Report on the Initial Loading of SS3 Specimens from the First Commercial Heat of Alloy 709 in Sodium Materials Test Loops for Long-term Exposure

Applied Materials Division

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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 report is a Level 2 deliverable in FY18 (M2NT-18AN050503011), under the Work Package NT-18AN05050301, “A709 Sodium Compatibility” performed by Argonne National Laboratory (ANL), as part of the Advanced Materials Development Program for Fast Reactors.

The focus of the FY18 activities was to continue the sodium exposure tests of the subsized tensile specimens from small lab heats of A709 and 316H (as a reference material) and to initiate the sodium exposure tests of subsized tensile specimens made from the first A709 commercial heat.

Sodium exposure tests at 550-650°C were initiated on the first commercial heat (heat 58776) of A709. Long-term sodium exposure tests are being conducted at 550°C for times up to 100,000 h on subsized tensile specimens made from two plates/lots, 58776-4-B2 (AOD solution annealed (SA) at 1100°C) and 58776-4-C2 (AOD SA at 1150°C). Subsize tensile specimens of six plates/lots, 58776-4-B2 (AOD SA at 1100°C), 58776-4-C2 (AOD SA at 1150°C), 58776-3RB-B2 (ESR SA at 1100°C), 58776-3RB-C2 (ESR SA at 1150°C), 58776-3RA-B2 (ESR+Homo SA at 1100°C), and 58776-3RA-C2 (ESR+Homo SA at 1150°C), are being tested in the SMT-1 at 650°C. Specimens of the first commercial heat are also being prepared for the 600°C sodium exposure tests.

Sodium exposure tests were also conducted on three laboratory-size heats of Alloy 709 as well as 316H (as a reference material). 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 for evaluating their compatibility. Sodium exposure tests were performed at 550, 600, and 650°C for times up to ~31,000 h. Parallel thermal aging experiments on the same lab heats were conducted to obtain thermal aging data for comparison with sodium exposure data to understand the direct effect of sodium. The major findings are:

- A709 specimens showed weight loss at all three exposure temperatures except a few outliers. The corresponding corrosion rate decreased with increasing exposure time, and no plateau was observed at the exposure time of ~30,000 h. The corrosion rates of A709 and 316H were < 1 $\mu\text{m/y}$ after 10,000 h exposure at 650°C, and their corrosion rates were comparable under the examined conditions. Overall, both 316H and A709 showed good sodium compatibility at 550-650°C up to ~30,000 h.
- The high-temperature tensile data indicate that sodium exposures caused virtually no additional effect on the tensile properties of A709 relative to thermal aging at 550-650°C. With increasing exposure time, the yield stress increased and the ultimate tensile strength decreased, and both the uniform and total elongations decreased under both thermal and sodium exposure conditions. The uniform and total elongations of A709 remained >20% after thermal/sodium exposures.
- In contrast, sodium exposures showed strong effects on tensile properties of 316H. Both the yield stress and the ultimate tensile strength of 316H increased after sodium exposures while the yield stress and ultimate tensile strength of 316H were relatively unchanged after thermal

aging for 10,000 h at 650°C. The 550- and 600°C-sodium data were too short term to draw conclusions.

- Microstructural characterization was conducted on A709 specimens exposed to sodium or aged at 650°C and the 650°C-Na-exposed 316H specimens. Several new phases were identified in the 650°C-Na-exposed 316H. $M_{23}C_6$ was the predominant precipitate phase with other two phases, Laves phase and Cr_3Ni_2SiX (X: C, N). These precipitates were distributed either at grain boundaries or within grains.
- Both thermal aging and sodium exposures at 650°C introduced several new phases in A709 including $M_{23}C_6$, Z-phase, Cr_3Ni_2SiN . These precipitates were distributed at grain and twin boundaries and in the matrix in the aged and sodium-exposed specimens. A high density of $M_{23}C_6$ carbides was observed at grain boundaries (GB), incoherent twin boundaries (ITB), coherent twin boundaries (CTB), and within grains. Z-phase precipitated intragranularly at dislocations and was often associated with $M_{23}C_6$ carbides. Cr_3Ni_2SiN often co-existed with $M_{23}C_6$ at interfaces, e.g. grain and twin boundaries, or residual MX particle interfaces.
- Significant precipitation, observed in 316H and A709, is consistent with the reduced serrations of the plastic flow curves due to the dynamic strain aging effect.

Future work will continue long-term sodium exposure tests of commercial and lab heats of A709, and to understand the carbon transfer kinetics, the phase transformation and stability in sodium environments, and their effect on tensile properties of A709.

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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.

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, 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. 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. It is necessary to evaluate and understand the influence of carbon transport in sodium on the microstructure and mechanical properties of advanced structural materials.

On the other hand, 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 [1]). In addition, the role of ingress of carbonaceous materials in altering the carbon concentration in sodium and its effect on materials performance have also been assessed.

It is necessary 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 ΔT in the sodium circuits. This report presents data on the corrosion performance, microstructural changes, and tensile properties of advanced Alloy 709 after exposures to sodium at temperatures in the range of 550-650°C, as well as the corrosion data of Alloy 709 weldments.

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 Sodium Exposure Tests of A709 Commercial Heat

2.1 Materials and specimens

Sodium exposure tests were conducted on the first commercial heat (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.

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	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 melt procedure involved three different processing conditions, namely, Argon-Oxygen-Decarburization (AOD), Electroslag Remelting (ESR), and Electroslag Remelting with subsequent homogenization (ESR-Homo). The AOD, ESR, and ESR-Homo plates were solution annealed at 1050°C (75 minutes), 1100°C (75 minutes), and 1150°C (60 minutes), respectively. Subsize sheet-type tensile specimens (Fig. 3) [1] were fabricated from the nine plates/lots listed in Table 2 for sodium exposure tests. Note that the thickness of the AOD plates was 1.2", and thickness of the ESR and ESR+Homo plates was 1.1".

Figure 3. Schematic drawing of subsize sheet-type tensile specimens.

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

Melt condition	Heat No	Solution anneal temperature (°C)	ANL Lot ID
AOD	58776-4	1050	58776-4-A2
AOD	58776-4	1100	58776-4-B2
AOD	58776-4	1150	58776-4-C2
ESR	58776-3R	1050	58776-3RB-A2
ESR	58776-3R	1100	58776-3RB-B2
ESR	58776-3R	1150	58776-3RB-C2
ESR+Homo	58776-3R	1050	58776-3RA-A2
ESR+Homo	58776-3R	1100	58776-3RA-B2
ESR+Homo	58776-3R	1150	58776-3RA-C2

2.2 Forced convection sodium loops

Two forced convection sodium 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) [1]) 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) [1]) 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 750°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.

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

2.3 Sodium exposure tests

Long-term sodium exposure tests are being conducted at 550°C for times up to 100,000 h. Eighteen specimens per plate/lot for two plates/lots, 58776-4-B2 and 58776-4-C2 were loaded in the SMT-2 for the 550°C sodium exposure tests. Four specimens per plate/lot for six plates/lots, 58776-4-B2, 58776-4-C2, 58776-3RB-B2, 58776-3RB-C2, 58776-3RA-B2, and 58776-3RA-C2 were loaded in the SMT-1 for the 650°C sodium exposure tests. Specimens of the first commercial heat are also being prepared for the 600°C sodium exposure tests.

2.4 Thermal aging tests

Parallel thermal aging experiments are planned to obtain thermal aging data for comparison with sodium exposure data. When a material is exposed to sodium at temperature, thermal aging will take place simultaneously. Thus sodium exposure data contain both sodium 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 programs, one for sodium and the other for thermal aging, are being conducted.

3 Sodium Compatibility of Laboratory-size Heats of A709

3.1 Materials and specimens

Sodium exposure tests were also conducted on three laboratory-size heats of Alloy 709 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.

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 for evaluating their compatibility.

316H stainless steel (Heat 8092297) was included as a reference material. It was an archive material provided by the Oak Ridge National Laboratory. It was in 1"-thick plate form, and solution annealed at 1065°C.

The chemical composition and heat treatment conditions of these alloys are given in Tables 3 and 4, respectively.

Subsize sheet-type tensile specimens with extended grip sections (Fig. 3) were used in sodium exposure tests, and post-exposure microstructural analysis and tensile tests. The design of extended grip sections 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 specimens were electrical-discharge-machined with the gage parallel to the rolling direction. The tensile specimen had nominal gage dimensions of $7.62 \times 1.52 \times 0.76$ mm.

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

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
316H	8092297	Bal	0.066	2.03	0.014	0.016	0.59	13.24	17.22	2.40	<0.001	-	-		0.031

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	8092297	Solution annealed at 1065°C

3.2 Sodium exposure tests and post- exposure examinations

Sodium exposure tests were performed at 550, 600, and 650°C for times up to ~31,000 h. Table 5 lists the total exposure times at each temperature for the three heats of A709, its weldments, and 316H.

Table 5. 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	5,346	5,564	16,406
709-02	5,346	10,058	31,422
709-01	---	---	5,143
316H	2,016	4,394	30,873
709 weldment (709 wire)	---	---	28,403
709 weldment (625 wire)	---	---	25,096

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 tests. The extended grip sections of the exposed tensile specimen were cut from both ends before the tensile test for microstructural analysis. Metallographic specimens were made 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). 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. 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.

3.3 Thermal exposure tests and post-exposure examinations

Thermal aging experiments were conducted at 550, 600, and 650°C on the three heats of A709, 709-01, 709-02, 709-05. Table 6 summarizes the total thermal aging times at each temperature for each heat. Thermal aging data of A709 were compared with the sodium exposure data to separate the thermal and sodium exposure effects. The literature data of thermally aged 316H SS of the same heat (8092297) [3] was used for comparison with the sodium exposure data of 316H generated in this project.

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. Microstructure of the thermally-aged specimens was characterized by optical microscopy (OM), high-resolution 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 air at a nominal strain rate of 0.001 s^{-1} .

Table 6. Thermal 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	9,186	9,573	21,490
709-02	21,931	15,700	27,350
709-01	---	---	40,071

3.4 Corrosion behavior

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. After sodium exposures, specimens lost surface finish, but the surface remained smooth, and no spallation or surface deterioration was visually observable. SEM of cross-sectional specimens showed little changes across the specimen thickness. Figure 6 shows the weight changes and corresponding corrosion rates as a function of exposure time. The weight data represent an average of three measurements for a specimen before and after sodium exposure. The corrosion rate, CR in μm per year ($\mu\text{m}/\text{y}$) was calculated from the weight loss, 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.

A709 specimens showed weight loss at all three exposure temperatures except a few outliers. The corresponding corrosion rate decreased with increasing exposure time, and no plateau was observed at the exposure time of ~30,000 h. The corrosion rates of A709 and 316H were $< 1 \mu\text{m}/\text{y}$ after 10,000 h exposure at 650°C, and were comparable under the examined conditions. The three heats of A709 showed similar corrosion performance. Overall, both 316H and A709 showed good sodium compatibility at 550-650°C up to ~30,000 h.

Figure 6. Weight change and corrosion rate as a function of Na exposure time for A709 base metal and weldment and 316H at (a) 550, (b) 600 and (c) 650°C.

3.5 Effect of sodium exposure on tensile properties

The tensile properties for A709 and 316H after sodium or thermal exposures at 550, 600 and 650°C are summarized in Table 7. The yield stress and ultimate tensile strength are plotted as a function of sodium exposure time in Fig. 7, and the uniform and total elongations vs. sodium exposure time are plotted in Fig. 8. All the tensile tests were conducted at the exposure temperature in air. To separate the effects of thermal and sodium exposures on the tensile properties, data of the thermally aged A709 specimens of the same heats are also included in Figs. 7-8 for comparison with the tensile data of the sodium-exposed specimens. The thermal aging data of 316H were taken from the reference [3], including both mill-annealed (as-received) and reannealed (laboratory annealed) data of the same heat (8092297) of 316H. The maximum aging time of the literature data was 10,000 h. The tensile properties of 316H at the aging temperature was obtained at a strain rate of $7 \times 10^{-5} \text{ s}^{-1}$, nearly one order of magnitude lower than the strain rate used in this project. While it is anticipated that the yield and ultimate tensile strength of 316H at a slower strain state are lower than those tested at a higher strain rate, the effect of strain rate on tensile properties is not studied in this project.

The high-temperature tensile data indicate that sodium exposures caused virtually no additional effect on the tensile properties of A709 relative to thermal aging at 550-650°C. The tensile properties of the sodium-exposed and thermally aged A709 specimens are indistinguishable in Figs. 7 and 8 for all three heats, and the effects of heat variations on tensile properties are negligible. With increasing exposure time, the yield stress increased and the ultimate tensile strength decreased, and both the uniform and total elongations decreased under both thermal and sodium exposure conditions. The effects of thermal/sodium exposures on tensile properties were stronger at higher temperatures. The uniform and total elongations of A709 remained >20% after thermal/sodium exposures.

In contrast, sodium exposures showed strong effects on tensile properties of 316H. As shown in Figs. 7(e) and (f), both the yield stress and the ultimate tensile strength of 316H increased after sodium exposures, and reached to the levels of the yield and ultimate tensile strength of A709 after ~30,000 h at 650°C. Nevertheless, the yield stress and ultimate tensile strength of 316H were relatively unchanged after thermal aging for 10,000 h at 650°C. The effects of sodium exposures on the uniform and total elongations were greater than the thermal effects under similar exposure conditions at 650°C (Figs. 8(e) and (f)). The 550- and 600°C- sodium data were too short to draw conclusions.

Figures 9 and 10 show representative stress-strain curves for 316H and 709-02 after sodium or thermal exposures at 650°C. An expanded view of the stress-strain curve was also given to show the effects of sodium/thermal exposures on the serrations of the plastic flow curves. The stress-strain curves of 316H and A709 before exposures showed pronounced serrations between yielding and necking due to the dynamic strain aging effects. Both thermal aging and sodium exposures significantly reduced the effects of dynamic strain aging in A709. While no stress-strain curves of thermally aged 316H were available to draw a conclusion, the serrations of the stress-strain curves of the sodium-exposed 316H specimens were much less pronounced compared to the as-received 316H specimen. 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 reduced serrations of the flow curves of thermally aged or sodium-exposed A709 and 316H imply that the amount of carbon in solution available for strain aging was reduced from carbide precipitation during thermal/sodium exposures.

Table 7. Tensile properties of sodium-exposed or thermally-aged A709 and 316H tensile tested at 550-650°C.

Heat/Alloy	Cond.	Exp T (°C)	Exp time (h)	Test T (°C)	YS (MPa)	UTS (MPa)	UE (%)	TE (%)
316H	AR	-	0	550	122	444	45	53
316H	AR	-	0	550	123	432	48	53
316H	Na	550	2016	550	130	431	45	50
316H	AR	-	0	600	118	413	48	55
316H	AR	-	0	600	121	400	40	44
316H	Na	600	4394	600	139	375	34	40
316H	Na	600	4394	600	158	387	29	36
316H	AR	-	0	650	127	345	42	54
316H	AR	-	0	650	105	343	45	54
316H	AR	-	0	650	117	336	45	54
316H	Na	650	504	650	99	314	36	47
316H	Na	650	1008	650	109	325	32	47
316H	Na	650	2037	650	114	341	30	41
316H	Na	650	5143	650	125	357	33	40
316H	Na	650	7154	650	117	354	29	39
316H	Na	650	7154	650	137	367	29	43
316H	Na	650	8953	650	140	358	25	31
316H	Na	650	9982	650	165	395	25	32
316H	Na	650	18480	650	173	401	25	36
316H	Na	650	18480	650	157	398	26	34
316H	Na	650	24987	650	160	405	26	30
316H	Na	650	30873	650	181	430	26	35
316H	Na	650	30873	650	211	428	23	30
709-01	AR	-	0	650	177	481	46	50
709-01	AR	-	0	650	173	502	46	54

709-01	Aged	650	491	650	205	463	35	41
709-01	Aged	650	491	650	201	440	34	41
709-01	Aged	650	1000	650	221	473	30	36
709-01	Aged	650	1000	650	210	478	32	38
709-01	Aged	650	5021	650	226	470	27	37
709-01	Aged	650	10197	650	242	457	23	37
709-01	Aged	650	13391	650	216	457	23	33
709-01	Aged	650	16931	650	209	452	21	34
709-01	Aged	650	17727	650	227	445	21	36
709-01	Aged	650	40071	650	243	450	20	30
709-01	Na	650	549	650	213	462	33	38
709-01	Na	650	1008	650	222	463	30	41
709-01	Na	650	5143	650	228	471	25	34
709-02	AR	-	0	550	186	558	52	58
709-02	AR	-	0	550	173	567	50	58
709-02	Aged	550	1631	550	173	566	50	54
709-02	Aged	550	1631	550	168	568	52	57
709-02	Aged	550	4590	550	183	567	50	55
709-02	Aged	550	8118	550	189	554	51	55
709-02	Aged	550	21931	550	180	530	40	42
709-02	Aged	550	21931	550	201	548	38	42
709-02	Na	550	3330	550	193	548	47	56
709-02	Na	550	5346	550	187	549	52	57
709-02	AR	-	0	600	169	521	46	51
709-02	AR	-	0	600	175	533	49	56
709-02	Aged	600	2018	600	178	515	43	47
709-02	Aged	600	2018	600	184	530	43	46
709-02	Aged	600	15700	600	199	496	34	38
709-02	Aged	600	15700	600	204	506	32	36
709-02	Na	600	4394	600	183	491	39	42
709-02	Na	600	4394	600	194	509	42	46
709-02	Na	600	10058	600	224	492	28	32
709-02	AR	-	0	650	170	509	45	52
709-02	AR	-	0	650	173	513	51	57
709-02	AR	-	0	650	164	502	48	54

709-02	Aged	650	500	650	171	470	40	47
709-02	Aged	650	500	650	186	483	41	49
709-02	Aged	650	1003	650	189	456	38	45
709-02	Aged	650	1003	650	198	459	32	38
709-02	Aged	650	3171	650	217	483	30	36
709-02	Aged	650	3171	650	196	465	28	36
709-02	Aged	650	4971	650	203	477	26	33
709-02	Aged	650	6331	650	238	470	25	37
709-02	Aged	650	6331	650	211	461	25	37
709-02	Aged	650	10537	650	211	453	25	35
709-02	Aged	650	10537	650	235	453	23	32
709-02	Aged	650	20105	650	198	454	24	34
709-02	Aged	650	20105	650	220	468	26	36
709-02	Aged	650	27350	650	232	465	22	34
709-02	Na	650	2540	650	213	472	29	39
709-02	Na	650	2540	650	208	466	28	36
709-02	Na	650	4340	650	217	459	26	34
709-02	Na	650	4340	650	204	445	27	35
709-02	Na	650	8030	650	212	461	25	32
709-02	Na	650	9830	650	214	452	25	34
709-02	Na	650	9830	650	223	448	23	27
709-02	Na	650	18520	650	233	474	23	29
709-02	Na	650	18520	650	233	488	24	31
709-02	Na	650	26075	650	232	479	22	28
709-02	Na	650	26075	650	235	473	19	22
709-02	Na	650	31422	650	232	477	22	33
709-02	Na	650	31422	650	242	488	21	29
709-05	AR	-	0	550	163	542	53	58
709-05	AR	-	0	550	176	533	50	54
709-05	Aged	550	9186	550	199	521	40	43
709-05	Na	550	3330	550	184	531	47	56
709-05	Na	550	5346	550	177	516	45	50
709-05	AR	-	0	600	170	506	52	56
709-05	AR	-	0	600	161	503	51	55
709-05	Aged	600	2018	600	191	497	42	47

709-05	Aged	600	2018	600	190	505	48	53
709-05	Aged	600	9573	600	206	506	35	40
709-05	Na	600	2304	600	169	476	46	51
709-05	Na	600	5564	600	171	472	42	47
709-05	AR	-	0	650	177	491	40	49
709-05	AR	-	0	650	178	482	44	51
709-05	Aged	650	1345	650	194	457	34	43
709-05	Aged	650	3384	650	187	455	30	39
709-05	Aged	650	9761	650	206	452	26	38
709-05	Aged	650	21490	650	206	436	24	35
709-05	Na	650	1820	650	181	431	32	37
709-05	Na	650	3504	650	212	455	28	34
709-05	Na	650	8684	650	228	468	27	30
709-05	Na	650	8684	650	217	461	27	33
709-05	Na	650	11059	650	222	466	28	34
709-05	Na	650	16406	650	228	464	22	29

Note: AR – as-received

Figure 7. Yield stress and ultimate tensile strength as a function of Na/thermal exposure time for A709 and 316H tensile tested at the exposure temperature of (a) and (b) 550°C, (c) and (d) 600°C, and (e) and (f) 650°C.

Figure 8. Uniform elongation and total elongation as a function of Na/thermal exposure time for A709 and 316H tensile tested at the exposure time of (a) and (b) 550°C, (c) and (d) 600°C, and (e) and (f) 650°C.

Figure 9. Stress-strain curves of 316H exposed to sodium at 650°C for 0 h, 9,982 h, and 24,987 h and tensile tested at 650°C (left: full curves; right: expanded view showing serrations of the plastic flow curves).

Figure 10. Stress-strain curves of 709-02 (a) exposed to sodium at 650°C for 0 h, 9,830 h, and 26,075 h (b) aged at 650°C for 0 h, 10,537 h, and 27,350 h. All tensile tests were conducted at 650°C (left: full curves; right: expanded view showing serrations of the plastic flow curves).

3.6 Effect of sodium exposure on microstructure

Microstructural characterization was conducted on A709 specimens exposed to sodium or aged at 650°C and the 650°C-Na-exposed 316H specimens. Prior to exposures, both 316H and A709 had a well-annealed microstructure with a low density of dislocations. The grain structure of the as-received 709-02 is shown in Fig. 11. Residual MX particles were observed at the grain boundaries and within grains in the as-received 709-02. MX particles showed a bimodal size distribution with micro-sized particles revealed by SEM (Fig. 11(b)), and nanometer-sized MX particles revealed by TEM shown in Fig. 12. The composition of the MX particles was verified by EDS.

Fig. 11. SEM images showing (a) the grain structure and (b) micron-sized MX particles in the as-received 709-02.

Fig. 12. Nanometer-sized MX particles observed in the as-received 709-02.

Effect of sodium exposure on microstructure of 316H

Several new phases were identified in the 650°C-Na-exposed 316H. $M_{23}C_6$ was the predominant precipitate phase. Other two phases observed were the Laves phase and Cr_3Ni_2SiX (X: C, N). These precipitates are located either at grain boundaries or within grains. The carbides at grain boundaries were large and densely distributed along the boundaries, forming an interlocked structure, while $M_{23}C_6$ carbides in the matrix were in cuboid shape with a broad size range. Laves phase and Cr_3Ni_2SiX often formed in association with $M_{23}C_6$ particles, as shown in Fig. 13(c). Laves phase is often in the plate form, and some Laves phase particles exhibit a layered structure. Significant precipitation observed in the 316H after sodium exposures at 650°C is consistent with the reduced serrations of the plastic flow curves shown in Fig. 9.

Figure 13. TEM images (a) and (b) showing precipitates observed at grain boundaries and within grains in the Na-exposed 316H (650°C/9,982 h), and (c) three phases identified in the Na-exposed 316H (650°C/30,873 h).

Effect of sodium exposure on microstructure of A709

Both thermal aging and sodium exposures at 650°C introduced several new phases in A709. The type, morphology and distribution of precipitates were similar in the thermally-aged and sodium-exposed A709 specimens. Figure 14 shows the XRD line profiles for 709-02 after aging for 10,537 h or sodium exposure for 8,660 h at 650°C, respectively. XRD and electron microscopy revealed several phases including $M_{23}C_6$, Z-phase, Cr_3Ni_2SiN , and MX, and these precipitates were distributed at grain and twin boundaries and in the matrix in the aged and sodium-exposed specimens. Note that Cr_3Ni_2SiN was observed in aged or Na-exposed A709, while Cr_3Ni_2SiX (X: C, N) formed in Na-exposed 316H.

Figure 14. XRD line profiles for the 709-02 specimens: top: aged (650°C/10,537 h); bottom: Na-exposed (650°C/8,660 h).

A high density of $M_{23}C_6$ carbides was observed at grain boundaries (GB), incoherent twin boundaries (ITB), coherent twin boundaries (CTB), and within grains, as shown in Fig. 15. The precipitate chemistry was verified by EDS (Fig. 16). Globular $M_{23}C_6$ carbides were often observed at grain boundaries, while $M_{23}C_6$ plates were observed at twin boundaries. As shown in Fig. 16(b), $M_{23}C_6$ grew into plates from incoherent twin boundaries, and further transformed into a different type of precipitates away from the ITBs. The phase away from the ITB was a Si rich phase, Cr_3Ni_2SiN according to the literature [4].

$M_{23}C_6$ carbides formed in the matrix often showed a density gradient with the carbide number density decreasing with increasing the distance to the boundary (Fig. 15). The $M_{23}C_6$ carbides formed in the matrix had a cuboid or globular shape with the mean size of ~170 nm. $M_{23}C_6$ precipitates were also observed intragranularly at dislocations (Fig. 17).

Z-phase precipitated intragranularly at dislocations, as shown in Fig. 17. Z-phase precipitates were often found to be associated with $M_{23}C_6$ carbides (Figs. 17 and 18). Residual MX particles in the as-received A709 were surrounded by $M_{23}C_6$ and Cr_3Ni_2SiN forming a core-shell structure after aging or sodium exposures, as shown in Fig. 19.

It is noted that $M_{23}C_6$ and Cr_3Ni_2SiN often co-existed at interfaces, e.g. grain and twin boundaries, or residual MX particle interfaces, and Z-phase was often found to be associated with $M_{23}C_6$ carbides in the matrix in the thermally-aged/Na-exposed A709. The close association of Z-phase with $M_{23}C_6$ at dislocations may explain the absence of MX that is expected to form during exposures. The formation of $M_{23}C_6$ can locally deplete carbon, favoring precipitation of Z-phase over MX precipitates.

Fig. 15. SEM image showing $M_{23}C_6$ particles in the aged 709-02 (650°C/10,537 h).

Fig. 16. EDS Cr (blue) and Si (yellow) overlapped map and element maps showing co-existed $M_{23}C_6$ carbides and Cr_3Ni_2SiN precipitates at (a) a grain boundary, and (b) an incoherent twin boundary in the Na-exposed 709-02 (650°C/8,030 h).

Fig. 17. $M_{23}C_6$ and Z-phase particles attached to dislocations observed in aged 709-02 (650°C/27,350 h).

Fig. 18. Z-phase formed on $M_{23}C_6$ particles observed in aged 709-02 (650°C/27,350 h).

Fig. 19. Residual MX particles surrounded by $M_{23}C_6$ and Cr_3Ni_2SiX precipitates observed in aged 709-02 (650°C/10,537 h).

4 Summary and Future Work

The focus of the FY18 activities was to continue the sodium exposure tests of the subsized tensile specimens from small lab heats of A709 and 316H (as a reference material) and to initiate the sodium exposure tests of subsized tensile specimens made from the first A709 commercial heat.

Sodium exposure tests at 550-650°C were initiated on the first commercial heat (heat 58776) of A709. Long-term sodium exposure tests are being conducted at 550°C for times up to 100,000 h on subsized tensile specimens made from two plates/lots, 58776-4-B2 (AOD solution annealed (SA) at 1100°C) and 58776-4-C2 (AOD SA at 1150°C). Subsize tensile specimens of six plates/lots, 58776-4-B2 (AOD SA at 1100°C), 58776-4-C2 (AOD SA at 1150°C), 58776-3RB-B2 (ESR SA at 1100°C), 58776-3RB-C2 (ESR SA at 1150°C), 58776-3RA-B2 (ESR+Homo SA at 1100°C), and 58776-3RA-C2 (ESR+Homo SA at 1150°C), are being tested in the SMT-1 at 650°C. Specimens of the first commercial heat are also being prepared for the 600°C sodium exposure tests.

Sodium exposure tests were also conducted on three laboratory-size heats of Alloy 709 as well as 316H (as a reference material). 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 for evaluating their compatibility. Sodium exposure tests were performed at 550, 600, and 650°C for times up to ~31,000 h. Parallel thermal aging experiments on the same lab heats were conducted to obtain thermal aging data for comparison with sodium exposure data to understand the direct effect of sodium. The major findings are:

- A709 specimens showed weight loss at all three exposure temperatures except a few outliers. The corresponding corrosion rate decreased with increasing exposure time, and no plateau was observed at the exposure time of ~30,000 h. The corrosion rates of A709 and 316H were < 1 $\mu\text{m}/\text{y}$ after 10,000 h exposure at 650°C, and their corrosion rates were comparable under the examined conditions. Overall, both 316H and A709 showed good sodium compatibility at 550-650°C up to ~30,000 h.
- The high-temperature tensile data indicate that sodium exposures caused virtually no additional effect on the tensile properties of A709 relative to thermal aging at 550-650°C. With increasing exposure time, the yield stress increased and the ultimate tensile strength decreased, and both the uniform and total elongations decreased under both thermal and sodium exposure conditions. The uniform and total elongations of A709 remained >20% after thermal/sodium exposures.
- In contrast, sodium exposures showed strong effects on tensile properties of 316H. Both the yield stress and the ultimate tensile strength of 316H increased after sodium exposures, and while the yield stress and ultimate tensile strength of 316H were relatively unchanged after thermal aging for 10,000 h at 650°C. The 550- and 600°C-sodium data were too short to draw conclusions.
- Microstructural characterization was conducted on A709 specimens exposed to sodium or aged at 650°C and the 650°C-Na-exposed 316H specimens. Several new phases were identified in

the 650°C-Na-exposed 316H. $M_{23}C_6$ was the predominant precipitate phase with other two phases, Laves phase and Cr_3Ni_2SiX (X: C, N). These precipitates were distributed either at grain boundaries or within grains.

- Both thermal aging and sodium exposures at 650°C introduced several new phases in A709 including $M_{23}C_6$, Z-phase, Cr_3Ni_2SiN . These precipitates were distributed at grain and twin boundaries and in the matrix in the aged and sodium-exposed specimens. A high density of $M_{23}C_6$ carbides was observed at grain boundaries (GB), incoherent twin boundaries (ITB), coherent twin boundaries (CTB), and within grains. Z-phase precipitated intragranularly at dislocations, and were often associated with $M_{23}C_6$ carbides. Cr_3Ni_2SiN often co-existed with $M_{23}C_6$ at interfaces, e.g. grain and twin boundaries, or residual MX particle interfaces.
- Significant precipitation observed in the 316H and A709 was consistent with the reduced serrations of the plastic flow curves due to the dynamic strain aging effect.
- Future work will continue long-term sodium exposure tests of commercial and lab heats of A709, and to understand the carbon transfer kinetics, the phase transformation and stability in sodium environments, and their effect on tensile properties of A709.

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