FY20 Status Report on Creep Rupture Testing to Support the Development of Alloy 709 Code Case

Applied Materials Division

About Argonne National Laboratory

Argonne is a U.S. Department of Energy laboratory managed by UChicago Argonne, LLC under contract DE-AC02-06CH11357. The Laboratory's main facility is outside Chicago, at 9700 South Cass Avenue, Argonne, Illinois 60439. For information about Argonne and its pioneering science and technology programs, see <http://www.anl.gov>.

DOCUMENT AVAILABILITY

Online Access: U.S. Department of Energy (DOE) reports produced after 1991 and a growing number of pre-1991 documents are available free at OSTI.GOV (<http://www.osti.gov/>), a service of the US Dept. of Energy's Office of Scientific and Technical Information.

Reports not in digital format may be purchased by the public from the National Technical Information Service (NTIS):

U.S. Department of Commerce
National Technical Information
Service 5301 Shawnee Rd
Alexandria, VA 22312
www.ntis.gov
Phone: (800) 553-NTIS (6847) or (703) 605-6000
Fax: (703) 605-6900
Email: **orders@ntis.gov**

Reports not in digital format are available to DOE and DOE contractors from the Office of Scientific and Technical Information (OSTI):

U.S. Department of Energy
Office of Scientific and Technical Information
P.O. Box 62
Oak Ridge, TN 37831-0062
www.osti.gov
Phone: (865) 576-8401
Fax: (865) 576-5728
Email: **reports@osti.gov**

Disclaimer

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor UChicago Argonne, LLC, nor any of their employees or officers, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of document authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof, Argonne National Laboratory, or UChicago Argonne, LLC.

FY20 Status Report on Creep Rupture Testing to Support the Development of Alloy 709 Code Case

Prepared by

X. Zhang

Nuclear Science and Engineering Division
Argonne National Laboratory

T.-L. Sham

Applied Materials Division
Argonne National Laboratory

August 2020

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. A creep test laboratory has been set up at ANL 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 temperature furnaces capable of operation up to 1000°C in an air environment. During FY18, uniaxial ASTM-sized creep specimens were fabricated from 9 Alloy 709 plates that were processed by AOD, ESR, and ESR-homogenized routes and subsequently solution annealed at 1050, 1100, and 1150°C, and creep-rupture tests were performed at 330MPa and 600°C. The test results led to the down selection of the ESR-1100°C condition to support the code case. In FY19, 15 creep-rupture tests of the ESR-1100°C material, requiring the use of Type K thermocouples, were initiated to support the 100,000-hour Code Case. 8 tests were completed in FY19. In FY20, 6 tests were completed at the time of writing this report. In addition, 1 more test were loaded in FY20, using Type S thermocouples, and was completed. This report presents the data from the completed tests in FY20 and also the microstructural characterization of the fractured specimens. The creep data enabled the preliminary analysis of the creep behavior of this material. The characterization showed that in brittle fractures, the pores were narrow and thin, and elongated in the direction perpendicular to the loading direction, while in ductile fractures, the pores were much rounder. Dense precipitates were observed in all the samples, the size of which grew with test temperature. Some of the precipitates could be identified to be M₂₃C₆ based on their cuboid shape.

TABLE OF CONTENTS

ABSTRACT.....	i
Table of Contents	iii
List of Tables	v
List of Figures	vii
1 Introduction	1
2 Material Specifications and Mechanical test facilities	3
2.1 Materials and specimens	3
2.2 Creep test facilities.....	4
2.3 Creep test procedure	7
3 Creep Test Data on Alloy 709 Commercial Heat ESR-1100°C Condition.....	9
4 Microstructural Analysis of Creep-Tested Specimens	15
4.1 Grain structures, deformation defects and precipitates	16
5 Summary and Future Work	25
ACKNOWLEDGEMENT	27
REFERENCES	29

LIST OF TABLES

Table 2-1.	Chemical composition of melt of Alloy 709 heat # 58776 (in wt.%).....	3
Table 3-1.	FY20 Alloy 709 code case testing progress.....	10

LIST OF FIGURES

Figure 2-1.	Schematic drawing of ASTM standard-sized creep specimens.....	3
Figure 2-2.	Creep test laboratory set up at ANL for testing of Alloy 709.....	5
Figure 2-3.	Photograph of a machine with furnace and a specimen loaded for the test, with Type K thermocouples spot welded to the shoulders of the specimen.....	6
Figure 2-4.	Photograph of a specimen loaded into the test rig, with Type S thermocouples fastened by platinum wires to the two ends of the specimen gauge section.....	7
Figure 2-5.	The loading curve of an Alloy 709 sample being loaded to 330 MPa at 625°C. ..	8
Figure 3-1.	Plots of the initial loading strain as a function of test temperature and stress for the ruptured A709 specimens.....	10
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..	11
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.	11
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 15 completed tests in FY19 and FY20.	12
Figure 3-5.	Larson-Miller plot of the completed tests of the commercial heat of Alloy 709 ESR-1100°C condition	12
Figure 3-6.	Minimum creep rate as a function of applied stress for tests at different temperatures.....	13
Figure 4-1.	Photos of the FY20 creep-ruptured A709 specimens	15
Figure 4-2.	A photo showing the polished surfaces of the cross section and axial section of the crept specimens.....	17
Figure 4-3.	Top: the axial sectional view of sample BB2-23 (550°C/385MPa), at lower (5x) and higher (50x) magnifications. Bottom: the cross sectional view of sample BB2-23 (550°C/385MPa), at lower (5x) and higher (50x) magnifications	18
Figure 4-4.	Top: the axial sectional view of sample BB2-19 (575°C/355MPa), at lower (5x) and higher (50x) magnifications. Bottom: the cross sectional view of sample BB2-19 (575°C/355MPa), at lower (5x) and higher (50x) magnifications.....	19
Figure 4-5.	Top: the axial sectional view of sample BB2-20 (575°C/330MPa), at lower (5x) and higher (50x) magnifications. Bottom: the cross sectional view of sample BB2-20 (575°C/330MPa), at lower (5x) and higher (50x) magnifications.....	20
Figure 4-6.	Top: the axial sectional view of sample BB2-12 (600°C/285MPa), at lower (5x) and higher (50x) magnifications. Bottom: the cross sectional view of sample BB2-12 (600°C/285MPa), at lower (5x) and higher (50x) magnifications.....	21

Figure 4-7.	The axial sectional view of sample BB2-16 (625°C/200MPa), at lower (5x) and higher (50x) magnifications	22
Figure 4-8.	Top: the axial sectional view of sample BB2-18 (650°C/175MPa), at lower (5x) and higher (50x) magnifications. Bottom: the cross sectional view of sample BB2-18 (650°C/175MPa), at lower (5x) and higher (50x) magnifications	23
Figure 4-9.	Top: the axial sectional view of sample BB2-29 (675°C/175MPa), at lower (5x) and higher (50x) magnifications. Bottom: the cross sectional view of sample BB2-29 (675°C/175MPa), at lower (5x) magnifications.....	24

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 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, 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 (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. 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.

In FY17, 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. 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 9 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. 2017).

In FY18, activities focused on the scoping tests for down selection from the 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 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 scoping tests and the code case. In FY18, 600°C/330 MPa tests were carried out on all 9 processing conditions (Natesan, et al. 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 100,000-hour code case testing. 15 creep-rupture tests at temperatures below or equal to 700°C, thus requiring the use of Type K thermocouples, were initiated. 8 tests were completed in FY19 (Zhang, et al. 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 5 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 100,000-hour code case testing. The addition of Type S thermocouples were completed on 5 test frames, and 1 tests at 675°C/175MPa were loaded. In total, 7 tests were completed in this FY.

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 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 material selected for the code case has a processing condition of ESR with solution annealing at 1100°C after rolling. 23 ASTM standard-sized specimens (Fig. 2-1) were fabricated for creep rupture tests. 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. Schematic drawing of ASTM standard-sized creep specimens.

2.2 Creep test facilities

A creep test laboratory was set up for the project at ANL in Building 212. Figure 2-2 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 2-3 shows the photograph of a single machine with a specimen being loaded. Two type-K thermocouples were spot welded to the shoulder sections of the specimen as the furnace temperature feedback. This direct contact method minimizes the errors in temperature readings due to incorrect electrical contact between the thermocouple wires and/or between the wires and the specimen. It also ensures the efficient heat conduction between the thermocouple and the specimen, thus providing an accurate measure of the specimen temperature.

Figure 2-2. Creep test laboratory set up at ANL for testing of Alloy 709.

Figure 2-3. Photograph of a machine with furnace and a specimen loaded for the test, with Type K thermocouples spot welded to the shoulders of the specimen.

Type K thermocouple is known to have drift issues during prolonged exposure to very high temperature. Communications with Oak Ridge National Laboratory suggested that Type S thermocouples should be used for test temperatures higher than 675°C and lifetime longer than 4,000 hours. Therefore, addition of Type S thermocouples to 5 of the creep frames were carried out. 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, which will change the thermocouple's property. 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 thermocouples were fastened by platinum wires to the specimen, as shown in Figure 2-4.

Figure 2-4. Photograph of a specimen loaded into the test rig, with Type S thermocouples fastened by platinum wires to the two ends of the specimen gauge section.

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 1 hour. The load was then applied in a step manner and fully loaded in about 5 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 2-5. In this case, the end stress was 330 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 2-5. The loading curve of an Alloy 709 sample being loaded to 330 MPa at 625°C.

3 Creep Test Data on Alloy 709 Commercial Heat ESR-1100°C Condition

15 creep tests were initiated in FY19 in support of the 100,000-hour code case. 1 creep test was initiated in FY20. The material is the commercial heat of the Alloy 709 ESR-1100°C condition. Table 3-1 lists the test parameters and the progress at the time of writing this report. 8 tests have been completed in FY19, under 600°C/355 MPa, 600°C/330 MPa, 625°C/330 MPa, 625°C/285 MPa, 650°C/200 MPa, 675°C/200 MPa, 700°C/175 MPa and 700°C/155 MPa conditions. 7 tests have been completed in FY20, under 625°C/200 MPa, 550°C/380 MPa, 650°C/175 MPa, 575°C/355 MPa, 575°C/330 MPa, 600°C/285 MPa, and 675°C/175 MPa. The elongation and area reduction are both calculated from the measurement of the rupture specimens. Figure 3-1 plots the initial loading strains as a function of test temperature and stress for all the 15 tests completed to date, ranging from 0.1% for the 700°C/155 MPa condition to 8.1% for the 550°C/380 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 higher temperature and lower stress leads to a more ductile failure. At temperatures equal and above 625°C, the elongations are more than 50% and the area reductions are more than 70%.

Figure 3-4 plots the creep curves of the 15 completed tests. Due to the limited range of the LVDTs, once the creep strain is over ~40%, the reading stops. Therefore, to reflect the real creep strain, in the figure the end creep strains, measured from the ruptured specimens, are plotted for those tests. The one test in progress is not plotted.

Figure 3-5 is the Larson-Miller plot of the 15 completed tests, together with the 2 tests in FY18 (600°C/330MPa and 600°C/300MPa) of the same ESR-1100°C material. The Larson-Miller parameter 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 chosen to be 14.04 because it gives the best linear regression. The fitted line gives

$$LMP = -4401.06 \times \log_{10} \sigma + 26163.52 .$$

From the above two equations, the creep life t_r of the material can be estimated knowing the stress and the temperature:

$$\log_{10} t_r = \frac{-4401.06 \times \log_{10} \sigma + 26163.52}{T + 273.15} - 14.04 .$$

Note that as more tests are completed in the future, the parameters in the above equations are subjected to revision.

Table 3-1. FY20 Alloy 709 code case testing progress.

test # (ANL)	sample ID	Stress (MPa)	Temp (°C)	Status	Completed in
1	BB2-15	355	550	running	--
2	BB2-16	200	625	ruptured	FY20
3	BB2-23	380	550	ruptured	FY20
4	BB2-18	175	650	ruptured	FY20
5	BB2-19	355	575	ruptured	FY20
6	BB2-20	330	575	ruptured	FY20
7	BB2-10	355	600	ruptured	FY19
8	BB2-8	330	600	ruptured	FY19
9	BB2-12	285	600	ruptured	FY20
10	BB2-3	330	625	ruptured	FY19
11	BB2-13	285	625	ruptured	FY19
12	BB2-14	200	650	ruptured	FY19
13	BB2-4	200	675	ruptured	FY19
14	BB2-5	175	700	ruptured	FY19
15	BB2-6	155	700	ruptured	FY19
16 (Type S TC)	BB2-29	175	675	ruptured	FY20

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

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 15 completed tests in FY19 and FY20.

Figure 3-5. Larson-Miller plot of the completed tests of the commercial heat of Alloy 709 ESR-1100°C condition.

The minimum creep rate of the A709 specimens followed a power law relationship with the applied stress, $\dot{\epsilon} = A\sigma^n$, as shown in Figure 3-6. The power exponents are indicated for different temperatures. This indicates that the primary creep mechanism is the dislocation creep.

Figure 3-6. Minimum creep rate as a function of applied stress for tests at different temperatures.

4 Microstructural Analysis of Creep-Tested Specimens

Figure 4-1 shows the photos of the FY20 creep-ruptured Alloy 709 specimens. All the specimens failed at the gauge section, with the majority failed around the gauge center. The deformation microstructures were studied by optical microscopy. Due to the facility access restrictions imposed during the COVID-19 pandemic period, investigation of the fracture surface using the scanning electron microscopy (SEM) was not carried out.

Figure 4-1. Photos of the FY20 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 7 creep-ruptured specimens that were ruptured in FY20. The cross sections (round/half-round pieces) were cut out very close to the fracture surface from one half of the broken specimen, and the axial sections (long pieces) were the mid-section of the gage region near the fracture surface of the other half of the broken specimen. Those surfaces were polished to mirror finish and were etched for optical microscopy study. Optical microscopy of the grain structures and deformation defects revealed that as the test temperature and stress varies, drastic differences in microstructures exist. Micrographs for the 6 specimens are shown in Figures 4-3 to 4-9. Figures 4-3 and 4-4 show the microstructures of BB2-23 (550°C/385MPa) and BB2-19 (575°C/355MPa), respectively. The cracks of those two specimens are narrow and thin, extending along grain boundaries in the direction perpendicular to the loading direction, indicating that the fracture mode is rather brittle. This agrees with the observation in Figure 4-1, where specimens BB2-23 and BB2-19 show little necking. Figures 4-5 to 4-9 show the microstructures of BB2-20 (575°C/330MPa), BB2-12 (600°C/285MPa), BB2-16 (625°C/200MPa), BB2-18 (650°C/175MPa), and BB2-29 (675°C/175MPa). The pores, although still located at grain boundaries, are much rounder compared to those in BB2-23 and BB2-19, indicating that the fracture mode is rather ductile.

The fracture mode can also be correlated with the initial loading strain. For the samples that have loading strains of 5% or higher, namely, BB2-23 (FY20), BB2-19 (FY20), BB2-20 (FY20), BB2-10 (FY19), BB2-8 (FY19), and BB2-3 (FY19), they were all rather brittle than ductile. However, we have to note that those samples were all tested at lower temperature end (lower than 625°C). Therefore, the fracture mode should be a result of the combined effect from the test temperature and the loading strain.

For all the specimens except BB2-18 (650°C/175MPa) and BB2-29 (675°C/175MPa), the original grain structure are preserved up to the fracture surface, with elongations in the loading direction. The observation is very different for BB2-18 and BB2-29, which was deformed at the highest temperatures (650°C and 675°C) among all. In those two specimens, the original grain structures are not very distinguishable near the fracture surface. As a result, it is hard to distinguish whether the pores are intergranular or intragranular. This observation is in agreement with that from the previous FY (Zhang, et al. 2019), where it is also observed that samples tested at or above 650°C had the original grain structure disappearing near the fracture surface, indicating a recrystallization mechanism being active at 650°C and above.

Another important feature is precipitation. Dense precipitates can be observed in all the samples at high magnification. The precipitate size grows with test temperature. Although no characterization has been done so far to characterize the precipitate types, Figure 4-6 and Figure 4-8 show that some the precipitates are cuboids, implying that they are $M_{23}C_6$ ($M = Cr$, with Ni, Fe, Mo often being substitutes).

Figure 4-2. A photo showing the polished surfaces of the cross section and axial section of the crept specimens.

Figure 4-3. Top: the axial sectional view of sample BB2-23 (550°C/385MPa), at lower (5x) and higher (50x) magnifications. Bottom: the cross sectional view of sample BB2-23 (550°C/385MPa), at lower (5x) and higher (50x) magnifications.

Figure 4-4. Top: the axial sectional view of sample BB2-19 (575°C/355MPa), at lower (5x) and higher (50x) magnifications. Bottom: the cross sectional view of sample BB2-19 (575°C/355MPa), at lower (5x) and higher (50x) magnifications.

Figure 4-5. Top: the axial sectional view of sample BB2-20 (575°C/330MPa), at lower (5x) and higher (50x) magnifications. Bottom: the cross sectional view of sample BB2-20 (575°C/330MPa), at lower (5x) and higher (50x) magnifications.

Figure 4-6. Top: the axial sectional view of sample BB2-12 (600°C/285MPa), at lower (5x) and higher (50x) magnifications. Bottom: the cross sectional view of sample BB2-12 (600°C/285MPa), at lower (5x) and higher (50x) magnifications.

Figure 4-7. The axial sectional view of sample BB2-16 (625°C/200MPa), at lower (5x) and higher (50x) magnifications.

Figure 4-8. Top: the axial sectional view of sample BB2-18 (650°C/175MPa), at lower (5x) and higher (50x) magnifications. Bottom: the cross sectional view of sample BB2-18 (650°C/175MPa), at lower (5x) and higher (50x) magnifications.

Figure 4-9. Top: the axial sectional view of sample BB2-29 (675°C/175MPa), at lower (5x) and higher (50x) magnifications. Bottom: the cross sectional view of sample BB2-29 (675°C/175MPa), at lower (5x) magnifications.

5 Summary and Future Work

A creep test laboratory has been set up at ANL 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 temperature furnaces capable of operation up to 1000°C in an air environment. In FY19, 15 creep-rupture tests on the first commercial heat of Alloy 709 with ESR-1100 were initiated to support the 100,000-hour Code Case. In FY20, 1 more test was initiated. At the time of writing this report, 15 tests have been completed. The results enabled the preliminary analysis of the creep behavior of this material. The deformation microstructures were characterized using optical microscopy, and was found that in brittle fractures, the pores were narrow and thin, and elongated in the direction perpendicular to the loading direction, while in ductile fractures, the pores were much rounder. Dense precipitates were observed in all the samples, the size of which grew with test temperature. Some of the precipitates could be identified to be $M_{23}C_6$ based on their cuboid shape.

Future work involves the testing and characterization of other code-case conditions, 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.

ACKNOWLEDGEMENT

Programmatic direction was provided by the Office of Nuclear Reactor Deployment of the Office of Nuclear Energy.

The authors gratefully acknowledge the support provided by Sue Lesica, Federal Manager, Advanced Materials, Advanced Reactor Technologies (ART) Program, Brian Robinson, Federal Manager, ART Fast Reactors (FR) Campaign, and Robert Hill of Argonne National Laboratory, National Technical Director, ART FR Campaign, and Meimei Li of ANL, group manager of the Nuclear Materials group.

The authors acknowledge the assistance of E. Listwan and R.L. Rink for specimen preparation, test set-up, and data acquisition of the creep tests.

REFERENCES

Busby, J. T., S. Byun, R. Klueh, P. Maziasz, and J. Vitek, K. Natesan, M. Li, R. Wright, S. Maloy, M. Toloczko, A. Motta, B. D. Wirth, G. R. Odette, and T. Allen, “Candidate Developmental Alloys for Improved Structural Materials for Advanced Fast Reactors,” ORNL/GNEP/LTR-2008-023, March 2008.

Natesan, K., X. Zhang, T.-L. Sham, and H. Wang, Argonne National Laboratory Report, ANL-ART-89, June 2017.

Natesan, K., X. Zhang, and M. Li, Argonne National Laboratory Report, ANL-ART-151, August 2018.

Zhang, X, and T.-L. Sham, Argonne National Laboratory Report, ANL-ART-172, August 2019.

Applied Materials Division

Argonne National Laboratory

9700 South Case Avenue

Argonne, IL 60439

www.anl.gov

Argonne National Laboratory is a U.S. Department of Energy
laboratory managed by UChicago Argonne, LLC