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ELEVATED TEMPERATURE FATIGUE OF TZC MOLYBDENUM ALLOY UNDER HIGH FREQUENCY AND HIGH VACUUM CONDITIONS

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by

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FOREWORD

The work described in this report was performed under National Aeronautics and Space Administration Contract NAS 3-6010. The purpose of the study was to obtain fatigue life data on refractory alloys for use in designing space power systems. The program is under the direction of Paul E. Moorhead, Technical Manager for the Space Power Systems Division of NASA-Lewis Research Center.

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ABSTRACT

High frequency fatigue tests were conducted in a high-vacuum environment on recrystallized molybdenum-base alloy TZC 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. Fatigue strengths at 2200°F (1204°C) and 10^9 cycles were as low as 16,000 psi. The application of a cyclic load to a statically-loaded specimen produced a marked acceleration in the degree of specimen extension. This increase was characterized by a relatively rapid extension during the first thirty minutes of testing followed by a period of steady state creep which was approximately two orders of magnitude greater than the creep rate observed under a comparable static peak stress.

Author

INTRODUCTION

In a variety of high temperature component applications refractory alloys are selected because of their superior creep resistance. However, in many instances the static creep load is accompanied by vibratory stresses which may actually represent a limiting design condition. Current data (1)* indicate that below approximately 900°F unalloyed molybdenum has a well-defined endurance limit with a ratio of fatigue limit to ultimate strength of about 0.7. At higher temperatures the fatigue curve exhibits no characteristic endurance limit and the fatigue strength appears to continuously decrease with an increasing number of test cycles. At 1050°F, the fatigue strength of unalloyed molybdenum is as low as 20,000 psi at 10^7 cycles (1).

The purpose of this investigation was to study the fatigue behavior of a high-strength molybdenum base alloy to determine whether the fatigue strength represents a more serious design limitation than static creep. In addition, the tendency for the cyclic load superimposed on a static load to promote accelerated creep was also evaluated. In order to minimize possible complications due to environmental effects, the tests were conducted in vacuums less than 1×10^{-7} torr. Loading frequencies between 19 and 20 kHz (Kcs) were used to facilitate study in the high cycle failure range.

MATERIALS AND PROCEDURE

The composition and processing history for the molybdenum-base TZC alloy used in the tests are given in Tables 1 and 2. The TZC was recrystallized at 3090°F (1700°C) for one hour in vacuum prior to testing and the conventional smooth tensile properties produced by this heat treatment are given in Table 2. The microstructure for the recrystallized TZC material is presented in Figure 1.

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* Numbers in parentheses pertain to references on Page 23.

TABLE 1

CHEMICAL COMPOSITION OF TZC ALLOY TESTED

			Composition-Weight %			
Heat	Form	Vendor	Zr	Ti	C	Mo
4345	Plate	Climax Molybdenum	0.15	1.24	0.13	Bal.

Processing History: (1) Machine vacuum-arc-melted ingot to 5.85" dia; (2) Extrude to 3" dia; (3) Heat treat in vacuum at 3000°F (1649°C); (4) Machine to 2.4-2.8 dia; (5) Upset forge 40% at 2400°F (1316°C); (6) Broad forge to 0.825" at 2400°F (1316°C); (7) Heat treat in vacuum at 2400°F (1316°C) for 1 hour; (8) Machine to 0.70" thick.

TABLE 2

SMOOTH TENSILE PROPERTIES* OF TZC ALLOY TESTED

Test Temperature		Tensile Strength		0.2% Yield Strength		% Red. Area	% Elongation	Elastic Modulus (psi) Used in this Investigation
°F	°C	Ksi	$10^8 N/m^2$	Ksi	$10^8 N/m^2$			
75	24	87.8	6.05	50.3	3.47	16.8	19.4	
1800	982	63.0	4.34	27.4	1.89	67.5	29.3	38.7×10^6
2000	1093	60.8	4.19	24.1	1.66	69.7	28.2	35.8×10^6
2200	1204	54.2	3.71	25.0	1.72	66.2	29.1	32.3×10^6

* Annealed 1 hour at 1700°C.

FIGURE 1 MICROSTRUCTURE OF MOLYBDENUM-BASE TZC ALLOY, ANNEALED 1700°C, 100X, ETCHANT: 15% HF, 15% H_2SO_4, 8% HNO_3, 62% H_2O.

The two test specimen geometries shown in Figure 2 were used for the fatigue studies. In the tests where the mean stress was essentially zero*, the notch geometry was required to allow sufficient stress to be generated to produce fatigue failure. Both smooth and notched specimens were used 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.

The tests were conducted in vacuum chambers, see Figure 3, equipped with ion pumps and tantalum resistance heated furnaces. 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, see Figure 4. The vibration train was driven at approximately 20 kHz (Kcs) by an externally mounted PZT piezoelectric transducer. Mechanical amplification was attained by suitable stepped-horn type velocity transformers (2) which provided a maximum displacement of approximately 0.001" at the 2000°F test temperature.

The test method involved mechanically mounting the specimen to the drive train, adjusting the capacitive vibration pick-up, making preliminary checks to insure 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 which 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. Due to breakage produced by the vibration, the thermocouple could not be attached directly to the specimen. The temperature was stabilized for approximately two 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

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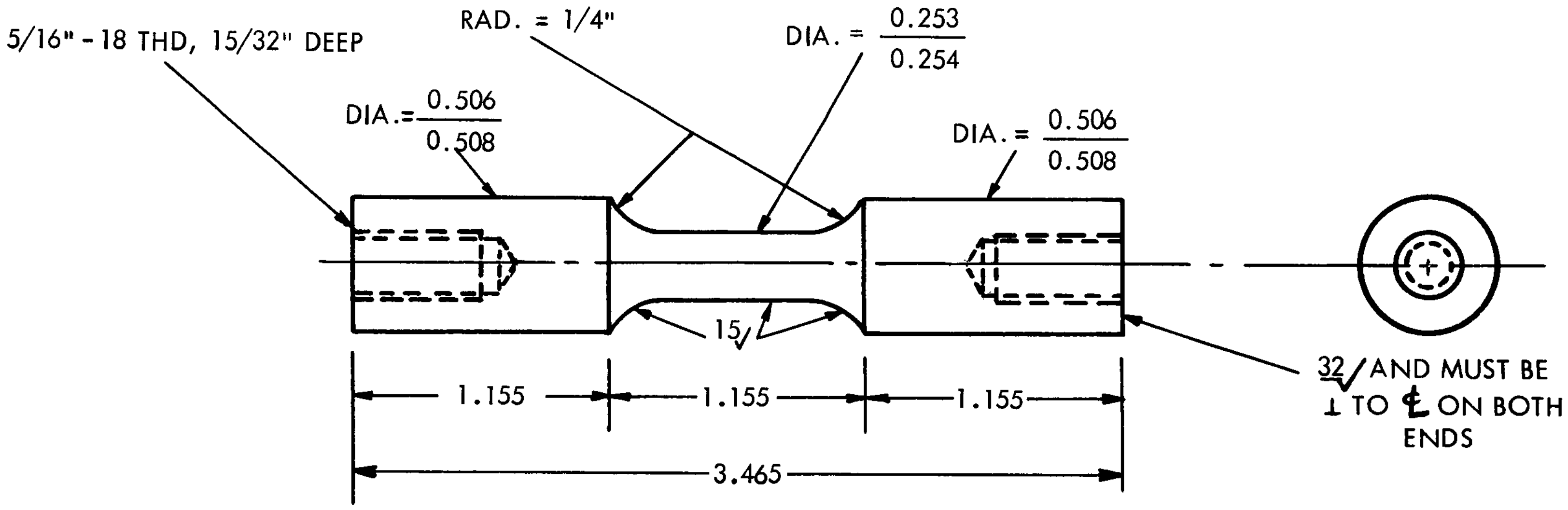
* In all cases a very slight static load was present on the specimens as a result of the weight of the fixture used to hold the capacitive pick-up for monitoring resonant conditions.

** 'A' ratio is defined as the dynamic stress amplitude divided by the mean stress.



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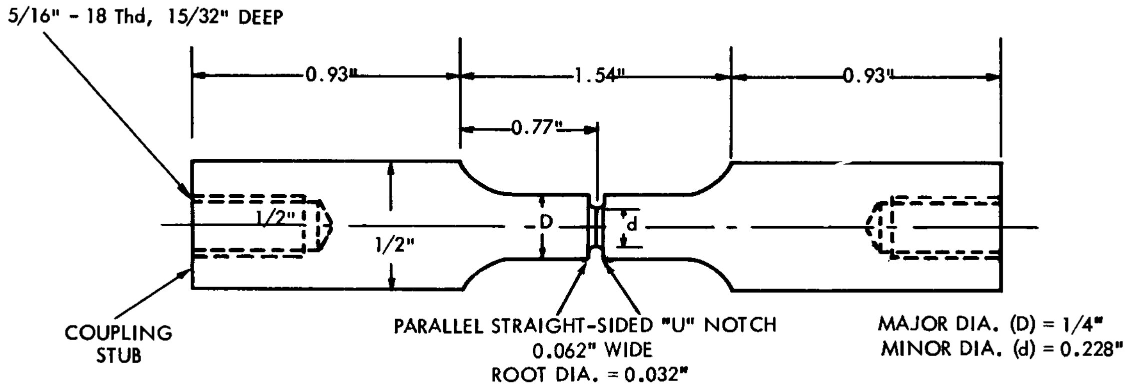

FIGURE 2 GEOMETRY OF RESONANT TZC TEST SPECIMENS

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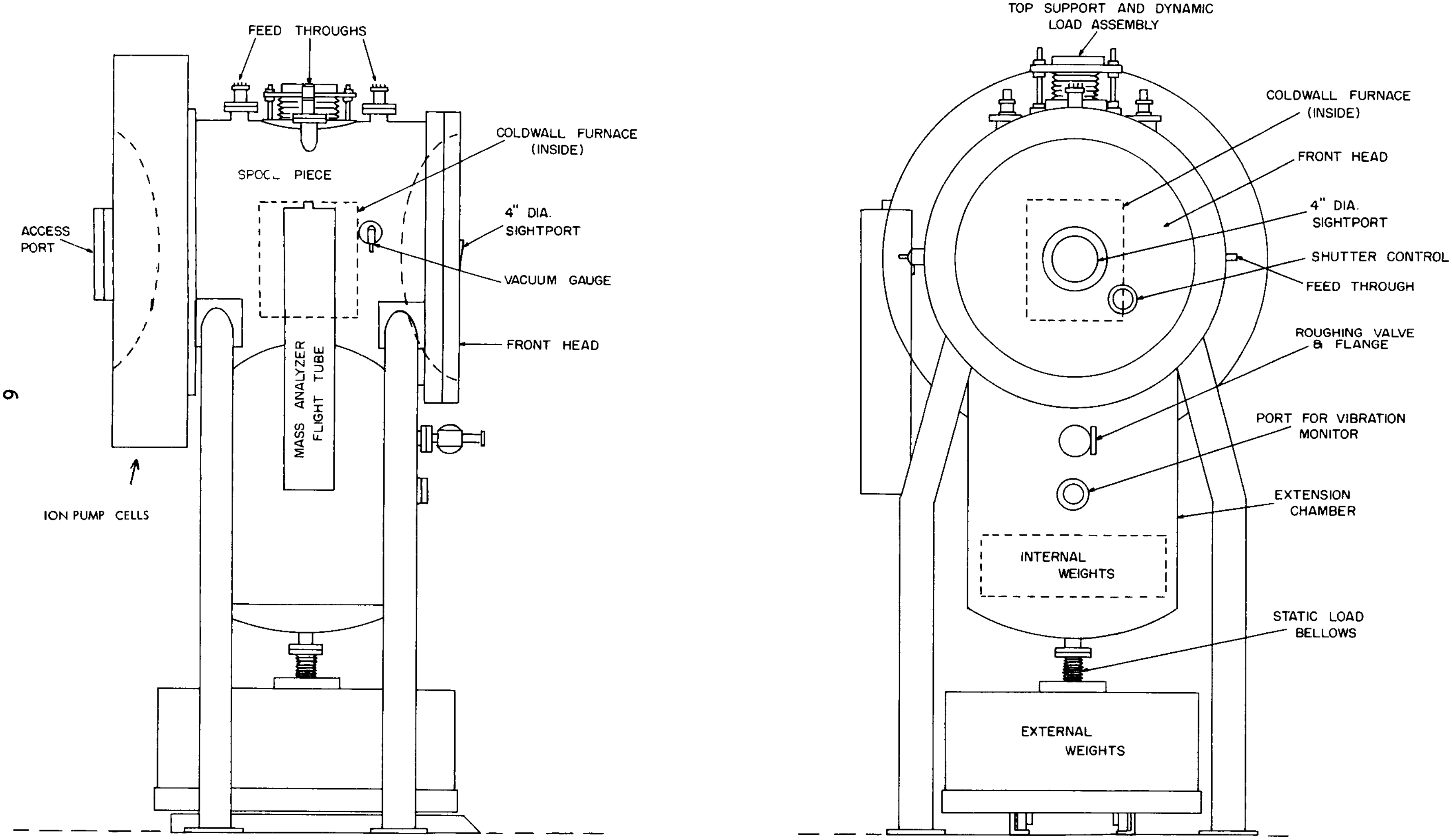


SIDE VIEW

FRONT VIEW

FIGURE 3 VACUUM FATIGUE SYSTEM

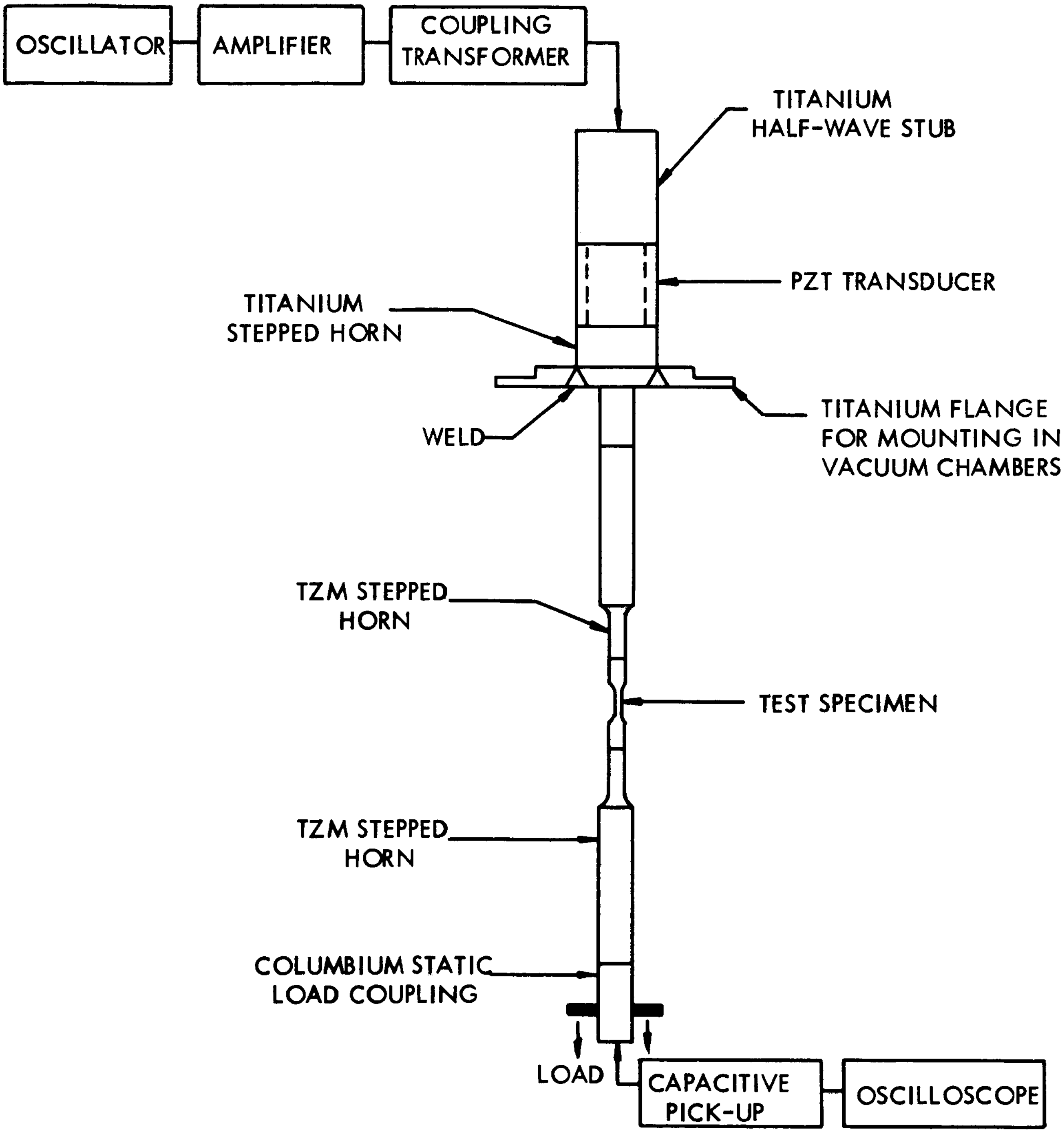


FIGURE 4 DESIGN OF ULTRASONIC DRIVE TRAIN

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 μ-inches. These reference points were slight perturbations in the 15 RMS μ-inch finish on the specimens which were well defined by the reflection of the cathetometer light source. The displacement along the specimen was assumed to follow the sinusoidal relationship:

$$\delta_x = \delta_o \sin \frac{2 \pi x}{\lambda} \tag{1}$$

where 2 δx 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 wave length.

The maximum strain (ϵ max) at the midpoint of the dumb-bell type specimen was then determined from the equation:

$$\epsilon_{\max} = \frac{2 \pi \delta_o}{\lambda} \tag{2}$$

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

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

On the basis of displacement measurements taken on a vibrating bar, an elastic modulus at 2000°F was determined which agreed very closely with previously reported dynamic measurements for molybdenum (3). On this basis, the modulus for molybdenum was used to compute the stresses at 1800 and 2200°F (982 and 1204°C). A summary of the moduli used is given in Table 2.

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

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

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 tests δ_o was determined from measurements of displacement (δx) 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 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 Hz (cps). This condition usually resulted in propagating the fatigue cracks through approximately one-half of the specimen cross-section (see Figure 5).

In the tests on smooth specimens at the an A ratio of approximately 0.45, the influence of the cyclic stress on creep properties was evaluated by measuring the specimen extension over the 1.155 inch distance between specimen shoulders. This procedure was necessary since gauge marks could not be placed on the specimens without significantly altering the fatigue behavior. Although the creep was measured over the 1.155 inch shoulder separation distance, the majority of the extension actually occurred over the 0.860 inch gauge section. An arbitrary guage length of one inch was assumed in reporting the creep test data.

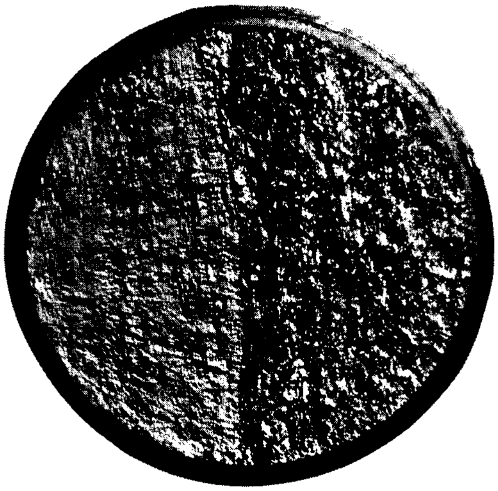


FIGURE 5 APPEARANCE OF FRACTURE SURFACE OF MOLYBDENUM-BASE TZC ALLOY, FATIGUE TESTED AT 2000°F (1093°C), 19 KHz, $<1 \times 10^{-7}$ TORR VACUUM, 10X.

RESULTS AND DISCUSSION

The fatigue curves for tests conducted at 1800, 2000, and 2200°F (982, 1093, and 1204°C) with both the notch and smooth specimens are presented in Figure 6. A well-defined endurance limit was not present at any of the test temperatures. At 2200°F (1204°C) and an A ratio of approximately 00, a fatigue strength as low as 16,000 psi was observed at 10^9 cycles. The fatigue strength-tensile strength ratios are shown in Table 4 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 (4) or the 0.7 value obtained for molybdenum when tested below 875°F (468°C) where dislocation locking can occur.

TABLE 4

Ratio of Fatigue-to-Tensile Strength TZC Molybdenum Alloy, Tested in Vacuum, A$\simeq$00 1 x 10^{-7} Torr at 20 Kcs

Test Temperature		Tensile Strength		Fatigue Strength at 10^9 cycles		Ratio Fatigue-to-Tensile Strength
°F	°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	4.19×10^8	17	1.17×10^8	0.28
2200	1204	54.2	3.71×10^8	16	1.10×10^8	0.30

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

In many high temperature applications where refractory alloys are employed, conditions of cyclic vibration are superimposed on the static load. A comparison of the fatigue life of the TZC alloy with the creep life is given in Figure 8. The comparison is made using the Larson-Miller parameter where T represents the test temperature in absolute units and t is the time to failure

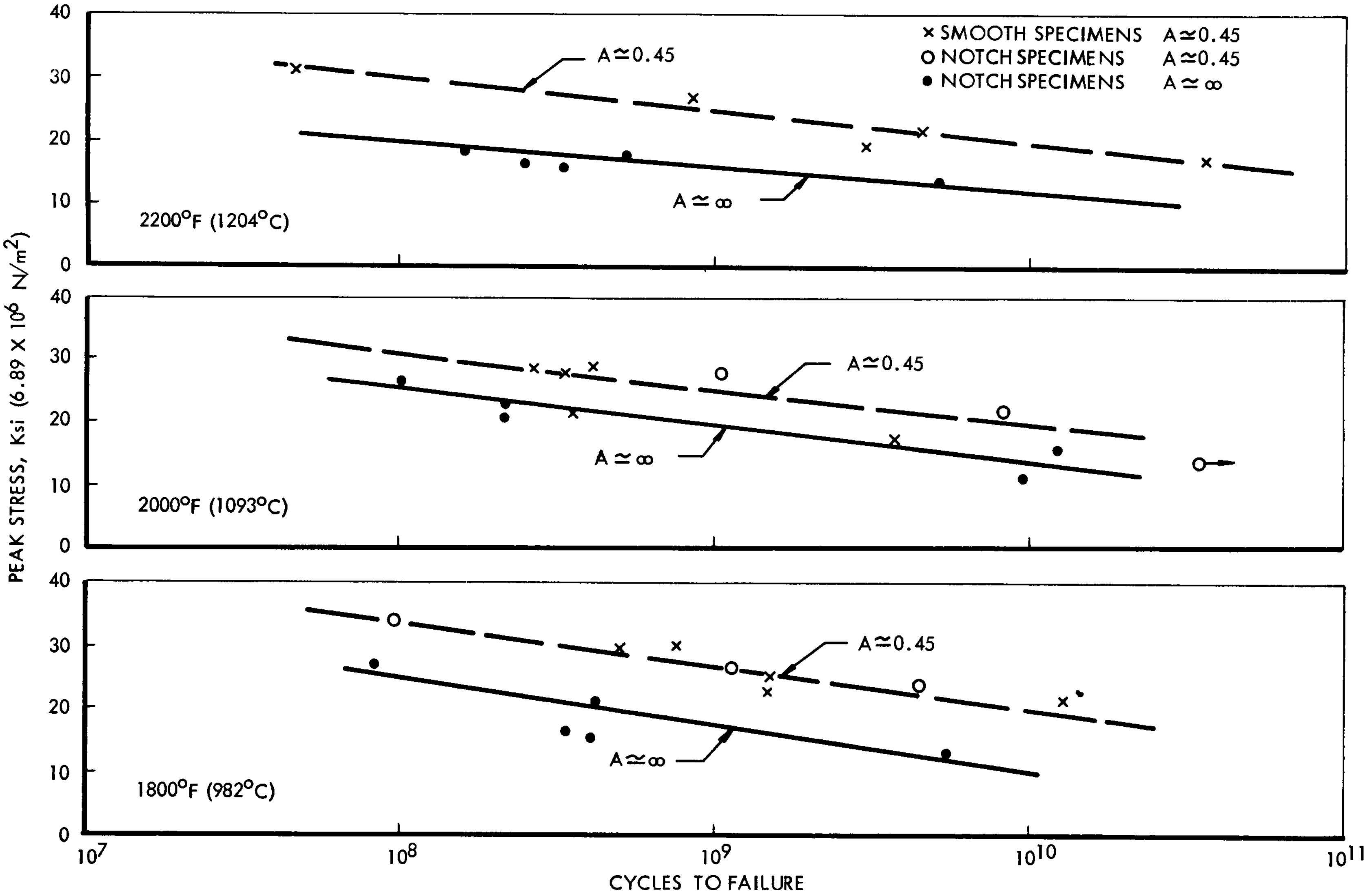


FIGURE 6 FATIGUE DATA FOR TZC RECRYSTALLIZED AT 3092°F (1700°C) AND TESTED AT~20 kHz IN VACUUM ENVIRONMENT $< 1 \times 10^{-7}$ TORR.

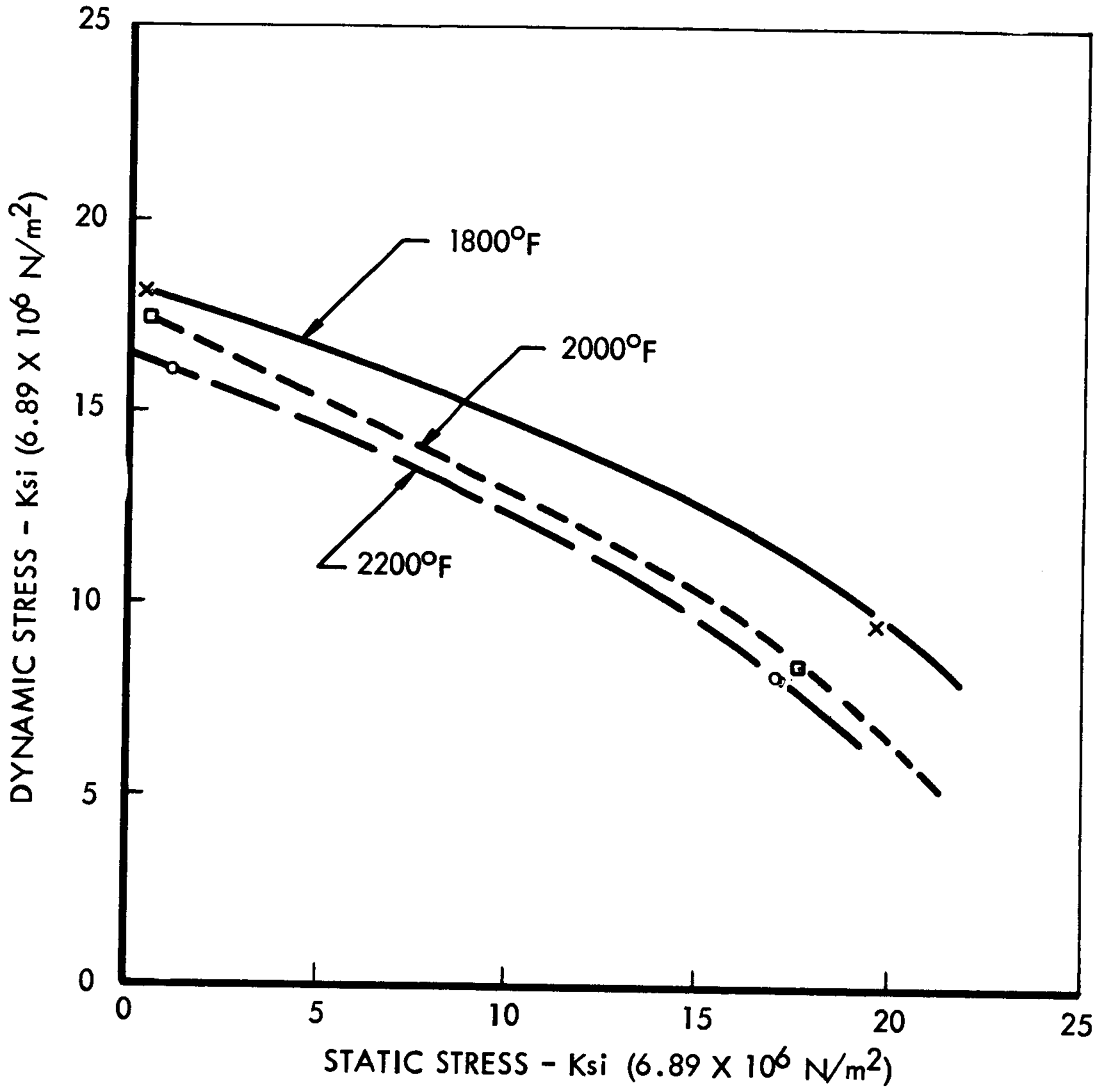


FIGURE 7 GOODMAN DIAGRAM FOR 20KC FATIGUE STRENGTH OF TZC ALLOY AT 10^9 CYCLES, TESTED IN VACUUM ENVIRONMENT $< 1 \times 10^7$ TORR.

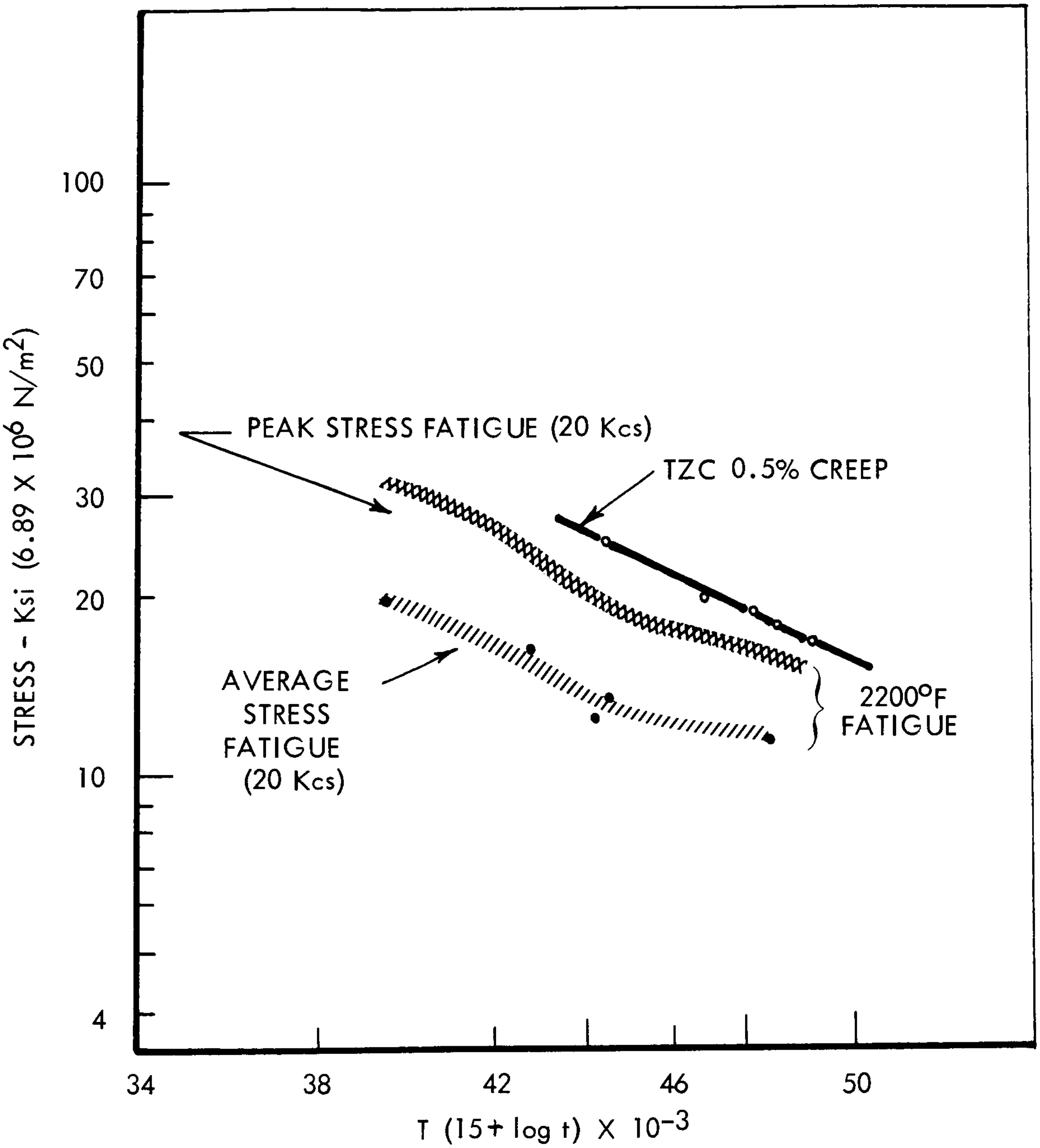


FIGURE 8 COMPARISON OF FATIGUE (20 Kcs) ON SMOOTH BAR SPECIMENS AND CREEP PROPERTIES OF TZC TESTED IN VACUUM ENVIRONMENT <1 X 10^{-7} TORR.

in the fatigue tests or the time to reach 0.5% in previously reported creep tests (5). Although the exact position of the fatigue curve on the time scale is a function of the loading frequency, the results indicate that under certain conditions fatigue can be a more restrictive failure mode in the refractory metals than creep.

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, 2000, and 2200°F (982, 1093 and 1204°C) are shown in Figures 9, 10, and 11. For comparative purposes, a creep curve obtained by testing under static load in a high vacuum environment is also presented in Figure 10. 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 test, 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, shown schematically in Figure 12, were plotted logarithmically as a function of peak stress in Figures 13 and 14. The initial specimen extension (ϵ_1) which occurred early in the test sequence was essentially independent of the temperature in the 1800 to 2200°F (982 to 1204°C) 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 constant creep rate (ϵ_2/t) shown in Figure 14 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 were almost two orders of magnitude less than those present in the tests where combined static-dynamic loading was used. The accelerated creep effects produced by cyclic loading were therefore apparent in both a very rapid increase in initial specimen extension and a significant increase in the steady state creep rate.

The accelerated creep produced under combined cyclic-static loading is not unique with the TZC alloy or the high frequency testing conditions employed in this evaluation. Feltner and Sinclair have summarized results obtained with cadmium, copper and aluminum and indicated that accelerated creep is produced under combined static-dynamic conditions when the test temperature is below approximately one-half the melting point of the material (6). Similar results

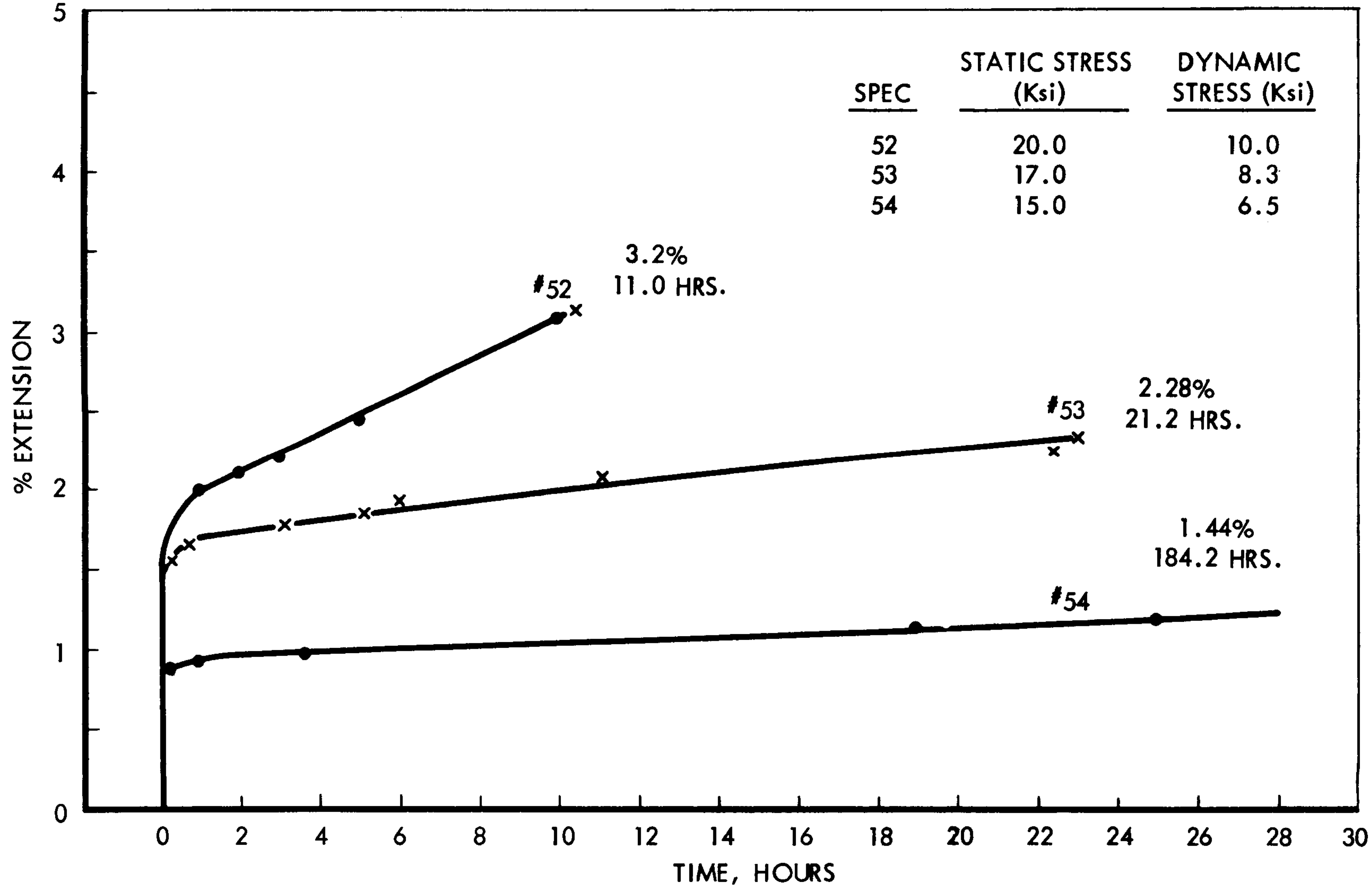


FIGURE 9 CREEP OF TZC UNDER DYNAMIC LOAD CONDITIONS, TEST TEMPERATURE 1800°F, VACUUM ENVIRONMENT $< 1 \times 10^{-7}$ TORR, A≃0.45.

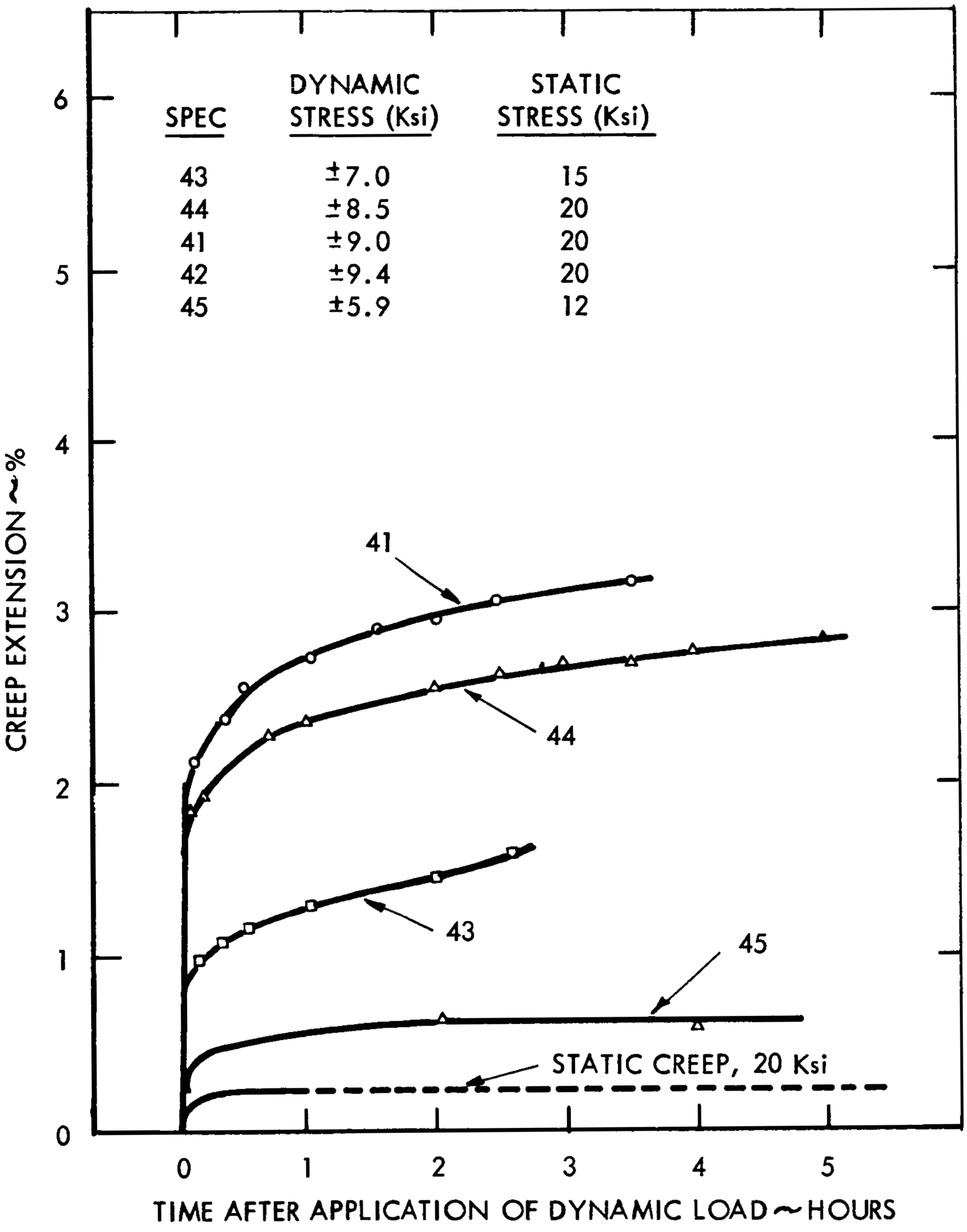
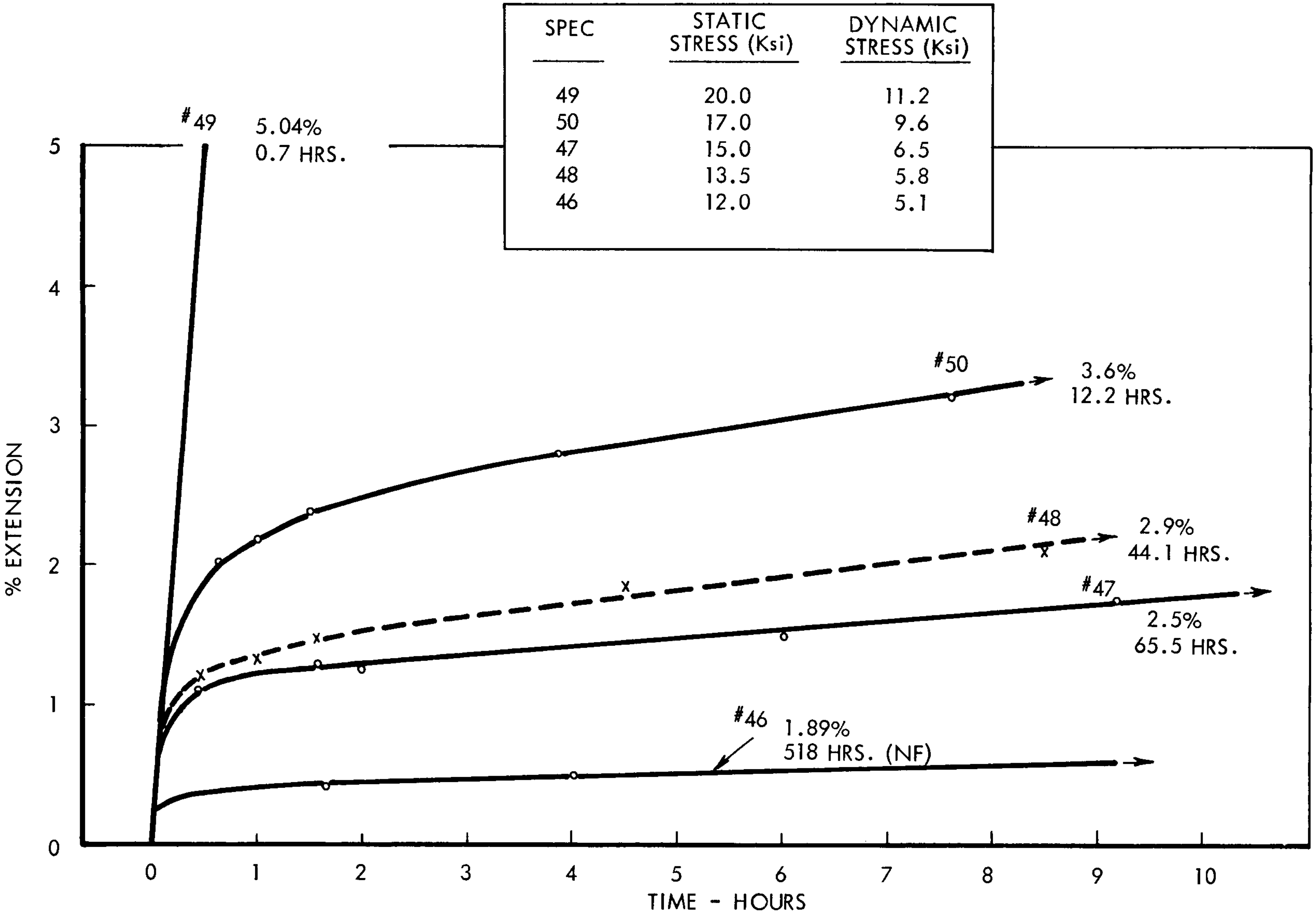


FIGURE 10 CREEP OF TZC UNDER STATIC AND DYNAMIC LOAD CONDITIONS, TEST TEMPERATURE 2000°F, VACUUM ENVIRONMENT $<1 \times 10^{-7}$ TORR.



SPEC	STATIC STRESS (Ksi)	DYNAMIC STRESS (Ksi)
49	20.0	11.2
50	17.0	9.6
47	15.0	6.5
48	13.5	5.8
46	12.0	5.1

FIGURE 11 CREEP OF TZC UNDER DYNAMIC LOAD CONDITIONS, TEST TEMPERATURE 2200°F, VACUUM ENVIRONMENT $<1 \times 10^{-7}$ TORR.

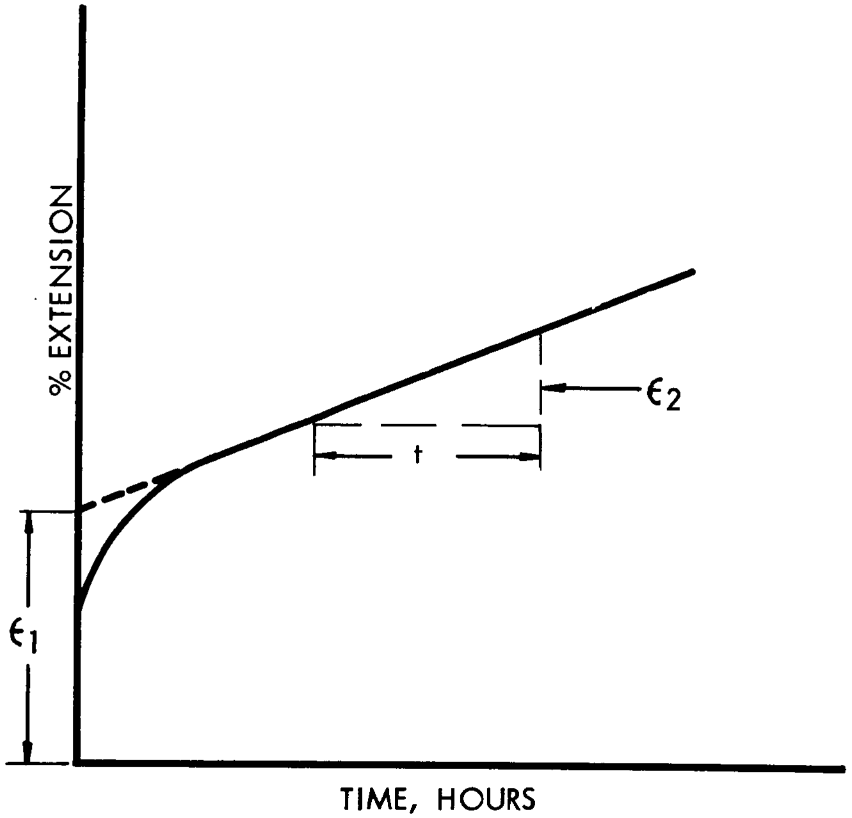


FIGURE 12 SCHEMATIC ILLUSTRATION OF TYPICAL CREEP BEHAVIOR UNDER COMBINED STATIC-DYNAMIC LOADING CONDITIONS.

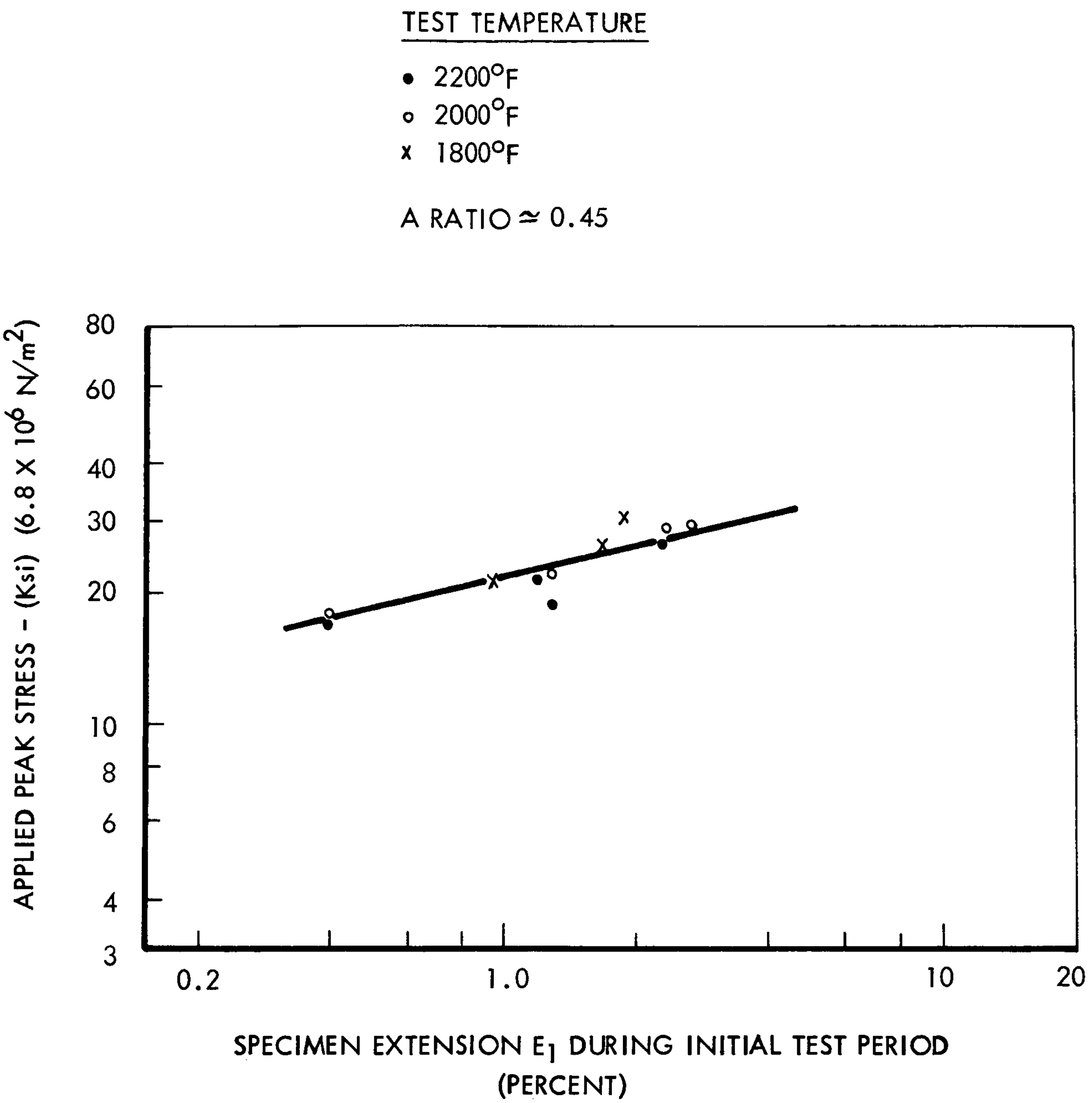


FIGURE 13 VARIATION IN INITIAL CREEP EXTENTION (E_1) WITH APPLIED PEAK STRESS IN CYCLICALLY - LOADED TZC ALLOY ($A \simeq 0.45$), VACUUM ENVIRONMENT $< 1 \times 10^{-7}$ TORR.

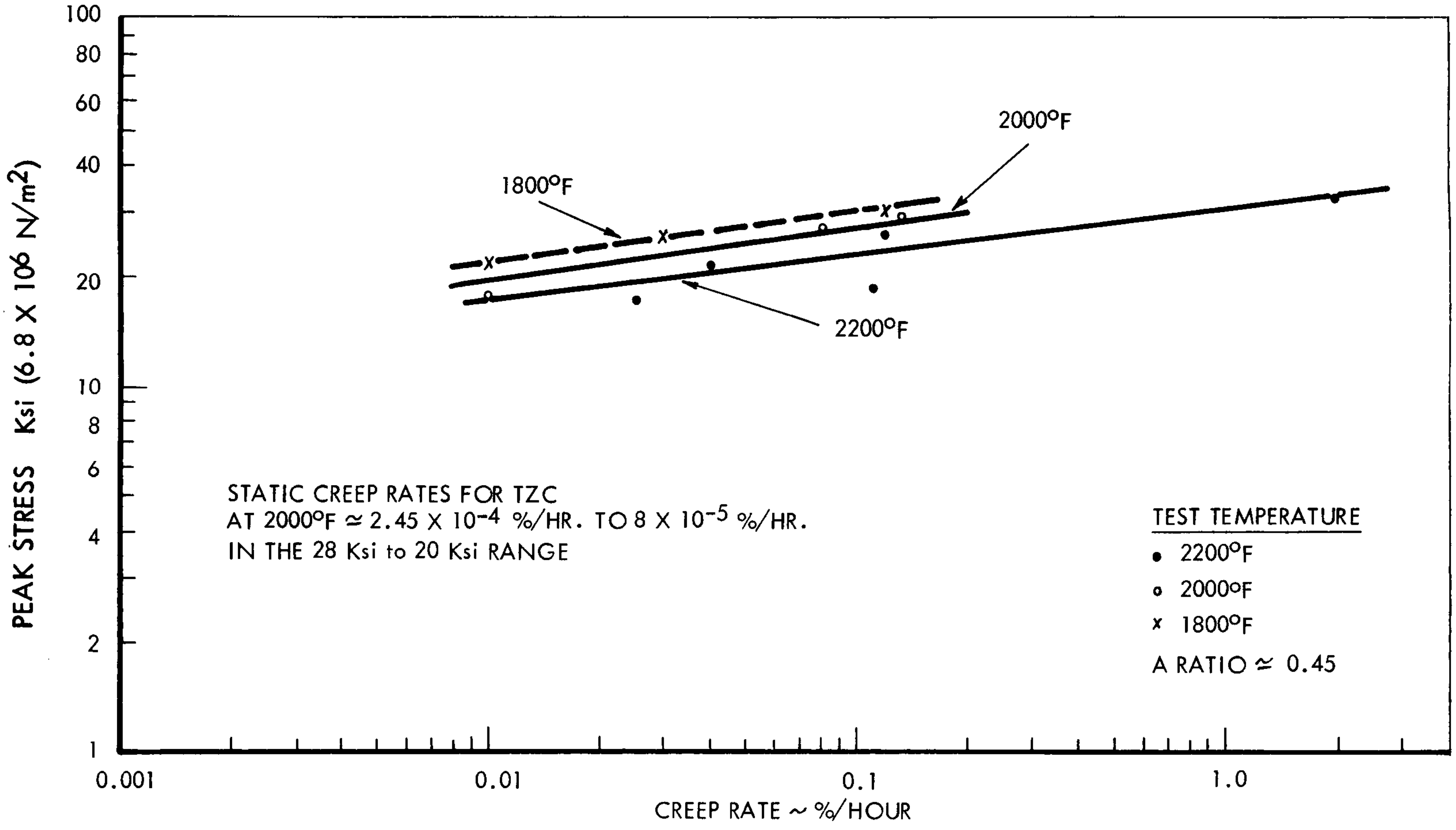


FIGURE 14 INFLUENCE OF PEAK STRESS IN DYNAMIC TESTS ($A \simeq 0.45$) ON THE CREEP RATE OF TZC, VACUUM ENVIRONMENT $< 1 \times 10^{-7}$ TORR.

have been obtained by a number of investigators, working with copper, lead, and nickel (7,8,9) who relate the increased creep to imperfections generated by the cyclic load application. In the case of the TZC alloy, the homologous test temperature is slightly below one-half so that the extensive increase in the creep extension is consistent with previously observed material behavior.

SUMMARY AND CONCLUSIONS

Fatigue tests were conducted in an ultra-high vacuum environment on molybdenum base alloy TZC at a test frequency of 20 kHz (Kcs) and temperatures of 1800,2000,& 2200°F (982, 1093 and 1204°C). A well-defined endurance limit did not occur at any of the test conditions, and fatigue strengths, as low as 16,000 psi, were observed at 10^9 cycles at 2200°F (1204°C). When compared on the basis of a Larson-Miller parameter, fatigue life at the 20 kHz (Kcs) test frequency and an A ratio of 0.45 represented a more restrictive design consideration than creep.

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. The steady-state creep produced under static-dynamic loading conditions was almost two orders of magnitude greater than that produced under comparable test conditions when only a constant load was involved. The results indicate that in the application of refractory alloys under conditions where a combination static-dynamic stress is applied, the acceleration of creep caused by the cyclic loading must be considered along with the problem of classical fatigue cracking.

REFERENCES

1. G. W. Brock and G. M. Sinclair "Elevated Temperature Tensile and Fatigue Behavior of Unalloyed Arc-Cast Molybdenum", Proc. ASTM, 60, 530, (1960).

2. E. A. Neppiras, "Techniques and Equipment for Fatigue Testing at Very High Frequencies", Proc. ASTM, 59, 691, (1959).

3. Molybdenum Metal, Climax Molybdenum Co., (1960).

4. H. A. Lipsitt and G. T. Horne, "The Fatigue Behavior of Decarburized Steel", Proc. ASTM, 57, 586, (1957).

5. J. C. Sawyer and E. A. Steigerwald, "Creep Properties of Refractory Metals in Ultra High Vacuum, Jl. of Materials, (1967).

6. C. E. Feltner and G. M. Sinclair, "Cyclic Stress Induced Creep of Close-Packed Metals:, Int. Conf. on Creep, ASME - Inst. of Mech. Eng., N.Y. (August 25-20, 1963).

7. A. H. Meleka and A.V. Evershed, "The Dependence of Creep Behavior on the Duration of a Superimposed Fatigue Stress", Jl. Inst. of Metals, 88, 411, (1960).

8. J. N. Greenwood, "The Influence of Vibration on the Creep of Lead", Proc. ASTM, 49, 834, (1949).

9. G. A. Webster and B. J. Piearcey, "The Effects of Load and Temperature Cycling on the Creep Behavior of a Nickel-Base Alloy", ASM Trans. Quart., 59, 847 (1966).