STRESS RELAXATION OF ZIRCALOY-4

by

ARTHUR PHILLIP MINAR

Submitted in Partial Fulfillment

of the Requirements for the

Degree of Bachelor of Science

at the

MASSACHUSETTS INSTITUTE OF TECHNOLOGY

June, 1976

Signature of Author. . . . . .

Department of Materials Science and Engineering

May 7, 1976

Certified by. . . . . .

Thesis Supervisor

Accepted by. . . . . .

Chairperson, Departmental Committee on Theses

A B S T R A C T

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Submitted to the Department of Materials Science and Engineering on May 7, 1976, in partial fulfillment of the requirements for the degree of Bachelor of Science.

The purpose of this investigation is to determine whether a mechanical equation of state exists for zircaloy-4 in the temperature regions of interest. For this purpose, stress relaxation experiments have been performed on samples of zircaloy-4 rod at temperatures of 22°C., 200°C., and 250°C. The results of the experiments were plotted on a graph of log stress versus log strain rate to determine whether the samples exhibited unique states of hardness which is characteristic of the mechanical equation of state.

The graphs indicated a tendency for the stress-strain rate data to follow parallel curves as would be expected for unique states of hardness but they also exhibited discontinuous behavior at a certain time into the relaxation run.

The conclusion reached is that a mechanical equation of state may exist for zircaloy-4 but discontinuous flow mechanisms make its existence uncertain.

Thesis Supervisor: Arden L. Bement
Title: Professor of Nuclear Materials

A C K N O W L E D G E M E N T S

I would like to thank Gene Lucas and Ron Ballinger for their helpful advice and a special thanks to Ron Ballinger for the loan of his equipment. I would also like to thank Mr. Bob Huston for his assistance in specimen preparation.

I wish to express my deep appreciation for the help and guidance in the preparation and writing of this thesis to Prof. Arden L. Bement, Jr., my thesis supervisor.

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I. INTRODUCTION

This investigation involves running load relaxation tests on specimens of zircaloy-4 at three temperatures in an attempt to determine whether a mechanical equation of state applies to zircaloy-4 at the test temperatures.

The most familiar example of an equation of state is the Ideal Gas Law, PV=nRT. This equation is a statement that, within appropriate temperature ranges for most gases, fixing any two of the three properties, pressure, volume, or temperature, for a given quantity of gas will automatically determine the third property without regard to the previous history of the gas.

Analogous to the Ideal Gas Law, a mechanical equation of state may exist for some metals. This equation would be a relationship whereby the plastic deformation behavior of metals can be predicted by summarizing prior deformation history from the readily measurable deformation parameters, stress, strain and strain rate. The mechanical equation of state can be useful in determining mechanical properties of a metal because if it applies, data obtained from one type of experiment can be used to predict the mechanical behavior of the metal in a different type of test.

In recent years, Hart[1] has introduced the concept of hardness or strength being a state property of a material. Hart proposed that at any point in the deformation history of a material, the stress and strain rate uniquely characterized the hardness of the material for isothermal conditions and in the

absence of metallurgical reactions.

Testing a material to determine if it satisfies a mechanical equation of state requires an experimental technique which allows plastic deformation to occur at a constant structure or hardness over a broad range of strain rates. Two of the methods previously employed to acquire stress-strain rate data, sudden load variations during creep tests and monitoring strain rate during constant extension rate tests, were found by Hart[2] to be dominated by the anelastic effects inherent in the sudden loading or unloading of an elastic material. Hart chose the load relaxation test as the optimum method for extractiong stress-strain rate information at constant hardness because the specimen is subjected to a gradual reduction in load at strain rates which allow continuous accomodation of anelastic effects.

Several load relaxation experiments have been run by various people to test the validity of an equation fo state for certain materials over specific temperature ranges. Among them have been Hart and Solomon's[3] work with aluminum, Yamada and Li's[4,5,6] work with nickel, TD nickel, niobium, iron and austenitic stainless steel, Wire, Yamada and Li's[7] work with lead and Lee and Hart's[8] tests on magnesium-aluminum alloy and pure zirconium. For each material, the authors concluded that a mechanical equation of state exists over the temperature ranges of interest.

II. THE LOAD RELAXATION TEST AND MECHANICAL EQUATION OF STATE

The load relaxation test is run by pulling a test specimen in a tensile testing machine at a constant extension rate to a

particular load level and then stopping the cross-head motion and recording the load as a function of time at that fixed cross-head position. The specimen continues to strain plastically under the action of the residual load exerted by the load train. As the specimen extends plastically, the applied load relaxes elastically.

The time rate of change of the applied load is a direct measurement of the plastic extension rate of the specimen. Since load is measured as a function of time, the load can be differentiated with respect to time to generate a relaxation history of non-elastic strain rate as a function of stress. Because there is only a small amount of plastic strain accomplished during the stress relaxation sequence, there is essentially no strain hardening of the specimen. Therefore, the stress-strain rate curve is characteristic of a single state of hardness.

If the imposed strain rate is known, the corresponding stress to produce that strain rate is a measure of the current mechanical strength of the specimen. The set of all possible pairs of values of stress and strain rate determine the mechanical state of the specimen at any instant. If the stress is solely a function of strain rate and temperature for a specific material and each state of plastic hardness of the material as a result of deformation is unique and is independent of the deformation path by which it was reached, then a mechanical equation of state exists for the material.

To determine if a material exhibits a mechanical equation

of state, it is necessary that, if two samples that have undergone different stress-strain rate history exhibit the datum point, s_1, $\dot{e}_1$, then both must, when the stress is changed to s_2, strain at the same rate $\dot{e}_2$.[2] In other words, it must not be possible for two constant hardness curves to intersect so that all curves for a given material must be parallel.

III. MODELING THE LOAD RELAXATION TEST

The loading of a plastic specimen in a tensile testing machine can be modeled in the following manner.[9] The specimen is considered to be purely plastic, loaded by a spring which in turn is extended by a movable crosshead. The spring represents the combined elasticity of the specimen, the load-measuring cell and the connecting linkages. The length of the specimen is given by L. The load on the specimen and spring as determined by the load cell is P. The crosshead displacement is L_c and at time t=0, L_c equals L_o, the original specimen length. K is the combined elastic constant of the specimen and machine as represented by the spring.

The load is the elastic extension multiplied by the elastic constant so that $P=K(L_c-L)$. Stress is defined as the load divided by the cross-sectional area, $s=P/A$. If the load is differentiated with respect to time and $\frac{d}{dt}$ is represented by a dot over the differentiated term, then $\dot{P}=K(\dot{L}_c-\dot{L})=A\dot{s}+s\dot{A}$. Isolating the stress rate results in: $\dot{s}=KL/A\ ((\dot{L}_c/L) - (\dot{L}/L)) - s\dot{A}/A$. Assuming constant specimen volume results in the equation $L_oA_o= LA$ and taking the derivative with respect to time gives $\dot{A}/A = -\dot{L}/L$. When substituted into the equation for stress

rate, the resulting equation is $\dot{s}=KL/A\ (L_c/L - (1-sA/KL)\dot{L}/L)$. From $L_oA_o = LA$, we can replace L/A with $(L_o/A_o)(L/L_o)^2$. For small crosshead displacement, P is much less than either KL or KL_c. Since $L=L_c-P/K = L_c(1-P/KL_c)$ and P/KL_c is much less than 1, then L_c is approximately equal to L. Also, P/KL and, subsequently, sA/KL are much less than 1 so that $\dot{s}=(KL_o/A_o)(L_c/L_o)^2\ ((\dot{L}_c/L) - (\dot{L}/L))$. Elastic strain rate($\dot{e}_e$) is defined as $\dot{L}_c/L_c$ and plastic strain rate ($\dot{e}_p$) is defined as $\dot{L}/L$. If the crosshead displacement is much less than the original value of L_c, then $\dot{s}=KL_o/A_o(\dot{e}_e - \dot{e}_p)$. Since the crosshead is held in a fixed position during stress relaxation and $\dot{L}_c=0$, the final equation relating stress rate and strain rate is $\dot{s}= -(KL_o/A_o)\ \dot{e}_p$.

IV. EXPERIMENTAL EQUIPMENT

All of the tests were performed in tension with a floor model Instron tensile testing machine. Room temperature (22°C. or 72°F.) tests were done using a D type load cell, and a GR load cell was used for the elevated temperature tests. The load-versus-time data was obtained from a standard Instron recorder which used the amplified load cell output signal to determine the tensile load on the sample and a constant speed chart to determine time.

A 14in.X16½in.X7½in. oven was used to maintain a constant temperature for the 250°C.(482°F.) and 200°C.(392°F.) tests. An argon atmosphere was maintained in the oven to prevent oxidation. A Leeds and Northrup controller and power supply regulated the oven temperature. The oven temperature was monitered with a thermocouple placed next to the sample.

The thermocouple was referenced to $0^{o}C$. by an ice bath, and its potential was read from a digital voltmeter.

V. EXPERIMENTAL PROCEDURE

To run a stress relaxation experiment a sample is first chosen and threaded into the grips which are attached to the pull rods on the tensile testing frame. For elevated temperature tests, the oven encloses the sample, and grips with the pull rods extending through the oven. The temperature of the sample is allowed to equilibrate at the desired test temperature. The sample is then loaded to approximately five pounds to take the slack out of the load train. It is then loaded at a constant extension rate of .02 inch per minute to a stress immediately above the elastic limit. The crosshead is stopped after the load-time record has begun to deviate from linearity, and the load immediately begins to decay at the fixed extension. For the first fifteen to twenty minutes of the experiment, the chart speed is set at 1 inch per minute and thereafter it is changed to .2 inch per minute. The load is allowed to decay until thermal fluctuations make the data unreliable. The specimen is then unloaded and the loading and relaxation sequence can be repeated for another experiment.

VI. REDUCTION OF DATA

The record of load as a function of time was reduced by computer to an equation of the form $P= B_1\exp(-B_2t) + B_3\exp(-B_4t) + B_5$ by use of a non-linear regression program utilizing the LSMARQ subroutine. The stress was computed using the formula, $s=P/A$. From the relationship between stress rate and strain rate

which was derived previously and the definition of stress, the resulting equation is $\dot{e}_p = -\frac{\dot{P}}{KL_o}$. $\dot{P}$ was obtained by differentiating the equation which was fit to the load-time record by the computer. $\dot{P}$ is in units of pounds per minute. K was found by taking the slope of the loading part of the load-time record. K is in units of pounds per inch.

VII. MATERIAL COMPOSITION AND SPECIMEN DIMENSIONS

All of the experiments were performed on zircaloy-4 obtained from Teledyne Wah Chang of Albany, Oregon. The chemical analysis of the material as obtained from the supplier is listed in Table 1. The material was received in the form of 0.25 inch (6.35 mm) diameter annealed rods. The specimens (see Figure 1) were made by cutting a three-inch section from the rod and turning down a center section gauge length of 0.640 inch (16.3 mm) to a 0.160-inch (4.06 mm) diameter. Three-quarters of an inch of both ends of the specimen were threaded with $\frac{1}{4}$-inch 20 threads to fit the grips on the tensile testing machine.

VIII. RESULTS OF EXPERIMENTS

The data from the stress relaxation experiments are tabulated in tables 2 through 7 and shown graphically in figures 2 through 5. As illustrated in the graphs, two anomalous shifts, or changes in slope, occur in the stress-strain rate data. The first shift occurs after about five minutes of relaxation and the second shift usually occurs after approximately twenty-five minutes. The changes in slope are most prominent at lower stresses during the first and possibly second relaxation run for a new sample.

The first change of slope is a result of using a sum of two exponentials to model the load decay. The first exponential models the anelastic effect and it has decayed away after the first five minutes. The second exponential is left to dominate the long term relaxation.

The second change in slope cannot be explained as easily. It is not likely to be a result of experimental procedure because nothing was done to the sample after the crosshead had been fixed. There is a possibility that the second shift in the stress-strain rate curve is the result of plotting an exponential function on a log-log graph. However, this would not explain why the curves approach linearity after a few experiments have been run on a sample.

Two possible micro-structural explanations for the changing slope of the stress-strain rate curve might be mixed mode deformation or thermally activated flow mechanisms. The mixed mode deformation could consist of a combination of twinning and

slip operating in parallel with one mechanism dominating the relaxation initially and the other taking over after a period of time.

The two thermally activated processes that may be responsible for the second discontinuity are dislocation climb and cross-slip. The relatively large concentration of oxygen in zircaloy-4 results in the presence of coupled pairs of oxygen atoms placed interstitially in the hexagonal lattice.[10.] Dislocation loops form around these paired atoms which eventually block the passage of further dislocations. The edge components of the dislocation loops can climb over the oxygen atoms which results in mutual annihilation and the screw components can continue the breakdown of the dislocation loop by slip.[11.] Therefore, the amount of slip is determined by the thermally activated dislocation climb. The competing mechanism with dislocation climb is the interchange of slip planes by screw dislocations known as cross-slip. These mechanisms have different activation energies and it might be possible for each mechanism to control deformation over different ranges of stress and strain rate.

IX. CONCLUSIONS

1. Two discontinuities appear in the graph for each relaxation run on zircaloy-4.

   a.) The discontinuities become less pronounced with increasing strain and stress.

   b.) The first discontinuity can be explained by the contribution of anelasticity to the relaxation curve.

c.) The cause for the second discontinuity is not known but is suspected to result from competing thermally activated mechanisms.

2. The stress-strain rate data exhibited some characteristics of a mechanical equation of state after repeated loadings at high initial stresses but the anomalous shape of the curve indicates that further work must be done to finally determine if a mechanical equation of state applies to the plastic deformation of zircaloy-4.

X. SUGGESTIONS FOR FURTHER WORK

There are three major areas where inaccuracies were introduced into the data. The first is the lack of proper temperature control for the specimen and load train. At 200°C., thermal fluctuations caused the load-time recaord to oscillate after approximately two hours of testing. At 250°C., thermal fluctuations made the data unreliable after only one hour of relaxation. Thermal oscillations became the limiting factor for low strain rate sensitivity.

The second area where errors were introduced was in the data acquisition system. Use of the standard analogue recorder necessarily limited the accuracy of the data for the high end of strain rate sensitivity. If high speed digital data recording equipment had been availble, this problem could have been reduced[9].

The third area where major inaccuracies were introduced was in fitting an equation to the load-time curve. High speed digital data acquisition would have been helpful here, also, by allowing numerical calculation of the change of slope at various discrete

points along the relaxation curve instead of fitting the entire curve to one equation. If these problems are rectified, further experiments need to be run to determine whether the data derived from the experiments outlined in this report correspond to actual physical phenomenon. If these results are physically justified, then an investigation into the mechanisms of the discontinuity would be warranted.

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TABLE 1

CHEMICAL ANALYSIS OF ZIRCALOY-4

supplied by Teledyne Wah Chang, Albany, Oregon

Impurities measured by weight in parts per million unless otherwise specified.

Iron (Fe)	2150
Chromium (Cr)	1140
Tin (Sn)	1.43%
Copper (Cu)	17
Nitrogen (N)	33
Carbon (C)	120
Oxygen (O)	1090
Hydrogen (H)	less than 6
Hafnium (Hf)	50
Nickel (Ni)	less than 35
Aluminum (Al)	44
Boron (B)	0.2
Cadmium (Cd)	less than 0.2
Cobalt (Co)	less than 10
Manganese (Mn)	less than 25
Silicon (Si)	92
Titanium (Ti)	23
Tungsten (W)	less than 25
Lead (Pb)	less than 50

TABLE 2

RESULTS OF TESTS AT 250°C.

time (min.)	load (lbs.)	stress (psi)X 10^4	strain rate (per min.)
0	399	2.00	
1	335	1.68	$2.00X10^{-5}$
2	324	1.62	$6.03X10^{-6}$
5	307	1.54	$2.73X10^{-6}$
10	292	1.46	$2.07X10^{-6}$
15	281	1.41	$1.58X10^{-6}$
25	265	1.33	$9.24X10^{-7}$
35	260	1.30	$5.39X10^{-7}$
50	249	1.25	$2.40X10^{-7}$
65	245	1.23	$1.07X10^{-7}$

$K=2.00X10^6$

time (min.)	load (lbs.)	stress (psi)X 10^4	strain rate (per min.)
0	500	2.50	
1	429	2.15	$1.59X10^{-5}$
2	420	2.10	$3.36X10^{-6}$
5	408	2.04	$1.12X10^{-6}$
10	403	2.02	$8.40X10^{-7}$
15	398	1.99	$6.40X10^{-7}$
20	395	1.98	$4.88X10^{-7}$
25	388	1.94	$3.72X10^{-7}$
35	385	1.93	$2.16X10^{-7}$
50	382	1.91	$9.56X10^{-8}$

$K=2.42X10^6$

TABLE 3

RESULTS OF TESTS ON FIRST SAMPLE AT 200°C.

First Run

time (min.)	load (lbs.)	stress (psi)X10^4	strain rate (per min.)
0	650	3.25	
1	551	2.76	2.75×10^{-5}
2	533	2.67	8.35×10^{-6}
5	511	2.56	1.90×10^{-6}
10	492	2.46	1.61×10^{-6}
15	479	2.40	1.43×10^{-6}
20	469	2.35	1.28×10^{-6}
39	440	2.20	8.37×10^{-7}
69	407	2.04	4.26×10^{-7}
99	397	1.99	2.16×10^{-7}
144	378	1.89	7.85×10^{-8}

$K = 2.63 \times 10^6$

Second Run

time (min.)	load (lbs.)	stress (psi)X10^4	strain rate (per min.)
0	700	3.51	
1	610	3.05	2.27×10^{-5}
2	593	2.97	6.87×10^{-6}
5	572	2.86	2.30×10^{-6}
10	551	2.76	1.91×10^{-6}
15	536	2.68	1.62×10^{-6}
25	512	2.56	1.15×10^{-6}
50	483	2.42	5.06×10^{-7}
100	459	2.30	9.61×10^{-8}
150	450	2.25	1.83×10^{-8}

$K = 2.75 \times 10^6$ lb./in.

TABLE 4

RESULTS OF TESTS ON FIRST SAMPLE AT 200°C.

Third Run

time (min.)	load (lbs.)	stress (psi)X10^4	strain rate (per minute)
0	750	3.75	
1	648	3.24	2.74×10^{-5}
5	614	3.07	9.26×10^{-7}
2	633	3.17	7.78×10^{-6}
10	601	3.01	7.20×10^{-7}
15	597	2.99	6.46×10^{-7}
25	589	2.93	5.19×10^{-7}
50	575	2.88	3.01×10^{-7}
100	561	2.81	1.01×10^{-7}
150	548	2.74	3.41×10^{-8}

$K = 2.69 \times 10^6$

Fourth Run

time (min.)	load (lbs.)	stress (psi)X10^4	strain rate (per min.)
0	800	4.00	
1	695	3.48	2.28×10^{-5}
2	680	3.40	5.75×10^{-6}
5	662	3.31	8.87×10^{-7}
10	652	3.26	7.54×10^{-7}
15	646	3.23	6.83×10^{-7}
25	633	3.17	5.61×10^{-7}
50	616	3.08	3.42×10^{-7}
100	594	2.97	1.27×10^{-7}
150	582	2.91	4.74×10^{-8}

$K = 3.08 \times 10^6$

TABLE 5

RESULTS OF TESTS ON FIRST SAMPLE AT 200°C.

Fifth Run

time (min.)	load (lbs.)	stress (psi)X10^4	strain rate (per min.)
0	830	4.15	
1	723	3.62	2.27×10^{-5}
2	709	3.55	5.37×10^{-6}
5	692	3.46	8.78×10^{-7}
10	683	3.42	7.10×10^{-7}
15	676	3.38	6.00×10^{-7}
25	665	3.33	4.28×10^{-7}
50	661	3.31	1.84×10^{-7}
100	653	3.27	3.42×10^{-8}

$K = 3.00 \times 10^6$

RESULTS OF TESTS ON SECOND SAMPLE AT 200°C.

First Run

time (min.)	load (lbs.)	stress (psi)X10^4	strain rate (per min.)
0	553	2.77	
1	463	2.32	2.57×10^{-5}
2	459	2.30	7.47×10^{-6}
5	427	2.14	3.79×10^{-6}
10	403	2.02	3.12×10^{-6}
15	388	1.94	2.58×10^{-6}
20	377	1.89	2.13×10^{-6}
50	335	1.68	6.78×10^{-7}
75	313	1.57	2.61×10^{-7}

$K = 2.00 \times 10^6$ lb./in.

TABLE 6

RESULTS OF TESTS ON SECOND SAMPLE AT 200°C.

Second Run

time (min.)	load (lbs.)	stress (psi)X10^4	strain rate (per min.)
0	650	3.25	
1	556	2.78	2.45×10^{-5}
2	542	2.71	6.53×10^{-6}
5	523	2.62	1.12×10^{-6}
10	513	2.57	8.50×10^{-7}
15	509	2.55	6.83×10^{-7}
25	502	2.51	4.41×10^{-7}
50	493	2.47	1.47×10^{-7}
100	482	2.41	1.65×10^{-8}

$K = 2.63 \times 10^6$ lb./in.

Third Run

time (min.)	load (lbs.)	stress (psi)X10^4	strain rate (per min.)
0	700	3.50	
1	604	3.02	2.11×10^{-5}
2	591	2.96	5.22×10^{-6}
5	572	2.86	1.41×10^{-6}
10	560	2.80	1.02×10^{-6}
15	554	2.77	7.60×10^{-7}
25	548	2.74	4.15×10^{-7}
50	539	2.70	9.19×10^{-8}

$K = 2.81 \times 10^6$

TABLE 7

RESULTS OF ROOM TEMPERATURE TESTS

time (min.)	load (lbs.)	stress (psi)	strain rate ($min.^{-1}$)
0	905	$4.53X10^4$	
1	850	$4.25X10^4$	$1.52X10^{-5}$
5	825	$4.13X10^4$	$1.37X10^{-6}$
10	811	$4.06X10^4$	$4.48X10^{-7}$
20	799	$4.00X10^4$	$3.84X10^{-7}$
50	783	$3.92X10^4$	$2.43X10^{-7}$
100	769	$3.85X10^4$	$1.14X10^{-7}$
150	764	$3.82X10^4$	$5.32X10^{-8}$
200	760	$3.80X10^4$	$2.49X10^{-8}$
250	757	$3.79X10^4$	$1.09X10^{-8}$

$K=3.20X10^6$

time (min.)	load (lbs.)	stress (psi)	strain rate ($min.^{-1}$)
0	1000	$5.00X10^4$	
1	958	$4.79X10^4$	$1.15X10^{-5}$
2	946	$4.73X10^4$	$4.90X10^{-6}$
5	931	$4.66X10^4$	$8.91X10^{-7}$
10	918	$4.59X10^4$	$5.62X10^{-7}$
15	911	$4.56X10^4$	$5.01X10^{-7}$
25	901	$4.51X10^4$	$3.98X10^{-7}$
50	888	$4.44X10^4$	$2.19X10^{-7}$
75	879	$4.40X10^4$	$1.23X10^{-7}$
100	872	$4.36X10^4$	$6.76X10^{-8}$
150	865	$4.33X10^4$	$2.10X10^{-8}$

$K=3.54X10^6$

FIGURE 1

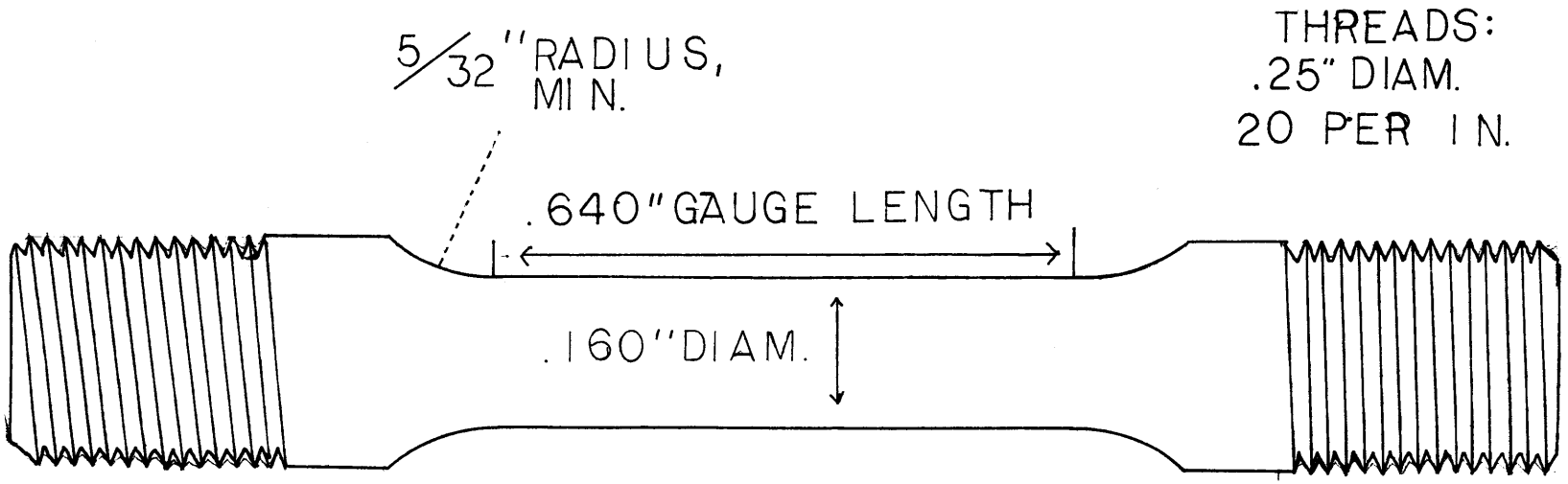
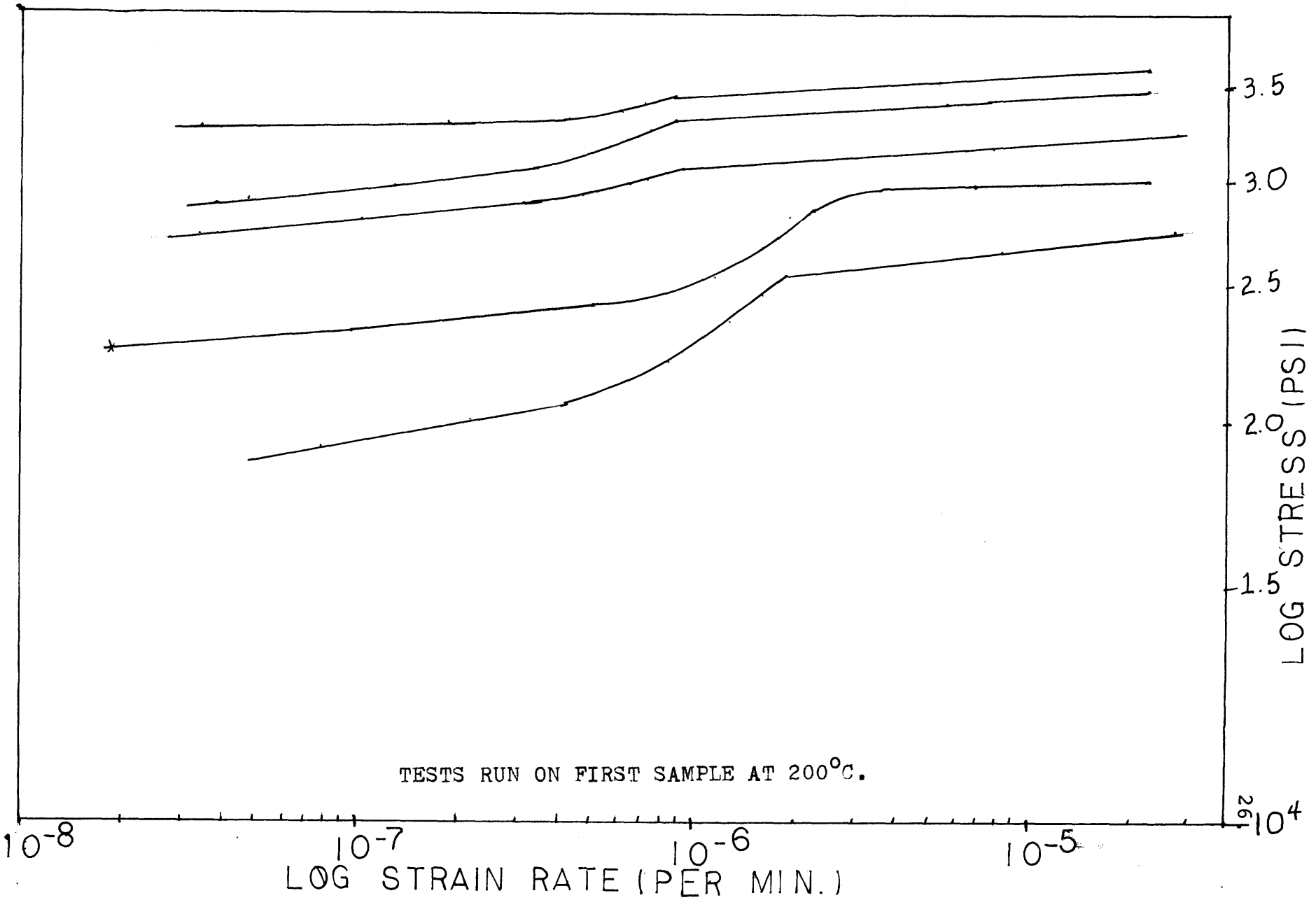

5/32" RADIUS, MIN.
THREADS:
.25" DIAM.
20 PER IN.
.640" GAUGE LENGTH
.160" DIAM.

FIGURE 2



TESTS RUN ON FIRST SAMPLE AT 200°C.

FIGURE 3

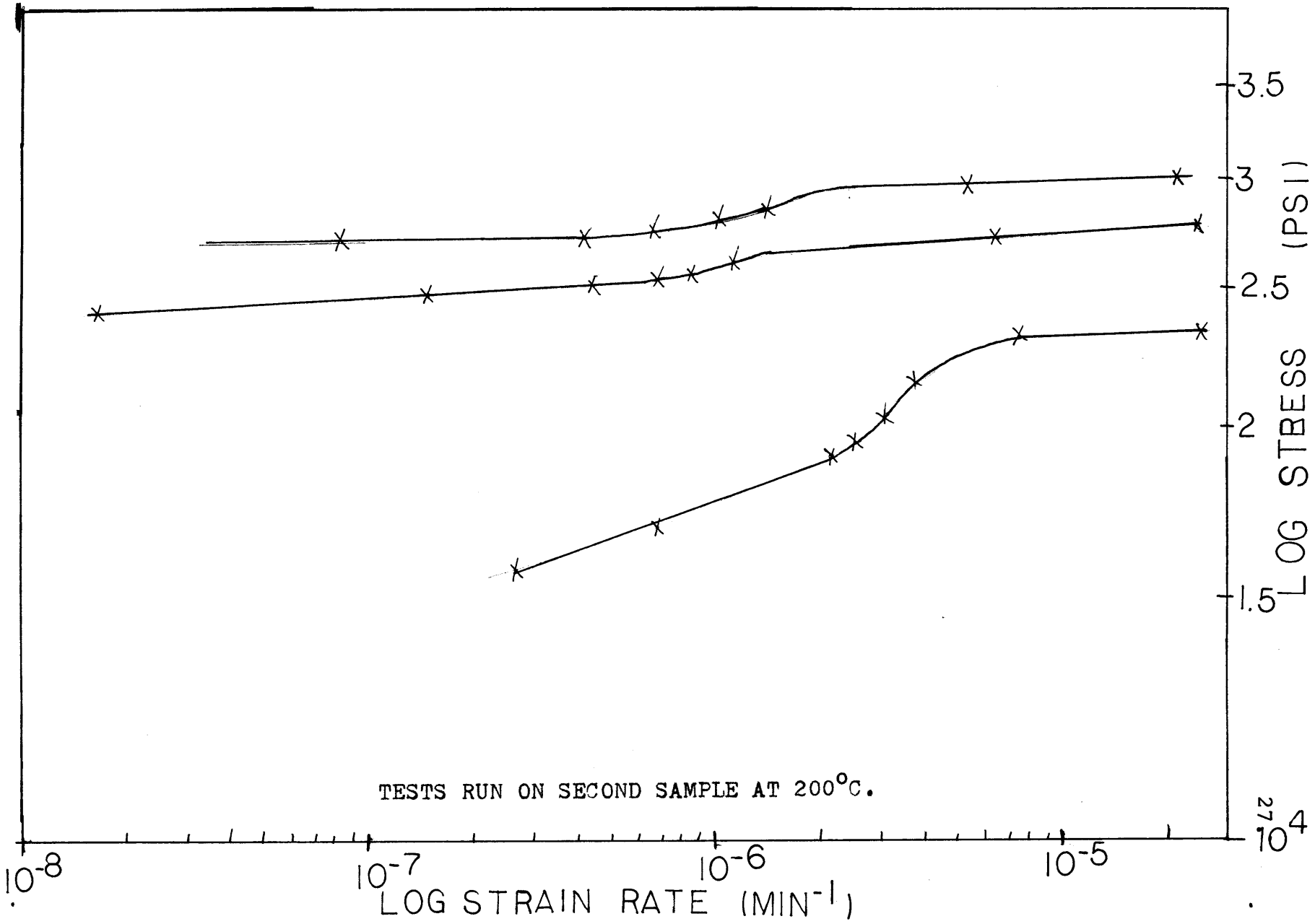

3.5
3
2.5
2
1.5
LOG STRESS (PSI)
TESTS RUN ON SECOND SAMPLE AT 200°C.
10^{-8}
10^{-7}
10^{-6}
10^{-5}
LOG STRAIN RATE (MIN^{-1})

FIGURE 4

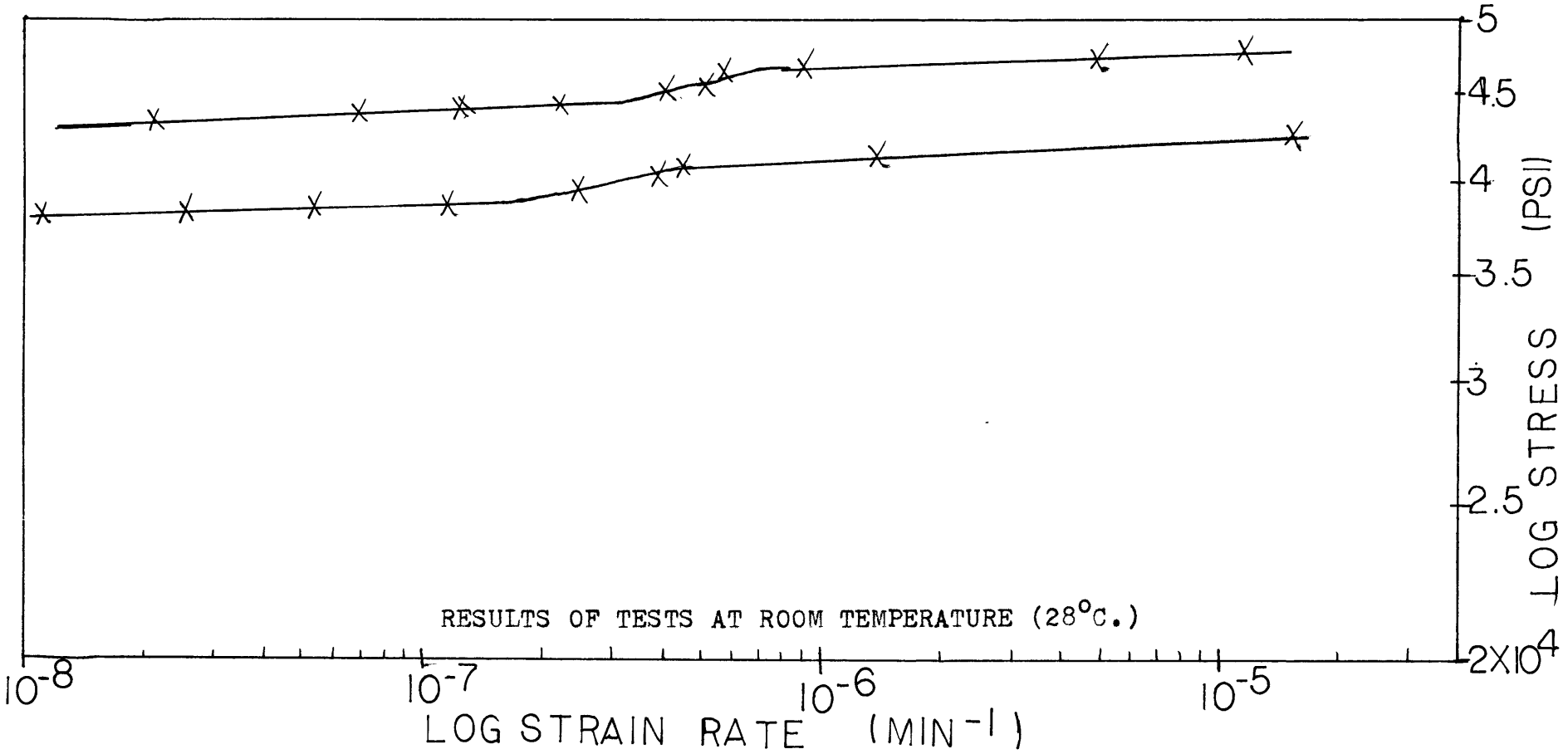
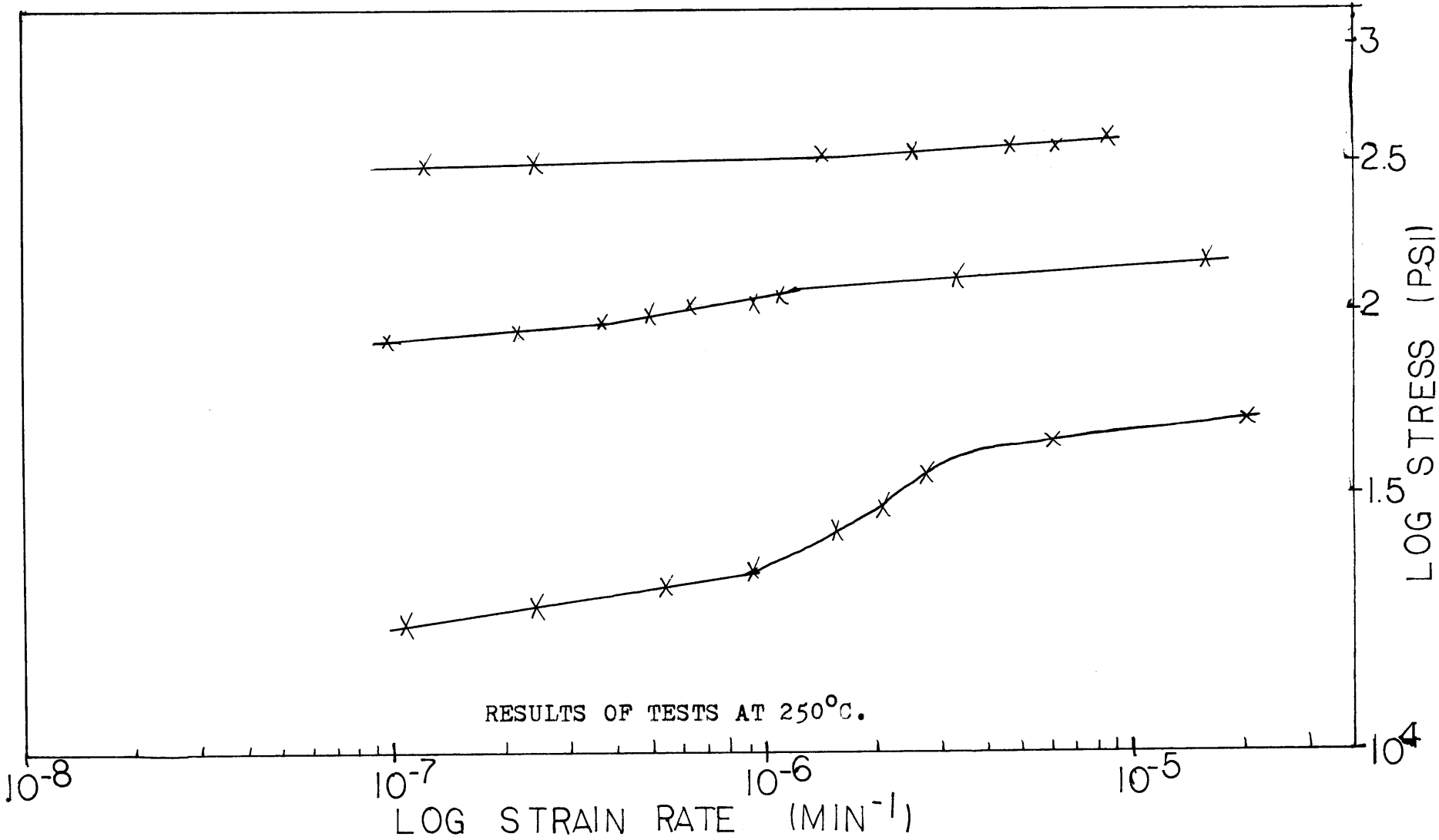

5
4.5
4
3.5
3
2.5
2X10^4
LOG STRESS (PSI)
RESULTS OF TESTS AT ROOM TEMPERATURE (28°C.)
10^-8
10^-7
10^-6
10^-5
LOG STRAIN RATE (MIN^-1)

FIGURE 5


3
2.5
2
1.5
10^4
LOG STRESS (PSI)
RESULTS OF TESTS AT 250°C.
10^-8
10^-7
10^-6
10^-5
LOG STRAIN RATE (MIN^-1)