

The Initiation of Long-Term Creep Rupture Tests on the First Alloy 709 Commercial Heat



Hong Wang
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Materials Science and Technology Division

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COMMERCIAL HEAT

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ABSTRACT

Systematic creep tests were conducted as part of a multi-lab effort to identify the material conditions for Alloy 709 Code Case Project. Nine candidate material conditions were studied with three melt conditions each with three S/A temperatures 1050, 1100 and 1150°C. The three melt conditions included AOD, ESR and ESR + Homogenization. The screening creep rupture tests were conducted under 600°C, 330MPa. AOD 1100 and ESR 1100 have been identified to be the top two material conditions for the Code Case project after a comprehensive evaluation.

Long-term creep tests were initiated on AOD 1100 at the ORNL. The six creep tests were started at temperatures from 550 to 800°C, stresses from 38 to 309 MPa to target rupture time 60,000 hours. The loading strains agreed with those obtained from tensile tests at the corresponding temperatures.

ACRONYMS AND ABBREVIATIONS

AAD	Advanced alloy development
AAT	Advanced alloy testing
ANL	Argonne National Laboratory
AOD	Argon-Oxygen-Decarburization
ART	Advanced Reactor Technologies
ASME	American Society of Mechanical Engineers
ASTM	American Society for Testing and Materials
BPV	Boiler and pressure vessel
DEG	Degree
DOE	US Department of Energy
EL	Elongation
ESR	Electroslag remelt
HOMO	Homogenization
INL	Idaho National Laboratory
LVDT	Linear variable differential transformer
LS	Loading strain
MCR	Minimum creep rate
NE	Office of Nuclear Energy
ORNL	Oak Ridge National Laboratory
S/A	Solution annealed
RA	Reduction of area

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1. INTRODUCTION

A multi-Laboratory effort among Argonne National Laboratory (ANL), Oak Ridge National Laboratory (ORNL), and Idaho National Laboratory (INL) under the DOE-NE, Office of Advanced Reactor Technologies (ART), has been initiated to develop an ASME Boiler and Pressure Vessel (BPV) Code Case for Alloy 709. The Code Case is to enable the construction of ASME Section III, Division 5, Class A components for sodium fast-reactor applications using this alloy [1].

A campaign to refurbish and upgrade enough creep frames was conducted at the ORNL in FY16 and FY17 under the sponsorship of the ART program, Advanced Alloy Testing (AAT) – ORNL to support the Alloy 709 Code Case. Creep curves and creep rupture times are critical components of the Code Case data package, which requires performing long-term creep tests with target rupture times of 60,000 and 100,000 hours. The test matrix for the long-term testing developed for this Code Case includes 36 tests from three heats of base metal at temperatures between 550 and 800° C, and 4 cross-weld tests for one filler material under similar temperature ranges. Totally, 40 creep frames are needed to accomplish the proposed matrix. The Mechanical Properties & Mechanics (MP&M) Group at ORNL is responsible for the operation of a large collection of mechanical testing machines with strain rate capabilities over 12 orders of magnitude from the creep regime to high-rate impact testing. Of these mechanical testing machines, 22 creep frames (#64, 65, 76 to 83, 156, 157, and 510 to 519) have been used and partially maintained by the ART program, and thus committed to the Code qualification of Alloy 709. Thereafter, ORNL identified 18 un-refurnished creep frames within the inventory of the MP&M group (#5 to 8, 301 to 307, and 88 to 95 with 92 excluded) as complementary frames for the potential project use. The shakedown tests for refurbished and upgraded creep frames were documented in a related report [2].

The screening creep tests of the candidate material conditions for the Code Case development continued at ORNL in FY18. In a work package sponsored by the ART program, Advanced Alloy Development (AAD) – ORNL, a large commercially-sized heat of Alloy 709 was procured in FY17 as part of fabrication scale-up effort (about 45,000 lbs.) [3]. The master heat had different processing conditions, such as argon-oxygen-decarburization (AOD), electroslag remelting (ESR), and electroslag remelting with subsequent homogenization (ESR+HOMO). The supplied plates were fabricated by hot-rolling, followed by solution annealing (S/A) at temperatures 1922°F (1050°C), 2012°F (1100°C), and 2102°F (1150°C). The tests at ORNL covered all the 9 heat conditions, namely, 3 melt conditions each with 3 S/A temperatures. A comprehensive evaluation has been performed on all the candidates by incorporating the screening data from the INL, ANL, and ORNL. AOD with S/A 1100°C (so-called AOD 1100), and ESR with S/A 1100°C (so-called ESR 1100) emerge to be the top two material conditions.

The long-term creep tests on the Alloy 709 were initiated at the ORNL in FY18. The long-term tests focused on AOD 1100 with 60,000 hours as a target life in this stage.

This report describes part of the ORNL activities related to screening creep tests and long-term creep tests of Alloy 709 in FY18 and the related results. All the work performed in this project followed either the ASME NQA-1-2008 requirements or ASTM E139-11.

2. CREENING CREEP TESTS

2.1 MATERIAL AND TEST CONDITION

Alloy 709 materials used for the creep rupture tests were from the hot-rolled plates fabricated from the first Alloy 709 commercially- sized heat procured to support testing for this program.

The mechanical property data obtained from room-temperature tests and microstructure screening results were reported previously [3]. The screening creep tests at ORNL covered the nine material conditions comprised of three melt conditions each with three S/A temperatures. The dimensions of the nine small plates allocated for the creep test task at the ORNL are given in Table 1. The chemical compositions for the two sub-heats tested at the ORNL are shown in Table 2 and Table 3, respectively.

Table 1. Heat condition and dimensions of small plates allocated for creep test tasks at the ORNL.

Melt cond.	Heat no.	Homo.	S/A, F	S/A, C	Lot Mark	ORNL ID	Dimensions* (inch ³)	Dimensions (mm ³)
AOD	5877 6-4	No	1922	1050	S/A 1922 DEG	776-4A1-S203	1.2×28.9×8.5	30×735×216
			2012	1100	S/A 2012 DEG	776-4B1-S203	1.2×28.9×8.5	30×735×216
			2102	1150	S/A 2102 DEG	776-4C1-S203	1.2×29×8.5	30×737×216
ESR	5877 6-3R	No	1922	1050	ESR S/A 1922 DEG	776-3RBA1-S203	1.1×29.7×8.5	28×754×216
			2012	1100	ESR S/A 2012 DEG	776-3RBB1-S203	1.1×29.7×8.5	28×752×216
			2102	1150	ESR S/A 2102 DEG	776-3RBC1-S203	1.1×29.9×8.5	28×759×216
ESR+ HOM O	5877 6-3R	Yes	1922	1050	ESR HOMO S/A 1922 DEG	776-3RAA1-S203	1.1×22×8.5	28×559×216
			2012	1100	ESR HOMO S/A 2012 DEG	776-3RAB1-S203	1.1×28.9×8.5	28×733×216
			2102	1150	ESR HOMO S/A 2102 DEG	776-3RAC1-S203	1.1×28.9×8.5	28×733×216

*Corresponding to dimensions in thickness, transverse and rolling directions, respectively.

Table 2. Chemical composition of Alloy 709 heat #58776-4, wt%

C	Mn	Si	P	S	Cr	Ni	Mo	N	Nb	Ti	Cu
.07	.91	.44	.014	< .000	19.93	24.98	1.51	.148	.26	.04	.06
Co	Al	B	Fe								
.02	.02	.0045	Balance								

Table 3. Chemical composition of Alloy 709 heat #58776-3R, wt%

C	Mn	Si	P	S	Cr	Ni	Mo	N	Nb	Ti	Cu
.066	.90	.38	.014	.001	20.05	25.14	1.51	.152	.26	.01	.06
Co	Al	B	Fe								
.02	.02	.003	Balance								

A scoping test matrix consisting of two creep rupture tests for each plate shown in Table 1, all with the same test condition of 600°C and 330 MPa, was developed. The test condition matches that in a previous property screening tests at ORNL for small-sized laboratory heats. The use of the proposed test condition is expected to produce a life time of 1,500 hours.

2.2 SPECIMEN AND FRAME PREPARATIONS

The specimen design was based on 3/8" (9.53 mm) gage diameter whose engineering drawing is shown in Fig. 1.

The initial creep tests were conducted by using a specimen design featuring dual ridges for extensometer mounting. Premature failures near the inner root of one of the mounting ridges suggested that a modification to the existing specimen design would be necessary. The modification considered either increasing radius of the ridge root or switching from ridging in reduced section into grooves in shoulder sections. The modification also considered minimizing that of the existing fitting of the extensometer and loading train to control the incurring cost. The design study finally recommended that the dual groove specimen (Fig. 1) be used in the project after a comprehensive examination. The related details can be found in a document addressed to the ORNL quality representative.^a

^a H. Wang, Technical Issue with Alloy 709 Creep Specimen and Re-Design, December 1, 2017, Oak Ridge National Laboratory, Oak Ridge, TN

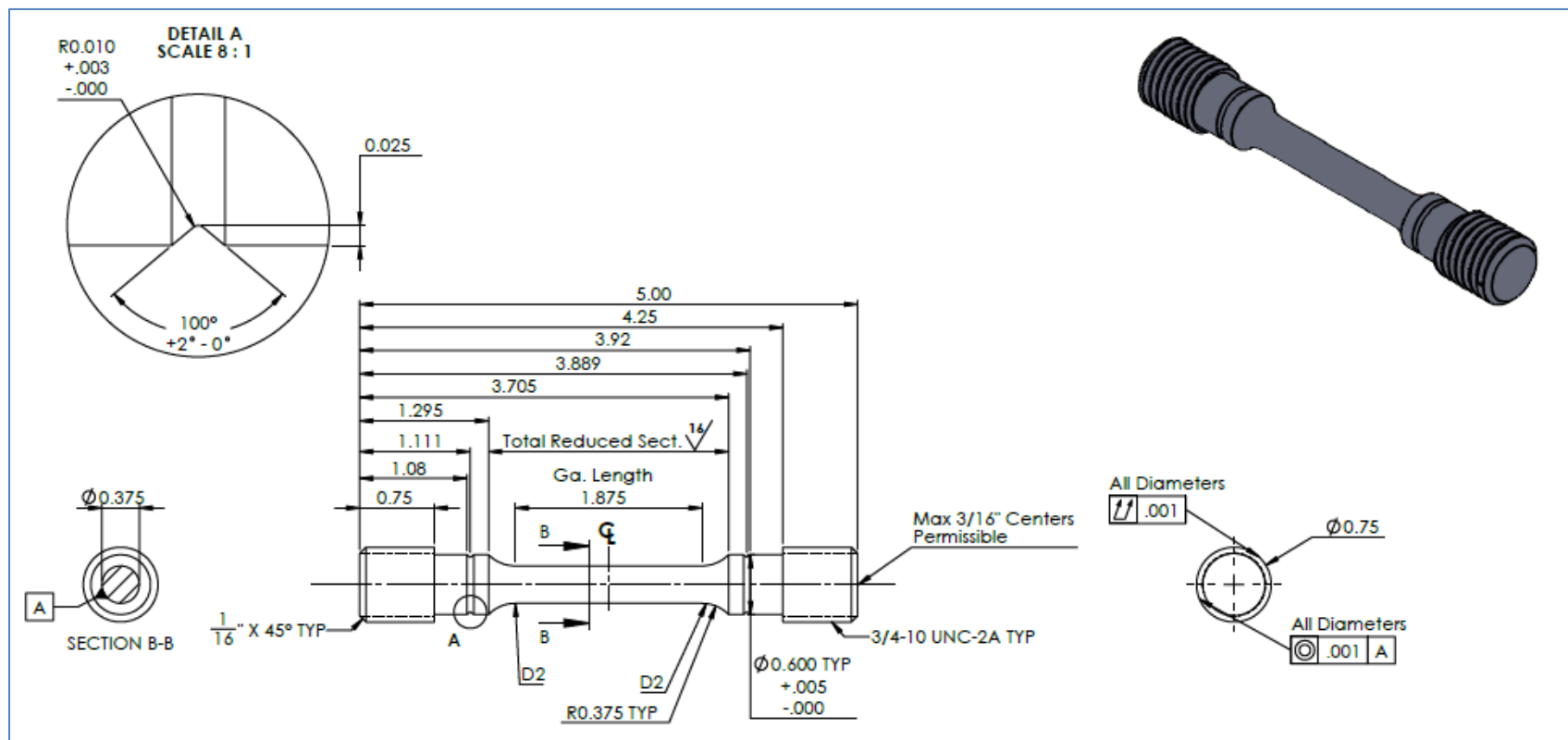


Fig. 1. Drawing used in machining Alloy 709 specimen (dimensions are in inches).

A small section, sized for two 3/8" (9.53 mm) gage diam. specimens, was cut by water-jet from each plate in Table 1 for use in the screening tests. The machining of 18 specimens was conducted by JV Precision (Oak Ridge, TN) through a quote solicitation process. The longitudinal axis of creep specimens was aligned with the rolling direction of the plates.

The required mechanical load under 330MPa was calculated to be 5,286 lbs. (23,513 N) for a 3/8" (9.53 mm) gage diameter specimen. Seven frames (#5 - #8 and #88 - #90) were identified for the screening tests because they have the desired load capacity of 10,000-lb. The main changes to these frames arising from using the new specimen design was to switch the specimen collet from groove type into ridge type for the compatibility of extensometer mounting. Machining of the new collets was also conducted by JV Precision (Oak Ridge, TN).

2.3 TEST RESULTS

2.3.1 Verification Test for Specimen Design

An accelerated test was performed to verify the design and fabrication of the creep specimens. An AOD specimen with S/A 1050°C was tested under 227MPa, 700°C (TN 33224, specimen #4A1-S21-2). Frame #307 was used due to a low level of expected mechanical loading. The specimen ruptured at 48 hours, which was less than 200 hour expected lifetime. The lower lifetime is understandable considering that an increasing S/A temperature tends to enhance the rupture life. The prediction of 200 hour lifetime was based on the data for the material whose S/A temperature was 1150°C, whilst the tested material had a lower S/A temperature.

The post-test examination indicated that the failure position was within the gage section and located away from the mounting groove as shown in Fig. 2. Significant elongation and reduction of area were developed with 44% and 53%, respectively. So, the new design was shown to be effective in preventing the creep specimen from the premature brittle failure seen in the specimen with ridges.



Fig. 2 Tested specimen of AOD with S/A 1050°C, under 227 MP, 700°C; rupture time 48 hours.

2.3.2 Screening Creep Tests

A total of 13 creep tests were conducted under the suggested testing condition for the nine material conditions. As of 8/13/2018, there have been 8 creep tests completed and 5 ongoing. A summary of the test status and test results is given in Table 4, in which MCR stands for minimum creep rate, and ϵ_c and ϵ_{tl} are the creep and total strains at the time of rupture or reporting. Creep curves in terms of creep strain and total strain are given in Fig. 3 and Fig. 4; ESRHM means ESR + HOMO (same comment applies to other figures whenever it appears).

The post-test measurements of the tests completed in terms of elongation (EL) and reduction of area (RA) are given in Table 5. The failures of these specimens were all located within the gage section as shown in Fig. 5.

Table 4. Summary of screening creep test results obtained under 330MPa, 600°C

TN	Mach	Spec	Melt	S/A, C	Time,* h	MCR,** %/h	ϵ_c , %	ϵ_{tl} , %	Status
33258	5	4A1-S21-1	AOD	1050	761	9.57E-03	12.67	14.92	Ruptured
33259	6	4B1-S21-1	AOD	1100	1,699	3.38E-03	9.07	15.82	Ruptured
33468	89	4B1-S21-2	AOD	1100	1,695	2.16E-03	4.66	10.04	Ongoing
33260	7	4C1-S21-1	AOD	1150	2,224	2.07E-03	6.85	14.51	Ruptured
33498	6	4C1-S21-2	AOD	1150	858	1.98E-03	2.29	9.74	Ongoing
33262	8	3RBA1-S22-1	ESR	1050	1,033	1.08E-02	26.53	28.04	Ruptured
33499	90	3RBA1-S22-2	ESR	1050	920	1.24E-02	26.89	28.36	Ruptured
33265	88	3RBB1-S22-1	ESR	1100	2,931	2.12E-03	21.02	25.26	Ruptured
33563	7	3RBB1-S22-2	ESR	1100	427	3.38E-03	1.79	6.13	Ongoing
33469	8	3RBC1-S22-1	ESR	1150	1,634	1.26E-03	2.82	7.78	Ongoing
33266	89	3RAA1-S22-1	ESR+HOMO	1050	817	9.47E-03	15.58	17.2	Ruptured
33267	90	3RAB1-S22-1	ESR+HOMO	1100	1,886	3.85E-03	14.85	20.23	Ruptured
33470	5	3RAC1-S22-1	ESR+HOMO	1150	1,611	1.51E-03	3.27	9.58	Ongoing

* As of 8/13/2018; **Minimum creep rate;

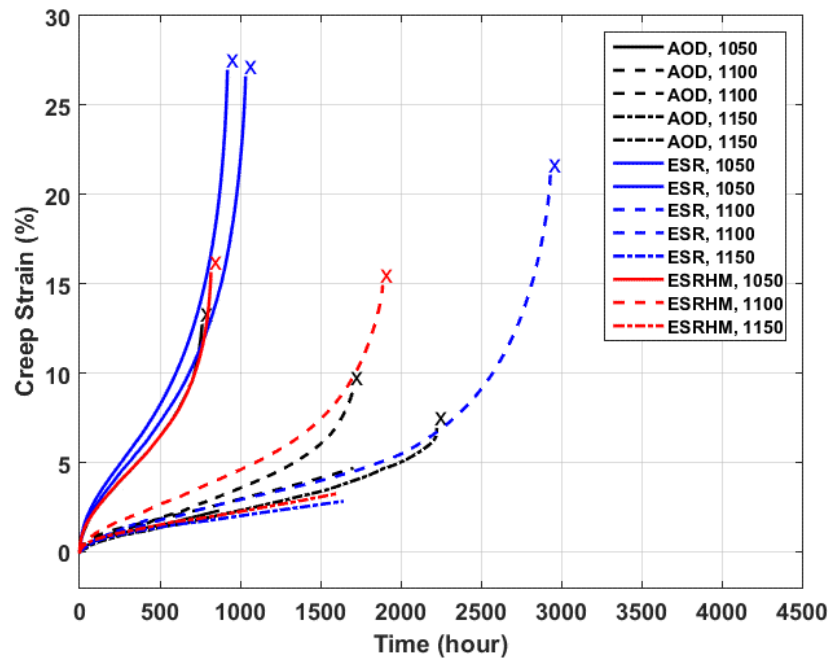


Fig. 3. Creep strain curves obtained under 330MPa, 600°C for various material conditions; symbol “x” indicates the failure of specimen.

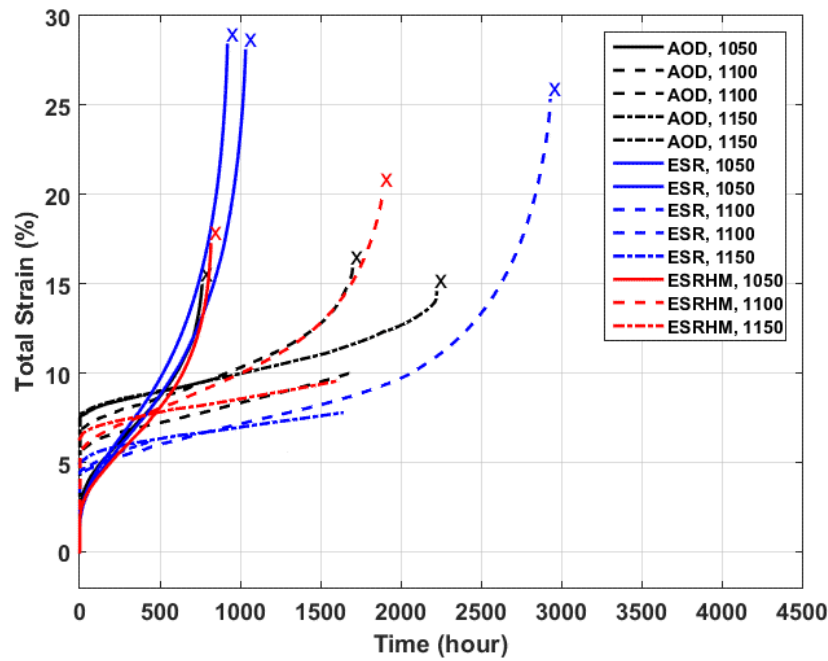


Fig. 4. Total strain curves obtained under 330MPa, 600°C for various material conditions; symbol “x” indicates the failure of specimen.

Table 5. Post-test measurements with testing condition 330MPa, 600°C

TN	Mach	Spec	Melt	S/A, C	Time, h	EL, %	RA, %
33258	5	4A1-S21-1	AOD	1050	761	14.78	17.06
33259	6	4B1-S21-1	AOD	1100	1,699	17.41	15.60
33260	7	4C1-S21-1	AOD	1150	2,224	14.19	12.22
33262	8	3RBA1-S22-1	ESR	1050	1,033	28.30	26.19
33499	90	3RBA1-S22-2	ESR	1050	920	28.96	30.03
33265	88	3RBB1-S22-1	ESR	1100	2,931	26.24	27.21
33266	89	3RAA1-S22-1	ESR+HOMO	1050	817	17.54	20.25
33267	90	3RAB1-S22-1	ESR+HOMO	1100	1,886	20.26	19.08



4A1-S21-1



4B1-S21-1



4C1-S21-1



3RBA1-S22-1



3RAA1-S22-1



3RAB1-S22-1

Fig. 5. Images of tested specimens with various material conditions; test condition was 330 MPa, 600°C.

2.4 DISCUSSION AND SUGGESTION

The refurbished creep frames have been shown to work as designed. Some of them have accumulated a use time of 7,260 hours (on the specimens of old and new designs). The displacement measurement has been shown to be reliable. The total strains measured by extensometers (dual LVDTs) agree with the elongations from post-test measurements as seen in Fig. 6. Minor discrepancy is related to the elastic strain captured online, or unclosed gap of mating fracture surfaces in the pots-test measurement.

The screening tests covered all the nine materials condition with 8 completed and 5 ongoing. Among the 13 tests, four are duplicate tests. The duplicates displayed a repeatable response as can be seen from the creep curves of ESR 1050 (Fig. 3 and Fig. 4), in which the creep variation, rupture time and final creep/total strain are all similar.

Loading strain (produced before the creep starts) increases systematically with S/A temperature as seen in Fig. 7. This is expected because of the grain growth with the increasing S/A temperature. Like the room temperature behavior, AOD exhibited more loading strain than other two melt conditions [3]. However, ESR behaved a little differently with the lowest loading strain among the three melt conditions.

S/A temperature had a noticeable effect on the rupture time, which can be seen from AOD that has the complete data series in Fig. 8. The trend observed is consistent with the data sets from a different source [4]. In general, more than half of rupture times exceeded the expected life 1,500 hours except those with S/A temperature 1050°C. Particularly, the rupture time with ESR 1100 reached 2,931 hours. A more conclusive observation can be obtained when more data are obtained.

S/A temperature had a considerable effect on the minimum creep rate (MCR) as well as can be seen in Fig. 9. A detailed examination indicates that the effect of S/A temperature decreases when it varies from 1100 to 1150°C. On the other hand, the effect of melt condition seems uncertain. Nevertheless, the MCRs agree with those reported for specific material conditions. For example, the MCRs for S/A 1150°C in this study are very close to that of NF709 (S/A 1150°C) under similar testing condition; namely, $1.40\text{E-}03$ %/h under 333 MPa, 600°C [5].

The effect of S/A temperature on the total strain or ductility appears to be less significant as shown in Fig. 10. This is different from that observed for room temperature tensile behavior where the elongation grew with the increasing S/A temperature. It seems that the loading consumed enough strain in the case of S/A 1150°C, resulting in the flat response. On the other hand, ESR displayed an impressive amount of total strain or elongation for both S/A 1050 and S/A 1100. Again, a clearer picture will be possible when the data series are completed.

Besides the creep rupture life, additional technical issue with the down-selection of the candidates for Code Case project lies in the creep-fatigue (C-F) of the material. The work on small-size laboratory heats revealed that the secondary re-crystallization at S/A temperatures like 1150°C or higher could be detrimental to the C-F performance. The map in Fig. 11 illustrates ESR 1100 seems better than other material conditions in terms of C-F and creep rupture lives. Next runners-up could be AOD 1100 and AOD 1150. However, the additional manufacturing cost with increased temperature and the deceased C-F life offset the marginal increase in the creep rupture time in the case of AOD 1150. Therefore, AOD 1100 and ESR 1100 are the two material conditions that are considered in next step.

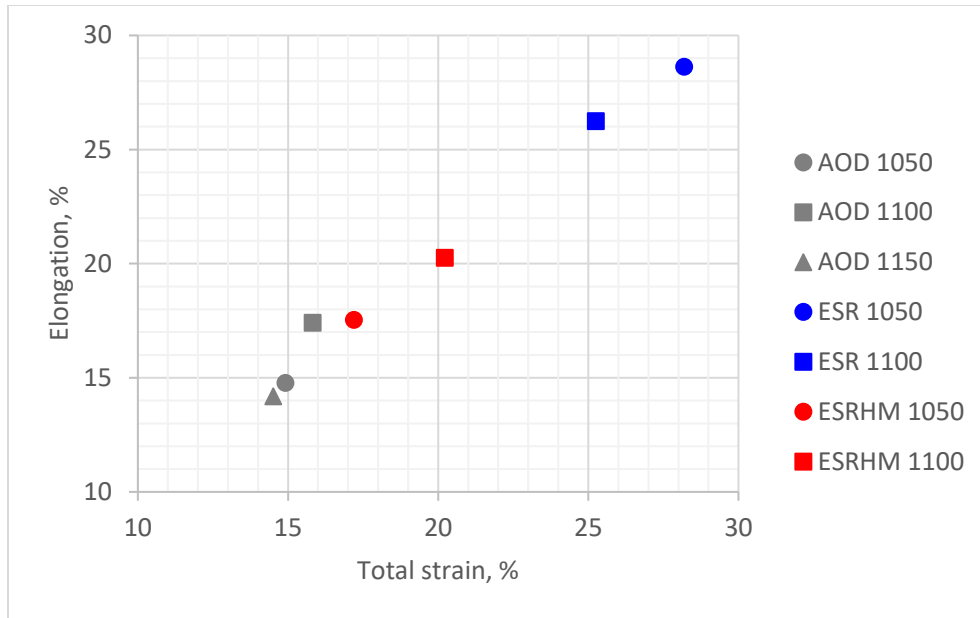


Fig. 6. Elongation based on post-test measurement vs. total strain measured by LVDTs; 330MPa, 600°C.

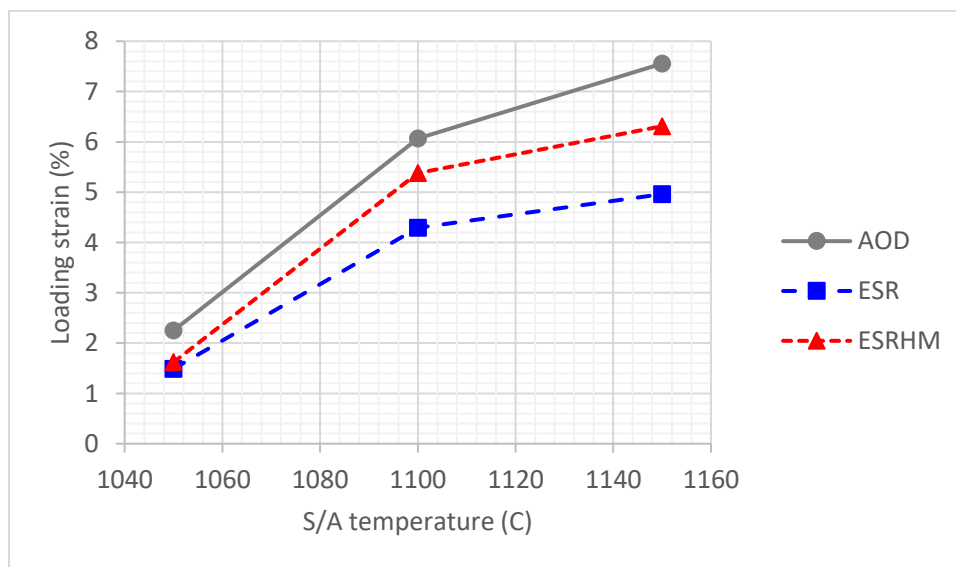


Fig. 7. Load strain vs. S/A temperature; 330MPa, 600°C.

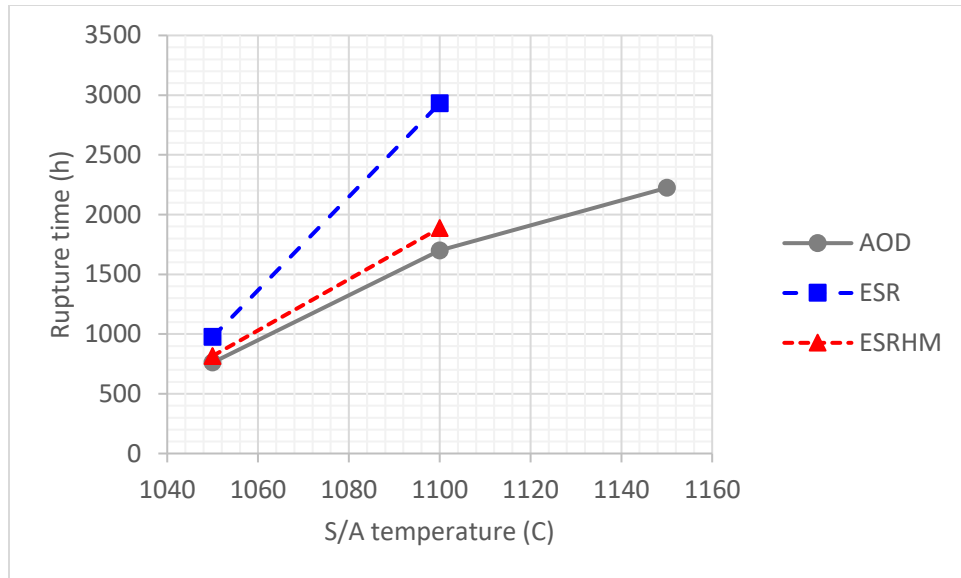


Fig. 8. Rupture time vs. S/A temperature; 330MPa, 600°C.

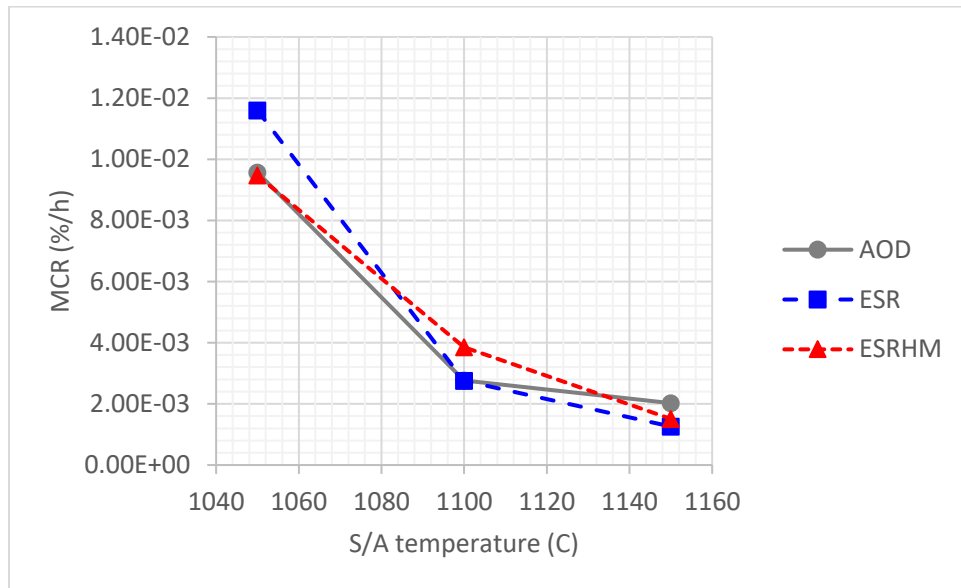


Fig. 9. MCR vs. S/A temperature; 330MPa, 600°C.

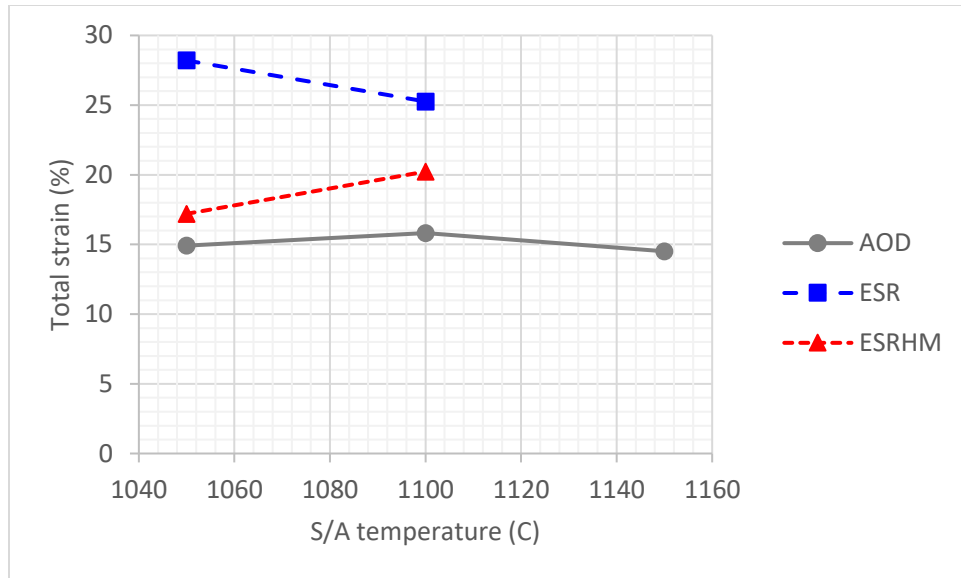


Fig. 10. Total strain vs. S/A temperature; 330MPa, 600°C.

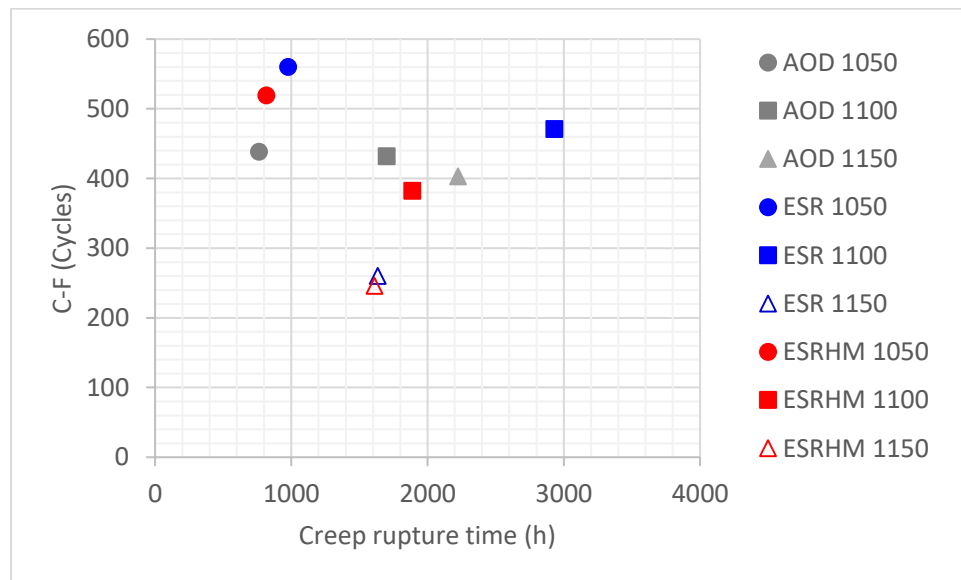


Fig. 11. Cycles to failure under C-F with 650°C, 1.0% total strain, 10^{-3} /s strain rate, 30 min. holding time vs. creep rupture time under 330 MPa, 600°C; open symbol indicates creep test is ongoing.

3. LONG-TERM CREEP TESTS

3.1 MATERIAL AND TEST MATRIX

AOD 1100 was used in the long-term tests in this stage of project. The chemistry composition of relevant heat 58776-4 can be found in Table 2.

The stress matrix related to 60,000- hour long-term test is given in Table 6.

Table 6. Stress matrix for 60,000- hour long-term tests.

Temperature, C	Stress, MPa
550	309
600	204
650	134
700	88
750	58
800	38

3.2 SPECIMEN AND FRAME PREPARATIONS

The 3/8-in. (9.53 mm) specimen design with dual mounting grooves as shown in Fig. 1 was used. Eight specimens were prepared from the AOD 1100 material reserved for creep test task at the ORNL, 776-4B1-S203, Table 2. The machining of the specimens was carried out by ORNL machine shop. Six of them were used in the long-term tests in this stage.

Frames #88, 91, 93, 95, 302, 303 were identified and prepared. These frames are equipped with load cells with various capacities as shown in Table 7 and capable of meeting the loading requirements.

Table 7. Frame conditions used in long-term tests

Frame Number	Lever arm ratio	Load cell capacity, lb
88	20	10,000
91	20	5,000
93	20	5,000
95	20	5,000
302	10	2,000
303	10	2,000

3.3 TEST RESULTS

Six long-term creep tests were started as planned for AOD 1100. A summary is given in Table 8, where ϵ_{td} has the same meanings as before. The tests all run at a relatively low level of creep.

The loading curve for one of the started tests is illustrated in Fig. 12. It is seen that the creep stress to target 60,000 hours of rupture time goes beyond the yield point. The estimated yield stress at 0.2% offset is 191 MPa, which is close to that obtained by tensile test for the corresponding material condition [6].

Overall, the loading strains agreed quite well with those extracted from the tensile strain-stress curves under relevant testing conditions, Table 9.

Table 8. Summary of long-term creep tests for material condition AOD 1100

TN	Mach	Spec	Temp, C	Stress, MPa	$\epsilon_{tcl},^*$ %	Status
33629	88	776-AOD-04	550	309	4.52	Ongoing
33630	93	776-AOD-02	600	204	0.73	Ongoing
33631	91	776-AOD-03	650	134	0.10	Ongoing
33632	95	776-AOD-01	700	88	0.05	Ongoing
33636	303	776-AOD-05	750	58	0.09	Ongoing
33635	302	776-AOD-06	800	38	0.05	Ongoing

*As of 8/31/2018

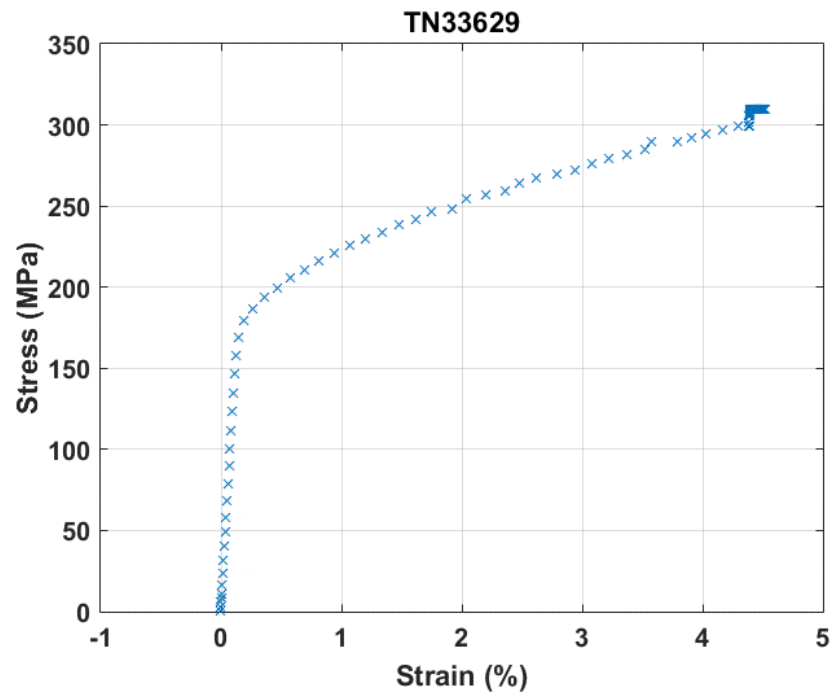


Fig. 12. Loading curve for AOD 1100 under 550°C

Table 9. Loading strains of AOD 1100 creep tests vs. strains based on tensile tests

TN	Temp, C	Stress, MPa	LS,* %	Strain from tensile test, %
33629	550	309	4.38	5.00 [9]
33630	600	204	0.70	
33631	650	134	0.10	0.09 [9]
33632	700	88	0.05	
33636	750	58	0.06	0.06 [10]
33635	800	38	0.04	

*LS: Loading strain

3.4 DISCUSSION

ORNL has substantial capability and expertise to conduct long-term creep experiments. Currently, there are four tests in the Creep Lab with an accumulated time of more than 60,000 hours, and one has run more than 100,000 hours.

The maintenance of the ART long-term creep tests will be based on the standard practice established at the ORNL. Routine inspection (weekly) of creep tests is implemented to ensure heating, loading and measurement systems work as designed. Data acquisition systems are also equipped with uninterruptible power supplies (UPSs) to handle the unexpected power outage. The responsibility of long-term creep tests is consistently relayed to next staff member whenever there is personnel change within the Lab. These procedures will be followed for the started long-term Alloy 709 tests to ensure that they run as designed.

4. SUMMARY

Systematic creep tests were conducted as part of a multi-lab effort to identify the material conditions for Alloy 709 Code Case Project. Nine candidate material conditions were studied with three melt conditions each with three S/A temperatures 1050, 1100 and 1150°C. The three melt conditions included AOD, ESR and ESR + Homogenization.

The screening creep rupture tests under 600°C, 330MPa suggested that both the S/A temperature and melt condition had a noticeable effect on the creep rupture time. The rupture time under AOD increased with the S/A temperature. Meanwhile, ESR exhibited a rupture time longer than other two, disregarding the S/A temperature. Although there is a marginal increase in creep rupture with S/A temperature increase from 1100 to 1150°C, the material suffers loss of C-F life with such increase. AOD 1100 and ESR 1100 emerged to be the top two candidates for the Code Case project after a comprehensive evaluation.

Long-term creep tests were initiated on AOD 1100. The six creep tests run at temperatures 550 to 800°C, stresses 38 to 309 MPa to target rupture time 60,000 hours. The loading strains agreed with those obtained from tensile tests at the corresponding temperatures.

5. REFERENCES

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