FY21 Status Report on Creep Rupture Testing at ANL to Support the Development of Alloy 709 Code Case

Nuclear Science and Engineering Division

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Prepared by

Xuan Zhang

Nuclear Science and Engineering Division

Argonne National Laboratory

Ting-Leung Sham

Reactor Systems Design & Analysis Division

Idaho National Laboratory

Meimei Li

Nuclear Science and Engineering Division

Argonne National Laboratory

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ABSTRACT

This report provides an update on the status of the creep rupture testing on the first commercial heat of Alloy 709 in support of the Alloy 709 Code Case development. Creep tests were performed in air on the lever-arm load frames in the Mechanical and Environmental Testing Laboratory at Argonne National Laboratory (ANL). In FY21, the focus was on the age-hardened ESR-1150°C material. Twenty two tests were initiated, among which ten were completed. This report presents the data from the completed tests. Metallographic characterization of the fractured specimens were performed and the results are included. The creep data enabled the preliminary analysis of the creep behavior of this material. The characterization showed the effect of test temperature and stress on the deformation microstructure.

ACRONYMS

AH	age hardening
ANL	Argonne National Laboratory
AOD	argon oxygen decarburization
ART	Advanced Reactor Technologies
ASME	American Society of Mechanical Engineers
ASTM	ASTM, International (formerly, the American Society for Testing and Materials)
ATS	Applied Test Systems
ESR	electro-slag remelting
FY	fiscal year
HOMO	homogenized
INL	Idaho National Laboratory
LVDT	linear variable differential transformer
NRC	Nuclear Regulatory Commission
S/A	solution anneal
SFR	sodium fast reactor
TEM	transmission electron microscopy

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1 Introduction

Advanced materials are a key element in the development of advanced nuclear energy systems. High-performance structural materials allow for a compact and simpler design of the reactor structure, and have the potential to reduce the construction and operational costs for next-generation advanced nuclear reactors. Due to the significant enhancement in time-dependent mechanical properties of the austenitic Alloy 709 relative to 316H stainless steel, a reference construction material for the sodium fast reactor (SFR) systems, code qualification of Alloy 709 was recommended in FY14. A comprehensive plan for the development of a 500,000-hour, 760°C ASME Code Case and the resolution of structural integrity issues identified by Nuclear Regulatory Commission (NRC) for Alloy 709 was developed in FY15. A Phase I implementation of this plan that includes a 100,000-hour, 760°C ASME code case and the initiation of very long-term creep tests, thermal aging, and sodium exposure of Alloy 709 was established.

Alloy 709 is derived from NF709 (Fe-20Cr-25Ni-1.5Mo-Nb,B,N), which was a commercial heat- and corrosion- resistant austenitic stainless steel developed by Nippon Steel Corporation in Japan for seamless boiler tubing applications. The high strength of NF709 is achieved by controlling the carbon content to 0.07–0.10% and precipitation strengthening by NbC (MX phase) and CrNbN (Z phase). NF709 also shows good fabricability properties and weldability. It is regarded as one of the best austenitic steels for elevated temperature applications among commercially-available austenitic alloy classes at the time that it was developed. The NF709 alloy provides time dependent strength nearly double that for conventional 304 and 316 stainless steels at sodium-cooled fast reactor relevant temperatures (Busby, et al., ORNL/GNEP/LTR-2008-023, 2008). While the cost for this alloy has been estimated at approximately 2-4 times that for 304 SS (and 1.5 to 3 times that for 316 SS), many fossil plants have found that the improved performance outweighs the commodity cost, and is still far below the cost for Ni-based superalloys at comparable strengths. Alloy 709 has the same chemical composition as NF709 but is intended for sodium fast reactor applications that include reactor vessel, core supports, primary and secondary piping, and possibly intermediate heat exchanger and compact heat exchanger. There is also interest in using Alloy 709 at high temperatures for other advanced reactor designs, e.g., the fluoride-salt cooled high temperature reactor. Hence development of processing conditions and fabrication scale up for different product forms such as plates, pipes, bars, forgings and sheets, in addition to seamless tubing, are required.

The fabrication scale-up effort for Alloy 709 was started in FY16 and completed in FY17. The effort culminated in the procurement of four ingots, totaling about 45,000 lb, that were bottom-poured from the melt by a commercial vendor in September 2016. Three ingots, processed by Argon-Oxygen-Decarburization (AOD), Electroslag Remelting (ESR), and ESR with subsequent Homogenization (ESR-homo) routes, were rolled into plates and each of them were solution annealed at 1050, 1100, and 1150°C. The nine conditions were subsequently delivered to ANL and ORNL. Several samples were cut from the as-rolled test pieces and solution annealed plates and were analyzed for their microstructures, hardness values, grain sizes, and tensile properties. The results showed that the scaled up heat of Alloy 709 fabricated using commercial practice exhibited tensile properties that exceeded the minimum values specified in the ASME Code Case for commercial heats of NF709 (Natesan, et al., ANL-ART-89, 2017).

In FY18, activities focused on the scoping tests for down selection from the nine (9) processing conditions. This task involves the creep rupture, fatigue, and creep-fatigue testing of Alloy 709 standard sized ASTM specimens from the first Alloy 709 commercial heat for the short and intermediate term durations. Creep testing equipment was set up at ANL through upgrade and refurbishment of existing equipment and procurement of new equipment to support the generation of creep rupture data for the Alloy 709 scoping tests and the code case. In FY18, 600°C/330 MPa tests were carried out on all nine processing conditions (Natesan, et al., ANL-ART-151, 2018). The results, in combination with the results from fatigue and creep-fatigue tests, facilitated the decision on carrying the ESR-1100°C material for the code case.

In FY19, activities at ANL focused on the preliminary design basis and the 100,000-hour code case testing of the ESR-1100°C material. Fifteen (15) creep-rupture tests at temperatures below or equal to 700°C, thus requiring the use of Type K thermocouples, were initiated. Eight (8) tests were completed in FY19 (Zhang, et al., ANL-ART-172, 2019). The results indicate that there is a critical transition from (more) brittle failure to (more) ductile failure between 625°C and 650°C. In addition, effort were initiated to add Type S thermocouples to five of the creep test frames, for performing long-term tests at temperatures higher than 675°C.

In FY20, activities at ANL continued to focus on the preliminary design basis and the 100,000-hour code case testing of the ESR-1100°C material. The addition of Type S thermocouples was completed on five test frames, and one tests at 675°C/175MPa was loaded. In total, seven tests were completed (Zhang, et al., ANL-ART-193, 2020).

In FY21, activities at ANL focuses on the preliminary design basis and the 100,000-hour code case testing of the age-hardened ESR-1150°C material. Age hardening at 775 °C for 10 hours was performed on the ESR-1150°C plates, from which test specimens were machined. Twenty two (22) tests were loaded; ten (10) were completed. This report details the test conditions, creep data, and post-test metallographic studies of the completed tests.

2 Material Specifications and Mechanical test facilities

2.1 Materials and specimens

U.S. Department of Energy, Office of Nuclear Energy's Advanced Reactor Technologies Program is conducting R&D on qualifying an advanced austenite stainless steel, Alloy 709, for ASME Section III Division 5 Class A constructions in support of development and deployment of sodium fast reactors.

The product chemistry of the first commercial heat conforms to the ranges or maximum values specified in wt.% in Table 2-1. The chemistry aims and the actual melt compositions are also shown in the table.

Table 2-1. Chemical composition of melt of Alloy 709 heat # 58776 (in wt.%)

Alloy 709	C	Cr	Ni	Mn	Mo	N	Si	P	Ti	Nb	B	Fe
Specification range	0.04-0.10	19.5-23	23-26	1.5	1.0-2.0	0.14-0.16	1.0	<0.025	0.2	0.1-0.4	0.002-0.01	Bal
Aim	0.07	20	25	0.9	1.5	0.15	0.40	*	0.05	0.25	0.002-0.005	Bal
Actual	0.066	19.93	24.98	0.91	1.51	0.148	0.44	0.014	0.04	0.26	0.0045	Bal

*The P shall not exceed 0.025 wt.%.

In FY21, the material selected for the code case testing has a processing condition of ESR with solution annealing at 1150°C after rolling, followed by age hardening at 775°C for 10 hours. The material is denoted as ESR1150AH from here on. Due to the thickness of the plate being 1.1", only one specimen was fabricated in the thickness direction, centering on the mid-section. Figure 2-1 presents the dimensions of the specimen.

A plate of the ESR1150AH condition was prepared at ORNL. Twenty (20) creep specimens were machined out of the plate and were sent to ANL, with IDs of A709-ESR1150AH-1 to A709-ESR1150AH-20. All 20 specimens were loaded in FY21.

A plate of the ESR1150AH condition was prepared at ANL. Eleven (11) creep specimens were machined out of the plate, with IDs of A709-BC2-AH-01 to A709-BC2-AH-11. Two (2) specimens, A709-BC2-AH-01 and A709-BC2-AH-02 were loaded in FY21.

Figure 2-1. Schematic drawing of ASTM standard-sized creep specimens (Natesan, et al., ANL-ART-151, 2018).

2.2 Creep test facilities

The creep tests were performed in the Mechanical and Environmental Testing Laboratory in Building 212 at ANL. Figure 2-2 shows a photograph of the 15 creep frames. The frames were ATS Series 2300 Lever Arm Creep Testing Systems integrated with WinCCS II computer control and data acquisition software package. ATS systems are precision knife-edge lever arm testers. Each creep machine is equipped with a three-zone split-tube furnace capable of operation up to 1100°C. The temperature in the system is controlled by the ATS temperature control system which regulates the power applied to the resistive heating elements and maintain the desired temperature (setpoint) as measured by a control thermocouple. Typically, Type S thermocouples will be used to measure the specimen temperature, and when doing so, two thermocouples are spot welded to the shoulder sections of the specimen as the furnace temperature feedback. Type S thermocouples are used for test temperatures higher than 675°C and lifetime longer than 4,000 hours. The attachment of the Type S thermocouples to the specimen is different from the Type K; spot welding cannot be used because of the issues with alloying at high temperatures. Instead, following the recommendation by ASTM E633-13 “Standard Guide for Use of Thermocouples in Creep and Stress-Rupture Testing to 1800°F (1000°C) in Air”, the two thermocouples are fastened by platinum wires to the specimen gauge areas towards the two ends.

Figure 2-2. Creep test laboratory set up at ANL for testing of Alloy 709 (Natesan, et al., ANL-ART-151, 2018).

2.3 Creep test procedure

The creep tests under this project are conducted according to the ASTM Standard E139-11, “Standard Test Methods for Conducting Creep, Creep-Rupture, and Stress-Rupture Tests on Metallic Materials.” A lever arm ratio of 20:1 was used for all frames. Before test, samples were measured, mounted on individual rigs with LVDTs retrieved to zero position, and loaded to the frames. To initiate a test, the required dead weight was first calculated based on the sample geometry and placed on the pan. Then the furnace was heated up to the set temperature, and soaked for one hour. The load was then applied in a step manner and fully loaded in about five minutes. A load cell was placed directly above the dead weight to measure the load applied on the sample at each step. A typical loading curve is shown in Figure 2-3. In this case, the end stress was 250 MPa. During the test, data was recorded by the WinCCS II software. Upon sample failure, the furnace was cooled down in air and the sample was removed with the rig. The samples were then measured for the overall length and the minimum diameter by carefully placing the two broken pieces back together for calculation of the elongation and the reduction of area.

Figure 2-3. The loading curve of an Alloy 709 sample being loaded to 250 MPa at 650°C.

3 Creep Test Data on Alloy 709 ESR1150AH Condition

As mentioned previously, a total of twenty two creep tests on the ESR1150AH material were initiated in FY21 in support of the preliminary design basis and the 100,000-hour code case. Table 3-1 lists the test parameters and the progress at the time of writing this report; twenty two tests were initiated, of which ten were completed. The completed tests were under the conditions of 625°C/250 MPa, 650°C/250 MPa, 650°C/200 MPa, 650°C/175 MPa, 675°C/175 MPa, 700°C/140 MPa, 725°C/140 MPa, 725°C/110 MPa, 750°C/110 MPa, and 775°C/82 MPa.

Figure 3-1 plots the initial loading strains as a function of test temperature and stress for all the ten (10) completed tests, ranging from 0.03% for the 775°C/82 MPa condition to 2.65% for the 650°C/250 MPa condition.

Figure 3-2 plots the creep rupture life and the minimum creep rate as a function of test temperature and stress. It is seen that higher temperature and larger stress leads to shorter creep life and higher minimum creep rate.

Figure 3-3 plots the elongation and the area reduction as a function of test temperature and stress. It is seen that the specimens under those test conditions were all very ductile.

Figure 3-4 plots the creep curves of the completed tests.

Figure 3-5 is the Larson-Miller plot of the completed tests. The Larson-Miller parameter LMP was calculated as

$$LMP = (T + 273.15) \times (\log_{10} t_r + C),$$

where T is the temperature in °C, and C is a constant assumed to be 15 in the calculation.

Table 3-1. FY21 Alloy 709 code case testing progress.

test # (ANL)	sample ID	Stress (MPa)	Temp (°C)	Status
1	A709-ESR1150AH-19	250	575	running
2	A709-ESR1150AH-18	200	600	running
3	A709-ESR1150AH-17	175	625	running
4	A709-ESR1150AH-16	140	650	running
5	A709-ESR1150AH-1	250	650	ruptured
6	A709-ESR1150AH-6	200	650	ruptured
7	A709-ESR1150AH-4	175	675	ruptured
8	A709-ESR1150AH-2	140	725	ruptured
9	A709-ESR1150AH-3	110	750	ruptured
10	A709-ESR1150AH-9	82	775	ruptured
11	A709-ESR1150AH-13	250	600	running
12	A709-ESR1150AH-14	200	625	running

13	A709-ESR1150AH-10	175	650	ruptured
14	A709-ESR1150AH-11	140	675	running
15	A709-ESR1150AH-12	110	700	running
16	A709-ESR1150AH-15	82	725	running
17	A709-ESR1150AH-7	250	625	ruptured
18	A709-ESR1150AH-5	140	700	ruptured
19	A709-ESR1150AH-8	110	725	ruptured
20	A709-ESR1150AH-20	150	675	running
21	A709-BC2-AH-01	150	700	running
22	A709-BC2-AH-02	75	750	running

Figure 3-1. Plots of the initial loading strain as a function of test temperature and stress for the ruptured A709 specimens in FY21.

Figure 3-2. Plots of the creep rupture life and the minimum creep rate as a function of test temperature and stress for the ruptured A709 specimens.

Figure 3-3. Plots of the elongation and the reduction of area as a function of test temperature and stress for the ruptured A709 specimens.

Figure 3-4. Creep strain as a function of time for the commercial heat of Alloy 709 in the code case testing program. The plot shows the ten completed tests in FY21.

Figure 3-5. Larson-Miller plot of the completed tests of the commercial heat of Alloy 709 ESR1150AH condition, assuming a Larson-Miller constant C of 15.

4 Microstructural Analysis of Creep-Tested Specimens

Figure 4-1 shows the photos of the FY21 creep-ruptured Alloy 709 specimens. All the specimens failed at the gauge center.

Figure 4-1. Photos of the FY21 creep-ruptured A709 specimens.

4.1 Grain structures, deformation defects and precipitates

Figure 4-2 shows a photo of polished surfaces near the fracture regions of the ten creep-tested specimens that were ruptured in FY21. Those axial sections were the mid-section of the gauge region near the fracture surface of a broken specimen, as illustrated in Figure 4-2. Those surfaces were polished to mirror finish and were etched for optical microscopy study. Metallographic images of the ten specimens are shown in Figures 4-3 to 4-12, revealing that as the test temperature and stress varies, drastic differences in the deformation microstructures exist. Several observations are listed below:

- In general, specimens that were tested at 700 °C or lower tend to have the original grain structures preserved at the end of deformation, elongated in the loading direction (Figures 4-3 to 4-8). In contrast, the original grain structures in those that were tested at 725 °C (Figure 4-9 to 4-12) or higher are not very distinguishable.

- In general, the higher the test temperature, the higher the void density. The voids in specimens that were tested at 675 °C or higher tend to align in the loading direction, which is also the rolling direction. In specimens whose grain boundaries are revealed (specimens that were tested at 700 °C or lower), the voids are observed to locate primarily along the grain boundaries. For the rest of the specimens, it is hard to tell whether the voids are intergranular or intragranular.
- Several specimens contain large, abnormally grown grains, including the specimens tested at 650 °C/250 MPa, 675 °C/175 MPa, and 700 °C/140 MPa. Those grains do not seem to have affected the creep properties of the specimens. Previous metallographic study of the as-received ESR-1150°C plate showed the general existence of the abnormally grown grains, especially towards the two surfaces of the plate (Natesan, et al., ANL-ART-89, 2017). This explains the presence of those grains in the specimens.
- Dense precipitates can be observed in all the samples at high magnification, at grain boundaries and within grains. The microstructure of the as-aged ESR1150AH material has been studied in detail using the transmission electron microscopy (TEM) and has been reported in ANL-ART-192 (Zhang, et al., ANL-ART-192, 2020). The microstructure of an ESR1150AH specimen after creep-testing at 600°C/330 MPa has been studied in detail using TEM and will be reported in ANL-ART-233 (Zhang, et al., 2021).

Figure 4-2. A photo showing the polished surfaces of the axial section of the crept specimens, and an illustration of where the surfaces were located. The labels in the photo are the number X in the specimen ID A709 ESR1150AH-X.

Figure 4-3. The axial sectional view of sample ESR1150AH-7 (625°C/250MPa), at lower (5x) and higher (20x) magnifications.

Figure 4-4. The axial sectional view of sample ESR1150AH-1 (650°C/250MPa), at lower (5x) and higher (20x) magnifications.

Figure 4-5. The axial sectional view of sample ESR1150AH-6 (650°C/200MPa), at lower (5x) and higher (20x) magnifications.

Figure 4-6. The axial sectional view of sample ESR1150AH-10 (650°C/175MPa), at lower (5x) and higher (20x) magnifications.

Figure 4-7. The axial sectional view of sample ESR1150AH-4 (675°C/175MPa), at lower (5x) and higher (20x) magnifications.

Figure 4-8. The axial sectional view of sample ESR1150AH-5 (700°C/140MPa), at lower (5x) and higher (20x) magnifications.

Figure 4-9. The axial sectional view of sample ESR1150AH-2 (725°C/140MPa), at lower (5x) and higher (20x) magnifications.

Figure 4-10. The axial sectional view of sample ESR1150AH-8 (725°C/110MPa), at lower (5x) and higher (20x) magnifications.

Figure 4-11. The axial sectional view of sample ESR1150AH-3 (750°C/110MPa), at lower (5x) and higher (20x) magnifications.

Figure 4-12. The axial sectional view of sample ESR1150AH-9 (775°C/82MPa), at lower (5x) and higher (20x) magnifications.

5 Summary and Future Work

A creep test laboratory has been set up at ANL that comprises of fifteen lever-arm test frames capable of testing ASTM-size specimens of the commercial heat of Alloy 709, in support of the Alloy 709 Code Case development. In FY21, the effort was focused on the preliminary design basis and the 100,000-hour code cast testing of the age-hardened ESR-1150°C material. Twenty two tests were initiated, of which ten were ruptured. The results enabled the preliminary analysis of the creep behavior of this material. The deformation microstructures were characterized using optical microscopy, and it was found that the variation in test temperature and stress resulted in systematic variation in grain structures and in the distribution of voids/cracks. Precipitates were observed in all the samples at grain boundaries and within grains.

Future work involves the completion of the code-case testings, and perform a systematic study of the creep performance of the Alloy 709 in a large temperature and stress window on different material conditions. The ultimate goal is to deliver the Alloy 709 code case to ASME.

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Argonne National Laboratory

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