Creep Behavior of MA957 and 14YWT (SM10 heat) - D.T. Hoelzer[1], J.P. Shingledecker[2], R.L. Klueh[3], E.T. Manneschmidt[1] and M.A. Sokolov[1] ([1]Oak Ridge National Laboratory; [2]Electric Power Research Institute and [3]ORNL, Retired)

OBJECTIVE

The objective of this work is to expand the analysis of the creep data obtained from creep tests of MA957 that was reported previously and to show results from the first set of creep tests on the advanced ODS ferritic alloy 14YWT-SM10. The 14YWT-SM10 alloy was a 1.2 kg heat produced at ORNL in July, 2007.

SUMMARY

This study reports the data obtained from creep tests for MA957 that were started in 2003 and the results obtained from the initial creep tests for 14YWT-SM10. The creep tests of the as-received MA957 showed typical three-stage creep behavior at 875ºC and above for stress of 70 and 100 MPa. However, the specimens failed in less than 1700 h. Abnormal creep behavior consisting of essentially no primary or tertiary creep was exhibited at 800ºC and 100 MPa. However, this specimen failed after 38,555 h with a total strain just prior to failure of only 0.361% and a very low minimum creep rate of ~1.2×10^{-11} s^{-1} ($d\dot{\varepsilon}/dt$).

Creep results for 14YWT-SM10 at 800ºC and stresses of 100 to 300 MPa are the first such data for this material. They show abnormal creep behavior at 200 to 300 MPa, with essentially no primary or tertiary creep. This was attributed to the high stress levels, more than 50% of the yield strength. The creep test at 100 MPa has now accumulated close to 20,000 h without failure.

PROGRESS AND STATUS

Background

A set of thermal creep tests were conducted on as-received MA957 starting in August 2003 shortly after discovering a high number density of Ti-, Y-, and O-enriched nanoclusters that were similar to those that had been discovered in 12YWT by APT in 1999 [1-3]. Some of the results obtained from creep tests conducted on six samples of MA957 at temperatures between 800ºC and 925ºC and constant loads of 70 or 100 MPa were reported previously [4]. However, did not include much analysis of the data and focused on the stability of the NC and microstructure of the failed MA957 specimen that was tested at 800ºC and 100 MPa. In addition, creep tests have now been initiated on the 14YWT-SM10 advanced ODS ferritic alloy produced at ORNL to obtain creep properties and to study creep deformation mechanisms at elevated temperatures and stresses. The initial creep tests were conducted at 800ºC with stresses of 100, 200, 250 and 300 MPa.

Advanced oxide dispersion strengthened (ODS) ferritic alloys, also referred to as nanostructured ferritic alloys, contain a high concentration of Ti-, Y-, and O-enriched nanoclusters (NC). The dispersion of NC was first discovered in the 12YWT ferritic alloy that was developed in Japan during the late 1990's and subsequently in MA957 during the FY02-05 I-NERI project between ORNL, plus UCSB subcontract, and CEA, Saclay, in France [1,2,5]. Interestingly, MA957 was patented by INCO in 1978 [6]. Unfortunately, 12YWT was produced only once in a relatively small heat and INCO discontinued production of MA957 shortly after it was introduced commercially in the 1980's. However, these developments plus the scientific interest that arose from the early studies of NC in 12YWT and MA957 prompted the development of 14YWT starting in early 2001 and continuing to the present date.

At ORNL, the focus for developing the 14YWT ferritic alloy was to produce the high concentration of NC and to reduce the grain size to a few hundred nanometers. The much smaller grain size achieved in producing 14YWT is the most significant microstructural feature that distinguishes it from 12YWT and MA957. A total of 14 small heats (<1.2 kg) of 14YWT have been produced using Ar gas atomized powders procured from Crucible Research (now ATI Powder Metals) and Special Metals in 2001. In

2007, the 14YWT-SM10 heat, which was a 1.2 kg extrusion, was produced with ball milled powders from Special Metals. The larger size of this heat permitted numerous types and quantities of specimens to be prepared in order to obtain tensile, fracture toughness and thermal creep properties and to study high temperature deformation mechanisms. A number of TEM, DCT and tensile specimens were also prepared from 14YWT-SM10 for the Phenix Matrix II neutron irradiation experiment conducted by DOE (LANL) and CEA, Saclay in the GNEP program.

One of the most important properties associated with high-temperature alloys being developed for applications in advanced nuclear reactors, including both fission and fusion reactor systems, is creep performance. Shortly after NC were discovered in the as-received MA957, a total of 6 creep tests were initiated in August of 2003 to compare the creep properties of MA957 with those of 12YWT, which had been obtained during the SEED and subsequent LDRD projects from 1999 to 2002 at ORNL. Creep testing was initiated on 14YWT-SM10 in 2008 since this became a milestone in the INERI project between ORNL and CEA, Saclay that was started in 2007. All of the creep tests on MA957 and 14YWT were conducted in air at temperatures ranging from 800ºC to 925ºC with stresses of either 70 or 100 MPa. The main objective of this report is to expand the analysis of the creep data obtained from the creep tests on MA957 that was reported previously [4] and to show results from the first set of creep tests on 14YWT-SM10.

Experimental Procedure

The sample of MA957 that used in the creep tests was provided to ORNL by CEA, Saclay in August, 2002. The sample was a thick walled tube that was 10 cm long with an external diameter of 6.5 cm and wall thickness of ~0.9 cm. The fabrication history for producing the tube is not known. A total of 7 creep specimens were machined from the tube wall section with the gage parallel to the length of the tube. The specimens were 3.0 in. long and had cylindrical gages, which were 0.8 in. long and 0.199 in. diameter. A total of 6 time-to-failure constant load creep tests were started in August 2003.

The processing and fabrication history of the 14YWT-SM10 plate used in the creep tests consisted of: (1) annealing the vacuum degassed can containing the ball milled powder for 1 h at 850ºC and subsequently extruding the can through a rectangular shaped die that had an opening of 1.5 in wide x 0.75 in high; (2) annealing 5 plates cut from the extruded bar for 1 h at 1000ºC in vacuum; (3) hot rolling the 5 plates to 40% reduction in thickness at 850ºC; (4) machining the creep specimens from 2 of the plates and (5) annealing the creep specimens for 1 h at 1000ºC in vacuum. Two types of creep specimens were prepared from the plates. Two 3.0 in long cylindrical specimens were prepared with threaded grips, extensometer grooves and gage that was 0.8 in long and 0.198 in diameter. One of these specimens was used in the initial creep test that was started in April, 2008 using a constant load equal to 100 MPa at 800ºC. Three 1.65 in long cylindrical specimens were prepared with threaded grips, extensometer grooves and gage that was 0.4 in long and 0.113 in diameter. These specimens were tested with an MTS unit in constant stress control using stresses of 300, 250 and 200 MPa at 800ºC.

The specimen codes and the temperatures and stresses used in the creep tests on MA957 and 14YWT-SM10 are shown in Table 1. All of the creep tests were conducted in air.

Results and Discussion

The results of the creep tests performed on the MA957 and 14YWT specimens are shown in Table 2. The results for MA957 were shown previously, but were included in this report for comparison with the new results for 14YWT. The results for MA957 showed that failure of the specimens occurred in shorter times in creep tests conducted at 875ºC and higher compared to much longer times to failure below this temperature. To emphasize this trend, the specimen being tested at 825ºC and 70 MPa is still in progress and has now been exposed to these conditions for more than 60,000 h, or close to 7 years. The

results for 14YWT showed that the time to failure decreased significantly with increasing stress at 800ºC. At the lowest stress, i.e. 100 MPa load, the test is still in progress after more than 19,000 h. However, increasing the stress to 200 MPa (constant stress control test) caused failure of the specimen in 102 h and the 300 MPa stress caused specimen failure after ~10 minutes. Although these are very short times to cause specimen failure, it should be mentioned that these stress levels are very high relative to the yield strength of 14YWT-SM10 at 800ºC. Tensile tests have been conducted on 14YWT-SM10 and have shown that the yield (0.2% offset) and ultimate tensile strengths are $\sigma_{0.2}$ = 395 MPa and σ_{UTS} = 429 MPa, respectively, at 800ºC. Thus, the creep tests conducted with stresses of 200, 250 and 300 MPa were more than 50% of the yield strength. The most extreme creep test used 300 MPa, which was 76% of the yield strength, which partially accounts for the rather short time of ~10 minutes to failure.

Table 1. The conditions used in the creep tests of MA957 and 14YWT.

Alloy	Specimen Code	Type of Creep Test	Stress (MPa)	Temperature (ºC)
MA957	ODS-01	Load	100	900
MA957	ODS-02	Load	70	925
MA957	ODS-03	Load	70	875
MA957	ODS-04	Load	100	800
MA957	ODS-05	Load	Not Tested	
MA957	ODS-06	Load	70	825
MA957	ODS-07	Load	100	875
14YWT	NFA00	Load	100	800
14YWT	NFA01	Load	Not Tested	
14YWT	NFA02	Constant Stress	250	800
14YWT	NFA03	Constant Stress	200	800
14YWT	NFA04	Constant Stress	300	800

Table 2. The time and strain to failure obtained from the creep tests of MA957 and 14YWT-SM10.

Alloy	Specimen Code	Time (h)	Strain at Failure (%)	Status
MA957	ODS-01	37	1.8	Ruptured
MA957	ODS-02	165	4.2	Ruptured
MA957	ODS-03	1640	3.4	Ruptured
MA957	ODS-04	38,555	6.5	Ruptured
MA957	ODS-06	60792*	N/A	In Test
MA957	ODS-07	42	6.6	Ruptured
MA957	NFA00	19776*	N/A	In Test
14YWT	NFA02	4.8	0.343	Ruptured
14YWT	NFA03	93.3	0.157	Ruptured
14YWT	NFA04	0.18	0.405	Ruptured

* Total time from start of test to July 27, 2010.

The Larson-Miller Parameters (LMP) calculated using the creep results obtained for MA957 and 14YWT are shown in Figure 1. This plot also shows the LMP for 12YWT and an advanced 9Cr-WMoVNb tempered martensitic steel (TMS) [7]. The 2 data points for MA957 at 650ºC were obtained from the

INCO patent [1]. The test temperature and the time-to-failure for all of the 12YWT data and a few of the MA957 data in Figure 2 to compare the creep test conditions. The linear line fit through the data demonstrates that the overall creep properties of 12YWT are slightly better than that of MA957, but both of these MA ferritic alloys show significant improvements over the 9Cr-WMoVNb TMS. The LMP data for 12YWT and MA957 are 4 to 6 orders of magnitude higher than that for the 9Cr-WMoVNb TMS at all stresses. The LMP allows a general comparison of the creep performance of these alloys since it does not provide information about the creep strain history, total strain at failure, or temperature of the creep test. This information is obtained from the creep curves.

Creep curves showing the dependence of strain with time for the MA957 specimens are shown in Figure 2. The creep curves are grouped by variations in temperature for each load, which was 70 MPa (Fig. 2a) and 100 MPa (Fig. 2b). The results showed very rapid transitions occurring in the creep strain from the initial primary creep regime to the secondary and tertiary creep regimes for tests that were conducted with high stresses and temperatures, i.e. 70 MPa at 925 ºC and 100 MPa at 875 and 900 ºC. These tests showed secondary, or steady state, creep over short durations that then ended with rapid transition to tertiary creep and subsequent failure. At lower stresses and temperatures, the tests exhibited much more extensive secondary creep regimes for the specimens. For example, the specimen that was tested at 800ºC and 100 MPa showed essentially no change in strain following the first 50 h of testing, which resulted in failure of the specimens tested at higher temperatures that are shown in Figure 2b.

Expanded creep curves of the specimen tested at 800ºC and 100 MPa are shown in Figure 3. The plot shown in Figure 3a shows the creep strain as a function of time with the data processed to 100 points due to the very large number of data points for this test. Interestingly, the results do not show evidence of primary creep and only a small tertiary creep regime. The small tertiary creep can only be observed when the data covers the last 1000 h prior to failure of the specimen as shown in Figure 3b. Tertiary creep occurs for ~300 h before the specimen experiences an increasing strain rate that leads to sudden failure. The results show that the data followed nearly steady state creep rate from the beginning of the test to ~38,165 h and that the vast majority of strain, i.e. ~6.14%, occurred in the last 0.1 h of the test. Thus, the extensometer creep strain prior to rupture of the specimen was ~0.361%, which corresponded to a displacement of ~0.003 in. for essentially the entire test duration of 38,555 h. The catastrophic failure mechanism is not clearly understood, but is an ongoing topic of research activities.

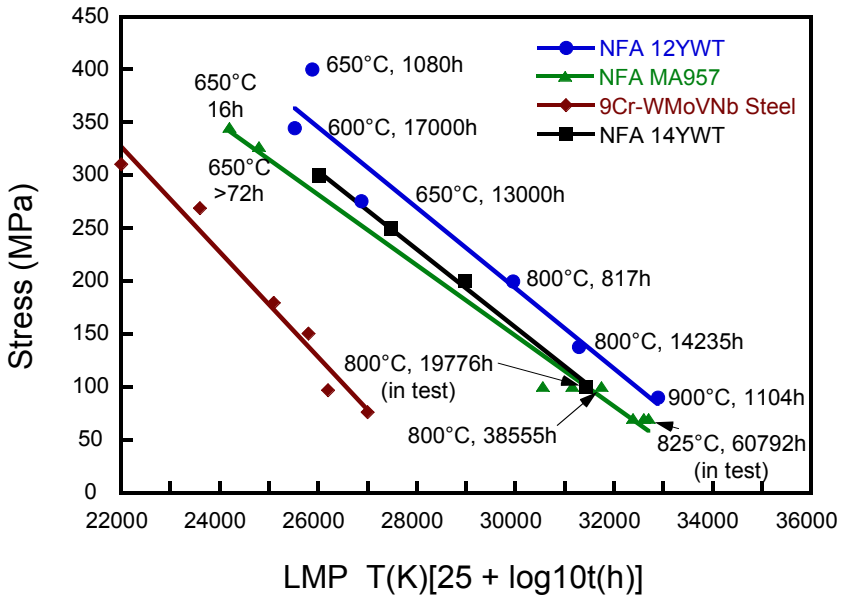

Figure 1. The Larson Miller Parameter plots of the new data for MA957 and 14YWT and those of MA957, 12YWT and 9Cr-WMoVNb TMS from previous studies [3,6,7].

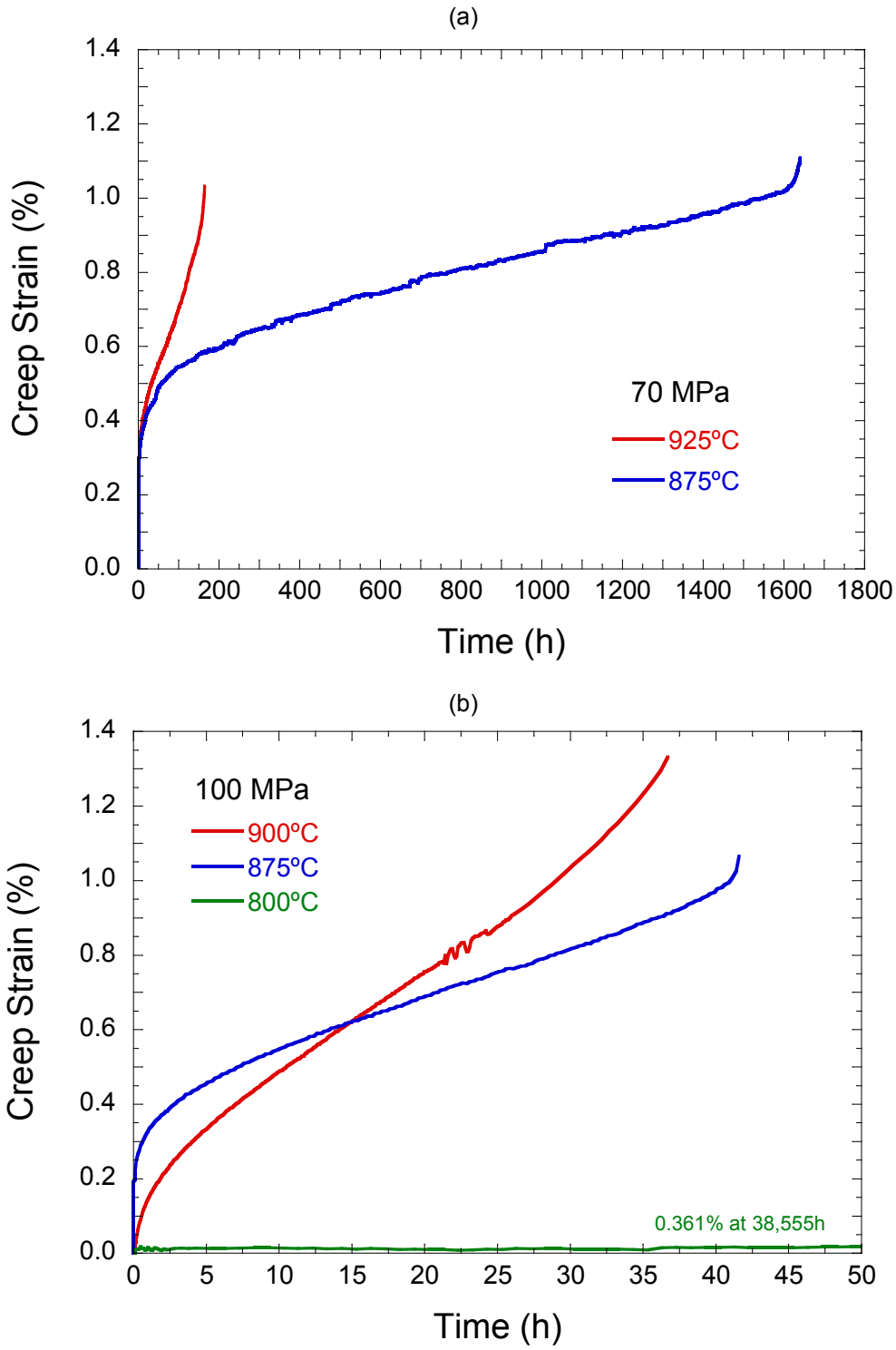

Figure 2. Creep curves of MA957 from tests conducted at elevated temperatures and with constant loads of: (a) 70 MPa and (b) 100 MPa.

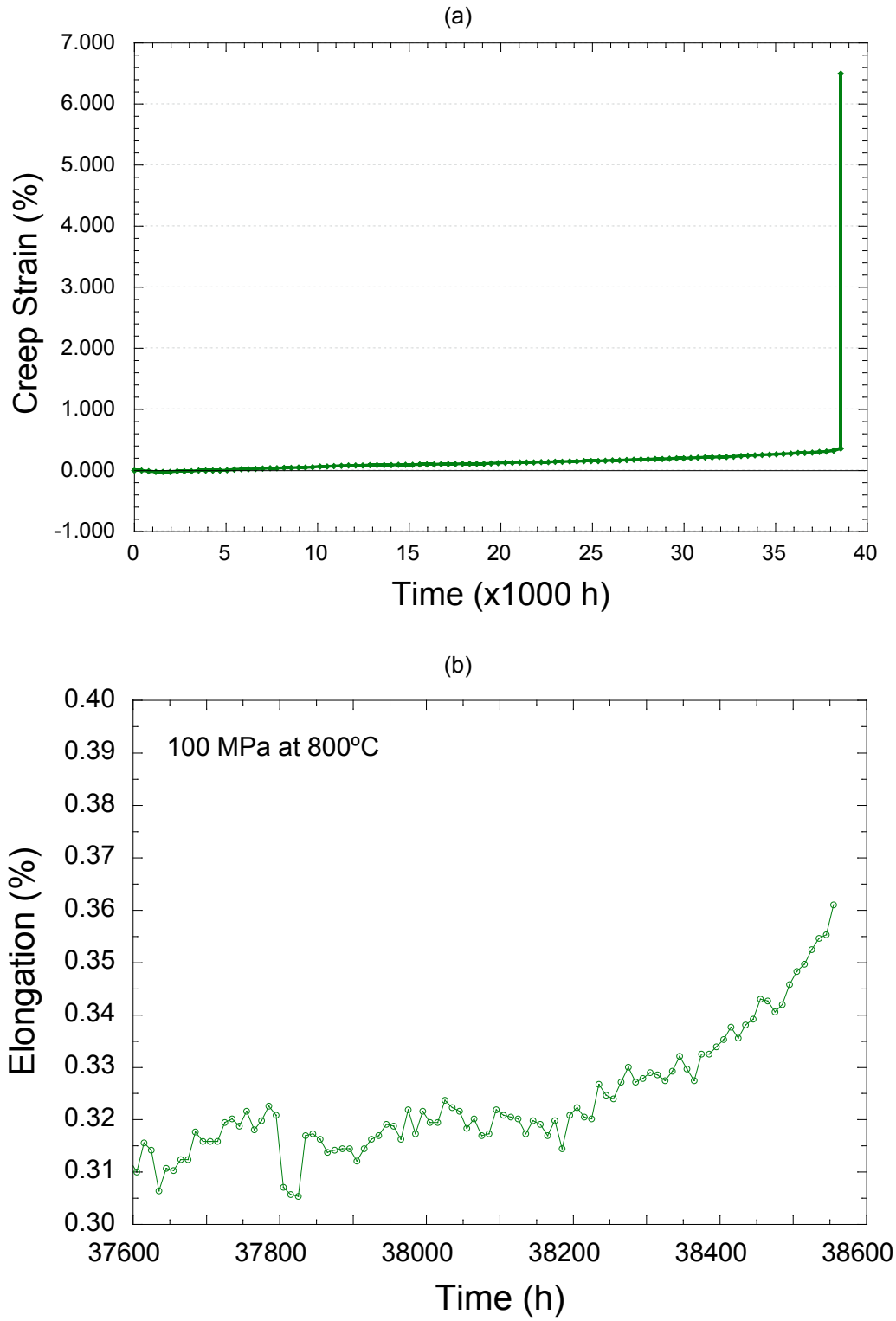

Figure 3. Creep curves of the MA957 specimen that failed after 38,555 h in the test the test that was conducted at 800ºC and 100 MPa in air. (a) Shows the creep curve covering the total length of the test and (b) shows the creep curve for the last 955 h prior to failure. The large number of data points has been reduced for the plots.

The creep curves showing the dependence of strain with time for specimens of 14YWT are presented in Figure 4. The results showed the strain increased rapidly during primary creep and transited to secondary creep in a short amount of time in the tests using 200, 250 and 300 MPa at 800ºC. The creep curve for the test at 200 MPa and 800ºC does not show the variation of strain with time during the first ~60 hours because of a problem that was encountered with Labview not storing the acquired data after the buffer became overloaded. However, screen images were saved and the results showed the same trend. All of the tests indicated that secondary creep occurred over short durations that then ended with subsequent failure with essentially no evidence of tertiary creep. In addition, all 3 tests resulted in failure of the specimens at low (<1%) levels of strain. As mentioned previously, the stresses used in these tests at 800 ºC were more than 50% of the yield strength for the 14YWT (SM10) heat and this contributed to the short time to failure experienced by the specimens. However, failure at low strains is unexpected. These characteristics of failure showing little to no tertiary creep and low strains are of major concern and will be the focus of further studies.

The minimum creep rates measured in secondary creep regime for tests on specimens of MA957 and 14YWT are shown in Table 3. With constant loads of 70 and 100 MPa, the creep results for MA957 indicated that the minimum creep rate (MCR) decreased as the test temperature was lowered. The lowest MCR value that was measured was for the test at 800ºC and 100 MPa, which was $d\dot{\varepsilon}/dt = 1.2 \times 10^{-11}\ s^{-1}$. For 14YWT, the MCR increased with stress at 800ºC. These trends are expected for typical creep behavior. For 14YWT, the results indicated that the minimum creep rate increased with increasing stress at 800ºC. The lowest MCR recorded for 14YWT was in the 200 MPa test, which was $3.2 \times 10^{-10}\ s^{-1}$ and close to an order of magnitude higher than the lowest MCR recorded for MA957 in the test using a load of 100 MPa at 800ºC.

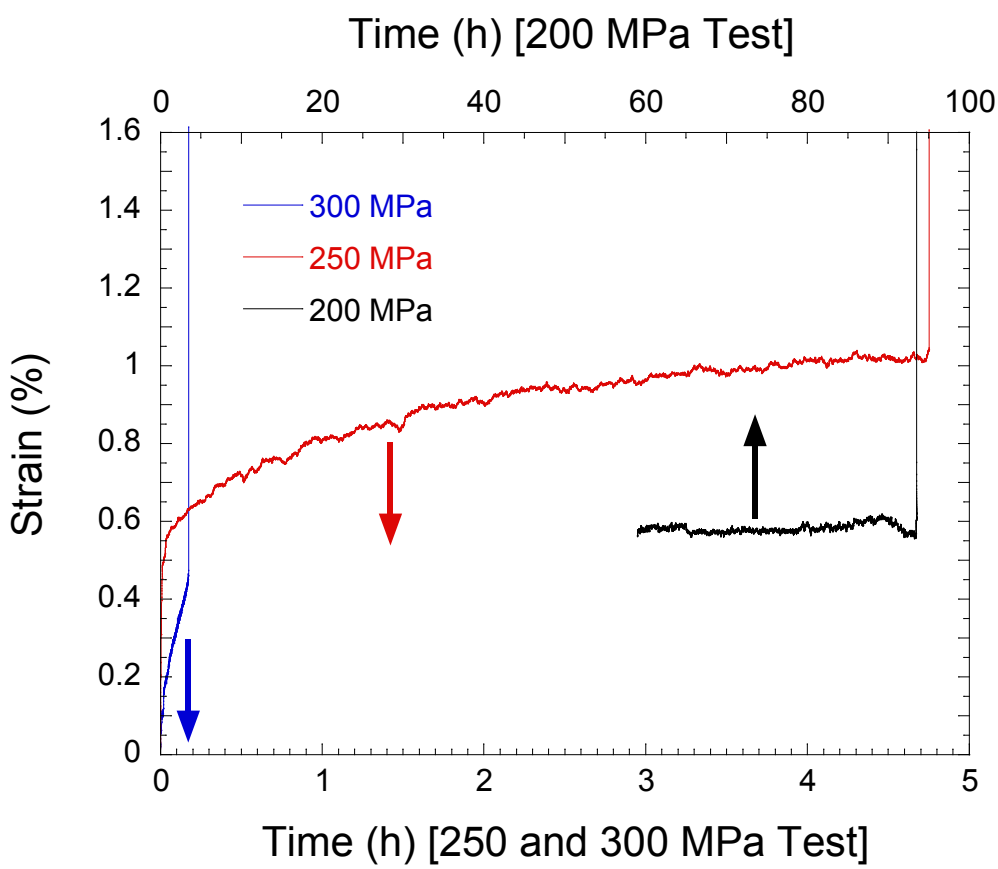

Figure 4. Creep curves of 14YWT from tests conducted at 800ºC with constant stresses of: 200, 250 and 300 MPa.

Table 3. The minimum creep rates measured from the creep results for MA957 and 14YWT.

Alloy	Specimen Code	MCR (s^{-1})
MA957	ODS-01	7.4×10^{-8}
MA957	ODS-02	8.8×10^{-9}
MA957	ODS-03	7.7×10^{-10}
MA957	ODS-04	1.2×10^{-11}
MA957	ODS-06	N/A
MA957	ODS-07	3.7×10^{-8}
14YWT	NFA00	N/A
14YWT	NFA02	2.0×10^{-7}
14YWT	NFA03	8.4×10^{-10}
14YWT	NFA04	6.4×10^{-6}

The dependences of stress with minimum creep rate obtained from creep tests for MA957 and 14YWT are shown in Figure 5. Data obtained from previous creep tests for 12YWT [2] and from a study by Wilshire and Lieu [8] for MA957 are also shown in this plot. The results show that the dependences of the minimum creep rate on stress at 800ºC for 14YWT can be described using the power law relationship for creep, which is of the form:

$$\dot{\varepsilon}_m = A\sigma^n \exp(-Q_c/RT)$$

where values of A, stress exponent (n) and activation energy for creep (Qc) typically vary with stress and temperature indicating different mechanisms are dominant. Thus, the linear fit to the values of MCR as a function of stress at 800ºC indicated that the stress exponent for 14YWT was ~24. This high value of stress exponent is consistent with high values of the stress exponent ($n \cong 35$) obtained for MA957 from creep tests at lower temperatures by Wilshire and Lieu [8]. These high values of stress exponent are commonly observed with dispersion strengthened alloys and suggest that the creep mechanism follows a threshold stress concept.

The data obtained for 12YWT cannot be plotted to obtain information about the stress exponent or activation energy for creep since these creep tests were not conducted using similar temperatures or stresses. Nevertheless, the limited results indicated that the creep performance of 12YWT was better than that obtained for MA957 by Wilshire and Lieu. For example, the MCR for 12YWT is similar to MA957 at 700ºC, but was tested with a stress that was ~70 MPa higher. However, there could be a number or reasons to explain this comparison such as differences in mechanical properties of MA957 related to different processing and fabrication histories between the remnants of material that were originally produced by INCO [6]. The specimens of MA957 tested at ORNL were from a sample that was provided by CEA, Saclay during a previous INERI project. This version of MA957 seemed to have very good creep performance as noted with the exceptionally low value of creep strain and minimum creep rate at 800ºC and 100 MPa.

Future Work

The creep testing of 14YWT will continue in the near future since a new heat, 14YWT-SM11, was recently produced. Specimens are being prepared. The research plan will be to perform creep tests at different temperatures using stresses that will allow the activation energy for creep to be obtained that will help elucidate creep mechanisms.

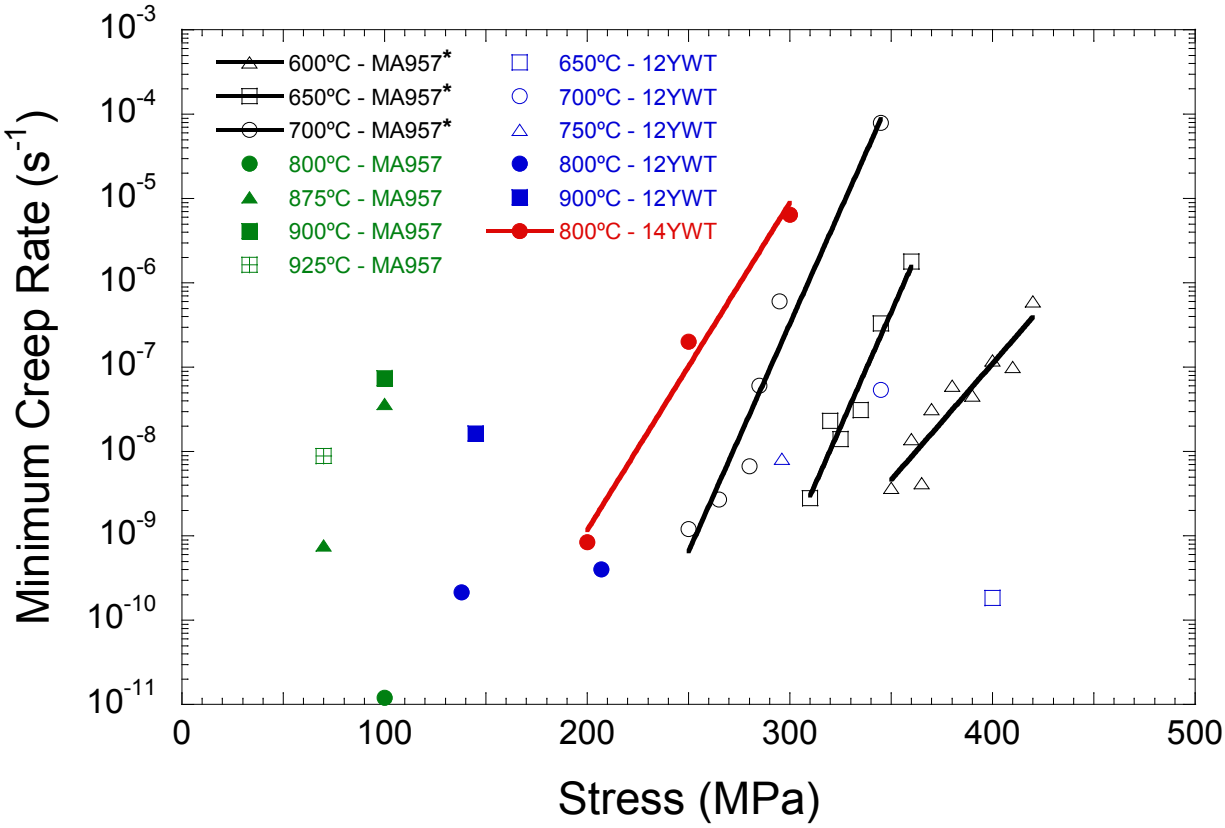

Figure 5. Shows the stress dependence of the minimum creep rates obtained from creep tests at high temperatures for MA957 and 14YWT. Also shown is data that was obtained from previous creep tests for 12YWT [3]. The * denotes data that was obtained from creep tests for MA957 from reference [8].

Conclusions

The results of the creep tests conducted on specimens prepared from the as-received MA957 showed:

- Typical creep behavior consisting of three regimes denoted by primary, secondary and tertiary creep was observed at temperatures of 875ºC and above for test loads of 70 and 100 MPa. However, the specimens failed in short amounts of time (<1700 h) in these creep tests.
- Abnormal creep behavior consisting of essentially no primary and tertiary creep was exhibited by the specimen that was tested at 800ºC and 100 MPa. However, this specimen failed after 38,555 h with a total strain just prior to failure of only 0.361% and a very low minimum creep rate of ~1.2 x 10^{-11} s^{-1} ($d\dot{\varepsilon}/dt$).

The creep results for 14YWT-SM10 at 800ºC and stresses of 100, 200, 250 and 300 MPa are the first such data for this material and showed:

- The creep tests conducted with constant stresses of 200, 250 and 300 MPa exhibited abnormal creep behavior showing essentially no primary and tertiary creep. The specimens in these tests showed low values of time-to-failure and sudden failure of the specimen at low strains. These characteristics of creep behavior were attributed to the high stress levels, which were more than 50% the yield strength of this heat.
- The creep test conducted at 100 MPa has now accumulated a total time of close to 20,000 h without failure.
- The stress exponent for creep at 800ºC and stresses greater than 200 MPa was determined to be ~24, which is consistent with the threshold stress concept.

Acknowledgements

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