9

IV. CREEP BEHAVIOR OF REFRACTORY ALLOYS IN ULTRAHIGH VACUUM*

K. D. Sheffler,[†] J. C. Sawyer,[†] and E. A. Steigerwald[†]

SUMMARY

Several Cb, Ta, Mo, and W base alloys have been creep tested with both constant and continuously increasing loads in a vacuum environment of $<1 \times 10^{-8}$ torr at temperatures and stresses chosen to provide between 1/2 and 1 percent creep in times up to 20 000 hours. Isostatic design data are presented in the form of a Larson-Miller plot for 1/2 or 1 percent creep life for each of these refractory alloys. Elevated temperature tension test data from the tantalum base alloy T-111 showed a strain aging reaction in the 1400° to 2200° F (760° to 1204° C) range which produced an unusual transient creep behavior. Analysis of the isostatic steady state creep data for this material showed that the minimum creep rate can be described by the expression

$$\dot{\epsilon} = 1.65 \times 10^9 \left[\sinh(6.6 \times 10^{-5} \sigma) \right]^{3.17} e^{-90\,000/RT}$$

in the temperature and stress ranges of 1600° to 2600° F (870° to 1427° C) and 500 to 45 000 psi (0.34×10^7 to 31.0×10^7 N/m²). These values of activation energy and stress exponent suggest that the isostatic steady state creep of T-111 is governed by a diffusion controlled microcreep mechanism rather than by nonconservative dislocation motion. The creep tests which were run on T-111 alloy with continuously increasing loads provided continuously increasing creep rates which were comparable to the isostatic steady state creep rates at equivalent stress levels. Methods have been developed for prediction of the 1 percent creep life under varying stress conditions from isostatic creep test data, and comparison of these predictions with the experimental results shows the dependence of analytical techniques on the character of the isostatic creep curves.

*Based on work done under NASA contracts NAS 3-2545 and NAS 3-9439.

[†]TRW Equipment Laboratories, Cleveland, Ohio.

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

INTRODUCTION

The exceptional elevated temperature strength of refractory alloys makes these materials ideally suited for use in space environments, where oxidation resistance is not a governing factor. The molybdenum base alloys TZC and TZM and the columbium alloys As-30 and CB-132 have therefore been considered for turbine components, while the tantalum and tungsten alloys are candidates for tubing or radioisotope capsule fabrication in space power systems. Long time creep strength of these materials is a critical design property under conditions where the partial pressure of reactive gases is extremely low. The creep strengths for these applications must be generated in ultrahigh vacuum to prevent contamination which would lead to erroneous results. The effects of testing in a good and poor vacuum are graphically illustrated by the comparison curves in figure IV-1. The work described in this paper was performed to develop creep data for molybdenum, tungsten, columbium, and tantalum alloys in an ultrahigh vacuum environment of less than 10^{-8} torr at temperatures and stresses chosen to provide 1/2 to 1 percent creep in times up to 20 000 hours. Screening tests were conducted on the molybdenum, columbium, tungsten, and some of the tantalum alloys, while a full series of design tests was run on the tantalum alloy T-111. Extensive creep testing is also in progress on ASTAR-811C, a relatively new dispersion strengthened tantalum base alloy developed at the Westinghouse Astronuclear Laboratory.

The consideration of T-111 for radioisotope encapsulation has added yet another dimension to the problem of service characterization for this alloy. This problem centers on the fact that the proposed fuel is an α -emitter, which means that after loading, the capsules are subjected to continuously increasing pressures because of the internal buildup of helium gas. In an effort to develop an understanding of the response of high temperature materials to continuously increasing stress, experiments were conducted under these conditions and compared to analytical predictions based on constant load data.

EXPERIMENTAL DETAILS

Creep Test Procedures

The creep program involved the testing of refractory alloys at temperatures ranging from 1100° to 3200° F (594° to 1760° C) and at stresses between 500 and 65 000 psi (3.4 to 448 MN/m^2). A combination of parameters was generally selected which would provide 1 percent total creep in 5000 to 20 000 hours. Two-inch

gage length, button-head bar-type specimens and double-shoulder, pin loaded, sheet-type specimens were used, respectively, for testing of plate and sheet-type materials. The orientation of the specimen with respect to the working direction is given in the following table:

Material form	Specimen axis parallel to
Disk forging	Radius
Plate	Extruding direction
Sheet	Rolling direction (except where indicated)
Tubing	Tube axis

The tubing was stressed parallel to the tube axis, with two flats being ground opposite one another to provide two webs in the gage section.

Both construction and operation of the ultrahigh vacuum creep test chambers and the service instruments in the laboratory have been described in detail elsewhere (refs. 1 and 2). The creep test procedure involved initial evacuation of the chamber to a pressure of less than 5×10^{-10} torr at room temperature, followed by heating of the test specimen at such a rate that the pressure never rose above 1×10^{-6} torr. Pretest heat treatments were performed in situ, and complete thermal equilibrium of the specimen was ensured by a 2 hour hold at the test temperature prior to load application. Pressure was always below 1×10^{-8} torr during the tests and generally fell into the 10^{-10} torr range as testing proceeded. Specimen extension was determined over a 2-inch gage length with an optical extensometer which measured the distance between two scribed reference marks to an accuracy of ± 50 microinches. Creep strains were calculated using the loaded gage length as the original length. Specimen temperature was established at the beginning of each test using a thermocouple. An optical pyrometer having a precision of $\pm 1^\circ$ F was calibrated against the thermocouple reading and was then used as the prime temperature reference throughout each test.

The response of T-111 alloy to variable stress conditions was studied by continuously increasing the load at various linear rates, starting at zero stress, while simultaneously measuring creep strain as a function of time. The steadily increasing load was achieved by using a motor driven screw to continuously feed lead shot at a uniform rate into a load pan attached to the bottom of the specimen. The weight of the shot added to the pan was monitored so that the exact load would be known at all times.

Characterization of Test Materials

Sources of the test materials and details of the available processing histories have been previously presented (ref. 3). Chemical analyses of each of the heats tested are shown in table IV-1, and pertinent details of the processing histories are summarized in table IV-2.

Molybdenum alloys. - TZM was evaluated in three forms: a bar (heat 7463), a conventionally processed disk forged at 2200° F (heat 7502), and a section of another disk which had a higher than normal carbon level and was both hot and warm forged to produce improved creep resistance (heat KDTZM-1175). All of these heats were evaluated primarily in the stress relieved condition. Photomicrographs of each of the materials (fig. IV-2) show that the bar material and the conventional forging were relatively fine-grained, while the special forging had a somewhat coarser structure. Electron micrographs of the two disk forgings (fig. IV-3) show a similar carbide morphology, but with a somewhat higher density of carbides in the higher carbon material.

TZC was also evaluated in three different forms. Two rolled plates were obtained with widely different drafting practices. One plate was given very small reductions on each pass and a high finishing temperature (heat M-80), while the other was given relatively large reductions and finished at a lower temperature (heat M-91). The third TZC plate was side forged in the 2400° F range from extruded bar stock (heat 4345).

Both the stress-relieved and recrystallized structures were studied in TZC. The two rolled plates had a uniform and relatively fine-grained microstructure in the stress-relieved condition, while the extruded and forged plate had a more irregular and somewhat coarser structure (fig. IV-4). The responses of the rolled plates to a 1-hour annealing treatment at 3092° F differed markedly; the plate from heat M-91, which was given large reductions, was fully recrystallized, while the M-80 plate was only partially recrystallized (fig. IV-5). The difference presumably results from the higher degree of residual cold work in the plate finished at lower temperatures.

Columbium alloys. - The columbium alloy AS-30 was evaluated in the as-received condition (rolled at 2100° F), while Cb-132M was annealed 1 hour at 3092° F (1700° C) prior to testing. Microstructures of these two alloys are shown in figure IV-6.

Tungsten alloys. - Both arc-melted and vapor-deposited tungsten were evaluated. The arc-melted material was received as 0.030-inch sheet and recrystallized for hours at 3200° F prior to testing. The vapor-deposited material was tested as a bar specimen machined from a thick tube wall. The W - 25 percent Re

was also tested as 0.030-inch sheet obtained from an arc-melted ingot. Microstructures of the tungsten alloys are presented in figures IV-7, IV-8, and IV-9. In the case of the vapor-deposited material (fig. IV-8) the gage section represents a creep tested structure, while the head section is characteristic of untested material.

Tantalum alloys. - Pure tantalum was tested in the form of tubing, while all of the other tantalum alloys tested were in the form of nominal-0.030-inch-thick cold rolled sheet. All of the tantalum alloys were tested in the fully recrystallized condition. Photomicrographs of the pure tantalum, Ta-10W, and ASTAR-811C are shown in figures IV-10 to IV-12. Electron micrographs showing the influence of annealing time and temperature on carbide morphology in the ASTAR-811C alloy are shown in figure IV-13.

Investigation of Strain Aging Behavior in T-111 Alloy

Because a complete characterization of mechanical properties was required for T-111 alloy, a more extensive test program was conducted to fully document the elevated temperature mechanical behavior of this material. Chemical analyses and room temperature mechanical properties of each heat tested are presented in table IV-3, while typical light and electron micrographs are shown in figures IV-14 and IV-15(a). Although the structure appears clean at optical magnifications, the electron micrographs show that the T-111 recrystallized 1 hour at 3000° F (1649° C) had an included second phase which was distributed preferentially at the grain boundaries. Electron probe micrographs emphasized this grain boundary network (fig. IV-16), but also showed a significant number of inclusions within the grains. Electron probe spectral distribution and emission scans indicated that these inclusions were hafnium oxides.

Elevated temperature tension tests were performed in a Brew furnace at pressures of the order of 10^{-5} torr at an extension rate of 0.005 inch per inch per minute (except where noted). The influence of temperature on the tensile properties of recrystallized T-111 is shown in figures IV-17. These overlapping data from several different heats as well as from the literature (ref. 4) show a pronounced ultimate strength peak near 1400° F, together with a maximum in the strain hardening exponent. In addition, a Portevin-LeChatlier effect (serrated stress-strain curve) was observed in this temperature range, as illustrated in figure IV-18. Schmidt et al. (ref. 5) have noted similar strain aging effects at a much lower temperature (about 600° F (316° C)) in pure tantalum and in tantalum containing between 225 and 955 ppm C, O, and N additions. Comparison of the T-111 data with Schmidt's results suggests that strain aging in T-111 may be caused by a complex atmosphere-

dislocation interaction rather than by simple interstitial dislocation pinning. Baird and Jamieson (ref. 6) have found the complex atmosphere to be a much more potent strengthener than straightforward interstitial atom strain aging in the analogous Fe-Mn-C system, especially at higher temperatures.

In order to study the strain aging phenomenon in greater detail and perhaps identify the responsible atom species, a series of tension tests was conducted on T-111 at different strain rates and temperatures in the strain aging range. Results of these tests, shown in figure IV-19, further emphasize the presence of the strain aging by showing a very large negative strain rate sensitivity in the strain aging temperature range. Two other interesting effects are also apparent in figure IV-19. First, strain age strengthening is much more effective at the lower strain rates, indicating that this factor may be important in creep behavior. Second, the strength peak shifts to higher temperatures with increasing strain rate, presumably because of the more rapid diffusion required for strain aging in the faster tests. The latter behavior provides a possible method for identifying the interstitial atom species responsible for strain aging. Kinoshita (ref. 7) has pointed out that the activation energy for strain aging can be determined from the strain rate sensitivity of the strength peak by plotting strain rate against the reciprocal of the peak temperature. This Arrhenius type of representation, which is shown in figure IV-20, yields an activation energy of 22 000 calories per mole for the strain aging reaction in T-111. This value is very close to the activation energy reported by Schmidt et al. (refs. 5 and 8) for diffusion of oxygen in tantalum (22 900 cal/mole), but is much lower than the values reported for diffusion of C and N in tantalum (39 600 and 41 100 cal/mole, respectively). It thus seems likely that oxygen is the critical interstitial specie in the strain age strengthening of T-111.

RESULTS AND DISCUSSION

Results from the refractory alloy creep program are divided into three basic categories. First, the data generated from the screening studies are presented and discussed briefly. Second, a detailed discussion of the creep behavior of T-111 alloy is presented, and finally the variable stress results are discussed.

Screening Studies

Molybdenum alloys. - Typical creep curves for TZC (fig. IV-21) show an irregular extension behavior characteristic of the molybdenum base alloys. Such

behavior is only observed in specific alloys, indicating that the discontinuities in the curves are real and do not represent instrument error. The accuracy of the extension measurements is apparently great enough to show some of the intrinsically random nature of the thermally activated creep process.

The 1/2 percent creep results for TZC are summarized on a Larson-Miller plot in figure IV-22. In the annealed condition the rolled plate with the partially recrystallized structure (heat M-80) had better creep resistance than the fully recrystallized plate (heat M-91). The higher creep strength may be partially due to the more complex substructure associated with the higher yield strength of the cold worked material or may result from the somewhat higher carbon level in heat M-80. Analysis of the creep data in conjunction with short time mechanical behavior indicates that, among the three TZC forms tested, heat 4345 represents the best combination of both short and long term creep strength with room temperature ductility.

The TZM test results, summarized in figure IV-23 on a 1/2 percent creep Larson-Miller plot, show the superior creep strength of the specially processed disk forging. Although the electron microscopy of the TZM alloys (fig. IV-3) revealed a somewhat higher number of carbides in the specially forged material, neither forging possessed a carbide dispersion which was dense enough to provide significant dispersion hardening. This indicates that the improved creep strength of heat KDTZM-1175 may also be associated with the coarser metallurgical structure shown in figure IV-2, with the increased carbide density playing a secondary role by providing an improved thermal stability to the cold worked structure.

Comparison of the TZC and TZM results indicated that at higher stress levels and lower temperatures the specially processed TZM was superior to TZC in the stress relieved condition, while at the lower stress levels the behavior of the two materials was comparable. The elevated temperature yield strength of TZM heat 1175 is superior to TZC (table IV-4), and this factor is believed to contribute to the improved creep strength at the lower test temperatures.

None of the molybdenum base alloys tested showed a significant change of structure or composition as a result of the long time elevated temperature exposure to ultrahigh vacuum, although a modest increase in strength resulted from the relatively small creep strains achieved.

Columbium alloys. - Creep test results from AS-30 and Cb-132M are compared on a Larson-Miller plot in figure IV-24. Although Cb-132M has significantly greater creep strength than AS-30, the specially processed TZM disk forging had creep resistance greater than either columbium alloy. Post-test examination indicated that no change in hardness or structure occurred in the columbium alloys as a result of creep exposure.

Tungsten alloys. - A summary of the creep data for tungsten and tungsten-25

percent rhenium is given in table IV-5. The Larson-Miller values for 1 percent creep in the table are based on a constant of 15. The arc-melted and vapor-deposited tungsten had comparable creep strength, while the tungsten-25 percent rhenium alloy had a greater creep strength but only at the higher stress levels.

The results obtained on the arc-melted and vapor-deposited tungsten are compared in figure IV-25 to published creep data for pure tungsten (refs. 9 and 10) using a Larson-Miller parameter for 1 percent creep. The results of Schmidt and Ogden (ref. 9) were obtained with a powder metallurgy product, and tests were performed in an argon environment. A powder metallurgy product was also used by the Southern Research Institute (ref. 10), but the test environment was a 10^{-4} torr vacuum. The creep strength of the arc-melted and vapor-deposited tungsten is definitely inferior to that of the powder metallurgy product of reference 9; however, the data from reference 10 appear to be an extension of those obtained with the arc-melted and vapor-deposited material.

Tantalum alloys. - A Larson-Miller plot of creep test results obtained on the tantalum base alloys T-111, Ta-10W, and ASTAR-811C and on pure tantalum is presented in figure IV-26 together with data from the literature on ASTAR-811C (ref. 11). The pure tantalum data show a significant variation of properties between heats. This effect is presumed to result from the fact that pure materials will be more sensitive to minor variations in trace impurity levels than the more heavily alloyed materials. However, the differences in creep strength may also be associated with the significant difference in grain size shown in figure IV-10. Further examination of the data in figure IV-26 indicates that the creep strengths of T-111 and Ta-10W alloys are comparable, while the ASTAR-811C alloy is significantly stronger. Heat treatment appears to have a considerable influence on the creep strength of ASTAR-811C, and the commercial heat is significantly stronger than either of the laboratory heats tested. Comparison of the various heat treatments studied with the carbide morphologies presented in figure IV-13 indicated that both grain size and carbide morphology significantly influenced ASTAR-811C creep behavior.

Creep Analysis of T-111 Alloy

Rationalization of T-111 transient creep behavior. - Conventional first stage creep was not always observed in T-111 alloys, and certain tests showed a very unusual behavior, with the creep rate initially attaining a relatively low value and then increasing again to a steady state. The tendency toward this type of behavior was temperature dependent, with tests at the lower temperatures (near $0.4 T_m$)

curving upward more sharply than those at higher temperatures (near $0.5 T_m$) (fig. IV-27).

Analysis of the unusual transient creep behavior in T-111 is facilitated by plotting true strain rate against true strain, as illustrated in figure IV-28 for the two tests shown in figure IV-27. This type of plot emphasizes the fact that the upward curvature is not classical third stage creep, since the right half of the low temperature curve in figure IV-28 shows a negative rather than a positive curvature. Both curves would presumably turn upward in true third stage creep if permitted to run long enough.

Comparison of the tensile and creep data on T-111 reveals that both the strain aging and the transient creep strengthening phenomenon are most pronounced at the lower test temperatures (in the 1600°F (870°C) range) and that both decrease gradually with increasing temperature and are essentially absent above about 2200°F (1204°C). This similarity of temperature dependence suggests that the two phenomena may be associated. Even so, it is difficult to explain why the effectiveness of a strain age strengthening mechanism should decrease with increasing test time. This phenomenon has been observed previously in an Fe-Cr-C alloy by DeBarbadillo (ref. 12), who attributed the decreasing strength to precipitation of chromium carbide. It was shown that both Cr and C were necessary by testing binary Fe-Cr and Fe-C alloys in which the unusual transient did not appear. Thus, as the chromium and carbon coprecipitated, the species necessary for strain aging was depleted from the matrix. DeBarbadillo further supported this supposition by showing that the kinetics of the precipitation reaction were such as to cause an abrupt creep rate transition of the observed form.

Examination of creep tested T-111 specimens showed that the precipitation mechanism was not directly applicable to the T-111 alloys. Figure IV-15(b) reveals an appreciable decrease rather than an increase in the number of inclusions after test. This decrease, which has been confirmed using the electron probe microanalyzer, is accompanied by a significant and consistent decrease in oxygen composition, as well as a drop in tensile strength and hardness in the tested material (table IV-6). Although sufficient creep tested material was not available to comprehensively evaluate the strength of the strain aging reaction in the post-tested specimens, a 56 percent decrease in the yield point drop was observed as a result of creep testing, indicating that less strain hardening occurred after creep test exposure.

On the basis of these results a mechanism similar to DeBarbadillo's involving softening of the creep tested material by depletion of the species responsible for strain age strengthening (oxygen) can be applied to the T-111 alloy to explain the transition in creep rate as a function of test time. Since oxygen removal proceeds

during creep testing, it causes a progressive weakening and a concomitant creep rate transition at the low test temperatures where strain aging is initially an important factor.

Analysis of T-111 steady state creep behavior. - Various standard analytical techniques were applied to the T-111 steady state creep data to gain better insight into the creep mechanisms involved. Neither the common engineering representation of log stress as a function of log minimum creep rate nor the Arrhenius plot would adequately correlate the results, so that it was necessary to utilize a temperature compensated creep rate parameter of the form

$$\dot{\epsilon} e^{\Delta H/RT}$$

in order to rationalize the steady state data. Although the usual approach to this treatment involves the use of the Arrhenius plot to obtain the activation energy, it is possible to obtain ΔH directly through statistical evaluation of the degree of correlation between the chosen stress function and $\dot{\epsilon} e^{\Delta H/RT}$.

Three stress functions are commonly found in the literature:

(1) Power function:

$$\dot{\epsilon} \propto \sigma^n$$

(2) Exponential function:

$$\dot{\epsilon} \propto e^{B\sigma}$$

(3) Hyperbolic sine function:

$$\dot{\epsilon} \propto [\sinh(\alpha\sigma)]^n$$

Garofalo (ref. 13) has discussed the stress dependence of creep rate and has shown that, for a number of materials tested over wide ranges of stress, the power law provides a plot of $\dot{\epsilon} e^{\Delta H/RT}$ against $f(\sigma)$, which is concave upward, while the exponential law provides a plot which is concave downward. Garofalo therefore proposed the hyperbolic sine relation to solve this problem, since it approximates the power law at low stresses and the exponential law at higher stresses. Figures IV-27, IV-30, and IV-31 show that these general observations apply to T-111, with the hyperbolic sine law being the only relation providing a linear correlation over the entire experimental stress range of 500 to 45 000 psi. It is interesting to note that the ultimate strength data at 1800° F (982° C), 2200° F (1204° C), and 2600° F (1427° C) all fall on the same line, with the creep data in figure IV-31 indicating

that at these temperatures and a strain rate of 0.005 inch per inch per minute tensile deformation is thermally activated in T-111 alloy. It should also be noted that, while the exponential and power laws do not fit the data over the entire stress range, there are limited stress ranges where each of these relations does provide a straight line fit of the data. For example, the exponential plot in figure IV-29 appears to provide a linear fit between 2.4 and 20 ksi despite the pronounced curvature over the wider stress range.

Evaluations of the straight line segments from each of the three stress representations all provided a ΔH value of about 90 000 calories per mole, which is close to the value of 100 000 calories per mole reported in the literature (ref. 6) for self-diffusion in tantalum. Since lattice strains introduced by substitution of the somewhat smaller W atom in the Ta lattice should enhance diffusivity, the apparent activation energy for creep is probably very close to that for either Ta or W diffusion in T-111. This indication of diffusion controlled creep seems somewhat surprising in view of the low homologous temperature range examined (0.39 to 0.52); however, Sherby and Burke (ref. 14) have suggested that at very low strain rates diffusion controlled creep may occur at lower than normal temperatures as a result of the long times available for diffusion to occur, and this is probably the explanation in the present case.

Diffusion controlled creep is normally assumed to result either from nonconservative motion of jogged screw dislocations or from climb of edge dislocations. However, the stress exponent for these two processes should be near 5, as opposed to the value of about 3 observed in the present work. A value of $n = 3$ conforms to that predicted by Weertman (ref. 15) for glide controlled creep, implying that steady state creep in T-111 may be controlled by substitutional solute atom drag. In this case, the ΔH value reported should represent the activation energy for diffusion of W in T-111 rather than Ta in T-111. Unfortunately, no data on the diffusion of W in Ta are available in the literature to check this possibility. However, alloys which display microcreep often do not undergo primary creep and do not exhibit a well developed substructure, both of which are consistent with the observed behavior in T-111. It therefore seems likely that steady state creep of T-111 is controlled by a microcreep mechanism in the temperature and stress ranges studied.

Analysis of Variable Stress Results on T-111 Alloy

The third major facet of the work presented in this paper deals with the behavior of T-111 alloy under continuously increasing loads. Typical creep curves obtained

from this type of test are presented in figure IV-32. The distribution of creep deformation through the test period depends on the relative loading rates and temperatures in these tests. At the higher temperatures and lower stress rate, creep strain is accumulated gradually throughout the test. On the other hand, at the lower temperatures and higher stress rates, very little creep occurs until the latter stages of the test, where most of the creep deformation is concentrated. For each temperature a stress rate existed above which the creep life was governed primarily by the rate of approach to the yield stress rather than by the creep rate, as indicated in table IV-7.

Using the time required to reach 1 percent extension as a creep life criterion, a parametric representation was developed for correlating the variable stress data. In this manner creep lives at different stress rates and temperatures could be combined and used for more reliable design predictions. McCoy (ref. 16) has pointed out that progressive stress results can be represented on the conventional log-log plot of stress against creep life, with stress rate substituted for stress. This type of plot provides a family of parallel, isothermal straight lines (fig. IV-33), each corresponding to the relation

$$\log \dot{\sigma} = M \log l + B \Big|_{\text{const. } T} \quad (1)$$

where $\dot{\sigma}$ is stress rate, l is creep life, and M and B are the slope and intercept. The influence of temperature on the intercept B can be expressed as

$$B(T) = B' - \log T^n \quad (2)$$

and combining equations (1) and (2) leads to the expression

$$\log(\dot{\sigma} T^n) = M \log l + B' \quad (3)$$

The progressive stress creep results can be represented by a single straight line when $\dot{\sigma} T^n$ is plotted as a function of creep life with $n = 5.5$ (fig. IV-33), and the desired parametric representation is thereby obtained.

Analytical treatments. - From both a theoretical and a practical standpoint it is desirable to compare the creep results obtained from continuously increasing stress tests with conventional isostatic creep behavior. Manson (ref. 17) has discussed this problem in his "time hardening" and "strain hardening" theories, which provide conceptual guide lines for the manual assembly of variable stress creep curves from selected segments of conventional creep curves. McCoy (ref. 16) and Nichols and Winkler (ref. 18) have approached the specific problem of

using isostatic data to predict variable stress behavior in radioisotope capsules by using a "life fraction" concept in which the useful life of a specimen is considered to be exhausted when the sum of the instantaneous life fractions equals unity. Each individual life fraction is equal to the time spent at any specific set of test conditions divided by the total creep life at those conditions. McCoy uses equation (1) to define creep life l_i and then integrates the life fraction for the case where stress increases steadily at the rate $\dot{\sigma}$:

$$1 = \int_0^L \frac{dt}{l_i} = \int_0^L \frac{dt}{e^{-B/M}(\dot{\sigma}t)^{1/M}} \quad (4)$$

The variable stress creep life is the upper limit L in equation (4):

$$L = \left(\frac{M-1}{M} e^{-B/M} \dot{\sigma}^{1/M} \right)^{(1-M)/M} \quad (5)$$

Nichols and Winkler have generalized this technique by using the temperature compensated Larson-Miller parameter (LMP) to generate the creep life function and have developed a computer program for numerical integration of the resulting life fraction expression. This program was written specifically for application to the radioisotope capsule problem and will interchangeably generate capsule dimensions or capsule life for any analytically expressed stress or temperature variation, when provided with the appropriate conventional creep test data. Nichols and Winkler's program contains a subroutine which fits the isostatic creep data to an expression of the form

$$\sigma = \left\{ \frac{(e^{\alpha} \sigma_u)^{\gamma}}{(e^{\alpha})^{\gamma} + [\sigma_u e^{-m(\text{LMP})}]^{\gamma}} \right\}^{1/\gamma} \quad (6)$$

This expression provides a linear relation between $\ln \sigma$ and the Larson-Miller parameter at high LMP values, while allowing σ to approach σ_u (ultimate strength) asymptotically at low LMP values. The constants M and α are, respectively, the slope and intercept of the straight line, and γ is an imposed constant of the order of 2.

Another technique for prediction of increasing load creep life was developed in the present study from a previous analysis of T-111 creep data obtained under iso-

static load conditions (ref. 19). The earlier study had shown that the steady state creep rate of T-111 recrystallized 1 hour at 3000° F can be described analytically in the temperature and stress ranges of 1800° to 2400° F (982° to 1316° C) and 2400 to 20 000 psi (1.65×10^7 to 13.8×10^7 N/m²) as

$$\dot{\epsilon} = A e^{B\sigma} e^{-\Delta H/RT} \quad (7)$$

with

$$\Delta H = 90\,000 \text{ cal/mole}$$

$$A = 4.68 \times 10^6 \text{ hr}^{-1}$$

$$B = 3.98 \times 10^{-4} \text{ psi}^{-1}$$

and R and T being, respectively, the universal gas constant and the absolute temperature in degrees Kelvin (ref. 19). This expression is convenient for comparison of conventional isostatic and increasing stress results since it allows steady state rates at different temperatures and stresses to be compared directly.

In the increasing stress tests the instantaneous strain rates were determined at various stress levels by numerical differentiation. These results are plotted in figure IV-34, where the increasing stress tests are compared with conventional test results. Although the scatter is somewhat greater, the creep strain rates observed in the increasing stress tests are with a few exceptions in the same range as the conventional test data. These results indicate that the creep rates obtained in the increasing stress tests are a unique function of stress and are relatively independent of creep strain for the conditions tested.

The above results also imply that integration of the conventional creep rates obtained under isostatic load conditions should predict the increasing stress creep lives. The strain at any point in an increasing stress test is simply the integral of the strain rate as a function of time:

$$\epsilon = \int_0^L \dot{\epsilon}(t) dt \quad (8)$$

If the functional relation of $\dot{\epsilon}$ to stress and the time dependence of stress are known, this equation can be expressed in the form

$$\epsilon = \int_0^L \dot{\epsilon}[\sigma(t)] dt \quad (9)$$

The form of $\dot{\epsilon}(\sigma)$ is known from equation (7), and $\sigma(t)$ is simply

$$\sigma = \dot{\sigma} t \quad (10)$$

for the linearly increasing stress test. Inserting these two expressions in equation (9) yields

$$\epsilon = \int_0^L A e^{B \dot{\sigma} t} e^{-\Delta H/RT} dt \quad (11)$$

which can be integrated analytically to evaluate the upper limit (which is creep life) for any desired strain. Choosing $\epsilon = 0.01$ results in an expression for 1 percent creep life of the form

$$0.01 = \frac{A e^{-\Delta H/RT}}{B \dot{\sigma}} (e^{B \dot{\sigma} L} - 1) \quad (12)$$

or, rearranging,

$$L = \frac{1}{B \dot{\sigma}} \ln \left(\frac{0.01 B \dot{\sigma}}{A e^{-\Delta H/RT}} + 1 \right) \quad (13)$$

where the constants A , B , and ΔH are as given for equation (7).

Comparison of creep life predictions with experimental data. - Predictions of each of the analytical techniques are compared with the experimental data in table IV-8. At the two lower test temperatures the strain rate integration technique predicts creep lives which are somewhat shorter than the experimental values, whereas the Nichols and Winkler predictions are somewhat larger than the observed lives. However, at 2200° F both techniques appear equally good, with statistical variation of the experimental results providing the major source of difference between predicted and observed values. The reason for this variability of predictions is illustrated schematically in figures IV-35 and IV-36. Figure IV-35 shows the basic relation between the life fraction and the strain rate integration techniques. The integrand in both cases is a creep rate, but whereas in the strain rate integration technique experimentally observed steady state creep rate $\dot{\epsilon}_1$ is integrated, the creep rate integrated in the life fraction technique is a hypothetical rate established by the reciprocal of the 1 percent creep life $\dot{\epsilon}_2$. Thus, the relative success of each of these techniques will depend upon the degree to which the instantaneous strain rates in the increasing stress tests approximate either the hypothetical or the experimentally observed steady state creep rates.

The specific application of the above concept to T-111 alloy is illustrated in figure IV-36, which shows that T-111 undergoes a creep rate transition at low test temperatures (in the 1600° to 2000° F range), whereas it shows normal primary creep at high temperatures (near 2600° F). At temperatures between these two extremes, creep of T-111 exhibits neither a pronounced rate transition nor a pronounced first stage.

The importance of the low temperature creep rate transition is that the isostatic steady state creep rate is higher than the hypothetical life fraction creep rate in this temperature range, whereas it ordinarily would be lower when first stage creep appears as it does in the high temperature T-111 creep tests. Previous work referred to earlier showed that the creep rate transition in T-111 alloy is caused by a vacuum induced loss of oxygen, which provides significant strain age strengthening in the early stages of a low temperature T-111 creep test (ref. 19). Since this vacuum induced deoxidation can proceed independently of the creep deformation, the creep test specimens may be partially degassed when the load in the increasing stress test reaches a high enough level for significant creep to occur.

Thus, throughout much of the active life of a low temperature increasing stress test the creep rate should approximate the isostatic steady state creep rates, as shown by the experimental results in figure IV-34. The reason that the creep rates in the increasing stress tests tend to be slower than isostatic rates in the early stages of the increasing stress tests now becomes clear, since creep at this stage of the test may be occurring in only partially deoxidized material. The significance of the creep rate transition is that, when the experimental isostatic creep rates are integrated at low temperatures, the integrated strain will be slightly greater than, and the predicted 1 percent creep life slightly shorter than, the experimentally observed increasing stress creep life. Conversely, when the hypothetical creep rate established by the reciprocal of the creep life $\dot{\epsilon}_2$ is integrated at low temperatures, the predicted creep life is slightly greater than the experimentally observed value. At high temperatures where primary creep occurs, the opposite would presumably be true; that is, the Nichols and Winkler life fraction integration should provide the conservative prediction while the steady state strain rate integration should predict creep lives which are longer than the experimentally observed values. While increasing stress data were not available at high temperatures to check this hypothesis, the data in table IV-8 show that at intermediate temperatures, where creep is essentially linear, the predictions of both methods are comparable and agree well with the experimental data.

SUMMARY OF RESULTS

A Larson-Miller analysis of creep test results on molybdenum and columbium-base turbine alloys showed that at higher stress levels and low temperatures a specially processed lot of TZM having a somewhat higher than normal carbon content is superior to TZC in the stress relieved condition, whereas at higher temperatures and lower stresses, the behavior of the two materials is comparable. Both of the molybdenum alloys were superior to the columbium-base alloys AS-30 and CB-132M.

The creep tests indicated that arc-melted and vapor-deposited tungsten had comparable creep behavior. A comparison between these data and literature values for the powder metallurgy product tested in vacuum showed no difference on a Larson-Miller plot. However, the literature values indicated that powder metallurgy material tested in argon appeared substantially more creep resistant.

A detailed analysis of tension and creep test results on T-111 revealed that a complex atmosphere strain aging phenomenon involving oxygen and probably hafnium provides a transient strengthening in the early stages of T-111 creep tests in the 1600° to 2200° F (870° to 1204° C) range. At long test times, ultrahigh vacuum removal of oxygen depletes the strain aging strengthening species from the matrix and causes a creep rate transition to occur. After the creep rate transition, steady state creep proceeds according to the relation

$$\dot{\epsilon} = 1.65 \times 10^9 \left[\sinh(6.6 \times 10^{-5} \sigma) \right]^{3.17} e^{-90\,000/RT}$$

where the values of activation energy and stress exponent suggest that steady state creep is governed by a diffusion controlled microcreep mechanism.

The creep tests conducted on T-111 alloy with continuously increasing loads provided continuously increasing creep rates which were comparable to the isostatic steady state creep rates at equivalent stress levels. Methods have been developed for prediction of the 1 percent creep life under varying stress conditions from isostatic creep test data, and comparison of these predictions with the experimental results shows the dependence of analytical techniques on the character of the isostatic creep curves.

REFERENCES

1. Sawyer, J. C.: Design and Operation of Ultra-High Vacuum Creep Equipment. Transactions Vacuum Metallurgy Conference. L. M. Bianchi, ed., American Vacuum Society, 1965, pp. 41-55.

2. Sawyer, J. C.; and Steigerwald, E. A.: Creep Properties of Refractory Metal Alloys in Ultrahigh Vacuum. *J. Materials*, vol. 2, no. 3, June 1967, pp. 341-361.
3. Sheffler, K. D.; and Steigerwald, E. A.: Generation of Long Time Creep Data on Refractory Alloys at Elevated Temperatures. TRW Equipment Labs. (NASA CR-72391), Dec. 14, 1967.
4. Schmidt, F. F.; and Ogden, H. R.: The Engineering Properties of Tantalum and Tantalum Alloys. DMIC Rep. 189, Battelle Memorial Inst., Sept. 13, 1963. (Available from DDC as AD-426344.)
5. Schmidt, Frank F. et al.: Investigation of the Properties of Tantalum and Its Alloys. Battelle Memorial Inst. (WADD-TR-59-13, DDC No. AD-236957), Mar. 1960.
6. Baird, J. D.; and Jamieson, A.: Effects of Manganese and Nitrogen on the Tensile Properties of Iron in the Range 20-600° C. *J. Iron Steel Inst.*, vol. 204, pt. 8, Aug. 1966, pp. 793-803.
7. Kinoshita, S.; Wray, P. J.; and Horne, G. T.: Some Observations on the Portevin-Le Chatelier Effect in Iron. *Trans. AIME*, vol. 233, no. 10, Oct. 1965, pp. 1902-1904.
8. Schmidt, F. F.: Tantalum and Tantalum Alloys. DMIC Rep. 133, Battelle Memorial Inst., July 25, 1960.
9. Schmidt, F. F.; and Ogden, H. R.: The Engineering Properties of Tungsten and Tungsten Alloys. DMIC Rep. 191, Battelle Memorial Inst., Sept. 27, 1963. (Available from DDC as AD-425547.)
10. Anon.: The Mechanical and Thermal Properties of Tungsten and TZM Sheet Produced in the Refractory Metal Sheet Rolling Program. Pt. 1. Rep. 7563-1479-XII, Southern Research Inst., Aug. 31, 1966. (Available from DDC as AD-638631.)
11. Buckman, R. W., Jr.; and Goodspeed, R. C.: Development of Dispersion Strengthened Tantalum Base Alloy. Rep. WANL-PR-(Q)-013, Westinghouse Electric Corp. (NASA CR-72316), Nov. 20, 1966.
12. deBarbadillo, John J.: The Effect of Substitutional Alloying Elements on the Elevated Temperature Mechanical Properties of Alpha Iron. Ph.D. Thesis, Lehigh Univ., 1967.
13. Garofalo, F.: Fundamentals of Creep and Creep-Rupture in Metals. Macmillan Co., 1965.

14. Sherby, O. D.; and Burke, P. M.: Mechanical Behavior of Crystalline Solids at Elevated Temperature. Progress in Material Science, vol. 13, no. 7, 1967. Pergamon Press, 1968, p. 325.
15. Weertman, J.: Creep of Indium, Lead, and Some of Their Alloys with Various Metals. Trans. AIME, vol. 218, no. 2, Apr. 1960, pp. 207-218.
16. McCoy, H. E.: Creep-Rupture Properties under Conditions of Constant and Varying Stresses. Nucl. Appl., vol. 2, no. 6, Dec. 1966, pp. 481-485.
17. Manson, S. S.: Thermal Stress and Low-Cycle Fatigue. McGraw-Hill Book Co., Inc., 1966, p. 118.
18. Nichols, J. P.; and Winkler, D. R.: A Program for Calculating Optimum Dimensions of Alpha Radioisotope Capsules Exposed to Varying Stress and Temperature. Rep. ORNL-TM-1735, Oak Ridge National Lab. (NASA CR-72172), Apr. 1967.
19. Sheffler, K. D.; Sawyer, J. C.; and Steigerwald, E. A.: Mechanical Behavior of Tantalum-Base T-111 Alloy at Elevated Temperature. NASA CR-1436, 1969.

TABLE IV-1. - CHEMICAL COMPOSITION OF ALLOYS EVALUATED IN CREEP TEST PROGRAM

Material	wt %									ppm		
	Cb	W	Re	Mo	Ta	Hf	C	Ti	Zr	N ₂	O ₂	H ₂
TZM												
(Heat 7463)	---	----	-----	Bal.	----	----	0.016	0.48	0.08	1	2	1
(Heat 7502)	---	----	-----	Bal.	----	----	.010	.51	.091	100	20	7
(Heat KDTZM-1175)	---	----	-----	Bal.	----	----	.035	.61	.120	43	34	9
TZC												
(Heat M-80)	---	----	-----	Bal.	----	----	.127	1.02	.17	18	41	10
(Heat M-91)	---	----	-----	Bal.	----	----	.113	1.17	.270	34	37	10
(Heat 4345)	---	----	-----	Bal.	----	----	.075	1.19	.16	9	19	2
AS-30	Bal.	21.0	-----	----	----	----	.09	----	1.04	100	60	15
Cb-132M	Bal.	15.6	-----	4.72	19.7	----	.16	----	2.10	24	4	4
W - Arc melted												
(Heat KC1357)	---	Bal.	-----	----	----	----	.0058	----	-----	16	9	3
Vapor deposited	---	Bal.	-----	----	----	----	.0012	----	-----	15	13	2
W-25 percent Re arc melted												
(Heat 35-75002)	---	----	24.88	----	----	----	.007	----	-----	7	61	13
ASTAR-811C												
(Heat NASV-20-WS)	---	7.3	1.0	----	Bal.	0.86	.024	----	-----	20	14	----
(Heat VAM-95)	---	7.6	1.1	----	Bal.	.65	.030	----	-----	3	4	.3
(Heat 650056)	---	8.2	1.2	----	Bal.	.9	.020	----	-----	14	30	3.5
Ta-10W												
(Heat 630002)	---	9.9	-----	----	Bal.	----	.0044	----	-----	25	100	5
Pure tantalum												
(Heat B-1962)	---	----	-----	----	Bal.	----	.0012	----	-----	21	20	5
(Heat 60249)	---	----	-----	----	Bal.	----	.0014	----	-----	20	51	3
T-111 - see table IV-3	---	----	-----	----	----	----	-----	----	-----	---	---	---

TABLE IV-2. - SUMMARY OF TEST MATERIAL PROPERTIES AND PROCESSING DETAILS

Material	Heat	Vendor	Pertinent processing details	Heat treatment	Tensile strength, ksi		Percent elongation	DPH hardness
					Ultimate	0.2 Percent yield		
TZC	M-80	General Electric	Extruded 2.3:1 at 3092° F, cross-rolled at 2925° F with 4 percent reduction per pass	3092° F, 1 hr	68.6	68.5	0.05	268
TZC	M-91	General Electric	Extruded 2.3:1 at 3092° F, cross-rolled with large deformations per pass and finished at 2372° F	3092° F, 1 hr 2500° F, 1 hr	85.0 106	49.0 99	7.0 4	240 303
TZC	4345	Climax Molybdenum	Extruded at 3000° F, heat treated at 3000° F, upset forged 40 percent at 2400° F, broad forged to 0.825 in. at 2400° F	2400° F, 1 hr	123	117	12	346
TZM	7502	Climax Molybdenum	Extruded from 10 $\frac{3}{4}$ to 6 $\frac{1}{4}$ in. diam, heat treated at 2700° F, upset forged at 2200° F	2200° F, 1 hr 2850° F, 1 hr	----- -----	----- -----	----- -----	304 209
TZM	KDTZM-1175	Air Research-Universal Cyclops	Extruded from 10 $\frac{3}{4}$ to 6 $\frac{1}{4}$ in. diam, recrystallized at 2800° F for 4 hr, forged to 4 in. diam at 3400° to 2800° F, recrystallized at 2950° F for 2 hr, forged to 3/4 in. flat disk, 11 blows, at 2800° to 2160° F	2300° F, 1 hr	122	111	17.4	316
TZM	7463	Climax Molybdenum	Extruded from 11 $\frac{1}{2}$ to 6 $\frac{1}{2}$ in. diam, recrystallized, rolled to 2 in. diam, recrystallized, rolled to 1 in. diam, swaged to 5/8 in. diam	2250° F, 1/2 hr	128	117	30	300
AS-30	C5	General Electric	Extruded from 4.8 to 4 by 1.625 in. sheet bar at 2825° F, cross rolled at 2100° F to 0.790 in.	-----	-----	-----	-----	293
Cb-132M	KC1454	Universal Cyclops	Extruded from 3 $\frac{7}{8}$ to 1 $\frac{1}{2}$ in. at 3130° F, cross-rolled to 3/4 in. at 2400° F	-----	-----	-----	-----	336
W - Arc-melted	KC1357	Universal Cyclops	Extruded 4:1 at 3100° F, forged at 2200° F, rolled at 2300°, 1800°, and 1400° F, stress relieved at 1700° F	-----	-----	-----	-----	487
W - Vapor deposited	-----	-----	-----	-----	-----	-----	-----	---
W - 25 percent Re	3.5-75002	Wah Chang	0.055 in. sheet stress relieved at 2375° F, rolled to 0.035 in., stress relieved at 2550° F	-----	-----	-----	-----	639
T-111	Typical	Wah Chang	6 $\frac{1}{2}$ in. diam ingot forged to 1 $\frac{1}{2}$ in. bar at 2200° F, annealed at 3000° F, rolled at 800° F to 1/4 in. thick, annealed at 3000° F, cold rolled to 0.030 in.	3000° F, 1 hr	94	Typical	-----	---
T-111	Typical	Fansteel	Extruded 3.25:1 at 2200° F, rod rolled and flat rolled to 0.030 in.	3000° F, 1 hr	94	74	35	250
Pure Ta	-----	-----	Double e.b. melt extruded to 2 in. o.d. by 1/4 in. wall tube, reduced and drawn to 3/4 in. o.d. by 0.040 in. wall	-----	-----	-----	-----	---
ASTAR-811C	Typical	Westinghouse	Side forged from 3.9 in. diam by 5 in. long ingot to 7.2 by 7.5 by 1.133 in. thick bar at 2550° F, annealed at 2700° F for 2 hr, rolled to 1/4 in. at 500° F, annealed 1 hr at 3000° F, cold rolled to 0.040 in.	-----	-----	-----	-----	253

TABLE IV-3. - CHEMICAL COMPOSITION AND ROOM TEMPERATURE MECHANICAL PROPERTIES OF T-111 ALLOYS ANNEALED 1 HOUR AT 3000⁰ F (1649⁰ C)

Heat	Composition						Mechanical properties		
	Weight percent			ppm			Tensile strength, ksi		Percent elongation
	W	Hf	C	N ₂	O ₂	H ₂	Ultimate	0.2 Percent yield	
70616	8.5	2.3	0.0044	20	55	6	(a)	(a)	(a)
65079	8.7	2.3	.0030	50	130	4	94.0	72.0	35.2
65076	8.6	2.0	.0040	20	100	3	93.2	79.9	31.0
D-1102	7.9	2.3	.0030	34	20	3	(a)	(a)	(a)
D-1670	7.9	2.4	.0010	20	72	<5	(a)	(a)	(a)
D-1183	8.7	2.2	.0036	10	25	6	94.3	81.0	32.8
650028	8.3	2.1	.0030	12	30	1.9	83.8	77.8	30.1
848001	7.9	2.0	.0010	13	21	1	79.3	70.0	30.7
650038	8.6	2.0	.0025	20	100	2.8	92.1	76.8	29.8

^aNot available.

TABLE IV-4. - TENSILE PROPERTIES OF STRESS RELIEVED

TZC AND TZM AT 2000⁰ F

Material	Stress relief temperature, °F	Tensile strength, ksi		Percent elongation	Percent reduction of area
		Ultimate	0.2 Percent yield		
TZM heat KDTZM-1175	2300	79	74	19	51
TZC heat 4345	2500	70	69	18	61

TABLE IV-5. - SUMMARY OF CREEP DATA FOR TUNGSTEN AND TUNGSTEN-BASE ALLOYS

Material	Specimen	Test temperature		Stress		Hours to 1 percent creep	Larson-Miller parameter - 1 percent creep, $T_{0R}(15 + \log t)$	Creep rate, in./in./hr
		$^{\circ}\text{F}$	$^{\circ}\text{C}$	ksi	N/m^2			
Tungsten, heat KC-1357	S-5	3200	1760	3	2.07×10^7	6	57.8×10^3	1.7×10^{-3}
	S-7	3200	1760	.4	2.76×10^6	(a)	(a)	1.7×10^{-6}
	S-9	3200	1760	1	6.89×10^6	675	65.4	1.5×10^{-5}
	S-17	2800	1538	4	2.76×10^7	20	53.1	5.0×10^{-4}
	S-18	2800	1538	3	2.07×10^7	125	55.8	8.0×10^{-5}
Vapor-deposited tungsten	B-17	3200	1760	1	6.89×10^6	1140	66.0×10^3	8.8×10^{-6}
	B-24	2800	1538	2	1.38×10^7	1500	59.2	6.7
Tungsten - 25 percent rhenium, heat 3.5-75002	S-3	3200	1760	5	3.44×10^7	12	58.9×10^3	8.3×10^{-4}
	S-4	3200	1760	3	2.07×10^7	25	60.0	4.0×10^{-4}
	S-6	3200	1760	.5	3.44×10^6	(a)	(a)	3.6×10^{-6}
	S-8	3200	1760	1.5	1.03×10^7	315	64.0	3.2×10^{-5}

^aToo little creep to extrapolate.TABLE IV-6. - EFFECT OF CREEP EXPOSURE ON ROOM TEMPERATURE MECHANICAL PROPERTIES
AND COMPOSITION OF T-111 ANNEALED 1 HOUR AT 3000^o F PRIOR TO CREEP TESTING

Creep test conditions					Before or after creep testing	Ultimate tensile strength, ksi	Percent elongation	Diamond pyramid hardness	Composition				
Test	Heat	Stress, ksi	Temper- ature, °F	Test duration, hr					Percent		ppm		
									Hf	C	N ₂	O ₂	H ₂
S-26	D-1670	17	1800	1624	Before	(a)	(a)	(a)	2.17	0.001	20	72	5
bS-31	65079	5	2200	6602	After				2.09	.0032	30	10	5
					Before	94.0	35.0	(a)	(a)	(a)	(a)	(a)	
S-33	65076	8	2200	2976	After	85.3	c8.2						
					Before	93.1	31.0	247	1.95	.004	20	100	3
S-32	D-1102	5	2200	4322	After	85.7	39.0	228	2.07	.004	30	20	31
					Before	(a)	(a)	(a)	2.28	.003	34	20	3
S-35	65079	5	2200	5522	After				2.17	.003	30	10	24
					Before	(a)	(a)	275	2.30	.003	50	130	4
S-43	65079	18	2000	361	After			257	2.14	.0046	30	10	5
					Before	(a)	(a)	(a)	2.30	.003	50	130	4
					After				1.33	.003	40	10	31

^aNot available.^bPerpendicular to rolling direction.^cReduced gage width.

TABLE IV-7. - LOADING RATES
 ABOVE WHICH INCREASING
 LOAD TESTS ARE YIELD
 RATHER THAN
 CREEP LIMITED

Temperature		Stress rate, psi/hr
°F	°C	
1800	982	0.2
2000	1093	8
2200	1204	166

TABLE IV-8. - PROGRESSIVE STRESS CREEP TEST RESULTS
 FOR T-111 ALLOY

Test temperature		Stress rate, psi/hr	1 Percent creep life		
°F	°C		Observed	Predicted ^a	Predicted ^b
1800	982	20	1660	1430	1809
1800	982	5	5500	4990	6466
2000	1093	13	1600	1540	1798
2000	1093	5	3850	3500	3957
2000	1093	2	^c 8000	7620	8342
2200	1204	16	1080	851	895
2200	1204	5	2240	2260	2122
2200	1204	1	7750	7340	7450

^aStrain rate integration technique.

^bLife fraction technique (Nichols and Winkler program).

^cExtrapolated.

Figure IV-1. - Influence of environment on creep behavior of T-111 alloy at identical test conditions.

CONVENTIONALLY PROCESSED TZM DISK FORGING (HEAT 7502);
EDGE PERPENDICULAR TO RADIAL DIRECTION

SPECIALLY PROCESSED TZM DISK FORGING (HEAT KDTZM-1175);
EDGE PERPENDICULAR TO RADIAL DIRECTION

TZM BAR (HEAT 7463); LONGITUDINAL SECTION

Figure IV-2. - Microstructures of stress relieved TZM. $\times 100$.

HEAT KDTZM-1175; X5000

HEAT KDTZM-1175; X10 000

HEAT 7502; X5000

HEAT 7502; X10 000

Figure IV-3. - Electron micrographs of pancake forged and stress relieved TZM disks. Two stage replicas (cellulose nitrate and carbon) with chromium shadowing on primary replica. Reduced 10 percent for reproduction.

ROLLED PLATE; HEAVY DRAFTING PRACTICE, LOW TEMPERATURE FINISH (HEAT M-91); LONGITUDINAL VIEW

ROLLED PLATE; LIGHT DRAFTING PRACTICE, HIGH TEMPERATURE FINISH (HEAT M-80); LONGITUDINAL VIEW

FORGED PLATE (HEAT 4345); EDGE OF PLATE PERPENDICULAR TO ORIGINAL EXTRUSION DIRECTION

Figure IV-4. - Microstructures of stress relieved TZC. $\times 100$.

HEAVY DRAFTING PRACTICE; LOW TEMPERATURE FINISH (HEAT M-91)

LIGHT DRAFTING PRACTICE; HIGH TEMPERATURE FINISH (HEAT M-80)

Figure IV-5. - Longitudinal microstructures of TZC rolled plates after annealing 1 hour at 3092°F . $\times 500$.

AS-30 AS ROLLED

Cb-132M AS ROLLED

Cb-132M ANNEALED 1 HOUR AT 3092°F (1700°C)

Figure IV-6. - Microstructures of columbium base alloys tested in ultrahigh vacuum creep program. $\times 100$.

EDGE PERPENDICULAR TO ROLLING DIRECTION

SURFACE OF SHEET

Figure IV-7. - Microstructure of tungsten sheet (heat KC 1357) annealed 1 hour at 3200°F (1760°C). $\times 100$.

⊕
TENSILE
AXIS

GAGE SECTION PERPENDICULAR TO SPECIMEN AXIS

HEAD SECTION PERPENDICULAR TO SPECIMEN AXIS

Figure IV-8. - Vapor deposited tungsten microstructure after test. $\times 100$.

EDGE PERPENDICULAR TO ROLLING DIRECTION

SURFACE OF SHEET

Figure IV-9. - Microstructure of tungsten-25 percent rhenium alloy sheet (heat 3.5-75002) annealed 1 hour at 3200°F (1760°C). $\times 100$.

HEAT B-1962 ANNEALED 1 HOUR AT 1832° F
(1000° C); HARDNESS, 94 KHN; X100

HEAT 60249 ANNEALED 1 HOUR AT 2290° F
(1254° C); HARDNESS, 90 KHN; X100

Figure IV-10. - Microstructures of pure tantalum tubing.

Figure IV-11. - Microstructure of Ta-10W alloy
annealed 1 hour at 3000° F. X100.

15 MINUTES AT 3520°F (1940°C); X100

9 MINUTES AT 3600°F (1983°C); X100

Figure IV-12. - Microstructure of ASTAR 811C heat VAM-95 annealed to grain size of 0.066 millimeter at 3520° and 3600° F (1940° and 1983° C).

Figure IV-13. - Influence of annealing time and temperature on precipitate morphology in tantalum-base ASTAR 811C, heat NASV-20-WS. $\times 7500$, reduced 50 percent for reproduction.

X100

X500

Figure IV-14. - Optical photomicrographs of T-111 heat 70616 recrystallized 1 hour at 3000° F (1649° C).

SPECIMEN S-40
TESTED 8717 HOURS
AT 1800° F (982° C)
AND 17 ksi (11.7×10^7
N/m²); TOTAL CREEP
STRAIN, 1.028 PERCENT;
HEAT D-1102

SPECIMEN S-26
TESTED 9624 HOURS
AT 1800° F (982° C)
AND 17 ksi (11.7×10^7
N/m²); TOTAL CREEP
STRAIN, 1.030 PERCENT;
HEAT D-1670

SPECIMEN S-33
TESTED 2976 HOURS
AT 2200° F (1204° C)
AND 8 ksi (5.51×10^7
N/m²); TOTAL CREEP
STRAIN, 1.048 PERCENT
HEAT 65076

SPECIMEN S-35
TESTED 5522 HOURS
AT 2200° F (1204° C)
AND 5 ksi (3.44×10^7
N/m²); TOTAL CREEP
STRAIN, 1.092 PERCENT
HEAT 65079

(a) Before creep testing.

(b) After creep testing.

Figure IV-15. - Electron micrographs of tantalum T-111 alloy (Ta-8%-2% Hf) recrystallized 1 hour at 3000° F, before and after creep testing at indicated test conditions. Total chamber pressure, less than 1×10^{-8} torr; two stage replicas (cellulose nitrate and carbon); arrows indicate direction of chromium shadowing on primary replica. $\times 7500$, reduced 55 percent for reproduction.

X550

X1650

Figure IV-16. - Electron microprobe analyzer current images of T-111 heat D-1102 recrystallized 1 hour at 3000° F (1649° C). Bright spots represent included second phase particles.

Figure IV-17. - Variation of ultimate strength, strain hardening exponent, 2 percent yield strength, and percent elongation with temperature in T-111 annealed 1 hour at 3000° F (1649° C).

Figure IV-17. - Concluded.

Figure IV-18. - Load-deflection curve or recrystallized T-111 at 1400° F. Arrow indicates decrease in chart speed.

Figure IV-19. - Variation of flow stress with temperature and strain rate at two levels of true strain in T-111 annealed 1 hour at 3000° F (1649° C). Tick on each curve indicates strain aging peak.

Figure IV-20. - Variation of strain aging peak temperature with strain rate at two levels of true strain in T-111 annealed 1 hour at 3000 $^\circ$ F (1649 $^\circ$ C). Slope of line represents activation energy for strain aging.

Figure IV-21. - Creep test data for TZC rolled plate (heat M-91) tested in vacuum environment of $<1 \times 10^{-8}$ torr. Arrows on curves indicate chamber pressure at various intervals during test.

Figure IV-22. - 1/2 Percent creep test results for TZC tested in vacuum environment of $<1 \times 10^{-8}$ torr.

Figure IV-23. - 1/2 Percent creep test results for TZM tested in vacuum environment of $<1 \times 10^{-8}$ torr.

Figure IV-24. - Larson-Miller plot for 0.5 and 1.0 percent creep in AS-30 and Cb-132M plate tested in vacuum environment of $<1 \times 10^{-8}$ torr.

Figure IV-25. - Larson-Miller plot of tungsten 1 percent creep data.

Figure IV-26. - Larson-Miller plot of 1 percent creep life data for tantalum-base alloys creep tested in vacuum of $<1 \times 10^{-8}$ torr.

Figure IV-27. - Creep test data for T-111, heat D-1670, annealed 1 hour at 3000° F (1649° C) and tested in vacuum environment of $<1 \times 10^{-8}$ torr.

Figure IV-28. - True strain rate as function of true strain in T-111 annealed 1 hour at 3000° F (1649° C) and creep tested at indicated conditions.

Figure IV-29. - Variation of temperature compensated true strain rate with stress in T-111 alloy annealed 1 hour at 3000° F (1649° C).

Figure IV-30. - Variation of temperature compensated true strain rate with log stress for T-111 alloy annealed 1 hour at 3000° F (1649° C).

TEST TEMP. °F	STRESS, psi	
▲ 1800	17 000	
● 1860	20 000	
• 2000	8 500	
▼ 2000	11 000	
▶ 2000	13 000	
▲ 2000	15 000	
• 2000	20 000	
× 2000	35 000	
■ 2000	35 000	
■ 2000	45 000	
■ 2120	12 000	
◇ 2200	5 000	
◇ 2200	8 000	
◇ 2200	12 000	
+ 2300	3 500	
◇ 2330	2 400	
△ 2400	3 500	
• 2600	500	
□ 1800	74 000	} ULTIMATE TENSILE STRENGTH, STRAIN RATE = 0.005 in./in./min)
□ 2200	49 000	
□ 2600	29 000	
× 2600	1 500	

Figure IV-31. - Variation of temperature compensated true strain rate with $\log [\sinh (\alpha \sigma)]^n$ in T-111 alloy annealed 1 hour at 3000° F (1649° C).

Figure IV-32. - Variable stress creep behavior of recrystallized T-111 alloy.

Figure IV-33. - Progressive stress 1 percent creep life data for T-111 alloy.

Figure IV-34. - Comparison of isostatic and variable stress creep rates in recrystallized T-111 alloy.

Figure IV-35. - Schematic creep curve showing hypothetical creep rate integrated in life fraction technique.

Figure IV-36. - Schematic creep curve for T-111 alloy showing relation between hypothetical strain rate integrated in life fraction technique and experimental isostatic creep rates integrated in strain rate integration technique.