Report on the initiation of planned FY18 short and intermediate term creep rupture tests and creep-fatigue tests on the first commercial heat of Alloy 709

Applied Materials Division

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Report on the initiation of planned FY18 short and intermediate term creep rupture tests and creep-fatigue tests on the first commercial heat of Alloy 709

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August 2018

ABSTRACT

This report provides an update on the initiation of planned FY18 short and intermediate term creep rupture tests and creep-fatigue tests on the first commercial heat of Alloy 709 in support of the design and operation of structural components in sodium-cooled fast reactors (SFRs). The report is a Level 3 deliverable in FY18 (M3NT-18AN050502031), under the Work Package NT-18AN05050203, “A709 Code Case Testing” performed by Argonne National Laboratory (ANL), as part of Advanced Reactor Technologies Program.

A creep test laboratory has been set up that comprises of 15 indirectly loaded test frames capable of testing ASTM-size specimens of the commercial heat of Alloy 709. The systems are equipped with high temperatures furnaces capable of operation up to 1000°C in an air environment. Type 316L stainless steel was used as the surrogate material to validate operation of creep machines, specimen design, and creep test procedure. Uniaxial creep specimens were fabricated from plates that were processed by AOD, ESR, and ESR-homogenized routes and subsequently solution annealed at 1050, 1100, and 1150°C. ASTM-size creep specimens were fabricated from all the 9 plates and initiated majority of testing at 330MPa and 600°C. Ten creep tests were completed and the results are presented in this report. In addition, fracture surfaces and polished sections in the region of the fractures were analysed using a scanning electron microscope.

Low-cycle fatigue and creep-fatigue tests are also planned on the commercial heat of Alloy 709. Specimens have been fabricated and have initiated fatigue tests.

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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 simple 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 SFR systems, code qualification of Alloy 709 was recommended (Sham et al. 2015). A comprehensive plan for the development of a 500,000-hour, 760°C ASME Code Case and the resolution of licensing issues for Alloy 709 was developed in FY15. A Phase I implementation of this plan that includes a 100,000-hour, 650°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 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 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. 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 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. 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.

One of the major concerns in applications of the advanced alloys in sodium-cooled fast reactors is their long-term microstructural stability and associated degradation of mechanical properties during service. Previous work (Li, et al. 2011, 2013) has showed that tensile properties of these advanced materials degrade with time, when exposed to high temperatures and to liquid sodium. Significant microstructural changes during prolonged thermal and sodium exposures can lead to dramatically different responses to service environments.

Last year, the fabrication scale-up effort was completed for Alloy 709 that was started in FY16. 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. The ingots were rolled into plates which were subsequently delivered to ANL and ORNL. Several samples were cut from the as-rolled test pieces and heat treated 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. 2017).

This report discusses the activities pertaining to the initiation of FY18 short and intermediate term creep rupture tests, fatigue, and creep-fatigue tests on the first commercial heat of Alloy 709. The execution of the Phase I plan was initiated in FY16. 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 stood 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 code case. Fatigue test frames were equipped with high-temperature furnaces and high temperature extensometers for the conduct of fatigue and creep-fatigue tests. In collaboration with material vendor, G.O. Carlson Inc. of Pennsylvania, the ART program successfully scaled the production of Alloy 709 from a laboratory heat of 500 pounds to a commercial heat of 55,000 pounds. Hot-rolled plates were fabricated from the first commercial heats to support testing. Plates from three different melting/casting processes (AOD, ESR, and ESR-Homogenized) and three different solution anneal treatments were used for the testing program.

2 Commercial Heat Specifications

2.1 Composition and Heat Treatments

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 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.%.

The melting and fabrication procedures as well as the heat treatments for the hot-rolled plates were discussed in an earlier report (Natesan et al. 2017). The master heat was produced to provide the project with sufficient material under different processing conditions such as Argon-Oxygen-Decarburization (AOD), Electroslag Remelting (ESR), and Electroslag Remelting with subsequent Homogenization (ESR-Homogenized). The AOD, ESR, and ESR-Homogenized

plates were solution annealed at 1050°C (75 minutes), 1100°C (75 minutes), and 1150°C (60 minutes).

In addition, information on the microstructural characterization, hardness property, and room-temperature tensile properties of the commercial heat Alloy 709 were also presented earlier. The results indicated 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 heat of NF709.

3 Mechanical Test Facilities

3.1 Creep Test Facilities

A creep test laboratory was set up for the project at ANL in Building 212. Figure 3-1 shows a photograph of the creep test laboratory. The laboratory contains twelve newly procured creep frames and three existing frames that were upgraded to meet the requirements. The creep machines 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 that incorporate a number of enhanced design features. Some of these features include:

- Wide-frame construction, which allows for a variety of environmental chambers, fixtures, and other accessories, while maintaining compact overall dimensions
- Counterbalanced lever arm with precision ratio adjustment and four-position rotatable hardened knife edges
- Precision drawhead guide assembly, providing automatic beam leveling, “weightless” specimen loading, hot-step loading, stress relaxation, constant stress, and more via WinCCS II software
- Durable vibration isolator mounts to prevent disturbance to other sensitive equipment upon specimen breakage
- On-center loading at high load ratio, providing optimum strength and minimum deflection
- Rugged vee-block supports for maximum linear knife-edge contact
- Knife edges of high-strength tool steel, designed for easy replacement of worn edges
- Unique lever arm design permits the construction of high load ratios, e.g., 50:1, with standard arm and machine dimensions

Each creep machine is equipped with a three-zone split-tube furnace capable of operation up to 1100°C. Following the setup of the systems, the furnaces were baked to remove any impurities in the refractory insulation. 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. The systems are also equipped with high-limit alarms and over-temperature controllers. Figure 3-2 shows the photograph of a single machine with furnace and sample being tested.

Figure 3-1. Creep test laboratory set up at ANL for testing of Alloy 709.

Figure 3-2. Photograph of a single machine with furnace and a specimen indirectly loaded for the test.

3.2 Creep Specimen Design and Validation of 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.” In order to validate the specimen design/fabrication, test set up and operation of creep test machines, ASTM-standard specimens were fabricated from Type 316L

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stainless steel and tested at 225 MPa at 600°C, in all the test frames. Figure 3-3 shows the creep specimen drawing used for the fabrication of 316L stainless steel specimens and tested at 650°C, 225 MPa. Figure 3-4 shows the macroscopic photographs of the 316L stainless steel specimens after rupture, indicating fracture in the center region of the gage section in each of the specimens.

3.3 Creep Test Data on Alloy 709 Commercial Heat

Creep specimens of commercial heat of Alloy 709 was fabricated similar to the stainless steel and Figure 3-5 shows the specimen dimensions along with the grooves for the extensometer attachment. In general tests were conducted at 600°C, 330 MPa, except the early three tests (one of AOD, ESR, and ESR-H annealed at 1100°C) were performed at 600°C, 300 MPa. In all, 10 tests were completed and all the others are in progress. The two fractured parts of each specimen was used to measure the final rupture strain for the specimen and compared the measured values with the extensometer readings. Figure 3-6 shows a photograph of a typical setup of a specimen for testing. Figure 3-7 shows photographs of the ruptured specimens, indicating that the fracture occurred in the center region of the gage section in all the tested specimens.

Figure 3-8 shows the creep strain as function of time for commercial heat of Alloy 709 with different processing and annealing conditions, tested at 600°C, 330 MPa. Duplicate tests are planned for each processing and annealing condition. The curves depict the behavior of the specimens that have ruptured thus far. Additional tests are in progress. The preliminary test results show that the annealing temperatures of 1100 and 1150°C exhibit superior performance under creep loading than at 1050°C, irrespective of the processing condition.

Figure 3-9 and 3-10 are the bargraphs for creep rupture life and creep elongation, for the commercial heat of Alloy 709 with different processing and annealing conditions, tested at 600°C, 330 MPa. Figures 3-11 and 3-12 show the reduction in area and minimum creep rate for the ruptured specimens of the alloy with different processing and annealing conditions, tested at 600°C, 330 MPa. The minimum creep rates are significantly lower for the specimens that were solution annealed at 1100 and 1150°C compared to those annealed at 1050°C, irrespective of the processing condition (AOD, ESR, and ESR-H).

Table 2-2 lists the rupture time, creep strain at rupture recorded using extensometer, and measured strain at the end of rupture, and the measured reduction of area. In addition, the table also lists the accumulated time and accumulated strain in the ongoing creep tests. In general, all the tests are conducted at 600°C, 330 MPa except specimens AOD/1100°C/specimen 1, ESR/1100°C/specimen 1, and ESR-H/1100°C/specimen 1 which are being conducted at 600°C, 300 MPa.

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Figure 3-5. Schematic of specimens fabricated from commercial heat of Alloy 709 for creep testing.

Figure 3-6. Typical set up of a specimen for testing.

Figure 3-7. Photographs of Alloy 709 specimens creep tested at 600°C, 330 MPa.

Figure 3-8. Creep strain as function of time for commercial heat of Alloy 709 with different processing and annealing conditions, tested at 600°C, 330 MPa.

Figure 3-9. Creep rupture life for commercial heat of Alloy 709 with different processing and annealing conditions, tested at 600°C, 330 MPa.

Figure 3-10. Creep-rupture elongation for commercial heat of Alloy 709 with different processing and annealing conditions, tested at 600°C, 330 MPa.

Figure 3-11. Creep-rupture reduction of area for commercial heat of Alloy 709 with different processing and annealing conditions, tested at 600°C, 330 MPa.

Figure 3-12. Minimum creep rate for commercial heat of Alloy 709 with different processing and annealing conditions, tested at 600°C, 330 MPa.

Table 2-2. ANL creep test data on Alloy 709 commercial heat

	Rupture Time (h)	Elongation Machine (%)	Elongation, Measured (%)	Reduction of Area (%)	Minimum Creep Rate (%/h)
AOD 1050°C Specimen 1	682.7	12.8	17.2	22.8	1.1×10^{-2}
AOD 1050°C Specimen 2	577.8	13.3	15.6	21.9	1.2×10^{-2}
AOD 1100°C Specimen 1* ♦	2469	5.0	-	-	-
AOD 1100°C Specimen 2	1752.3	20.5	26.0	34.4	3.7×10^{-3}
AOD 1150°C Specimen 1*	1802	6.9	-	-	-
AOD 1150°C Specimen 2*	1655	6.7	-	-	-
ESR 1050°C Specimen 1	714.1	29.0	30.5	30.8	1.7×10^{-2}
ESR 1050°C Specimen 2	741.6	32.0	30.7	31.3	1.7×10^{-2}
ESR 1100°C Specimen 1* ♦	2470	5.5	-	-	-
ESR 1100°C Specimen 2	2168	30.4	33.6	45.1	3.3×10^{-3}
ESR 1150°C Specimen 1*	1780	5.1	-	-	-
ESR 1150°C Specimen 2*	1680	4.6	-	-	-
ESR-H 1050°C Specimen 1	682.2	15.1	16.4	18.0	1.2×10^{-2}
ESR-H 1050°C Specimen 2	700.0	16.1	17.6	19.5	1.2×10^{-2}
ESR-H 1100°C Specimen 1* ♦	2487	5.0	-	-	-
ESR-H 1100°C Specimen 2	1624.1	17.6	23.9	29.5	3.7×10^{-3}
ESR-H 1150°C Specimen 1*	1802	6.1	-	-	-
ESR-H 1150°C Specimen 2	1703	7.0	14.9	23.0	2.3×10^{-3}

The tests are conducted at 600°C, 330MPa unless indicated otherwise.

*Indicates test is in progress and the accumulated time and strain are given in the table.

♦ indicates tests are conducted at 600°C, 300 MPa.

3.4 Microstructural Analysis of Creep-Tested Specimens

The fracture surfaces and polished sections of the regions near the fracture of several of the ruptured specimens were examined using scanning electron microscopy. Figure 3-13 shows SEM photomicrographs of fracture surfaces of creep-tested specimens with AOD, ESR, and ESR-H processing conditions and post-annealed annealed at 1050°C. The micrographs indicate a ductile fracture and are similar in all processing conditions. Figure 3-14 shows SEM photomicrographs of fracture surfaces of creep-tested specimens with AOD and ESR-H processing and annealed at 1100°C. There is little difference between the specimens annealed at 1050 and 1100°C. Figures 3-15 through 3-17 show the SEM photomicrographs of polished surfaces near the fracture region of creep-tested specimens with AOD, ESR, and ESR-H processing, respectively, for 1050°C solution anneal treatment. The figures show classic creep cavitation and intergranular fracture in entire cross section of the specimens in all cases. Figures

3-18 and 3-19 show the SEM photomicrographs of polished surfaces near the fracture region of creep-tested specimen with AOD and ESR-H processing conditions and annealed at 1100°C. The cracks are intergranular and similar to those observed in specimens annealed at 1050°C. Microstructural analysis of additional specimens is in progress and will continue as additional tests are completed.

Figure 3-13. SEM photomicrographs of fracture surfaces of creep-tested specimens with different processing conditions, annealed at 1050°C.

Figure 3-14. SEM photomicrographs of fracture surfaces of creep-tested specimens with AOD and ESR-H processing conditions, annealed at 1100°C.

Figure 3-15. Optical photomicrographs of polished surfaces near the fracture region of creep-tested specimen with AOD processing condition and annealed at 1050°C.

Figure 3-16. Optical photomicrographs of polished surfaces near the fracture region of creep-tested specimen with ESR processing condition and annealed at 1050°C.

Figure 3-17. Optical photomicrographs of polished surfaces near the fracture region of creep-tested specimen with ESR-H processing condition and annealed at 1050°C.

Figure 3-18. Optical photomicrographs of polished surfaces near the fracture region of creep-tested specimen with AOD processing condition and annealed at 1100°C.

Figure 3-19. Optical photomicrographs of polished surfaces near the fracture region of creep-tested specimen with ESR-H processing condition and annealed at 1100°C.

3.5 Fatigue and Creep-Fatigue Testing of Commercial Heat of Alloy 709

Fatigue and creep-fatigue tests are also planned for the commercial heat of Alloy 709. ASTM standard-size round creep-fatigue specimens are fabricated from two heats of A709, namely, AOD solution annealed at 1100°C (plate ID 4B-1) and AOD solution annealed at 1150°C (plate ID 4C-1). The geometry and dimensions of the creep-fatigue specimens are shown in Fig. 3-20. Gauge sections of creep-fatigue specimens are polished longitudinally with 1-μm diamond paste to remove surface scratches and oxide layer, if any, before creep-fatigue testing.

Creep-fatigue tests are performed on closed-loop servo-hydraulic test frames at ANL. Specimen heating is achieved by a three-zone split furnace. The test temperature is controlled and monitored by two Type K thermocouples wired at the top and bottom grip sections of the specimen. A high temperature extensometer is used to control the total axial strain. All creep-fatigue tests are carried out in air. The creep-fatigue test systems are shown in Fig. 3-21.

Creep-fatigue tests are conducted in the strain-controlled, fully-reversed mode. Creep-fatigue damage is introduced by applying a hold time at peak strains either in tension, or compression, or in both tension and compression. The Instron WaveMatrix™ Dynamic and Fatigue Materials Testing Software is used for applying various loading conditions. The initial round-robin tests are planned for the total strain amplitude of 0.5% with a tension hold of 30 min.

Figure 3-21. High temperature creep-fatigue test systems at ANL.

4 Summary

We have initiated the planned FY18 short and intermediate term creep rupture tests and creep-fatigue tests on the first commercial heat of Alloy 709 in support of the design and operation of structural components in sodium-cooled fast reactors (SFRs). The report summarizes the experimental approach and preliminary data generated on creep properties of AOD, ESR, and ESR-Homogenized plates of Alloy 709.

The creep test laboratory has been set up that comprises of 15 indirectly loaded test frames capable of testing ASTM-size specimens of the commercial heat of Alloy 709 at temperatures up to 1000°C. Type 316L stainless steel was used as the surrogate material to validate operation of creep machines, specimen design, and creep test procedure. Uniaxial creep specimens were fabricated from plates that were processed by AOD, ESR, and ESR-homogenized routes and subsequently solution annealed at 1050, 1100, and 1150°C. ASTM-size creep specimens were fabricated from all the 9 plates and initiated testing at 330MPa and 600°C. Ten creep tests were completed and the results are presented in this report. In addition, fracture surfaces and polished sections in the region of the fractures were analysed using a scanning electron microscope.

Low-cycle fatigue and creep-fatigue tests are also planned on the commercial heat of Alloy 709. Specimens have been fabricated and have initiated fatigue tests.

Acknowledgement

The research was sponsored by the U.S. Department of Energy, under Contract No. DE-AC02-06CH11357 with Argonne National Laboratory, managed and operated by UChicago Argonne LLC. Programmatic direction was provided by the Office of Nuclear Energy.

The authors gratefully acknowledge the support provided by Alice Caponiti, Director, Office of Advanced Reactor Technologies (ART), Sue Lesica, Federal Manager, ART Advanced Materials Program, Robert Hill of ANL, National Technical Director, ART Fast Reactors Campaign, and Sam Sham of ANL, ART Technology Area Lead on Advanced Materials.

The authors acknowledge the assistance of E. Listwan, D. L. Rink, J. Listwan, and R. Kmak for specimen design/preparation, tests set up, and data acquisition for the creep and fatigue tests.

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