the data package needed for the determination of the material-specific design parameters for inclusion in the Alloy 709 Code Case.
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ACKNOWLEDGMENTS

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<tr>
<th>ACRONYMS</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>AGR</td>
<td>Advanced Gas-cooled Reactor</td>
</tr>
<tr>
<td>ANL</td>
<td>Argonne National Laboratory</td>
</tr>
<tr>
<td>AOD</td>
<td>Argon-Oxygen-Decarburization</td>
</tr>
<tr>
<td>ART</td>
<td>Advanced Reactor Technologies</td>
</tr>
<tr>
<td>ASME</td>
<td>American Society of Mechanical Engineers</td>
</tr>
<tr>
<td>ASTM</td>
<td>American Society for Testing and Materials</td>
</tr>
<tr>
<td>CC</td>
<td>Code Case</td>
</tr>
<tr>
<td>DOE</td>
<td>Department of Energy</td>
</tr>
<tr>
<td>ESR</td>
<td>Electroslag Remelting</td>
</tr>
<tr>
<td>FR</td>
<td>Fast Reactors</td>
</tr>
<tr>
<td>INL</td>
<td>Idaho National Laboratory</td>
</tr>
<tr>
<td>NEUP</td>
<td>Nuclear Energy University Program</td>
</tr>
<tr>
<td>ORNL</td>
<td>Oak Ridge National Laboratory</td>
</tr>
<tr>
<td>PT</td>
<td>Precipitation Treatment</td>
</tr>
<tr>
<td>SMT</td>
<td>Simplified Model Test</td>
</tr>
<tr>
<td>TTT</td>
<td>Time-Temperature-Transformation</td>
</tr>
<tr>
<td>UNS</td>
<td>Unified Numbering System</td>
</tr>
</tbody>
</table>
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A709 Qualification Plan Update and Mechanical Properties Data Assessment

1. INTRODUCTION

Improved material performance of advanced reactor components allows designers to increase the operating temperature of the reactor plant to achieve higher thermal efficiency and power output, to extend the component design lifetimes, and to reduce the commodity requirements. Hence the use of advanced materials can reduce the levelized cost of power generation and improve the economics of advanced reactor deployment. Advanced materials can also provide greater structural safety margins of the reactor plant and improve the reliability of the reactor components. Further, the enhanced structural properties can spur improvements in elevated-temperature design methodology and thereby allow more flexibility in the design, construction, and operation of advanced reactors. Both the development and qualification of advanced materials was identified as one of the objectives of the Fast Reactors Campaign of the Advanced Reactor Technologies (ART) Program to support cost reduction in the deployment of advanced reactors.

To achieve this objective, a multi-laboratory advanced materials development effort, involving the Argonne National Laboratory (ANL), the Idaho National Laboratory (INL), and the Oak Ridge National Laboratory (ORNL), based on an established alloy development priority list and followed by an intermediate term testing program, was carried out to down-select one ferritic-martensitic steel and one austenitic stainless steel for Code qualification consideration. It was concluded that two advanced materials, which have overall structural performance advantages as compared with their respective reference construction material, were suitable for Code qualification. The down-selected materials were the Grade 92 ferritic-martensitic steel, with a special thermal mechanical treatment, and the Alloy 709 austenitic stainless steel. However, due to resource constraints, only the Code qualification of Alloy 709 was selected to move forward because of its more compelling structural strength advantage over Type 316 stainless steel. The focus of this report is on Alloy 709.
2. ALLOY 709 DEVELOPMENT HISTORY

2.1 Early Development

The 20Cr25Ni/Nb stainless steel was originally developed by the British in the 1950s and was used as a fuel cladding material in the British Advanced Gas-cooled Reactor (AGR) fleet since 1962. The cladding tubes had a diameter of 14.5 mm, a wall thickness of 0.38 mm, and a length of 1 m. Each cladding tube was exposed to neutron damage for about 6 years. There were approximately 90,000 fuel pins in each AGR plant, and there were 14 plants constructed and operated. The creep strength of 20Cr25Ni/Nb was relatively low, but it was adequate for the intended application.

In the 1980s, the Nippon Steel Corporation was developing advanced stainless steels for use in ultra-supercritical boilers. They modified the base chemical composition of 20Cr25Ni/Nb and added B, Mo, and Ti to strengthen the creep resistance. The resulting austenitic stainless steel was trademarked NF 709. The chemical composition specification of NF 709 and a typical chemistry of NF 709 and 20Cr25Ni/Nb are shown in Table 1.

Table 1. Comparison of chemical compositions.

<table>
<thead>
<tr>
<th>Element</th>
<th>Typical 20Cr25Ni/Nb Specification</th>
<th>Typical NF 709 TP310MoCbn (S31025) Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Wt %</td>
<td>Wt %</td>
</tr>
<tr>
<td>C</td>
<td>0.015</td>
<td>0.07</td>
</tr>
<tr>
<td>Mn</td>
<td>0.5</td>
<td>0.97</td>
</tr>
<tr>
<td>Si</td>
<td>0.6</td>
<td>0.40</td>
</tr>
<tr>
<td>p</td>
<td>0.01</td>
<td>0.001</td>
</tr>
<tr>
<td>S</td>
<td>0.02</td>
<td>0.007</td>
</tr>
<tr>
<td>Cr</td>
<td>20</td>
<td>20.68</td>
</tr>
<tr>
<td>Ni</td>
<td>25</td>
<td>24.7</td>
</tr>
<tr>
<td>Nb</td>
<td>0.6</td>
<td>0.24</td>
</tr>
<tr>
<td>B</td>
<td>0.0001</td>
<td>0.005</td>
</tr>
<tr>
<td>Mo</td>
<td>1.0-2.0</td>
<td>1.45</td>
</tr>
<tr>
<td>Ti</td>
<td>0.02-0.20</td>
<td>0.04</td>
</tr>
<tr>
<td>N</td>
<td>&lt; 0.004</td>
<td>0.17</td>
</tr>
</tbody>
</table>

The composition of NF 709 was designed to produce the following characteristics:

- A stable austenite devoid of the sigma and other intermetallic phases under long-term elevated-temperature service conditions
- Creep-strengthening by carbonitride M(CN) precipitated in a stable, fine dispersion
- Solid solution strengthening from Mo and N in solution
- Excellent steam-side corrosion resistance
• Good coal ash corrosion resistance.

There were approximately 500 metric tons of NF 709 tubing materials produced by the Nippon Steel Corporation from 1995 to 2005 for superheater and reheater applications in the power boiler industry in Japan. When the patent expired, the material received a grade designation of TP310MoCbN with an Unified Numbering System (UNS) S31025 in ASTM A213, “Standard Specification for Seamless Ferritic and Austenitic Alloy-Steel Boiler, Superheater, and Heat-Exchanger Tubes.” The chemical specification for TP310MoCbN is also shown in Table 1. It is noted that modifications of the specification for some elements, as highlighted in the table, were made.

An ASME Code Case requested for Section I power boiler application was proposed by the Mitsubishi Heavy Industries, Ltd. of Japan in 2006 based on an extensive data package developed by the Nippon Steel Corporation. It was approved for construction to 1500°F (816°C) in Code Case 2581.

2.2 ART Program Development

To support the objective of the ART Program to reduce the construction and operational costs in the deployment of fast reactors, two austenitic stainless steels, HT-UPS and NF 709, were identified as candidate materials to replace Type 316 stainless steel, a reference construction material for sodium fast reactors. The HT-UPS austenitic stainless steel with a nominal composition of Fe-14Cr-16Ni-Mo-Mn in weight percent was developed in the late 1980s at the ORNL [1]. It exhibited superior creep strength compared to other heat-resistant steel alloys in similar grades. The NF 709 austenitic stainless steel developed by the Nippon Steel Corporation in the early 1990s, as described in the previous section, exhibits superior creep resistance among the commercially available advanced austenitic heat-resistant steels. Both steels have considerably higher creep strengths than Type 316 stainless steel.

Laboratory-scale heats, approximately 100 to 150 lbs, in plate product form were procured to support the testing efforts. Scoping tests on tensile, creep, fatigue, creep-fatigue, thermal aging, toughness, sodium exposure, and weldability were conducted for the base composition of HT-UPS, its two variants and Alloy 709. Most of the test specimens are sub-sized specimens. The resulting high-temperature tensile properties, thermal stability, creep strength, creep-fatigue resistance, sodium compatibility, and weldability for these candidate alloys and reference Type 316 stainless steel were compared. Alloy 709 exhibited the best overall performance as illustrated in the property ranking chart in Figure 1.

![Property Rank Chart](image)

Figure 1. Property ranking chart for Alloy 709, HT-UPS, its variants and Type 316 stainless steel. Alloy 709 showed the best overall performance.

Since the scoping tests were conducted primarily on sub-sized specimens and with relatively short test times, an intermediate term test program was initiated with the objective to ascertain the improvement
in the mechanical properties of Alloy 709 by testing standard-sized ASTM specimens and for longer times. Four larger laboratory-scale heats of Alloy 709 in plate product form were procured from one U.S.-based vendor and one vendor from Japan, ranging from 275 to 330 lbs for each heat. Some materials from these heats were also provided to universities to support their projects under the Nuclear Energy University Program (NEUP) on Alloy 709. In addition, a weldment program was initiated to develop the welding parameters and to fabricate Alloy 709 weldment using matching filler metal and gas-tungsten arc welding process to support the intermediate term testing. The findings of the intermediate term test program and the weldment program are as follows:

- Based on the thermal aging and sodium compatibility studies in terms of tensile properties, hardness measurements, and microstructural analysis, it was concluded that Alloy 709 is a stable material when operated in liquid sodium at elevated temperatures.

- The heat treatment process developed by the Nippon Steel Corporation was optimized to promote high-creep strength. This was confirmed by the creep test data.

- Creep-rupture data continued to show the enhanced strength of Alloy 709 over the reference Type 316 stainless steel.

- Since ASME Section I power boiler designs do not consider the creep-fatigue failure mode, power boiler materials are usually not optimized for creep-fatigue resistance. The creep-fatigue test data from the intermediate term testing showed that the creep-fatigue resistance was less than optimal and could be very poor under certain processing conditions.
  
  - Based on the fabrication processes of the four laboratory-scale heats, processing guidelines for the hot rolling and heat treatment of Alloy 709 plates were recommended to guard against the potential for the reduction in creep-fatigue resistance.

- A limit on the P content in the chemical composition of the matching filler metal was necessary to produce an ASME Section IX qualified Alloy 709 weldment as fabricated by the gas-tungsten arc welding process.

- It was recommended to Code qualify Alloy 709 for sodium-fast reactor applications.

  Following this recommendation, a comprehensive Code qualification plan was developed to support the incorporation of Alloy 709 in ASME Section III, Division 5 for Class A component construction. This involved the generation of an Alloy 709 data package and the development of material-specific design parameters required for Class A component design. Table 2 shows the design parameters for Class A design rules and the corresponding data categories required for their development.
Table 2. Design parameters for Section III, Division 5 Class A rules and the corresponding data source for their determination.

<table>
<thead>
<tr>
<th>Design Parameters</th>
<th>Required Test Data</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Allowable Stresses</strong></td>
<td></td>
</tr>
<tr>
<td>( S_m ): based on yield and ultimate strengths at temperature</td>
<td>Tensile data at temperature (time independent)</td>
</tr>
<tr>
<td>( S_t ): based on time to 1% total strain, time to onset of tertiary creep, time to rupture</td>
<td>Creep-rupture data with full creep curves (time dependent)</td>
</tr>
<tr>
<td>( S_s ): based on stress to rupture</td>
<td></td>
</tr>
<tr>
<td>( S_{mt} ): lesser of ( (S_m, S_t) )</td>
<td>Derived design parameters</td>
</tr>
<tr>
<td>( S_0 ): lesser of ( (S, S_{mt}@300,000h) )</td>
<td></td>
</tr>
<tr>
<td>( R ): Stress rupture factor - based on rupture strengths of base metal and weldment</td>
<td>Stress rupture data from base metal and weldment (time dependent)</td>
</tr>
<tr>
<td><strong>Thermal aging factors on yield and ultimate strengths</strong></td>
<td></td>
</tr>
<tr>
<td>Isochronous stress-strain curves constructed based on the creep behavior</td>
<td>Tensile stress-strain curves (time independent), and creep strain data up to 3% (time dependent)</td>
</tr>
<tr>
<td>Fatigue design curves</td>
<td>Strain-controlled continuous cycling tests</td>
</tr>
<tr>
<td>Creep-fatigue interaction diagram</td>
<td>Strain-controlled cyclic tests with hold times</td>
</tr>
<tr>
<td>Elastic, perfectly plastic design parameters</td>
<td>Two-bar test and Simplified Model Test (SMT); cyclic stress-strain curves</td>
</tr>
<tr>
<td>Inelastic material model parameters</td>
<td>Test data for other design parameters; and strain rate change and thermomechanical cycling</td>
</tr>
<tr>
<td>Huddleston effective stress parameters</td>
<td>Multiaxial creep-rupture data</td>
</tr>
<tr>
<td>External pressure charts</td>
<td>Tensile stress-strain curves (time independent)</td>
</tr>
<tr>
<td>Time-temperature limits for external pressure charts</td>
<td>Isochronous stress-strain curves</td>
</tr>
</tbody>
</table>

It is noted that the ASME Section III, Division 5 design-by-analysis Code rules for high-temperature reactor components address many additional failure modes that the Section I design-by-formula Code rules for power boilers do not cover. Also, the Section I Code rules do not have provisions to cover variable design lifetimes. Hence the data requirements for Division 5 Class A components are much more comprehensive. The Alloy 709 test plan was developed by closely following the recommendations in Division 5, Appendix-HBB-Y, “Guidelines for Design Data Needs for New Materials.”

For time-dependent design parameters, extrapolations from shorter term test data using an engineering model are generally necessary, particularly for the very long design lifetimes of 300,000 to 500,000 hours. Appendix-HBB-Y recommends a time-extrapolation factor of three for ferritic-martensitic steels and a factor of five for austenitic stainless steels and nickel alloys. As shown in Table 2, the time-dependent allowable stresses are driven primarily by the creep-rupture data. Thus, the creep-rupture tests would dictate the required test durations of the Alloy 709 Code Case test program. In order for the advanced reactor designers to conduct design activities using Alloy 709 at various design phases of a demonstration plant and, eventually, a commercial plant deployment, a staged approach was developed for the Code Case development. It involved the development of the 100,000, 300,000, and 500,000-hour Code Cases sequentially, and as soon as the relevant time-dependent data are available. This staged Code qualification approach for Alloy 709 is illustrated in Figure 2.
A “Staged” approach for the Code qualification of Alloy 709.

In addition to seamless tubing, product forms such as plate, bar, pipe, and forging are required for the construction of high-temperature reactor components. Data from a minimum of three commercial heats are required for some data categories in support of a new ASME Section III, Division 5 Material Code Case. However, data submitted on three heats of one wrought product form may be considered applicable for all other wrought product forms having the same chemistry. Thus, the test plan called for the majority of testing to be performed on plates. The plate data would be supplemented by a smaller data set from other product forms.

A description of the Alloy 709 Code qualification plan was given by Sham and Natesan [2].

3. COMMERCIAL ALLOY 709 HEATS

3.1 Procurement of the First Commercial Heat

To initiate the Alloy 709 Code Case testing effort, commercial heats of Alloy 709 in plate product form were needed. Unlike the laboratory-scale heats with a heat size of 330 lbs or less, the commercial heats for stainless steels had a minimum heat size requirement of 40,000 to 45,000 lbs. Additionally, the preferred hot rolling schedule developed from the laboratory-scale heats of Alloy 709 could not be implemented in a large-scale industrial production setting. After an exhaustive effort, an agreement was reached with a U.S.-based stainless steel vendor to procure Alloy 709 plates from a master heat of approximately 45,000 lbs. Since the master heat was large, it was decided to get hot-rolled plates from ingots melted from the master heat with different commercial melt practices and different heat treatments to gain experience with the commercial heats and to assess variabilities. The plate rolling schedule used by the vendor for this class of stainless steels was adopted. The procurement of the first Alloy 709 commercial heat was documented in [3].

Three processing steps, argon-oxygen-decarburization (AOD), electroslag remelting (ESR), and homogenization (HOMO) treatment at 1200°C, were applied in sequence to obtain three types of ingots from the master heat. They were (i) AOD only, (ii) AOD followed by ESR (labeled as ESR), and (iii) AOD followed by ESR and a subsequent HOMO treatment (labeled as ESR+HOMO). Hot-rolled plates from each ingot type were solution annealed at three different temperatures of 1050, 1100 and 1150°C. That led to the nine combinations of processing conditions as shown in Table 3.

Table 3. Processing conditions of the first commercial heat.

<table>
<thead>
<tr>
<th>Ingot Processing Step(s)</th>
<th>Solution Anneal Temperature, °C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1050</td>
</tr>
<tr>
<td>AOD</td>
<td>AOD-1050</td>
</tr>
<tr>
<td>ESR</td>
<td>ESR-1050</td>
</tr>
<tr>
<td>ESR+HOMO</td>
<td>ESR+HOMO-1050</td>
</tr>
</tbody>
</table>
The acceptance testing of the first commercial heat showed that the tensile properties of all the processing conditions are satisfactory. Creep-rupture, fatigue, and creep-fatigue acceptance tests were also conducted for each processing condition. The test conditions were 330 MPa at 600°C for creep-rupture tests, fully-reversed 1% strain range at 650°C for fatigue tests, and fully-reversed 1% strain range at 650°C with 30 minutes tensile strain hold for creep-fatigue tests. The results on the rupture times, fatigue cycles to failure, and creep-fatigue cycles to failure are shown in Table 4. The data are based on the average of two test results for the same test condition, when available.

Table 4. Results from acceptance testing of the first Alloy 709 commercial heat.

<table>
<thead>
<tr>
<th>Ingot processing step(s)</th>
<th>Rupture time (h)</th>
<th>Fatigue (cycles to failure)</th>
<th>Creep-fatigue (cycles to failure)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AOD</td>
<td>670</td>
<td>1706 3004</td>
<td>1562 1739 1673 438 432 403</td>
</tr>
<tr>
<td>ESR</td>
<td>1564 2361 3480</td>
<td>1150 1021 1237 568 471 232</td>
<td></td>
</tr>
<tr>
<td>ESR + HOMO</td>
<td>707 2195 2835</td>
<td>1850 1323 1458 519 438 281</td>
<td></td>
</tr>
<tr>
<td>Solution Anneal Temperature, °C</td>
<td>1050 1100 1150</td>
<td>1050 1100 1150</td>
<td>1050 1100 1150</td>
</tr>
</tbody>
</table>

The results indicate that higher solution anneal temperature leads to higher creep strength. But the creep-fatigue resistance follows an opposite trend. The solution anneal temperature dependence of the fatigue cycles to failure is rather weak. The degree of reduction of fatigue resistance due to the creep-fatigue interaction can be characterized by the fraction $f$, defined as the ratio of creep-fatigue cycles to failure at the same temperature and strain range conditions. The smaller the value of $f$ the more severe is the interaction. The values of $f$ determined from the fatigue and creep-fatigue data of Table 4 are shown in Table 5. The creep-fatigue interaction is the most severe for the solution anneal condition of 1150°C.

Table 5. Fatigue resistance reduction due to creep-fatigue interaction.

<table>
<thead>
<tr>
<th>Ingot processing step(s)</th>
<th>$f$, ratio of creep-fatigue to fatigue cycles to failure</th>
</tr>
</thead>
<tbody>
<tr>
<td>AOD</td>
<td>0.46 0.25 0.24</td>
</tr>
<tr>
<td>ESR</td>
<td>0.49 0.46 0.19</td>
</tr>
<tr>
<td>ESR + HOMO</td>
<td>0.28 0.33 0.19</td>
</tr>
<tr>
<td>Solution Anneal Temperature, °C</td>
<td>1050 1100 1150</td>
</tr>
</tbody>
</table>

The data in Table 4 from the limited acceptance test conditions together with the results in Table 5 showed that the ESR plates with a solution anneal temperature of 1100°C (ESR-1100) gave the most balanced mechanical properties.

3.2 Precipitation Treatment

As discussed earlier, 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. However, under low-temperature (below 500°C) and low-stress conditions, there are concerns that (1) those precipitates are not going to form in a reasonable amount of time due to the slow kinetics, and (2) different types of precipitates, such as the intermetallic laves phase, which is believed to be detrimental to creep properties, would be formed preferentially over M(CN). Thus, the development of a precipitation treatment (PT) procedure was pursued [4] with the objective to bring out the desired nano-sized precipitates on dislocations for improved mechanical performance in low-stress, low-temperature regions of a component.
The chemical compositions of the first commercial Alloy 709 heat were used in a commercial software to investigate the thermodynamic stability as well as the time-temperature-transformation (TTT) kinetics. Figure 3 shows these results.

![Figure 3. (Left) Simulated equilibrium phase diagram of Alloy 709. (Right) Simulated TTT diagram of Alloy 709 for 0.1% precipitation.](image)

Based on the results of the simulations, the 775°C/10h and 900°C/10h PTs were selected as candidate protocols to avoid the formation of the potentially deleterious sigma, chi, and laves phases and to promote the formation of the carbide phases. The 10-hour duration of the PT was selected for practicability in the industrial settings. These two PTs were first applied to samples from the first Alloy 709 commercial heat in the AOD-1100 condition. It was found in [4] that the grain boundary M23C6 precipitates were less dense and more distinct in the 900°C/10h PT sample. The 775°C/10h PT sample had a higher density of dislocations and a much higher density of 20 to 30 nm-sized Nb-rich precipitates decorating the dislocations compared to the 900°C/10h PT sample. Based on these observations, the 775°C/10h PT was down-selected for further studies.

### 3.2.1 Cyclic Properties

Plate materials from the first commercial heat in the ESR-1100 and ESR-1150 conditions (see Table 3) were heat treated in the box furnace for 10 hours at 775°C [5]. The plate materials that have received the 775°C/10h PT condition were given the designation of ESR-1100-PT and ESR-1150-PT. Strain-controlled fatigue and creep-fatigue scoping tests were conducted on these materials. The test temperature was 650°C, the applied fully-reversed strain range was 1%, and the magnitude of the strain rate was 0.001 m/m/s. Additionally, a hold time of 30 minutes was imposed at the maximum applied strain within each cycle for the creep-fatigue tests.

The maximum and minimum stresses within a cycle are plotted versus the cycle counts in Figure 4 for the fatigue tests and in Figure 5 for the creep-fatigue tests. Additionally, some data from prior testing on ESR-1100 and ESR-1150 at INL and ORNL and on Type 316 stainless steel, all under the same test conditions, are included in these figures.
Figure 4. Maximum and minimum stresses in fatigue cycles for Alloy 709 in the solution anneal and the PT conditions. Type 316 stainless steel fatigue data are included for comparison.

Figure 5. Maximum and minimum stresses in creep-fatigue cycles for Alloy 709 in the solution anneal and the PT conditions. Type 316 stainless steel creep-fatigue data are included for comparison.

The cycles to failure for the precipitation-treated and non-treated Alloy 709 materials and for Type 316 stainless steel are shown in Table 6. The values in the table are averages from duplicate tests, ranging from 2 to 5.
Table 6. Information on cycles to failure from fatigue and creep-fatigue tests for different Alloy 709 material conditions and for Type 316 stainless steel. Scoping test conditions: strain range = 1%, temperature = 650°C, strain rate = 0.001 m/m/s, and hold time = 30 min (only for creep-fatigue tests).

<table>
<thead>
<tr>
<th>Material</th>
<th>Fatigue cycles to failure</th>
<th>Creep-fatigue cycles to failure</th>
<th>f, ratio of creep-fatigue to fatigue cycles to failure</th>
</tr>
</thead>
<tbody>
<tr>
<td>ESR-1100</td>
<td>1021</td>
<td>471</td>
<td>0.46</td>
</tr>
<tr>
<td>ESR-1150</td>
<td>1237</td>
<td>232</td>
<td>0.19</td>
</tr>
<tr>
<td>ESR-1100-PT</td>
<td>1672</td>
<td>1113</td>
<td>0.67</td>
</tr>
<tr>
<td>ESR-1150-PT</td>
<td>1473</td>
<td>986</td>
<td>0.67</td>
</tr>
<tr>
<td>316H</td>
<td>1389</td>
<td>903</td>
<td>0.65</td>
</tr>
</tbody>
</table>

The PT was originally developed to improve the mechanical performance of Alloy 709 in the low-stress and low-temperature regime. However, it is seen from Table 6 that it has also improved the fatigue and creep-fatigue resistance of Alloy 709 quite significantly for the scoping test conditions. Within the large data scatter of cyclic tests, the difference in the cyclic properties of ESR-1100-PT and ESR-1150-PT is considered to be small. Referring to the acceptance test data from the first commercial Alloy 709 heat in Table 4, it is noted that the ESR-1150 had a higher creep strength as compared with ESR-1100. Thus, it was recommended to apply the PT protocol of 775°C/10h to ESR-1150 as the base condition to develop the Alloy 709 Code Case data package.

### 3.3 Procurement of Additional Commercial Heats

As the data package for the Alloy 709 Code Case requires data from a minimum of three commercial heats, a second commercial heat of Alloy 709 in plate product form was procured from a second U.S.-based stainless steel vendor. The ESR ingots were produced from a master heat of approximately 40,000 lbs. The ingots were hot rolled into plates of thickness 1.75 in. (7 plates) and 2 in. (2 plates), followed by a solution anneal treatment at a minimum temperature of 1150°C for all plates, except one 1.75 in. plate which remains in the as-rolled condition. The plates were delivered in Fiscal Year (FY) 2021. Figure 6 shows the plates in controlled storage at INL.

![Figure 6. Alloy 709 plates from the second commercial heat in controlled storage at INL.](image)

A third commercial heat with the same specification was procured from the same vendor, and the plates were delivered in April 2022. Figure 7 shows the plates in a staging area at ORNL.
4. ALLOY 709 CODE CASE TESTING

In this section, the data generated to date to support the development of the data package for the Alloy 709 Code Case are assessed. These include tensile, creep, fatigue, and creep-fatigue data.

4.1 Tensile Data

4.1.1 Room Temperature Tensile Properties

The ASTM A213 specification for seamless tubing provides the following room temperature tensile requirements for Grade TP310MoCbN (UNS S31025):

<table>
<thead>
<tr>
<th>Tensile strength min, ksi [MPa]</th>
<th>Yield strength min, ksi [MPa]</th>
<th>Elongation in 2 in. or 50 mm, min, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>93 [640]</td>
<td>39 [270]</td>
<td>30</td>
</tr>
</tbody>
</table>

These minimum room temperature tensile requirements are adopted for the Alloy 709 Code Case for all product forms. The room temperature yield and tensile strength data and elongation data for Grade TP310MoCbN in the solution anneal condition from the data package of Code Case 2581 and for the two commercial Alloy 709 heats are shown in Figure 8, Figure 9, and Figure 10, respectively. The data from the first commercial heat include all the nine combinations of process and solution anneal conditions shown in Table 3 and the precipitation-treated condition of ESR-1150-PT. For the second commercial heat, the data include those in the solution anneal condition and the precipitation-treated condition. All room temperature tensile data met the minimum room temperature tensile requirements.
Figure 8. Available Alloy 709 room temperature yield strength data from different processing conditions. The specification minimum requirement is met.

Figure 9. Available Alloy 709 room temperature tensile strength data from different processing conditions. The specification minimum requirement is met.
Figure 10. Available Alloy 709 room temperature elongation data from different processing conditions. The specification minimum requirement is met.

### 4.1.2 Tensile Properties at Elevated Temperatures

The temperature dependence of the yield and tensile strength and elongation for Grade TP310MoCbN from the data package of Code Case 2581, and the first Alloy 709 commercial heat in the PT condition (ESR-1150-PT) and the second Alloy 709 commercial heat, also in the PT condition, are shown in Figure 11, Figure 12, and Figure 13, respectively. The tensile data from the commercial heats in the PT condition to date are limited; more data will be generated. At higher temperatures, the elongations from the PT conditions are significantly higher than those from Grade TP310MoCbN in the solution anneal condition, indicating higher tensile ductility.
Figure 11. Comparison of yield strength data from commercial heats in PT condition with Grade TP310MoCbN in solution anneal condition.

Figure 12. Comparison of tensile strength data from commercial heats in PT condition with Grade TP310MoCbN in solution anneal condition.
Figure 13. Comparison of elongation data from commercial heats in PT condition with Grade TP310MoCbN in solution anneal condition.

4.2 Creep-Rupture Data

The original rationale for recommending Alloy 709 for Code qualification was its creep strength advantage over Type 316 stainless steel, which is a reference construction material for advanced reactors. In this section, the creep-rupture data generated to date from the commercial Alloy 709 heats in the PT condition are assessed against Type 316 stainless steel. Additional creep rupture data for Type 316 stainless steel was used to revise the allowable stress values in Section III, Division 5 are shown in Figure 14 in the form of a Larson-Miller plot.

Figure 14. Creep-rupture data for Type 316 stainless steel.
The Larson-Miller plot of the creep-rupture data from the two commercial heats of Alloy 709 in the PT conditions is shown in Figure 15.

![Figure 15. Creep-rupture data for Alloy 709 from the commercial heats in the PT condition.](image)

A preliminary assessment of the precipitation-treated Alloy 709 can be made by comparing its creep-rupture strengths with those of Type 316 stainless steel by extrapolating the creep-rupture data to different design lifetimes using the results from the Larson-Miller statistical analysis of the data shown in Figure 14 and Figure 15. In order to provide another perspective on the creep-rupture properties of the precipitation-treated Alloy 709, comparison is also made with Alloy 800H, an ASME Section III, Division 5 qualified high-temperature alloy. Table 7 shows the ratios of the value of the expected minimum stress to rupture of precipitation-treated Alloy 709 to that of Type 316 stainless steel and Alloy 800H, respectively, for design lifetimes of 65,000, 100,000, and 300,000 hours. The following parameters are used in Table 7:

\[
\begin{align*}
    r_{316} & = \frac{\text{Minimum Stress to Rupture of precipitation}\text{-treated Alloy 709}}{\text{Minimum Stress to Rupture of Type 316 stainless steel}} \\
    r_{800H} & = \frac{\text{Minimum Stress to Rupture of precipitation}\text{-treated Alloy 709}}{\text{Minimum Stress to Rupture of Alloy 800H}}
\end{align*}
\]

Table 7. Comparisons of extrapolated minimum creep-rupture strength of precipitation-treated Alloy 709 relative to Type 316 stainless steel and Alloy 800H.

<table>
<thead>
<tr>
<th>Temp (°C)</th>
<th>65,000 h</th>
<th>100,000 h</th>
<th>300,000 h</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$r_{316}$</td>
<td>$r_{800H}$</td>
<td>$r_{316}$</td>
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<tr>
<td>550</td>
<td>1.52</td>
<td>1.56</td>
<td>1.63</td>
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<tr>
<td>575</td>
<td>1.56</td>
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<td>600</td>
<td>1.58</td>
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</tr>
<tr>
<td>675</td>
<td>1.59</td>
<td>1.60</td>
<td>1.57</td>
</tr>
<tr>
<td>700</td>
<td>1.57</td>
<td>1.60</td>
<td>1.55</td>
</tr>
</tbody>
</table>
The results shown in Table 7 should be considered as trends rather than quantitatively as the extrapolation of the creep-rupture properties of precipitation-treated Alloy 709 was based on short-term data. However, the trends continue to show that the creep-rupture strengths of precipitation-treated Alloy 709 are higher than Type 316 stainless steel and Alloy 800H.

### 4.3 Continuous Cycling Data

Fully-reversed, strain-controlled continuous cycling testing on the precipitation-treated Alloy 709 materials was conducted to generate fatigue data for the construction of the fatigue design curves. The fatigue tests were performed to the ASTM E606 standards. Figure 16 summarizes the initial set of fatigue data generated to date. The test conditions were selected over a coarse grid for the purpose of establishing the general trends of the fatigue resistance of Alloy 709-PT. The information would guide the selection of test conditions over a finer grid to generate fatigue data to populate the full fatigue design curves.

<table>
<thead>
<tr>
<th></th>
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<th>1.53</th>
<th>1.94</th>
<th>1.51</th>
<th>2.00</th>
<th>1.45</th>
</tr>
</thead>
<tbody>
<tr>
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<td>2.01</td>
<td>1.49</td>
<td>2.04</td>
<td>1.46</td>
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<td>2.16</td>
<td>1.40</td>
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<td>1.25</td>
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<td>1.53</td>
<td>1.94</td>
<td>1.51</td>
<td>2.00</td>
<td>1.45</td>
</tr>
</tbody>
</table>

Figure 16. Strain-controlled fatigue data for Alloy 709 from the commercial heats in the PT condition.

The initial set of fatigue data is not sufficient for the construction of a full fatigue design curve at any temperature. However, a review of the fatigue data suggested that the data from the 1400 and 1500°F tests followed a similar trend. Thus, these data were grouped together to construct one fatigue curve in order to provide an initial assessment of the fatigue resistance of Alloy 709-PT as compared with Type 316 stainless steel. The average fatigue curve from such a construction is shown in Figure 17. For Type 316 stainless steel, fatigue design curves were provided in ASME Section III, Division 5, but only up to 1300°F (704°C). The average fatigue curve for Type 316 stainless steel at 1300°F was reconstituted from the Division 5 fatigue design curve using the “two-on-strain-range and twenty-on-cycles” criterion. This curve is also included in Figure 17.

It is seen from Figure 17 that the 1400–1500°F curve for Alloy 709-PT lies above the 1300°F curve for Type 316 stainless steel. Generally, the fatigue resistance decreases as the temperature increases. Thus
the results from Figure 17 continue to show that Alloy 709-PT has a higher fatigue resistance over Type 316 stainless steel.

![Average Fatigue Curves](image1.png)

**Figure 17.** Average fatigue curves of precipitation-treated Alloy 709 and Type 316 stainless steel, showing higher fatigue resistance of Alloy 709-PT as compared with Type 316 stainless steel even for a higher temperature for Alloy 709-PT.

To further explore the general fatigue behavior of Alloy 709-PT, the fatigue data of Alloy 617, which is a high-temperature alloy, were used to compare with the Alloy 709-PT fatigue data. Fatigue data from these two materials are plotted as a function of temperature at a strain range of 1%, 0.6%, and 0.3% in Figure 18, Figure 19, and Figure 20, respectively. The results show that the fatigue resistance of Alloy 709-PT is comparable to Alloy 617 at these strain ranges.

![1% Strain Range Fatigue Data](image2.png)
Figure 18. Comparison of Alloy 709-PT and Alloy 617 fatigue data at 1.0% strain range.
Figure 19. Comparison of Alloy 709-PT and Alloy 617 fatigue data at 0.6% strain range.

Figure 20. Comparison of Alloy 709-PT and Alloy 617 fatigue data at 0.3% strain range.
4.4 Creep-Fatigue Data

Strain-controlled creep-fatigue tests on the precipitation-treated Alloy 709 materials were conducted to generate creep-fatigue data for the construction of the creep-fatigue interaction diagram or the D-diagram. The creep-fatigue tests were performed to the ASTM E606 standards. In a strain-controlled creep-fatigue test, a triangular wave form in strain, with a specified, fully-reversed strain range and a hold time imposed at peak strain, is applied to the test specimen until failure. The strain rate is typically set at 0.001 in./in./s. The time histories of stress and strain and the cycles to failure are the output of the creep-fatigue test.

Cycles to failure from the creep-fatigue test and from the fatigue test of the same material with the same strain range, strain rate, and temperature are used to determine the fatigue-damage fraction for the D-diagram construction. Due to large data scatter in cyclic tests, averages from duplicate tests are usually used to determine the fatigue-damage fraction.

The creep-damage fraction of the D-diagram is determined based on the time-fraction criterion. The stress relaxation profiles during the strain holds in the creep-fatigue test, together with the time-to-rupture Larson-Miller correlation from the creep-rupture data, are used to calculate the total creep-damage fraction for the strain range, temperature, and hold time of the creep-fatigue test. Averages from duplicate tests are also used to determine the creep-damage fraction.

The fatigue and creep-fatigue data generated to date for Alloy 709-PT were used to determine the creep damage fraction and the fatigue damage fraction and are shown in Figure 21.

![Figure 21. Creep-fatigue interaction diagram for Alloy 709-PT.](image-url)
Various bi-linear creep-fatigue interaction envelopes were added to Figure 21 to provide a visual
guide. It is noted that the envelope is not intended as a lower bound on the creep- and fatigue-damage
fractions in the Division 5 creep-fatigue evaluation procedures. The Alloy 709-PT creep-fatigue data
generated to date do not indicate any abnormal behavior.

4.5 Assessment of Alloy 709 Data Generated to Date

The tensile, creep, fatigue, and creep-fatigue data generated to date for precipitation-treated Alloy 709
continued to show that this advanced austenitic stainless steel outperforms Type 316 stainless steel. The
data also showed that the PT is effective in enhancing the creep-fatigue resistance while maintaining a
significant creep strength advantage over Type 316 stainless steel. It is recommended to continue the
Alloy 709 Code Case Testing Program to develop the data package needed for the determination of the
material-specific design parameters for inclusion in the Alloy 709 Code Case.

5. UPDATE TO THE ALLOY 709 TEST PLAN

Based on the creep, fatigue, and creep-fatigue data generated to date for the precipitation-treated
Alloy 709, the test conditions for these tests are updated below to provide a more even coverage of the
mechanical properties for the Alloy 709 Code Case data package. Data for some the test conditions in the
updated test matrices have already been generated to date.

5.1 Creep-Rupture Test Conditions

The creep-rupture data for the precipitation-treated Alloy 709 as shown in the Larson-Miller plot of
Figure 15 allow the creep-rupture test matrix to be updated in order to avoid getting rupture times that are
either too short or too long as estimated from the preliminary rupture data generated previously. The
updated test matrix is shown in Table 8.

Table 8. Updated creep-rupture test matrix.

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Temp (°C)</th>
<th>Stress (MPa)</th>
<th>Code Case</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>550</td>
<td>250</td>
<td>500,000-hour Code Case</td>
</tr>
<tr>
<td>2</td>
<td>650</td>
<td>110</td>
<td>500,000-hour Code Case</td>
</tr>
<tr>
<td>3</td>
<td>750</td>
<td>45</td>
<td>500,000-hour Code Case</td>
</tr>
<tr>
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<td>27</td>
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<td>925</td>
<td>24</td>
<td>Preliminary Code Case</td>
</tr>
<tr>
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<td>925</td>
<td>20</td>
<td>Preliminary Code Case</td>
</tr>
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<td>950</td>
<td>24</td>
<td>Preliminary Code Case</td>
</tr>
<tr>
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<td>950</td>
<td>20</td>
<td>Preliminary Code Case</td>
</tr>
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</tr>
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</tr>
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<td>62</td>
<td>1000</td>
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<td>Preliminary Code Case</td>
</tr>
</tbody>
</table>
5.2 Fatigue Test Conditions

Based on the dependence of the cycles to failure on the temperature and strain range from the fatigue data generated to date, an update to the overall fatigue test matrix is provided in Table 9.

Table 9. Updated fatigue test matrix.

<table>
<thead>
<tr>
<th>Temp (°F)</th>
<th>Temp (°C)</th>
<th>Strain Ranges, %</th>
<th>No. of Test Conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td>800</td>
<td>427</td>
<td>3, 2, 1, 0.6, 0.4, 0.3</td>
<td>6</td>
</tr>
<tr>
<td>1000</td>
<td>538</td>
<td>3, 2, 1, 0.6, 0.4, 0.3</td>
<td>6</td>
</tr>
<tr>
<td>1200</td>
<td>649</td>
<td>3, 2, 1, 0.6, 0.4, 0.3</td>
<td>6</td>
</tr>
<tr>
<td>1400</td>
<td>760</td>
<td>3, 2, 1, 0.6, 0.4, 0.3, 0.25</td>
<td>7</td>
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<td>1500</td>
<td>816</td>
<td>3, 2, 1, 0.6, 0.4, 0.3, 0.25</td>
<td>7</td>
</tr>
<tr>
<td>1750</td>
<td>954</td>
<td>3, 2, 1, 0.6, 0.4, 0.3, 0.25</td>
<td>7</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td></td>
<td></td>
<td><strong>39</strong></td>
</tr>
</tbody>
</table>

5.3 Creep-fatigue Test Conditions

Based on the dependence of the cycles to failure on the temperature, strain range, and hold time from the creep-fatigue data generated to date, an update to the overall creep-fatigue test matrix is shown in Table 10. The updated test conditions would avoid excessive test times for some of the test conditions from the original test matrix which was developed without too much information on the creep-fatigue behavior of Alloy 709 at the time.

Table 10. Updated creep-fatigue test matrix.

<table>
<thead>
<tr>
<th>Temp (°F)</th>
<th>Temp (°C)</th>
<th>Strain Range, %</th>
<th>Hold Times, min</th>
<th>No. of Test Conditions</th>
</tr>
</thead>
<tbody>
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<td>1200</td>
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<td>10, 30, 60</td>
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<td>649</td>
<td>0.3</td>
<td>10</td>
<td>1</td>
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<td>1500</td>
<td>816</td>
<td>1.0</td>
<td>10, 30, 60, 600</td>
<td>4</td>
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<td>1500</td>
<td>816</td>
<td>0.6</td>
<td>10, 30, 60</td>
<td>3</td>
</tr>
<tr>
<td>1500</td>
<td>816</td>
<td>0.4</td>
<td>10, 30</td>
<td>2</td>
</tr>
<tr>
<td>1500</td>
<td>816</td>
<td>0.3</td>
<td>10</td>
<td>1</td>
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<tr>
<td>1750</td>
<td>954</td>
<td>1.0</td>
<td>10, 30, 60, 600</td>
<td>4</td>
</tr>
<tr>
<td>1750</td>
<td>954</td>
<td>0.6</td>
<td>10, 30, 60</td>
<td>3</td>
</tr>
<tr>
<td>1750</td>
<td>954</td>
<td>0.4</td>
<td>10, 30</td>
<td>2</td>
</tr>
<tr>
<td>1750</td>
<td>954</td>
<td>0.3</td>
<td>10</td>
<td>1</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td></td>
<td></td>
<td></td>
<td><strong>25</strong></td>
</tr>
</tbody>
</table>
6. CONCLUSIONS

The development effort for the Alloy 709 Code Case was summarized in this report. An assessment was made of the mechanical properties data generated to date from the tensile, creep, fatigue, and creep-fatigue tests of the precipitation-treated Alloy 709 from the first two commercial heats. It was concluded that the mechanical properties of Alloy 709 with the PT continued to outperform those of Type 316 stainless steel, affirming the recommendation for its Code qualification to support the effort to reduce construction and operating costs for advanced reactor deployment.

The data also showed that the PT is effective in enhancing the creep-fatigue resistance of Alloy 709 while maintaining a significant creep strength advantage over Type 316 stainless steel.

Based on the test data generated to date, in particular the creep-rupture data, the test conditions for the creep, fatigue, and creep-fatigue test matrices have been updated in order to provide a better coverage of the Code Case data package.

It is recommended to continue the Alloy 709 Code Case Testing Program to develop the data package needed for the determination of the material-specific design parameters for inclusion in the Alloy 709 Code Case.
7. REFERENCES


