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Robert H. Titran and William D. Klopp
Lewis Research Center
Cleveland, Ohio

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Summary

The creep behavior of the niobium alloy C-103 (Nb-10Hf-1Ti-0.7Zr) has been studied as a function of stress, temperature, and grain size for test times to 19 000 hr. Over the temperature range 827° to 1204° C and the stress range 6.89 to 138 MPa, only tertiary (accelerating) creep was observed. The creep strain can be related to time by an exponential relation recently developed by the authors:

$$\epsilon = \epsilon_0 + K(e^{st} - 1)$$

where ϵ_0 is the initial creep strain, K is the tertiary creep strain parameter, and s is the tertiary creep rate parameter. The initial tertiary creep rate Ks (the zero-time intercept of the differential of the strain-time relation) can be related to stress, temperature, and grain size by a modified power-law relation:

$$Ks = A(\sigma/E)^n(1 + f/d^2)e^{-Q/RT}$$

where A is the proportionality constant, σ is stress, E is Young's modulus, f is the grain-size factor, d is grain size, Q is the activation energy, R is the gas constant, and T is temperature. The observed stress exponent 2.87 is similar to the three-power law generally observed for secondary (linear) creep of Class I solid solutions. The apparent activation energy 374 kJ/g mol is close to that observed for self-diffusion of pure niobium. The initial tertiary creep rate was slightly faster for fine-grained than for coarse-grained material. The strain parameter K can be expressed as a combination of power functions of stress and grain size and an exponential function of temperature. Strain-time curves generated by using calculated values for K and s showed reasonable agreement with observed curves to strains of at least 4 percent. The time to 1 percent strain was related to stress, temperature, and grain size in a similar manner as the initial tertiary creep rate.

Introduction

Expressions relating creep strain to time during tertiary creep have been proposed for several alloys, for example, Nimonic 80 (ref. 1), iron (ref. 2), alpha-brass (ref. 3), and stainless steel (ref. 4). Although these expressions appear capable of mathematically describing tertiary creep curves, they all require as input the time to initiation of tertiary creep. This requirement limits the usefulness of these expressions in predicting tertiary creep curves as functions of the usual inputs of stress, temperature, and grain size.

Power terms of time have also been used to represent creep curves containing segments of tertiary (accelerating) creep (ref. 1). For refractory alloys we previously proposed relations of one or more power terms of the total creep time to represent tertiary creep in tantalum and niobium alloys, such as $(At)^{3/2}$ (refs. 5 and 6) and $(At)^{3/2} + (Bt)^{5/2}$ (ref. 7). This type of relation does not require knowledge of the time to the initiation of tertiary creep, but the requirement for multiple power terms to model extensive portions of tertiary creep makes this approach cumbersome.

More recently we proposed an exponential term for tertiary creep that overcomes at least some of the objections to previous approaches. This tertiary creep term is similar to the Garofalo exponential term proposed for primary creep (ref. 8) and can be combined with that primary creep term to express curves exhibiting both primary and tertiary creep:

$$\epsilon = \epsilon_0 + \epsilon_p(1 - e^{-rt}) + K(e^{st} - 1) \quad (1)$$

This relation is capable of describing well the creep curves for the tantalum alloy Astar 811C to creep strains of 3 percent (ref. 9).

The usefulness of the new exponential tertiary creep term in describing the creep behavior of the niobium alloy C-103 is examined in this paper. The original purpose for conducting this creep study was to provide design data to facilitate use of C-103 in the heat exchanger and related components of a small, 2-kW, electric, isotope-fueled Brayton cycle space power system (ref. 10). An analysis of the time to 1 percent strain for C-103 alloy is included, since it was the design criterion for this system.

The nonlinear regression programs used in this study were designed by Steven M. Sidik and Raymond W. Palmer of the Lewis Research Center.

Symbols

A	proportionality constant, various dimensions
a	stress exponent, dimensionless

B	proportionality constant, various dimensions
b	grain-size exponent, dimensionless
c	temperature factor, dimensionless
d	grain diameter, μm
E	Young's modulus, MPa
f	grain-size factor, cm^2
K	tertiary creep strain parameter, dimensionless
n	stress exponent, dimensionless
n_1	grain-size-independent stress exponent
n_2	grain-size-dependent stress exponent
Q	activation energy, kJ/g mol
R	gas constant, kJ(g mol)(deg)
r	primary creep rate parameter, sec^{-1}
s	tertiary creep rate parameter, sec^{-1}
T	temperature, K
t	time, sec
t_{1pct}	time to 1 percent strain, sec
ϵ	creep strain, dimensionless
ϵ_p	total primary creep strain, dimensionless
ϵ_0	initial creep strain, dimensionless
$\dot{\epsilon}$	creep rate, sec
σ	stress, MPa

Experimental Procedure

The niobium C-103 alloy was commercially procured as 0.076-cm-thick sheet. The principal constituents of the alloy were determined to be:

Hafnium, wt%	9.75
Titanium, wt%	1.11
Zirconium, wt%	0.45
Tantalum, wt%	0.31
Tungsten, wt%	0.25
Oxygen, wt ppm	214
Nitrogen, wt ppm	62
Carbon, wt ppm	37
Hydrogen, wt ppm	0.8
Niobium	Balance

Creep specimens having a 0.635-cm-wide by 2.54-cm-long gage section were machined from the 0.076-cm-thick sheet. These specimens were degreased, rinsed in distilled water and alcohol, wrapped in tantalum foil, and annealed in a vacuum of 10^{-10} Pa at 1204° to 1760° C for 1 to 5 hr before creep testing. Average grain diameters (measured by the intercept method) ranged from 15 μm after annealing for 1 hr at 1204° C to 83 μm after annealing for 1 hr at 1760° C. Weight changes associated with annealing generally amounted to only a few milligrams, equivalent to changes in interstitial content of a few tens of ppm.

Constant-load creep tests were conducted in the internally loaded, high-vacuum creep units described in reference 11. A tantalum split-sleeve resistance heater was used for heating the specimens. The pressure was generally about 10^{-10} Pa at the start of a creep test and decreased into the 10^{-12}-Pa range after several hundred hours. Strains were measured by frequent optical readings of fiducial marks in the reduced gage section during creep. Strains on loading were of the order of 0.05 percent and are not included in the creep strain data.

Test temperatures ranged from 827° to 1204° C, and stresses from 6.89 to 138 MPa. Tests were generally terminated after 1 percent strain, although a few were continued to strains as high as 4 percent.

Grain sizes measured after creep are included in table I; these values are essentially the same as those measured on annealed and uncrept specimens, an indication that the grain sizes were stable under the creep conditions studied.

Results and Discussion

Shapes of Creep Curves

Typical long-time creep curves for C-103 are shown in figures 1 and 2. These curves consist entirely of tertiary (accelerating) creep, with no apparent period of primary (decelerating) or secondary (linear) creep. All strain-time data were computer fitted by using nonlinear regression techniques based on the exponential relation recently developed by the authors to described tertiary creep in the tantalum alloy Astar 811C (ref. 9):

$$\epsilon = \epsilon_0 + K(e^{st} - 1) \tag{2}$$

The good fits of the data to this relation seen in figures 1 and 2 are representative of those obtained in the 35 tests included in this analysis. Table I presents the creep curve parameters derived from regression fitting of the individual curves and other pertinent creep data from this study.

Differentiating equation (2) gives the creep rate as a function of time:

$$\dot{\epsilon} = Kse^{st} \tag{3}$$

This relation predicts a linear increase in log creep rate with time. In contrast, the Garofalo primary creep term (ref. 8) predicts a linear decrease in log creep rate with time. Creep rates determined by linear regression over sequential segments of several creep tests are seen in figure 3 to be fairly well related to time by equation (3). The initial tertiary creep rate, which is equal to *Ks*, is the creep rate at zero time (intercept of the rate-time curve with the ordinate in fig. 3). Equation (3) appears valid to at least a threefold range in creep rates.

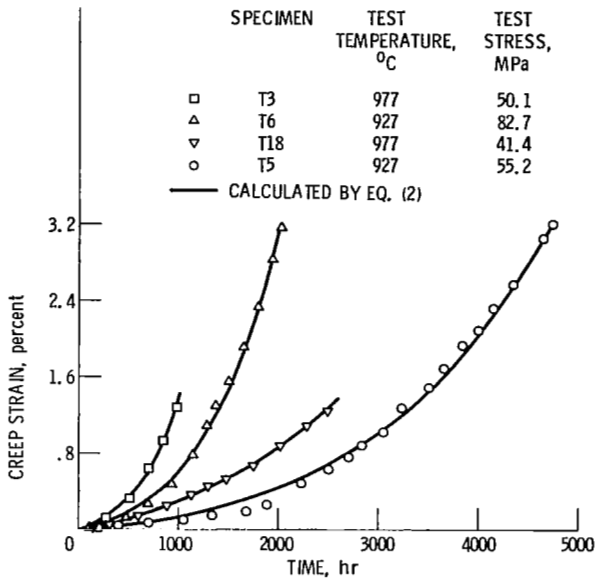

Figure 1. - Typical creep curves for C-103 tested at 927° and 977° C, showing only tertiary creep. All specimens annealed 1 hr at 1327° C to average grain diameter of 27 μm.

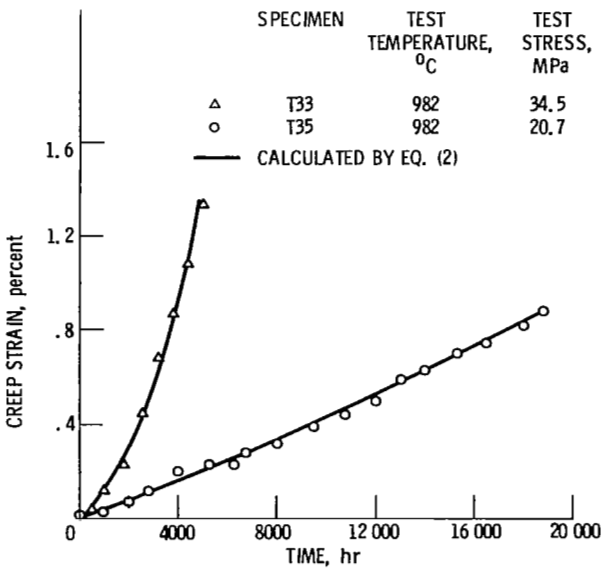

Figure 2. - Typical creep curves for C-103 tested at 982° C, showing only tertiary creep. Both specimens annealed 3 hr at 1593° C to average grain diameter of 75 μm.

Relation of Tertiary Creep Rate to Stress, Temperature, and Grain Size

In determining the effects of stress, temperature, and grain size on tertiary creep, the initial tertiary creep rate *Ks* was used rather than the rate parameter *s*. Stresses were compensated for modulus by dividing the applied stress by the modulus for unalloyed niobium at the creep temperature (ref. 11), since dynamic moduli data for C-103 are not available. It is expected that the moduli for C-103 are close to those for unalloyed niobium.

The initial tertiary creep rate *Ks* was found to vary as a power function of the stress modulus ratio, as shown in figures 4 and 5. The exponent *n* was determined to be 2.87 and did not vary significantly with stress, temperature, or grain size. The proximity of the stress exponent to 3 suggests that C-103 behaves like a Class I solid solution (ref. 12). The rate-controlling reaction for secondary (linear) creep for this type of alloy is considered to be solute viscous drag on gliding dislocations and is termed microcreep. Although C-103, under the conditions of this study, showed only accelerating creep, a similar type of reaction may be rate controlling.

The initial tertiary creep rate was also found to vary exponentially with inverse temperature, as shown in figure 6. The apparent activation energy was determined from the slope of figure 6 to be 374 kJ/g mol and did not vary with stress, temperature, or grain size. This activation energy is close to the 402 kJ/g mol determined for self-diffusion in niobium (ref. 13) and is consistent with solute-drag-controlled creep.

The effects of grain size were evaluated at 982° C and 48.3 MPa. The variation of the initial tertiary creep rate *Ks* with grain size under these conditions is shown in figure 7. The finest-grained material, with an average grain diameter of 15 μm, had a significantly higher creep rate than materials with grain sizes from 27 to 83 μm. Based on previous observations of the variation of creep rate with grain size for niobium and tantalum alloys (refs. 5, 6, and 9), it was assumed that the creep rate tended toward a $1/d^2$ variation with grain size at small grain sizes but became constant at large grain sizes.

TABLE I. - CREEP DATA FOR NIOBIUM ALLOY C-103

Test	Annealing conditions		Test temperature, °C	Stress, MPa	Stress-modulus ratio, σ/E	Initial tertiary creep rate, Ks, sec^{-1}	Tertiary creep strain parameter, K	Tertiary creep rate parameter, s, sec^{-1}	Time to 1 percent strain, hr	Grain diameter, d, μm	Test duration	
	Time, hr	Temperature, °C									Time, hr	To strain, percent
11	1	1204	982	48.3	4.61×10^{-4}	17.9×10^{-10}	0.299×10^{-2}	60.0×10^{-8}	670	15	818	1.36
7	1	1327	827	138	13.41	1.67	.101	16.5	3 960	22	4 867	1.81
5	↓	↓	927	55.2	5.30	3.68	.230	16.0	2 980	26	4 897	3.37
6			927	82.7	7.94	8.47	.224	37.8	1 260	24	2 034	3.17
18			977	41.4	3.96	6.34	.396	16.0	2 210	27	2 493	1.24
3			977	50.1	4.79	8.04	.112	71.8	876	31	982	1.28
4			977	60.1	5.75	5.66	.063	89.9	867	29	960	1.30
17			1027	20.7	1.97	2.68	.134	20.0	2 930	24	3 016	1.10
23			1027	41.4	3.95	31.5	.884	35.6	545	30	1 294	3.35
9			1027	55.2	5.26	66.4	.481	138	229	27	433	3.55
13	5	1427	982	48.3	4.61	11.2	.354	31.5	1 135	42	1 387	1.42
14	1	1538	982	48.3	4.61	11.2	.531	21.0	1 330	49	2 353	2.59
38	3	1593	927	68.9	6.62	1.64	.075	21.8	3 370	80	3 931	1.44
30			954	34.5	3.31	1.39	.177	7.88	6 467	73	6 575	1.01
35			982	20.7	1.98	1.07	1.89	.565	[a]20 800	77	18 873	.96
33			982	34.5	3.30	2.83	.283	10.0	4 125	65	5 041	1.39
61			982	48.3	4.61	9.68	.410	23.6	1 440	70	1 717	1.30
36			1002	27.6	2.63	3.91	.983	3.98	5 200	62	5 230	1.06
50			1002	34.5	3.29	11.9	1.51	7.89	1.820	85	2 013	1.18
37			1052	17.2	1.64	2.10	.733	2.86	8 215	74	9 237	1.14
32			1093	13.8	1.32	3.92	.847	4.63	4 630	71	4 748	1.07
51			1093	17.2	1.64	14.6	2.42	6.05	1 524	86	1 790	1.25
39			1093	34.5	3.29	81.2	.812	100	228	70	295	1.62
34			1149	10.3	.984	7.85	6.89	1.14	3 245	69	3 715	1.27
52			↓	13.8	1.32	20.9	1.73	12.1	1 052	85	1 827	2.10
49				20.7	1.98	43.6	1.38	31.6	510	83	1 030	3.04
40				34.5	3.30	262	2.06	127	89	80	240	4.11
45			1204	6.89	.661	4.76	1.83	2.60	4 630	80	5 066	1.10
44	↓	↓	1204	10.3	.998	15.0	.665	22.6	1 200	72	1 415	1.32
29	3	1649	871	89.6	8.65	1.38	.305	4.51	8 975	71	9 023	1.02
28	↓	↓	927	62.1	5.97	6.54	.873	7.49	2 790	77	3 861	1.67
27			982	41.4	3.95	8.38	.755	11.1	2 030	85	2 275	1.18
26			1093	20.7	1.97	15.8	1.04	15.2	1 170	92	1 676	1.57
16	1	1760	982	48.3	4.61	11.4	.857	13.3	1 600	83	2 541	2.12
15	1	1649	982	48.3	4.61	14.8	.467	31.6	1 060	63	1 153	1.19

[a]Estimated.

Based on these observations of C-103 and prior observations of other refractory alloys (refs. 5, 6, and 9), the relations for initial tertiary creep rate as a function of stress, temperature, and grain size is expressed as

$$Ks = A(\sigma/E)^n(1 + f/d^2)e^{-Q/RT} \quad (4)$$

Values for the constants in equation (4) are given in table II. The plot of temperature- and grain-size-compensated initial tertiary creep rate as a function of stress-modulus ratio in figure 8 shows a reasonable fit to equation (4).

The strain parameter K was also studied and was determined to vary as a power function of stress and grain size and an exponential function of inverse

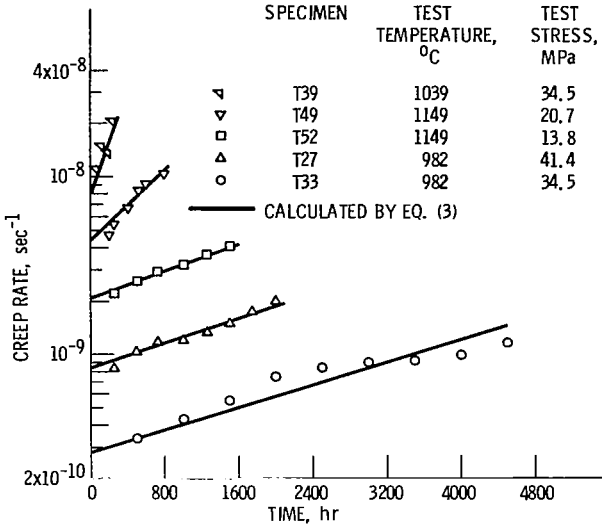

Figure 3. - Creep rates as a function of time. Specimen T-27 annealed 3 hr at 1649° C; others preannealed 3 hr at 1593° C.

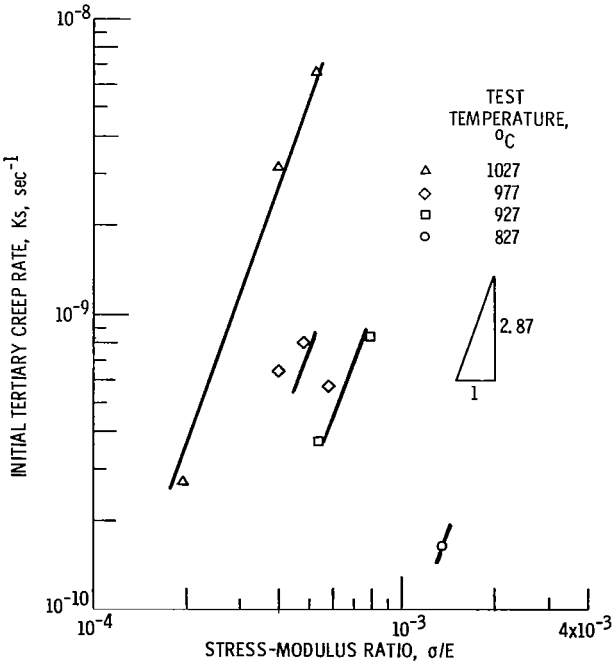

Figure 4. - Initial tertiary creep rate as a function of stress-modulus ratio for material annealed 1 hr at 1327° C to average grain diameter of 27 μm.

temperature. The relation for K is expressed as

$$K = A(\sigma/E)^a d^b e^{c/T} \quad (5)$$

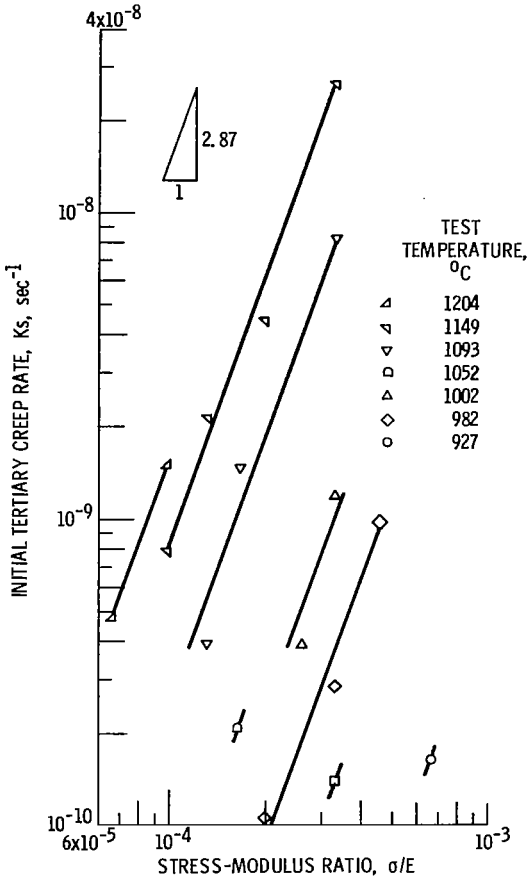

Figure 5. - Initial tertiary creep rate as a function of stress-modulus ratio for material annealed 3 hr at 1593° C to average grain diameter of 75 μm.

with the proportionality constant A determined by regression fitting to be 17.9 cm^{-b}; the exponents a and b to be -0.23 and 0.717, respectively; and c to be -7960 K^{-1}. Figure 9 compares calculated and observed values of K.

The variation of K with stress and temperature for C-103 is different from that observed previously for Astar 811C (ref. 9), although for both materials K increased with increasing grain size.

Comparison of Calculated and Experimental Creep Curves

It is of interest to compare calculated creep curves with experimental data. The strain parameter K can be calculated from equation (5); the rate parameter s can be obtained by dividing Ks from equation (4) by K. The creep curve can then be generated by inserting these calculated parameters into equation (2) and

TABLE II. - CREEP CONSTANTS FOR C-103

Constant	Equation	
	(4)	(6)
Proportionality constant, A, sec^{-1}	1.31×10^{16}	1.17×10^{17}
Stress exponents:		
n	2.87	---
n_1 (grain-size independent)	---	2.95
n_2 (grain-size dependent)	---	2.52
Grain-size factor, f, cm^2	1.2×10^{-6}	1.27×10^{-7}
Activation energy, Q, kJ/g mol	374	336
Correlation coefficient, R^2	0.962	0.988

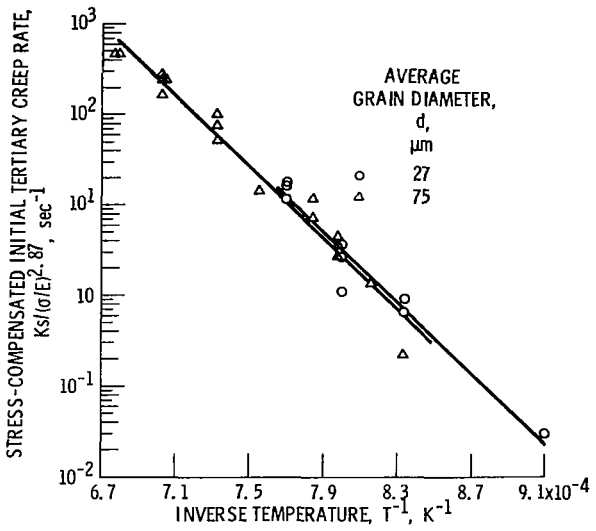

Figure 6. - Stress-compensated initial tertiary creep rate as a function of inverse temperature for C-103 of two different average grain diameters.

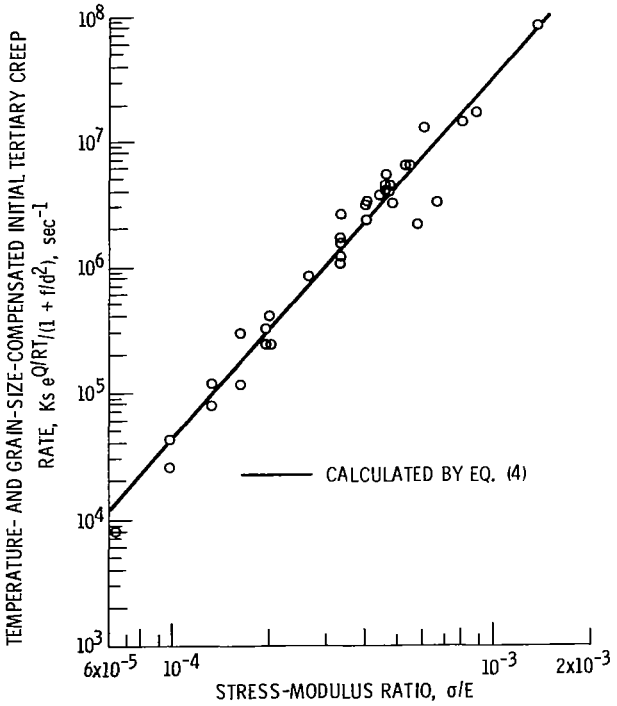

Figure 8. - Temperature- and grain-size-compensated initial tertiary creep rate as a function of stress-modulus ratio.

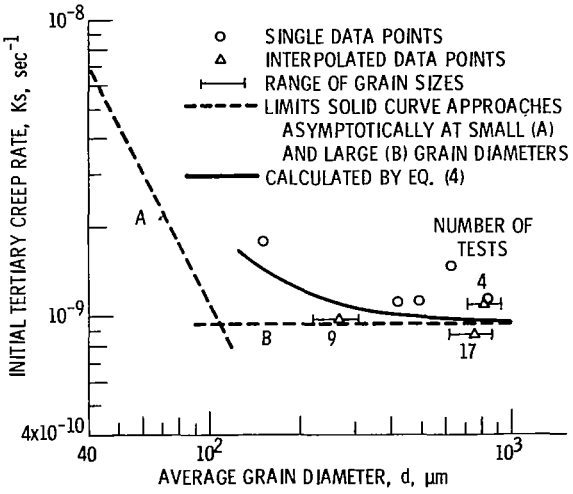

Figure 7. - Initial tertiary creep rate as a function of grain diameter for C-103 creep tested at 48.3 MPa at 982° C.

assuming ϵ_0 to be zero. Figure 10 and 11 compare calculated curves with experimental data points. The agreement is reasonable, especially considering the probable error in calculation of the strain parameter *K* as indicated by the scatter in figure 9.

Relation of Time to 1 Percent Strain to Stress, Temperature, and Grain Size

The time-to-1-percent-strain data were analyzed in a manner similar to that used for analyzing the initial tertiary creep rate *Ks*. The time to 1 percent strain varied as a power function of the applied stress; however, unlike for *Ks*, the stress exponent *n* for the time to 1 percent strain decreased from 2.97 to 2.75 as the grain diameter decreased from 75 to 27 μm. A decrease in *n* with decreasing grain size had been observed previously for Ta-10W (ref. 5), tantalum T-222 (ref. 6), and stainless steel (ref. 14). Matlock

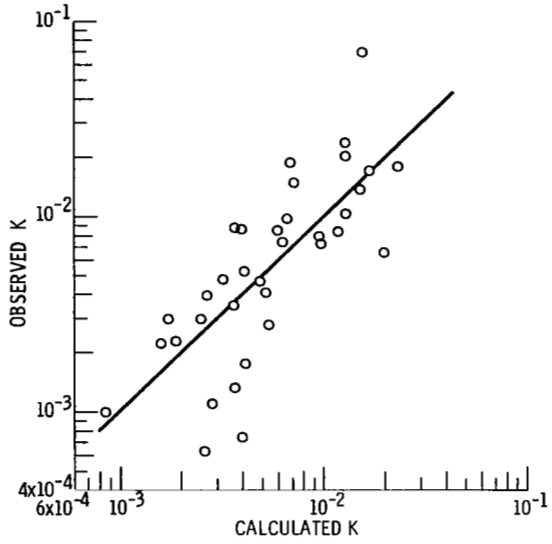

Figure 9. - Observed strain parameter K as a function of K as calculated from equation (5).

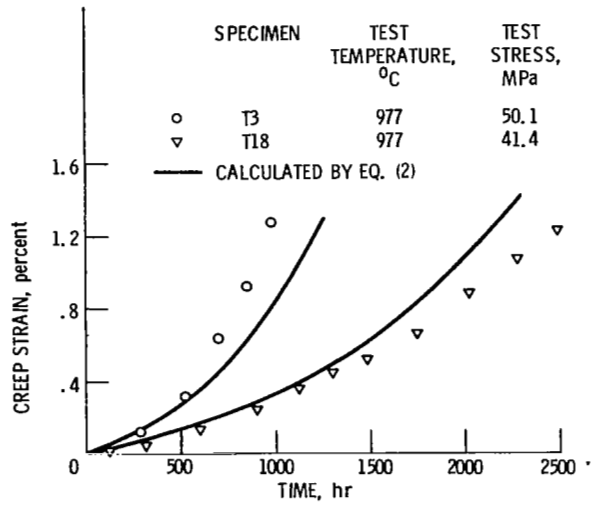

Figure 10. - Strain-time curves for C-103 annealed for 1 hr at 1327° C.

and Nix (ref. 15) also have suggested that the n for grain-boundary sliding, which predominates at small grain sizes, is about 1 less than the n for intragranular creep.

The apparent activation energy for time to 1 percent strain was determined to be 336 kJ/g mol, less than the 402 kJ/g mol for self-diffusion of niobium (ref. 13). The apparent activation energy did not vary significantly with stress, temperature, or grain size.

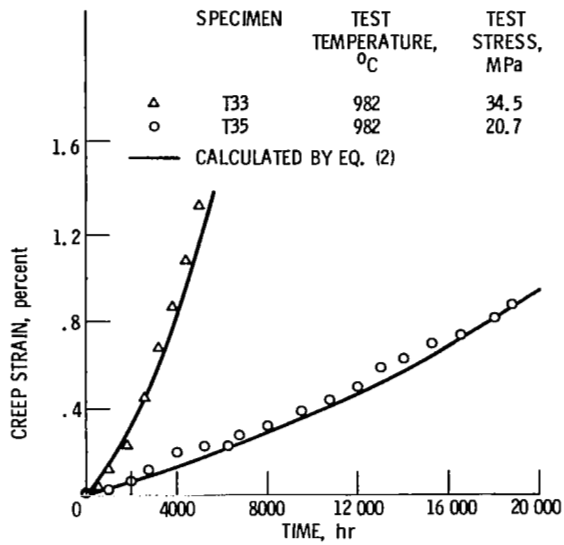

Figure 11. - Strain-time curves for C-103 annealed for 3 hr at 1593° C.

The time to 1 percent strain varied with grain size in a similar manner as observed for Ks. The inverse time (proportional to a linear creep rate to 1 percent strain) increased with decreasing grain size. The data were analyzed as done previously for Ks by assuming that the inverse time to 1 percent strain was proportional to $1/d^2$ at fine grain sizes but invariant at large grain sizes. The variation of inverse time to 1 percent strain with grain size is shown in figure 12.

A relation expressing time to 1 percent strain as a function of stress, temperature, and grain size was developed as

$$1/t_{1pct} = A\left[(\sigma/E)^{n_1} + f/d^2(\sigma/E)^{n_2}\right]e^{-Q/RT} \quad (6)$$

Values for the constants in equation (6) are given in table II. Here, two stress exponents are required since the stress dependency varies with grain size. Figure 13 shows the good correlation between temperature-compensated inverse time to 1 percent strain and stress for C-103 with average grain diameters of 27 and 75 μm.

The calculated stress for 1 percent creep strain in 10 000 hr for C-103 is shown in figure 14. This stress ranges from about 50 MPa a 900° C to about 6 MPa at 1100° C and is about 15 percent greater for coarse-grained than for fine-grained material. C-103 is about one-fourth as strong as the commonly used niobium alloy FS-85 (ref. 16).

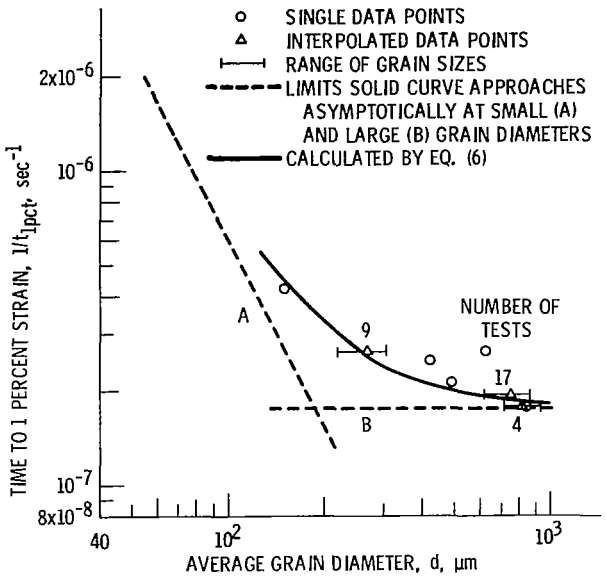

Figure 12. - Time to 1 percent strain as a function of grain diameter for C-103 creep tested at 48.3 MPa and 982° C.

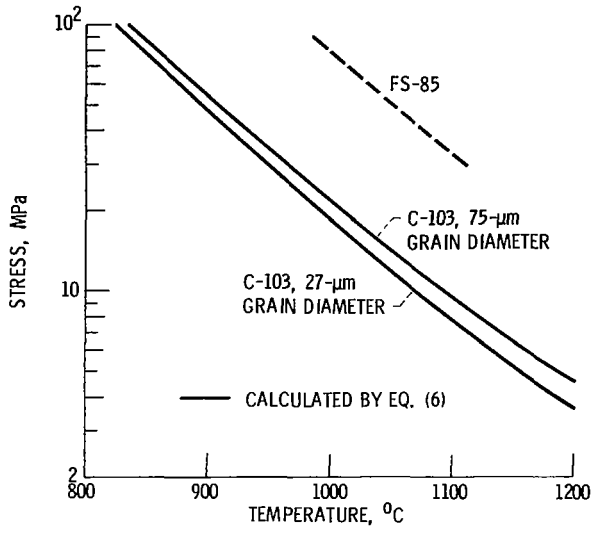

Figure 14. - Calculated stresses for 1 percent creep strain in 10 000 hr as a function of temperature for C-103 and FS-85.

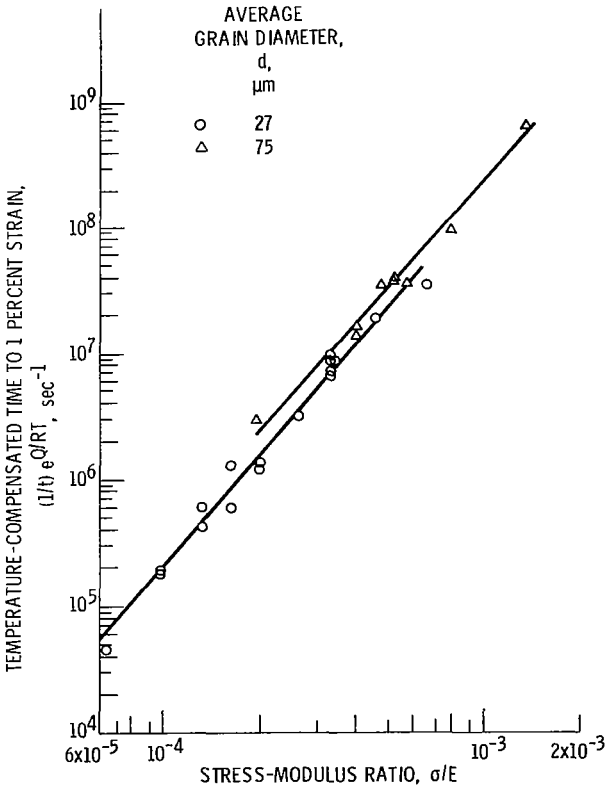

Figure 13. - Temperature-compensated time to 1 percent strain as a function of stress-modulus ratio for C-103 of two different average grain diameters.

Conclusions

From a study of the creep behavior of C-103 as a function of stress, temperature, and grain size the following conclusions were drawn:

1. The exponential relation for tertiary creep strain as a function of time can well describe creep curves for C-103, which shows only tertiary creep at 827° to 1204° C at stresses of 6.89 to 138 MPa. This relation is

$$\epsilon = \epsilon_0 + K(e^{st} - 1)$$

where ϵ is creep strain, ϵ_0 is initial creep strain, K is the tertiary creep strain parameter, s is the tertiary creep rate parameter, and t is time.

2. The initial tertiary creep rate Ks can be related to stress through the well-known power law. The rate Ks can be expressed by a single activation energy (over the range of this study) that is slightly less than the self-diffusion activation energy for niobium. Fine-grained C-103 creeps slightly faster than does coarse-grained C-103.

3. The strain parameter K can be expressed as a function of grain size, stress, and temperature.

4. Strain-time curves calculated from predicted values of K and s agree fairly well with observed strain-time curves.

Lewis Research Center,
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15. Supplementary Notes

16. Abstract

The creep behavior of C-103 (Nb-10Hf-1Ti-0.7Zr) has been studied as a function of stress, temperature, and grain size for test times to 19 000 hr. Over the temperature range 827° to 1204° C and the stress range 6.89 to 138 MPa, only tertiary (accelerating) creep was observed. The creep strain ϵ can be related to time t by an exponential relation recently developed by the authors: $\epsilon = \epsilon_0 + K(e^{st} - 1)$, where ϵ_0 is initial creep strain, K is the tertiary creep strain parameter, and s is the tertiary creep rate parameter. The initial tertiary creep rate Ks (the zero-time intercept of the differential of the strain-time relation) can be related to stress, temperature, and grain size by a modified power-law relation: $Ks = A(\sigma/E)^n(1 + f/d^2) \times e^{-Q/RT}$, where A is the proportionality constant, σ is stress, E is Young's modulus, f is the grain-size factor, d is grain size, Q is the activation energy, R is the gas constant, and T is temperature. The observed stress exponent 2.87 is similar to the three-power law generally observed for secondary (linear) creep of Class I solid solutions. The apparent activation energy 374 kJ/g mol is close to that observed for self-diffusion of pure niobium. The initial tertiary creep rate was slightly faster for fine-grained than for coarse-grained material. The strain parameter K can be expressed as a combination of power functions of stress and grain size and an exponential function of temperature. Strain-time curves generated by using calculated values for K and s showed reasonable agreement with observed curves to strains of at least 4 percent. The time to 1 percent strain was related to stress, temperature, and grain size in a similar manner as the initial tertiary creep rate.

17. Key Words (Suggested by Author(s))

Creep; Refractory metals; Niobium (columbium); C-103; Primary creep rate; Secondary creep rate; Tertiary creep rate; Grain size; Stress dependency; Activation energy

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