FY19 Report on the Status of Sodium Exposure Tests of the A709 First Commercial Heat

Nuclear Science and Engineering Division

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Prepared by
Meimei Li, Wei-Ying Chen, Yoichi Momozaki
Nuclear Science and Engineering Division
Argonne National Laboratory

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ABSTRACT

This report provides an update on the understanding of the effects of sodium exposures on tensile properties of advanced alloy A709 in support of the design and operation of structural components in sodium-cooled fast reactors (SFRs). The focus of the FY19 program was (a) to complete the sodium exposure of sub-sized specimens from A 709 lab heats and 316H (a reference material) and (b) to continue sodium exposure of sub-sized specimens from A 709 commercial heat 58776.

Sodium exposure tests were conducted on H58776 of A709 involving six different processing and heat treatment conditions. The melt procedure of H58776 included three processing conditions, namely, Argon-Oxygen-Decarburization (AOD), Electro-slag Remelting (ESR), and Electro-slag Remelting with subsequent homogenization (ESR-Homo). The A709 of AOD, ESR, and ESR-Homo was solution annealed (SA) at either 1100°C or 1150°C, respectively. Specimens in six different processing/heat treatment conditions, namely, AOD-SA1100°C, AOD-SA1150°C, ESR-SA1100°C, ESR-SA1150°C, Homo-SA1100°C, and Homo-SA1150°C were tested at 650°C in sodium for down-selection of the processing/heat treatment conditions. A 3,000-h sodium exposure test was completed, and a 10,000-h exposure test is in progress. Specimens in the AOD-SA1100°C and AOD-SA1150°C conditions were also tested at 550 and 600°C because of their overall good mechanical performance from initial evaluation. Sodium exposure tests at 550, 600, and 650°C of three lab heats, 709-01, 709-02, and 709-05 were completed. 316H SS was tested as a reference material for comparison with A709. Parallel thermal aging experiments were carried out to obtain thermal aging data for comparison with sodium exposure data to separate the thermal and sodium exposure effects. The major findings are:

- A709 commercial heat and lab heats showed good sodium compatibility showing a corrosion rate of $< 1 \mu\text{m/y}$ at 650°C after the initial transient stage in the oxygen-controlled sodium environment.
- Sodium exposure at 650°C for ~3,000 h increased the yield stress and decreased the ultimate tensile strength, uniform elongation and total elongation for all six processing/heat treatment conditions of A709 H58776.
- Thermal aging at 650°C for ~3,000 h increased the yield stress for the AOD-SA1100°C, AOD-SA1150°C, Homo-SA1150°C specimens, but had a minimal effect on the yield stress for the ESR-SA1100°C, ESR-SA1150°C, and Homo-SA1100°C specimens. It decreased the ultimate tensile strength for all six processing/heat treatment conditions, equivalent to the 650°C sodium exposure effect, and reduced the uniform elongation and the total elongation, either equivalent to or less than the 650°C sodium effect.
- Compared to A709 lab heats, A709 H58776 showed comparable yield stress, ultimate tensile strength, and total elongation, but had lower uniform elongation after thermal aging or sodium exposure for ~3,000 h at 650°C. The distinct difference of the tensile behavior between the commercial heat and lab heats was the complete removal of the dynamic aging effect (DSA) after thermal/sodium exposures in the commercial heat while the DSA effect still existed in the lab heats.
- Thermal aging or sodium exposure at 650°C for ~3,000 h introduced several new phases in A709, mainly M_{23}C_6 carbides, $\text{Cr}_3\text{Ni}_2\text{SiX}$ (X: C, N) particles, and Z-phase precipitates. The matrix precipitates were dominated by Z-phase particles decorating dislocation lines. The grain and twin boundaries were covered by M_{23}C_6 carbides, $\text{Cr}_3\text{Ni}_2\text{SiX}$ (X: C, N) particles. Some

$M_{23}C_6$ carbides, Cr_3Ni_2SiX (X: C, N) particles were also found in the matrix, either being isolated or co-existing forming a shell structure with a carbide surrounded by Cr_3Ni_2SiX .

- Thermal aging 650°C for ~3,000 h produced $M_{23}C_6$ carbides, Cr_3Ni_2SiX (X: C, N) particles predominantly at grain or twin boundaries and a low density of matrix precipitates of Z-phase decorating dislocations. Sodium exposure 650°C for ~3,000 h had a similar effect on grain or twin boundary precipitation, but produced a much higher density of matrix precipitates of Z-phase and dislocations. Sodium exposure also generated a $M_{23}C_6$ carbides segregation zone adjacent to the grain boundary observed only the AOD-SA1100°C specimen but not in the ESR-SA1100°C and Homo-SA1150°C specimens.

This initial assessment has indicated that the sodium exposure effects on microstructure and tensile properties of A709 commercial heat is dependent on the processing/heat treatment condition. A better understanding will be developed by examining the 650°C/10,000 h specimens when they become available.

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1 Introduction

The Advanced Reactor Technologies campaign has focused on the sodium-cooled fast reactor concept because of its technical maturity. The current needs are for more reliable, more flexible, and more economical reactors suitable for commercial nuclear power generation. Research and development focuses on three areas including advanced materials, innovative components and systems, and computer modeling and simulation enabling predictions of long-term performance. Inherent in this research and development is the selection and adequate long-term performance of reactor structural materials that are directly exposed to the sodium coolant at elevated temperatures.

Figure 1 shows a schematic of a sodium-cooled fast reactor (SFR) [1]. In these reactors, heat is generated from fission reactions in a nuclear-fueled core. The heat is removed by liquid sodium flowing through the core, transferred to a steam system, and the steam is used to drive a turbine. Because of excellent heat transfer properties, liquid sodium has been selected as the coolant and heat transport medium. In general, austenitic stainless steels are used for in-core structural components, piping, and intermediate heat exchanger. High chromium ferritic steels are considered for primary piping system, intermediate heat exchanger, secondary piping and steam generator in SFRs. The selection of a particular material and combinations that will be used in both primary and secondary circuits is based upon a number of factors. For in-core applications, the stability of the material in the fast neutron environment, compatibility with the sodium coolant and elevated temperature performance are of primary concern. In the case of the steam generator, the resistance of the material to aqueous chloride and caustic cracking as well as minimization of corrosion wastage rates due to sodium-water reaction products in the event of a tube failure have important bearing on the material selection.

Figure 1. A schematic diagram of a sodium-cooled fast reactor.[1]

In general, structural materials can undergo a variety of interactions upon exposure to liquid sodium. The extent of interaction depends upon the exposure conditions of temperature, temperature gradient, sodium velocity and purity, the materials of construction, and external sources and sinks for the constituent elements of the structural materials. The interactions can be broadly classified into either metallic or nonmetallic element mass transfer. Metallic element mass transfer usually establishes a go/no-go type of evaluation of an alloy for use in sodium environment, based upon the established limits of material wastage or component section loss. A classic example of this is the unacceptable performance of vanadium base alloys in sodium of normal reactor purity. Upon selection of an alloy for sodium service, corrosion allowances that account for metallic element transfer can be incorporated into the design of specific components.

Nonmetallic elements such as oxygen, carbon, nitrogen, and hydrogen are known to migrate in structural materials/sodium systems as a result of chemical activity differences that occur in isothermal or non-isothermal systems. Transfer of these elements also occurs in systems where combinations of materials of different compositions are used. By examining the partitioning of nonmetallic elements between the structural alloy and sodium, the extent to which oxygen, carbon, and nitrogen interact in the structural material-sodium systems can be assessed. From the operation of the sodium fast reactor standpoint, the oxygen impurity in sodium can be controlled to ~1 wppm which would result in acceptable corrosion of austenitic and ferritic steels and nickel-base alloys at anticipated operating temperatures. The solubility of nitrogen in sodium is low in the reactor at the operating temperature, and its effect on materials performance may not be significant. On the other hand, decarburization/carburization in sodium environments can significantly affect the microstructural stability and long-term mechanical performance of austenitic and ferritic steels. It is necessary to evaluate and understand the influence of carbon transport in sodium on the microstructure and mechanical properties of advanced structural materials in SFR sodium environments. Additionally, observations by various investigators that materials of nominally the same composition with different degrees of decarburization/carburization in sodium loops operating over similar temperature ranges have resulted in questions regarding the extrapolation of results obtained in small scale sodium loops to large reactor systems [2]. Since carbon concentration in a given system is established by a dynamic equilibrium between the carbon sources and carbon sinks present in the system, it could vary from system to system as well as over time within a system. An analysis of the carburization-decarburization behavior of austenitic stainless steels in the primary heat-transport systems of the EBR-II and FFTF has been made based upon a kinetic model for the diffusion process and the surface area of steel in contact with flowing sodium at various temperatures in the two systems (shown in Fig. 2 [2]). The role of ingress of carbonaceous materials in altering the carbon concentration in sodium and its effect on materials performance have also been assessed.

The goal of this program is to evaluate and understand the influence of carbon in sodium on the microstructure and mechanical properties of advanced structural materials as well as establish the performance window for the materials in terms of temperature, carbon level in sodium, materials of construction, and the temperature gradient in the sodium circuits. This report presents the status and progress in assessing the corrosion performance, microstructural changes, and tensile properties of advanced alloy, A709 with the focus on the tests on A709 first commercial heat 58776.

Figure 2. Carburization-Decarburization regimes for Types 316 and 304L stainless steels in the FFTF and EBR-II primary sodium systems, respectively, in terms of the carbon concentration in sodium and temperature.[2]

2 Experimental

2.1 Materials and specimens

A709 Commercial Heat 58776

Sodium exposure tests were conducted on the first commercial heat (58776) of A709. The product chemistry of the heat is given in Table 1. It conforms to the ranges or maximum values specified in wt% in Table 1.

The melt procedure involved three different processing conditions, namely, Argon-Oxygen-Decarburization (AOD), Electro-slag Remelting (ESR), and Electro-slag Remelting with subsequent homogenization (ESR-Homo). The A709 of AOD, ESR, and ESR-Homo was solution annealed (SA) at 1100°C (75 minutes) and 1150°C (60 minutes), respectively. Specimens used for sodium exposure tests were fabricated from the six plates/lots in six different processing/heat treatment conditions listed in Table 2. Note that the thickness of the AOD plates was 1.2", and thickness of the ESR and ESR-Homo plates was 1.1".

Subsize sheet-type tensile specimens (Fig. 3 [3]) were electrical-discharge-machined with the gage parallel to the rolling direction. The design of extended grip sections of a subsize tensile specimen was to ensure that the microstructural characterization and the tensile test were carried out on the sample exposed to the same sodium condition. The tensile specimen has nominal gage dimensions of $7.62 \times 1.52 \times 0.76$ mm.

Table 1. Chemical composition of melt of A709 commercial heat 58776 (wt%)

Alloy 709	C	Cr	Ni	Mn	Mo	N	Si	P	Ti	Nb	B
Specification	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
Aim	0.07	20	25	0.9	1.5	0.15	0.40	*	0.05	0.25	0.002-0.005
Actual	0.066	19.93	24.98	0.91	1.51	0.148	0.44	0.014	0.04	0.26	0.0045

*The P shall not exceed 0.025 wt.%.

Table 2. . Six plates/lots of A709 commercial heat 58776 used for sodium exposure tests

Processing/heat treatment ID	Processing condition	Solution anneal (SA) temperature (°C)	ANL Lot ID
AOD-SA1100°C	AOD	1100	58776-4-B2
AOD-SA1150°C	AOD	1150	58776-4-C2
ESR-SA1100°C	ESR	1100	58776-3RB-B2
ESR-SA1150°C	ESR	1150	58776-3RB-C2
Homo-SA1100°C	ESR-Homo	1100	58776-3RA-B2
Homo-SA1150°C	ESR-Homo	1150	58776-3RA-C2

Figure 3. Schematic drawing of subsize sheet-type tensile specimens (unit: in).[3]

A709 Lab Heats

Sodium exposure tests were also conducted on three laboratory-size heats of A709 developed under the DOE Advanced Reactor Technologies Program. Heat 709-01 (SAI Heat 12013806-709) was made by Sophisticated Alloys Inc. It was hot rolled at 1150°C and stress relieved at 1150°C for 0.5 h, followed by water quenching. Heat 709-02 (CarTech Heat 011367) and Heat 709-05 (CarTech Heat 011502) were made by Carpenter Technology. Heat 709-02 was hot forged and annealed at 1150°C for 1 h, and then water quenched, and Heat 709-05 was forged and hot rolled at 1100°C, and annealed for 2 h at 1100°C, and then water quenched. The chemical composition and heat treatment conditions of these three lab heats are given in Tables 3 and 4, respectively.

In addition to base metal specimens, weldment specimens of A709 welded with filler metals Alloy 709 or Alloy 625 were exposed to flowing sodium environment to evaluate their sodium compatibility.

Table 3. Chemical composition of A709 lab heats (wt%)

Alloy	Heat	Fe	C	Mn	P	S	Si	Ni	Cr	Mo	B	Ti	Nb	V	N
709-01	12013806	Bal	0.05	1.0	<0.01	<0.01	0.39	24.5	20.9	1.5	0.003	0.07	0.23	0.01	0.19
709-02	011367	Bal	0.078	0.90	<0.005	<0.001	0.30	25.03	20.04	1.51	0.003	<0.01	0.26	-	0.17
709-05	011502	Bal	0.063	0.88	<0.005	<0.001	0.28	25.00	19.69	1.46	0.0022	<0.01	0.23	-	0.14

Table 4. Heat treatment conditions of A709 lab heats

Alloy	Heat No.	Heat treatment
709-01	12013806	Hot rolled at 1150°C and stress relieved at 1150°C for 0.5 h, water quenched
709-02	011367	Hot forged and annealed at 1150°C for 1 h, water quenched
709-05	011502	Forged and hot rolled, and annealed at 1100°C for 2 h, water quenched

316H Stainless Steel (reference material)

316H stainless steel (SS) (Heat 8092297) was used as a reference material for comparison with an advanced alloy A709. It was an archive material provided by the Oak Ridge National Laboratory. The chemical composition of 316H SS is given in Table 5. It was in 1"-thick plate form, and solution annealed at 1065°C.

Table 5. Chemical composition of 316H SS (wt%)

Alloy	Heat	Fe	C	Mn	P	S	Si	Ni	Cr	Mo	B	Ti	Nb	V	N
316H	8092297	Bal	0.066	2.03	0.014	0.016	0.59	13.24	17.22	2.40	<0.001	-	-		0.031

2.2 Forced convection sodium loops

Two forced convection sodium materials testing loops were constructed at the Argonne National Laboratory for sodium exposure tests of advanced materials. The Sodium Materials Test Loop 1 (SMT-1) (shown in Fig. 4(a) [3]) consists of a single specimen exposure vessel, an electromagnetic pump, two electromagnetic flow meters, an economizer, and a cold trap. The total amount of sodium in the loop is approximately 10 kg. The vessel has large penetrations at the top for sample loading as well as small penetrations for a level probe, a thermocouple, and an argon/vacuum line. The maximum operating pressure and temperature of the specimen vessel are 5 psig and 700°C, respectively.

The Sodium Materials Test Loop 2 (SMT-2) (shown in Fig. 4(b) [4]) is a dual sample-vessel loop for sodium exposure tests at two temperatures. The maximum operating pressure and temperature for the two sample vessels are 5 psig and 700°C, respectively, and the total amount of sodium in the loop is approximately 20 kg. The loop is equipped with electromagnetic pumps, electromagnetic flow meters, economizers, and a cold trap. Each vessel has two penetrations at the top for sample loading as well as smaller penetrations for a level probe, thermocouple, and argon/vacuum line.

Both loops are constructed of austenitic stainless steels. The oxygen content of the sodium is controlled by the cold trap in both loops. The temperature of the cold trap is maintained at 125°C to ensure that the oxygen concentration is at ~1 wppm during specimen exposure.

(b)
Figure 4. Isometric view of (a) the Sodium Materials Test Loop 1 (SMT-1) and (b) the Sodium Materials Test Loop 2 (SMT-2).[4]

2.3 Sodium exposure tests

Specimens in six different processing/heat treatment conditions of the commercial heat 58776 were tested at 650°C in sodium for down-selection of the processing/heat treatment conditions. Specimens in AOD-SA1100°C and AOD-SA1150°C conditions were also tested at 550 and 600°C because of their overall good mechanical performance from initial evaluation. Table 6 lists the approximate accumulated exposure times at each temperature for each processing-heat treatment condition for H58776.

Table 6. Sodium exposure temperatures and total exposure times of A709 commercial heat 58776.

Processing/Heat Treatment	Total Exposure Time at 550°C (h)	Total Exposure Time at 600°C (h)	Total Exposure Time at 650°C (h)
AOD-SA1100°C	11,000	8,500	10,000
AOD-SA1150°C	11,000	8,500	10,000
ESR-SA1100°C	-	-	10,000
ESR-SA1150°C	-	-	10,000
Homo-SA1100°C	-	-	10,000
Homo-SA1150°C	-	-	10,000

Sodium exposure tests of the A709 lab heats were performed at 550, 600, and 650°C. Table 7 lists the total exposure times at each temperature for the three lab heats, its weldments, and 316H reference material.

Table 7. Sodium exposure temperatures and total exposure times of A709 lab heats.

Heat No.	Total Exposure Time at 550°C (h)	Total Exposure Time at 600°C (h)	Total Exposure Time at 650°C (h)
709-05	6,691	9,003	16,406
709-02	6,691	13,397	31,422
709-01	---	---	5,143
316H	10,917	10,757	30,873
709 weld (709 wire)	---	---	30,899
709 weld (625 wire)	---	---	27,592

Prior to the sodium exposure test, each specimen was weighed three times using an OHAUS Model AP250 analytical balance to a precision of 10 µg. The thickness of the specimen was measured three times using a micrometer to a precision of 0.0001 in. After the sodium exposure test, the specimen was removed from the loop, cleaned with alcohol with final cleaning

with water to remove sodium residues. Specimen surfaces were visually inspected and recorded by a digital camera if needed. Mass and thickness measurements were conducted to determine the mass and thickness changes resulted from sodium exposures. The post-exposure weight and thickness data were compared with the pre-exposure weight and thickness values to evaluate the corrosion performance. If only non-destructive (weight and thickness) measurements were performed on a specimen, the specimen was reloaded into the sodium loop for continued exposure.

Destructive examinations after sodium exposures include microstructural characterization and tensile testing. The extended grip sections of the exposed tensile specimen were cut from both ends before the tensile test for microstructural analysis. A metallographic specimen was prepared from the cross section for microstructural characterization by optical microscopy (OM), scanning electron microscopy (SEM), and micro-hardness measurements. Thin foil disk specimens of 3-mm in diameter were made for microstructural characterization by transmission electron microscopy (TEM). TEM specimens were electropolished to perforation using a Tenupol twin-jet polishing unit. Synchrotron X-ray diffraction (XRD) measurements were conducted on bulk specimens.

Sodium-exposed sheet-type subsize tensile specimens were tested under uniaxial tension at the Na exposure temperature in an air furnace to examine the effect of sodium exposure on tensile properties. Figure 5 shows a setup for high temperature tensile tests in air at the ANL [5]. Tensile tests were performed in an electromechanical testing system equipped with a three-zone air furnace. The applied load was recorded by a load cell. The specimen displacement was measured by the crosshead extension. The engineering tensile properties were determined from the analysis of the load and displacement data. All the tests were conducted at a nominal strain rate, 0.001 s^{-1} .

Figure 5. Experimental setup for high temperature tensile tests.[5]

2.4 Thermal aging tests

Parallel thermal aging experiments were conducted to obtain thermal aging data for comparison with sodium exposure data to separate the thermal and sodium exposure effects. When a material is exposed to sodium at temperature, thermal aging will take place simultaneously. Thus sodium exposure data contain both sodium exposure and thermal aging effects. If additional design factors are developed directly from the sodium exposure data, the thermal aging contribution will be accounted for twice as thermal aging factors for yield and ultimate strengths are already implemented in the ASME Code, leading to unnecessary conservatism. In order to understand the direct effect of sodium on the tensile properties, two companion exposure testing programs, one for sodium and the other for thermal aging, are being conducted.

Thermal aging experiments were performed in air furnaces using the same type of specimens shown in Fig. 3. Each specimen was wrapped in Ta foil and encapsulated in a quartz tube under vacuum. Encapsulated specimens were loaded in an air furnace that was heated to the target temperature for a desired period.

Thermal aging experiments were conducted at 650°C for six different processing/heat treatment conditions of the commercial heat 58776. Specimens in AOD-SA1100°C and AOD-SA1150°C conditions were also tested at 550 and 600°C. Table 8 summarizes the total accumulated thermal aging times at each temperature for each processing/heat treatment condition.

Thermal aging experiments were also conducted on the three lab heats of A709, 709-01, 709-02, 709-05 at 550, 600, and 650°C. Table 9 summarizes the total thermal aging times at each temperature for each heat. The literature data of thermally aged 316H SS of the same heat (8092297) [6] was used for comparison with the sodium exposure data of 316H generated in this project.

Microstructure of the thermally-aged specimens was characterized by optical microscopy (OM), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and energy dispersive spectroscopy (EDS). Tensile tests were conducted under uniaxial tension at the aging temperature in an air furnace at a nominal strain rate of 0.001 /s.

Table 8. Thermal aging temperatures and total exposure times of A709 commercial heat 58776.

Processing/Heat Treatment	Total Exposure Time at 550°C (h)	Total Exposure Time at 600°C (h)	Total Exposure Time at 650°C (h)
AOD-SA1100°C	7,000	7,000	7,000
AOD-SA1150°C	7,000	7,000	7,000
ESR-SA1100°C	-	-	7,000
ESR-SA1150°C	-	-	7,000
Homo-SA1100°C	-	-	7,000
Homo-SA1150°C	-	-	7,000

Table 9. Thermal aging temperatures and total exposure times of A709 lab heats.

Heat No.	Total Exposure Time at 550°C (h)	Total Exposure Time at 600°C (h)	Total Exposure Time at 650°C (h)
709-05	9,186	9,573	21,490
709-02	21,931	15,700	27,350
709-01	28,319	-	40,071

3 Results

3.1 Corrosion behavior of A709

The corrosion behavior of A709 in sodium was studied over the temperature range of 550-650°C at the oxygen level of ~1 wppm controlled by the cold trap. The corrosion data for the 550 and 600°C tests for the A709 lab heats can be found in [7]. The corrosion data for the 650°C tests for the A709 lab heats and commercial heat 58776 are shown in Fig. 6, where the corrosion rate is plotted as a function of exposure time. The corrosion rate, CR in μm per year ($\mu\text{m}/\text{y}$) was calculated from the weight change, w using Eq. (1):

$$CR = \frac{w}{\rho A t} \quad (1)$$

where ρ is the density ($7.8 \text{ g}/\text{cm}^3$), A is the specimen surface area, and t is the exposure time. The weight data represent an average of three measurements for a specimen before and after sodium exposure. A709 specimens showed weight loss at 650°C after sodium exposures except a few outliers. The corrosion rate decreased with increasing exposure time, and leveled out at the exposure time of ~30,000 h. The corrosion rate showed large variations in the initial stage, likely due to selective corrosion resulted from dissolution of alloying elements. In the isothermal sodium condition, the corrosion slows down when the dissolved elements gradually reach saturation at the exposure temperature. No significant difference in corrosion rate was observed among A709 lab heats, commercial heat, and 316H SS. The corrosion rates of A709 and 316H were $< 1 \mu\text{m}/\text{y}$ at 650°C after the initial transient stage. Overall, A709 showed good sodium compatibility at 650°C when the oxygen content in sodium is controlled.

Figure 6. Corrosion rate as a function of Na exposure time at 650°C for (a) A709 lab heat base metal and weldment and 316H and (b) commercial heat 58776 in six different processing/heat treatment conditions.

3.2 Effect of sodium exposure on tensile properties of A709

Table 10 summarizes the tensile properties, i.e. the yield stress (YS), ultimate tensile strength (UTS), uniform elongation (UE), and total elongation for A709 commercial heat 58776 in six different processing/heat treatment conditions before exposure, after thermal aging at 650°C for 3,144 h, and after sodium exposure at 650°C for 3,336 h. All the tensile tests were conducted at the exposure temperature, 650°C in air. Comparison of the tensile properties of the three different processing conditions, AOD, ESR, ESR-Homo in the 1100°C-solution-annealed condition is given in Fig. 7. The AOD specimens showed an increase in YS after aging, and a further increase after sodium exposure, while the ESR and ESR-Homo specimens showed no increase in YS after thermal aging, but an increase in YS after a sodium exposure. Thermal aging reduced the UTS, and no further reduction in UTS after sodium exposure in all three processing conditions. A similar effect of the thermal aging and sodium exposure was found on the uniform and total elongations. The total elongation, however, was only slightly reduced after thermal aging or sodium exposures compared to a more significant reduction in uniform elongation.

Figure 8 compares the tensile properties of the three different processing conditions, AOD, ESR, and ESR-Homo in the 1150°C-solution-annealed condition. Both the AOD and ESR-Homo specimens showed an increase in YS after aging, and a further increase in YS after sodium exposure, while the YS of the ESR-Homo specimens showed an obvious increase in YS only after sodium exposure. Thermal aging or sodium exposure equally reduced the UTS of the AOD and ESR specimens, but had nearly no effect on UTS in the ESR-Homo specimens. Similar to the 1100°C-solution-annealed specimens, there was a significant reduction in uniform elongation after aging or sodium exposure for all three processing conditions, and the reduction in total elongation was smaller.

In general, the solution annealing (SA) temperature, 1100 or 1150°C has a minimal effect on the tensile properties of the AOD specimens. The effect of the SA temperature is stronger on the ESR specimens, and the strongest on the ESR-homo specimens. The YS and UTS are generally lower in the 1150°C-SA specimens than in the 1100°C specimens.

Figure 9 compares the tensile properties of A709 commercial heat 58776, lab heats and 316H reference material. The tensile properties of H58776 showed larger variations than the lab heats due to different processing and heat treatment conditions. A unique finding is the lower uniform elongation of H58776 compared to the lab heats of A709. The low uniform elongation of H58776 is clearly revealed in Fig. 10, where the representative stress-strain curves of H58776 (ESR-SA1100°C) and lab heats (709-05) are compared. It was found that the stress-strain curves of the lab heat 709-05 showed more pronounced serrations of the plastic flow curve (due to the dynamic strain aging effect) than that of H58776. More importantly, the A709 lab heat retained the serrations in the stress-strain curve after aging or sodium exposure, while the A709 commercial heat H58776 showed no serrations in the stress-strain curves after thermal or sodium exposures. It is apparent that either thermal aging or sodium exposures at 650°C for ~3,000 h completely removed the dynamic strain aging effect in the commercial heat, but not in the lab heat. Dynamic strain aging is an interaction between free interstitials (e.g. carbon, nitrogen) and dislocations. Because of the high mobility of interstitial atoms, they segregate to the dislocations, and cause repeated locking and unlocking of dislocations during plastic flow, resulting in the serrated flow. The elimination of serrations of the flow curves of H58776 after aging or sodium exposure imply that carbon or nitrogen in solution available for strain aging was removed from the solution and

form precipitates during aging or sodium exposures, while some interstitials remained in the solution in the lab heats under similar thermal aging and sodium exposure conditions.

Table 10. Tensile properties at 650°C of A709 H58776 after sodium or thermal exposures at 650°C.

Processing/Heat Treatment	Exposure Cond.	Exposure T (°C)	Exposure time (h)	Test T (°C)	YS (MPa)	UTS (MPa)	UE (%)	TE (%)
AOD-SA1100C	AR	-	-	650	191	501	39.2	49.2
AOD-SA1100C	Aged	650	3144	650	226	435	20.1	39.0
AOD-SA1100C	Na	650	3336	650	243	458	20.8	39.9
AOD-SA1150C	AR	-	-	650	186	501	41.2	50.1
AOD-SA1150C	Aged	-	3144	650	219	444	20.4	37.5
AOD-SA1150C	Na	650	3336	650	240	448	20.4	39.6
ESR-SA1100C	AR	-	-	650	232	509	32.2	47.0
ESR-SA1100C	Aged	650	3144	650	218	446	21.2	39.7
ESR-SA1100C	Na	650	3336	650	258	459	18.3	41.9
ESR-SA1150C	AR	-	-	650	200	522	38.3	49.7
ESR-SA1150C	Aged	650	3144	650	206	460	22.5	39.7
ESR-SA1150C	Na	650	3336	650	245	455	21.5	35.0
Homo-SA1100C	AR	-	-	650	205	506	35.9	48.8
Homo-SA1100C	Aged	650	3144	650	199	442	23.2	40.5
Homo-SA1100C	Na	650	3336	650	259	455	19.3	36.7
Homo-SA1150C	AR	-	-	650	141	434	47.8	52.7
Homo-SA1150C	Aged	650	3144	650	191	449	26.2	42.8
Homo-SA1150C	Na	650	3336	650	212	420	24.5	31.6

Note: AR – as-received

Figure 7. Comparison of yield stress, ultimate tensile strength, uniform elongation and total elongation for 1100°C-solution-annealed A709 H58776. A709 was thermally aged or sodium exposed at 650°C and tensile tested at 650°C.

Figure 8. Comparison of yield stress, ultimate tensile strength, uniform elongation and total elongation for 1150°C-solution-annealed A709 H58776. A709 was thermally aged or sodium exposed at 650°C and tensile tested at 650°C.

Figure 9. Yield stress, ultimate tensile strength, uniform elongation and total elongation as a function of Na/thermal exposure time. 709 and 316H A709 were thermally aged or sodium exposed at 650°C and tensile tested at 650°C.

Figure 10. Stress-strain curves for A709 commercial heat 58776 (ESR-SA1100°C) (a) as-received, (c) aged at 650°C/3,144 h, (e) Na exposed at 650°C/3,336h, and A709 lab heat 709-05 (b) as-received, (d) aged at 650°C/3,384 h, (f) Na exposed at 650°C/3,504h.

3.3 Effect of sodium exposure on microstructure of A709 H58776

Nine samples of A709 H58776 were examined by transmission electron microscopy (TEM) (see Table 11). Figures 11-13 shows the microstructure of the AOD-SA1100°C specimens in the as-received condition, after aging at 650°C/3,144 h, and after sodium exposure at 650°C/3,336 h, respectively. Three bright-field images are included in each figure illustrating the microstructure inside a grain, at a grain boundary, and at a twin boundary. The as-received specimen shows a well-annealed structure with a low dislocation density and free of precipitates. Both thermal aging and sodium exposures at 650°C introduced several new phases in A709. After thermal aging at 650°C, a low number density of precipitates was formed inside grains, and a large amount of precipitates was observed at grain and twin boundaries. Sodium exposure at 650°C produced a much high density of precipitates both inside grains and at boundaries. EDS maps of the aged (Figs. 14-15) or sodium-exposed (Figs. 16-18) AOD-SA1100°C specimens showed that the precipitates decorating dislocations in the grain matrix are rich in Nb and N, and they are Z-phase particles further verified by electron diffraction. Two other phases observed in the grain matrix are $M_{23}C_6$ carbides and Cr_3Ni_2SiX (X: C, N) particles. While most of $M_{23}C_6$ carbides are distributed randomly in the matrix, some carbides co-exist with Cr_3Ni_2SiX particles, forming a shell structure with a carbide surrounded by Cr_3Ni_2SiX . Both globular-shaped and needle-shaped carbides were observed inside grains. Precipitates formed at twin boundaries and grain boundaries are $M_{23}C_6$ carbides and Cr_3Ni_2SiX . Globular $M_{23}C_6$ carbides were often observed at grain boundaries, while $M_{23}C_6$ plates were observed at twin boundaries. The region adjacent to the grain boundary has a high density of $M_{23}C_6$ cuboids and is depleted of Z-phase precipitates in the sodium-exposed specimen (Fig. 13(b)).

Figures 19-21 shows the microstructure of the ESR-SA1100°C specimens in the as-received condition, after aging at 650°C/3,144 h, or after sodium exposure at 650°C/3,336 h, respectively. Figures 22-24 shows the microstructure of the Homo-SA1100°C specimens in the as-received condition, after aging at 650°C/3,144 h, or after sodium exposure at 650°C/3,336 h, respectively. The precipitation behavior in the ESR-SA1100°C and Homo-SA1100°C specimens is qualitatively similar to that observed in the AOD-SA1100°C after aging or sodium exposure at 650°C. The effect of sodium exposure on precipitation is much stronger in forming Z-phase in the grain matrix. Furthermore, the $M_{23}C_6$ carbide segregation zone near the GB observed in the Na-exposed AOD-SA1100°C specimen was not found in the Na-exposed ESR-SA1100°C and Homo-SA1100°C specimens.

Table 11. A709 H58776 samples examined by TEM.

	As-received	Aged 650°C/3,144 h	Na-exposed 650°C/3,336 h
AOD-SA1100°C	X	X	X
ESR-SA1100°C	X	X	X
Homo-SA1100°C	X	X	X

Figure 11. TEM images showing microstructure of AOD-SA1100°C of H58776 in the as-received condition (a) inside grains, (b) at grain boundary, and (c) at twin boundary.

Figure 12. TEM images showing microstructure of AOD-SA1100°C of H58776 after aging for 3,144 h at 650°C (a) inside grains, (b) at grain boundary, and (c) at twin boundary.

Figure 13. TEM images showing microstructure of AOD-SA1100°C of H58776 after sodium exposure for 3,336 h at 650°C (a) inside grains, (b) at grain boundary, and (c) at twin boundary.

Figure 14. EDS elemental maps showing chemistry of precipitates inside grains in AOD-SA1100°C of H58776 after aging for 3,144 h at 650°C

Figure 15. EDS elemental maps showing chemistry of precipitates at twin boundary in AOD-SA1100°C of H58776 after aging for 3,144 h at 650°C.

Figure 16. EDS elemental maps showing chemistry of precipitates inside grains in AOD-SA1100°C of H58776 after sodium exposure for 3,336 h at 650°C.

Figure 17. EDS elemental maps showing chemistry of precipitates at twin boundary in AOD-SA1100°C of H58776 after sodium exposure for 3,336 h at 650°C.

Figure 18. EDS elemental maps showing chemistry of precipitates at grain boundary in AOD-SA1100°C of H58776 after sodium exposure for 3,336 h at 650°C.

Figure 19. TEM images showing microstructure of ESR-SA1100°C of H58776 in the as-received condition (a) inside grains, (b) at grain boundary, and (c) at twin boundary.

Figure 20 TEM images showing microstructure of ESR-SA1100°C of H58776 after aging for 3,144 h at 650°C (a) inside grains, (b) at grain boundary, and (c) at twin boundary.

Figure 21 TEM images showing microstructure of ESR-SA1100°C of H58776 after sodium exposure for 3,336 h at 650°C (a) inside grains, (b) at grain boundary, and (c) at twin boundary.

Figure 22. TEM images showing microstructure of Homo-SA1100°C of H58776 in the as-received condition (a) inside grains, (b) at grain boundary, and (c) at twin boundary.

Figure 23. TEM images showing microstructure of Homo-SA1100°C of H58776 after aging for 3,144 h at 650°C (a) inside grains, (b) at grain boundary, and (c) at twin boundary.

Figure 24. TEM images showing microstructure of Homo-SA1100°C of H58776 after sodium exposure for 3,336 h at 650°C (a) inside grains, (b) at grain boundary, and (c) at twin boundary.

4 Summary

The focus of the FY19 program was (a) to complete the sodium exposure of sub-sized specimens from A 709 lab heats and 316H (a reference material) and (b) to continue sodium exposure of sub-sized specimens from A 709 commercial heat 58776.

Sodium exposure tests were conducted on H58776 of A709 involving six different processing and heat treatment conditions. The melt procedure of H58776 included three processing conditions, namely, Argon-Oxygen-Decarburization (AOD), Electro-slag Remelting (ESR), and Electro-slag Remelting with subsequent homogenization (ESR-Homo). The A709 of AOD, ESR, and ESR-Homo was solution annealed (SA) at either 1100°C or 1150°C, respectively. Specimens in six different processing/heat treatment conditions, namely, AOD-SA1100°C, AOD-SA1150°C, ESR-SA1100°C, ESR-SA1150°C, Homo-SA1100°C, and Homo-SA1150°C were tested at 650°C in sodium for down-selection of the processing/heat treatment conditions. A 3,000-h sodium exposure test was completed, and a 10,000-h exposure test is in progress. Specimens in the AOD-SA1100°C and AOD-SA1150°C conditions were also tested at 550 and 600°C because of their overall good mechanical performance from initial evaluation. Sodium exposure tests at 550, 600, and 650°C of three lab heats, 709-01, 709-02, and 709-05 were completed. 316H SS was tested as a reference material for comparison with A709. Parallel thermal aging experiments were carried out to obtain thermal aging data for comparison with sodium exposure data to separate the thermal and sodium exposure effects. The major findings are:

- A709 commercial heat and lab heats showed good sodium compatibility showing a corrosion rate of $< 1 \mu\text{m/y}$ at 650°C after the initial transient stage in the oxygen-controlled sodium environment.
- Sodium exposure at 650°C for ~3,000 h increased the yield stress and decreased the ultimate tensile strength, uniform elongation and total elongation for all six processing/heat treatment conditions of A709 H58776.
- Thermal aging at 650°C for ~3,000 h increased the yield stress for the AOD-SA1100°C, AOD-SA1150°C, Homo-SA1150°C specimens, but had a minimal effect on the yield stress for the ESR-SA1100°C, ESR-SA1150°C, and Homo-SA1100°C specimens. It decreased the ultimate tensile strength for all six processing/heat treatment conditions, equivalent to the 650°C sodium exposure effect, and reduced the uniform elongation and the total elongation, either equivalent to or less than the 650°C sodium effect.
- Compared to A709 lab heats, A709 H58776 showed comparable yield stress, ultimate tensile strength, and total elongation, but had lower uniform elongation after thermal aging or sodium exposure for ~3,000 h at 650°C. The distinct difference of the tensile behavior between the commercial heat and lab heats was the complete removal of the dynamic aging effect (DSA) after thermal/sodium exposures in the commercial heat while the DSA effect still existed in the lab heats.
- Thermal aging or sodium exposure at 650°C for ~3,000 h introduced several new phases in A709, mainly M_{23}C_6 carbides, $\text{Cr}_3\text{Ni}_2\text{SiX}$ (X: C, N) particles, and Z-phase precipitates. The matrix precipitates were dominated by Z-phase particles decorating dislocation lines. The grain and twin boundaries were covered by M_{23}C_6 carbides, $\text{Cr}_3\text{Ni}_2\text{SiX}$ (X: C, N) particles. Some M_{23}C_6 carbides, $\text{Cr}_3\text{Ni}_2\text{SiX}$ (X: C, N) particles were also found in the matrix, either being isolated or co-existing forming a shell structure with a carbide surrounded by $\text{Cr}_3\text{Ni}_2\text{SiX}$.
- Thermal aging 650°C for ~3,000 h produced M_{23}C_6 carbides, $\text{Cr}_3\text{Ni}_2\text{SiX}$ (X: C, N) particles predominantly at grain or twin boundaries and a low density of matrix precipitates of Z-phase

decorating dislocations. Sodium exposure 650°C for ~3,000 h had a similar effect on grain or twin boundary precipitation, but produced a much higher density of matrix precipitates of Z-phase and dislocations. Sodium exposure also generated a $M_{23}C_6$ carbides segregation zone adjacent to the grain boundary observed only the AOD-SA1100°C specimen but not in the ESR-SA1100°C and Homo-SA1150°C specimens.

This initial assessment has indicated that the sodium exposure effects on microstructure and tensile properties of A709 commercial heat is dependent on the processing/heat treatment condition. A better understanding will be developed by examining the 650°C/10,000 h specimens when they become available.

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Nuclear Science and Engineering Division

Argonne National Laboratory

9700 South Cass Avenue, Bldg. 208

Argonne, IL 60439-4842

www.anl.gov

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