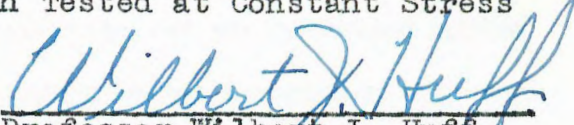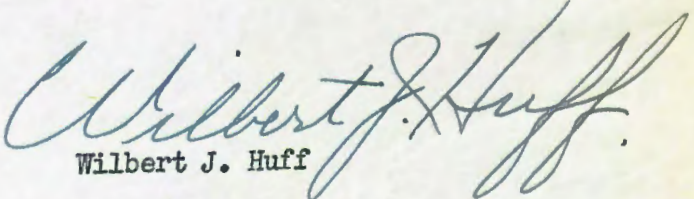John E. Breen, M.S., 1954

Creep Rupture Properties of Tin Tested at Constant Stress

Thesis and Abstract Approved: _____
Professor Wilbert J. Huff
Chairman, Chemical Engineering
Department

The thesis by John E. Breen on "Creep Rupture Properties of Tin Tested at Constant Stress" bears my name as director. However, in this capacity I merely took responsibility for the execution of the thesis and should not be listed in publications as contributing to the direction, which correctly should instead be accorded to Dr. Johannes Weertman alone when public announcement is made.

Wilbert J. Huff

CREEP RUPTURE PROPERTIES OF TIN TESTED AT CONSTANT STRESS

by
John E. Breen

ACKNOWLEDGEMENTS

The author wishes to acknowledge the guidance and help
of Professor Wilbert J. Huff and Dr. Johannes Weertman
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material.

TABLE OF CONTENTS

SECTION I

INTRODUCTION

Purpose and Scope. This investigation was initiated to study the creep characteristics of a simple, relatively pure, material at constant stress as compared to that tested at constant load. Tin was selected for the investigation.

A series of creep tests were run to rupture at constant stress and constant load. The test variables were adjusted in these tests so that time to rupture varied from approximately 300 minutes to 33500 minutes (5 hours to 558 hours) at room temperature, and from 40 minutes to 2500 (0.67 hours to 41.7 hours) at 200 °F.

General Considerations. The phenomenon of creep has been extensively investigated especially in the last twenty years as the result of the need for materials that will withstand high stresses at elevated temperatures. Most of this testing has been of an empirical nature on the conventional engineering alloys, or modifications of these alloys. Practically all of this testing has been done at constant load. All of these investigations have emphasized the fact that creep is an extremely complex phenomenon which is structure, strain-rate, and temperature sensitive. The complexity of the metallurgical structure of most creep-resistant alloys together with their structural instabilities makes fundamental studies of the creep curve with these alloys very difficult. Investigators of the creep curve, as such, have generally used single phase materials such as aluminum, copper, lead, and tin.

In the usual creep test at constant load the stress pro-
gressively increases as the specimen elongates. As the creep
rate under most circumstances is a power function of the
stress it is readily seen that test conditions are continually
changing in any constant load test. The suggestion has been
advanced by many investigators that fundamental studies of
creep should be done at constant stress. Furthermore, the
results of such tests could be more readily applied to creep
under compression or torsion loading than the results of
constant load tests.

Strangely enough some of the first work on creep was
done at constant stress. Andrade (1)[1] in his study on the
creep of lead wire made use of the sinking hyperbolic weight
method for maintaining the stress constant, see Figure 1.
This was also the method used in this present investigation.
The basic philosophy of any constant stress test is that the
load should be relaxed as the cross sectional area is decreas-
ed such that the ratio of load to area remains constant, e.g.,
$\sigma = \frac{L}{A} = K$. In all constant stress tests known to the
author it is assumed that the volume of the test-specimen
remains constant and that the cross-sectional area remains
uniform. The length of the specimen is considered inversely
proportional to the area and also to the load. The necessary
condition for constant stress can be obtained by allowing the

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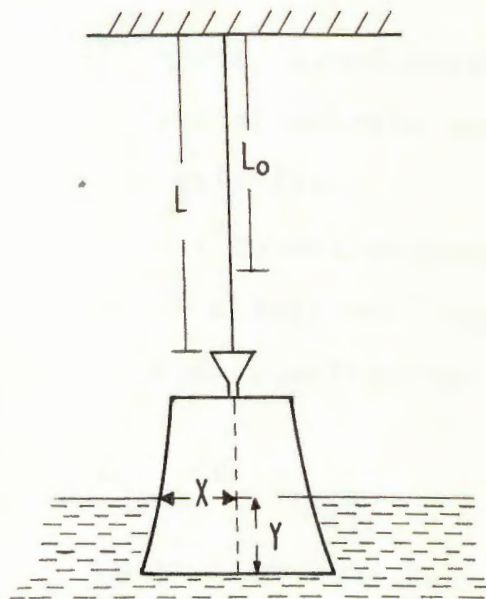[1]The figures appearing in parentheses pertain to the
Bibliography.

Figure 1 – Sinking Hyperbolic Weight Method for
Maintaining Constant Stress

weight causing the stress to sink into a liquid. It can be shown that the necessary shape is a hyperbola of revolution whose equation is given by $Y = \left(\dfrac{M\,L_O}{\rho\,\pi}\right)^{\frac{1}{2}} \cdot \dfrac{L_O}{L_O + X}$. X and Y are shown in Figure 1. M is the mass of the weight; L_O is the initial length of the specimen, and ρ is the density of the fluid. The method is very simple, but rather inflexible as each weight has a unique mass and corresponds to a definite specimen length.

Other constant stress creep tests have been devised. The most famous is that of Andrade and Chalmers (2) which uses a lever arm-cam relation.

As a result of his investigations on the creep of lead, iron, copper, lead-tin alloy, and frozen mercury, Andrade (1)(3) postulated the following equation for the creep curve:

$L = L_O (1 + \beta t^{1/3}) e^{Kt}$, where

L = length of specimen at time t,

L_O = length of specimen immediately after the application of the load,

t = time,

β and K are constants depending on the temperature, stress, and material.

For small values of t, e^{Kt} will approach one and the curve can be represented by $L = L_O (1 + \beta t^{1/3})$. Differentiating

to obtain the creep rate we get $\dfrac{dL}{dt} = \dfrac{1}{3} \beta L_O t^{-2/3}$ which

shows a rate that diminishes with time. At times such that

the β term contributes little to the creep, the curve can be represented by $L = L_o e^{Kt}$ which is the equation for constant true strain rate. Differentiating we obtain $\frac{1}{L} \frac{dL}{dt} = K$. Most investigators still divide the creep curve into the β or transient stage and the K or steady state stage which is also called the quasi-viscous stage.

Andrade claimed that during constant stress creep tests on simple metals that there was no third stage creep. The author will discuss this later in connection with his own work on constant stress creep testing.

There have been many other equations offered for the creep curve. As a typical example we might take that of Hasiguti and Owadano (4). $S = A - \frac{1}{bt - c} + Kt$. S equals the logarithm of the ratio of the length of specimen at the time t to its initial length. A,b,c, and K are constants independent of time. Considering only the transient component it is readily seen that $\frac{ds}{dt} = b (a - s)^{-2}$ which is similar to the equation of a second order chemical reaction. Taking only the steady state component it is seen that the true strain is a straight line function with time as indicated by Andrade.

Excellent text books for a complete review of this subject are those by Sully (5) and Rotherham (6).

Previous Work on the Creep of Tin. The creep of Tin has been investigated by Freeman and Quick (7), Hanson and Sandford (8), Chalmers (9), and Hanson and Pell-Walpole (10).

Freeman and Quick tested pure tin and pure lead and a 1:1 tin-lead alloy in the form of wire. Hanson and Sandford ran creep tests of long duration (one year) on tin and tin alloys of silver bismuth, antimony, and cadmium. These tests were of a practical nature to obtain information on the behavior of tin solders under load at room temperature. No attempt was made to hold the room temperature constant because the authors felt that the temperature differences would counter balance each other in tests of such long duration. Chalmers' work was concerned with the detection of extremely small amounts of plastic deformation. He was able to measure strains of 10^{-7} cm/cm. The data is applied to theoretical considerations of micro-creep and creep recovery in the β range. Hanson and Pell-Walpole (10) also investigated the creep of tin solders.

Tyte (11) found that the rate of viscous flow in tin was an exponential function of load at a given temperature, and an exponential function of absolute temperature at a given load. This work is important because from it Kauzmann (12) calculated the activation energy for the creep of tin.

SECTION II

TEST PROCEDURE

A program was initiated to examine the constant stress creep test as a tool and to determine its advantages if any over the constant load creep test. It was decided to use the sinking hyperbolic weight method. Weights of 5.0 Kg, 4.0 Kg, 3.2 Kg, 2.5 Kg, 2.2 Kg, and 1.9 Kg were machined. All test-specimens were 10 cm. in length. All room temperature tests were run in a constant temperature room which maintained a temperature of 70° F $\pm 2^{\circ}$ F. All specimens were annealed for one hour, in air, at 212° F.

Periodic measurements of elongation were made so that each creep curve could be fully plotted. All elongation measurements were made optically. An electrical timer and switching device were used to determine the time to rupture. The specimen was loaded directly to avoid any friction in pulleys or levers. The water-level into which the hyperbolic weight sank was maintained constant.

The tin wire used as specimen material was analyzed spectrochemically; the results are shown in Table I. The total impurities present are less than 0.173%.

A transverse photomicrograph of the specimen material is shown in Figure 2.

Table I

Results of Spectrochemical Analysis on Specimen Material

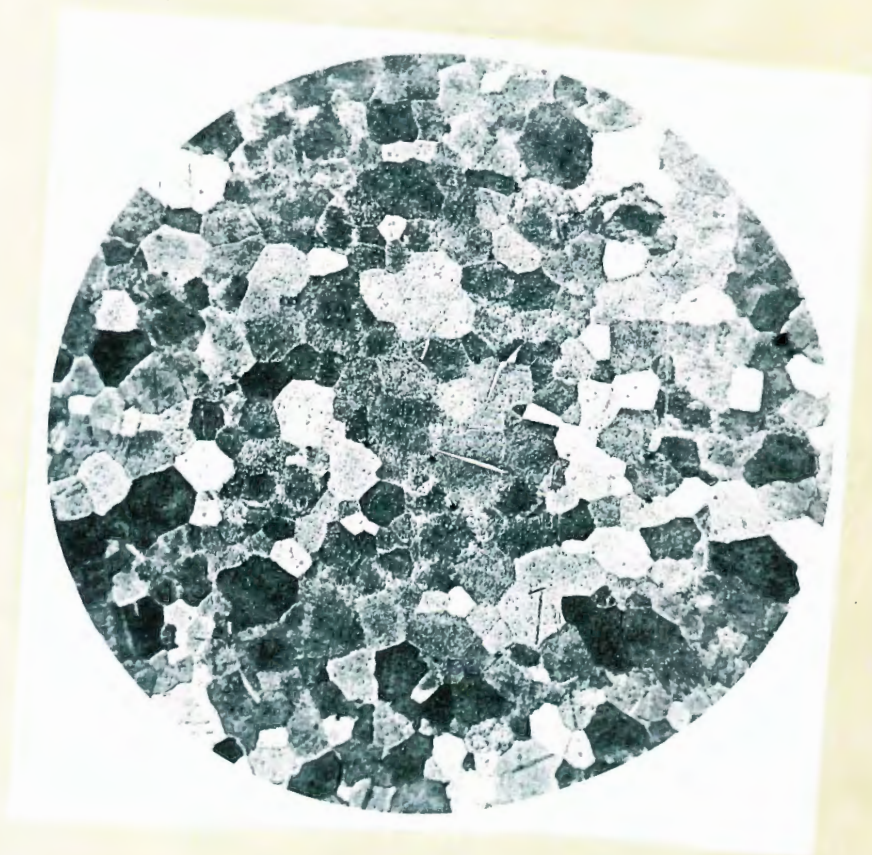| Element	Maximum Amount Present
Cu	0.1 %
Pb	0.01
Si	0.01
Fe	0.01
Bi	0.01
In	0.01
As	0.01
Sb	0.01
Ag	0.001
Mg	0.001
Ca	0.001
Al	Not detected
Zn	Not detected
Total Impurities	0.173%

Figure 2 - Photomicrograph of Specimen
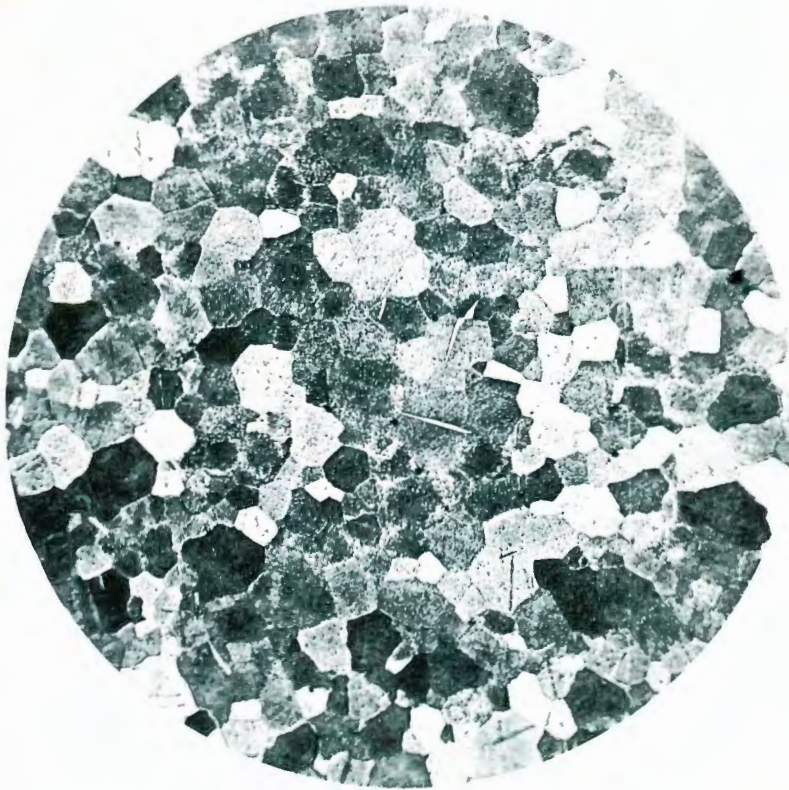Material (Tin) at 125 Diameters

Figure 2 - Photomicrograph of Specimen
Material (Tin) at 125 Diameters

SECTION III

RESULTS

A total of thirty-six creep-rupture tests were completed.
Twenty-five tests were run at room temperature (70° F). Of
these, twelve were run at constant stress, and thirteen at
constant load. Eleven tests were completed at 200° F. Of
these six were run at constant stress and five at constant
load. Five stress levels were used in the tests at room
temperature and four stress levels were used in the tests
at 200° F.

A typical data sheet of a creep-rupture test is shown
in Table II. This is the data for test No. 6 which was run
under constant stress, at 1059.5 psi and 70° F. Similar
data sheets were taken for all tests.

A graphical representation of these data, commonly
called a creep curve, is shown in Figure 3 (a) and Figure 3 (b).
The usual creep curve shows elongation as a function of
time. Here, the true strain has been shown as a function
of time as this method will yield more fundamental information
about test material. If the initial length is called L_1
and the length at any time t called L, then the elongation
is simply $L - L_1$. On the other hand the instantaneous strain

rate is $\frac{dL}{L}$ and the true strain is $\int_{L_1}^{L} \frac{dL}{L}$ which is $\ln \frac{L}{L_1}$.

The length is measured in centimeters and the initial length

Table II

Typical Data Sheet For Creep-Rupture Test

Test No. 6; Material - Sn wire, 0.092 inches diameter;
Stress = 1059.5 psi (Initial Load = 3.2 Kg); Temp. = 70°F.

Time (Min.)	Elongation (%)	0.1 L (cm.)	log 0.1 L	Strain 2.3 log 0.1 L
0.0	0.00	1.0000	0.00000	0.00000
1.0	0.29	1.0029	0.00126	0.00290
7.5	0.31	1.0031	0.00134	0.00308
14.7	0.38	1.0038	0.00164	0.00377
25.7	0.47	1.0047	0.00204	0.00469
34.6	0.52	1.0052	0.00216	0.00497
43.5	0.62	1.0062	0.00269	0.00619
54.7	0.69	1.0069	0.00299	0.00688
107.5	1.03	1.0103	0.00455	0.01024
126.7	1.17	1.0117	0.00505	0.01162
175	1.52	1.0152	0.00676	0.01555
261	2.09	1.0209	0.00899	0.02068
295	2.33	1.0233	0.01001	0.02302
647	4.88	1.0488	0.02070	0.04761
1286	9.60	1.0960	0.03981	0.09156
1372	10.24	1.1024	0.04234	0.09738
1432	10.69	1.1069	0.04411	0.10145
1488	11.10	1.1110	0.04571	0.10515
1547	11.74	1.1174	0.04821	0.11088
1665	12.42	1.1242	0.05085	0.11696

Table II (cont.)

Typical Data Sheet For Creep-Rupture Test

Test No. 6; Material = Sn wire, 0.092 inches diameter;
Stress = 1059.5 psi (Initial Load = 3.2 Kg); Temp. = 70° F.

Time (Min.)	Elongation (%)	0.1 L (cm.)	log 0.1 L	Strain 2.3 log 0.1 L
1769	13.19	1.1319	0.05381	0.12376
2959	22.48	1.2248	0.08807	0.20256
3524	27.13	1.2713	0.10425	0.23978
4285	32.82	1.3282	0.12326	0.28350
4990	40.10	1.4010	0.14644	0.33681
5614	46.24	1.4624	0.16507	0.37966
5692	47.01	1.4701	0.16736	0.38493
5793	48.04	1.4804	0.17038	0.39187
5904	49.16	1.4916	0.17365	0.39940
5999	50.17	1.5017	0.17658	0.40613
6083	51.03	1.5103	0.17906	0.41184
6442	54.93	1.5493	0.10013	0.43730
7072	62.46	1.6246	0.21075	0.48473
7176	63.77	1.6377	0.21423	0.49273
7313	65.56	1.6556	0.21896	0.50361
7413	66.93	1.6693	0.22254	0.51184
7530	68.56	1.6856	0.22676	0.52155
7830	72.97	1.7297	0.23797	0.54733
*8396	86.2	1.862	0.2700	0.6210

*Specimen Fractured

in all cases was 10 centimeters. Therefore in this case the true strain is ln 0.1 L. The above analysis is correct only so long as the specimen area remains uniform. However, this assumption is also necessary if the stress on the specimen is to be constant. Therefore, a series of measurements were made optically on the diameter of the specimen at various positions along the gauge length during the entire creep test. These measurements showed that the maximum deviation of the specimen area from the mean area was + 1.5% to - 3.0%. During the first sixty percent of the test time, the maximum deviation was + 0.5% to - 1.0%. The author feels that these deviations are well within the allowable errors for this type of work.

Figures 3 (a) and 3 (b) are typical examples of the type of curves that were drawn for all the tests that were conducted. Figure 3 (a) shows the entire curve. Figure 3 (b) shows the initial portion of the curve on an enlarged scale which allows a more detailed analysis of this portion. It can be seen that at constant stress the strain is a straight line function with time. Therefore the curve, except for the small portion at the end lies entirely within the steady state region. The slope of this line is the true strain rate and is constant. The equation for a curve of this type is simply $e = e_0 + Kt$. Where e is the strain at any time t; K is the strain rate, and e_0 is the instantaneous strain when the load is applied. This equation in terms of its length would be $L = L_0 e^{Kt}$. This is Andrades' equation minus
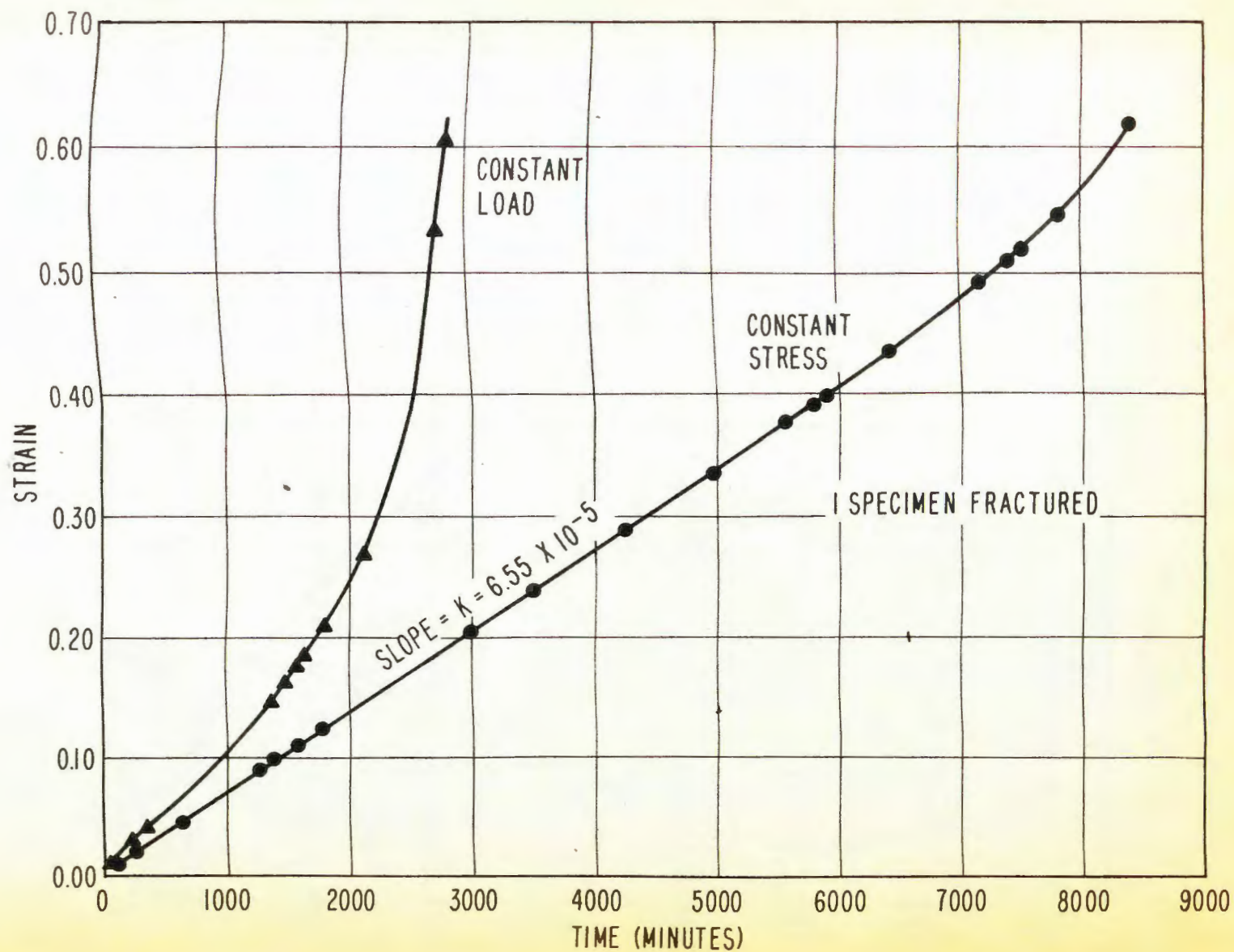
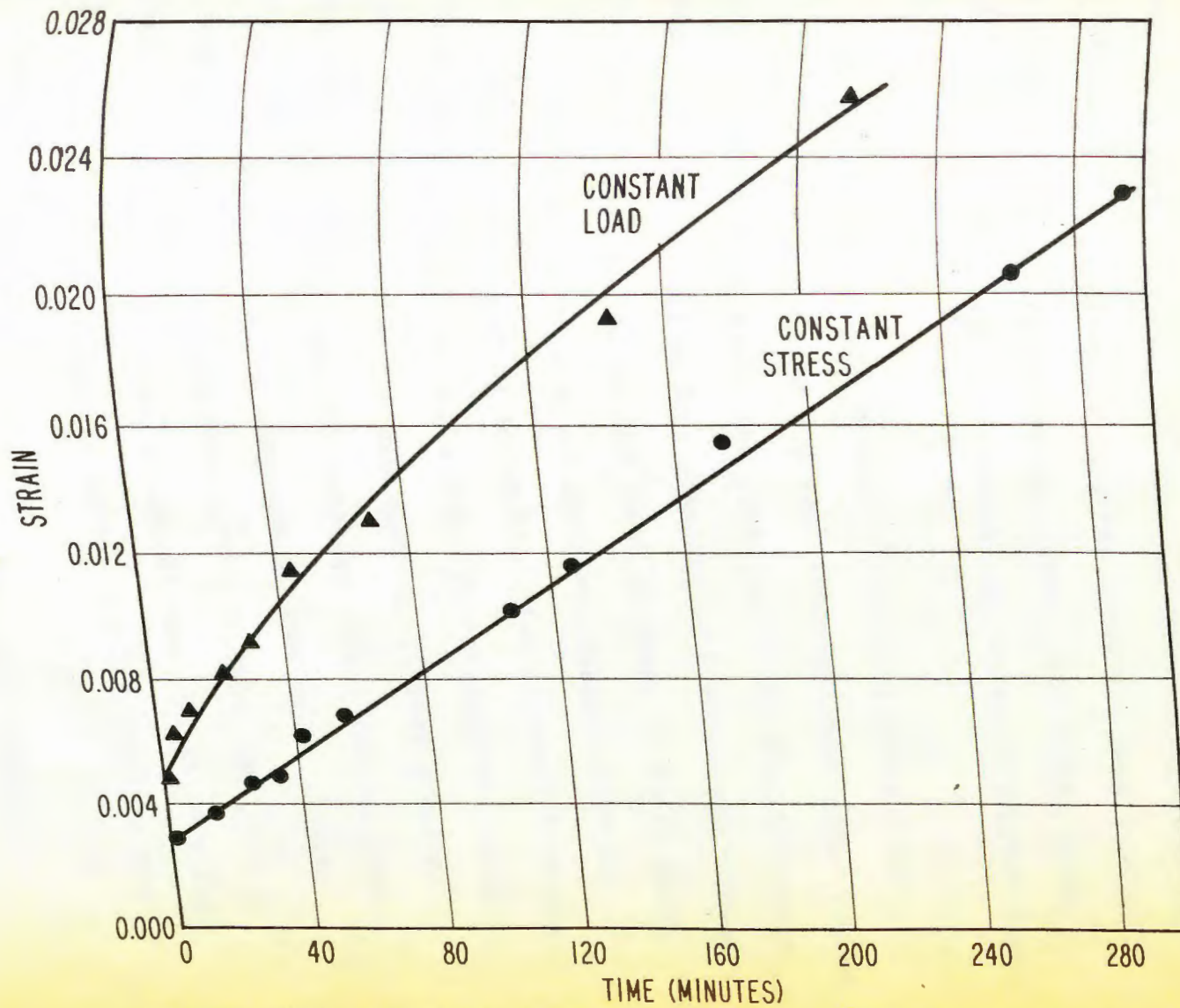Figure 3(a) - Creep of Tin at 70 °F and 1059.5 psi

Figure 3(b) - Initial Creep of Tin at 70 °F and 1059.5 psi

its component for the β flow.

In Figures 3 (a) and 3 (b) is also plotted the creep curve for a specimen at the same initial stress and temperature but under conditions of constant load. In this case there is a small amount of β flow as shown in Figure 3 (b). The minimum strain rates for the two cases are very similar but the rapid acceleration of the strain rate during later stages for the test run at constant load makes interpretation difficult.

Creep curves similar to those shown in Figures 3 (a) and 3 (b) were plotted for all the tests that were run. Scales large enough to facilitate the plotting of the data and large enough to allow accurate determination of K were used. These graphs were usually two to ten times as large as those shown in Figure 3 (a). A summary of the data obtained from the creep rupture tests and of the information obtained from the creep curves is given in Table III. Stress-Rupture curves at 70° F and 200° F for tin are shown in Figures 4 and 5 respectively. The values for constant stress and constant load are both included for purposes of comparison. These are conventional stress-rupture curves in which log. time to rupture is a linear function of log. stress. Plots were made of log. time to rupture against stress but the function was not linear.

Figures 6 and 7 show the functional relationship between the true strain rate (k) and the applied stress. It can be seen that log. K is a linear function of stress at a given temperature.

Table III

Summary of Creep-Rupture Tests

Test No.	Type Test	Test Temperature (°F)	Initial Stress (psi)	Time to Rupture (Minutes)	Total Elongation (%)	K (X 10^{-5})
25	Constant Stress	70	1655.5	1221.3	105.2	64.8
26	"	"	"	945.5	77.0	54.0
1*	"	"	1324.4	2948	104.7	21.5
2*	"	"	"	3202	93.4	18.8
3	"	"	"	3031	73.2	16.7
4	"	"	"	3132	78.8	18.1
5	"	"	1059.5	9510	85.0	6.0
6	"	"	"	8396	86.2	6.6
12	"	"	827.8	23500	72.7	2.1
13	"	"	"	32157	102.2	2.0
16	"	"	"	25035	105.5	2.5
24	"	"	728.4	33508	91.0	1.6
18	Constant Load	"	1655.5	399.0	92.3	67.0
19	"	"	"	401.5	61.8	57.0
22	"	"	"	449.6	77.0	50.0
23	"	"	"	338.0	96.9	73.2
9	"	"	1324.4	1112	93.3	19
10	"	"	"	1164	104.5	17
7	"	"	1059.5	2845	83.6	11.3

*Specimen was not annealed before test.

Table III cont.

Summary of Creep-Rupture Tests

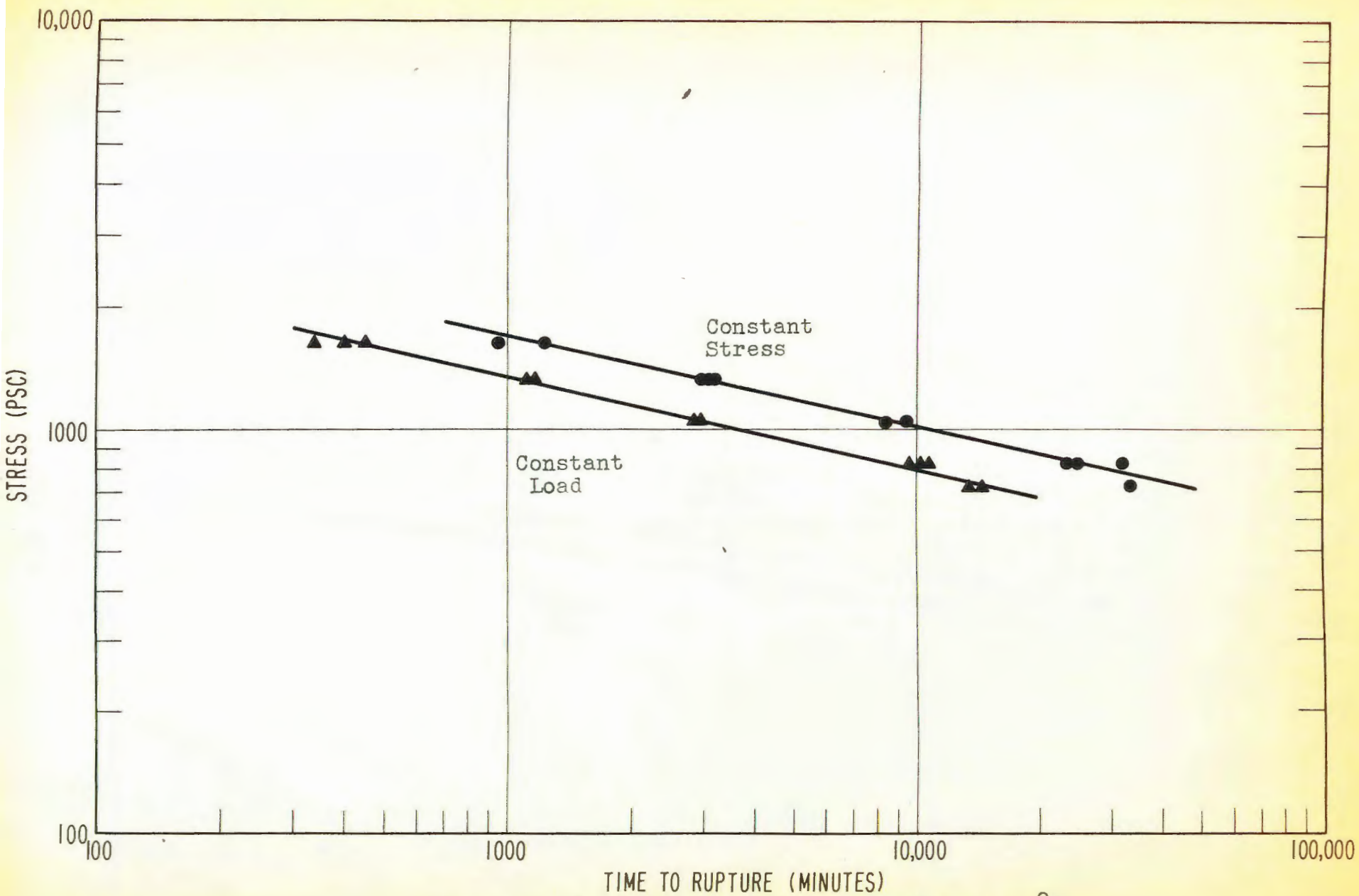| Test No.	Type Test	Test Temperature (°F)	Initial Stress (psi)	Time to Rupture (Minutes)	Total Elongation (%)	K (X 10^{-5})
11	Constant Load	70	1059.5	2914	84.7	10.3
14	"	"	827.8	10868	93.6	—
15	"	"	"	10295	67.4	2.9
17	"	"	"	9596	81.6	3.2
20	"	"	728.4	14622	86.9	2.3
21	"	"	"	13400	83.6	2.7
C - 5	Constant Stress	200	1059.5	109.2	41.2	260.
C - 3	"	"	827.8	317.9	31.1	75.
C - 4	"	"	"	463.5	24.1	38.
C - 6	"	"	728.4	1360.3	84.9	59.
C - 8	"	"	"	1139.6	60.0	37.
C - 7	"	"	629.1	2579	43.3	19.
C - 9	Constant Load	"	1059.5	39.7	82.4	Too Fast to Measure
C - 10	"	"	827.8	148.7	48.8	160
C - 11	"	"	728.4	345.8	42.7	80
C - 13	"	"	"	326.5	31.3	70
C - 12	"	"	629.1	977.5	48.3	29

Figure 4 Stress- Rupture Curve for Tin at 70°F
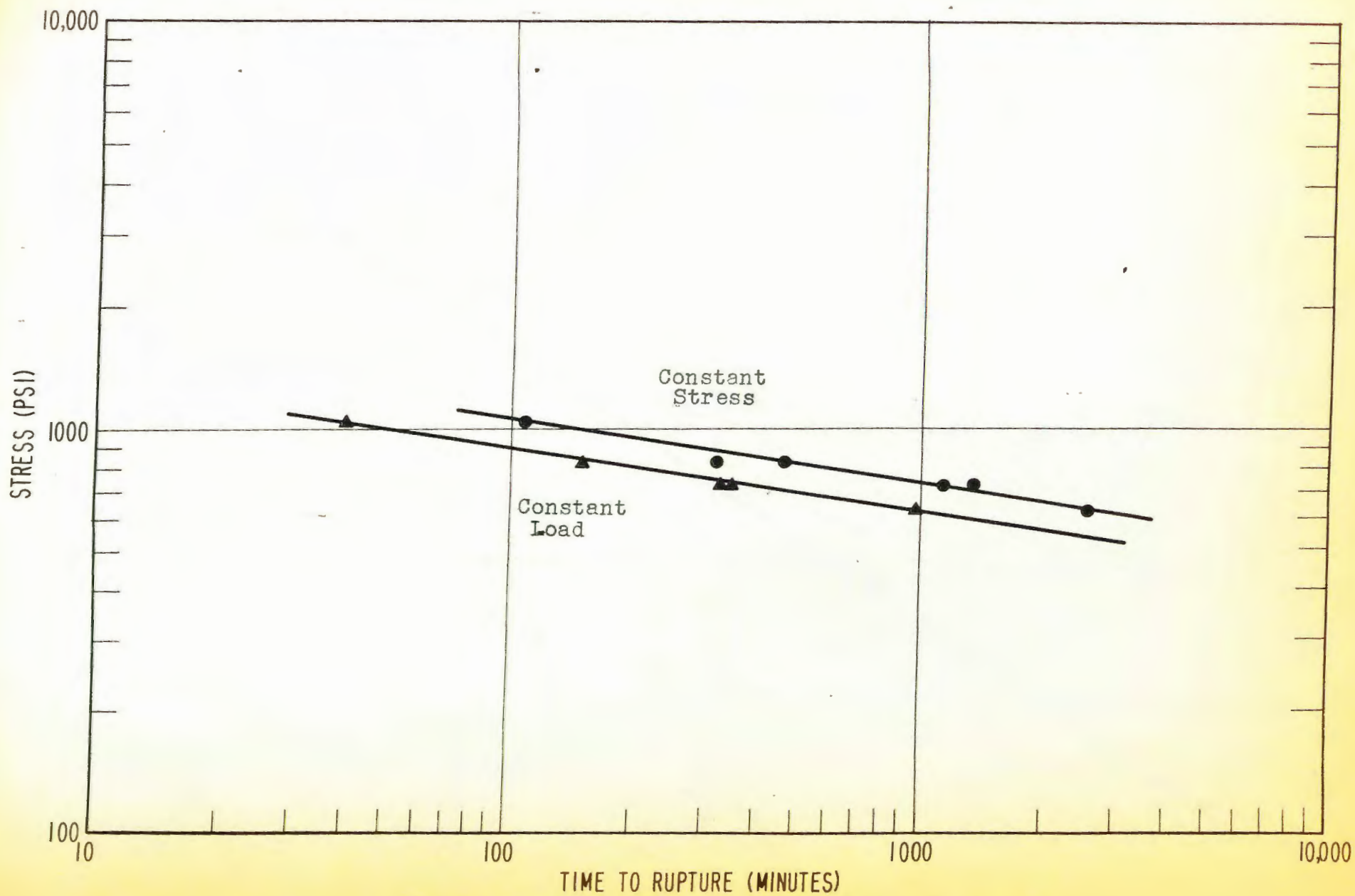
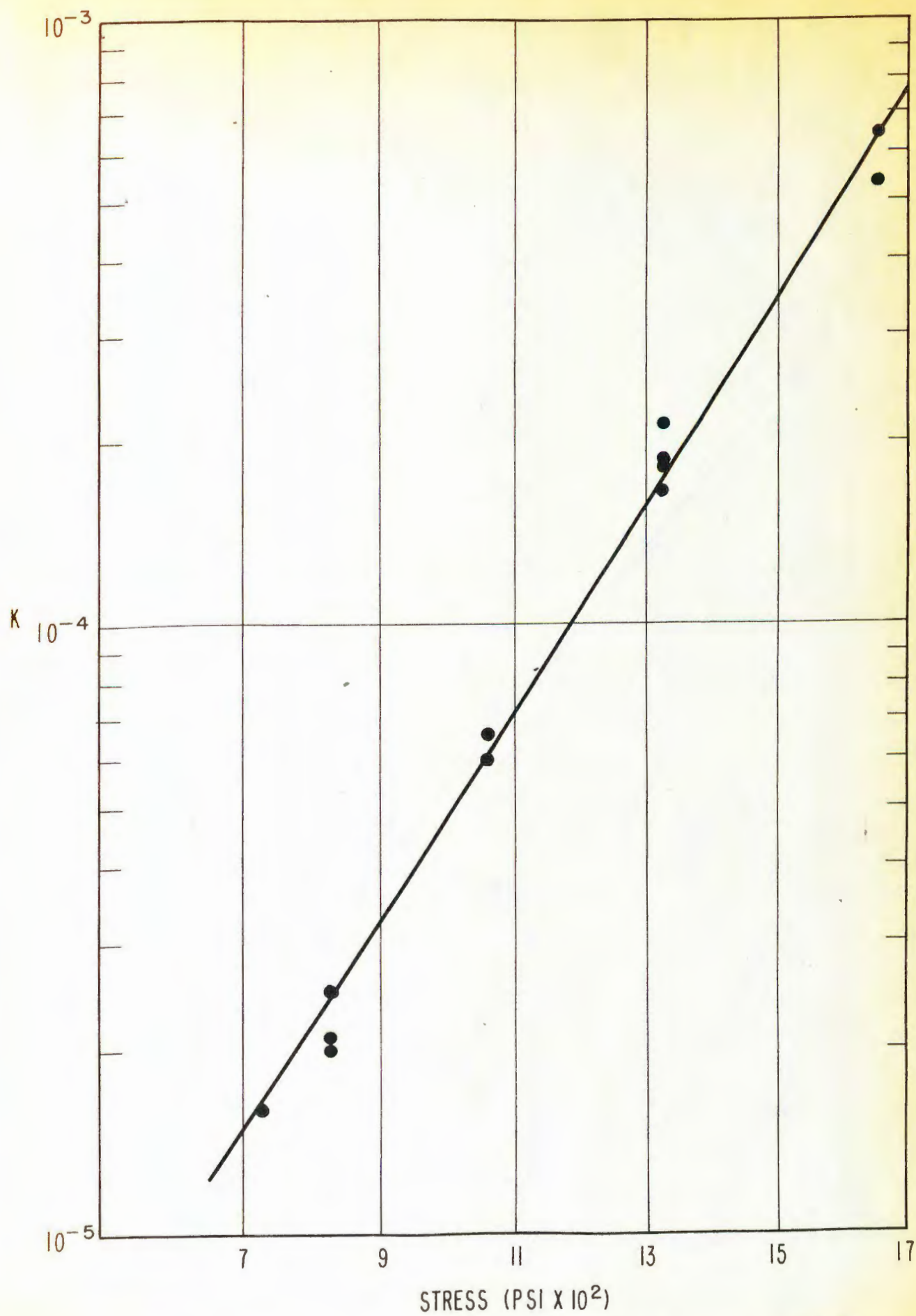Figure 5 Stress-Rupture Curve for Tin at 200 °F

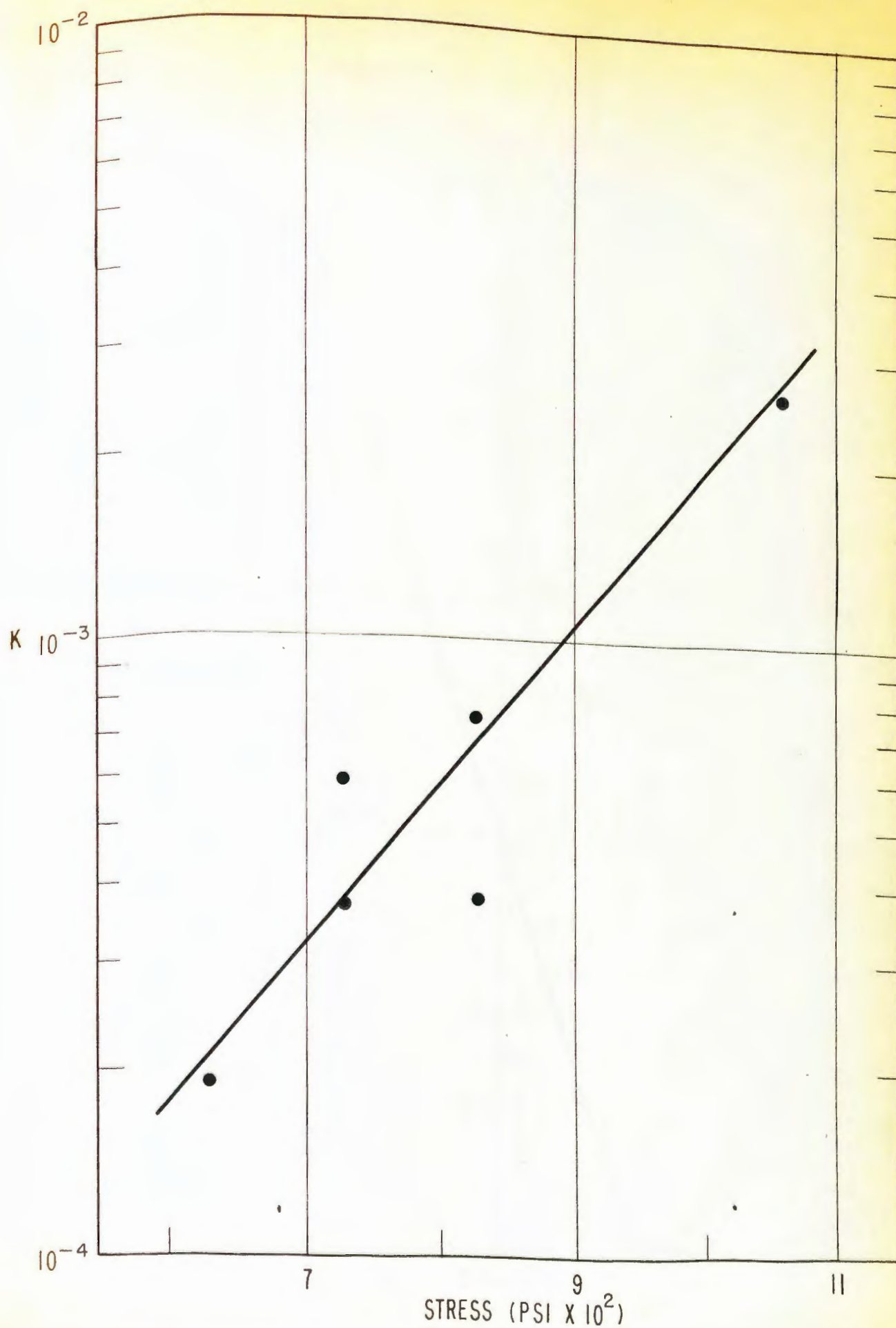Figure 6- Strain Rate as a Function of Stress at 70 °F

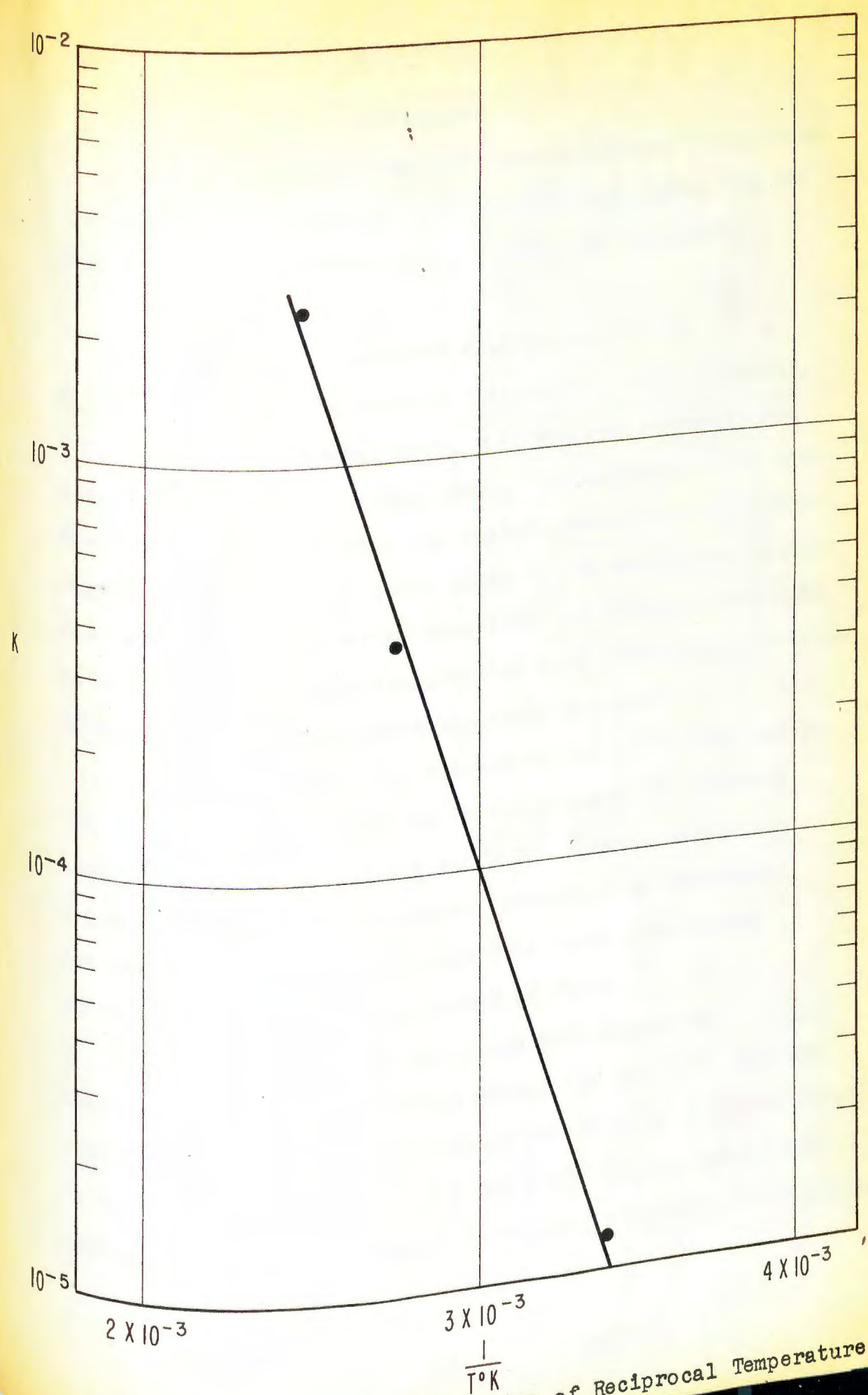Figure 7 - Strain Rate as a Function of Stress at 200°F

Figure 8 - Strain Rate as a Function of Reciprocal Temperature

SECTION IV

DISCUSSION

Many investigators who have correlated and interpreted
creep data have suggested several equations giving the re-
lationship between creep rate, stress, and temperature.

The most widely accepted equation is $K = Ae^{-\frac{Q}{RT}}$,
where K is the strain rate or creep rate, A is a constant,
T is the absolute temperature, R is the gas constant, and
Q is the activation energy. Among others Sherby, Orr, and
Dorn (13) have shown that for a given metal the activation
energy is constant and about equal to the activation energy
for self-diffusion. Using data from the literature as well
as their own they have demonstrated this for aluminum, iron,
nickel, copper, zinc, platinum, gold, and lead.

Boas and Fenshaw (14) determined the activation energy
for self-diffusion in tin to be $5.9 \pm 0.4 \times 10^3$ calories
along the a axis and $10.5 \pm 0.5 \times 10^3$ calories along the
c axis. Tin because its crystal structure is tetragonal
has two activation energies and thus is an interesting
material to study from this point of view.

Rotherham, Smith and Greenough (15) showed no
correlation between activation energy for internal friction
peak for grain boundary relaxation and that for self-diffusion
in tin; their value being $19 \pm 2.5 \times 10^3$ for the activation
energy of internal friction. On the basis of work on aluminum,

alpha brass and alpha iron, Ke (16) proposed that the activation energies for steady state creep in single crystals, and those for self-diffusion and internal friction peak for grain boundary relaxation should be approximately equal as a general rule. Kauzmann (12) using the data of Tyte (11) calculated the activation energy for creep in tin as 5.35×10^3 calories. This agrees with the Q value for self-diffusion along the a axis.

In the present work the activation energy was calculated from the data shown in Figures 6 and 7 as follows:

$$\frac{K_1}{K_2} = e^{-\frac{Q}{R}\left(\frac{1}{T_1} - \frac{1}{T_2}\right)}$$

$$\ln \frac{K_1}{K_2} = -\frac{Q}{R}\left(\frac{1}{T_1} - \frac{1}{T_2}\right)$$

$$\ln \frac{1.94 \times 10^{-3}}{4.8 \times 10^{-5}} = -\frac{Q}{1.99}\left(\frac{1}{366} - \frac{1}{294}\right)$$

$$Q = 11.0 \times 10^3$$

This value of Q is equal to that for self-diffusion along the c axis. To check this value an additional test was run at 271 $^\circ$F (406 $^\circ$K). As in the above calculation a stress of 728 psi was used. Values of ln K were plotted against reciprocal temperature as shown in Figure 8. The slope of this curve is $\frac{Q}{R}$ and is constant. It is interesting to note that

neither Kauzmann (12) nor the present author found a Q for
creep that was the average of those for self-diffusion in
the two crystallographic directions. Further investigations
should be made to determine the conditions under which the
various activation energies are dominant. The work of
Tyte (11) from which Kauzmann (12) calculated his Q was
done using comparitively short time tests in which the
total strain was small. The present work was done at
constant stress using longer time tests that were run to
rupture. Purity of material, metallographic structure,
or grain orientation may all play a part.

The relation of strain rate, stress, and temperature
as shown in Figures 6 and 7 can best be expressed by an

equation of the type $K = A^* e^{-\frac{Q}{RT}} e^{B\sigma}$ where A^* is
considered a constant, although it may be a slowly
changing variable depending on T and σ whose magnitude
of change is small compared to the exponential functions.
Dorn et al. (17) found that B did not change with temp-
erature. However, Cotrell and Aytekin (18) believe that
Q varies slightly with stress and the B term varies with
temperature. They propose that the equation should be:

$$K = A^* e^{-\frac{Q_0}{RT}} e^{\frac{C(\sigma_0 - \sigma)}{RT}}$$

where $C = BRT$, σ_0 is any reference stress and $Q_0 = Q$ at σ_0.

The magnitude of the difference between the equation of
Dorn and that of Cotrell are small and the present data have

not been developed over a sufficiently wide range of
temperature to resolve the difference.

SECTION V

CONCLUSIONS

(a)   The creep of tin above 70 °F under constant stress
and stresses greater than 628 psi lies entirely in the
steady state region and can be represented by the equation:

$$1 = 1_0 \, e^{Kt}$$

(b)   The activation energy for the creep of tin is
11.0×10^3 calories which is approximately equal to that
for self-diffusion along the c axis.

(c)   Log rupture-time is a linear function of log stress.

(d)   Log strain rate is a linear function of stress.

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