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High-Temperature Tensile and Creep Test Results on Thin Wall Tube Specimens of ODS Alloys 14YWT and OFRAC

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May 2022

OAK RIDGE
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Materials Science and Technology Division

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ABSTRACT

High-temperature tensile and strain rate jump (SRJ) creep tests were conducted on axial specimens fabricated from thin wall tubes of the oxide dispersion strengthened (ODS) alloys 14YWT and OFRAC. The dimensions of the 14YWT thin wall tube was 10.7 mm OD, 0.5 mm WT and 0.92 m and of the OFRAC thin wall tube was 8.5 mm OD + 0.5 mm WT and 1.78 m in length. The axial specimens fabricated from the thin wall tubes were based on a dual-gauge design with gauge dimensions of 4 mm long and 2 mm wide. The tensile tests were conducted in air at 800ºC, 900ºC and 1,000ºC and the SRJ creep tests were conducted in air at 800ºC. Two axial specimens were used in each tensile and SRJ creep tests. The results of the tensile tests showed much lower yield stress (YS) compared to ultimate tensile strength (UTS) indicative of large work hardening properties. The results showed the UTS of OFRAC was higher than that of 14YWT by ~35 MPa at 800ºC. At 900ºC, the UTS of OFRAC and 14YWT was similar at 900ºC and at 1000ºC, the UTS of 14YWT was higher than that of OFRAC by ~10 MPa. The ductility properties of OFRAC were greater than that of 14YWT with higher values of uniform elongation (UE) and total elongation (TE) at all tensile test temperatures. The creep properties of 14YWT and OFRAC were evaluated by the strain rate jump test method at 800ºC. The stress exponent (n) for creep was obtained from the SRJ test data by plotting the values of the log strain rate against the log stress, which is measured from the stress-strain curve generated during the SRJ test. The results showed the highest stress exponents of n = 19.8 and n = 35.0 were measured from the two SRJ tests on 14YWT. The stress exponent measured from one successful SRJ test on OFRAC was n = 13.3, which was lower than that of 14YWT. Nevertheless, these high values of stress exponent are consistent with ODS ferritic alloys such as 14YWT and OFRAC since they indicate dislocation-particle interactions are the dominant creep mechanism for specific ranges of temperature and stress. The results obtained from the tensile and SRJ creep tests conducted on axial specimens in this study indicate that 14YWT and OFRAC retain high strengths at temperatures up to 1,000ºC and good creep performance at 800ºC.

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1. INTRODUCTION

The nanostructured ferritic alloys (NFA) 14YWT and OFRAC are candidates for fuel cladding in future fast reactors. Although the high strength of these NFA's at low temperatures presents significant challenges for fabricating thin wall tubing, recent efforts using cold pilger rolling and cold high precision tube rolling (HPTR) successfully demonstrated that thin wall tubing can be fabricated from 14YWT and OFRAC. Recently, a thin wall tube of 14YWT with dimensions of 10.7 mm OD, 0.5 mm WT and 0.92 m in length was fabricated by cold pilger rolling campaign in collaboration with CEA, Saclay, France. Two recent campaigns in collaboration with Nippon Nuclear Fuel Development (NFD) Co., LTD, Japan, resulted in the fabrication of a thin wall tube of OFRAC that was 8.5 mm OD + 0.5 mm WT and 1.78 m in length by cold pilger rolling and most recently in 2020, four thin wall tubes (6 mm OD + 0.5 mm WT and >2.2 m in length) were fabricated by HPTR. The successful fabrication of thin wall tubes from NFA 14YWT and OFRAC may allow for other applications such as heat pipes in micro-reactors.

The mechanical properties of 14YWT and OFRAC have been acquired from numerous tensile and creep tests conducted over the past 20 years at Oak Ridge National Laboratory (ORNL). The report ORNL/LTR-2021/1910 "Summary of Previous Mechanical Test Data on ODS Alloys 14YWT and OFRAC up to 1,000ºC" summarized the previously obtained tensile and creep data on 14YWT and OFRAC [1]. However, since the fabrication of thin wall tubes from 14YWT and OFRAC were achieved recently, only the tensile properties of specimens fabricated from the thin wall tube have been obtained at room temperature [2]. The tensile properties were obtained using a dual gauge axial specimen design and corresponding specially designed grips. This report covers the tensile and thermal creep properties of the thin wall tubes of 14YWT and OFRAC using the same dual-gauge axial specimen design.

2. THIN WALL TUBE FABRICATION

The ODS 14YWT and OFRAC were both produced at ORNL. The composition of each NFA was different, but similar mechanical alloying conditions were used for producing the rods for pilger fabrication into thin wall tubes. Table 1 shows the chemical composition of 14YWT and OFRAC in the as-extruded condition. The alloy powders of 14YWT and OFRAC were both produced by Ar gas atomization by ATI Powder Metals. The OFRAC powder produced by Ar gas atomization and 0.3 wt.% Y_2O_3 powder were blended together for mechanical alloying. For the 14YWT powder, ~ 0.21 wt.% Y was added to the melt consisting of nominal Fe-14Cr-3W-0.4Ti prior to producing the powder by Ar gas atomization. To elevate the O level in the 14YWT powder, ~0.35 wt.% FeO powder was added for mechanical alloying. The blended powder of 14YWT was ball milled using the CM100 Simoloyer at Zoz, GmbH, Germany, while the blended powder of OFRAC was ball milled using the CM08 Simoloyer at ORNL. Similar ball milling conditions were used for both NFA powders that consisted of varying rotor rotation speeds, 40 h duration and in Ar gas atmosphere. Following ball milling, the NFA powders were encapsulated in steel cans, degassed under vacuum for 24 h at 300ºC for OFRAC and 400ºC for 14YWT followed by extrusion at 850ºC through a circular die to produce the starting rods for pilger fabrication. Figure 1 shows the extruded rods of 14YWT and OFRAC. The extruded rod of 14YWT had a 2.8 cm outer diameter (OD) and 19.7 cm length (L) and the OFRAC rod had a 2.5 cm OD and 25 cm L.

Table 1. Chemical composition of 14YWT and OFRAC in weight percent (wt.%).

ODS	Fe	Cr	W	Mo	Ti	Nb	Y	O	C	N
14YWT	81.76	14.40	3.10	-	0.39	-	0.21	0.116	0.016	0.008
OFRAC	85.90	12.35	-	0.95	0.20	0.30	0.18	0.087	0.026	0.011

(a) (b)

Figure 1. The extruded rods of (a) 14YWT and (b) OFRAC.

2.1. 14YWT

The fabrication of the 14YWT thin wall tube was performed during 2018 in collaboration with CEA, Saclay, France. The extruded rod of 14YWT was the center mass was removed by gun drilling to produce the starting thick wall tube. Prior to the first pilger run, the thick wall tube was annealed in vacuum at 1200ºC for 3 h to reduce the initial hardness to acceptable levels by causing recovery and recrystallization processes in the microstructure of the 14YWT thick wall tube. The thin wall tube of 14YWT was produced with 4 cold (room temperature) pilger passes of 40% reductions each. Intermediate annealing treatments at 1200ºC for 1 h were applied to the tube after the first three pilger passes followed by an annealing at 750ºC for 1 h after the final pilger pass. The purpose of the 1200ºC annealing treatments was to reduce the deformation structure that was produced in the microstructure after each cold pilger pass by inducing recovery and recrystallization processes while that of the final 750ºC annealing treatment was for stress relief of the deformation structure. The dimensions of the final thin wall tube of 14YWT shown in Figure 2 was 10.73 mm OD with a wall thickness (WT) of 0.5 mm and length (L) of 915 mm.

Figure 2. Final thin wall tube of 14YWT produced by cold pilger at CEA, Saclay, France.

2.2. OFRAC

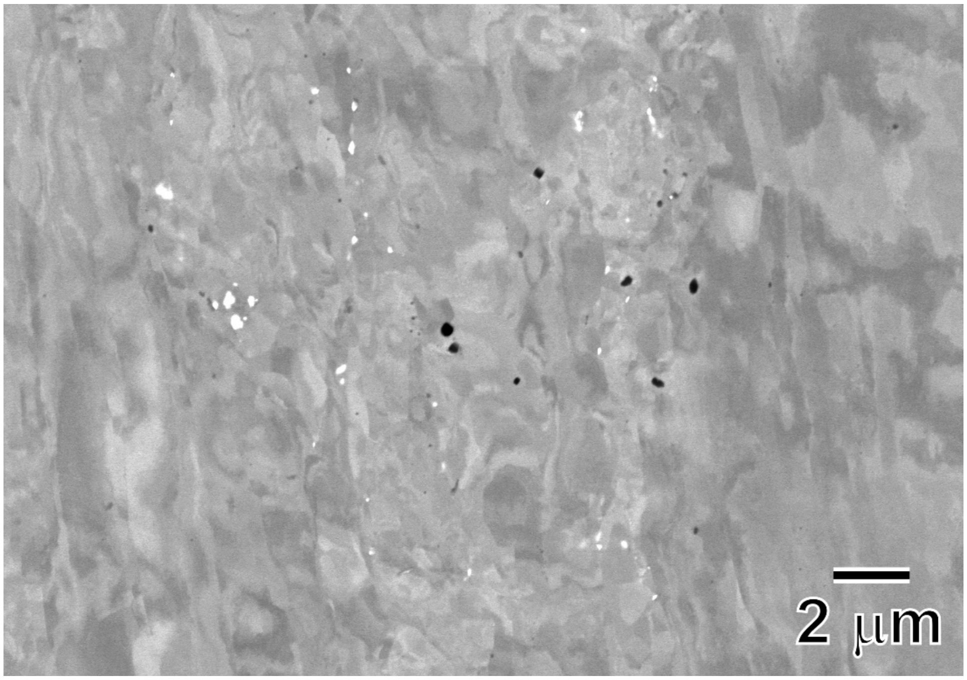
The fabrication of the OFRAC thin wall tube was performed during 2016-2017 in collaboration with Nippon Nuclear Fuel Development, Ltd, Japan. The center mass of the extruded rod of OFRAC was removed by gun drilling to produce the initial thick wall tube. The thick was tube was subjected to four cold pilger passes, with each pass resulting in 50% reduction in cross-section area. Following each pilger pass, an optimum annealing condition was determined using a small sample that was cut from the end of the tube and annealed at temperatures ranging from 700ºC to 1100ºC for 30 min followed by characterization using optical microscopy and micro-hardness measurements. The annealing conditions used between each pilger pass run were at temperatures that did not cause recrystallization or coarsening of the Y-, Ti- and O-enriched nanoclusters. The dimensions of the final thin wall tube of OFRAC shown in Figure 3 was 8.5 mm OD, 0.5 mm WT and 915 mm L.

Figure 3. Final thin wall tube of OFRAC produced by cold pilger at NFD, Japan.

2.3. MICROSTRUCTURE COMPARISON

The microstructures of the 14YWT and OFRAC thin wall tubes obtained by Scanning Electron Microscopy (SEM) with backscattered electrons (BSE) are shown in Figure 4. The grains in both tubes are elongated in the pilger rolling direction. The grains in the 14YWT tube (Figure 4a) are larger than those in the OFRAC tube (Figure 4b). The larger grains were formed since the intermediate pilger annealing temperature of 1200ºC to induce dislocation recovery and recrystallization processes. The final anneal at 750ºC was for residual stress reduction that also induced formation of deformation substructure within the large grains causing large amounts of intragranular orientation changes. The grains in the OFRAC tube are much smaller than the grains in the 14YWT tube and are highly aligned in the pilger rolling direction. This grain structure characteristics of the OFRAC tube was due to no heat treatment after the final pilger rolling pass.

(a)



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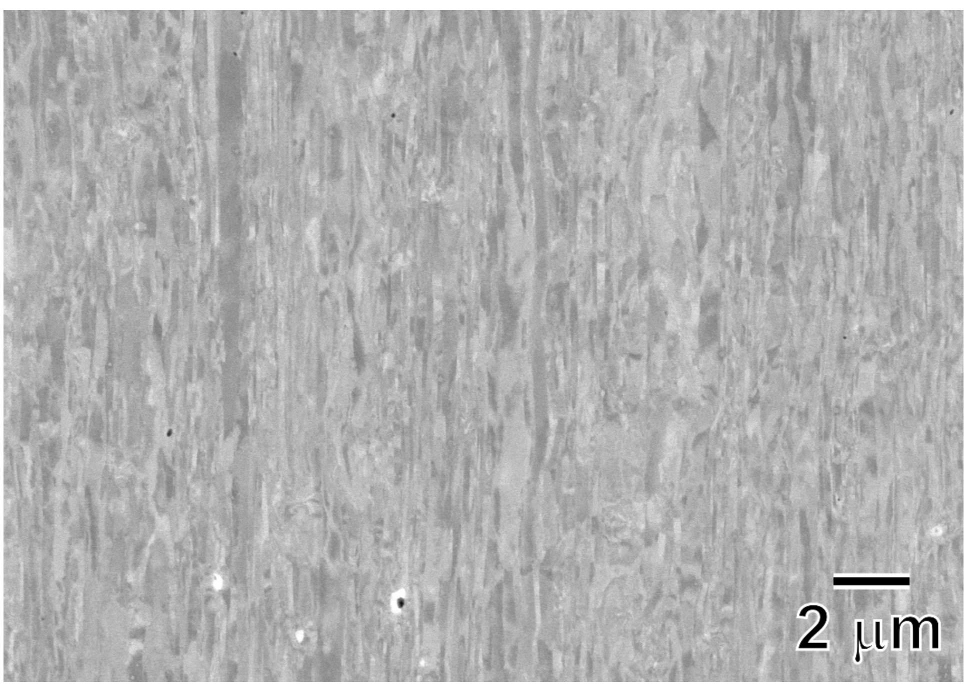


Figure 4. SEM with BSE imaging of the grain structures observed in the thin wall tubes of: (a) 14YWT and (b) OFRAC).

3. PROCEDURES

3.1 SPECIMEN PREPARATION

Axial specimens based on a dual-gauge design were fabricated from the thin wall tubes of 14YWT and OFRAC. Figure 5 shows the dimensions of the axial specimens at two orthogonal views. The dimension of the gauges for both axial specimens was 4 mm long and 2 mm wide with gauge head that was 4 mm long. The outside diameter of the 14YWT axial specimen was 10.73 mm and that of the OFRAC axial specimen was 8.50 mm. Therefore, the gauge widths have a slight curvature of 2πr. The axial specimens have 1 mm radius shoulders (R1) at the base of the gauge where it meets the gauge head. This design permitted shoulder loading during the tensile and strain rate jump tests that allowed for load-displacement curves. Figure 6 shows digital images of the fabricated axial specimens of 14YWT and OFRAC. A total of 8 axial specimens were fabricated from each thin wall tubes of 14YWT and OFRAC for conducting the tensile and strain rate jump creep tests at elevated temperatures.

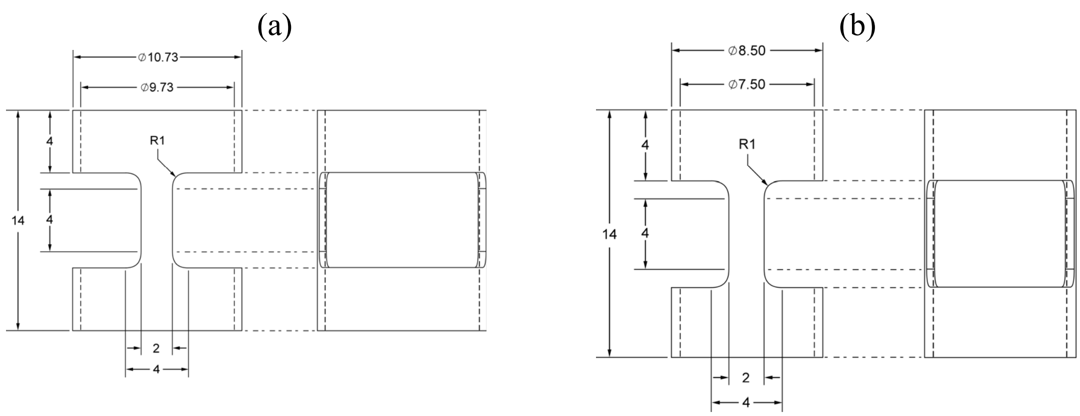


Figure 5. Dimensions of axial specimens fabricated from thin wall tubes of (a) 14YWT and (b) OFRAC. *Units in mm.*

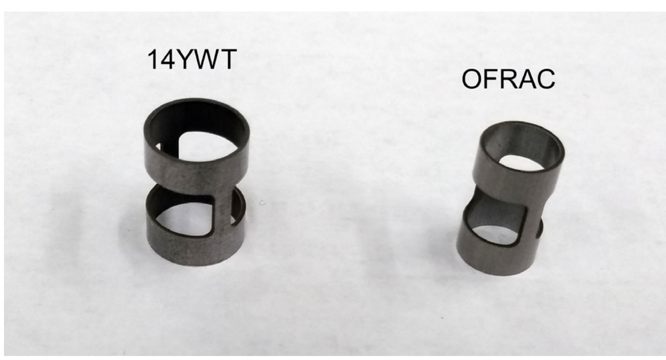


Figure 6. Image of the axial specimens fabricated from the thin wall tubes of 14YWT and OFRAC.

3.2 TENSILE AND STRAIN RATE JUMP TESTS

A drawing of the grips used for the axial specimens in the tensile and strain rate jump (SRJ) tests is shown in Figure 7 [1]. The grips were fabricated from Rene 11 with 1 mm radius rounded edges for shoulder loading the axial specimens of 14YWT and OFRAC. The tensile and SRJ tests were performed on an Instron 5900R screw driven tensile machine that was coupled with an induction furnace for heating the specimens to the test temperature. A thermocouple was spot welded to the gauge head of the axial specimen prior to the tensile and SRJ tests. A reference thermocouple was spot welded to the lower end of the upper grip. The heating rate of the induction furnace was rapid and typically reached the test temperature in <7 minutes. After reaching the test temperature, the temperature was held constant for 10 minutes followed by starting the tensile or SRJ test. All tests were conducted in air. The temperatures of the tensile tests were 800ºC, 900ºC and 1000ºC and the strain rate was 10^{-3} s^{-1}. In the SRJ tests, the start of the test is at the lowest attainable strain rate of the tensile machine and the corresponding stress is measured as a function strain. The stress increases with strain until it saturates and does not change with increasing strain. The strain rate is increased by one order of magnitude and the stress changes in response until it saturates and this procedure is then repeated until the final strain rate is reached that leads to specimen failure. The SRJ tests were conducted at 800ºC. Two axial specimens of 14YWT and OFRAC were tested at each of the three temperatures for the tensile tests and at 800ºC for the SRJ tests.

Figure 7. The specimen grips used for the axial tensile tests [1].

4. TENSILE TEST RESULTS

The stress-strain curves obtained from tensile tests of two axial tube specimens of 14YWT and OFRAC at 800ºC, 900ºC and 1000ºC are shown in Figures 8, 9 and 10, respectively. The results for all the tensile tests showed a much lower yield stress (YS) compared to the ultimate tensile strength (UTS), which indicates a large work hardening capacity. This deformation behavior was not observed for tensile tests conducted on 14YWT and OFRAC from room temperature to 800ºC using type SS-3 and SS-J3 flat tensile specimens. From the report ORNL/LTR-2021/1910, the stress-strain curves at 800ºC are shown in Figure 12 for 14YWT-NFA1 and Figure 17 for OFRAC. The stress-strain curves show a small increase in stress from YS to UTS and small uniform strain (UE). The flow stress decreases very gradually with increasing strain and shows a small total decease in stress at total elongation (TE) compared to UTS. In the tensile tests that were conducted at lower temperatures, there is a much larger decrease in stress with increasing strain after reaching plastic instability at UTS due necking in the specimen gauge.

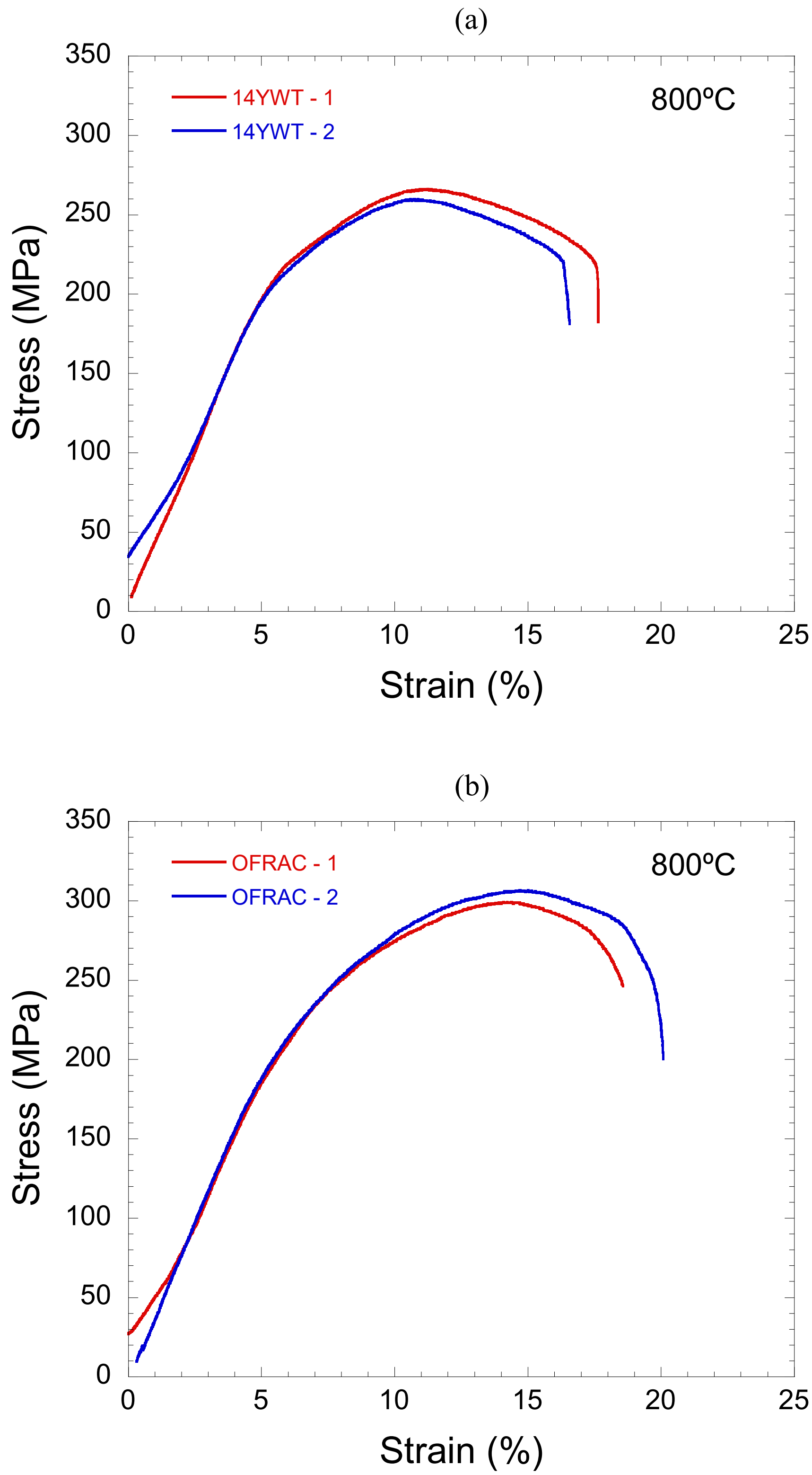
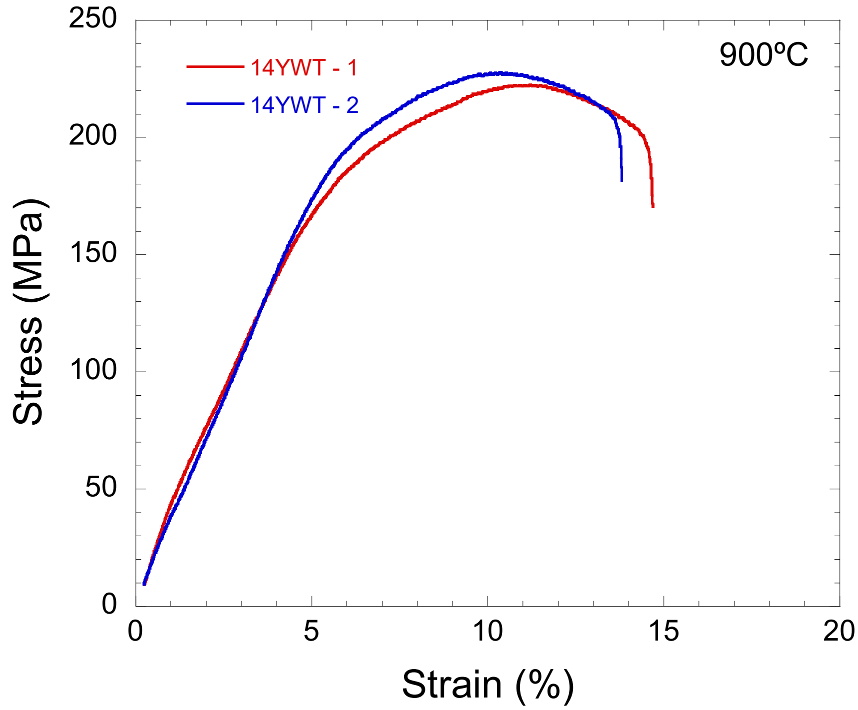


Figure 7. Stress-strain curves for the axial tube specimens tensile tested at 800ºC. (a) 14YWT and (b) OFRAC.

(a)



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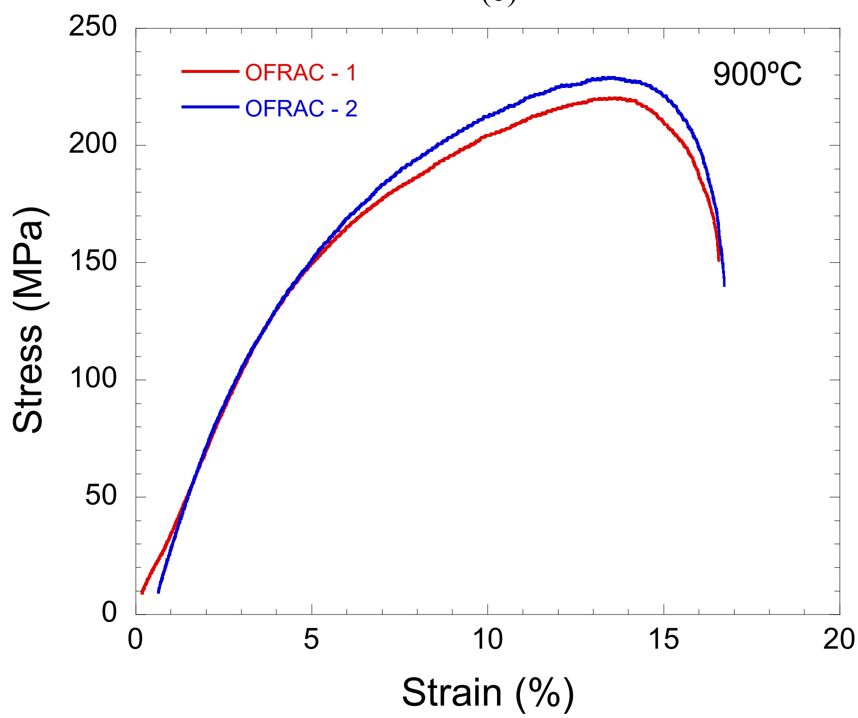
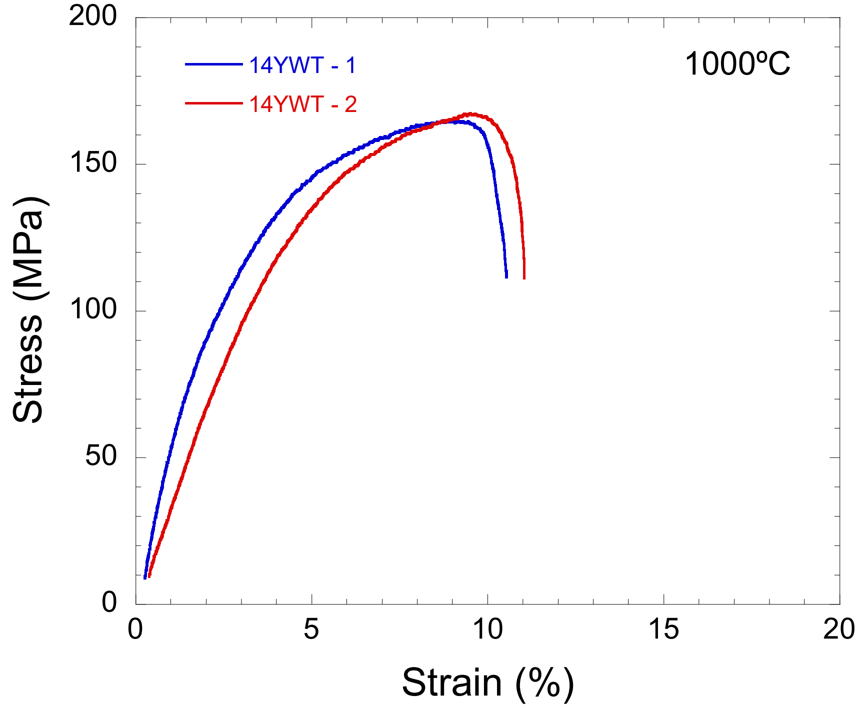


Figure 8. Stress-strain curves for the axial tube specimens tensile tested at 900ºC. (a) 14YWT and (b) OFRAC.

(a)



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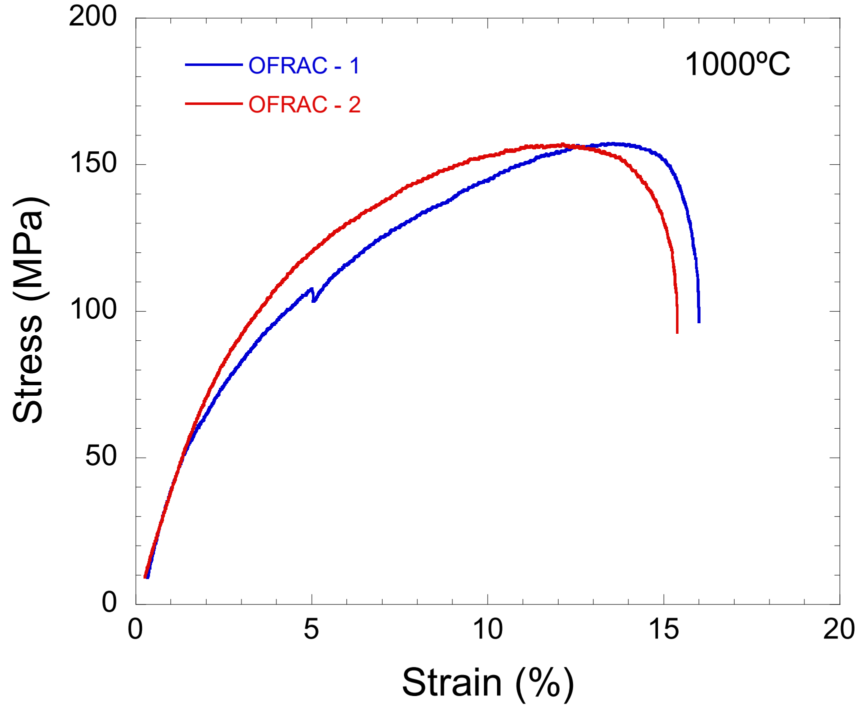

Figure 9. Stress-strain curves for the axial tube specimens tensile tested at 1000ºC. (a) 14YWT and (b) OFRAC.

The tensile properties (YS, UTS, UE and TE) measured from the stress-strain curves of the axial specimens of 14YWT and OFRAC at 800ºC, 900ºC and 1000ºC are shown in Table 1. The 0.2% proof stress was used for obtaining the YS values. In general, the YS of OFRAC was lower than that of 14YWT at all temperatures. The UTS of OFRAC was higher than that of 14YWT by ~35 MPa at 800ºC and were similar at 900ºC. However, at 1000ºC, the UTS of 14YWT was higher than that of OFRAC by ~10 MPa. This trend for UTS may be related to the different grain structures between 14YWT and OFRAC. The smaller grain size of OFRAC compared to 14YWT may have promoted a larger amount of grain boundary sliding that would cause plastic flow deformation at a lower stress. The ductility of OFRAC was greater than that of 14YWT with higher values of UE and TE at all temperatures.

Table 1. The measured tensile properties of two axial specimens of 14YWT and OFRAC tested at 800ºC, 900ºC and 1000ºC.

Specimen Number	Temp. (ºC)	14YWT				OFRAC			
		YS (MPa)	UTS (MPa)	UE (%)	TE (%)	YS (MPa)	UTS (MPa)	UE (%)	TE (%)
1	800	194	266	4.6	11.1	169	299	6.6	12.2
2	800	200	260	4.8	12.3	189	307	6.9	14.9
1	900	175	222	4.1	8.1	100	220	7.5	12.3
2	900	153	228	4.9	9.0	102	229	7.2	12.7
1	1000	74	165	7.0	8.7	58	157	9.8	13.5
2	1000	92	167	4.8	7.7	66	157	8.3	13.0

After completing each tensile test, it was observed that the circular shape of the axial specimens was deformed to an elliptical shape. Figure 10 shows digital images of the gauge heads following the tensile tests at 800ºC, 900ºC and 1000ºC on 14YWT and OFRAC. The results showed that the gauge heads of the OFRAC axial specimens were deformed to a greater extent compared to the 14YWT axial specimens. The maximum distortions occurred on opposite ends of the gauge head where the gauges are located. It is possible that the differences in diameter of the axial specimens for 14YWT and OFRAC may have contributed to the distortions. The loading characteristics on the axial specimens by the grips during the tensile test depends on the contact area between the gauge head and the surface of the grip. With the wall thickness of 0.5 mm, the larger outer diameter of 14YWT axial specimens (10.73 mm) compared to the OFRAC axial specimens (8.50 mm) indicates that a larger contact surface area exists between the gauge head and grip. The gauge width of 2 mm is the same for both axial specimens of 14YWT and OFRAC, which means that the OFRAC axial specimen possesses a smaller percent of the contact area between the surface of the gauge head and grip compared to the 14YWT axial specimens. During the tensile test at high temperatures, the stress concentration on the two gauges will exert forces on the contact surface area between the gauge head and grip, which may lead to stresses on the gauge heads by the grips. In this case, the difference in contact surface may have contributed to the larger distortions observed for the axial specimens of OFRAC compared to 14YWT.

(a) (b)

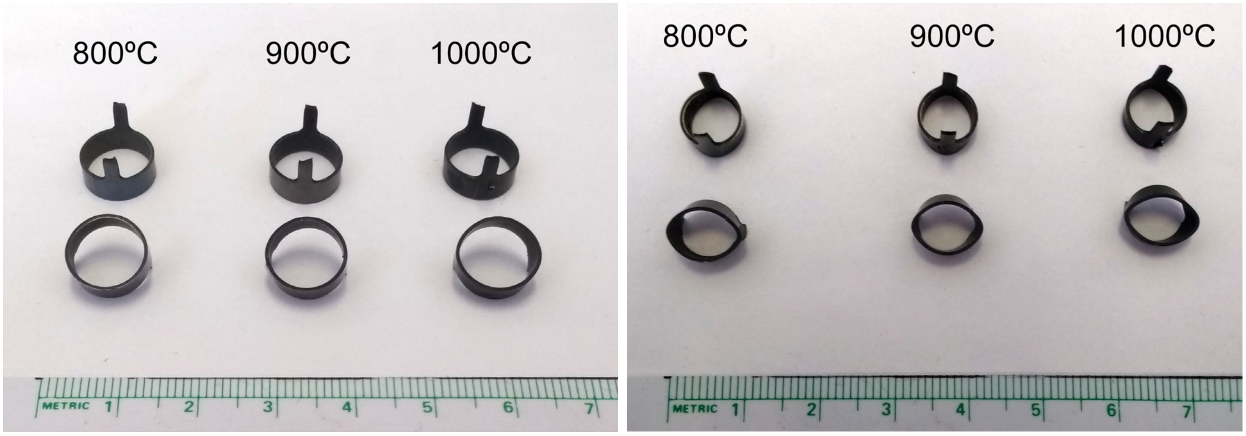

Figure 10. Digital images of the gauge sections of the axial specimens following the high temperature tensile tests. (a) 14YWT and (b) OFRAC.

5. STRAIN RATE JUMP CREEP TEST RESULTS

The stress-strain curve obtained from the SRJ test at 800ºC for the dual gauge axial specimen of OFRAC is shown in Figure 11. This curve illustrates the concept of the SRJ test. At the start of the SRJ test the stress increases very slowly at the lowest strain rate of 5.1×10^{-5} s^{-1} (first arrow) that the Instron screw driven test frame could obtain until yielding occurs and the stress becomes nearly constant with increasing strain. After confirming the region of constant stress, the strain rate was then increased to 1.8×10^{-4} s^{-1} (second arrow), which is accommodated by an increase in stress to the next yield stress followed by a region of constant stress. These steps are repeated two more times with increases in strain rates of 5.1×10^{-4} s^{-1} followed by 1.0×10^{-3} s^{-1}. After starting the last strain rate increase, the axial specimen quickly yields and reaches the ultimate tensile stress followed by failure. The two gauges associated with the axial specimen may deform differently, which contributed to the noise in the stress-strain curves.

The two plots shown in Figure 12 illustrate how the creep properties obtained from the SRJ test correlate with that from the time-to-failure constant stress creep test. Figure 12a shows the creep behavior of a specimen with constant stress. In primary creep (I), the strain rate increases rapidly and then slowly decreases as the specimen experiences hardening. The specimen exhibits secondary creep (II) when the strain rate reaches a steady state (SS). Tertiary creep (III) shows an acceleration in the strain rate that ends with failure of the specimen. Figure 12b shows the creep behavior of a specimen when constant strain rate. The primary creep (I) also shows a rapid increase in the strain rate that decreases due to hardening of the specimen. In secondary creep (II), the stress remains constant with increasing time. The stress then decreases with time until failure of the specimen occurs in tertiary creep (III). This type of creep behavior is observed in the strain rate jump test, except that the strain rate is increased prior to specimen failure that causes the stress to increase further with time until the next constant stress regime is reached.

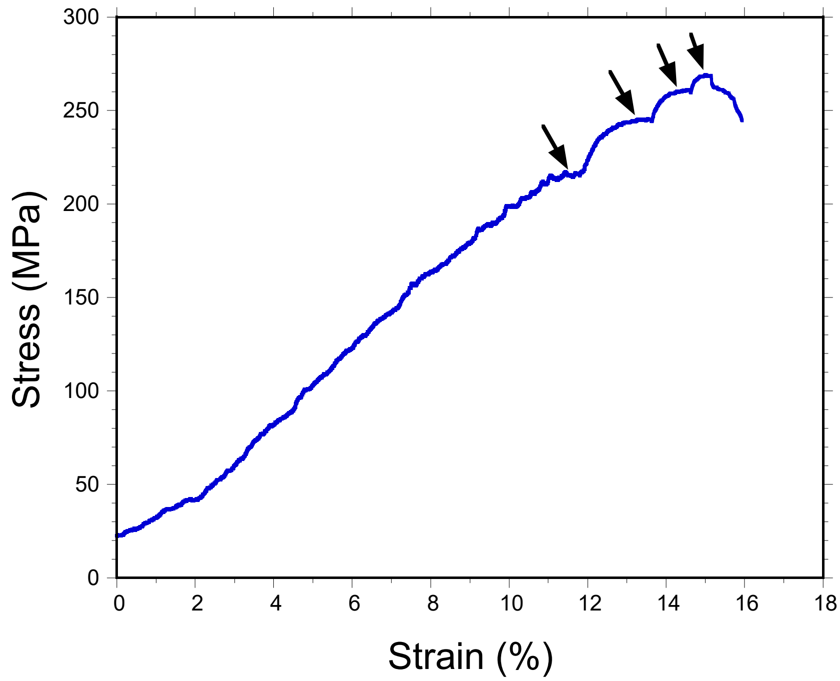

Figure 11. Stress-strain curve of SRJ creep test conducted on axial specimen of OFRAC at 800ºC.

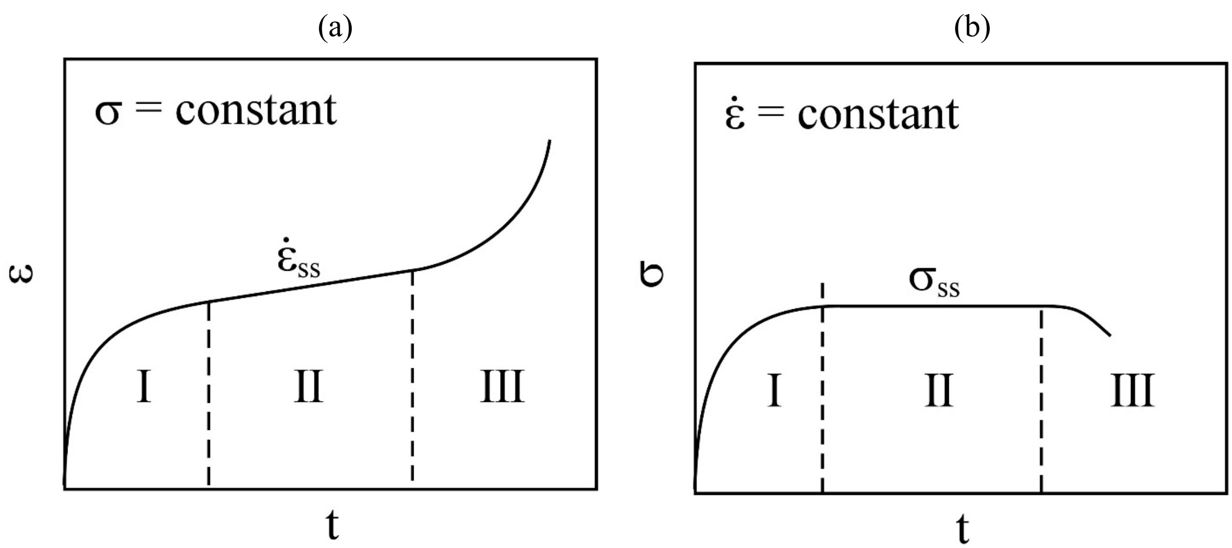

Figure 12. Plots illustrating the stress-strain curves for (a) creep tests using a constant stress and (b) creep tests using a constant strain rate.

The creep properties obtained from the SRJ tests on the axial specimens of 14YWT and OFRAC at 800ºC are shown in Figure 13. The values of the log minimum creep rate ($\dot{\varepsilon}_m$) stress, or strain rate used in the SRJ test, are plotted against the measured values of the log stress (σ). The stress exponent (n) is determined from the following relationship:

$$n = \left(\frac{log\dot{\varepsilon}_m}{log\sigma}\right)$$

The slope obtained from the linear fit to the log values gives stress exponent. The highest stress exponents of n = 19.8 and n = 35.0 were measured from the two SRJ tests on 14YWT. The first SRJ test performed on OFRAC experienced considerable noise and instabilities in the stress-strain curve that resulted in premature failure of the axial specimen. The second SRJ test on the axial specimen of OFRAC was successful, but yielded the lowest stress exponent of 13.3. The stress exponent provides information about creep mechanisms. For materials that are tested at high homologous temperatures, the typical values for *n* are ~1, which indicates that creep diffusion is the dominant mechanism. For oxide dispersion strengthened ferritic alloys such as 14YWT and OFRAC, the high values of *n* indicate threshold stress behavior that indicates dislocation-particle interactions are the dominant creep mechanism for specific ranges of temperature and stress.

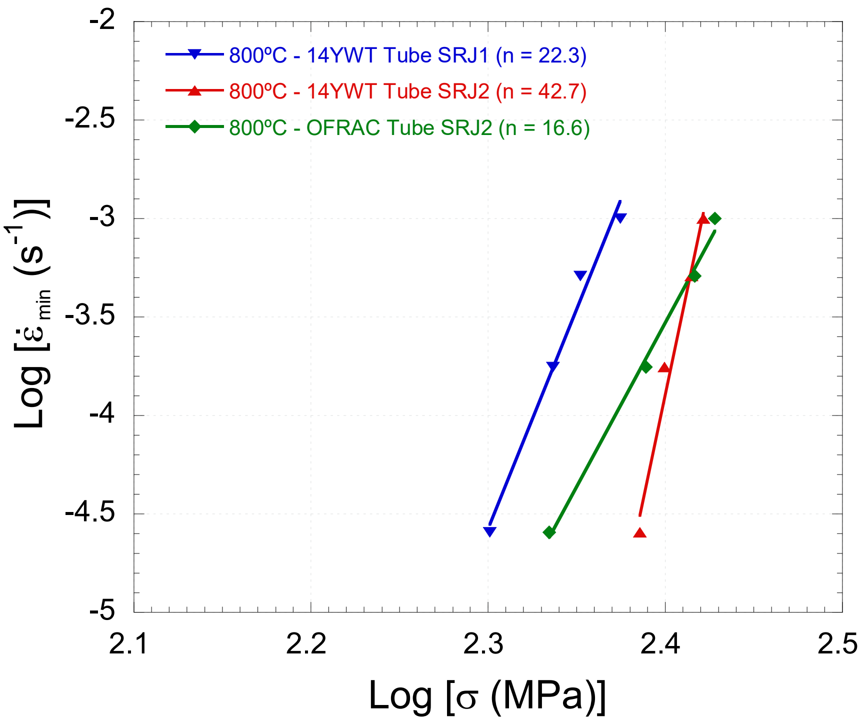

Figure 13. Comparison of the thermal creep behavior of 14YWT with OFRAC from SRJ tests conducted on axial specimens at 800ºC. The calculated stress exponents (n) are shown for each set of creep data.

The creep properties of OFRAC have been obtained from SRJ tests that were conducted on dual gauge axial specimens fabricated from the thin wall tube covered in this report and previously on SS-3 flat specimens fabricated from an extruded bar [3]. The plot in Figure 14 compares the SRJ test results between the axial specimen at 800ºC and the SS-3 specimens at 550ºC, 600ºC, 700ºC and 800ºC. The data for the axial specimens covers a smaller range of strain rates compared to that of the SS-3 specimens at the 4 temperatures. The reason for this is partly due to the smaller gauge length of the axial specimen compared to the SS-3 specimen, which are 4 mm and 7.62 mm, respectively. In addition, the crosshead of the servo-hydraulic driven MTS test frame used for the SS-3 specimens can achieve lower travel speeds than that of screw driven Instron test frame, resulting in lower strain rates during the SRJ tests. The results of the SRJ tests indicate that the calculated stress exponent of the data for the axial specimens is 13.3, which is lower than that for the SS-3 specimens at all the temperatures. Although there is no clear explanation for these differences, it is possible that the dual gauge design of the axial specimens may cause differences in the deformation behavior during creep in the SRJ tests compared to the singly gauge design of the SS-3 flat tensile specimens. The deferent grip designs may also have an effect on the deformation behavior during creep. More research on creep properties using axial tube specimens and further modifications to the specimens grips are needed.

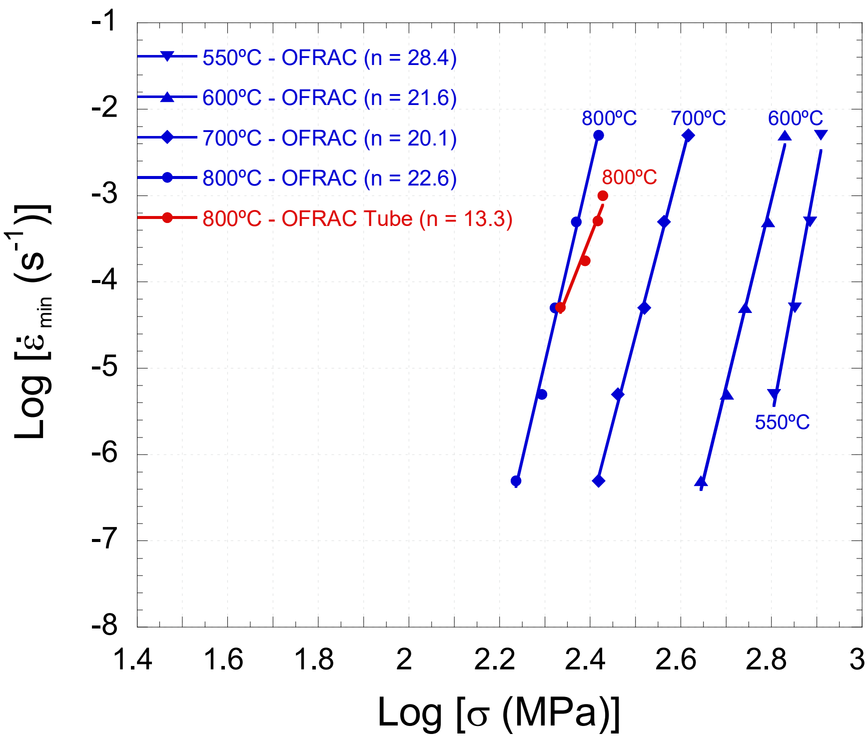

Figure 14. Comparison of the thermal creep behavior of OFRAC from SRJ tests conducted on the axial tube specimen at 800ºC and on the SS-3 flat tensile specimens at 550ºC. 600ºC, 700ºC and 800ºC [3]. The calculated stress exponents (n) are shown for each set of creep data.

SUMMARY

High-temperature tensile and strain rate jump (SRJ) creep tests were conducted on dual-gauge axial specimens fabricated from thin wall tubes of the ODS alloys 14YWT and OFRAC. Two axial specimens from both ODS alloys were used in tensile tests that were conducted at 800ºC, 900ºC and 1,000ºC and SRJ creep tests that were conducted at 800ºC. All tests were performed in air. In all the tensile tests, the results showed much lower yield stress than ultimate tensile strength, which is indicative of large work hardening properties. The uniform tensile strength of OFRAC was ~35 MPa higher than that of 14YWT at 800ºC. The results showed similar uniform tensile strengths for 14YWT and OFRAC at 900ºC. However, the uniform tensile strength of 14YWT was ~10 MPa higher than that of OFRAC at 1,000ºC. The ductility properties of OFRAC were greater than that of 14YWT with higher values of uniform elongation and total elongation at all tensile test temperatures. The creep properties of 14YWT and OFRAC were evaluated using the SRJ method at 800ºC. The regions of constant stress were measured after each change in strain rate so that the exponent (n) for creep could be obtained by plotting the values of log strain rate against the log stress. The results showed the highest stress exponents of n = 19.8 and n = 35.0 were measured from the two SRJ tests on 14YWT. The stress exponent measured from one successful SRJ test on OFRAC was n = 13.3, which was lower than that of 14YWT. These high values of stress exponent are consistent dislocation-particle interactions are the dominant creep mechanism over specific ranges of temperature and stress, which is commonly observed for ODS alloys. Thus, the results obtained in this study consisting of tensile and SRJ creep tests using axial specimens indicate that 14YWT and OFRAC retain high strengths at temperatures up to 1,000ºC and good creep performance at 800ºC.

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