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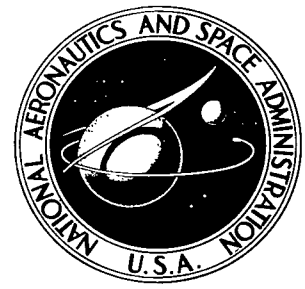

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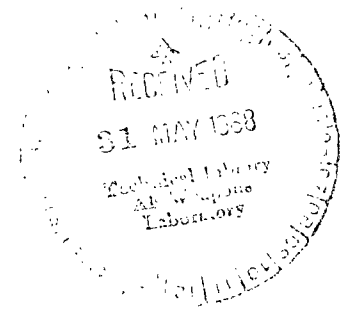
CREEP OF TANTALUM T-222 ALLOY IN ULTRAHIGH VACUUM FOR TIMES UP TO 10 000 HOURS

by Robert H. Titran

Lewis Research Center
Cleveland, Ohio



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ABSTRACT

The long-time creep behavior of fine-grained (0.016-mm) Ta-9.11W-1.91Hf-0.01C was determined over the temperature range of 2000° to 2600° F (1093° to 1426° C) and in a vacuum of 10^{-8} torr (1.33×10^{-6} N/m^2). Creep stresses ranged from 2500 to 20 000 psi (17.5×10^6 to 138×10^6 N/m^2) for test times up to 10 000 hours. At temperatures ranging from 2000° to 2600° F (1093° to 1426° C), stress levels to limit creep to 1 percent in 10 000 hours are 28 000 and 800 psi (193×10^6 and 6×10^6 N/m^2), respectively. The stress dependence of the steady creep rate was 3, and the temperature dependence of steady creep was equivalent to an apparent activation energy of 150 kcal/g-mole (627 450 J/g-mole).

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CREEP OF TANTALUM T-222 ALLOY IN ULTRAHIGH VACUUM FOR TIMES UP TO 10 000 HOURS

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SUMMARY

The creep behavior of one heat of fine-grained (0.016-mm), recrystalized tantalum T-222 alloy, tantalum (Ta) - 9.11 tungsten (W) - 1.91 hafnium (Hf) - 0.01 carbon (C), was characterized in the temperature range of 2000° to 2600° F (1093° to 1426° C) at stress levels of 2500 to 20 000 psi (17.5×10^{6} to 138×10^{6} N/m^2). Creep tests were conducted at low pressures, generally in the region of 10^{-8} to 10^{-9} torr (1.33×10^{-6} to 1.33×10^{-7} N/m^2), for times up to 10 000 hours.

For conceptual design purposes, an estimate of the maximum allowable stress levels to limit total creep strain to 1 percent in 10 000 hours is desired. These stress levels range from 28 000 psi (193×10^{6} N/m^2) at 2000° F (1093° C) to 800 psi (6×10^{6} N/m^2) at 2600° F (1426° C).

The stress dependence of the steady-state creep rate $\dot{e}_s$ follows the power relation $\dot{e}_s = A\sigma^n$, where A is a constant, σ is the engineering stress, and n is the stress dependence of the creep rate and is equal to 3. The apparent activation energy for creep is 150 kilocalories per gram-mole (627 450 J/g-mole). Extensive precipitation of carbides during creep testing probably accounts for the high activation energy and suggests that a microcreep mechanism is rate controlling.

INTRODUCTION

Advanced space-electric-power systems, such as those of the nuclear turbogenerator type, will require the use of readily fabricated refractory materials for various components. In the Rankine cycle system, for example, alloys of tantalum and/or columbium are of interest as tubing for the containment of liquid alkali-metal working fluids. The tubing material will be subjected to stress levels determined primarily by the vapor pressure of the working fluid in the temperature range of 1800° to 2400° F (982° to 1316° C) for times of 10 000 hours or longer. Therefore, creep resistance is a critical design parameter.

Preliminary studies to evaluate the long-time creep strength of candidate materials at elevated temperatures in a high vacuum showed that the tantalum T-222 alloy is the most creep resistant of the readily fabricated columbium- and tantalum-base alloys on a density-compensated basis (ref. 1). (Fabricable is understood herein to mean the ability to be drawn into thin-wall tubing by conventional methods.) This report describes the results of further creep studies conducted on the T-222 alloy in the temperature range of 2000° to 2600° F (1093° to 1427° C). The purpose of this study is to provide preliminary engineering design data and to characterize the temperature and stress dependency of creep for this alloy.

SYMBOLS

A, c	constants
$\dot{e}_s$	steady creep rate, sec^{-1}
ΔH_C	apparent activation energy for creep, kcal/g-mole; J/g-mole
ΔH_D	activation energy for self-diffusion, kcal/g-mole; J/g-mole
n	stress dependence of creep rate
R	gas constant, 1.987 cal/g-mole
T	temperature, K
t	time, sec
σ	engineering stress, psi; N/m^2

MATERIALS

The material evaluated in this study was procured from a commercial vendor in the form of sheets 0.030 inch (0.762 mm) thick and 14 inches (356 mm) long by 10 inches (254 mm) wide. This heat of the material was prepared by first double-electron-beam melting a tantalum-plus-tungsten master alloy. Hafnium and carbon were added during the remelting of the master alloy by the vacuum-consumable, direct-current, arc-furnace process. Extrusion was used as the primary breakdown method.

The sheet was cold worked approximately 85 percent following the last in-process anneal. Prior to shipping, the sheet was given a stress-relief anneal for 2 hours at 2400° F (1315° C).

The composition, in weight percent, of the as-received sheet, as determined by chemical analysis, is given in the following table:

Tantalum	Tungsten	Hafnium	Carbon	Oxygen	Nitrogen	Hydrogen
Balance	9.11	1.91	0.0113	0.0045	0.0014	0.0002

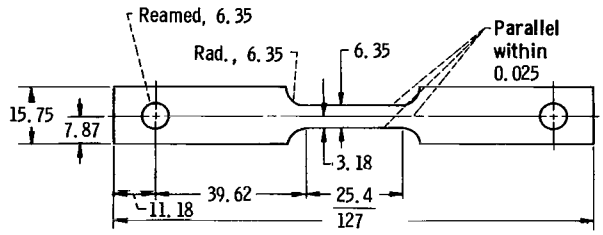


Figure 1. - Standard sheet creep-test specimen. (All dimensions are in millimeters.)

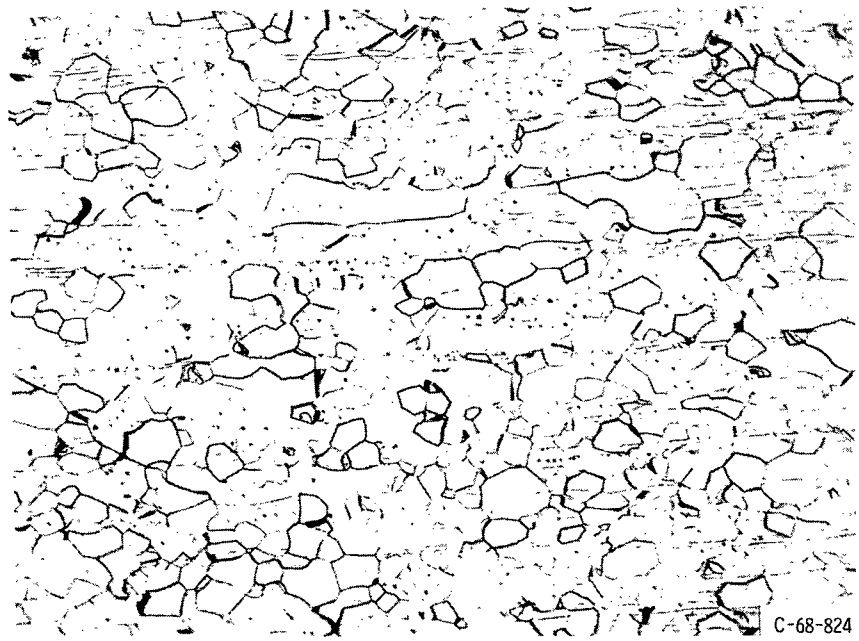


Figure 2. - Microstructure of tantalum T-222 alloy material prior to creep testing. Annealed 1 hour at 2800° F (1538° C) at 10^{-6} torr (1.33×10^{-4} N/m^2); etchant, 50 milliliters nitric acid, 20 milliliters water, 30 grams ammonium bifluoride; X250.

Creep specimens with a 1-inch (25.4-mm) gage length and a 0.250-inch (6.35-mm) gage width, as shown in figure 1, were machined from the as-received sheet with the specimen axis parallel to the final rolling direction. These specimens were annealed prior to testing for 1 hour at 2800° F (1538° C) in a vacuum of 10^{-6} torr (1.33×10^{-4} N/m^2) to give a fine-grained (0.016-mm) equiaxed structure, as shown in figure 2. All creep specimens were weighed to the nearest 0.1 milligram before and after the annealing to determine whether or not any possible contamination had resulted from the heat treatment. In all cases, no significant weight changes were observed; that is, the total contamination observed amounted to a maximum of only 15 ppm by weight.

APPARATUS AND PROCEDURE

The creep facilities used in this study are described in detail in references 2 and 3. The major features of one ultrahigh-vacuum creep unit are shown in figure 3.

The procedure for preparing the sheet specimens for creep testing is similar to that previously described in reference 1. Briefly, Knoop hardness impressions placed 1.0 inch (25.4 mm) apart served as fiducial marks. Three platinum-13-percent-rhodium - platinum thermocouples were equally spaced along the reduced gage section and tied with tantalum wire. The temperature gradient along the length of the gage section was less than 4° C. The reduced gage section and the thermocouples were shielded with tantalum foil in which fiducial-mark sight holes were cut.

Following the insertion of the specimen into the creep chamber, the direct-weight load was supported on a pedestal. The chamber was checked for leaks with a helium mass spectrometer, and the chamber and pump were baked at approximately 650° F (350° C) for 2 days. After the bakeout, the ultimate pressure was generally in the low 10^{-9}-torr (10^{-7}-N/m^2) regime. The specimen was then heated to the test temperature at a controlled rate so that the pressure never exceeded 5×10^{-7} torr (6.65×10^{-5} N/m^2). Generally, the pressure was in the low 10^{-8}-torr (10^{-6}-N/m^2) range 20 hours after the test temperature was reached. Temperature stability, as indicated by the thermocouples, was checked by periodically measuring the surface brightness temperature with a micro-optical pyrometer and by monitoring the heater power input. The temperature was held within ±3° F (±2° C) of the desired temperature for the duration of the test.

Creep-Strain Measurement

Specimen strain was measured optically with a cathetometer that was clamped to the vacuum chamber frame. The precision of creep-strain measurements is estimated to

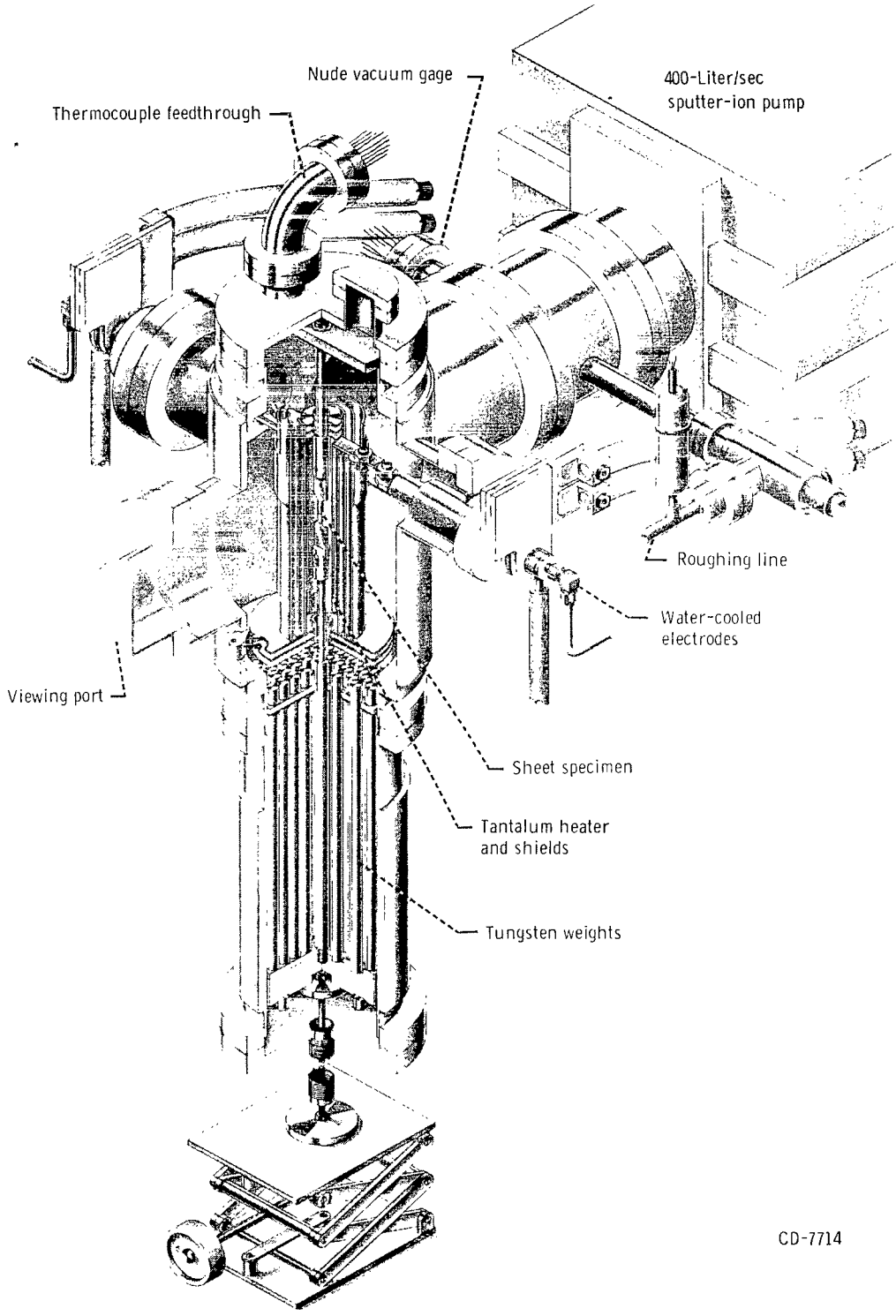


Figure 3. - Cutaway drawing of ultrahigh-vacuum creep unit.

be ±0.02 percent for the nominal 1-inch (25.4-mm) gage length used. In all instances, an initial gage length was read at the test temperature prior to the loading of the specimen. The strain on loading was measured and is incorporated in the reported total creep strain. Generally, this initial strain was less than 0.05 percent. Testing was usually terminated after a total creep strain of 1 or 2 percent was achieved.

Post-Test Examination

After the creep tests, the specimens were reweighed and hardness readings were taken on the major surface. Sections were sheared from the gage length for both metallographic and chemical analysis. Data from the chemical analysis were determined primarily to indicate the extent of any changes that occurred in the oxygen content during testing.

RESULTS AND DISCUSSION

Creep Curves

Creep curves for the fine-grained T-222 alloy tested at 2000° F (1093° C), 2200° F (1204° C), 2400° F (1315° C), and 2600° F (1426° C) are shown in figures 4 to 7, respectively. These curves are unusual in that no primary stage of creep was observed. Similar curves have been reported previously for columbium alloys (ref. 3) at high temperatures and low stress levels. The absence of a primary creep stage suggests that,

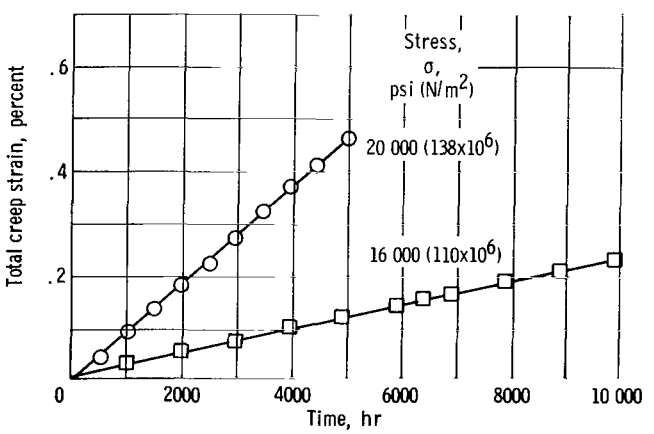


Figure 4. - Creep curves for annealed tantalum T-222 alloy at 2000° F (1093° C).

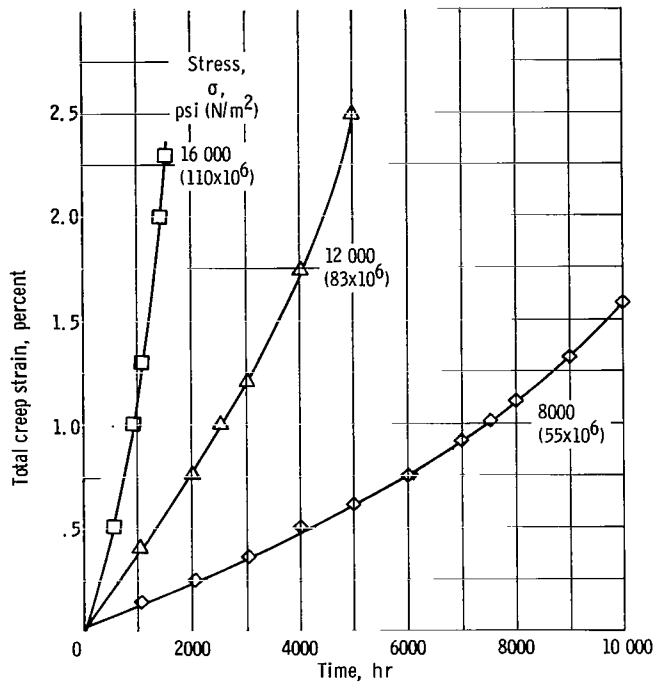


Figure 5. - Creep curves for annealed tantalum T-222 alloy at 2200° F (1204° C).

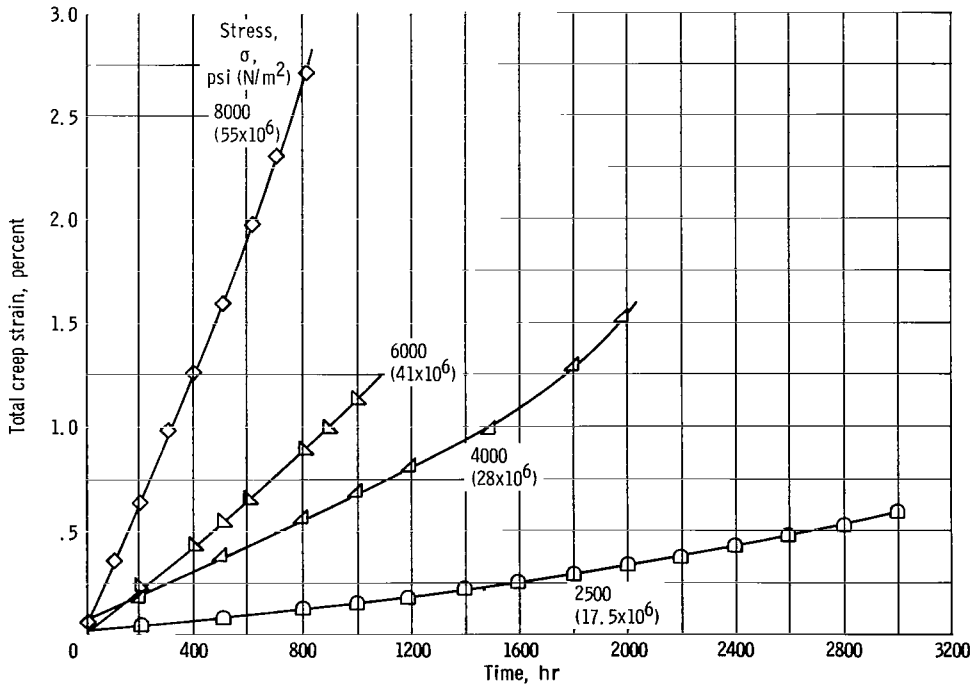


Figure 6. - Creep curves for annealed tantalum T-222 alloy at 2400° F (1315° C).

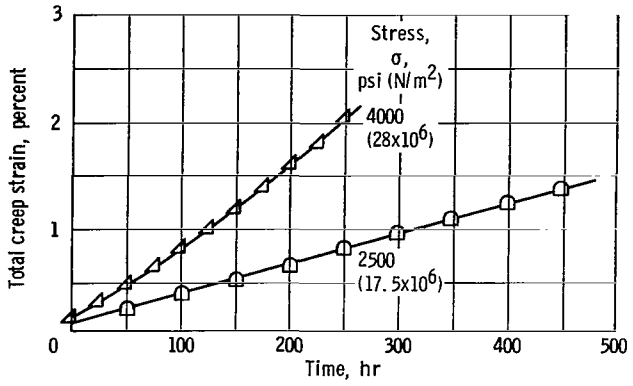


Figure 7. - Creep curves for annealed tantalum T-222 alloy at 2600° F (1426° C).

for the combination of very low creep rates and relatively high test temperatures investigated, any work hardening accompanying the initial stage of deformation is rapidly recovered.

Steady creep rates were calculated from the linear portions of these creep curves. Generally, the linear portion of the creep curve extended from the start of the test to that point where at least 1 percent strain was attained. In some instances, 1 percent strain was not achieved prior to the termination of the test, or the creep rate increased only slightly during the test.

In view of the linearity of the creep curves from the start of the tests, the steady creep rate and the time to achieve 1 percent total strain may be represented by the relation (ref. 4)

$$\dot{e}_s t_1 = 0.01$$

where

$\dot{e}_s$ steady creep rate, sec^{-1}

t_1 time to reach 1 percent strain, sec

In figure 8, a plot of the time required for 1 percent strain against the creep rate shows this linear relation. For the material investigated, a steady creep rate of 2.7×10^{-10} per second will limit creep to 1 percent in 10 000 hours.

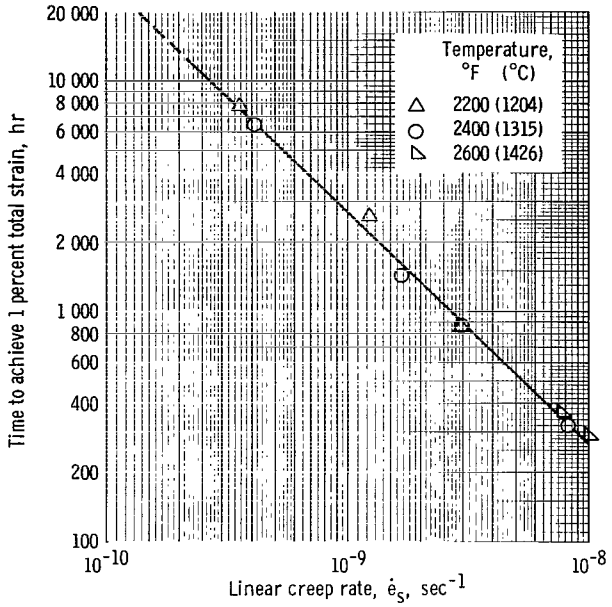


Figure 8. - Relation between time required to achieve 1 percent total creep strain and linear creep rate for fine-grained, recrystallized tantalum T-222 alloy.

Stress and Temperature Dependency

The steady creep rates can be related to stress and temperature by the relation (ref. 5)

$$\dot{e}_s = A\sigma^n \exp\left(\frac{-\Delta H_C}{RT}\right)$$

where

A	structure constant
σ	engineering stress, psi; N/m^2
n	exponential stress dependence
ΔH_C	apparent activation energy for creep, kcal/g-mole; J/g-mole
R	gas constant, 1.987 cal/g-mole
T	temperature, K

TABLE I. - TANTALUM T-222 CREEP RATES AND TIMES TO ACHIEVE 1 PERCENT STRAIN

[Vacuum environment, 10^{-8} torr (1.33×10^{-6} N/m^2).]

Temperature		Stress, σ		Creep rate, $\dot{e}_s$, sec^{-1}	Time to achieve 1 percent strain, hr
^oF	^oC	psi	N/m^2		
2000	1093	16 000	110×10^6	4.8×10^{-11}	(a)
2000	1093	20 000	138	1×10^{-10}	(b)
2200	1204	8 000	55	3.6×10^{-10}	7500
2200	1204	12 000	83	1.2×10^{-9}	2600
2200	1204	16 000	110	2.8×10^{-9}	880
2400	1315	2 500	17.5	4.0×10^{-10}	5100
↓	↓	4 000	28	1.6×10^{-9}	1500
		6 000	41	3.8	880
↓	↓	8 000	55	8.1	320
2600	1426	2 500	17.5	7.7	330
2600	1426	4 000	28	1.8×10^{-8}	135

[a]Test terminated after 10 000 hr with 0.22 percent strain.

[b]Test in progress; 0.45 percent strain at 5100 hr.

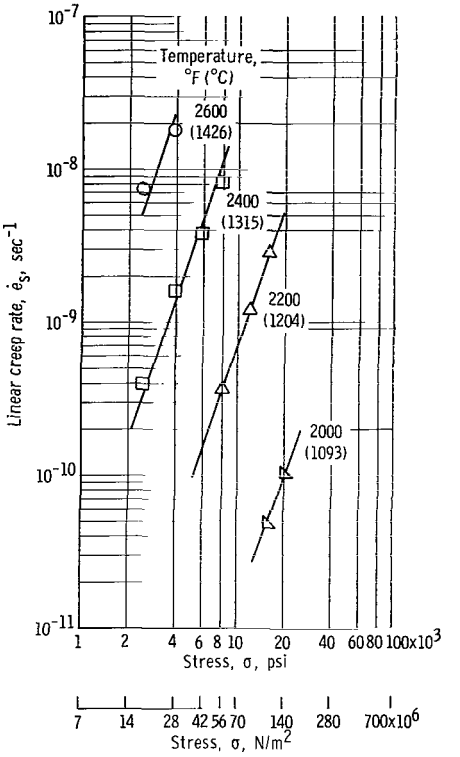


Figure 9. - Stress dependency of linear creep rate for fine-grained, recrystallized tantalum T-222 alloy in temperature range of 2000° to 2600° F (1093° to 1426° C).

Table I gives the creep test conditions, the resulting creep rates, and the time required to achieve 1 percent strain. A log-log plot of the steady creep rate as a function of stress is shown in figure 9. The slope of these curves is 3, the probable significance of which is discussed in the section Creep-Strengthening Mechanism.

With the determination of the stress dependency and the linear relation of creep rate and time for 1 percent strain, the engineering design data sought are readily available. In figure 10, a plot is presented of the maximum stress level necessary to limit total strain to 1 percent in 10 000 hours at the temperatures of interest. At 2000° F (1093° C), this stress is 28 000 psi (193×10^6 N/m^2). At higher temperatures, the limiting stress level decreases rapidly to 7400 psi (51×10^6 N/m^2) at 2200° F (1204° C); 2400 psi (17×10^6 N/m^2) at 2400° F (1315° C); and 800 psi (6×10^6 N/m^2) at 2600° F (1426° C).

On the basis of the observed creep rates, at temperatures of about 2400° F (1315° C) and above, the T-222 alloy does not appear to be suitable for the containment of liquid alkali-metal in lightweight systems. It is realized that the fine-grained, recrystallized structure evaluated in this study is probably not the optimum metallurgical structure for maximum creep strength. Studies of the effects of microstructure and heat treatment on the creep properties of the T-222 alloy are currently being conducted.

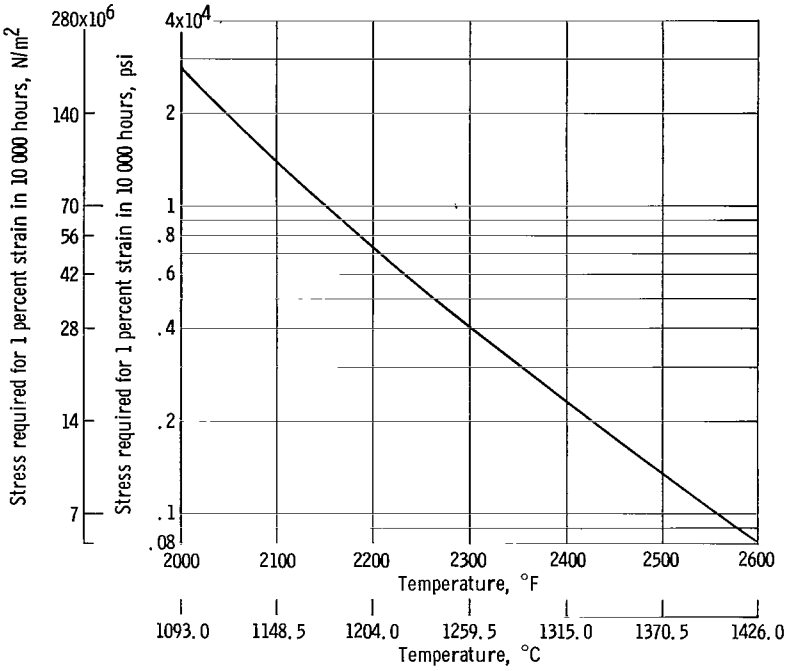


Figure 10. - Effect of temperature on allowable stress level required to achieve 1 percent total strain in 10 000 hours for fine-grained, recrystallized tantalum T-222 alloy.

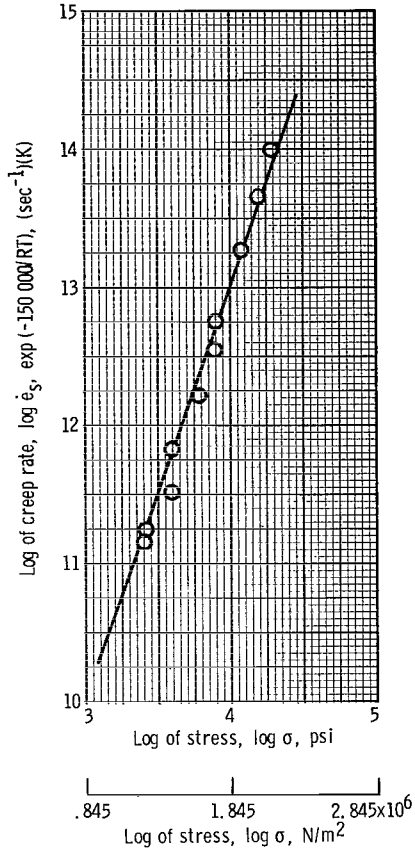


Figure 11. - Temperature-compensated linear creep rate as function of stress for fine-grained, recrystallized tantalum T-222 alloy.

The absence of a primary creep stage in the creep curves suggests that the microstructure is relatively independent of temperature during steady creep. If this is the case, an apparent activation energy for creep may be calculated from the data of these creep tests. A calculated value of 150 kilocalories per gram-mole (627 450 J/g-mole), the apparent activation energy, was used in the log-log plot of figure 11, which presents the temperature-compensated creep rate as a function of stress. The significance of this rather high activation energy, as compared with that for the self-diffusion of tantalum, is discussed in the following section.

Creep-Strengthening Mechanism

Evident from the stress- and temperature-dependency data is the fact that the T-222 alloy exhibits remarkable long-time creep strength in comparison with other tantalum-

and columbium-base alloys. In the consideration of the possible strengthening mechanisms for this alloy, two features of the creep behavior are of prime importance. The first is the stress-dependency factor of 3, and the second is the temperature dependence, which is equivalent to an apparent activation energy of 150 kilocalories per gram-mole (627 450 J/g-mole).

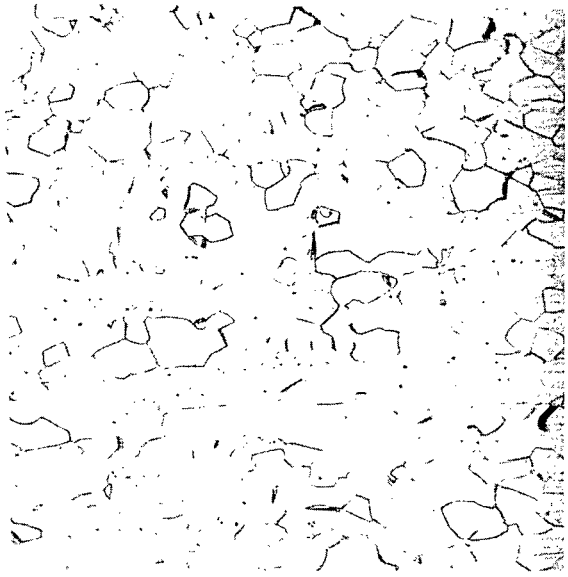
The stress-dependency factor of 3 is significantly smaller than the values of 4 to 5 that are associated with the creep controlled by dislocation climb. However, Weertman (ref. 6) proposed various "microcreep" mechanisms that have a stress dependency of 3 and the common feature that dislocation motion in the slip plane is rate controlling (as opposed to dislocation climb out of the slip plane). Dislocation mobility in the slip plane can be reduced by solute atoms, which drag on the dislocation line, by a preexisting fine particle that pins the dislocation, or by the precipitation of a particle directly on the migrating dislocation. In the present alloy, it seems reasonable that the hafnium carbide (HfC) particles, precipitated either prior to or during dislocation migration, are reducing dislocation mobility in the slip planes and giving rise to the stress-dependency factor of 3.

Weertman's model for microcreep further states that the activation energy for creep should be that for movement of the dragging atoms or the pinning particles in the slip plane. The observed activation energy of 150 kilocalories per gram-mole (627 450 J/g-mole) is significantly larger than that for self-diffusion in tantalum, 110 kilocalories per gram-mole (460 130 J/g-mole) (ref. 7), and also larger than the 80 kilocalories per gram-mole (334 640 J/g-mole) for fine-grained Ta-10W alloy (estimated from unpublished data). The significant difference in activation energies would suggest that solute dragging is not the rate-controlling microcreep reaction in T-222. However, large activation energies are commonly observed in the creep of precipitation-strengthened alloys, and Conrad, et al. (ref. 8) considered the reactions involved and concluded that the creep activation energy for this type of alloy includes a term related to volume diffusion in the alloy plus a term related to the temperature dependency of the precipitation reaction. Thus, the creep activation energy would be higher than that for self-diffusion.

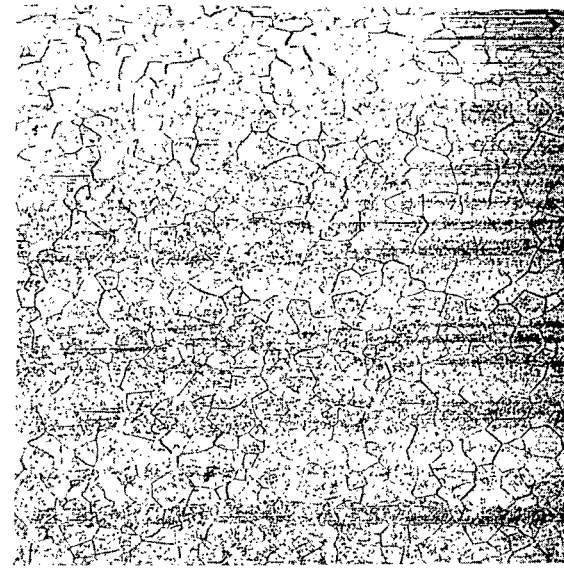
In summary, the probable rate-controlling mechanism for creep in T-222 is the precipitation of HfC particles (or coherent clusters) on the dislocation lines during creep straining. These particles prevent the dislocation loops from expanding into the stress fields of other loops, where they would affect recovery by climbing toward and annihilating each other.

Metallography

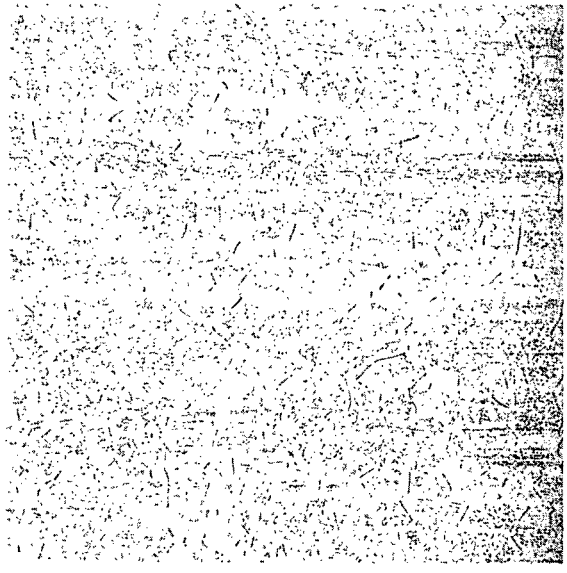
Photomicrographs of representative creep-tested specimens (fig. 12) show that extensive precipitation took place during testing. The photomicrographs also show that the



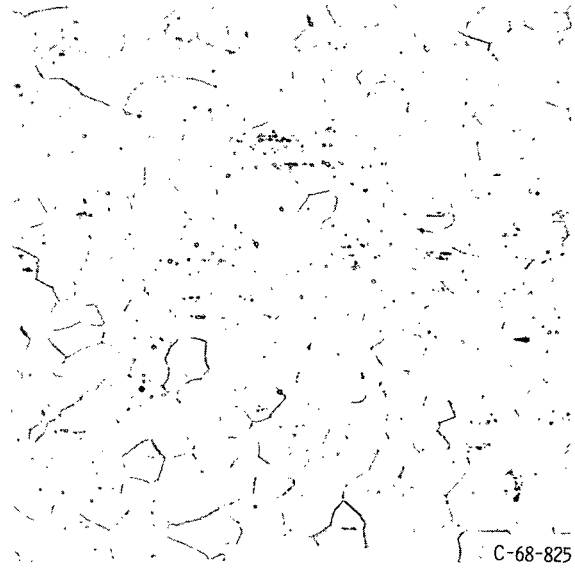
(a) As annealed.



(b) Creep tested at 2000° F (1093° C) and 16 000 psi (110×10^6 N/m^2) for 10 000 hours.



(c) Creep tested at 2200° F (1204° C) and 12 000 psi (83×10^6 N/m^2) for 5062 hours.



(d) Creep tested at 2400° F (1315° C) and 8000 psi (55×10^6 N/m^2) for 740 hours.

Figure 12. - Microstructure of annealed and creep-tested tantalum T-222 alloy specimens. Annealed 1 hour at 2800° F (1538° C) at 10^{-6} torr (1.33×10^{-4} N/m^2); etchant, 50 milliliters nitric acid, 20 milliliters water, 30 grams ammonium bifluoride; X250.

size of the precipitate increased with increasing test temperature. The precipitates are uniformly dispersed throughout the matrix, except at the higher temperatures where some particles are found in the grain boundaries and at triple points. Unfortunately, one creep specimen cannot be directly compared with another, since in all cases, the temperatures, stress levels, and test times varied considerably. It should be noted that the particles shown in figure 12 are not those believed to be responsible for the observed high creep strength; rather, unresolvable coherent particles are believed to be responsible for the high creep strength.

Results of Vacuum Fusion Analysis for Oxygen

All specimens were analyzed for oxygen content to determine the extent of contamination that occurred during creep testing. Presented in table II are the results from a vacuum fusion analysis for oxygen, along with the pertinent test times, temperatures, and pressures. With one exception, the oxygen pickup during testing at 2000° F (1093° C), 2200° F (1204° C), and 2400° F (1315° C) was less than 100 ppm. For the 2600° F (1426° C) tests, oxygen pickup was substantially higher as the result of increased outgassing of furnace components. The effects of these small amounts of oxygen contamination on the observed creep rates are unknown but are believed to be small. The observed linearity of the creep curves up to at least 1 percent strain suggests the absence of any significant effects on creep rate due to contamination during testing.

TABLE II. - RESULTS OF VACUUM FUSION ANALYSIS FOR OXYGEN CONTAMINATION OF CREEP SPECIMENS AFTER TESTING

Temperature		Stress, σ		Time, hr	Start of test		End of test		Increase in oxygen content during testing, ppm[a]
					Pressure				
°F	°C	psi	N/m^2		torr	N/m^2	torr	N/m^2	
2000	1093	16 000	110×10^6	10 121	2.5×10^{-7}	3.33×10^{-5}	2.5×10^{-9}	3.33×10^{-7}	96
2200	1204	8 000	55	10 000	1.7×10^{-7}	2.26	6.1	8.1	162
2200	1204	12 000	83	5 062	1.2×10^{-7}	1.60	3.1	4.12	75
2200	1204	16 000	110	1 411	8.2×10^{-8}	1.10	3.3	4.38	53
2400	1315	4 000	27.5	1 966	3.2×10^{-7}	4.26	4.9	6.52	78
2400	1315	6 000	41	1 001	6.8	9.05	8.8	1.18	97
2400	1315	8 000	55	740	1.2	1.60	2.0×10^{-8}	2.66	55
2600	1426	2 500	17.5	460	2.0	2.66	3.8×10^{-8}	5.05	375
2600	1426	4 000	27.5	595	5.8	7.72	2.6×10^{-8}	3.46	235

[a]Original oxygen content was 45 ppm by weight.

SUMMARY OF RESULTS

A study of the long-time creep behavior of fine-grained (0.016-mm), recrystallized tantalum T-222 alloy in the temperature range of 2000° to 2600° F (1093° to 1426° C) and at stress levels from 2500 to 20 000 psi (17.5×10^{6} to 138×10^{6} N/m^2) yielded the following results:

1. The stress levels necessary to limit creep strains to 1 percent in 10 000 hours at 2000° F (1093° C), 2200° F (1204° C), 2400° F (1315° C), and 2600° F (1426° C) are 28 000, 7400, 2400, and 800 psi (193×10^{6}, 51×10^{6}, 17×10^{6}, and 6×10^{6} N/m^2), respectively.

2. The T-222 alloy exhibits steady creep from the onset of testing, and generally, up to at least 1 percent strain. A steady creep rate of 2.7×10^{-10} per second is equivalent to 1 percent creep strain in 10 000 hours.

3. The steady creep rate has an exponential stress dependency of 3. The temperature dependency for steady creep corresponds to an apparent activation energy of 150 kilocalories per gram-mole (627 450 J/g-mole). These data suggest that a microcreep mechanism, such as interactions of dislocations with precipitates, is rate controlling.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, March 4, 1968,
129-03-02-03-22.

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