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Robert H. Titran
National Aeronautics and Space Administration
Lewis Research Center

and

Mehmet Uz
Lafayette College
Easton, Pennsylvania

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Robert H. Titran
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SUMMARY

Effects of thermomechanical processing on the mechanical properties of Nb-1wt.%Zr-0.1wt.%C, a candidate alloy for use in advanced space power systems, were investigated. Sheet bars were cold rolled into 1-mm thick sheets following single, double, or triple extrusion operations at 1900 K. All the creep and tensile specimens were given a two-step heat treatment of 1 hr at 1755 K + 2 hr at 1475 K prior to testing. Tensile properties were determined at 300 as well as at 1350 K. Microhardness measurements were made on cold rolled, heat treated, and crept samples. Creep tests were carried out at 1350 K and 34.5 MPa for times of about 10,000 to 19,000 hr. The results show that the number of extrusions had some effects on both the microhardness and tensile properties. However, the long-time creep behavior of the samples were comparable, and all were found to have adequate properties to meet the design requirements of advanced power systems regardless of thermomechanical history. The results are discussed in correlation with processing and microstructure, and further compared to the results obtained from the testing of Nb-1wt.%Zr and Nb-1wt.%Zr-0.06wt.%C alloys.

INTRODUCTION AND BACKGROUND

Space power systems are expected to produce electrical power ranging from hundreds of kilowatts to several megawatts. To provide such high levels of power, advanced nuclear power systems currently appear to be the only feasible choice. It is critical to select the appropriate materials to meet the design requirements for such an advanced power system. These requirements currently include a service life of 7 years or more under stresses of 5 to 25 MPa at 1350 to 1450 K in an environment of a liquid alkali metal such as lithium, and total allowable strain of 2 percent maximum (refs. 1 and 2).

Refractory metals and their alloys are the leading candidates for high-temperature applications due to their high melting points. In particular, various alloys of niobium (Nb, melting point = 2742 K) with zirconium (Zr) and/or carbon (C) have been selected for use in advanced space power systems (ref. 1). This choice was based primarily on the resistance of Nb and its alloys to liquid alkali metal corrosion and their lower densities as compared to the other refractory metals and alloys. Initially, solid-solution strengthened Nb-1Zr alloy (this and all the compositions that follow in this paper are expressed in wt.%) was chosen for use in space power conversion systems with a service temperature of about 1000 K and a maximum stress of around 10 MPa (refs. 3 and 4). However, this alloy was not developed for applications requiring long-time stability at temperatures over 1100 K or those with higher stress levels. Because the design requirements of the advanced power systems are currently more stringent involving higher stresses at higher temperatures for longer times, long-term creep resistance of a material becomes the primary concern.

In earlier studies (refs. 5 and 6), a double-annealed (DA: 1 hr at 1755 K + 2 hr at 1475 K both steps followed by furnace cooling) Nb-1Zr-0.06C alloy was reported to have about five times the creep strength of a similarly heat-treated Nb-1Zr alloy at 1350 K and 10 MPA. Under these conditions, the alloy containing 0.06 wt.% carbon had no measurable creep strain after 34,500 hr. However, the Nb-1Zr alloy achieved 2 percent strain in nearly 18,000 hr, and it had a total creep strain of more than 4 percent after about 31,000 hr of testing. The Nb-1Zr alloy containing carbon owes its superior creep resistance to the presence of extremely stable cubic carbides of Zr and Nb (refs. 7 and 8). These earlier studies clearly show that the carbide-strengthened Nb-1Zr alloy has much better creep resistance than the solid solution-strengthened Nb-1Zr.

The current investigation was undertaken as part of a larger project assessing the feasibility of using Nb-1Zr-C alloys in advanced power system applications. In particular, this paper deals with the effects of thermomechanical processing history on the microhardness, tensile, and creep properties of a Nb-1Zr-0.1C alloy, which is also known as PWC-11 (ref. 9). Also included are the tensile and/or creep properties of Nb-1Zr and Nb-1Zr-0.06C alloys in order to assess the effects of the carbon-content on these properties.

EXPERIMENTAL

Materials and Apparatuses

The nominal compositions of the materials evaluated in this study are given in table I. All the samples were in sheet form with a thickness of about 1 mm. The condition in which each specimen was tested and/or examined can be seen from table II. The Nb-1Zr and Nb-1Zr-0.06C samples are included for comparison purposes, and detailed information about these samples can be found in earlier work reported (refs. 6 to 8 and 10).

The designation of the samples will be as given in tables I and II in the remainder of this manuscript. The Nb-1Zr samples are designated as such, those containing 0.1C are labeled with the heat number (064-), and the samples containing 0.06C are labeled LC- (lower carbon as compared to 064 samples). Each alphanumeric identification is followed by extension(s) descriptive of processing and/or testing. The label of each creep-tested sample contains a number giving the test stress in megapascals following the process indicator. It should be noted that the temperature of the creep tests at 10 and 34.5 MPa was 1350 K, and that of the tests at 24 MPa was 1450 K. Then, for example the sample designated LC-DA10 would be a double-annealed Nb-1Zr-0.06C sample creep tested at 1350 K and 10 MPa for the time indicated in table II.

The Nb-1Zr-0.1C samples were all fabricated from a vacuum arc-melted ingot by a combination of hot extrusion and cold rolling. The extrusion operations were performed at 1900 K with an extrusion ratio of 4:1. The primary difference between 064A, 064B, and 064C in table II was that they were, respectively, single-, double-, and triple-extruded prior to cold rolling.

An internally-loaded, constant-load, ultrahigh vacuum chamber was used for the creep tests. It was equipped with split-sleeve resistance heaters (ref. 11) and a viewing port to facilitate observation of

TABLE I.—CHEMICAL COMPOSITIONS OF DOUBLE-ANNEALED (DA: 1 HR AT 1755 K + 2 HR AT 1475 K) Nb-1Zr AND Nb-1Zr-C ALLOYS

Sample	Chemical composition, wt.%				
	0[a]	N[a]	C[b]	Zr[c]	Nb
Nb-1Zr	0.0170	0.0041	0.0016	1.1	Balance
LC-DA	.0080	.0053	.0630	.90	↓
064A-DA	.0028	.0019	.0921	.95	
064B-DA	.0033	.0022	.0914	.95	
064C-DA	.0022	.0009	.0944	.95	

[a] Inert-gas fusion method.
[b] Combustion extraction method.
[c] Inductively-coupled plasma method.

TABLE II.—PROCESSING HISTORIES OF Nb-1Zr, Nb-1Zr-0.06C, AND Nb-1Zr-0.1C SHEET SAMPLES

Sample	Number of extrusions[a]	Heat treatment/condition
Nb-1Zr-A	-	1 hr at 1475 K
Nb-1Zr-A34		1 hr at 1475 K + 218 hr at 1350 K and 34.5 MPa
Nb-1Zr-DA	-	DOUBLE-ANNEALED, DA[d]
Nb-1Zr-DA10		DA + 31 780 hr at 1350 K and 10 MPa
LC-DA	-	DOUBLE-ANNEALED, DA
LC-DA10		DA + 34 500 hr at 1350 K and 10 MPa
LC-DA34		DA + 4288 hr at 1350 K and 34.5 MPa
LC-DA/AGE	-	DOUBLE-ANNEALED, DA, + AGED[e]
LC-DA/AGE10		DA + AGE + 32 500 hr at 1350 K and 10 MPa
064A	1	AS COLD ROLLED (96%CW)
064A-DA	1	DOUBLE-ANNEALED, DA
064A-DA34(E)[b]	1	DA + 18 780 hr at 1350 K
064A-DA34(M)[c]	1	DA + 18 780 hr at 1350 K and 34.5 MPa
064B	2	AS COLD ROLLED (88%CW)
064B-DA	2	DOUBLE-ANNEALED, DA
064B-DA34(E)[b]	2	DA + 15 460 hr at 1350 K
064B-DA34(M)[c]	2	DA + 15 460 hr at 1350 K and 34.5 MPa
064C	3	AS COLD ROLLED (60%CW)
064C-DA	3	DOUBLE-ANNEALED, DA
064C-DA34(E)[b]	3	DA + 9950 hr at 1350 K
064C-DA34(M)[c]	3	DA + 9950 hr at 1350 K and 34.5 MPa

[a] Number of hot extrusions employed in fabricating sheet bars prior to cold rolling.
[b] (E): Stress-free end portions of crept sample.
[c] (M): Stressed middle portion of crept sample.
[d] DA: A 2-step anneal of 1 hr at 1755 K + 2 hr at 1475 K.
[e] AGE: A heat treatment of 1000 hr at 1350 K.

the sample. Ultrahigh vacuum chambers were also used for all the heat treatments. The pressure in these high-temperature chambers during testing was of the order of 10^{-6} Pa. A screw-driven tensile testing machine was used for both the room and high-temperature tensile tests. The temperature in a chamber during high-temperature tests was measured using an R-type (Pt/Pt-13%Rh) thermocouple. The temperature in the chambers was maintained within ±5° of the intended test temperature. A microhardness tester equipped with a diamond pyramid indentor and a digital processor/printer was used for the hardness measurements.

Procedure

All the testing and evaluation in this study were conducted on longitudinal samples, i.e., the direction of applied load in tensile and creep tests and the surfaces examined were parallel to the rolling direction of the sheet. Prior to testing and annealing, the samples were chemically cleaned using a solution of H_2O:HNO_3:HF with a volume ratio of 3:1:1. Furthermore, to avoid interstitial impurity contamination during exposure to elevated temperatures, the samples were wrapped in chemically-cleaned tantalum foil prior to heat treating and high-temperature testing.

The creep and tensile test specimens were similar in geometry. The sketch of a typical sample showing dimensions is shown in figure 1. Fiducial marks were placed in the narrow gage section of each sample to facilitate the measurement or monitoring of the changes in sample length.

One sample from each of the 064A-DA, 064B-DA, and 064C-DA (see table II) was creep tested uniaxially at 1350 K under a stress of about 34.5 MPa. The test was carried out for a period of 18,780 hr for 064A-DA, 15,460 hr for 064B-DA, and 9950 hr for 064C-DA. The times and conditions of the creep tests on the samples from the Nb-1Zr and Nb-1Zr-0.06C alloys were as given in table II. The elongation, i.e., the change in the distance between the fiducial marks, was measured optically through the holes punched in the tantalum wrap using a cathetometer.

Tensile tests on samples from each of 064A-DA, 064B-DA, and 064C-DA were made in duplicate at 300 and 1350 K. The cross-head speed during these tests was about 21.17×10^{-6} m/s which corresponds to a strain rate of about 8×10^{-4} s^{-1} assuming that the deformation was restricted to the 25-mm gage section of the sample. The tensile properties of ultimate tensile strength (UTS), 0.2 percent offset yield strength (YS), ductility (ε), and uniform elongation (ε_u) were determined from a load-time plot obtained for each test using the widely-known equations and conventional methods (ref. 12).

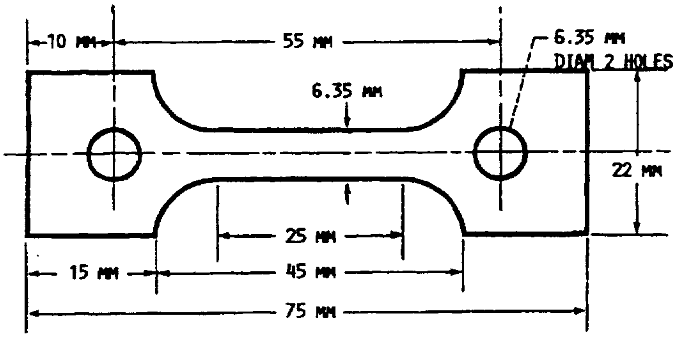

Figure 1.—A schematic of the specimens used in tensile and creep tests.

Vickers hardness measurements were made on all of the 064-samples listed in table II. The number of readings varied from 6 to 20 per sample depending on the sample surface available.

Optical micrographs of the 064-samples were taken in the as-polished condition before and after creep testing to correlate the mechanical properties to microstructure and processing.

RESULTS AND DISCUSSION

Microstructure

The microstructures of the Nb-1Zr and Nb-1Zr-0.06C alloys as well as the effects of thermomechanical processing and various heat treatments on the microstructure of Nb-1Zr-0.1C sheets (064-samples in table II) were discussed in earlier publications (refs. 5 to 8 and 13). Hence, they will be discussed here as they are pertinent to the present study with the emphasis on the double-annealed and as-crept microstructures of the samples from 064A, 064B, and 064C.

Prior to heat treating, all the samples had typical cold-rolled microstructures with grains elongated and the precipitates aligned along the rolling direction. In the samples containing carbon, the precipitates were mainly orthorhombic Nb_2C with some fine cubic carbides of Zr and Nb (refs. 8 and 13). The only precipitates found in the Nb-1Zr samples were fine ZrO_2 (ref. 5).

The samples from the Nb-1Zr alloy were either given a heat treatment of 1 hr at 1475 K (sample Nb-1Zr-A in table II) or they were double-annealed, 1 hr at 1755 K + 2 hr at 1475 K (Nb-1Zr-DA in table II) before being creep tested. Both heat treatments resulted in a recrystallized microstructure with some dispersion of fine ZrO_2 particles throughout the matrix (ref. 5).

The microstructures of the samples from the Nb-1Zr-0.1C sheets before and after creep testing at 1350 K and 34.5 MPa are shown in figure 2. An examination of the micrographs in (a) to (c) shows that the effects of the number of extrusions on the double-annealed microstructures were quite noticeable. The sample from the single-extruded sheet, 064A-DA, had a fully recrystallized microstructure with relatively large grains and rather coarse precipitates along the grain boundaries and within the grains alike. Sample 064B-DA was also recrystallized with a microstructure similar to 064A-DA, but with smaller grains. The double-annealed sample from the triple-extruded sheet, 064C-DA, had a microstructure with highly-elongated grains giving no indication of full recrystallization. The precipitates in 064A-DA were primarily orthorhombic Nb_2C with some cubic (Zr,Nb)C. The relative amount of the more stable cubic carbides of (Zr,Nb)C increased with the number of extrusions in both the as-rolled and double-annealed samples (ref. 13).

The micrographs in figures 2(d) to (f) show the microstructures of the stressed middle portions of the crept samples. The microstructure of the stress-free end of each sample was similar to that of its middle. There does not appear to be any discernable difference between the microstructures of the crept samples 064A-DA34, 064B-DA34, and 064C-DA34. It is evident, however, that prolonged exposure to 1350 K gave rise to significant changes in the double-annealed microstructure of each sample. Such exposure caused the rather coarse Nb_2C, which was the primary carbide in the double-annealed samples, to transform to the finer and more stable (Zr,Nb)C finely distributed throughout the matrix in all the crept samples regardless of the processing history. The double-annealed and as-crept microstructures of the samples from the Nb-1Zr-0.06C sheet (LC-DA34) were similar to those of 064A-DA34 shown in figure 2. However, the microstructure of LC-DA34 had smaller grains and less amount of precipitates throughout its matrix (refs. 5, 7, and 8).

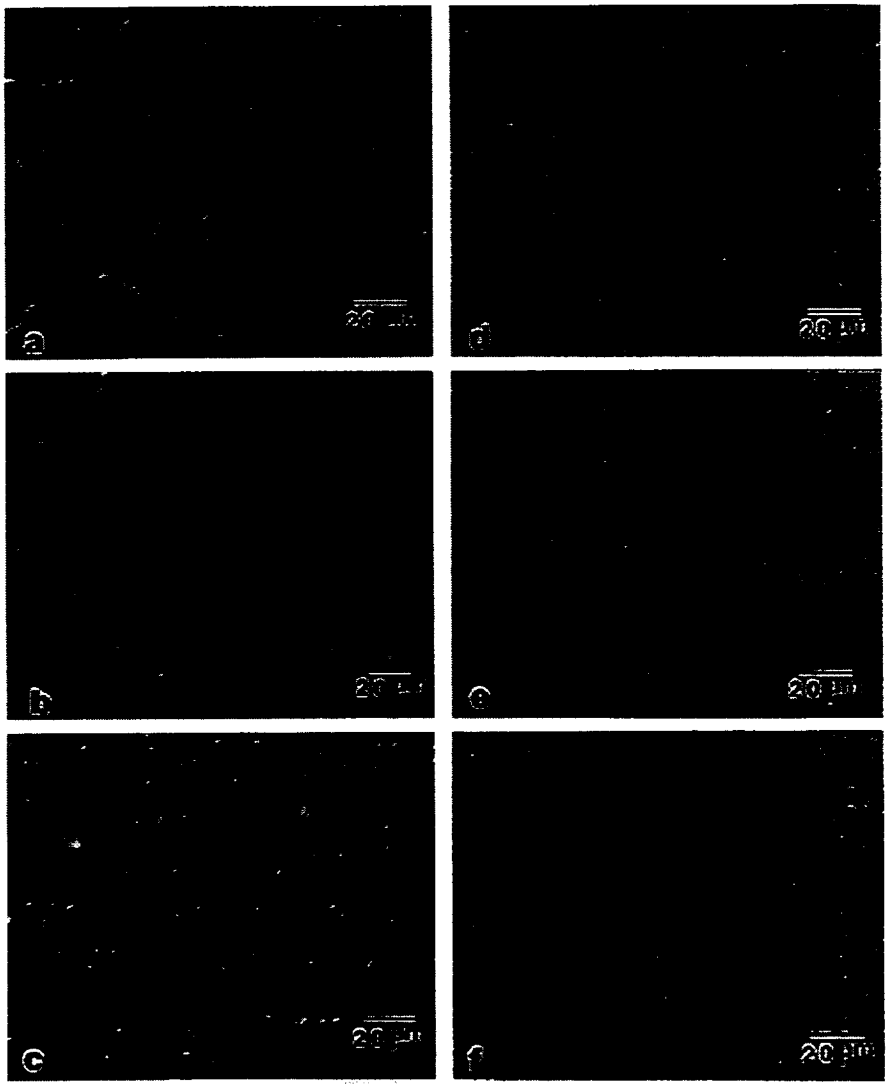

Figure 2.—Optical micrographs of the Nb-1Zr-0.1C samples before and after creep testing at 1350 K and 34.5 MPa. All differential interference contrast (DIC) images of as-polished samples, and the longitudinal or rolling direction is parallel to the lines of text. (a) 064A-DA. (b) 064B-DA. (c) 064C-DA. (d) 064A-DA34(M). (e) 064B-DA34(M). (f) 064C-DA34(M).

Microhardness

Microhardness measurements were made on the samples from the Nb-1Zr-0.1C sheets in the as-rolled and double-annealed conditions as well as on their stressed middle and stress-free end portions following creep testing. The results are tabulated in table III and are also plotted as bar graphs in figure 3 to facilitate better visualization of the differences. The results show that in a given condition, the microhardness of the specimens from the single-, double-, and triple-extruded sheets fall within about one standard deviation of one another. This would indicate that the number of extrusions prior to cold rolling did not have a significant effect on the microhardness of these samples regardless of the condition. As expected, double-anneal resulted in a marked decrease of about 40 percent in the microhardness of each sample. This is attributable to the relief of internal stresses and the effects of cold work by recovery and recrystallization during such heat treatment. Also expected was that the stressed middle of each sample

TABLE III.—MICROHARDNESS OF Nb-1Zr-0.1C SHEET SAMPLES DESCRIBED IN TABLE II

Sample	Vickers hardness number, VHN	Standard deviation, $\mp S_n$
064A	179.1	5.8
064A-DA	104.0	4.0
064A-DA34(E)	90.3	4.6
064A-DA34(M)	112.2	10.1
064B	163.0	10.0
064B-DA	95.5	9.1
064B-DA34(E)	101.9	10.7
064B-DA34(M)	128.3	3.8
064C	157.5	6.5
064C-DA	104.0	5.2
064C-DA34(E)	100.4	5.4
064C-DA34(M)	112.9	4.3

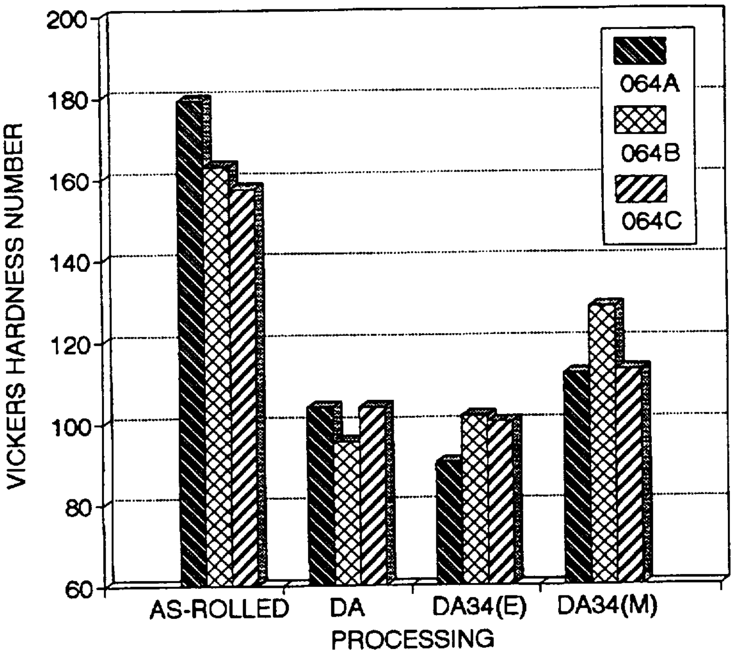

Figure 3.—Bar graphs of the microhardness of samples from as-rolled, as double-annealed and as-crept Nb-1Zr-0.1C specimens.

had a slightly higher microhardness than its stress-free ends. This should be due to the strain induced in the reduced gage section of the crept sample even though the total creep strain in each was extremely small as will be discussed shortly. It is very interesting to note from the results that prolonged exposure to 1350 K for approximately 10,000 to 19,000 hr had no measurable effect on the microhardness of the double-annealed samples. This can be attributed to the predominant presence of the extremely stable and finely-distributed cubic carbides of (Zr,Nb)C in the crept samples.

Tensile Properties

The results of the tensile tests at 300 and 1350 K are tabulated in table IV. The difference between the duplicate values was less than 5 percent relative to the averages reported in the table. Ultimate tensile strength, yield strength, and ductility data are also presented in bar-graph form in figures 4 to 6, respectively.

The tensile strengths of 064A-DA, 064B-DA, and 064C-DA were comparable to one another with 064C-DA being a little higher than the other two at both 300 and 1350 K (see table IV and fig. 4). This indicates that multiple extrusions did not have a significant effect on the tensile strength either as was the case for the microhardness of these samples presented above. Slightly higher tensile strength of 064C-DA should be expected because this sample had an oriented microstructure with grains elongated in the load (longitudinal) direction. The tensile strength of the solid-solution strengthened Nb-1Zr-A was lower than all of the precipitation-strengthened 064-sheets at 300 K, but they were comparable to one another at 1350 K.

The trends concerning yield strengths (see table IV and fig. 5) were different than those observed for the tensile strength. The yield strengths of 064A-DA and 064B-DA were comparable to one another, but both were measurably lower than 064C-DA at 300 K as well as at 1350 K. The yield strength of Nb-1Zr-A was close to that of 064C-DA at 300 K, and it was lower than all of the 064-samples at 1350 K.

The ductility data in table IV and figure 6 show that the values for 064A-DA and 064B-DA were both about 35 percent at 300 K and increased to about 40 percent at 1350 K. The ductility of 064C-DA was lower, and that of Nb-1Zr-A was higher than either (of 064A-DA and 064B-DA) at both temperatures. Rather unexpected was that the ductility of Nb-1Zr-A as well as of 064C-DA was considerably lower at 1350 K than at 300 K.

It is evident from the above results that each of the samples tested has tensile properties which are more than adequate for the advanced power system applications considering the maximum service stress of about 25 MPa. None of these materials should have brittle failure, nor should they have any strength-related problems during operation.

TABLE IV.—RESULTS OF TENSILE TESTS ON SAMPLES FROM Nb-1Zr AND Nb-1Zr-0.1C SHEETS AT 300 AND 1350 K.

Sample	Test conditions		Test results			
	Temperature, K	$\varepsilon \times 10^4$, 1/sec[b]	UTS, MPa	0.2%YS, MPa	ε, %[b]	ε_u, %[b]
Nb-1Zr-A[a]	300	4	224	127	41	26
	1350	4	126	76	33	18
064A-DA	300	8.33	263.9	108.1	35.8	24.8
	1350	8.33	126.3	88.1	42.1	14.9
064B-DA	300	8.33	257.3	105.4	35.2	24.4
	1350	8.33	115.9	84.1	39.6	13.1
064C-DA	300	8.33	281.0	134.2	28.1	19.8
	1350	8.33	134.9	113.1	24.0	5.3

[a] From reference 10.

[b] Based on the assumption that the elongation was restricted to 25-mm gage region.

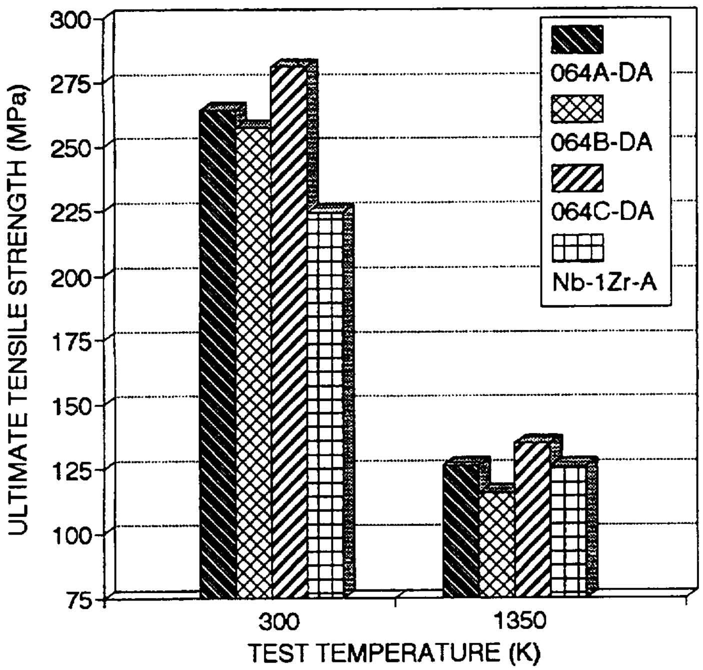

Figure 4.—Bar graphs showing the ultimate strength of samples from Nb-1Zr and Nb-1Zr-0.1C at 300 and 1350 K.

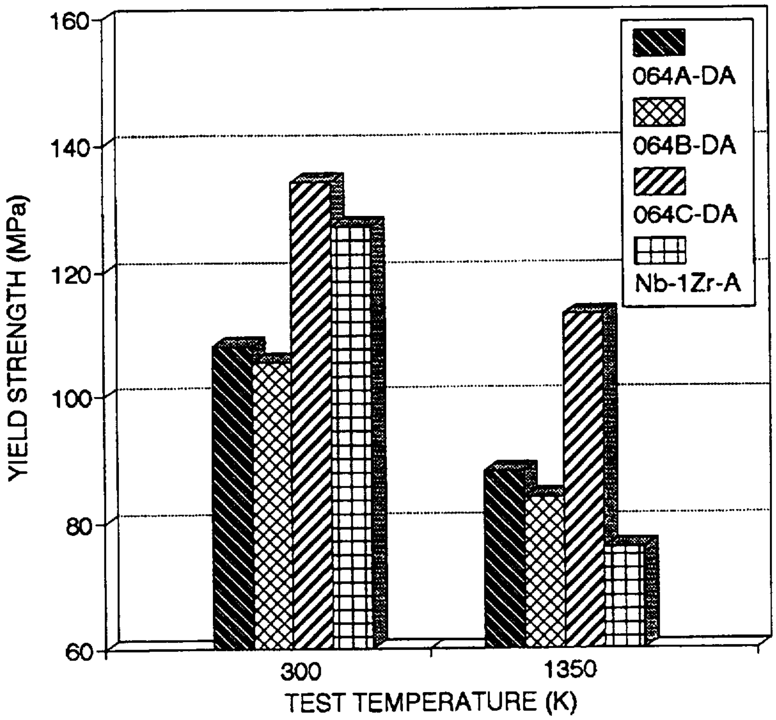

Figure 5.—Bar graphs showing the yield strength of samples from Nb-1Zr and Nb-1Zr-0.1C at 300 and 1350 K.

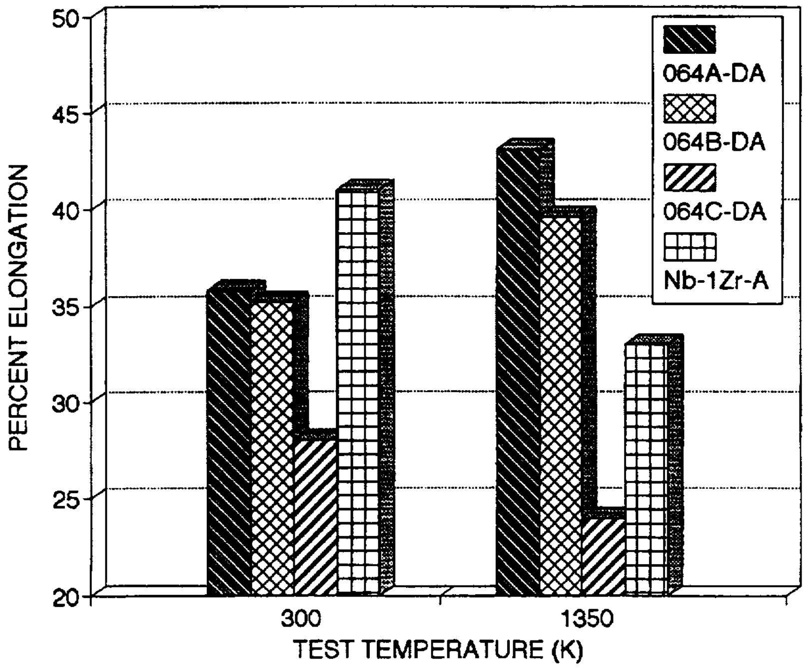

Figure 6.—Bar graphs showing the ductility of samples from Nb-1Zr and Nb-1Zr-0.1C at 300 and 1350 K.

Creep Properties

The conditions and the results of the creep tests on various samples from Nb-1Zr, Nb-1Zr-0.06C (LC-samples) and Nb-1Zr-0.1C (064-samples) are given in table V. To better illustrate the creep behavior of each sample the results are plotted for the test conditions of 1350 K and 10 MPA in figure 7, 1350 K and 34.5 MPa in figure 8, and for 1450 K and 24 MPa in figure 9. It will be noted that all the creep tests have been concluded with the exception of those at 1450 K. These latter tests are still in progress at over 10,000 hr, and each appears to have reached the linear or steady state creep stage already (around 3000 hr).

The first four columns in table V are self explanatory. The total creep strain in each sample as measured at the time indicated is given in column 5. The steady state creep rate in column 6 is calculated from the slope of the linear portion of the strain-time plot for each sample. This rate is then used to estimate the time it would take each sample to reach 1 and 2 percent strain given in columns 7 and 8, respectively. These times are given as a range with its lower and higher ends corresponding to the lower and higher ends of the ± error in each. For samples which crept 1 or 2 percent during testing, the experimentally-determined times are provided in parentheses. It is noteworthy that the comparison of the experimental and estimated values for such samples shows that the estimated times to reach 1 or 2 percent strain are more conservative.

The results in table V and figure 7 for the creep tests at 1350 K and 10 MPa show that Nb-1Zr-DA10 reached a strain of 2 percent in less than 20,000 hr, and it crept over 4 percent after nearly 32,000 hr of testing. Of the carbon-containing alloys tested under similar conditions, LC-DA10 showed no measurable strain after 34,500 hr and the total strain in LC-DA/AGE10 was about 0.1 percent after nearly 32,500 hr. The estimated time to reach 2 percent creep strain for either of the crept LC-samples was well over 25 years. Considering that LC-DA10 showed no measurable creep strain after 4 years of testing, this number may be quite realistic and perhaps even a little conservative. Furthermore, it was reported in the earlier studies (refs. 5 and 6) that the stress necessary to reach 1 percent strain in 7 years was about 4 Pa

TABLE V. RESULTS OF THE CREEP TESTS AT 1350 K AND 34.5 MPa AND AT 1450 K AND 24 MPa

Sample	Test conditions			Test results		Time to strain[c]	
	Time, hr	Temperature, K	σ, MPa	Total ε, %	Linear[b] ε°, h^{-1}	ε = 1%, 1000 hr	ε = 2%, 1000 hr
Nb-1Zr-DA10	31 780	1350	10	4.06	(14±0.07)x10^{-5}	7.1-7.2 (11)[d]	14.2-14.4 (18)[d]
LC-DA10	34 500	1350	10	(e)	(e)	(e)	(e)
LC-DA/AGE10	32 500	1350	10	0.1	(3.2±0.56)x10^{-6}	266-378	532-756
Nb-1Zr-A34	218	1350	34.5	5.18	(233±2.3) 10^{-5}	0.04-0.05 (0.075)[d]	0.08-0.10 (0.125)[d]
LC-DA34	4 288	1350	34.5	1.65	(39.7±5)x10^{-5}	2.30-2.90 (3.2)[d]	4.6-5.8
064A-DA34	18 780	1350	34.5	0.29	(11.6±1.7) 10^{-6}	75.2-101	150.4-202
064B-DA34	15 460	1350	34.5	0.21	(14±0.8)x10^{-6}	67.5-75.5	135-151
064C-DA34	9 950	1350	34.5	0.27	(17±1.1) 10^{-6}	55-63	110-126
064A-DA24[a]	11 800	1450	24	0.82	(64±2)x10^{-6}	15.2-16.2	30.4-32.4
064B-DA24[a]	10 000	1450	24	0.29	(16.1±2.5) 10^{-6}	53.5-73.5	107-147
064C-DA24[a]	11 100	1450	24	0.16	(9.7±1.4)x10^{-6}	90-120	180-240

[a] Creep test in progress.
[b] Linear or steady-state creep rate as calculated from the slope of the strain versus time plot.
[c] Estimated using the linear strain rate in column 6.
[d] Experimental values as observed during the creep test.
[e] This sample had no measurable strain at the conclusion of the creep test.

for double-annealed Nb-1Zr alloy and 20 MPa for a similarly heat-treated Nb-1Zr-0.06C alloy at 1350 K. These results clearly indicate that the carbide-strengthened Nb-1Zr-0.06C alloy has much better creep resistance than the solid solution-strengthened Nb-1Zr alloy at 1350 K and 10 MPa.

The data in table V clearly shows that the creep resistance of the Nb-1Zr alloys containing carbon are superior to that without carbon at 1350 K and 34.5 MPa as well. Nb-1Zr-A34 had a total strain of over 5 percent in only 218 hr, and the total strain in the alloy with 0.06C (LC-DA34) was about 1.6 percent after nearly 4300 hr. The total strain in each of the samples from the Nb-1Zr-0.1C sheets was less than 0.3 percent after creep tests of nearly 19,000 hr for 064A-DA34, 15,000 hr for 064B-DA34, and 10,000 hr for 064C-DA34.

The creep plots in figure 8 would indicate that the creep behavior of the single-, double-, and triple-extruded sheets from the Nb-1Zr-0.1C alloy (064A-DA34, 064B-DA34, and 064C-DA34, respectively) are similar to one another at 1350 K and 34.5 MPa. It is evident from the figure and table V, however, that each of these alloys has a much better creep resistance than either Nb-1Zr-A34 or LC-DA34. The linear or steady state creep rate of the LC-DA34 is one order of magnitude and those of the 064-sheets are

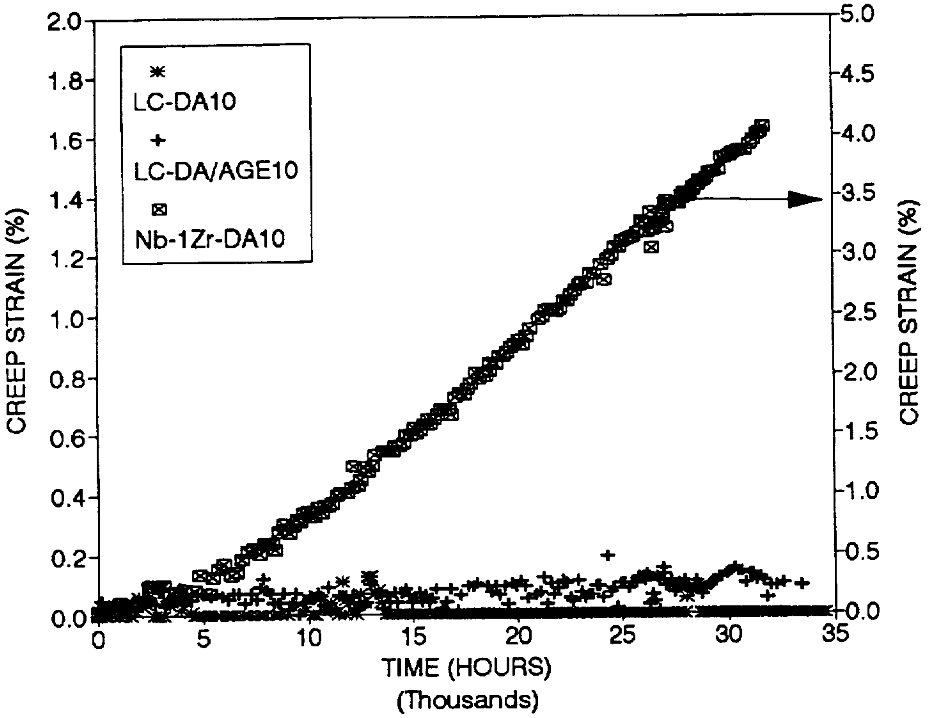

Figure 7.—Strain versus time plots for samples from Nb-1Zr and Nb-1Zr-0.06C specimens creep tested at 1350 K and 10 MPa. The specimens were double-annealed (DA: 1 h at 1755 K + 2 h at 1475 K) or double-annealed and aged (AGE: 1000 h at 1350 K) as indicated. (Read right strain axis for Nb-1Zr-DA10.)

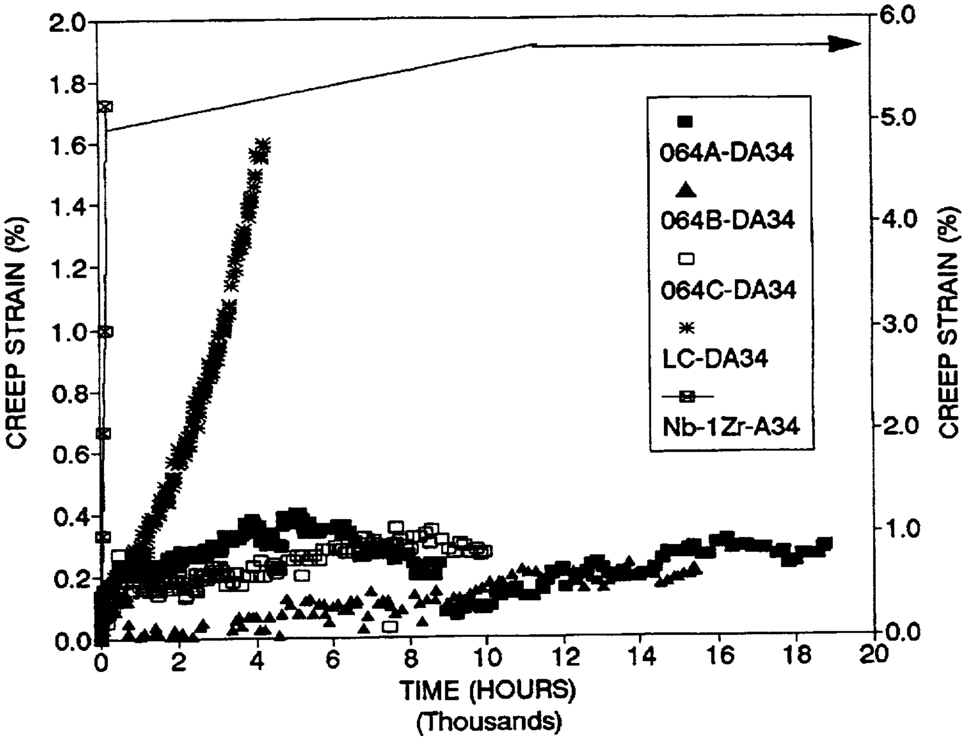

Figure 8.—Strain versus time plots for specimens from Nb-1Zr, Nb-1Zr-0.06C and Nb-1Zr-0.1C creep tested at 1350 K and 34.5 MPa. (Read right strain axis for Nb-1Zr-A34.)

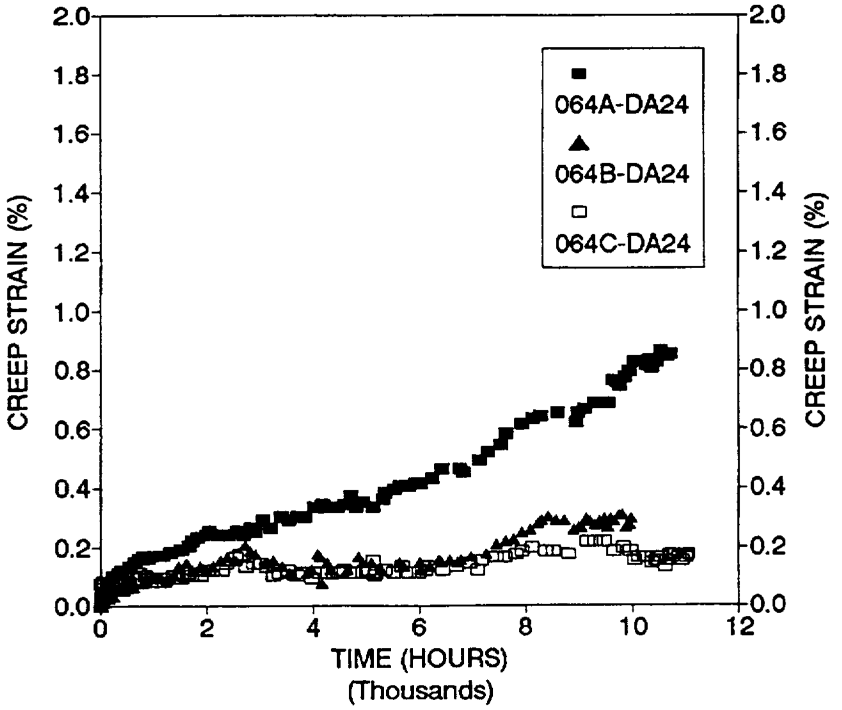

Figure 9.—Strain versus time plots for specimens from Nb-1Zr-0.1C creep tested at 1450 K and 24 MPa.

at least two orders of magnitude less than the linear creep rate of the Nb-1Zr alloy. At 1350 K and 34.5 MPa, the estimated time to reach a creep strain of 2 percent is around 100 hr for Nb-1Zr-A34 (≈125 hr as measured during testing) and less than 6000 hr for Nb-1Zr-0.06C sample LC-DA34. Under the same conditions, the Nb-1Zr-0.1C sheet with the highest creep rate (064C-DA34) is estimated to reach 2 percent strain in at least about 110,000 hr, approximately twice the design service life of 61,000 hr (7 years). The data in table V would further indicate that of the Nb-1Zr-0.1C sheets, 064A-DA34 is the most and 064C-DA34 is the least creep resistant at 1350 K.

The data from the creep tests that are in progress at 1450 K and 24 MPa (see table V and fig. 9) show a somewhat different trend. Among these samples, 064C-DA24 appears to be the most and 064A-DA24 the least creep resistant, which is a reversal of the order at 1350 K and 34.5 MPa. The total strain in 064A-DA24 is about 0.8 percent after nearly 11,000 hr, and its linear strain rate is anomalously high. This sheet is estimated to reach 2 percent strain in less than 4 years which is less than the service life requirement of 7 years. Of the other two samples being tested, the estimated time to reach 2 percent strain is about 12 years for 064B-DA24 and nearly 20 years for 064C-DA24.

It is evident from the creep data presented here that the carbide strengthened Nb-1Zr alloys have much better creep resistance than the solid solution-strengthened Nb-1Zr alloy at 1350 K and should be the case at 1450 K as well. This must be expected because of the presence of extremely stable cubic carbides of (Zr,Nb)C in the carbon-containing alloys. These carbides are shown to form during processing and especially during high-temperature exposure mainly as a result of transformation from rather coarse Nb_2C (refs. 8 and 12). It is also obvious from the results that increasing the carbon content from 0.06 to 0.1 percent gives rise to a marked increase in the creep resistance of the alloy. This can be attributed to an increase in the number density of the carbides, because more carbon goes out of solution and become available for precipitation with increasing carbon content. Furthermore, it is quite clear that the carbide-strengthened alloys would be the only choices if a service life of 7 years with a total creep strain of less than 2 percent are to be required at temperatures around 1350 K and stresses approaching or exceeding 10 MPa.

CONCLUSIONS

Microhardness, tensile, and long-term creep behavior of Nb-1Zr-0.1C sheets with different processing histories were studied. The sheets studied were fabricated by cold rolling following single-, double-, or triple-extrusion operations at 1900 K. The purpose was to determine the effects of thermomechanical processing on the properties and to assess the feasibility of the use of these materials in advanced power system applications. From the results obtained in this study on the Nb-1Zr-0.1C sheets and the previous results on Nb-1Zr and Nb-1Zr-0.06C sheets, the following conclusions could be drawn:

1. The number of 1900 K extrusions prior to cold rolling does not have a significant effect on the micro-hardness of Nb-1Zr-0.1C sheets in a given condition, e.g., as-rolled, as double annealed. Microhardness decreases (≈40 percent) significantly upon double annealing, but is not affected by subsequent exposure to 1350 K for as long as 19,000 hr.

2. The samples from the single-, double-, and triple-extruded Nb-1Zr-0.1C sheets (064A-DA, 064B-DA, and 064C-DA, respectively) and those of the Nb-1Zr alloy that was annealed for 1h at 1475 K (Nb-1Zr-A) all have more than adequate strength and ductility for advanced power applications intended.

3. Multiple extrusions appear to have very little if any beneficial effect on the creep behavior of the Nb-1Zr-0.1C sheet at 1350 K. At this temperature under either 10 or 34.5 MPa, the carbide-strengthened sheets containing 0.06 or 0.1%C all have much superior creep resistance than the solid-solution strengthened Nb-1Zr owing to the presence of the extremely stable fine carbides of Zr and Nb, (Zr,Nb)C. Furthermore, the creep resistance of the Nb-1Zr-C alloy increases with increasing carbon content.

4. If a service life of 7 years with a total creep strain of 2 percent or less is desirable at 1350 K, either of the carbon-containing alloys are viable candidates provided that the service stresses stay around 10 MPa. If the service stresses approach 34 MPa, the alloy with 0.1%C should be the only choice, because at this stress level it takes Nb-1Zr a little over 100 hr and Nb-1Zr-0.06C less than 1 year to reach 2 percent strain while the estimated time to creep 2 percent is over 12 years for Nb-1Zr-0.1C.

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13. ABSTRACT ***(Maximum 200 words)***

Effects of thermomechanical processing on the mechanical properties of Nb-1wt.%Zr-0.1wt.%C, a candidate alloy for use in advanced space power systems, were investigated. Sheet bars were cold rolled into 1-mm thick sheets following single, double, or triple extrusion operations at 1900 K. All the creep and tensile specimens were given a two-step heat treatment of 1 hr at 1755 K + 2 hr at 1475 K prior to testing. Tensile properties were determined at 300 as well as at 1350 K. Microhardness measurements were made on cold rolled, heat treated, and crept samples. Creep tests were carried out at 1350 K and 34.5 MPa for times of about 10,000 to 19,000 hr. The results show that the number of extrusions had some effects on both the microhardness and tensile properties. However, the long-time creep behavior of the samples were comparable, and all were found to have adequate properties to meet the design requirements of advanced power systems regardless of thermomechanical history. The results are discussed in correlation with processing and microstructure, and further compared to the results obtained from the testing of Nb-1wt.%Zr and Nb-1wt.%Zr-0.06wt.%C alloys.

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