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Revision 0

Status of INL Aged A709 Mechanical Testing

Changing the World's Energy Future

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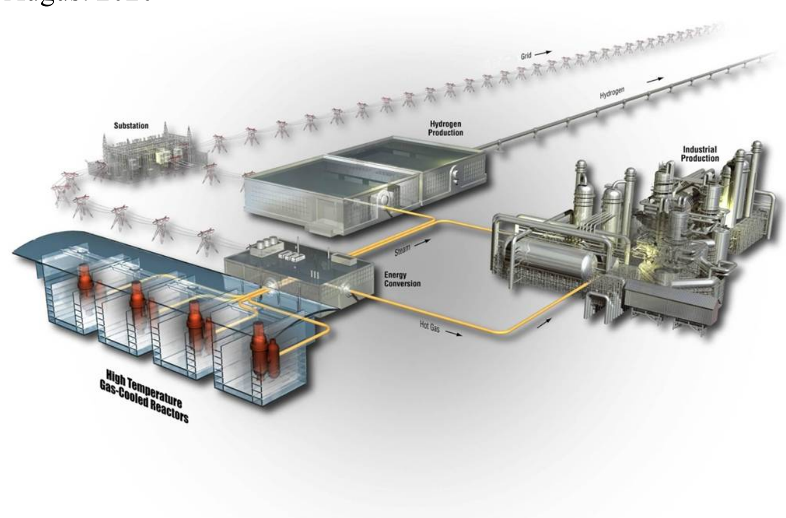
August 2020



Idaho National Laboratory

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SUMMARY

A709 was selected to be qualified in Section III, Division 5 of the American Society of Mechanical Engineers Boiler and Pressure Vessel Code. Idaho National Laboratory, Argonne National Laboratory and Oak Ridge National Laboratory are collaborating to develop and qualify A709 plate. Aging A709 to form beneficial precipitates prior to service is being investigated. One tensile test, five creep-rupture tests, and eight cyclic tests have been completed on aged A709. Two creep-rupture tests are in progress.

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TABLES

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ACRONYMS

ART	Advanced Reactor Technologies
ASME	American Society of Mechanical Engineers
ASTM	American Society for Testing and Materials
BPVC	Boiler and Pressure Vessel Code
ESR	Electro-slag remelting
FY19	Fiscal year 2019
FY20	Fiscal year 2020
FY21	Fiscal year 2021
INL	Idaho National Laboratory
UTS	Ultimate tensile strength

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1. INTRODUCTION

A709, an Fe-20Cr-25Ni austenitic stainless steel, is strengthened by a combination of solid solution strengthening from molybdenum and nitrogen and precipitate strengthening from carbo-nitrides. The properties of A709 plate were evaluated and compared to 316H stainless steel, which is qualified in Section III, Division 5 of the American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code (BPVC) for elevated-temperature nuclear construction. It was recommended that A709 be code qualified in Section III, Division 5 of the ASME BPVC for fast-reactor structural applications. A collaborative program with Argonne National Laboratory, Idaho National Laboratory (INL) and Oak Ridge National Laboratory is ongoing to develop and qualify A709 plate [1]. As part of this program, the first commercial heat of A709 was procured. The fabrication process parameters of this heat were investigated in order to optimize mechanical properties. A tradeoff between creep and creep-fatigue properties was identified. A709 formed by electro-slag remelting (ESR) with an 1100°C solution anneal was determined to have the best balance of properties [2, 3].

In the solution annealed condition, the majority of the solutes are in solution with some residual MX (Nb, Ti, V: C, N) precipitates present. During service, dynamic precipitation on dislocations is expected. This hinders dislocation movement, strengthening the material. Some challenging questions emerge from this expectation which includes the following: i) Does accelerated testing which utilizes higher stresses and temperatures result in precipitate kinetics that are representative of service conditions? ii) What is the kinetics associated with precipitate formation and coarsening, and how does this impact their effectiveness? iii) Is little to no precipitation or static precipitation a more realistic expectation than dynamic precipitation at service conditions? Aging A709 prior to service, so that beneficial precipitates are present at the start of service, may circumvent some of these concerns. A loss in A709's potential strength is expected as static precipitation is typically less effective at hindering dislocation movement than dynamic precipitation. Precipitate kinetics, primarily coarsening, remain a concern.

A heat treatment of 775°C for ten hours was developed to age A709. This heat treatment was chosen to promote the formation of $M_{23}C_6$ (Cr, Fe, Mo, Ni: C) while avoiding intermetallic laves phases (Fe_2Mo, Fe_2Nb). ESR A709 that was solution annealed at 1100°C and 1150°C was heat treated. These processing conditions will be identified as ESR 1100 SA and ESR 1150 SA, respectively, for the solution annealed condition for the remainder of this report. The aged conditions will correspondingly be referred to as ESR 1100 SA-A and ESR 1150 SA-A. These processing conditions were chosen because ESR 1100 SA had the best overall properties. ESR 1150 SA had the best creep properties but poor creep-fatigue properties. Testing to evaluate the impact of aging so that the tradeoffs between solution annealing and aging can be adequately assessed is in progress. The purpose of this report is to document the status of the INL testing of the aged A709.

2. EXPERIMENTAL PROCEDURE

2.1 Material

A709 plate, procured from Electralloy G. O. Carlson, was investigated. The plate had a nominal thickness of 1.1 inches. The chemistry of the ESR ingot is provided in Table 1 below. Two different solution anneal temperatures, 1100 and 1150°C, were investigated. The 1100 and 1150°C solution annealed plates received an additional heat treatment of 775°C for ten hours in order to age the material. Specimens were machined so that the longitudinal direction was parallel to the rolling direction of the plate.

Table 1. Chemical composition in weight percent of the ESR A709 ingot, heat 58776-1R.

C	Mn	Si	P	S	Cr	Ni	Mo
0.066	0.90	0.39	0.014	0.001	20.06	25.12	1.50
N	**Nb**	**Ti**	**Cu**	**Co**	**Al**	**B**	**Fe**
0.160	0.26	0.02	0.06	0.02	0.02	0.0035	Bal[1]

[1] Bal = balance

2.2 Tensile Testing

Room-temperature tensile testing was performed in accordance with the American Society for Testing and Materials (ASTM) standard E8 with an electromechanical test frame [4]. Testing was performed with a constant crosshead displacement of 0.030 inches per minute (0.75 millimeters per minute). Strain was recorded with an extensometer. Tests were executed to the full measuring potential of the extensometer and then paused so that the extensometer could be removed. Tests were then resumed to completion. After extensometer removal, strain was determined from the crosshead displacement.

A cylindrical test specimen geometry with a 0.25-inch diameter and 1.260-inch reduced section was used. Important specimen dimensions were measured before and after testing with calipers. Prior to testing, the gage was marked with two indentations that were one inch apart. The distance between the indentations was measured with calipers post-test. Elongation and reduction in area were calculated from these measurements.

2.3 Creep Testing

Uniaxial creep-rupture testing in accordance to PLN-3386 and following the general guidelines of ASTM E139 were performed [5, 6]. These tests were conducted in air at a constant elevated-temperature with a fixed applied load. The test frames were manufactured by Applied Test Systems and generally utilized a 20:1 lever arm. Creep strain with a resolution greater than 0.01% was measured using an extensometer connected to dual averaging linear variable differential transformers or Heidenhain linear encoded photoelectric gauges. Specimen temperature was monitored by two thermocouples located in the vicinity of the top and bottom of the specimen gage. K-type thermocouples were generally used for tests at temperatures of 750°C and below. Otherwise, R-type thermocouples were used. Specimen temperature was primarily maintained within 2°C of the desired test temperature as recommended by ASTM E139 [6].

Creep testing utilized the same specimen geometry as tensile testing. Critical dimensions were measured with a 50x optical comparator prior to testing and calipers after rupture when the specimen was at room temperature. Elongation and reduction in area were calculated from these measurements.

2.4 Elevated-temperature Cyclic Testing

Elevated-temperature axial strain-controlled cyclic testing were performed in accordance to PLN-3346 [7]. Fatigue and creep-fatigue testing widely met ASTM E606 and E2714 specifications, respectively [8, 9]. Testing was performed in air using servo-hydraulic test frames fitted with three-zone resistance furnaces, both manufactured by Materials Test Systems. Temperature was monitored by two R-type thermocouples spot welded to the specimen shoulders. Strain was measured using a direct-mounted extensometer manufactured by Epsilon Technology. The extensometer had a 0.47-inch (12 mm) gage length with a 0.098-inch (2.5 mm) travel range in both tension and compression.

Fatigue testing was at 650°C with a fully-reversed 1% total strain range and 10^{-3} s^{-1} strain rate. This utilized a symmetric triangular waveform. Creep-fatigue testing included the addition of a 30-minute hold at the peak tensile strain. A button-head specimen geometry with a 0.295-inch diameter was used. Critical dimensions were measured with a 50x optical comparator prior to testing.

Cyclic data was analyzed to determine the number of cycles to failure, N_f. This required identifying the number of cycles to crack initiation, N_o. N_o is the cycle when the maximum peak stress deviated from linearity. N_f was defined as a 25% drop in peak maximum stress from N_o. Please refer to Figure 1 in ASTM E2714 for schematics explaining the determination of N_o and N_f [9]. The optimal cyclic test involves the primary crack forming within the gage length of the extensometer. This location is referred to IX in this report. Sometimes, however, the primary crack can occur outside the extensometer or at the extensometer knife edge. These locations are correspondingly referred to as OX and AX. When the primary crack forms outside the extensometer, the extensometer is unable to measure crack initiation and propagation. This leads to the sample, upon crack initiation, being strained greater than the desired amount. Consequently, N_o is not able to be determined and N_f is reported as the number of machine cycles to failure. While this test is not considered valid under the strictest sense, the test can still provide valuable information. The validity of a test with an AX primary crack is dependent on the individual test. Sometimes the test can be analyzed in the same manner as when the primary crack is IX. Other times it is treated the same as when the primary crack is OX.

3. RESULTS

3.1 Tensile Testing

In fiscal year 2019 (FY19), an ESR 1100 SA-A room-temperature tensile test was completed. The results from the test are shown in Table 2 below. An issue with the test resulted in approximately ten minutes of data from not being collected during the middle of the test. This did not impact evaluation of the yield stress, elongation, or reduction in area. It did prevent the exact value of the ultimate tensile strength (UTS) from being measured. The largest stress recorded during the test was 687 MPa. Therefore, the UTS is equal to or greater than 687 MPa.

Table 2. Results from a room-temperature ESR 1100 SA-A tensile test.

Specimen	**Yield stress**	**UTS**	**Elongation**	**Reduction in area**
	MPa	MPa	%	%
D775-C4	344	≥ 687	42.4	53.7

3.2 Creep Testing

A total of seven creep tests of the aged A709 have been completed or are in progress. The results from these tests are shown in Table 3 below. The ESR 1100 SA-A creep-rupture test was completed in FY19. The six ESR 1150 SA-A tests were started in fiscal year 2020 (FY20).

Table 3. Results from ESR 1100 SA-A and ESR 1150 SA-A creep-rupture tests.

Specimen	Temperature (°C)	Stress (MPa)	Rupture time (hour)	Elongation (%)	Reduction in area (%)
			ESR 1100 SA-A		
D775-C1	600	330	1,524	43.3	55.0
			ESR 1150 SA-A		
BCHT-IC-1	600	330	1,387	45.1	51.5
BCHT-IC-2	925	35	196	66.8	76.9
BCHT-IC-3	700	175	357	68.8	77.6
BCHT-IC-4	925	27	686	60.0	59.6
BCHT-IC-5	650	175	In progress		
BCHT-IC-6	650	155	In progress		

3.3 Elevated-Temperature Cyclic Testing

A total of eight elevated-temperature cyclic tests of the aged A709 have been completed. For each solution anneal temperature investigated, two fatigue and two creep-fatigue tests were performed. The results from these tests are shown in Table 4 below. ESR 1100 SA-A and ESR 1150 SA-A cyclic testing was completed in FY19 and FY20, respectively.

Table 4. Results from ESR 1100 SA-A and ESR 1150 SA-A cyclic tests.

Specimen	Hold time	N_o	N_f	Primary crack location	Cycle 10				Cycle Midlife			
					σ_{max}	σ_{min}	$\sigma_{hold\ start}$	$\sigma_{hold\ end}$	σ_{max}	σ_{min}	$\sigma_{hold\ start}$	$\sigma_{hold\ end}$
	minutes	cycle	cycle		MPa	MPa	MPa	MPa	MPa	MPa	MPa	MPa
ESR 1100 SA-A												
D775-F1	0	1,773	1,955	IX	287	-291	-	-	331	-337	-	-
D775-F2	0	-	1,388	OX	293	-300	-	-	332	-337	-	-
D775-F3	30	-	941	OX	293	-307	273	205	315	-348	313	190
D775-F4	30	935	1,285	AX	293	-307	287	191	302	-336	301	193
ESR 1150 SA-A												
BCHT-IF-1	0	1,764	1,913	AX	273	-278	-	-	325	-330	-	-
BCHT-IF-2	0	1,701	1,791	IX	286	-293	-	-	330	-333	-	-
BCHT-IF-3	30	1,048	1,064	IX	281	-293	192	162	306	-338	303	198
BCHT-IF-4	30	1,155	1,169	IX	285	-296	240	175	307	-338	303	185

4. SUMMARY

A tensile test, five creep tests, and eight cyclic tests of the aged A709 have been completed at INL. Creep-rupture testing of two ESR 1150 SA-A specimens are in progress. In fiscal year 2021 (FY21), testing of a second commercial heat of solution annealed and aged A709 will commence at INL.

5. Works Cited

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