VI. HIGH FREQUENCY FATIGUE PROPERTIES OF TZC AND T-111 ALLOYS*

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

SUMMARY

High frequency fatigue tests were conducted in an ultrahigh vacuum environment on recrystallized molybdenum base alloy TZC and tantalum base alloy T-111 at temperatures between 1800° and 2200° F (982° and 1204° C). The results indicated that in this temperature range no well-defined endurance limit existed for either material. At A ratios of 0.45, fatigue strengths at 2000° F (1093° C) and 10^9 cycles were as low as 16 000 psi (11.0×10^7 N/m²) for the TZC and 34 000 psi (24.8×10^7 N/m²) for the T-111. The application of a cyclic load to a statically-loaded specimen produced a marked acceleration in the degree of specimen extension. In both the TZC and T-111 alloy the increase was characterized by a relatively rapid first stage creep extension (on the order of 1 to 10 percent) compared to the very small extensions (less than 0.01 percent) observed at the same temperatures and peak stresses in the isostatic creep tests. In the TZC alloy the dynamic loading increased the steady state creep rate by approximately two orders of magnitude; however, this acceleration was not observed for the T-111 alloy. Possible reasons for this behavior are discussed in terms of previous observations concerning the isostatic creep behavior of these materials.

INTRODUCTION

In space power systems, refractory metal alloys are selected as materials for high temperature components because of their compatibility with the environment and their strength and creep resistance. However, in many instances the static creep load is accompanied by high frequency vibratory stresses which may actually represent a limiting design condition. For example, literature data indicate that

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[†]TRW Equipment Laboratories, Cleveland, Ohio.

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below approximately 900° F (482° C) unalloyed molybdenum has a well-defined endurance limit with a ratio of fatigue strength to ultimate strength of approximately 0.7 (ref. 1). At higher temperatures the fatigue curve exhibits no characteristic endurance limit, and the fatigue strength continuously decreases with an increasing number of test cycles. In addition to representing a specific failure mode, the presence of vibratory stresses superimposed on a static load can also lead to a rapid acceleration of creep extension when metals are tested below a homologous temperature of approximately 0.5 (refs. 2 and 3). A consideration of this type of behavior indicates that a component could fail by extensive creep which is induced by the presence of the cyclic load. Constant load creep data obtained without regard to the influence of cyclic stresses would then provide a nonconservative design stress value.

The purpose of this investigation was to study the high frequency, high temperature fatigue behavior of the molybdenum-base alloy TZC and the tantalum-base alloy T-111. The data obtained from this study will provide an indication of the relative importance of fatigue as a possible failure mode and a measure of the influence which cyclic stressing can have on the creep behavior.

MATERIALS AND PROCEDURE

The compositions and processing histories for the TZC and T-111 alloys used in the tests are given in tables VI-1 and VI-2. The TZC was recrystallized at 3092° F (1700° C) for 1 hour, and the T-111 at 3000° F (1649° C) for 1 hour in vacuum prior to testing. The conventional smooth tensile properties produced by these heat treatments are given in table VI-2. The microstructure for the recrystallized TZC material is presented in figure VI-1, while the structure of the T-111 is shown in figure VI-2.

The two test specimen geometries shown in figure VI-3 were used for the fatigue studies. In the tests on TZC where the mean stress was essentially zero, the notch geometry was required to allow sufficient stress to be generated to produce fatigue failure. (In all cases a very slight static load was present on the specimens as a result of the weight of the fixture used to hold the capacitive pickup for monitoring resonant conditions.) Both smooth and notched specimens were used on TZC for A ratios of approximately 0.45 to provide a measure of the validity of the method used to calculate peak stress in the notch specimens. (A ratio is defined as the dynamic stress single (mean to peak) amplitude divided by the mean stress.)

The tests were conducted in vacuum chambers equipped with ion pumps and tantalum resistance heated furnaces (see fig. VI-4, ref. 3). A resonant drive

train was used to produce the dynamic load with nodes at both the top port seal and the lower attachment point for the static load. The majority of tests were performed with a vibration train driven at approximately 20 kilohertz (kcps) by an externally mounted PZT piezoelectric transducer. Mechanical amplification was attained by suitable stepped-horn type velocity transformers (ref. 4), which provided a maximum displacement of approximately 0.001 inch at the 2000° F test temperature. Selected tests were also conducted at 10 and 15 kilohertz. In these cases appropriate changes were made in the specimen and load train geometry to produce resonance.

The test method involved mechanically mounting the specimen to the drive train, adjusting the capacitive vibration pickup, making preliminary checks to ensure that the system was in resonance, and pumping the unit to a vacuum better than 1×10^{-7} torr at room temperature. Testing was performed at this vacuum except during heating, when it was controlled so that the pressure never exceeded 1×10^{-6} torr.

A W-3%Re/W-25%Re thermocouple placed approximately 1/8 inch from the surface at the specimen midpoint was used for temperature measurement. Because of breakage produced by the vibration, the thermocouple could not be attached directly to the specimen. The temperature was stabilized for approximately 2 hours prior to the initiation of testing.

The application of the high frequency cyclic load produced heating of the fatigue specimen. As a result of a series of preliminary tests, the increment of temperature increase was determined as a function of drive level by optical pyrometer readings. It was then possible to adjust the initial furnace temperature setting to produce the desired temperature level in the specimen at the nodal point when the drive was applied.

The dynamic stress produced by the ultrasonic vibration was determined from displacement measurements made directly on the specimen with a cathetometer. In the smooth specimens two reference points were selected approximately equidistant from the specimen midpoint, and the displacements at these points were determined by averaging 10 readings which showed a variation of approximately 50 microinches. These reference points were slight perturbations in the 15 rms microinch finish on the specimens which were well defined by the specimen was assumed to follow the sinusoidal relation

$$\delta_x = \delta_0 \sin \frac{2\pi x}{\lambda} \quad (1)$$

where $2\delta_x$ is the total measured displacement at a distance x from the node at

the specimen midpoint, δ_o is the maximum amplitude at an antinode, and λ is the resonant wavelength.

The maximum strain $\epsilon_{\max}$ at the midpoint of the dumbbell type specimen was then determined from the equation

$$\epsilon_{\max} = \frac{2\pi\delta_o}{\lambda} \quad (2)$$

and the dynamic stress σ was obtained from the product of the strain and the elastic modulus at the particular test temperature

$$\sigma_{\max} = \epsilon_{\max} E \quad (3)$$

When the notch specimen was employed, the maximum stress σ was calculated on the basis of the following equation:

$$\sigma = K_T \left(\frac{D}{d}\right)^2 \left(\frac{2\pi}{\lambda}\right) \delta_o E \quad (4)$$

where K_T is the elastic stress concentration factor (equal to 1.87), D/d is the ratio of major to minor diameter in the notch specimen (equal to 1.20), and δ_o , E , and λ are defined in equation (1).

In the notch test δ_o was determined from measurements of displacement (δ_x) taken on the major diameter and calculated from equations (1) and (4). The validity of using equation (4) to calculate the effective dynamic stress in notch specimens was determined by conducting both smooth and notch fatigue tests on TZC at an A ratio of 0.45. The results indicated that comparable fatigue curves were generated by both specimen configurations.

Although cracking of the test specimen was accompanied by a significant decrease in the resonant frequency, the tests were continued until the resonant frequency decreased 150 hertz (cps). This condition usually resulted in propagating the fatigue cracks through approximately one-half of the specimen cross section (see fig. VI-5). The time required for this crack propagation was generally short enough so that the fatigue lives reported can be considered as essentially the time required to initiate a fatigue crack.

In the tests on smooth specimens at the A ratios between 0.15 and 0.45, the influence of the cyclic stress on creep properties was evaluated by measuring the specimen extension over the distance between specimen shoulders. This procedure was necessary since gage marks could not be placed on the specimens without significantly altering the fatigue behavior.

RESULTS AND DISCUSSION

Molybdenum-Base Alloy TZC

The fatigue curves for tests conducted on TZC at 1800^o, 2000^o, and 2200^o F (982^o, 1093^o, and 1204^o C) with both the notch and smooth specimens are presented in figure VI-6. No variation in properties was observed as a function of heat of material, and the data for both heats are combined in the figures. A well-defined endurance limit was not present at any of the test temperatures. At 2200^o F (1204^o C) and an A ratio of approximately zero, a fatigue strength as low as 16 000 psi (11.0×10^7 N/m²) was observed at 10^9 cycles. The fatigue strength-tensile strength ratios are shown in table VI-3 for each of the test temperatures. The ratios are considerably lower than the 0.5 to 0.4 usually observed in materials which possess a well-defined endurance limit, such as steels, or the 0.7 value obtained for molybdenum when tested below 875^o F (468^o C), where dislocation locking can occur (ref. 1).

The fatigue results are plotted in the form of a modified Goodman diagram in figure VI-7. At the lower A ratios, the 2000^o and 2200^o F (1093^o and 1204^o C) test temperatures showed comparable fatigue behavior while the strength at 1800^o F (982^o C) was significantly greater.

In addition to actually causing total fatigue failure, the superposition of a dynamic load on a static load can produce a significant acceleration in creep. Typical creep curves obtained for TZC under cyclic load conditions at 1800^o, 2000^o, and 2200^o F (982^o, 1093^o, and 1204^o C) are shown in figures VI-8, VI-9, and VI-10. For comparative purposes, a creep curve obtained by testing under static load in a high vacuum environment is also presented in figure VI-9. The superposition of a dynamic stress on the static stress caused a very marked increase in total extension. Under the static-dynamic loading, the specimen exhibited a large initial extension within the first 30 minutes of testing, followed by a period where an approximately constant creep rate occurred. To further analyze the factors which control both the initial extension and the constant creep rate, the parameters ϵ_1 and ϵ_2/t were defined as shown schematically in figure VI-11 and were plotted logarithmically as a function of peak stress in figures VI-12 and VI-13. The initial specimen extension (ϵ_1) which occurred early in the test sequence was essentially independent of the temperature in the 1800^o to 2200^o F (982^o to 1204^o C) range and A ratios in the 0.10 and 0.45 range. In cases where the dynamic drive on the specimen was removed for a period of time, and then reinitiated, the values of ϵ_1 were greatly reduced. The influence of peak stress on the steady state creep rate (ϵ_2/t) shown in figure VI-13 indicated that the creep rate under combined static-dynamic loading

conditions increased with increasing test temperature. By way of comparison, the creep rates obtained under static conditions at essentially the same peak stress values (i. e., $A = 0$) were almost two orders of magnitude less than those present in the tests where combined static-dynamic loading was used. Conversely, at the high A ratios ($A \approx \infty$), no creep was observed so that the creep rate as a function of A ratio reached a maximum value at about $A = 0.45$ (see fig. VI-14). The results indicate that relatively low values of vibratory stress superimposed on a static load can produce a significant increase in the steady state creep of TZC in the 2000° F (1093° C) temperature range.

The variation of fatigue strength as a function of frequency of load application is shown in figure VI-15. Decreasing the loading rate at 2000° F (1093° C) from 20 to 15 kilohertz resulted in a marked increase in fatigue life, while a further decrease to 10 kilohertz did not produce any additional variation. The decreasing test frequency also produced a retardation in the creep rate at an A ratio of 0.45. The variation in steady state creep rate at 2000° F (1093° C) could be expressed over the 10 to 20 kilohertz range as

$$\dot{\epsilon}_2 = Ae^{Bf}$$

where $\dot{\epsilon}_2$ is the steady state creep rate ϵ_2/t (in %/hr), f is the test frequency (in kHz), and A and B are constants equal to 5.42×10^{-3} and 0.281, respectively.

Tantalum-Base Alloy T-111

The fatigue strength of T-111 as a function of cycles to failure is shown in figure VI-16 along with the comparative data for TZC. At 2000° F (1093° C) and 20 kilohertz the fatigue strength of T-111 was considerably greater than that obtained on the TZC alloy, despite the fact that the two materials had comparable tensile strengths.

The influence of ultrasonic vibration on the creep behavior of T-111 is illustrated in figure VI-17, which shows the very rapid first stage extension immediately upon application of the cyclic stress. The magnitude of this extension was comparable in both the T-111 and TZC alloys despite the higher fatigue strength of the T-111.

The T-111 was not susceptible to the dramatic acceleration of steady state creep when exposed to high frequency cyclic stresses as was the case for the TZC when comparisons were made with long time steady state creep rates. The iso-static and dynamic creep rates for T-111 are compared in figure VI-18 on the basis

of a temperature compensated creep parameter (paper IV of this volume). When the dynamic creep data were corrected for the nonlinear dependence of creep on stress which was present during the application of the cyclic loads, the agreement between the creep under steady state and steady state plus dynamic loads was excellent.

General Discussion

In the case of the T-111 alloy no rapid steady state creep rate was observed when the creep behavior under dynamic conditions was compared to the constant load steady state creep which occurred in T-111 after the transition in creep rate. This isostatic creep rate transition which is shown schematically in figure VI-19 has been directly correlated with the removal of hafnia particles from the microstructure and a loss of oxygen to the high vacuum test environment, which eliminates a strain aging mechanism that retards creep (paper IV of this volume). In the case of the fatigue tests, electron micrographs indicated that the same structural refinement which occurred in several thousand hours of creep testing occurred in less than 100 hours when the dynamic load was applied. A significant oxygen loss was also measured in the fatigue tested samples; for example, post-test analyses were generally below 10 ppm as compared to 100 ppm before testing. On the basis of these results the cyclic stressing was believed to have accelerated the oxygen removal mechanism and produced a rapid steady state creep which was comparable to the steady state creep obtained in T-111 under constant load conditions after long periods of time.

In the case of TZC no microstructural variation was observed after fatigue testing and no creep transition was observed after long time isostatic creep exposure. The explanation for the observed behavior in T-111 cannot then be applied to TZC, and presently no satisfactory mechanism has been developed to quantitatively account for the accelerated creep behavior in this material. In addition, the very rapid first stage extension which was observed in both T-111 and TZC still requires further investigation to determine both its basic cause and its importance to component design situations.

SUMMARY OF RESULTS

High frequency fatigue tests were conducted in an ultrahigh vacuum environment on molybdenum-base TZC and tantalum-base T-111 alloys in the temperature range of 1800° and 2200° F (982° and 1204° C). In this temperature range no well-

defined endurance limit existed for either material, and at 10^9 cycles fatigue strengths as low as 16 000 psi (11.0×10^7 N/m²) were observed for TZC and 34 000 psi (24.8×10^7 N/m²) for T-111.

In addition to classical fatigue failure, the superposition of a cyclic stress on a static stress produced a substantial increase in specimen extension. This increase occurred as an initially rapid creep rate during approximately the first 30 minutes of testing followed by a period where the creep rate was essentially constant. In both TZC and T-111, the rapid first stage creep was linearly related to the applied peak stress value, and this relation was independent of material, A ratio, and temperature over the range of variables studied. Decreasing the test frequency from 20 to 10 kilohertz increased the fatigue strength in TZC and minimized the acceleration in creep behavior produced by the application of cyclic stresses. In the steady state creep range the presence of the cyclic stresses produced a several order of magnitude increase in creep in the TZC alloy. However, in T-111 this acceleration was not observed when the comparisons were made on the basis of the long time isostatic creep where the structural refinement and loss of oxygen occurred in the alloy. In T-111 the application of the high frequency cyclic stresses increased the rate at which this loss of oxygen took place by several orders of magnitude. In the case of TZC the accelerated creep rate due to the superposition of cyclic stresses on a static load could not be explained in terms of a structural or compositional variation, and the specific mechanism was not defined.

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TABLE VI-1. - CHEMICAL COMPOSITIONS OF TZC AND T-111 ALLOYS

(a) TZC alloy

Material	Heat	Form	Vendor	Composition, wt %			
				Zr	Ti	C	Mo
TZC	4345	Plate	Climax Molybdenum	0.15	1.24	0.13	Bal.
TZC	4350	Plate	Climax Molybdenum	.17	1.30	.125	Bal.

(b) T-111 alloy

Material	Heat	Form	Vendor	Composition							
				wt %			ppm				
				W	Hf	Ta	C	O	N	H	
T-111	650038	Plate	Wah Chang	8.6	20	Bal.	25	100	20	2.8	

TABLE VI-2. - SMOOTH TENSILE PROPERTIES OF TZC AND T-111 ALLOYS

AFTER 1 HOUR RECRYSTALLIZATION

Test temperature		Tensile strength		0.2 Percent yield strength		Reduction in area, %	Elongation, %
°F	°C	ksi	N/m ²	ksi	N/m ²		
TZC recrystallized at 3090 ^o F (1700 ^o C)							
75	24	87.8	6.05×10 ⁸	50.3	3.47×10 ⁸	16.8	19.4
1800	982	63.0	4.34	27.4	1.89	67.5	29.3
2000	1093	60.8	4.19	24.1	1.66	69.7	28.2
2200	1204	54.2	3.71	25.0	1.72	66.2	29.1
T-111 recrystallized at 3000 ^o F (1649 ^o C)							
75	24	92.1	6.35×10 ⁸	76.8	5.29×10 ⁸	47.5	29.8
1800	982	62.9	4.33	32.1	2.21	76.7	29.9
2000	1093	55.2	3.80	31.5	2.17	50.0	28.5
2200	1204	43.6	3.00	28.1	1.94	48.7	34.2

TABLE VI-3. - RATIO OF FATIGUE TO TENSILE STRENGTH FOR
TZC MOLYBDENUM ALLOY, TESTED IN VACUUM
(1×10^{-7} TORR) at 20 KILOHERTZ

Test temperature		Tensile strength		Fatigue strength at 10^9 cycles		Ratio of fatigue to tensile strength
$^{\circ}\text{F}$	$^{\circ}\text{C}$	ksi	N/m^2	ksi	N/m^2	
1800	982	63.0	4.34×10^8	18	1.24×10^8	0.29
2000	1093	60.8	4.19	17	1.17	.28
2200	1204	54.2	3.71	16	1.10	.30

Figure VI-1. - Microstructure of molybdenum-base TZC alloy annealed at 1700°C . Etchant, 15 percent Hf , 15 percent H_2SO_4 , 8 percent HNO_3 , 62 percent H_2O . $\times 100$.

Figure VI-2. - Microstructure of T-111 heat 650038 recrystallized 1 hour at 3000°F (1649°C). $\times 100$.

Figure VI-3. - Geometry of resonant test specimens. All diameter and thread ends must be concentric ± 0.002 total indicator reading, and minimum relief must be used on thread ends.

Figure VI-4. - Vacuum fatigue system.

Figure VI-5. - Appearance of fracture surface of molybdenum-base TZC alloy fatigue tested at 2000° F (1093° C), 19 kilohertz, and $<1 \times 10^{-7}$ torr vacuum. $\times 10$.

Figure VI-6. - Fatigue data for TZC recrystallized at 3092° F (1700° C) and tested at ~20 kilohertz in vacuum environment ($<1 \times 10^{-7}$ torr).

Figure VI-7. - Goodman diagram for 20 kilocycle fatigue strength of TZC alloy at 10⁹ cycles. Tested in vacuum environment ($<1 \times 10^{-7}$ torr).

Figure VI-8. - Creep of TZC under dynamic load conditions. Test temperature, 1800° F; vacuum environment of $<1 \times 10^{-7}$ torr; $A \approx 0.45$.

Figure VI-9. - Creep of TZC under static and dynamic load conditions. Test temperature, 2000° F; vacuum environment of $<1 \times 10^{-7}$ torr.

Figure VI-10. - Creep of TZC under dynamic load conditions. Test temperature, 2200° F; vacuum environment of $<1 \times 10^{-7}$ torr.

Figure VI-11. - Schematic illustration of typical creep behavior under combined static-dynamic loading conditions. $\epsilon_1 > \epsilon_4$; $\epsilon_2 > \epsilon_3$.

Figure VI-12. - Variation in initial creep extension with applied peak stress in cyclically loaded TZC alloy. Vacuum environment of $<1 \times 10^{-7}$ torr; $A \approx 0.45$ to 0.10 .

Figure VI-13. - Influence of peak stress in dynamic tests on creep rate of TZC. Vacuum environment of $<1 \times 10^{-7}$ torr; $A \approx 0.45$.

Figure VI-14. - Variation of $\dot{\epsilon}_2$ with A ratio for fatigue tests at 20 kilocycles per second and 2000° F; TZC annealed in ultrahigh vacuum environment. $\dot{\epsilon} = 0$ at $A = \infty$.

Figure VI-15. - Fatigue data for smooth specimens of TZC recrystallized at 3092° F (1700° C) and tested at 10 and 15 kilohertz in vacuum environment of $<1 \times 10^{-7}$ torr. Test temperature, 2000° F (1093° C); A ratio = 0.45.

Figure VI-16. - Fatigue data for T-111 compared to TZC at 20 kilohertz and 2000° F (1093° C).
A ratio = 0.25.

Figure VI-17. - Schematic representation of T-111 dynamic creep behavior at 2000° and 2200° F (1093° and 1204° C), shown superimposed on a static creep curve at same temperature and mean stress.

Figure VI-18. - Comparison of ultrasonic fatigue and conventional steady state creep rates in T-111 tested in ultrahigh vacuum.

Figure VI-19. - Schematic representation of T-111 creep behavior in the 1600° to 2200° F (870° to 1204° C) range, showing unusual nature of transient first stage creep in this alloy.