FY 2017 – Thermal Aging Effects on Advanced Structural Materials

Nuclear Engineering Division

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ABSTRACT

This report provides an update on the evaluation of the effect of thermal aging on tensile properties of existing laboratory-sized heats of Alloy 709 austenitic stainless steel and the completion of effort on the thermal aging effect on the tensile properties of optimized G92 ferritic-martensitic steel. The report is a Level 3 deliverable in FY17 (M3AT-17AN1602081), under the Work Package AT-17AN160208, “Advanced Alloy Testing - ANL” performed by the Argonne National Laboratory (ANL), as part of the Advanced Reactor Technologies Program.

Thermal aging experiments at 550, 600, and 650°C were carried out on three laboratory-sized heats of Alloy 709 austenitic stainless steel, 709-01 (SAI Heat 12013806-709), 709-02 (CarTech Heat 11367), 709-05 (CarTech Heat 011502). Tensile tests were conducted at the aging temperature in air at a nominal strain rate of 0.001 s^{-1} . It was found that thermal aging at 550°C had minimal effect on the tensile properties for times $< 10,000\text{ h}$. Thermal aging at 600°C for $\sim 15,000\text{ h}$ slightly increased the yield stress and decreased the ultimate tensile strength (UTS). The effect of thermal aging at 650°C was more pronounced on the tensile properties of Alloy 709. An increase in the yield stress and a decrease in the UTS were accompanied by the reduction in the uniform and total elongations. The yield stress was increased by $\sim 34\%$, the UTS was reduced by $\sim 9\%$, and the uniform and total elongations were reduced by $\sim 50\%$ and $\sim 36\%$, respectively after aging for $\sim 20,000\text{ h}$ at 650°C. The effect of thermal aging on tensile properties showed little variations among three heats of Alloy 709.

Four heats of optimized G92 ferritic-martensitic steel with or without thermo-mechanical treatment (TMT), including Opt. G92-3, Opt. G92-4, Opt. G92-4T, and Opt. G92-8T were investigated to evaluate the effect of thermal aging on the tensile properties of optimized G92. Conventional G92 (Heat G92-0) was included in the study to provide the baseline data. Thermal aging at 550 - 650°C caused a decrease in the yield stress and the ultimate tensile strength but had an insignificant effect on tensile ductility of optimized G92. The effect of thermal aging was stronger at higher temperatures and longer exposure times. The effect of thermal aging on tensile strength became evident after $\sim 10,000\text{ h}$ at 550°C, $\sim 1,000\text{ h}$ at 600°C, and $< 1,000\text{ h}$ at 650°C. The aging-induced reduction in tensile strength was much more pronounced at 650°C than at temperatures of 550-600°C, and exhibited large variations among different heats of optimized G92. The heat with a higher initial tensile strength (e.g. Opt. G92-3 and Opt. G92-8T) showed a much greater reduction in strength than those with lower initial tensile strength (e.g. Opt. G92-4 and Opt. G92-4T). Microstructural characterization of optimized G92 showed subgrain coarsening, coarsening of $M_{23}C_6$ and MX particles, and precipitation of Laves phase after thermal aging at 650°C. These microstructural changes can be correlated well with aging-induced tensile property degradation at 650°C.

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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. 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 nuclear reactors. Several advanced alloys were selected for further development in support of sodium-cooled fast reactors (Busby et al 2008). Advanced materials development efforts under the Advanced Reactor Concepts from FY2008 to FY2012 led to the down-selection of an advanced austenitic stainless steel, Alloy 709 and a ferritic-martensitic steel, optimized Grade 92 with thermo-mechanical treatment (TMT).

Alloy 709 (Fe-20Cr-25Ni-1.5Mo-Nb,B,N) has a significantly higher strength than conventional Type 304 and 316 austenitic stainless steels, and is considered as one of the best austenitic stainless steels for elevated temperature structural applications among commercially-available austenitic alloy classes. The high strength of Alloy 709 is achieved by controlling the carbon content to 0.07–0.10% and precipitation strengthening by MX precipitates. Alloy 709 also shows good fabrication properties and weldability.

Optimized Grade 92 (Fe-9Cr-0.5Mo-2W-V,Nb) is a variant of commercially available Grade 92 ferritic-martensitic steels with tighter control of chemistry and thermo-mechanical treatment to achieve improved high temperature performance. Optimized G92 with and without thermomechanical treatment (TMT) has been developed in the United States for its potential applications for structural components in advanced sodium-cooled fast reactors. Though the alloy shows enhanced creep properties, good cross-weld creep performance, and comparable fatigue/creep-fatigue performance to commercial G92 steels [1], its sodium compatibility and the effect of sodium exposures on microstructure and mechanical properties have not been fully addressed.

While these advanced alloys have showed enhanced performance over current generation of high temperature structural materials, one of the major concerns in their structural applications in sodium-cooled fast reactors is their long-term microstructural stability and associated degradation of mechanical properties during service. Our previous work has showed that tensile properties of these advanced materials degrade with time, when exposed to high temperatures and liquid sodium. Significant microstructural changes during prolonged thermal and sodium exposures can lead to dramatically different responses to service environments. The objective of this project is to evaluate the effect of long-term thermal aging on the tensile properties of advanced alloys, Alloy 709 and optimized G92 (Opt. G92). This report presents continued effort on evaluating the effect of long-term thermal aging on tensile properties of Alloy 709 of laboratory-sized heats and the completion of effort on understanding the thermal aging effect on tensile properties of Opt. G92.

2 Experimental Procedure

2.1 Materials and specimens

Three laboratory-sized heats of Alloy 709 were studied, i.e. 709-01, 709-02, and 709-05:

- Alloy 709-01 (SAI Heat 12013806-709) was made by Sophisticated Alloys Inc. It was hot rolled at 1150°C from ~4" thickness to ~1" with 10-15% reduction each pass, and stress relieved at 1150°C for 0.5 h, followed by water quenching.
- Alloy 709-02 (CarTech Heat 11367) was made by Carpenter Technology. Two 1"-thick plates were received in FY14. The lot numbers of the two plates are "67-7" and "67-9". They were hot forged, and annealed at 1150°C for 1 h under a reducing atmosphere, and then water quenched.
- Alloy 709-05 (CarTech Heat 011502) was also made by Carpenter Technology. It was annealed for 2 h at 1100°C, and then water quenched.

The chemical composition and heat treatment conditions of the three heats of Alloy 709 are given in Tables 1 and 2, respectively.

Four heats of optimized G92 were examined, i.e. Opt. G92-3, Opt. G92-4, Opt. G92-4T, and Opt. G92-8T. Commercial G92 (Generic Heat No. G92-0) was included to provide the baseline data and compare with Opt. G92. All the materials were provided by the Oak Ridge National Laboratory (ORNL).

- Opt. G92-3 (Heat 1105364201, Generic Heat No. G92-3) was made by vacuum-induction melting (VIM) by Sophisticated Alloys, and was in 1"-thick plate form. It was heat treated at 1177°C for 4 h, hot-forged/rolled, air cooled, followed by normalization at 1080°C for 1 h, air cooling, and tempering at 750°C for 2 h, and air cooling.
- Opt. G92-4 (Heat 011364, Generic Heat No. G92-4) was made by VIM and electroslag remelting (ESR) by Carpenter Technology. It was in 3"-thick plate form. It was normalized at 1150°C for 1 h and water quenched, followed by hot forging at 1050°C and water quench, and tempered at 750°C for 1 h and air cooled.
- Opt. G92-4T (Heat 011364, Generic Heat No. G92-4T) was made by VIM and ESR by Carpenter Technology. It was in 1"-thick plate form. The plate was normalized at 1150°C for 1 h, followed by hot forging at 1050°C and water quench, and tempered at 750°C for 1 h and air cooled.
- Opt. G92-8T was produced via VIM route and was in 1.6"-thick plate form. It was hot forged from 1130°C and held at 1130°C for 0.5h and water quenched, followed by tempering at 600°C for 1 h at 600°C and air cooled. It was re-tempered at 750°C for 4 h and air cooled.
- G92-0 was in 1"-thick plate form. It was normalized at 1050°C and tempered at 750°C for 2 h and air cooled.

The chemical composition and heat treatment conditions of Opt. G92 and commercial G92 are given in Tables 3 and 4, respectively.

Table 1. Chemical composition of Alloy 709 (in wt%)

Generic Heat No.	Heat	Fe	C	Mn	P	S	Si	Ni	Cr	Mo	B	Ti	Nb	N	Al
709-01	120113806	Bal	0.05	1.0	<0.01	<0.01	0.39	24.5	20.9	1.5	0.003	0.07	0.23	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 2. Heat treatment conditions of Alloy 709

Generic Heat No.	Heat	Heat treatment
709-01	120113806	Anneal at 1150°C for 30 min, water quenched
709-02	011367	Anneal at 1050°C for 30 min, water quenched
709-05	011502	Anneal at 1100°C for 120 min, water quenched

Table 3. Chemical composition of Opt. G92 and G92 (in wt%)

Alloy	Generic Heat No.	Heat	Fe	C	Cr	Mn	Mo	N	Nb	Ni	Si	Ti	V	W
Opt.G92	G92-3	11053642-1	Bal	0.05	9	0.5	0.5	0.05	0.1	0.1	0.2	-	0.2	1.9
Opt.G92	G92-4	011364	Bal	0.09	8.74	0.36	0.36	0.029	0.06	0.12	0.01	0.001	0.19	1.88
Opt.G92	G92-4T	011364	Bal	0.09	8.74	0.36	0.36	0.029	0.06	0.12	0.01	0.001	0.19	1.88
Opt. G92	G92-8T	011448	Bal	0.091	8.81	0.4	0.36	0.046	0.08	0.12	0.1	0.005	0.18	1.78
G92	G92-0	-	Bal	0.11	9.1	0.46	0.43	0.057	0.08	0.06	0.03	-	0.20	1.84

Table 4. Heat treatment conditions of Opt. G92 and G92

Alloy	Generic Heat No.	Heat	Heat treatment
Opt.G92	G92-3	11053642-1	1177°C/4h + hot-forging/rolling + 1080°C/1h/AC ⁽¹⁾ + 750°C/2h/AC
Opt.G92	G92-4	011364	1150°C/1h/WQ ⁽²⁾ + 750°C/1h/AC
Opt.G92	G92-4T	011364	1150°C/1h/WQ + Hot Forging at 1050°C/WQ + 750°C/1h/AC
Opt. G92	G92-8T	011448	Hot forging at 1130°C + 1130°C/0.5h/WQ + 600°C/1h/AC + 750°C/4h/AC
G92	G92-0	-	Normalized at ~1050°C and tempered at 750°C/2h/AC

⁽¹⁾AC – air cooling; ⁽²⁾WQ – water quenching

Subsize sheet-type tensile specimens with extended grip sections (Fig. 1) were used in thermal aging experiments and tensile tests. The extended grip sections were designed for microstructural characterization and tensile test to be conducted on the same sample to minimize data scatter from specimen variations. The tensile specimens were electrical-discharge-machined with the gage parallel to the rolling direction. The tensile specimen has nominal gage dimensions of $7.62 \times 1.52 \times 0.76$ mm.

Figure 1. Schematic drawing of subsize sheet-type tensile specimens used for thermal aging tests (a) the extended-grip tensile specimen used for microstructural characterization and tensile tests and (b) the specimen used for tensile tests.

2.2 Thermal aging experiments

Thermal aging experiments were performed at 550, 600, and 650°C for various exposure times to examine the effect of thermal aging on tensile properties and microstructure. Each tensile specimen was wrapped in Ta foil and encapsulated in a quartz tube under vacuum (shown in Fig. 2(a)). Encapsulated specimens were loaded in an air furnace (Fig. 2(b)) that was heated to the target temperature for an extended period.

Figure 2. Thermal aging experiment (a) vacuum-encapsulated specimens and (b) high-temperature furnaces used for thermal aging tests.

Table 5 provides the total exposure times at different temperatures for various heats of Alloy 709, Opt. G92 and G92.

Table 5. Total thermal exposure times at 550, 600, and 650°C for Alloy 709, Opt. G92 and G92.

Alloy	Heat No.	Total Exposure Time at 550°C (h)	Total Exposure Time at 600°C (h)	Total Exposure Time at 650°C (h)
Alloy 709	709-01	-	-	40,071
Alloy 709	709-02	8,118	15,700	20,105
Alloy 709	709-05	-	-	9,761
Opt. G92	G92-3	34,132	29,594	21,480
Opt. G92	G92-4	8,843	13,748	5,284
Opt. G92	G92-4T	8,144	13,748	5,284
Opt. G92	G92-8T	-	3,695	5,284
G92	G92-0	19,926	14,979	36,061

2.3 High temperature tensile tests

Tensile specimens were tested under uniaxial tension in an air furnace. Figure 3 shows a setup for high temperature tensile tests in air at the Argonne National Laboratory. 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 analysis of load versus displacement data files. As-received specimens were tested at a range of temperatures of 20-650°C. Thermally-aged specimens were tested at the thermal aging temperature. All the tests were conducted at a nominal strain rate, 0.001 s^{-1} .

Figure 3. Experimental setup for high temperature tensile tests.

3 Results

3.1 Effect of thermal aging on tensile properties of Alloy 709

Figures 4-6 show the yield stress, the ultimate tensile strength, the uniform elongation, and the total elongation as a function of aging time for Alloy 709 thermally aged at 550, 600, and 650°C, respectively. The as-received condition is plotted as “1 h- exposure” for convenience. Table 6 summarizes the tensile property data of thermally-aged Alloy 709. Note that a few data exist for thermal aging at 550 - 600°C, and for one heat only, 709-02.

Thermal aging at 550°C has minimal effect on the tensile properties for times < 10,000 h, as shown in Fig. 4. A slight increase in the yield stress and a decrease in the ultimate tensile strength (UTS) was observed after thermal aging at 600°C for ~15,000 h, shown in Fig. 5. The effect of thermal aging on the tensile properties of Alloy 709 is more pronounced at 650°C. An increase in the yield stress and a decrease in the UTS are accompanied by the reductions in uniform and total elongations, as shown in Fig. 6. The yield stress was increased by ~34%, the UTS was reduced by ~9%, and the uniform and total elongations were reduced by ~50% and ~36%, respectively after aging for ~20,000 h at 650°C. The effect of thermal aging on tensile properties showed little variations among three heats of Alloy 709.

Figure 4. The yield stress, ultimate tensile strength, uniform elongation and total elongation as a function of exposure time for Alloy 709 tensile tested at 550°C after thermal aging at 550°C.

Figure 5. The yield stress, ultimate tensile strength, uniform elongation and total elongation as a function of exposure time for Alloy 709 tensile tested at 600°C after thermal aging at 600°C.

Figure 6. The yield stress, ultimate tensile strength, uniform elongation and total elongation as a function of exposure time for Alloy 709 tensile tested at 650°C after thermal aging at 650°C.

Table 6. Tensile properties of thermally-aged Alloy 709.

Heat No.	Aging T (°C)	Aging time (h)	Test T (°C)	YS (MPa)	UTS (MPa)	UE (%)	TE (%)
709-01	-	0	650	177	481	46	50
709-01	-	0	650	173	502	46	54
709-01	650	491	650	205	463	35	41
709-01	650	491	650	201	440	34	41
709-01	650	1,000	650	221	473	30	36
709-01	650	1,000	650	210	478	32	38
709-01	650	5,021	650	226	470	27	37
709-01	650	10,197	650	242	457	23	37
709-01	650	13,391	650	216	457	23	32.5
709-01	650	16,931	650	209	452	21	34
709-01	650	17,727	650	227	445	21	36
709-01	650	40,071	650	243	450	20	30
709-02	550	0	550	186	558	52	58
709-02	550	0	550	175	519	36.8	40.5
709-02	550	4,590	550	183	567	50	55
709-02	550	8,118	550	189	554	51	55
709-02	-	0	600	169	521	46	51
709-02	-	10	600	175	533	49.2	55.8
709-02	600	15,700	600	199	496	34	38
709-02	-	0	650	170	509	45	51.5
709-02	-	0	650	173	513	51	57
709-02	-	0	650	164	502	48	54
709-02	650	500	650	171	470	40	47
709-02	650	500	650	186	483	41	49
709-02	650	1,003	650	189	456	38	45
709-02	650	1,003	650	198	459	32	38
709-02	650	3,171	650	217	483	30	36
709-02	650	3,171	650	196	465	28	35.5
709-02	650	4,971	650	203	477	26	32.5
709-02	650	6,331	650	238	470	25	36.5
709-02	650	6,331	650	211	461	25	36.5
709-02	650	10,537	650	211	453	25	35
709-02	650	10,537	650	235	453	23	32
709-02	650	20,105	650	198	454	24	34
709-02	650	20,105	650	220	468	25.6	35.7
709-05	-	0	650	177	491	40	49
709-05	-	0	650	178	482	44	51
709-05	650	1,345	650	194	457	34.1	43.1
709-05	650	3,384	650	187	455	30	39
709-05	650	9,761	650	206	452	26	38

3.2 Effect of thermal aging on tensile properties of optimized G92

The effects of thermal aging on the tensile properties of optimized G92 are illustrated in Figs. 8 – 10, where the yield stress, the ultimate tensile strength (UTS), the uniform elongation, and the total elongation are plotted as a function of aging time for optimized G92 thermally aged at 550, 600, and 650°C, respectively. The data of the thermally-aged G92 specimens are included in Figs. 8-10 as the baseline data to compare with the performance of optimized G92, as well as the data of the sodium-exposed optimized G92 and conventional G92. The tensile property data of thermally-aged optimized G92 and commercial G92 are summarized in Table 7.

Thermal aging at 550 - 650°C decreased the yield stress and the ultimate tensile strength of optimized G92. The effect of thermal aging was stronger at higher temperatures and longer exposure times. The effect of thermal aging on tensile strength became evident after ~10,000 h at 550°C, ~1,000 h at 600°C, and <1,000 h at 650°C. The aging-induced reduction in tensile strength was much more pronounced at 650°C than at temperatures of 550-600°C. The reductions in tensile strength resulted from thermal aging at 650°C varied among different heats of optimized G92. Heats of Opt. G92-3 and Opt. G92-8T have the highest UTS in the as-received condition. The UTS of these two heats was reduced by ~30% after thermal exposure at 650°C for 5,000 h. Heats of Opt. G92-4 and Opt. G92-4T have the lowest UTS prior to thermal exposures, and their UTS values were reduced by ~10% after thermal exposure at 650°C for 5,000 h. While the commercial G92 has the moderate UTS compared to various heats of optimized G92, its UTS was decreased by ~15% from thermal exposures at 650°C. Ductility of thermally-aged optimized G92 and commercial G92 remained adequate with >15% of total elongation.

Sodium exposures at 550 and 600°C caused virtually no additional effect on the tensile strength and the elongations when compared with the thermally-aged specimens of optimized and commercial G92, while sodium exposures at 650°C have a much stronger effect than thermal exposures alone under comparable temperature and time conditions for all heats of optimized G92 and commercial G92.

Figure 7. The yield stress, ultimate tensile strength, uniform elongation and total elongation as a function of exposure time for Opt. G92 and G92 tensile tested at 550°C after thermal aging at 550°C.

Figure 8. The yield stress, ultimate tensile strength, uniform elongation and total elongation as a function of exposure time for Opt. G92 and G92 tensile tested at 600°C after thermal aging at 600°C.

Figure 9. The yield stress, ultimate tensile strength, uniform elongation and total elongation as a function of exposure time for Opt. G92 and G92 tensile tested at 650°C after thermal aging at 650°C.

Table 7. Tensile properties of thermally-aged Opt. G92 and G92

Heat No.	Aging T (°C)	Aging time (h)	Test T (°C)	YS (MPa)	UTS (MPa)	UE (%)	TE (%)
Opt. G92-3	-	0	550	574	604	1.5	18.5
Opt. G92-3	550	10,353	550	539	592	2.0	17
Opt. G92-3	550	34,132	550	515	576	2.3	17.6
Opt. G92-3	-	0	600	514	537	1	18.5
Opt. G92-3	600	5,417	600	454	490	1.7	18.1
Opt. G92-3	600	10,007	600	439	485	1.8	18.4
Opt. G92-3	600	29,594	600	417	464	1.8	25.5
Opt. G92-3	-	0	650	436	451	1.1	17
Opt. G92-3	650	499	650	360	365	0.8	26
Opt. G92-3	650	1,033	650	366	373	0.7	24
Opt. G92-3	650	2,011	650	322	327	0.7	25
Opt. G92-3	650	7,891	650	274	287	0.9	29
Opt. G92-3	650	12,574	650	287	293	0.5	26
Opt. G92-3	650	21,480	650	254	267	1	26
Opt. G92-4	-	0	550	389	422	1.6	18
Opt. G92-4	550	8,843	550	418	463	2.0	14.1
Opt. G92-4	-	0	600	306	358	2	17
Opt. G92-4	600	1,152	600	323	341	1.1	23.8
Opt. G92-4	600	4,344	600	300	342	1.5	17
Opt. G92-4	600	8,110	600	294	334	1.7	16.4
Opt. G92-4	600	13,748	600	303	343	1.8	20.1
Opt. G92-4	-	1	650	267	292	1.5	23.0
Opt. G92-4	-	1	650				
Opt. G92-4	650	2,116	650	254	267	1.4	25.0
Opt. G92-4	650	5,284	650	241	263	1.3	22.5
Opt. G92-4T	-	0	550	391	431	1.5	13
Opt. G92-4T	550	8,144	550	372	425	2.1	14.8
Opt. G92-4T	-	0	600	351	365	1	19
Opt. G92-4T	-	0	600	333	367	1.5	17
Opt. G92-4T	-	0	600	316	363	1.7	15.7
Opt. G92-4T	600	1,152	600	335	356	1.2	21.1
Opt. G92-4T	600	2,009	600	338	357	1.1	21
Opt. G92-4T	600	4,562	600	345	368	1.5	23.5
Opt. G92-4T	600	4,562	600	332	360	1	20.3
Opt. G92-4T	600	6,445	600	318	351	1.4	19
Opt. G92-4T	600	13,748	600	316	355	2.0	17.9
Opt. G92-4T	-	0	650	272	303	1.6	20.2

Opt. G92-4T	650	2,116	650	243	260	1.2	18.4
Opt. G92-4T	650	5,284	650	256	280	1.5	31
Opt. G92-8T	-	0	600	449	475	1	13.3
Opt. G92-8T	600	1,152	600	447	479	1.2	17.1
Opt. G92-8T	600	3,695	600	435	484	1.7	16.0
Opt. G92-8T	-	1	650	385	428	1.3	16.3
Opt. G92-8T	650	2,116	650	321	341	1.8	21.3
Opt. G92-8T	650	5,284	650	298	318	1.1	21.7
G92-0	-	0	550	459	479	1.7	18
G92-0	550	5,000	550	451	476	1.4	20
G92-0	550	12,025	550	435	483	2.1	19.7
G92-0	550	19,926	550	448	490	1.5	20
G92-0	-	0	600	386	419	1.5	27
G92-0	-	0	600	395	413	1	26.3
G92-0	600	2,009	600	394	409	1	27.5
G92-0	600	4,589	600	335	392	2	17.8
G92-0	600	4,589	600	353	390	1.8	23.3
G92-0	600	7,557	600	382	394	1.3	20
G92-0	600	7,557	600	382	401	1	26.8
G92-0	600	14,979	600	370	404	1.7	23.2
G92-0	-	0	650	315	333	1.5	28
G92-0	650	1,348	650	279	295	1.3	26
G92-0	650	5,011	650	270	283	1.4	30
G92-0	650	10,546	650	257	268	1.5	32
G92-0	650	20,531	650	261	275	1.1	30
G92-0	650	36,061	650	250	270	1.2	30

3.3 Effect of thermal aging on microstructure

Microstructural characterization was conducted on the Heat Opt. G92-4 of optimized G92 in the as-received condition and after thermal aging for 5,284 h at 650°C. The microstructure of the as-received Opt. G92-4 has tempered martensitic structure with a high density of dislocations, $M_{23}C_6$ carbides decorated primarily along grain or subgrain boundaries, and a fine distribution of vanadium/niobium carbonitrides (MX) within subgrains. Thermal aging at 650°C caused significant microstructural changes, e.g. dislocation density and structural changes, subgrain coarsening, carbides and carbonitrides coarsening, and precipitation of a new phase, intermetallic Laves phase. Figures 10 (a) – (d) show representative TEM/SEM images of subgrains, dislocations within a subgrain, $M_{23}C_6$, MX and Laves phase precipitates in the specimen thermally aged for 5,284 h at 650°C. Quantitative measurements of dislocation densities within subgrains, subgrain widths, and size distributions of $M_{23}C_6$, MX and Laves phase precipitates were conducted on the as-received and thermally-aged specimens, and the data are compared in Fig. 10(e). Note that the microstructural data of the sodium-exposed Opt. G92-4 specimen (650°C/4,370h) was also included in Fig. 10(e).

Subgrains coarsened rapidly during thermal aging at 650°C. The mean width of subgrains in the as-received Opt. G92-4 was 277 nm, while the mean width of subgrains in the thermally-aged Opt. G92-4 was 492 nm. The subgrain size distribution became broader, and subgrains with a much larger width formed during aging. The dislocation density within subgrains showed minimal changes after aging. Coarsening of $M_{23}C_6$ carbides and MX particles was observed in the thermally-aged Opt. G92-4 specimen. The mean size of $M_{23}C_6$ precipitates in the thermally-aged Opt. G92-4 specimen was 119 nm, relative to 90 nm in the as-received specimen. The mean size of MX precipitates increased from 46 nm to 71 nm after aging for 5,284 h at 650°C. Precipitation of Laves phase occurred during thermal exposures at 650°C in Opt. G92-4. The mean size of the Laves phase particles was 270 nm. Laves phase precipitates were distributed primarily along grain/subgrain boundaries. Microstructural changes in Opt. G92-4 during sodium exposure were much more pronounced than under thermal exposure at 650°C.

Figure 10. Microstructure of Opt. G92-4 after thermal aging for 5,284 h at 650°C (a) TEM image of subgrains, (b) TEM image of dislocations, (c) TEM image of $M_{23}C_6$ (colored in blue) and MX (colored in green) precipitates, (d) SEM image of Laves phase, and (e) comparison of subgrain width, dislocation density, mean sizes of $M_{23}C_6$, MX and Laves phase particles in the as-received, thermally-aged (650°C/5,284h), and sodium-exposed (650°C/4,370h) conditions.

4 Summary

This report provides an update on the evaluation of the effect of thermal aging on tensile properties of existing laboratory-sized heats of Alloy 709 austenitic stainless steel and the completion of effort on the thermal aging effect on the tensile properties of optimized G92 ferritic-martensitic steel.

Thermal aging experiments at 550, 600, and 650°C were carried out on three laboratory-sized heats of Alloy 709 austenitic stainless steel, 709-01 (SAI Heat 12013806-709), 709-02 (CarTech Heat 11367), 709-05 (CarTech Heat 011502). Tensile tests were conducted at the aging temperature in air at a nominal strain rate of 0.001 s^{-1} . It was found that thermal aging at 550°C had minimal effect on the tensile properties for times $< 10,000 \text{ h}$. Thermal aging at 600°C for $\sim 15,000 \text{ h}$ slightly increased the yield stress and decreased the ultimate tensile strength (UTS). The effect of thermal aging at 650°C was more pronounced on the tensile properties of Alloy 709. An increase in the yield stress and a decrease in the UTS were accompanied by the reduction in the uniform and total elongations. The yield stress was increased by $\sim 34\%$, the UTS was reduced by $\sim 9\%$, and the uniform and total elongations were reduced by $\sim 50\%$ and $\sim 36\%$, respectively after aging for $\sim 20,000 \text{ h}$ at 650°C. The effect of thermal aging on tensile properties showed little variations among three heats of Alloy 709.

Four heats of optimized G92 ferritic-martensitic steel with or without thermo-mechanical treatment (TMT), including Opt. G92-3, Opt. G92-4, Opt. G92-4T, and Opt. G92-8T were investigated to evaluate the effect of thermal aging on the tensile properties of optimized G92. Conventional G92 (Heat G92-0) was included in the study to provide the baseline data. Thermal aging at 550 - 650°C caused a decrease in the yield stress and the ultimate tensile strength but had an insignificant effect on tensile ductility of optimized G92. The effect of thermal aging was stronger at higher temperatures and longer exposure times. The effect of thermal aging on tensile strength became evident after $\sim 10,000 \text{ h}$ at 550°C, $\sim 1,000 \text{ h}$ at 600°C, and $< 1,000 \text{ h}$ at 650°C. The aging-induced reduction in tensile strength was much more pronounced at 650°C than at temperatures of 550-600°C, and exhibited large variations among different heats of optimized G92. The heat with a higher initial tensile strength (e.g. Opt. G92-3 and Opt. G92-8T) showed a much greater reduction in strength than those with lower initial tensile strength (e.g. Opt. G92-4 and Opt. G92-4T). Microstructural characterization of optimized G92 showed subgrain coarsening, coarsening of M_{23}C_6 and MX particles, and precipitation of Laves phase after thermal aging at 650°C. These microstructural changes can be correlated well with aging-induced tensile property degradation at 650°C.

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