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Shakedown Tests for Refurbished and Upgraded Frames and Initiation of Alloy 709 Creep Rupture Tests

Hong Wang
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Materials Science and Technology Division

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ABSTRACT

This report describes the shakedown tests performed on the upgraded frames and the initiation of creep rupture tests on the refurbished frames. The shakedown tests were conducted to verify the operation of the frames' instrumentation. SS316H, a reference material for Alloy 709, was used for these tests, which were conducted at 816° C under three stress levels to accumulate 1% creep strain. The diameter of the gage section of the test specimens was 1/4″ (6.35 mm).

The creep rupture tests on Alloy 709 were initiated at 600° C under stress 330MPa to target 1,500 hour rupture time. The experimental work is part of an ongoing effort to screen the material conditions of Alloy 709. Twelve specimens with 3/8″ (9.53 mm) gage diameter were prepared from the materials with six heat treatment conditions, two from each. Frames #5 - #8 and #88 - #90 equipped with a 10,000-lb. load cell were used for the seven tests in this stage of the project. The test results indicated that all the refurbished and upgraded creep frames would work properly.

ACRONYMS AND ABBREVIATIONS

AAD	Advanced alloy development
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
ATS	Applied Test Systems
BPV	Boiler and pressure vessel
DAQ	Data acquisition
DEG	Degree
DOE	US Department of Energy
ESR	Electroslag remelt
HOMO	Homogenization
ID	Inside diameter
INL	Idaho National Laboratory
LVDT	Linear variable differential transformer
MCR	Minimum creep rate
NE	Office of Nuclear Energy
NI	National Instruments
OD	Outside diameter
ORNL	Oak Ridge National Laboratory
S/A	Solution annealed
TC	Thermocouple
UTS	Ultimate tensile strength
YS	Yield stress

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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.

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. The 40 long-term tests dominate the majority of use time on the creep frames and also determine the total number of creep frames required for the project.

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 to perform tests from the creep regime to high-rate impact testing. Of these mechanical testing machines, 22 creep testing frames (#64, 65, 76 to 83, 156, 157, and 510 to 519) are being used to support DOE's ART program and have been committed to the Code qualification of Alloy 709. Therefore, 18 more frames are needed to perform this project. Work in FY 2016 and part of FY2017 was carried out to refurbish the 18 frames identified within the inventory of the Creep Laboratory, MP&M group (#5 - #8, #301 - #307, and #88 - #95 with #92 excluded), and also to upgrade the existing 22 frames.

In the last stage of the project, ORNL continued addressing remaining issues associated with:

- data acquisition (DAQ) system that had an outdated Labview program and insufficient computer memory for data storage,
- instruments overdue for calibration, and
- thermocouples that were not suitable for long term high temperature use like 800°C and had to be replaced.

It is important to verify the instruments before the Code Case data are developed, and shakedown tests serve this purpose. SS316H was employed. This is a reference material for the Alloy 709 codification so that data generated can be used as a baseline as well. The proposed testing condition is temperature 816°C with various stress levels from 18 to 27 MPa. If allowed to run, the tests would be finished with different rupture times. However, it was suggested to discontinue the tests whenever 1% creep strain is reached.

The tests on Alloy 709 materials with six heat treatment conditions were initiated all under same condition 330 MPa, 600°C. In a work package sponsored by DOE NE ART program, Advanced Alloy Development (AAD) – ORNL, a large commercially-sized heat of Alloy 709 was procured in FY2017 as part of fabrication scale-up effort (about 45,000 lbs.) [1]. 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 cover the following six heat treatment conditions: ESR with three S/A temperatures 1050 °C, 1100 °C, and 1150°C, and ESR+HOMO with the same three S/A temperatures. The results such as rupture times obtained on these plates can be used for property screening along with those to be obtained by INL on AOD with the same S/A temperatures. It is worthwhile noting that, because data on small size heats of Alloy 709 are available, the results obtained can be compared with the previous data. The short-term creep tests for property screening in this report can also serve as a part of verification of instruments for the refurbished creep frames.

The shakedown tests on SS316H for the upgraded creep frames and the Alloy 709 creep rupture tests on the refurbished frames are described in this report. All the work performed in this project followed either the ASME NQA-1-2008 requirements or ASTM E139-11.

2. SHAKEDOWN TESTS ON SS316H FOR REFURBISHED AND UPGRADED FRAMES

2.1 MATERIAL AND TEST CONDITIONS

SS 316H was used in the shakedown creep tests. This material serves as a reference material for the Alloy 709 code case project. The chemical composition is given in Table 1 according to Tech Steel & Materials (heat #101076, Holbrook, NY).

Table 1. Chemical composition of SS 316H, wt%

C	**P**	**Si**	**Ni**	**Cu**	**N**	**Ti**	**Sn**	**V**	**Mn**	**S**	**Cr**
.045	.028	.650	10.120	.353	.053	.002	.006	.060	1.420	.024	16.230
Co	**Mo**	**Nb**	**Al**	**B**	**Fe**						
.279	2.090	.014	.004	.004	Balance						

It was planned to test SS 316H at 816° C under three stress levels, and tests will be discontinued when the accumulated creep strain reaches 1%. As a result, the expected time for the test interruption would be different. The load strain is monitored and recorded as well. The test matrix is shown in Table 2.

Table 2. Test matrix of shakedown tests

Material	**Test #**	**Temp, (ºF)**	**Temp, (ºC)**	**Diameter, Inches**	**Diameter, mm**	**Stress, MPa**	**Stress, ksi**	**Load, lbs**	**Load, N**
SS 316H	1	1500	816	0.25	6.35	27	3.916	192.2	854.9
SS 316H	2	1500	816	0.25	6.35	24	3.481	170.9	760.2
SS 316H	3	1500	816	0.25	6.35	18	2.611	128.2	570.3

2.2 SPECIMENS

The shakedown on the upgraded frames using SS 316H was performed by using a specimen design whose gage diameter is 0.25″ (6.35 mm), and gage length 1.25″ (31.75 mm). The engineering drawing of specimen is shown in Fig. 1. The three specimens were prepared from 1″ (25.40 mm) diameter bar stock.

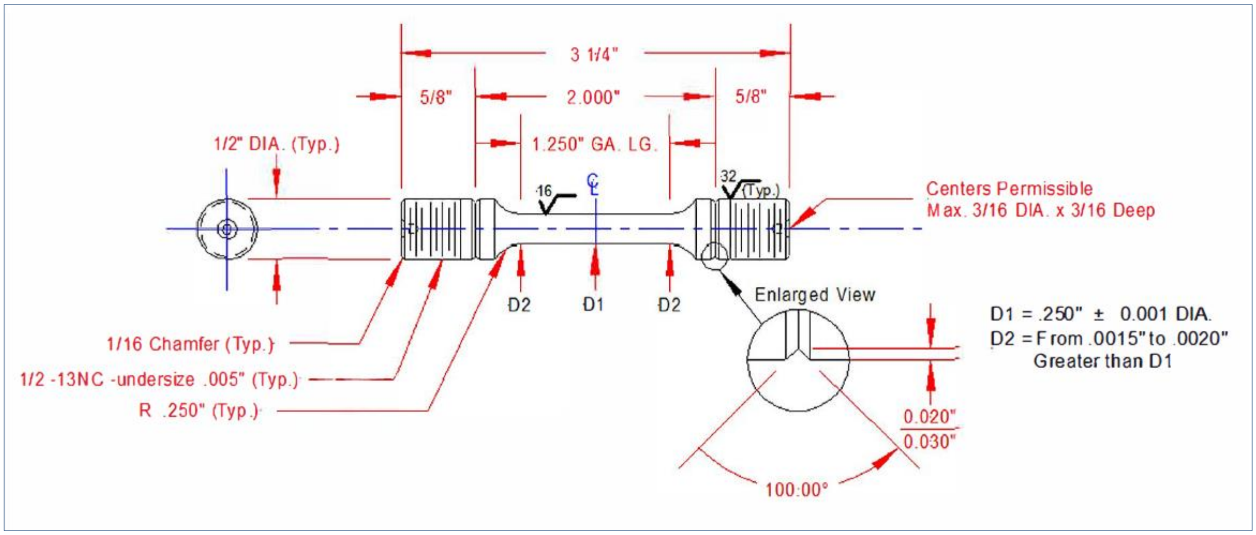


Fig. 1. Specimen drawing used in machining of SS 316H specimen.

2.3 FRAME PREPARATION AND TEST BUILD-UP

Upgraded frames #78, #80, and #81 were identified for this subtask. The following preparations were made in order for the frames to be ready for the tests:

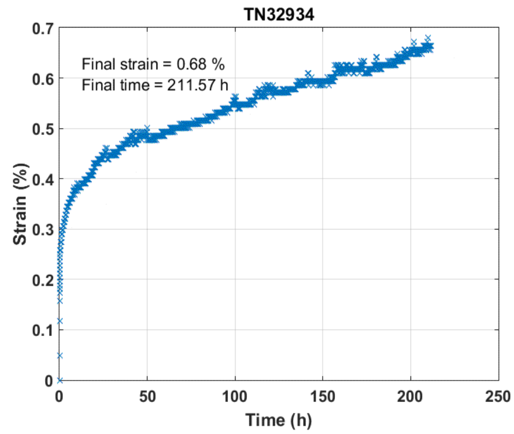
1) Change-over of thermocouples (TCs) from Type K to Type S was made on these frames to accommodate the requirement of testing three 316H specimens at 816° C. Nine Type S TCs were fabricated and calibrated for the requested tests.
2) The data acquisition (DAQ) system and the LabVIEW program (installed in the previous upgrade stage) were modified accordingly. Particularly, new TC wires were installed, and a new NI module SCXI-1102B dedicated to Type S TCs was added to the NI chassis.
3) Temperature controllers and limits were calibrated. In addition, the load cells and LVDTs were calibrated; these instruments were overdue for calibrations but had not been re-calibrated because the frames were being used for tests in the previous stage of the project.

The attachment of TC on the specimen surface caused a malfunction of the temperature limit. This issue was eventually resolved after a number of cycles of debugging. At the same time, the load cell on frame #81 was discovered to be working abnormally with a very low reading in load cell channels wired to frames #81 and #80. It has been found out that the inline amplifier in the load cell was defective and that load cell replaced. Shown in Fig. 2 are frames #80 and #81 with tests ongoing on SS 316H.

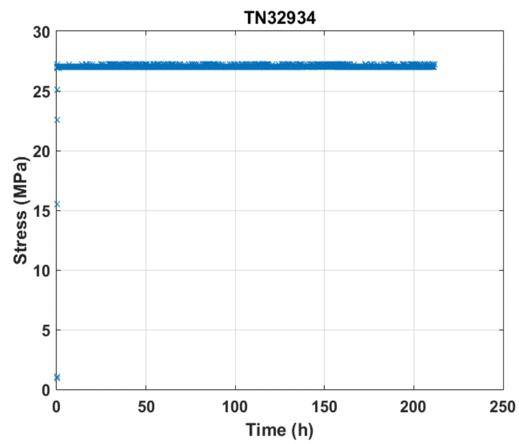
Fig. 2. Tests on 316H are ongoing on frames #78 (not shown), #80 and #81 with three Type S TCs installed in each frame.

2.4 TEST RESULTS

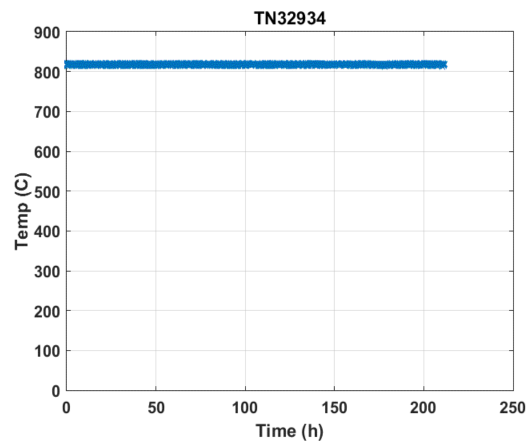
A set of test data can be seen in Fig. 3 for the testing condition under 27 MPa, 816° C, including input stress and temperature and output strain (loading strain included).



(a)



(b)



(c)

Fig. 3. Time series of (a) strain, (b) stress, and (c) temperature for tests on SS 316H, TN32934; loading stage is included.

The creep curve for this test is given in Fig. 4. It can be seen that the creep entered into the secondary stage of creep around 50h, and now is still in the secondary stage. A minimum creep rate (MCR) can be obtained using linear curve-fitting into the linear segment of creep curve and that is shown to be 1.10E-03 %/h. The final strain is the strain reached when the data were reported.

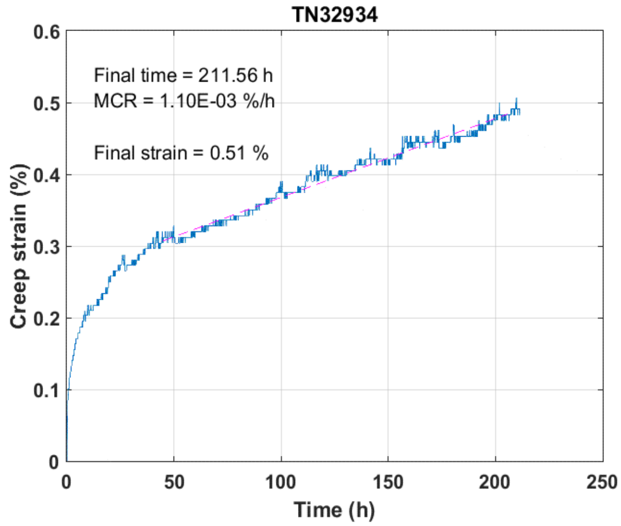


Fig. 4. Creep curve of TN 32934 under 27 MPa, 816° C.

The LVDT results for the test on frame #78 are shown to be very noisy. However, a steady increase in strain can still be detected in the secondary stage. The creep tests are summarized in Table 3, including loading strains, creep strains at the accumulated hours, and MCRs. At a specified time, the creep strain increases with the increasing applied stress, and so does the loading strain or MCR. As expected, the maximum creep strain occurs under stress level 27 MPa, but it is still less than 1%.

Table 3. Summary of creep tests on SS 316H*

TN	Mech.	Spec. #	Temp, C	Stress, MPa	Load strain, %	Time, h	Creep strain, %	MCR, %/h	Status
32934	81	SS-316-001	816	27	0.17	212	0.51	1.10E-03	Ongoing
32935	80	SS-316-002	816	24	0.10	211	0.17	3.83E-04	Ongoing
32936	78	SS-316-003	816	18	0.04	212	0.10	8.71E-05	Ongoing

*As of 8/22/2017

3. CREEP RUPTURE TESTS ON ALLOY 709

3.1 MATERIAL AND TEST CONDITIONS

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 Alloy 709 melt of approximately 45,000 lbs. in weight was used to produce three slab ingots and two round ingots. The slabs were in the AOD, ESR and ESR + HOMO conditions, respectively, and the round ingots were in ESR and ESR + HOMO conditions. The AOD slab ingot was hot-rolled into a 1.2-inch thick plate and the ESR and ESR + HOMO ingots were hot-rolled into 1.1-inch thick plates. Each as-rolled plate was sectioned into three equal sections and they were solution annealed at 1050, 1100 and 1150ºC, respectively. The dimensions of the nine solution annealed plates are given in Table 4. The mechanical properties data obtained from room-temperature tests and microstructure screening results were reported in [1].

Table 4. Dimensions of nine solution-annealed plates.

Melt cond.	Heat no.	Homo.	S/A, F	S/A, C	Lot Mark	ORNL lot ID	Dimensions (inch)	Dimension (mm)
AOD	58776-4	No	1922	1050	S/A 1922 DEG	58776-4-A1	1.2×60×74.1	30×1524×1882
			2012	1100	S/A 2012 DEG	58776-4-B1	1.2×60×74.8	30×1524×1900
			2102	1150	S/A 2102 DEG	58776-4-C1	1.2×60×75	30×1524×1905
ESR	58776-3R	No	1922	1050	ESR S/A 1922 DEG	58776-3RB-A1	1.1×62×43.3	28×1575×1100
			2012	1100	ESR S/A 2012 DEG	58776-3RB-B1	1.1×62×49.5	28×1575×1257
			2102	1150	ESR S/A 2102 DEG	58776-3RB-C1	1.1×62×50	28×1575×1270
ESR+ HOMO	58776-3R	Yes	1922	1050	ESR HOMO S/A 1922 DEG	58776-3RA-A1	1.1×46×47.5	28×1168×1207
			2012	1100	ESR HOMO S/A 2012 DEG	58776-3RA-B1	1.1×60×47.9	28×1524×1217
			2102	1150	ESR HOMO S/A 2102 DEG	58776-3RA-C1	1.1×60×47.8	28×1524×1214

A small section was water-jet cut from each plate listed in Table 4. A scoping test matrix consisting of two creep rupture tests for each plate shown in Table 4, all with the same test condition of 600ºC and 330 MPa, was developed. Six tests for the AOD condition are to be conducted at the INL and the twelve tests for the ESR and ESR + HOMO conditions are to be tested at the ORNL and discussed in this report. The test condition matches with some of the tests previously performed at ORNL from small-sized heats.

The sub-heat tested by ORNL has a designation of #58776-3R and the chemical composition is shown in Table 5.

Table 5. 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								

3.2 SPECIMENS

The specimen design was based on 3/8" (9.53 mm) gage diameter whose engineering drawing of the specimen is shown in Fig. 5.

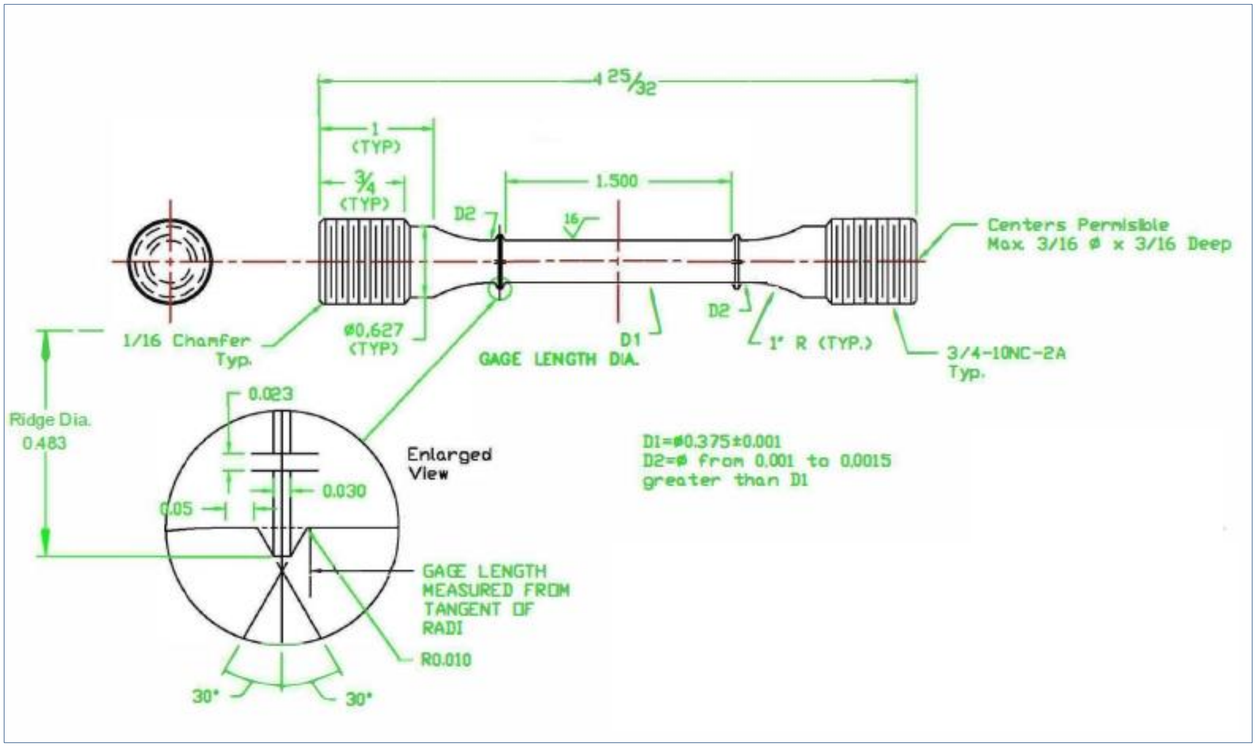


Fig. 5. Drawing used in machining Alloy 709 specimen (dimensions are in inches).

The loading axis of a creep specimen was aligned with the rolling direction of the plate. The machining of 12 specimens was conducted in Machine Shop, Fabrication Complex at ORNL. Table 6 lists the melt and annealing temperatures of the 12 specimens to be used in the creep rupture tests at ORNL (Sect. 3.1).

Table 6. Melt and annealing temperatures of Alloy 709 specimens to be tested at ORNL.

No	Melt	S/A, C	Lot Mark	GOC Lot ID	ORNL ID	Qty
1	ESR	1050	ESR S/A 1922 DEG	58776-3RB-A1	76-3RBA1-S20301	2
2	ESR	1100	ESR S/A 2012 DEG	58776-3RB-B1	76-3RBB1-S20301	2
3	ESR	1150	ESR S/A 2102 DEG	58776-3RB-C1	76-3RBC1-S20301	2
4	ESR+HOMO	1050	ESR HOMO S/A 1922 DEG	58776-3RA-A1	76-3RAA1-S20301	2
5	ESR+HOMO	1100	ESR HOMO S/A 2012 DEG	58776-3RA-B1	76-3RAB1-S20301	2
6	ESR+HOMO	1150	ESR HOMO S/A 2102 DEG	58776-3RA-C1	76-3RAC1-S20301	2

3.3 FRAME PREPARATION AND TEST BUILD-UP

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 equipped with a 10,000-lb load cell (#5 - #8 and #88 - #90) were used because they have the load capacity required. Preparation mainly involved the calibration of lever arm ratio and baking-out of furnace for the initial use of a new furnace.

The plan is to test seven specimens first, and then move to the remaining five whenever any of the seven tests is completed. For the seven tests in start-up, one specimen was picked from each of heat treatment conditions #1 through #6, and then repeat #1 (Table 6).

Images in Fig. 6 show the tests that were built up and are ongoing on frames #5 - #8 and #88 - #90.

(a)

(b)

Fig. 6. Tests are ongoing on (a) frames #5 - #8 and (b) #88 - #90.

3.4 TEST RESULTS

A typical set of strain, stress, and temperature curves is given in Fig. 7 for specimen 3RBA1-001, while the creep curve for this test is given in Fig. 8. It can be observed the creep seems still in the primary stage.

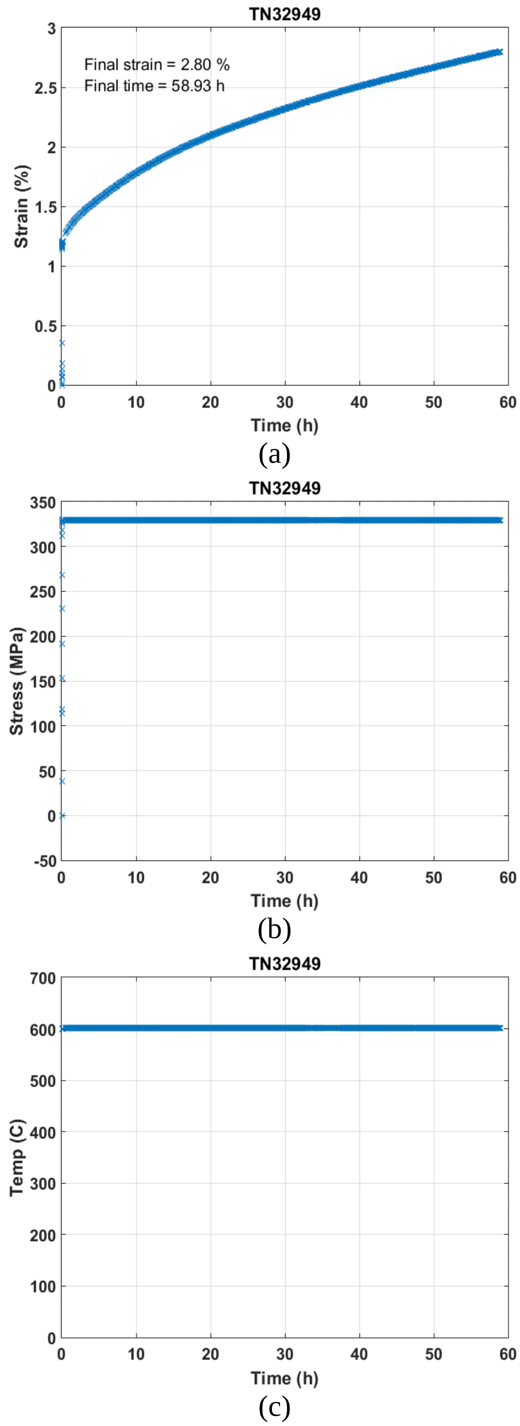


Fig. 7. Typical time series of (a) strain, (b) stress, and (c) temperature for 3RBA1-001, 330 MPa and 600° C; loading stage is included.

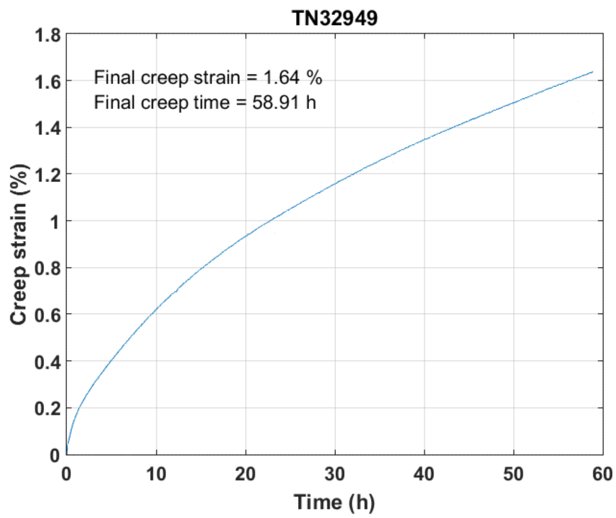


Fig. 8. Creep curve of 3RBA1-001, 330 MPa and 600° C.

Results for all the seven tests are summarized in Table 7. It was learned that one test was loaded with 300 MPa (one of the dead weights was not loaded up), which was a little less than the designed stress. No MCR data can be extracted at the time of reporting; given a creep time (for example, 60 hrs.), the creep strains tend to decrease with the increasing solution annealing temperature.

Table 7. Summary of Alloy 709 test conditions and results*

TN	Mach	Spec #	Melt	S/A	Temp, C	Stress, MPa	Load strain, %	Time, hrs	Creep strain, %	Status
32949	88	3RBA1-001	ESR	1050	600	330	1.16	59	1.64	Ongoing
32948	89	3RBA1-002	ESR	1050	600	330	0.9	59	1.37	Ongoing
32946	5	3RBB1-001	ESR	1100	600	330	3.65	64	0.44	Ongoing
32947	90	3RBC1-001	ESR	1150	600	330	4.97	64	0.42	Ongoing
32943	8	3RAA1-001	ESR+HOMO	1050	600	330	1.6	62	1.75	Ongoing
32944	7	3RAB1-001	ESR+HOMO	1100	600	300	3.57	62	0.31	Ongoing
32945	6	3RAC1-001	ESR+HOMO	1150	600	330	6.78	64	0.38	Ongoing

*as of 8/22/2017

It can be seen that the loading strain (Table 7) increases with the solution annealing temperature for ESR with and without homogenization. This observation is similar to tensile tests results [1]. It has been shown that increasing annealing temperature enhances ductility (Table 8). Note that the tensile tests were conducted at room temperature. Now it has been demonstrated that same behavior occurs at elevated temperature such as 600° C.

Table 8. Tensile properties at room temperature [1]

Melt	S/A, DEG C	YS, MPa	UTS, MPa	Elongation, %
ESR	1050	372.3	740.5	41.6
ESR	1100	356.7	732.7	45.6
ESR	1150	287.1	664.4	48.9
ESR+HOMO	1050	486.1	802.6	32.6
ESR+HOMO	1100	390.2	761.9	43.1
ESR+HOMO	1150	334.4	725.8	44.4

4. SUMMARY

Shakedown tests were conducted by using SS 316H under various stress levels at 816° C. As expected, strain and MCR increase with applied stress. All three tests are ongoing; even for the test under stress 27 MPa, the creep strain is still less than 1%.

Alloy 709 creep rupture tests have been initiated. The tests were conducted under stress 330 MPa at temperature 600° C. Seven tests were started with all the heat treatment conditions. The creep curves resulting from these tests exhibit a primary creep stage with decreasing creep rate; at the time of the completion of this report, the MCR has not been reached yet. Finally, the effect of solution annealing on the loading strains at the elevated temperature agrees with the room temperature tensile results. The experimental results confirmed that all the refurbished and upgraded creep frames would work as designed.

5. REFERENCES

[1] K. Natesan, X. Zhang, T.-L. Sham, and H. Wang, *Report on the completion of the procurement of the first heat of Alloy 709*, ANL-ART-89, Argonne National Laboratory, Lemont, IL, June 2017.