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DETERMINATION OF ELEVATED-TEMPERATURE FATIGUE DATA ON REFRACTORY ALLOYS IN ULTRA-HIGH VACUUM

FIFTH QUARTERLY REPORT

Prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
LEWIS RESEARCH CENTER
UNDER CONTRACT NAS 3-6010

TRW *EQUIPMENT LABORATORIES*

CLEVELAND, OHIO

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Fifth Quarterly Report

for

1 July 1965 to 1 October 1965

DETERMINATION OF ELEVATED-TEMPERATURE FATIGUE DATA ON REFRACTORY ALLOYS IN ULTRA-HIGH VACUUM

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
CONTRACT NO. NAS 3-6010

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October 15, 1965

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FOREWORD

The work described herein is being performed by TRW Inc. under the sponsorship of the National Aeronautics and Space Administration under Contract NAS 3-6010. The purpose of this study is to obtain fatigue life data on refractory metal alloys for use in designing space power systems.

The program is administered for TRW Inc. by E. A. Steigerwald, Program Manager, C. R. Honeycutt and J. C. Sawyer are the Principal Investigators.

TABLE OF CONTENTS

		Page No.
I.	INTRODUCTION	1
II.	MATERIALS	1
III.	PROCEDURE	8
IV.	RESULTS AND DISCUSSION	10
	1. Notch Tests	10
	2. Smooth Specimen Tests	15
	3. Comparative Creep and Fatigue Properties	19
V.	FUTURE WORK	19
VI.	BIBLIOGRAPHY	21

I. INTRODUCTION

The purpose of this investigation is to generate fatigue data for refractory alloys at elevated temperatures in ultra-high vacuum environments. The ultimate objective is to determine whether fatigue life or creep is the limiting design parameter in turbine applications involving space-power systems.

During this report period, notched fatigue tests were conducted on TZM alloy in the stress-relieved condition at an ambient temperature of 1800°F (982°C) and on TZC after annealing at 3092°F (1700°C) at an ambient temperature of 2000°F. No well-defined endurance limit was observed in either alloy and fracture occurred in TZC at peak stresses as low as 9,030 psi ($6.22 \times 10^{-9} N/m^2$) in 143 hours ($9.75 \times 10^9$ cycles).

II. MATERIALS

The initial program plan involved testing columbium-base alloy Cb132M and molybdenum-base alloy TZC. The Cb132M, however, could not be forged satisfactorily by the vendor and attempts are currently being made to select an alternate material or material form. The TZC plate material was fabricated according to the two processing cycles shown in Table 1 and the chemical composition of the two heats are shown in Table 2. In addition to the TZC, a TZM alloy in bar form was evaluated and the composition of this material is also given in Table 2.

The TZC material was tested after annealing at 3092°F (1700°C) for 1 hour. This treatment was selected to provide a direct comparison with results of creep tests which are being performed on TZC with a comparable processing history.(1)* The TZM was tested in the as-received condition which consisted of a one hour stress relief treatment at 2250°F (1232°C). The room temperature properties of the test materials are presented in Table 3. In the tensile tests, the TZC specimens were oriented with their tensile axes perpendicular to the rolling direction, while in the TZM samples, the tensile axis was parallel with the axis of the bar stock. This same orientation was maintained during subsequent fatigue tests. The microstructures of the test materials are presented in Figures 1, 2, and 3. The 3092°F (1700°C) annealing treatment produced a recrystallized structure in both heats of TZC material.

* Numbers in parentheses pertain in references in the Bibliography.

TABLE 1

Processing Cycles Used to Fabricate TZC Alloy

I. Processing Cycle No. 1, Heat M-89

a. Vacuum arc melt ingot, 5.88" dia.
b. Machine to 5" dia.
c. Extrude 2.30:1 at 1700°C to 4-1/8" x 2.22" plate,
d. Cut to 4" lengths,
e. Cross-roll at 2925°F (1585°C) in 4" direction to 0.740" using 12" dia. rolls, 4% reduction per pass, hydrogen atmosphere.

II. Processing Cycle No. 2, Heat M-91

Steps a, b, and c same as processing Cycle No. 1
d. Cross-roll on large mill, 28" dia. to produce relatively large degrees of deformation and a finishing temperature of approximately 2372°F (1300°C),
e. Grit blast and cut to final length with abrasive saw.

TABLE 2

Chemical Composition of Alloys Tested

	% Weight				ppm		
	Mo	Zr	Ti	C	H	N	O
TZC Heat M-89	Bal.	0.20	1.45	0.13	5	1	7
TZC Heat M-91	Bal.	0.18	1.25	0.14	1	4	2
TZM Heat 7463	Bal.	0.08	0.48	0.016	-	3	2

TABLE 3

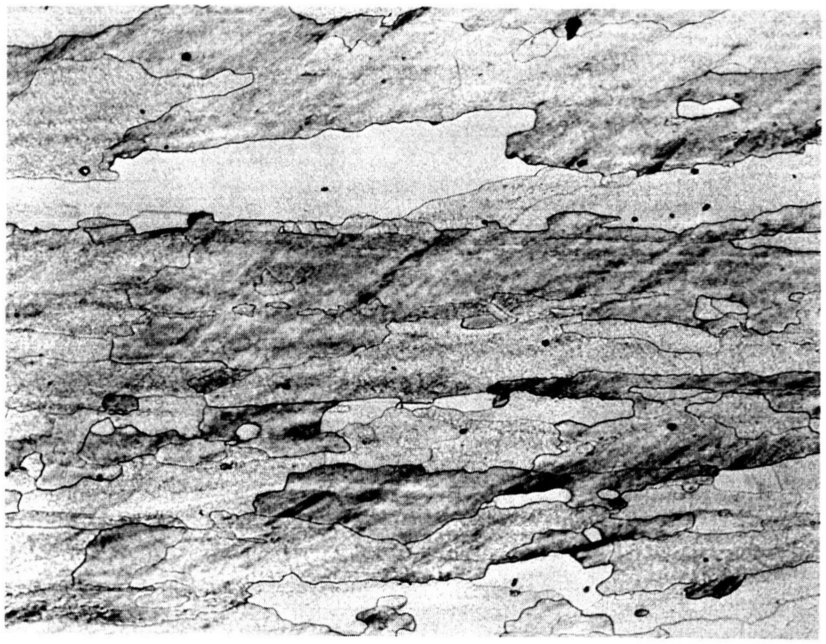
Room Temperature Tensile Properties of TZC and TZM Test Materials

Material	Ultimate Tensile Strength (Ksi, $6.89 \times 10^6 N/m^2$)	0.2% Yield Strength (Ksi, $6.89 \times 10^6 N/m^2$)	Reduction in Area (%)
TZC,* Heat M-80 (same processing as Heat M-89, See Table I) annealed 3092°F (1700°C), 1 Hour.	68.6	68.5	0
TZC, Heat M-91 Annealed 1700°C, 1 Hour,	88.2	47.8	5.6
TZM**, Heat 7463	125.3	112.3	29% Elongation in 4D length

Strain rate 0.005"/"/min.

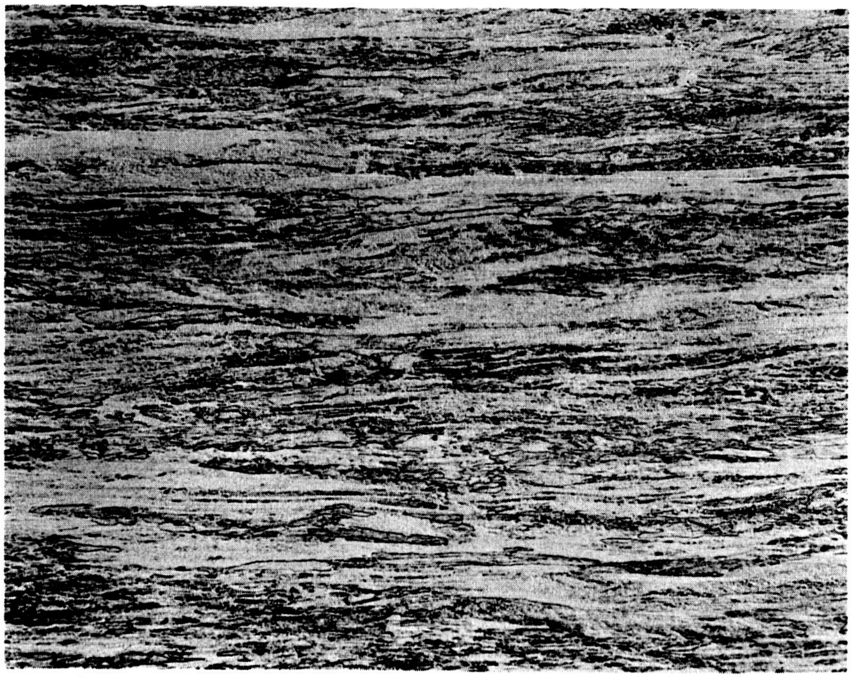
* Specimen failed in a brittle manner so that strength values may not be representative.

** Properties determined by vendor tests.

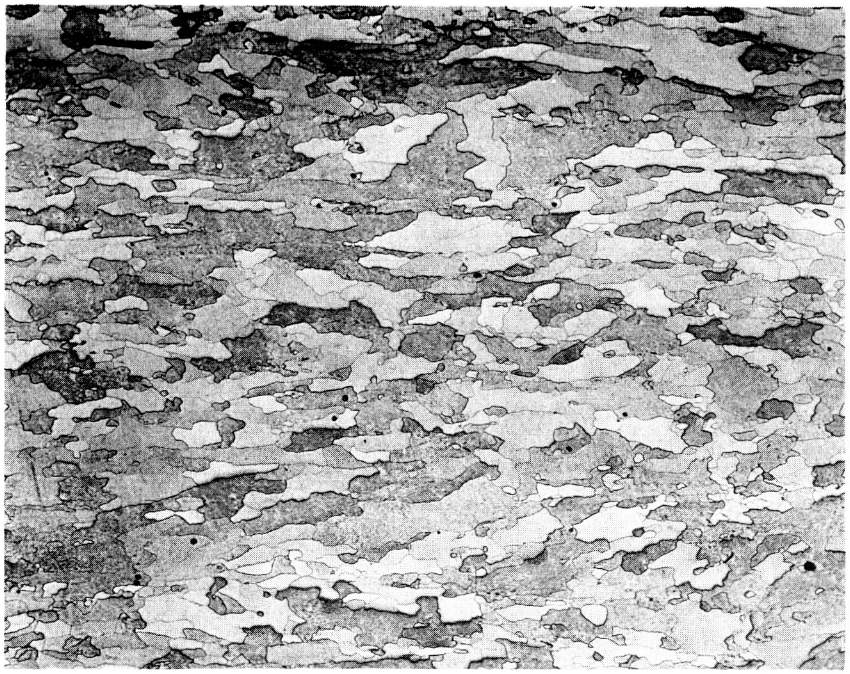


Recrystallized 3092°F
(1700°C), 1 Hour

Figure 1. Microstructure of TZC (Heat M-89), Etchant: 15%HF, 15%H_2SO_4, 8%HNO_3, 62%H_2O, 100X.



As-Received Plate
Cross-Section



Recrystallized 3092°F
(1700°C), 1 Hour

Figure 2. Microstructure of TZC (Heat M-91), Etchant: 15%HF, 15%H_2SO_4, 8%HNO_3, 62%H_2O, 100X.

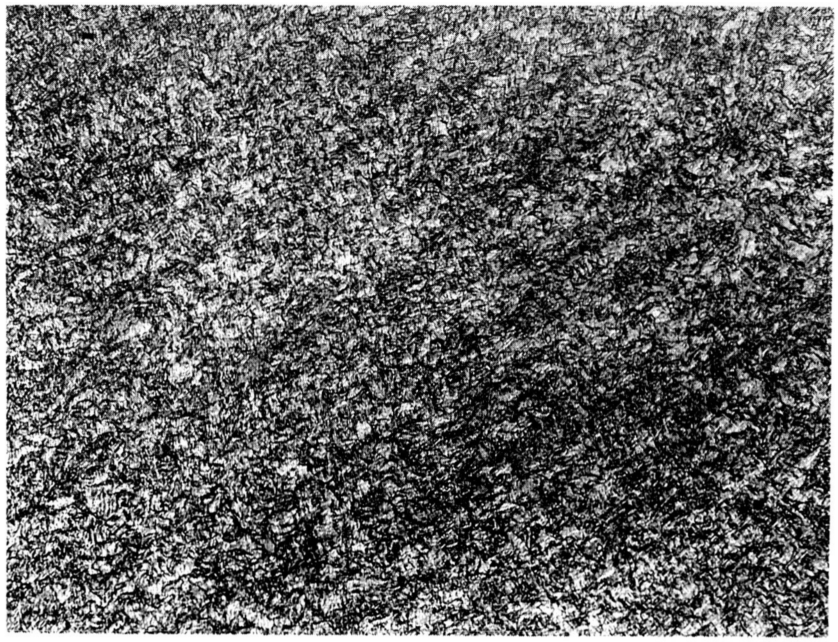


Figure 3. Microstructure of TZM, Bar Stock, Perpendicular to Axis of Bar, Etchant: 15%HF, 15%H_2SO_4, 8%HNO_3, 62%H_2O, 100X.

III. PROCEDURE

The program plan involves fatigue testing the selected alloys as both notch and smooth specimens. Although a 1/8 inch diameter smooth specimen of TZM has been fatigue tested to failure, the major effort during this report period on the unnotched specimen geometry has been devoted to obtaining resonant conditions in the load train which would provide sufficient drive to crack (fatigue) 1/4 inch diameter specimens at temperatures in the 1800 to 2200°F (982 to 1204°C) range. Notch tensile specimens were tested and S-N curves were obtained for TZM and TZC material. The specimen geometry, shown in Figure 4, consisted of 1/4 inch major diameter, a 0.170 inch minor diameter, and a parallel-sided notch with a root radius of 1/32 inch. This geometry represented a theoretical stress concentration factor (K_T) of 1.75 (2). The specimens were tested with an as-machined surface finish which produced an RMS finish of $< 15\mu$-inch.

The specimen was mounted on the drive train and a W-3%Re/W-25%Re thermocouple was placed approximately 1/8 inch from the surface at the specimen midpoint. Due to breakage produced by the vibration, the thermocouple could not be attached directly to the specimen. The system was pumped to a vacuum less than 10^{-8} Torr, and then the sample was heated to the test temperature at a rate slow enough so that the vacuum never exceeded 1×10^{-6} Torr. The temperature was stabilized for approximately two hours and then the cyclic load was applied. The initial tests were conducted with a very slight static tensile load (7.4 Kg) consisting of the weight of the lower half of the specimen and the bracket for mounting the capacitance pick-up gauge.

As a result of the application of the high frequency cyclic load, heating of the fatigue specimen took place. The degree of heating was dependent upon the power applied to the system. In determining the S-N curve, the ambient test temperature; i.e., the temperature recorded by the thermocouple, was set at a fixed value for each test. At the high stress levels where significant heating of the specimen occurred, the test time was sufficiently short so that a readjustment of the furnace temperature to compensate for the self-heating could not be accomplished. At the low values of applied static stress, the temperature increase was very slight and no adjustment of the furnace temperature was usually necessary. Although the data are presented for constant values of the ambient temperature, the actual specimen temperature is also recorded in cases where the test duration was sufficient to allow time for accurate readings. The temperature increase due to self-heating was obtained by measuring the difference in specimen brightness temperature before and after the application of the cyclic load with an L-N optical pyrometer.

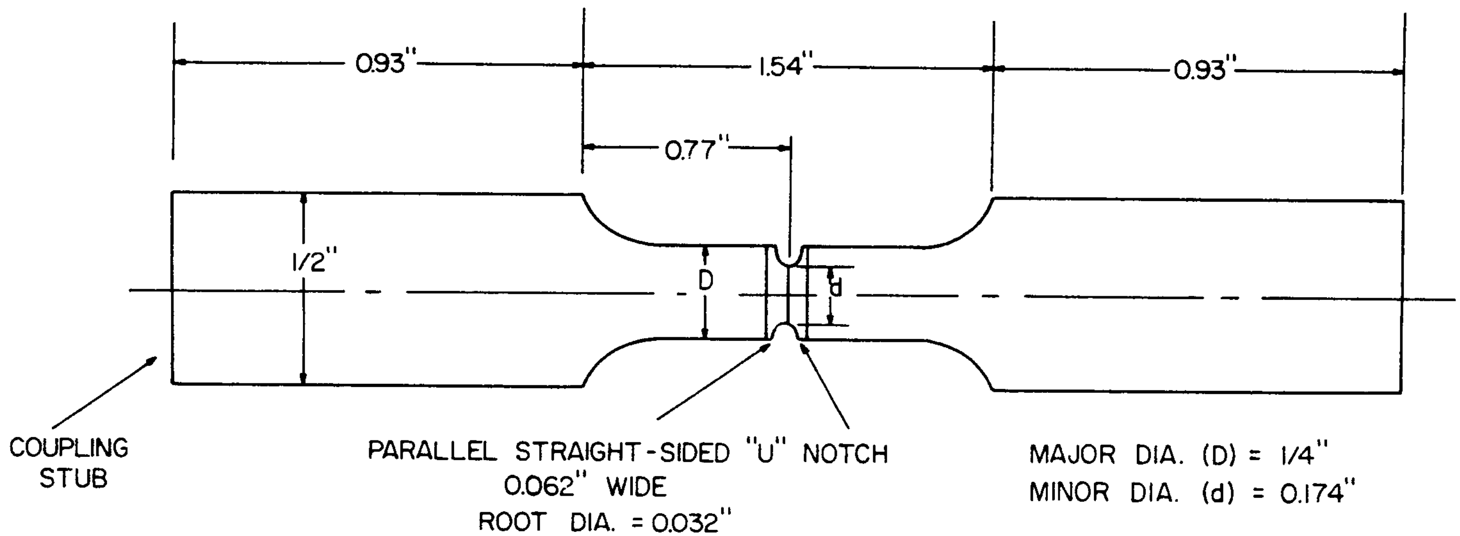


FIG. 4: GEOMETRY OF NOTCH TENSILE SPECIMEN.

The magnitude of the cyclic stress was determined by measuring the displacement of a reference mark on the specimen. Displacement measurements were made at selected positions on the major specimen diameter equidistant from the notch. These displacement values were then used to calculate the strain at the specimen midpoint, assuming that no notch was present. The strain was converted to stress by using the modulus of elasticity at the test temperature. The stress at the base of the notch is higher than that present on the major diameter due to ultrasonic stress amplification produced by the decreased area at the notch root. This amplification factor is equal to the ratio of the area of the major to the minor diameter $(\frac{D}{d})^2$. In addition, an effective stress concentration factor (K_f) based on the notch radius also multiplies the peak stress value (Ref. 3). The method of determining the effective stress concentration factor (K_f) which is less than the theoretical stress concentration factor (K_T) has been described in the previous quarterly report (1). For the notch geometry employed in this program, a K_f value of 1.50 was used.

The accuracy of the stress determinations is not only dependent upon the displacement measurements but is also sensitive to the value of the elastic modulus. Modulus measurements reported in the literature (4,5,6) for both dynamic and static measurements on TZM and unalloyed molybdenum are plotted in Figure 5. The results indicate that there is a significant difference between the dynamic and static modulus measurements. For a given test method, however, the values obtained for unalloyed molybdenum and the TZM alloy were comparable. Although dynamic modulus tests are currently being conducted on the alloys under test in this program, the presently-reported stress values shown in Figure 6 were calculated with moduli obtained by extrapolation of the dynamic test data in Figure 5. A value of 37.5×10^6 psi (2.58×10^{11} N/m^2) was used for the TZM at an ambient test temperature of 1800°F (982°C) and a value of 35.0×10^6 psi (2.4×10^{11} N/m^2) for the TZC data at 2000°F (1093°C).

The fatigue tests were conducted in the 18 to 19 Kcs (kHz) frequency range. When cracking in the test specimen occurred, a significant decrease in resonance frequency was apparent. The end point of the test was defined by this variation in the drive characteristic.

IV. RESULTS AND DISCUSSION

1. Notch Tests

Fatigue tests were conducted on notched specimens (K_T = 1.75) of TZM at 1800°F (982°C) and TZC (Heat M-89) at 2000°F (1093°C). The test data are presented in Tables 4 and 5 and summarized in Figure 6. The stress is plotted as the peak value taking into account the calculated intensification due to the presence of the notch. A compression-tension cycle was employed with a constant static load equal to the weight

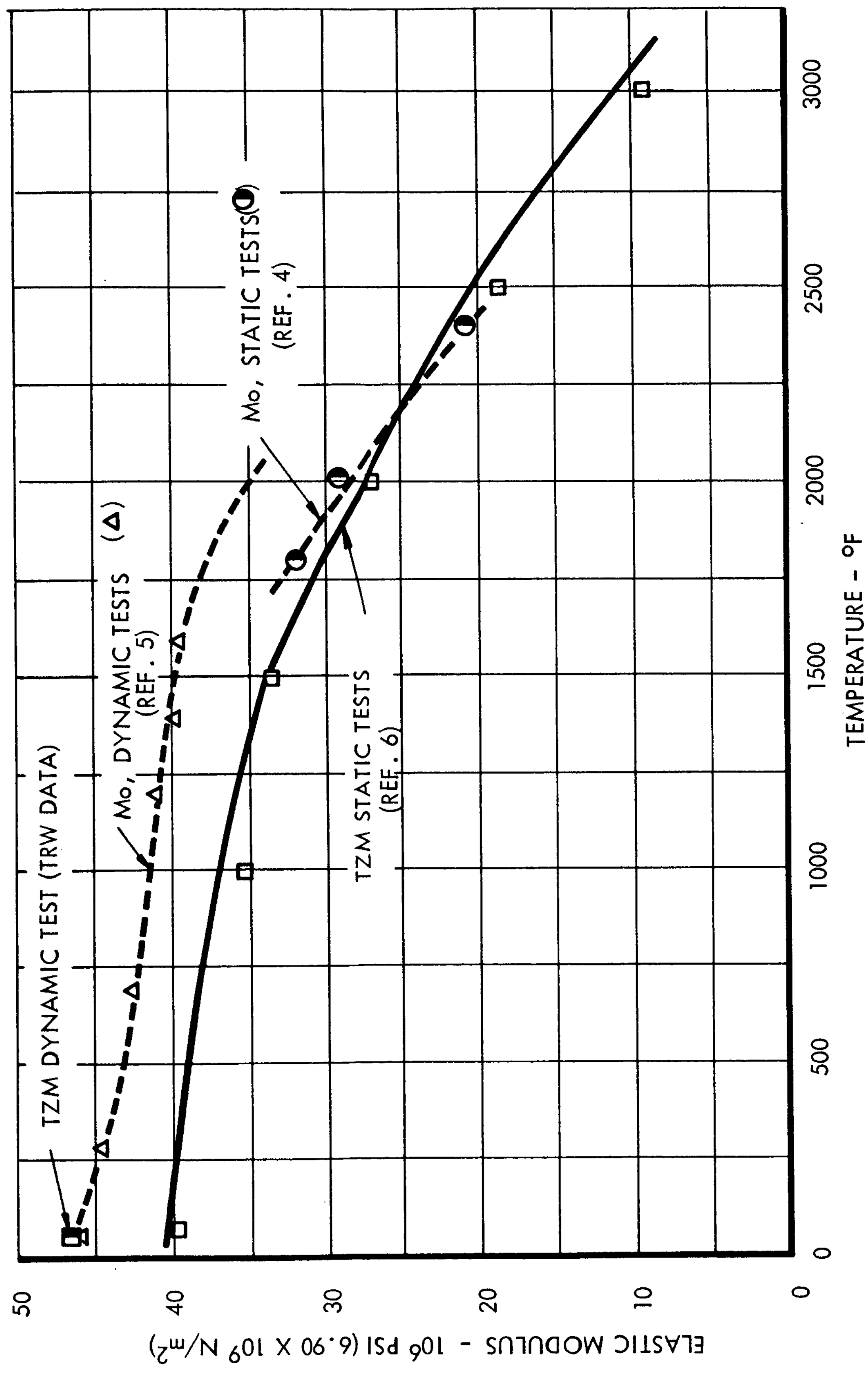


FIGURE 5 VARIATION OF ELASTIC MODULUS WITH TEMPERATURE FOR MOLYBDENUM ALLOYS

TABLE 4

Summary of Data Obtained from Notched Fatigue Tests on TZM Specimens, Stress Relieved Condition
Ambient Test Temperature 1800°F (981°C), Frequency 18.6 Kcs (kHz)

Specimen	(A) Displacement Magnitude (μ-in.)	(B) Displacement Dist. from Center (in)	(C) Strain (in/in)	(D) Stress Based on Smooth Specimen (Ksi, $6.89 \times 10^6 N/m^2$)	(E) Peak Dynamic Stress (Ksi)	(F) Static Notch Stress (Ksi)	(G) Total Peak Stress (Ksi)	(H) Cycles to failure (KHz)	(I) Time to failure (hours)
9AA	55	0.350	157	5.90	18.7	1.02	19.7	8.20×10^6	0.13
10	54	0.355	152	5.70	18.1	1.02	19.1	6.22×10^8	9.3
11	56	0.399	141	5.30	16.8	1.02	17.8	1.61×10^8	2.4
12	43	0.378	114	4.28	13.6	1.02	14.6	1.61×10^{10}	239

Column C: calculated from displacement measurements on major diameter, assuming no notch

Column D: calculated by multiplying strain by elastic modulus, 37.5×10^6 psi

Column E: column D multiplied by K_f=1.50 and ratio of major-to-minor diameter squared $(D/d)^2 = 2.11$

Column F: static average stress (drive train below specimen) multiplied by effective stress concentration factor (K_f=1.50)

Column G: summation of columns E and F.

TABLE 5

Summary of Data Obtained from Notched Fatigue Tests on TZC Specimens, Recrystallized 3092°F (1700°C)
Ambient Test Temperature 2000°F (1093°C), Frequency 19.0 Kcs (kHz)

Specimen	(A) Displacement Magnitude (μ-in)	(B) Displacement Dist. from Center (in)	(C) Strain (in/in)	(D) Stress Based on Smooth Specimen (Ksi, 6.89 x $10^6 N/m^2$)	(E) Peak Dynamic Stress (Ksi)	(F) Static Notch Stress (Ksi)	(G) Total Peak Stress (Ksi)	(H) Cycles to failure (KHz)	(I) Time to failure (hours)	(J) Temperature Increase Due to Drive (°F)	(J) (°C)
2	> 237	0.590	>401	>14.0	> 44.4	1.02	>45.4	8.9 x 10^6	0.08	*	
3	203	0.557	365	12.8	40.6	1.02	41.6	2.06 x 10^7	0.20	*	
4	151	0.511	296	10.4	32.8	1.02	33.8	2.17 x 10^8	3.10	27	15
7	111	0.573	196	6.86	21.7	1.02	22.7	3.77 x 10^8	5.5	*	
9	53	0.551	96.0	3.36	10.6	1.02	11.6	1.81 x 10^9	26.5	*	
12	50	0.523	95.5	3.34	10.5	1.02	11.5	3.01 x 10^9	44.0	28	15
11	42	0.582	72.1	2.53	8.01	1.02	9.03	9.75 x 10^9	143.0	0	0

Column C: calculated from displacement measurements on major diameter, assuming no notch

Column D: calculated by multiplying strain by elastic modulus, 35 x 10^6 psi

Column E: column D multiplied by K_f = 1.50 and ratio of major-to-minor diameter squared $(D/d)^2$ = 2.11

Column F: static average stress (drive train below specimen) multiplied by effective stress concentration factor (K_f = 1.50)

Column G: summation of columns E and F.

* No temperature measurements obtained.

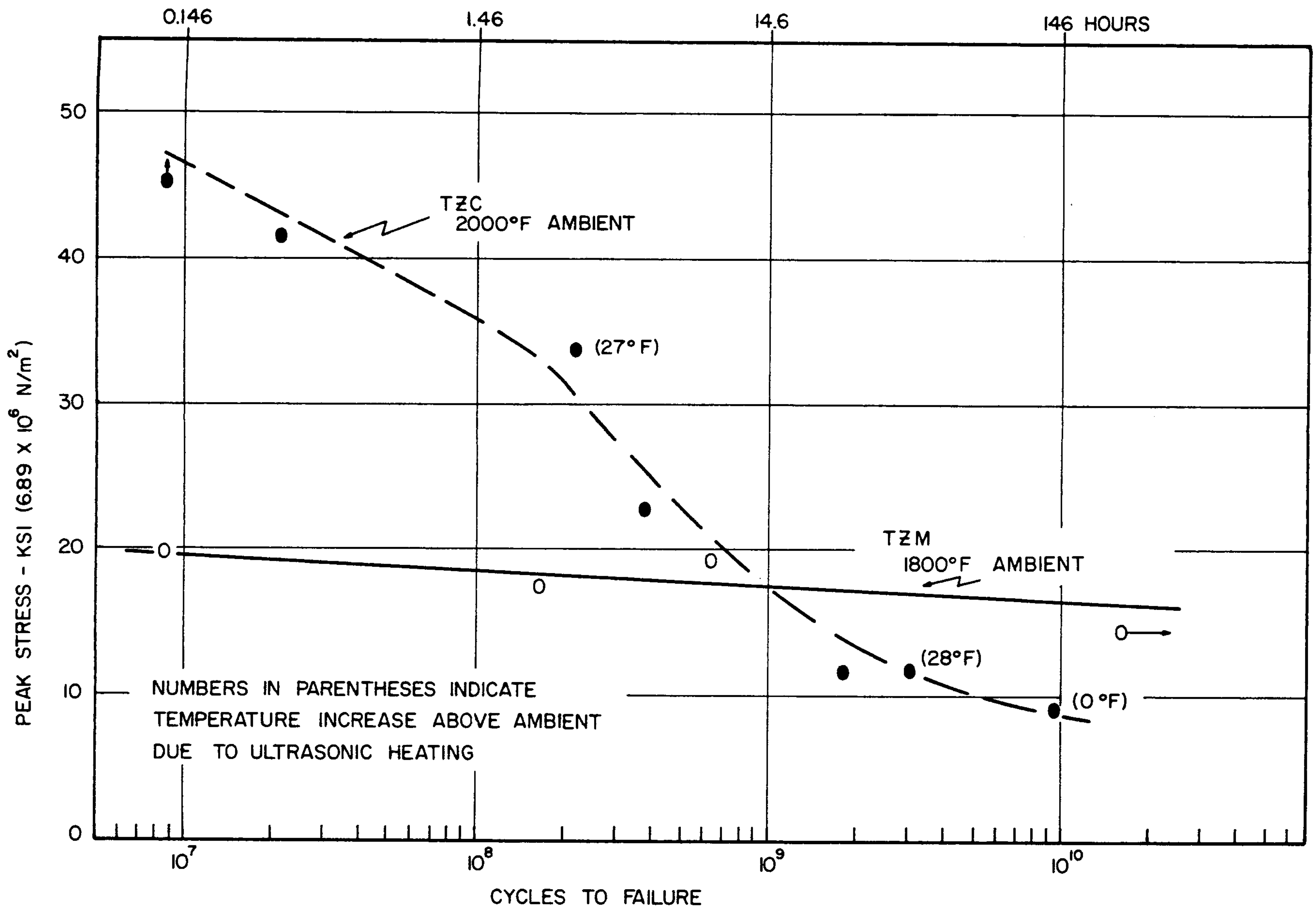


FIG. 6: FATIGUE CURVES FOR TZM (RECRYSTALLIZED AT 3092°F) AND TZM (STRESS-RELIEVED CONDITION) TESTED IN VACUUM ENVIRONMENT $<10^{-8}$ TORR.

of the lower half of the specimen and the fixture for holding the capacitance pick-up (see Column F in Tables 4 and 5).

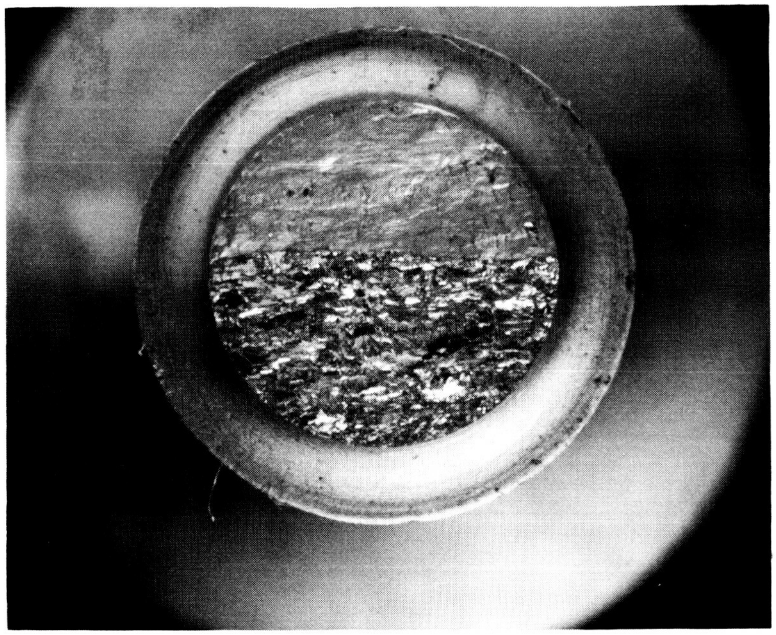
The TZM exhibited a fatigue curve that was extremely sensitive to stress level in the 10^7 to 10^{10} cycle range. By comparison the fatigue strength of the TZC decreased from a calculated stress of 45 ksi ($3.1 \times 10^8 N/m^2$) to less than 10 ksi ($6.89 \times 10^7 N/m^2$) over approximately the same range of test cycles. Neither material showed a true endurance limit at the selected test temperatures.

The appearance of representative fracture surfaces are shown for both the TZC and TZM in Figures 7 and 8. The fracture appearance clearly indicates the region of fatigue crack growth which normally covers approximately one-half of the specimen cross-section. The appearance of "beach marks" is also more apparent in the TZM specimens which were tested in the stress-relieved condition.

The rather steep slope of the S-N curve for the TZC material (see Figure 6) is somewhat unexpected since conventional results generally indicate a very stress sensitive relationship at high cycles similar to that present for TZM. The appearance of the TZC surfaces (see Figure 9) indicates that appreciable surface rippling has occurred at the base of the notch. This effect indicates that localized flow has occurred and suggests that the flow may result in an effective stress concentration factor in the high stress range which is less than that employed to calculate the peak stress values.

2. Smooth Specimen Tests

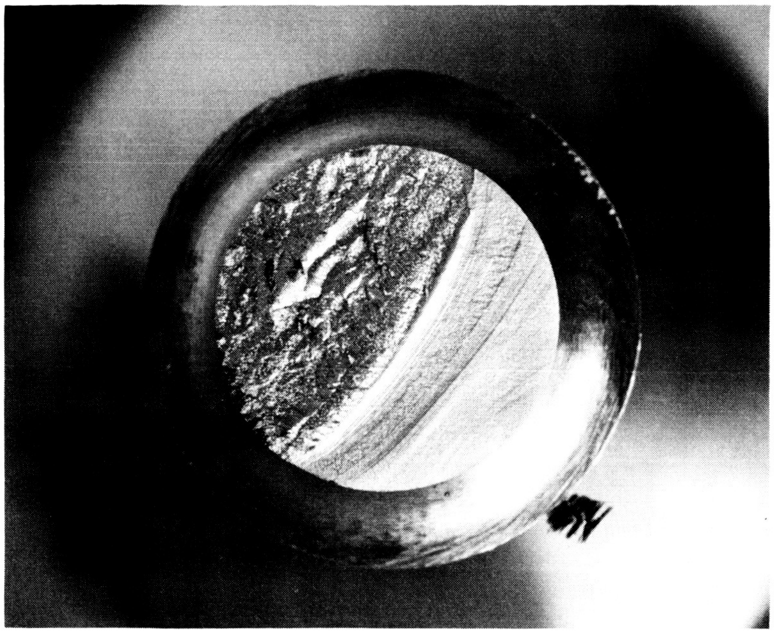
Thus far, the ultrasonic drive system has not produced sufficient cyclic stress to cause fatigue fracture in either a TZC or TZM smooth specimen at test temperatures in the 1800 to 2000°F (981 to 1093°C) range. During this report period, a tension-compression test was conducted on 1/4 inch diameter smooth specimen of TZC (Heat M-89, annealed 3200°F, 1 hour) at a peak stress of 15,000 psi ($1.03 \times 10^8 N/m^2$) for 69 hours (4.8×10^9 cycles) at 2000°F (1093°C) without producing any indication of fracture. At present, no explanation is apparent as to why the notch specimens fractured at a calculated peak stress as low as 9.03 ksi ($6.21 \times 10^7 N/m^2$), while the smooth specimens did not fail at a considerably higher stress. Results obtained by several investigators (7,8) using notch specimens on a variety of alloys indicate reasonably good agreement between S-N curves based on notch specimens under ultrasonic conditions and conventional fatigue test results on smooth specimens. The explanation for the difference does not appear to be in the method of calculating peak stress in the notched specimens since any error in the application of either the ultrasonic amplification factor or the stress concentration factor should tend to decrease the calculated stress value.



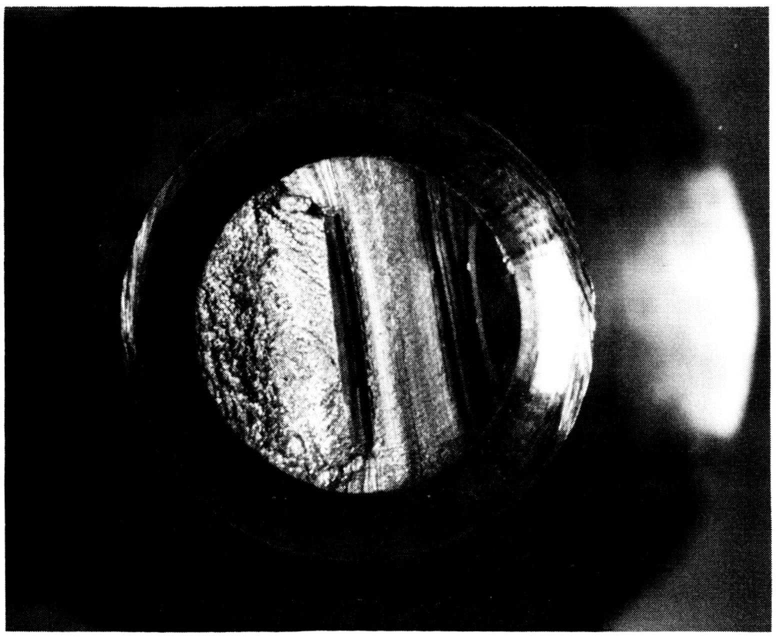
Peak Stress 22.7 Ksi 10X
3.77×10^8 Cycles

Peak Stress 11.6 Ksi 10X
1.81×10^9 Cycles

Figure 7. Fracture Appearance of Notch Fatigue Specimens, TZC (Heat M-89), Annealed 3092°F (1700°C), 1 Hour, Tested at 19.0 Kcs (KHz), 2000°F (1093°C), Vacuum Environment 10^{-8} Torr.

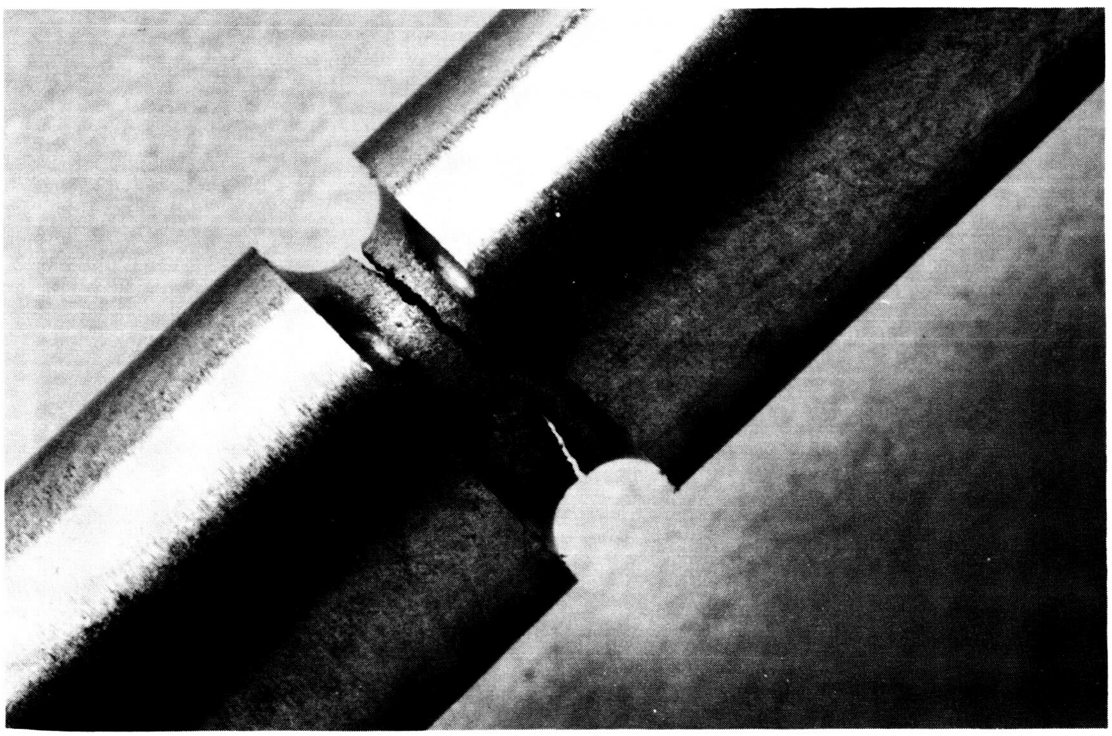


Peak Stress 19.1 Ksi
6.22 x 10^8 Cycles



Peak Stress 17.8 Ksi
1.61 x 10^8 Cycles

Figure 8. Fracture Appearance of Notch Fatigue Specimens, TZM, Stress-Relieved Condition, Tested at 18.6 Kcs (KHz), 1800°F (982°C), Vacuum Environment 10^{-8} Torr.



Peak Stress 45.4 Ksi 10X
8.9×10^6 Cycles

Figure 9. Appearance of Specimen Surface in Notch Area Showing Localized Deformation at Notch Root. TZC Annealed 3200°F (1700°C), 1 Hour, Tested at 2000°F (1093°C) Vacuum Environment 10^{-8} Torr.

The attempts to produce increased displacement in the ultrasonic drive system which would enable testing of smooth specimens have been devoted to the following three areas:

(1) Modification of the flange which provides the seal between the piezoelectric drive system and the vacuum chamber.

(2) Improvement of the horn design to allow greater deflections to be obtained, and

(3) Improved tuning of the horn-specimen system at the elevated test temperature.

The use of a thin Viton seal substituted for the weld at the flange, along with the application of hollow horns to the drive train, has increased the displacement in the system by a factor of approximately three. Fracture has been obtained in 1/8 inch diameter smooth specimens of TZM at ambient room temperature. When comparable specimens were heated to 2000°F (1093°C), however, significant loss in displacement occurred as a result of detuning and fatigue failure could not be produced. At present, specimen design is being optimized to provide a resonant system under the test conditions which involve a uniformly-heated specimen and a temperature gradient along the horns which pass through the furnace section.

3. Comparative Creep and Fatigue Properties

Although questions still exist concerning the significance of notch fatigue tests from a design standpoint, it is informative to compare the relative susceptibilities of the test materials for creep and fatigue failure on the basis of the existing results. Figure 10 presents a Larson-Miller plot of 0.5 percent creep data for TZC (1). Superimposed on the creep curve is the fatigue data presented in Figure 6 using the failure times obtained with the loading frequency of 19.0 Kcs (kHz).

Although the fatigue results when presented on a time scale will vary depending on the frequency of load application, the preliminary results, particularly for TZC, indicate that fatigue may be a limiting factor in components operating in a high vacuum environment at high frequencies of load application.

V. FUTURE WORK

Emphasis will be placed on optimizing the design of the loading system so that fatigue failure can be produced in 1/4" diameter test specimens. Additional tests will be performed on notch specimens at varying ratios of dynamic to static load.

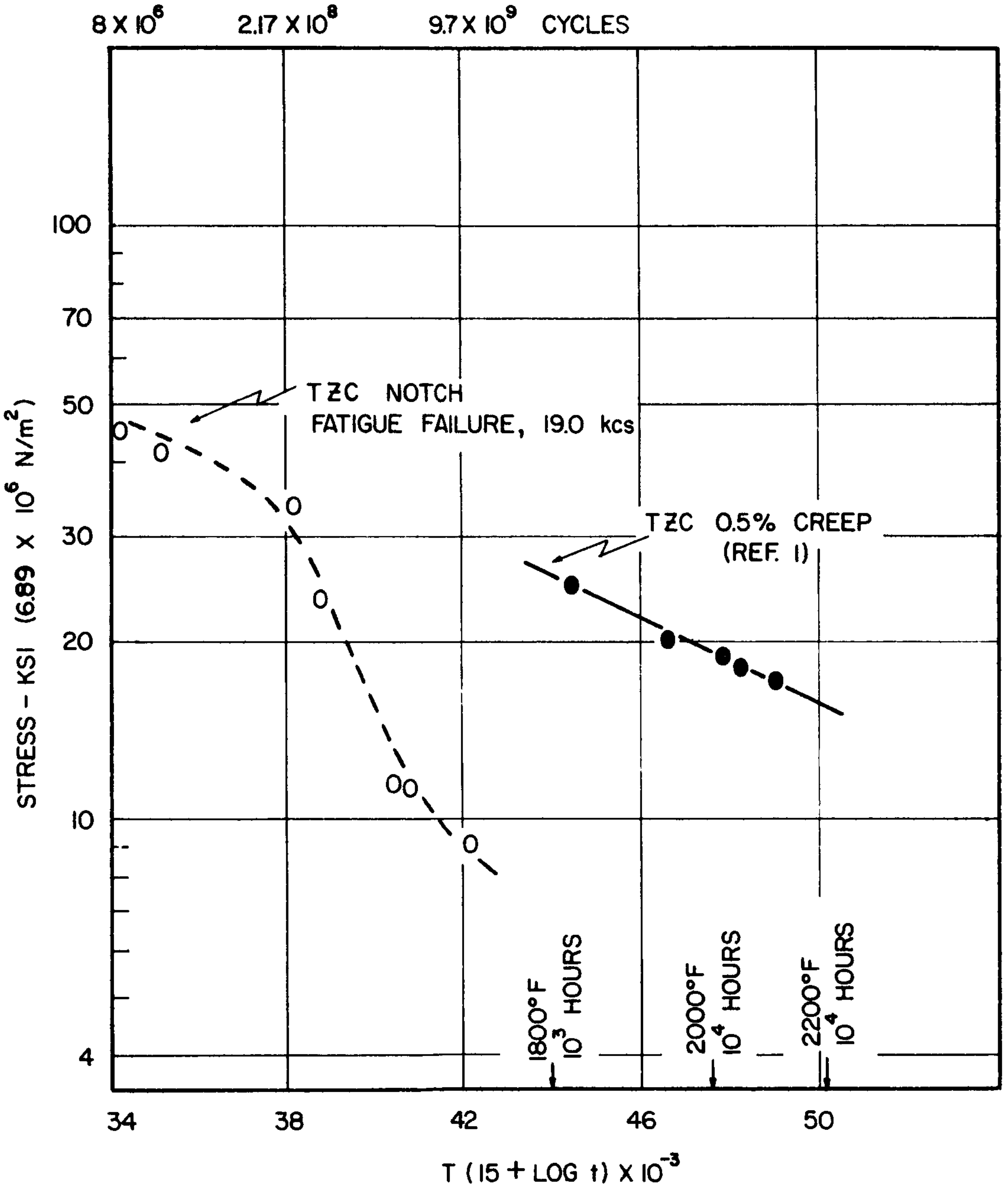


FIG. 10: COMPARISON OF FATIGUE AND CREEP PROPERTIES OF TZC ANNEALED AT 3092°F (1760°C) TESTED IN HIGH VACUUM ENVIRONMENT.

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