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Submitted in partial fulfillment of
the requirements for the degree of

MASTER OF SCIENCE

IN

MECHANICAL ENGINEERING

United States Naval Postgraduate School
Monterey, California

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the thesis requirements for the degree of
MASTER OF SCIENCE
IN
MECHANICAL ENGINEERING

from the
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ABSTRACT

An investigation was conducted to predict the creep behavior of a beryllium-copper alloy under cyclic temperature conditions by use of a temperature compensated time parameter, θ.

$$\theta = \int_0^t e^{-Q/RT}\, dt$$

where Q = activation energy

R = gas constant

t = time in hours

T = temperature, degrees Kelvin.

Temperature was cycled in the $\alpha+\beta$, $\alpha+\gamma$ and over the $\alpha+\gamma \rightleftharpoons \alpha+\beta$ regions. Isothermal tests were also conducted in these regions to determine the activation energies. The isothermal creep data was then correlated to the cyclic data by use of the θ parameter. Thus the effect of microstructural changes induced by the cycling temperature on creep was studied.

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INTRODUCTION

Applications for metals at high temperatures have greatly increased in recent years. Thus there is an increasing necessity for designing with creep as one of the limiting factors. Today there is available a fair amount of creep data for isothermal constant load conditions. However only upon rare occasions will these ideal conditions be found in actual applications. Since it has been reported that cyclic temperature environment may accelerate creep strain, [1,2,3] it follows that a design based upon isothermal tests will have a tendency to be on the unsafe side. A solution to this problem would be to conduct creep tests under the actual conditions to which the part in question would be subjected. However, this would be both an expensive and time consuming task. Therefore, the engineer must have a method of correlating constant load isothermal creep data to cyclic creep data. Sherby and Dorn have shown that if the creep - strain - time relation is known at a given stress and at a high temperature, the strain - time curve can be predicted for any other temperature or varying temperature by use of the temperature - compensated time parameter, θ. [4] The basic definition of θ is given by equation (1) which is

$$\theta = \int_o^t e^{-Q/RT} dt \qquad (1)$$

$$\theta = te^{-Q/RT} \qquad (2)$$

where:

t = time in hours
Q = activation energy for creep
R = gas constant
T = temperature in degrees Kelvin

valid for variable temperature conditions. For a constant temperature (temperature independent of time) this reduces to equation (2). Thus

a graphical representation of strain versus θ for tests conducted at various constant temperatures but at the same constant load will superimpose. Also the strain versus θ plot for a (variable) temperature history at the same constant load as the isothermal tests should superimpose on them. Thus isothermal data could be utilized to predict cyclic temperature creep curves. Although investigations to prove the validity of this theory are scarce, results of the few tests conducted show that in some cases the predicted strain will be less than that obtained experimentally.[5,6,7] This acceleration in the creep rate may be due to microstructural instabilities induced by the varying temperature. These microstructural changes may result in eliminating the hardening state normally associated with creep under isothermal temperatures. This hardening state is the result of very fine precipitates which impede the dislocation motion associated with slip by acting as barriers upon which the dislocations tend to pile up. But if the temperature is varied, these precipitates tend to form and then dissolve or decompose. Thus barriers originally effective in blocking slip break down, and the dislocations that have piled up are again free to move. The creep rate is therefore accelerated by the instability of the microstructure so that a particular strain would occur in a shorter time.

This investigation was undertaken to investigate the creep behavior of a beryllium-copper alloy under a cycling temperature-constant load condition by use of the θ parameter. Temperature was cycled in the $\alpha+\beta$ and $\alpha+\gamma$ regions and through the eutectoid reaction, $\beta \rightleftharpoons \alpha+\gamma$. Isothermal tests, conducted in these regions, were used to obtain the activation energy for creep and to predict the creep curve for the variable temperature tests. Comparison of these curves on a strain versus θ plot would

show the effect of changes in the amount of β or γ phase present as the temperature is cycled in the $\alpha + \beta$ or $\alpha + \gamma$ region. When temperature is varied in the $\alpha + \beta \rightleftharpoons \alpha + \gamma$ region, the effect of the phase change from $\beta \rightleftharpoons \alpha + \gamma$ can be determined. Examination of Fig. 10 reveals that temperature changes cause a definite innovation in the microstructure of the test specimen. Hence general test procedures as outlined above on a beryllium-copper alloy should give a good indication of the effect of microstructural changes on creep rate.

EXPERIMENTAL PROCEDURE

1. Test Material

Specimens were made from a beryllium-copper alloy received from The Beryllium Corporation of Reading, Pennsylvania. The composition of the alloy is given in Table I. The alloy, received in the solution—treated

TABLE I - CHEMICAL COMPOSITION*

Beryllium	2.00%	Silver	0.01%
Cobalt	0.22%	Tin	0.01%
Iron	0.14%	Nickel	0.01%
Silicon	0.12%	Chromium	0.01%
Aluminum	0.03%	Copper	Balance

*Chemical analysis supplied by The Beryllium Corporation

condition, was cold-rolled 8% from 0.10-inch strips to .092 inch thickness in order to obtain the maximum number of specimens per strip. The specimens were then machined to size from 4-1/2 x 3/4-inch blanks. Dimensions of the specimens are shown in Fig. 1.

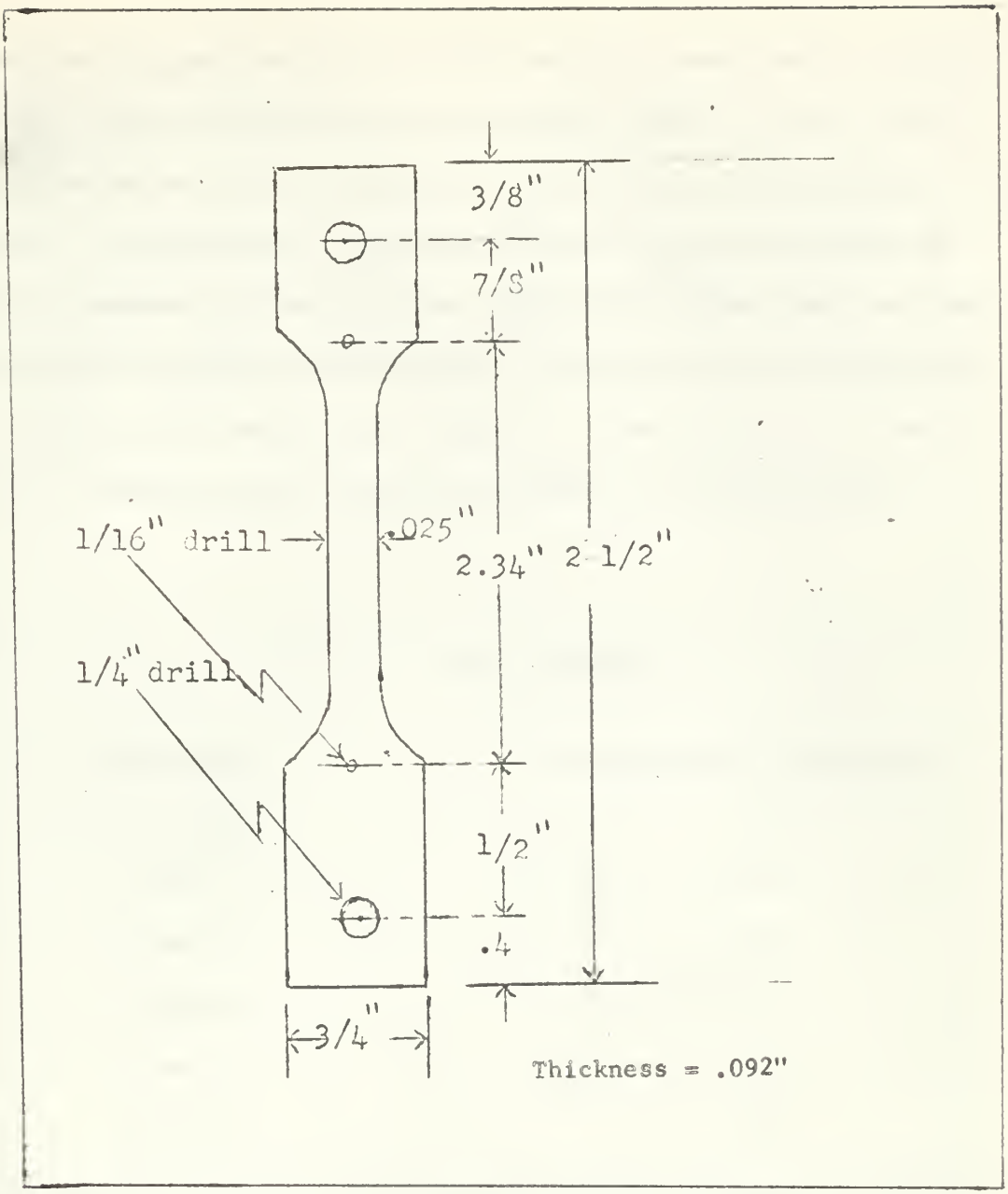


FIG. 1 DIAGRAM OF SPECIMEN WITH ITS DIMENSIONS.

As a stable grain structure is desirable for creep tests, the effect of various elevated temperatures and annealing times on grain growth was investigated by annealing samples at 750°C and 820°C for periods of 1 and 10 hours. Microstructural examination revealed that grain growth was greatly influenced by annealing time. Therefore further tests were conducted at 820°C to determine the period of time in which the major portion of grain growth had occured. Results of these tests are shown in Table II. Microstructure of sample specimens is pictured in Fig. 2.

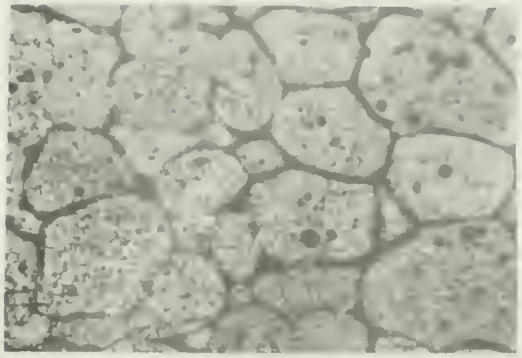
TABLE II - GRAIN SIZE DATA

Time Heated	Grains/Width of Specimen
1/2 hour	18.2
1 hour	14.1
2 hours	13.3
3 hours	12.4
4 hours	11.8
10 hours	9.2

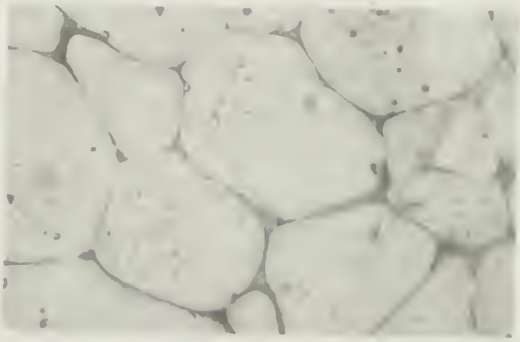
From phase diagram information, Fig. 3, it was decided that creep tests might be conducted at temperatures as high as 750°C. Thus test specimens were annealed at 820°C for a period of 2-1/2 hours. Referring to Fig. 2, it can be seen that there will still be come grain growth during creep tests.

750°C for 1 hour

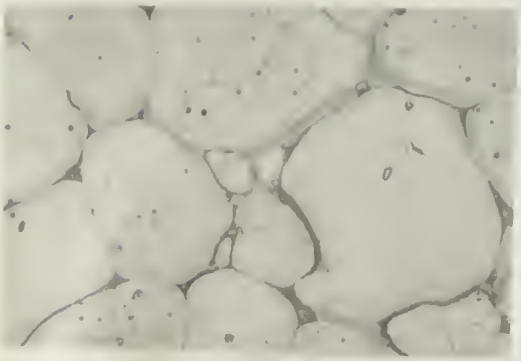
750°C for 10 hours



820°C for 1/2 hour



820°C for 1 hour



820°C for 3 hours

820°C for 10 hours

FIG. 2 EFFECT OF HEAT TREATMENT ON [illegible]

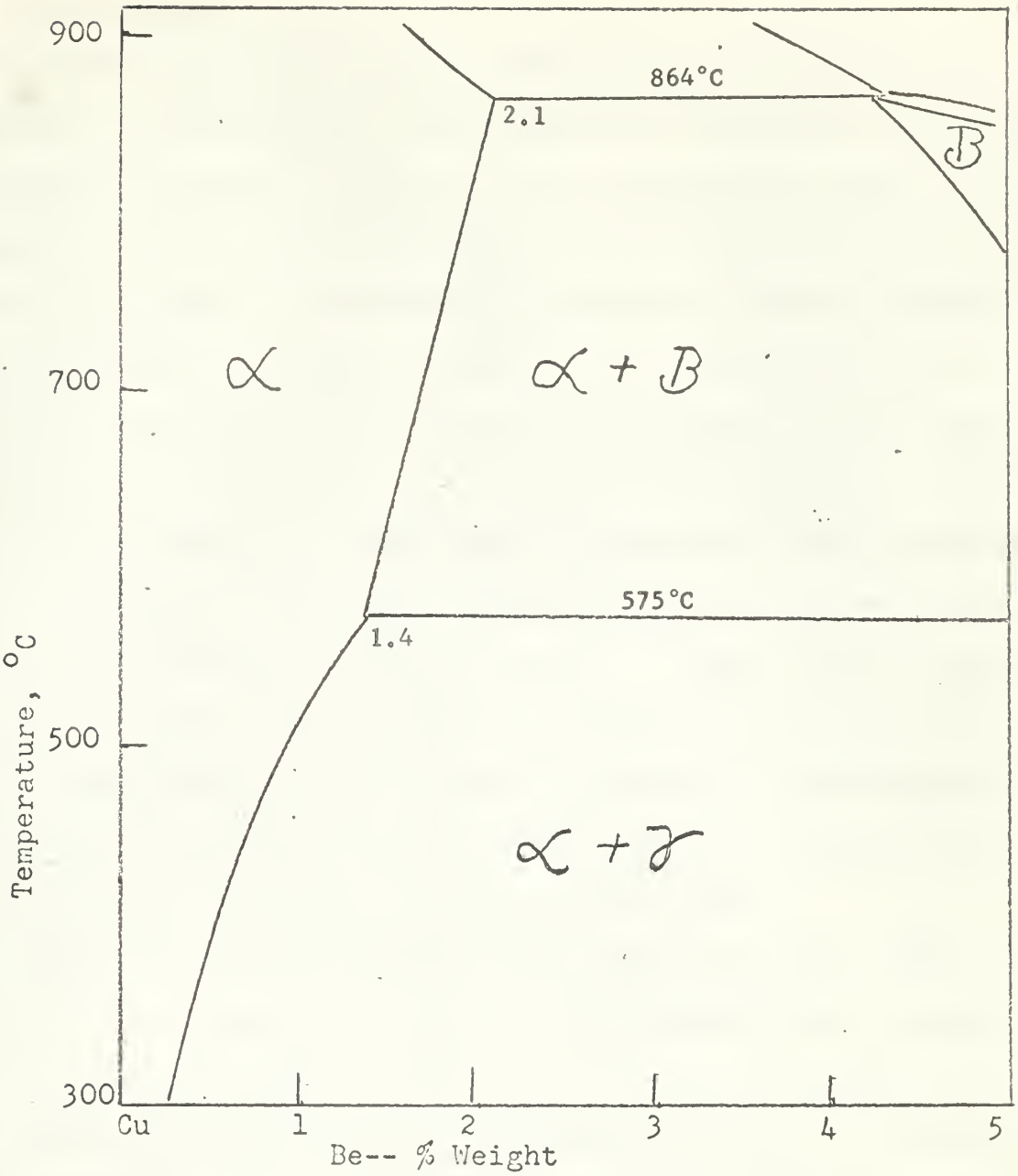


FIG. 3 PHASE DIAGRAM FOR COPPER-BERYLLIUM ALLOY.

2. Test Equipment

A conventional constant load - single lever arm type creep machine was used. The load, applied on the weight pan, is transmitted through the lever arm to the vertical linkages to the extensometer and finally to the specimen. A double universal joint was attached to the swivel block on the lever arm to provide the system with three degrees of freedom and insure uniaxial loading. A turnbuckle was attached to the universal joint to allow for tightening the linkage system with the lever arm in the raised position. Also, as the specimens displayed extensive elongation, it was necessary to tighten the linkages during the experiment to keep the lever arm from hitting the lower stops. The time the load was removed while tightening the lkinages was kept to a minimum -- usually fifteen seconds. Examination of creep curves revealed no irregularities due to these no load periods so that no errors in results is expected. A movable weight on the front part of the lever arm enabled the system to be balanced for no load conditions. A creep test unit is pictured in Fig. 4.

Conventional tube-type nichrome wound furnaces were used. Taps located at close intervals along the winding permitted vertical temperature adjustment by attaching external shunts. This is important so that the temperature along the specimen is maintained constant. The importance of this feature is further emphasized as the specimens elongated almost an inch before fracture resulting in a length of almost 3-1/2 inches over which the temperature must be maintained constant.

For isothermal tests, furnce temperature was controlled by a Leeds and Northrop controller.

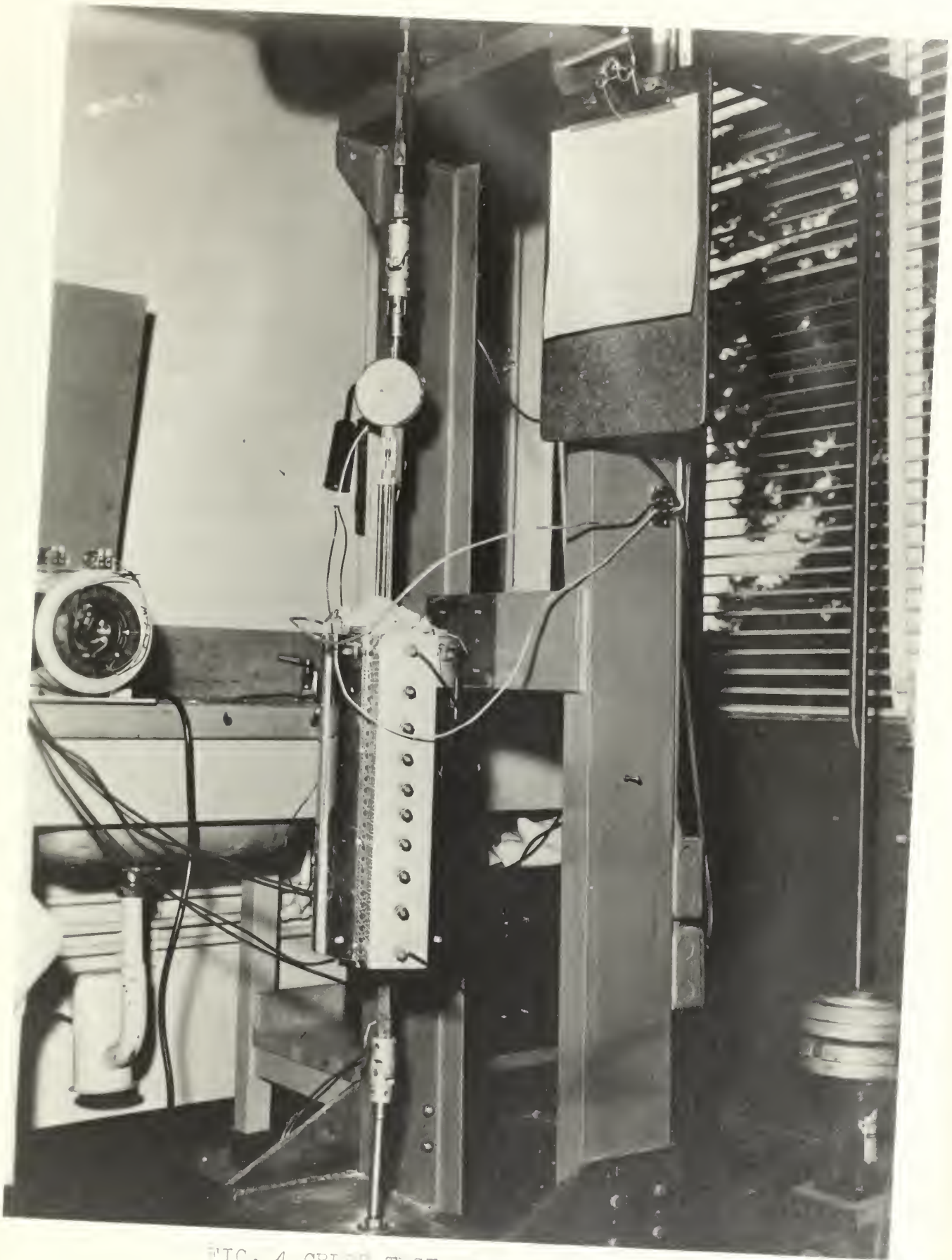


FIG. 4 CRIPP TEST UNIT.

For cyclic tests the temperature was varied by changing the current input to the furnace. This was accomplished by attaching a motor to the shaft of a variac. Motor speed, reduced by a 200,000: 1 reduction gear system, could be varied by changing the armature current. A slotted glass plate was attached to the front of the variac. Two micro-switches based in the slot were actuated by a cam on the variac shaft to change the motor direction. Thus their location in the slot controlled the minimum and maximum current to the furnace and the time for a cycle. As the output of the variac was in series with the main Leeds and Northrop controller, the maximum temperature of the furnace could also be controlled there. Trial and error adjustment of the various components of this system insured that the cycle would repeat itself. A cycle pattern in the general shape of a sine wave resulted. The temperature cycle was recorded by a Leeds and Northrop recorder, type G.

A schematic diagram of the control system is shown in Fig. 5. The entire system including the recorder is pictured in Fig. 6.

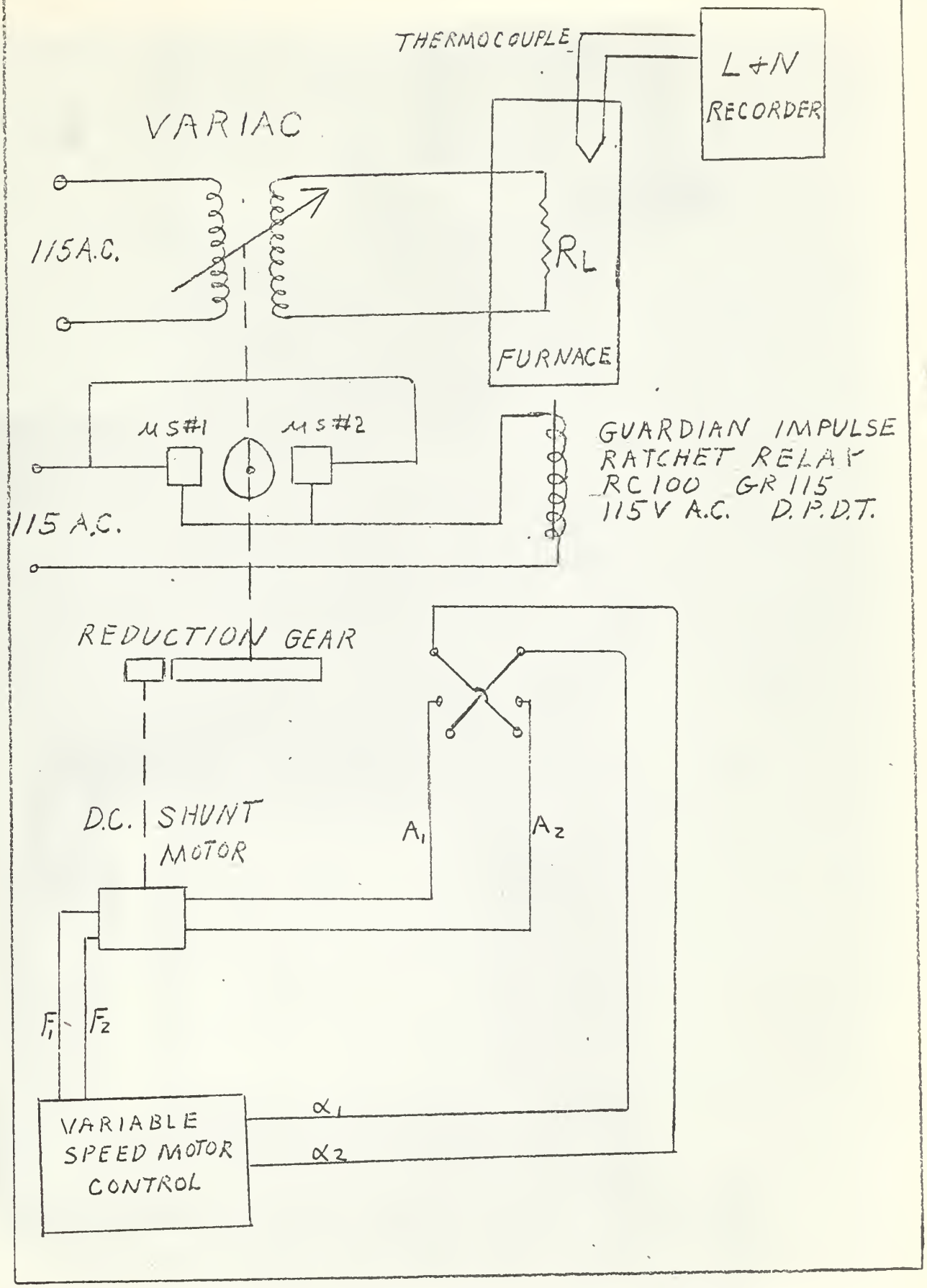


FIG. 5 SCHEMATIC DIAGRAM, FURNACE CONTROL SYSTEM FOR CYCLIC TEMPERATURE TESTS.

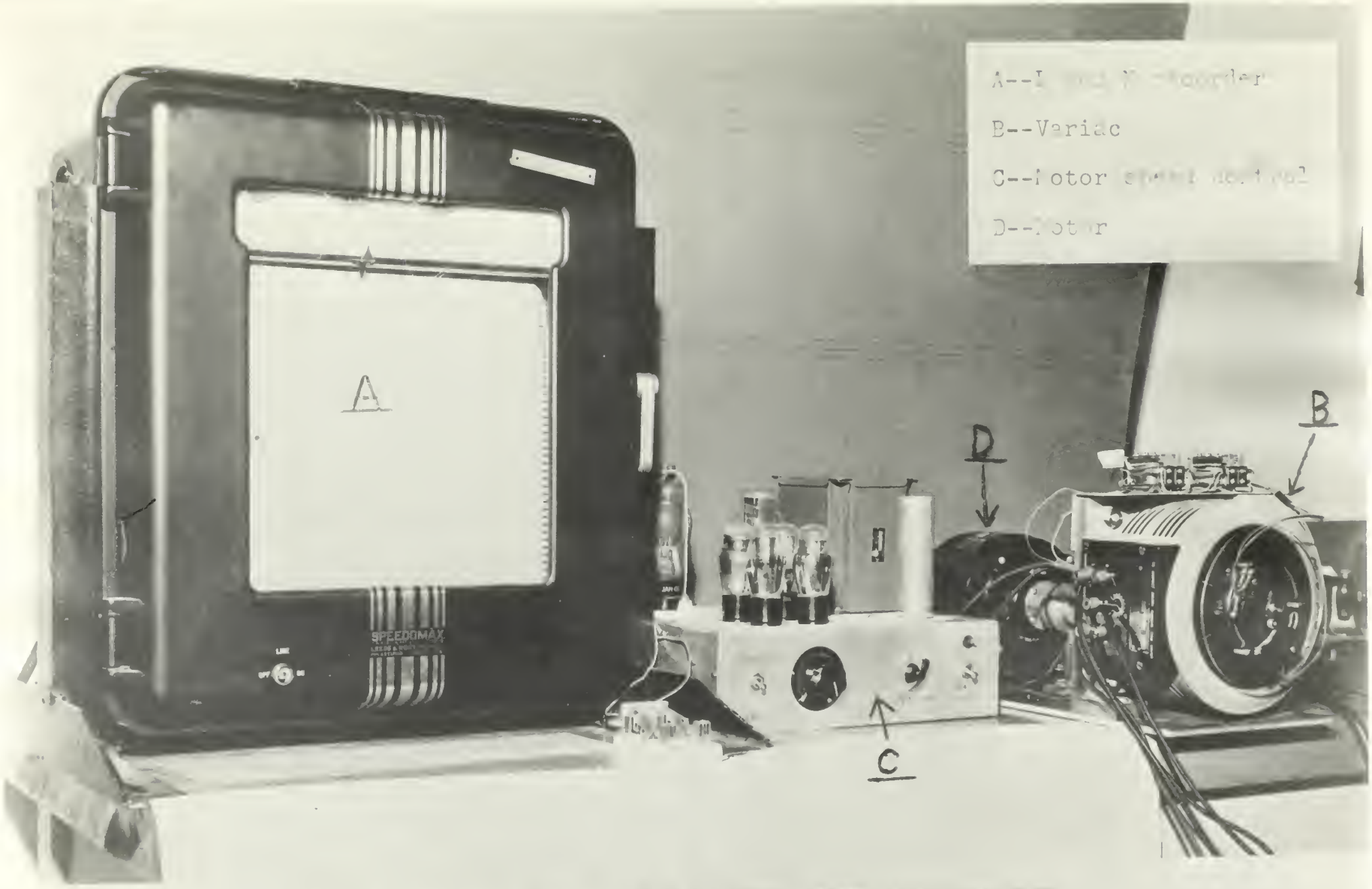

A--X and Y - Recorder
B--Variac
C--Motor speed control
D--Motor
A
B
C
D
SPEEDOMAX

FIG. 4. [illegible]

3. Experimental Technique

The thickness and width of each specimen was measured at various intervals along its reduced section. The minimum cross sectional area was computed from these readings. The load required to produce the desired initial stress was then computed by equation (3).

$$L = \frac{\sigma \quad A_{min.}}{\text{Lever Arm}} \qquad (3)$$

This load was measured to $\pm$ .002 lb.

The specimen was then attached to the extensometer and two chromel-alumel thermocouples were attached to the surface of the specimen by thin glass wire. The extensometer was then lowered into the furnace and connected to the upper and lower pulling tabs. The loading pan was then held in its upward position by a hydraulic jack. This placed the lever arm in its raised position. The load was then placed on the loading pan and all linkages tightened. Asbestos packing was placed around the furnace openings. After a period of approximately two hours, temperature conditions were steady; and the tests were commenced by lowering the jack, permitting the load to hang free. Changes in specimen length to the nearest one ten thousandth of an inch and time to the nearest second were recorded.

Cyclic tests were conducted in a like manner except that specimen temperature was recorded on a Leeds and Northrop Speedomax, type G. Temperature was also taken at frequent intervals with a potentiometer to check the accuracy of the recorder. Any recorder errors were thus removed in the final temperature versus time plot.

EXPERIMENTAL RESULTS AND DISCUSSION

Data from the isothermal creep tests is summarized in Table III, Appendix I. Creep curves of stress versus time for initial stresses of 2,500 psi., 5,000 psi., and 10,000 psi. are also shown in Appendix I, Fig. 11, Fig. 12, and Fig. 13. Fig. 14 shows fast creep rate curves.

Minimum creep rate and time to rupture are plotted versus reciprocal temperature for isothermal tests in Fig. 15 and Fig. 16, Appendix III. From these curves average values of activation energy were calculated.

$750° C > T > 550°C$, Q = 80,000 cal/mole

$500°C > T > 450°C$, Q = 30,000 cal/mole

$550°C > T > 500°C$, Q undetermined

Data for cyclic temperature tests is summarized in Appendix II, Table IV. Temperature and corresponding $e^{-Q/RT}$ cycles for variable heating tests are shown diagrammtically in Fig. 17, Fig. 18, and Fig. 19, Appendix IV. Referring to the Be - Cu phase diagram, Fig. 3, it can be seen that these temperature cycles fall in the $\alpha + \gamma$, $\alpha + \beta \rightleftarrows \alpha + \gamma$ and $\alpha + \beta$ regions respectively.

Results in $\alpha + \beta$ phase. Tests in this phase region were conducted at an initial stress of 2500 psi. Fig. 7 displays isothermal and cyclic temperature (613°C to 650°C) strain results versus the temperature-compensated time parameter, θ. There appears to be no detectable increase in creep rate due to cycling the temperature. Isothermal creep curves give a good approximation of the cyclic temperature creep curves.

Results over the Eutectoid Region. Tests in this region were conducted at 5,000 psi. Fig. 8 shows isothermal and cyclic temperature (556°C to 613°C) strain versus the temperature-compensated time parameter, θ. There appears

to be a slight increase in the creep rate and decrease in time to rupture for the cyclic temperature test as compared to the isothermal tests. However, isothermal tests results still give a fair approximation of cyclic temperature results.

Results in $\alpha + \gamma$ phase. These tests were conducted at 10,000 psi. Fig. 9 shows isothermal and cyclic temperature (427°C to 468°C) creep strain versus temperature-compensated parameter, θ. Isothermal creep curves for 520°C and 533°C tests could not be plotted as an accurate value of activation energy could not be determined for these temperatures (see Appendix III). There does not appear to be any appreciable increase in strain rate due to cyclic temperature conditions. Isothermal creep curves give an excellent prediction for the cyclic temperature creep curve.

It is noted that in the region where the eutectoid reaction, $\beta \rightleftarrows \alpha + \gamma$, is occuring that the creep rate is increased by cycling the temperature. However in phase regions where only slight solubility changes are occuring there is no noticeable increase in creep rate for cyclic tests. This is not unexpected as microstructural changes associated with phase changes are greater than those occuring during solubility changes. Fig. 10 shows the microstructure of the copper-beryllium alloy when quenched in brine from 550°C, 650°C and 750°C. Only a slight change in the structure is noted between temperatures 650° and 750°C. Both these temperatures are in the $\alpha + \beta$ phase region. But the microstructure at 550°C is very much different due to the presence of the γ phase, resulting from the eutectoid reaction, $\beta \rightleftarrows \alpha + \gamma$. Thus the theory that microstructural changes tend to augment creep rate is supported. However the increase is not so much as to make isothermal tests of no use in predicting cyclic temperature creep curves.

It is noted that constant temperature tests do not exactly coincide on the strain versus θ graphs. This is believed due to five reasons. First, any deformities in the specimens would result in increasing creep rates. Second, these tests were all of a short time nature so that any jerking upon application of load would be magnified. Third, any error in temperature recorded would be magnified exponentially ($e^{-Q/RT}$) in calculating θ. It was noted that the fast creep rate of these tests caused small temperature fluctuations by altering the asbestos insulating packing around the extensometer rods. Fourth, the Be-Cu alloy was very susceptible to grain growth at elevated temperatures. Thus the microstructure changed as the creep tests progressed. Fifth, there may be slight changes in activation energy in regions where it was assumed to be constant. Such error would not change the shape of the creep curve but would shift its position along the θ axis.

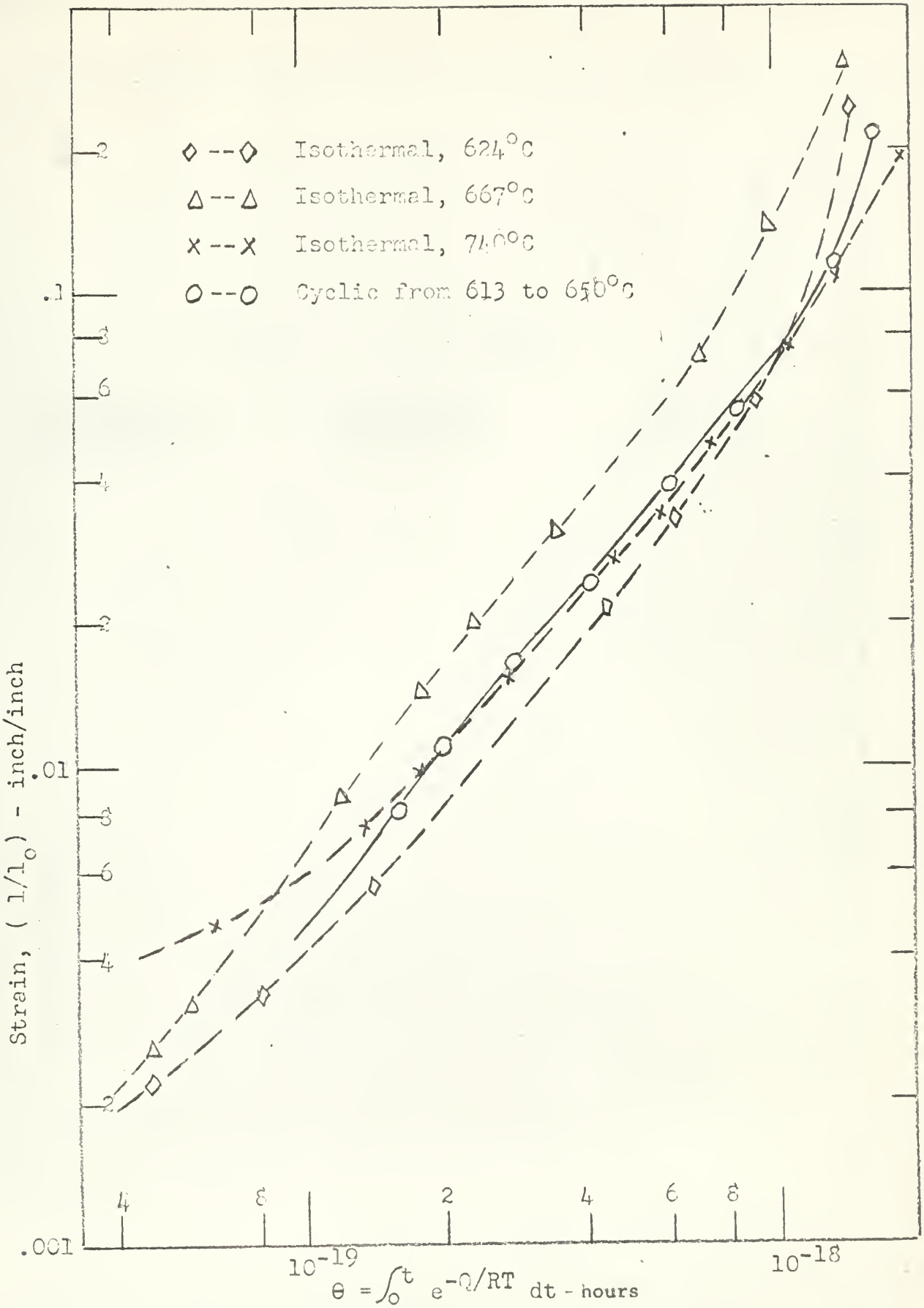


FIG. 7 ISOTHERMAL AND CYCLIC TEMPERATURE CREEP CURVES FOR AN INITIAL STRESS OF 2500 psi.

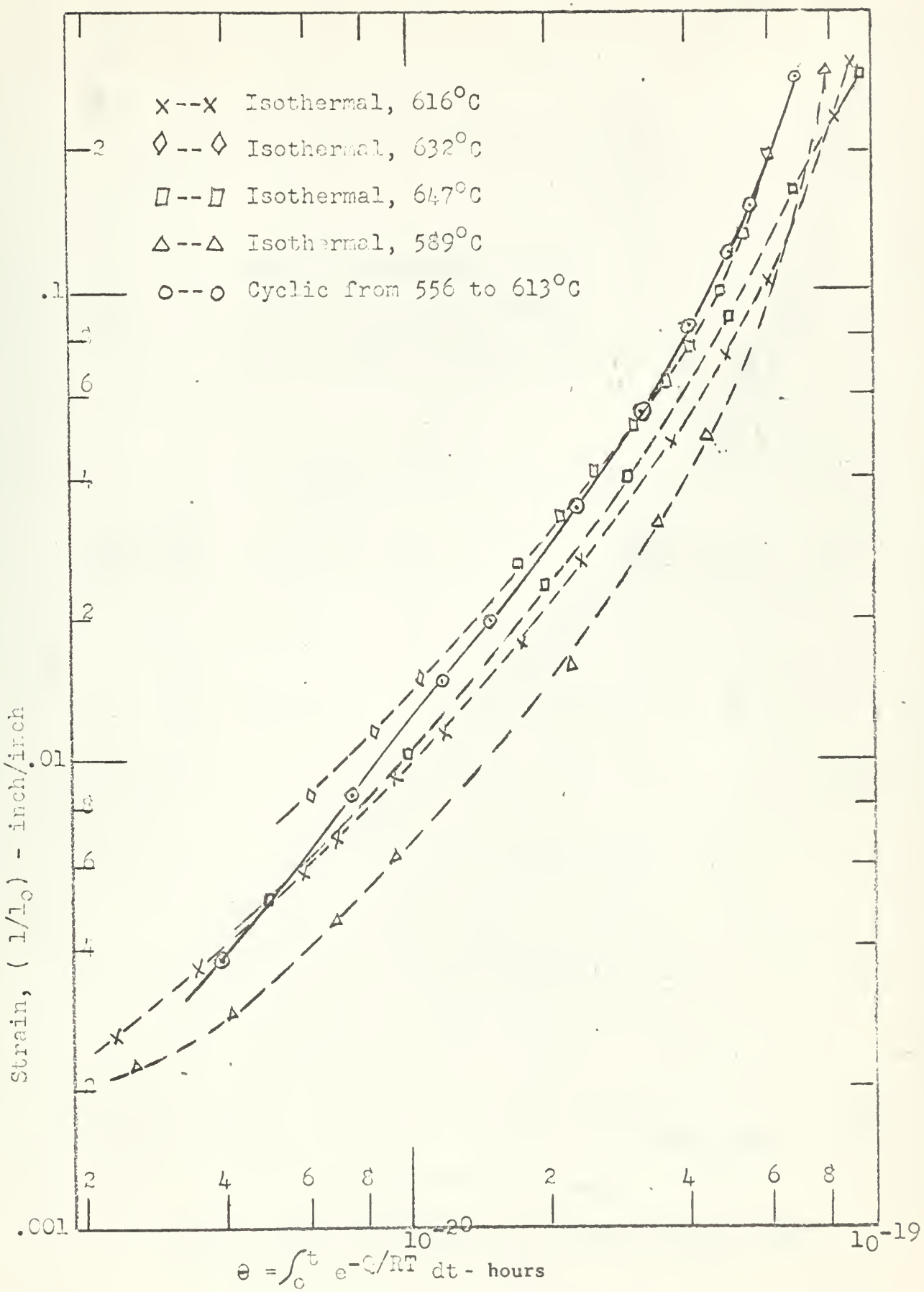


FIG. 8 ISOTHERMAL AND CYCLIC TEMPERATURE CREEP CURVES FOR AN INITIAL STRESS OF 5000 psi.

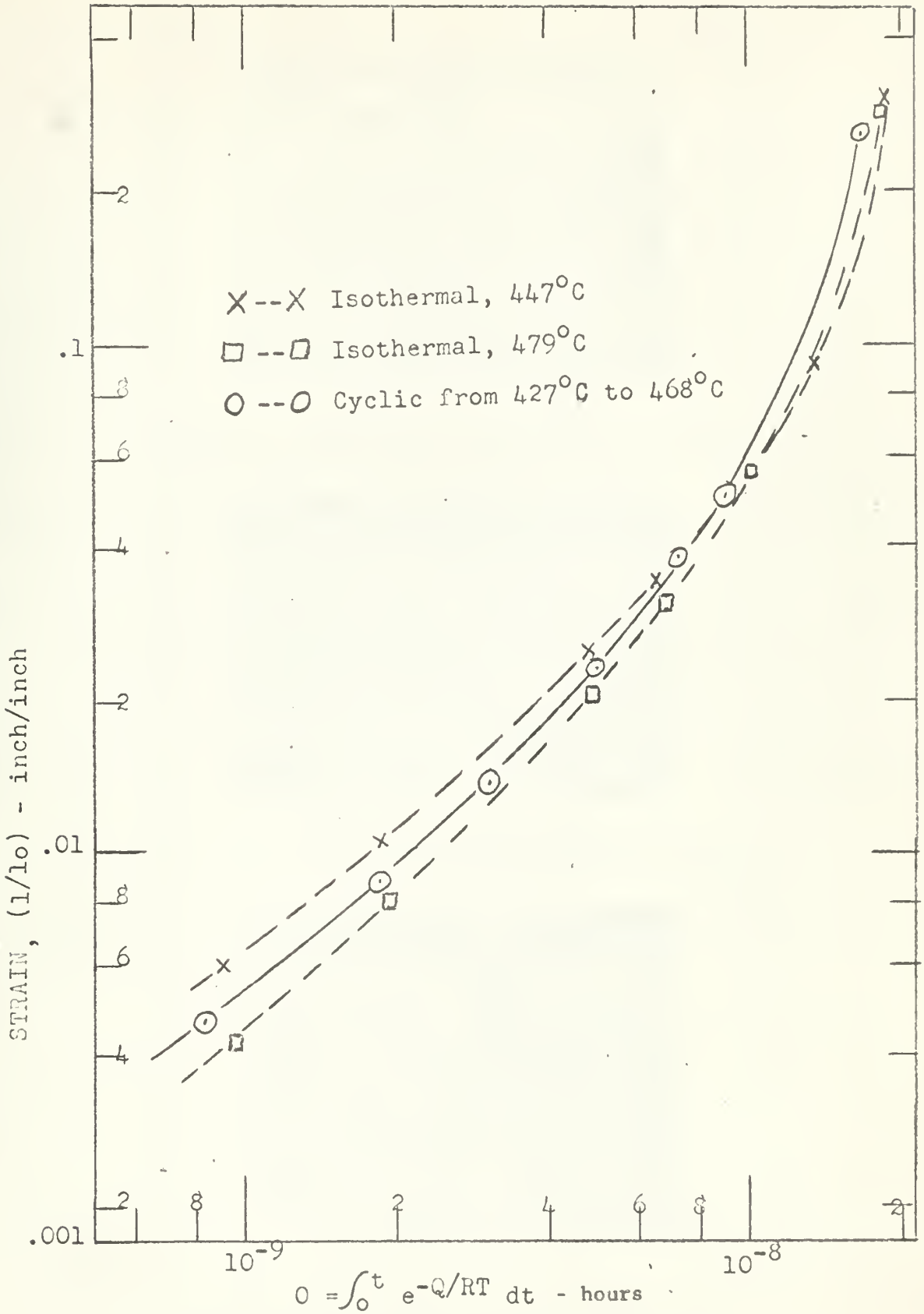
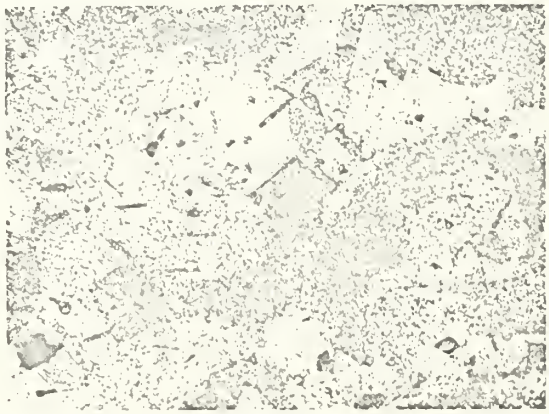


FIG. 9 ISOTHERMAL AND CYCLIC TEMPERATURE CREEP CURVES FOR AN INITIAL STRESS OF 10,000 psi.

Quenched from 750°C.

Quenched from 650°C.



Quenched from 550°C

FIG. 10 PHOTOGRAPH ILLUSTRATING THE CHANGE IN MICROSTRUCTURE OF A CU-BE ALLOY AT VARIOUS TEMPERATURES.

CONCLUSIONS AND RECOMMENDATIONS

The following conclusions are drawn:

1. Prediction of creep curves for a beryllium-copper alloy under constant load-varying temperature conditions can be made with a fair degree of accuracy by the θ parameter method. However the accuracy of predictions are only as accurate as the values of activation energy used to calculate θ. Thus in regions where Q varies, the predicted strain curve may be unreliable.

2. The effect of microstructural changes is to cause an increase in creep rate. But in cases where these changes are small, such as the case where relatively small changes in the proportion of phases present occur, any increase in creep rate is within the experimental accuracy of these tests. However in cases where phase changes occur there is a small increase in creep rate, but not enough to make isothermal predictions for cyclic temperature creep curves valueless for design purposes.

It is recommended that:

(1). Tests be conducted in a particular region under different temperature cycles to determine the effect of cycle period and amplitude on creep rate.

(2). Tests be conducted under a temperature cycle with a large amplitude which extends into regions where Q is not constant, and then attempt to predict the creep curve by combining the θ parameter method for the part of the cycle where Q remains constant with the actual creep curve for the region where Q is varying. This would enable an engineer to predict the creep curve for any cycle by using isothermal data and strain vs. θ data for regions of changing Q.

(3). Slower creep rate tests be conducted thereby increasing the accuracy of results.

ACKNOWLEDGEMENTS

The writer wishes to express his appreciation for the assistance given by his Faculty Advisors, Alfred Goldberg, Associate Professor of Metallurgy, and Peter M. Burke, Assistant Professor of Metallurgy, and to Robert C. Smith, Office of Naval Research Technician, for his assistance in designing and fabricating a variable speed control unit for cyclic temperature control.

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APPENDIX I

ISOTHERMAL CREEP CURVES AND DATA

Conventional creep curves for isothermal tests at constant loads of 2,500 psi., 5,000 psi., and 10,000 psi. are shown in Figures 11, 12, and 13 respectively. Fig. 14 shows the creep curves for faster creep rate tests at the above loads as these curves are only partially displayed on their respective load figures. Engineering strain, change of length in inches divided by initial length in inches, is used for all curves.

Table III gives the initial stress, temperature, time to rupture, minimum creep rate, and values of $e^{-Q/RT}$ for isothermal tests.

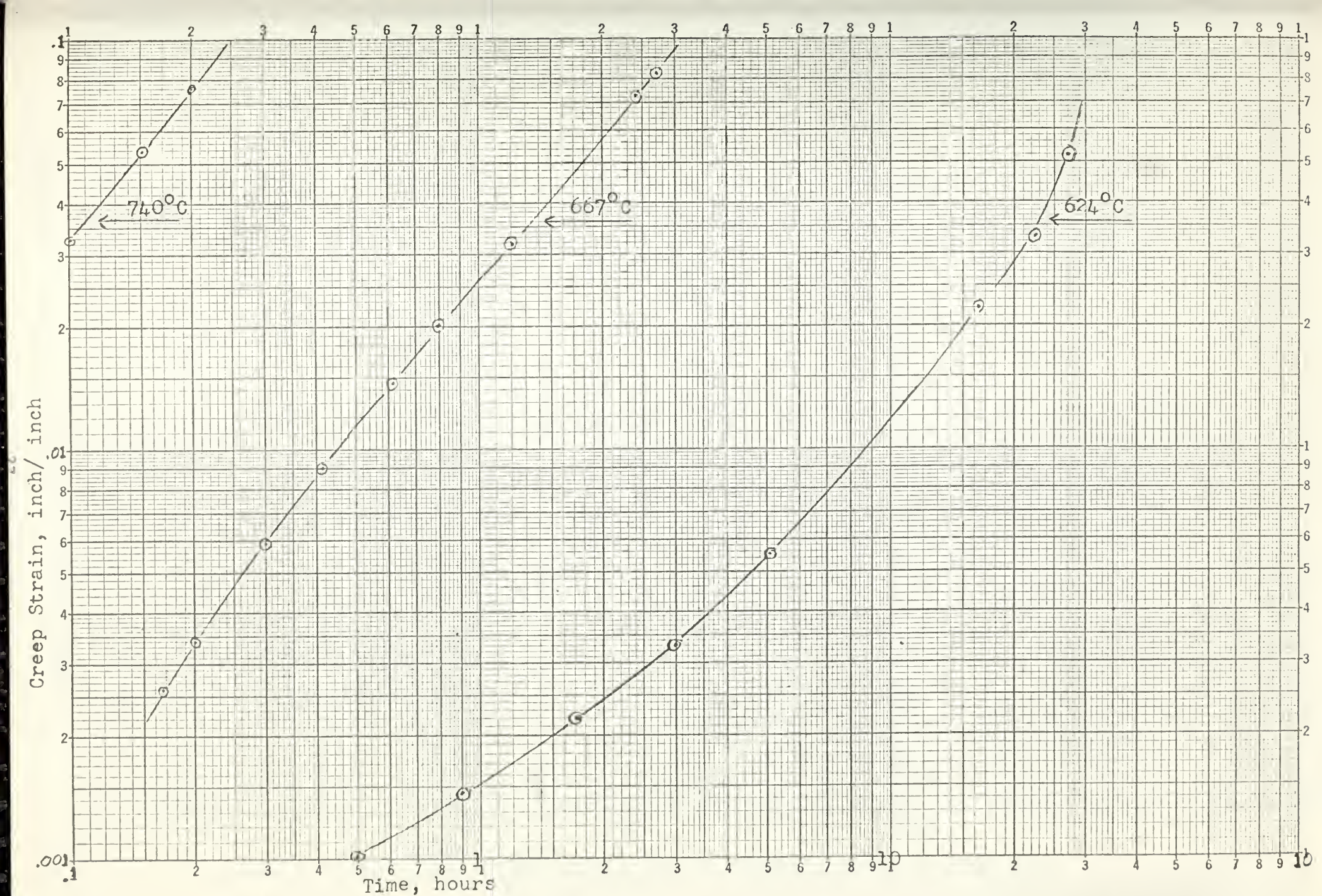

FIG 11 ISOTHERMAL CREEPCURVES FOR AN INITIAL STRESS OF 2500 psi.

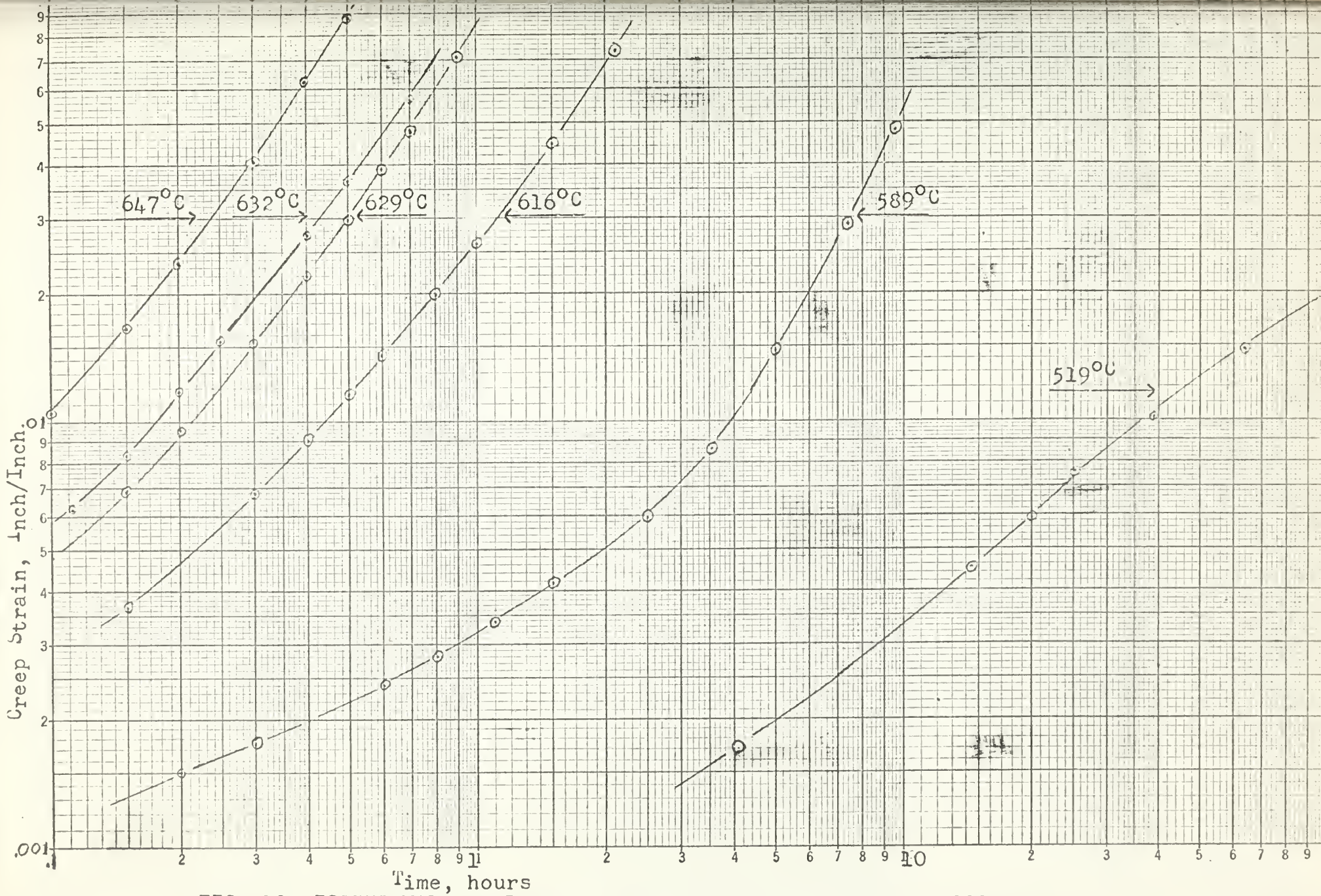

FIG. 12 ISOTHERMAL CREEP CURVES FOR AN INITIAL STRESS OF 5000 psi.

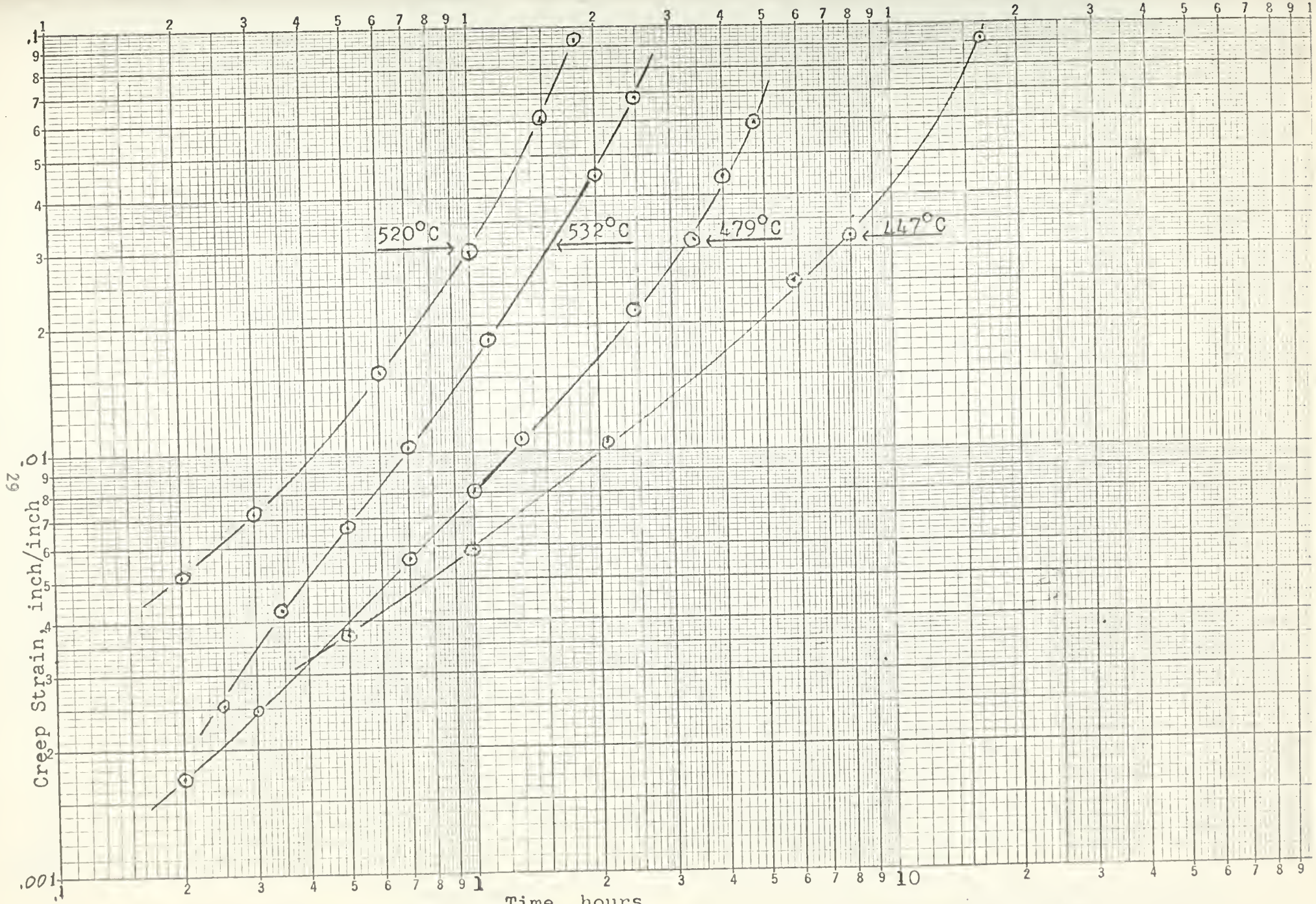

FIG. 13 ISOTHERMAL CREEP CURVES FOR AN INITIAL STRESS OF 10,000 psi.

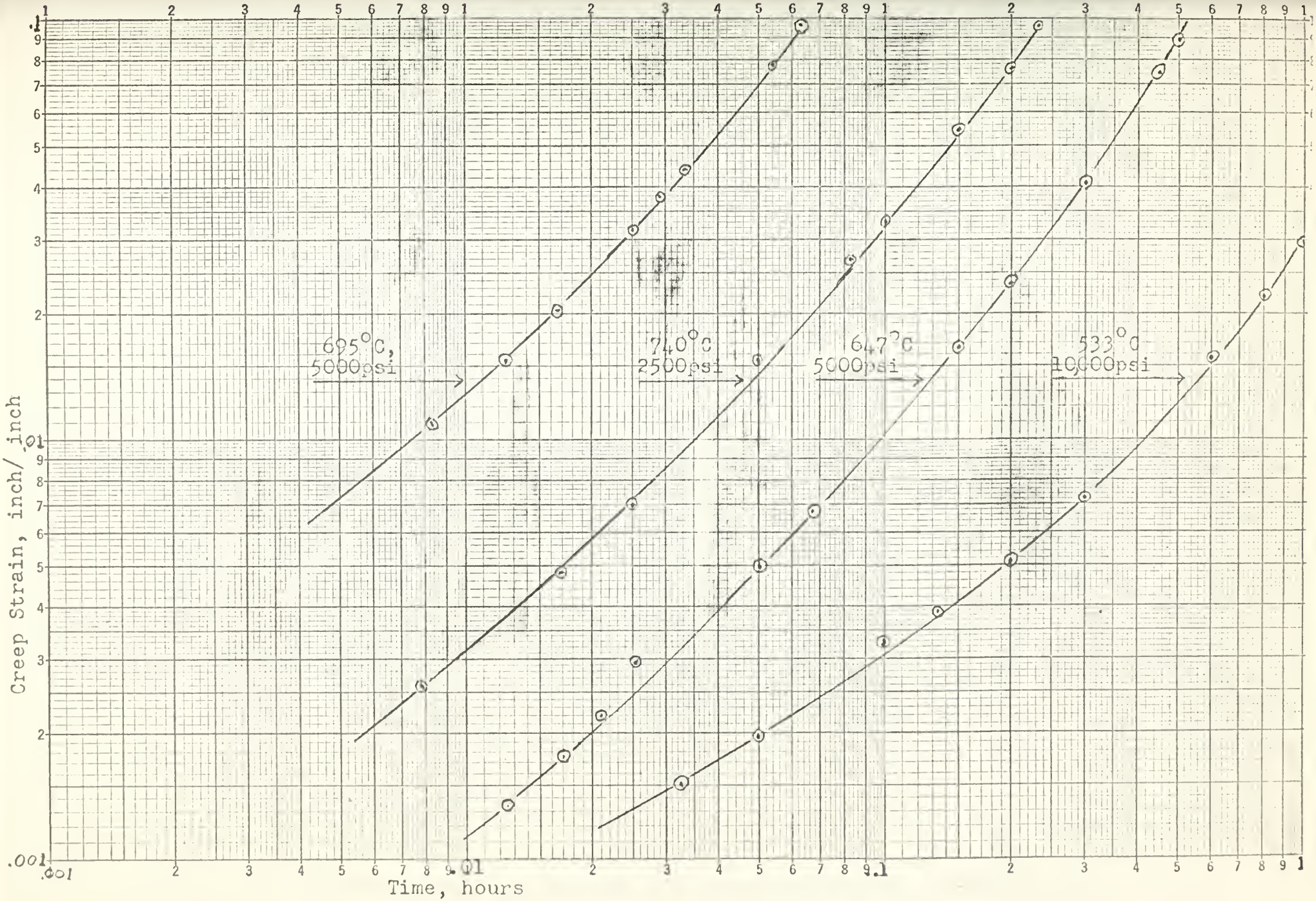

FIG. 14 ISOTHERMAL CREEP CURVES FOR FAST CREEP RATE TESTS AT VARIOUS INITIAL STRESSES.

TABLE III - ISOTHERMAL TEST DATA.

Stress psi	Temp. °C	Temp. °K	Hours to Rupture	Minimum Creep Rate	$e^{-Q/RT}$
2500	740	1013	.401	0.8	$5.4\ 10^{-18}$
2500	667	940	4.9	0.03	$2.84\ 10^{-19}$
2500	624	897	32.6	0.0055	$2.74\ 10^{-20}$
5000	695	968	.139	2.65	$8.57\ 10^{-20}$
5000	647	920	.95*	0.25	$8.4\ 10^{-19}$
5000	632	905	1.362	0.118	$4.1\ 10^{-20}$
5000	629	902	1.32*	0.11	$3.78\ 10^{-20}$
5000	616	889	3.846	0.048	$2.3\ 10^{-20}$
5000	589	862	14.95	0.008	$4.9\ 10^{-21}$
5000	582	855	16.5*	---	$3.3\ 10^{-21}$
5000	519	792	----	0.0003	----- ---
10000	533	806	2.03	0.045	----- ---
10000	520	793	3.58	0.02	----- ---
10000	479	752	8.0	0.008	1.96×10^{-9}
10000	447	720	21.74	0.0045	8.1×10^{-10}

* Jerked upon loading

APPENDIX II

TABLE IV. CYCLIC TEMPERATURE TEST DATA.

Phase Cycled In	Temperature °C	Time to Rupture
$\alpha + \beta$	427-468	17.82 hours
Eutectoid reaction	556-613	9.87 hours
$\alpha + \delta$	613-650	18.35 hours

APPENDIX III

CALCULATION OF ACTIVATION ENERGY

The activation energy for creep was found by two methods. The average value was used for calculation of the temperature - compensated time parameter as outlined below.

From the relationships (4), (5), and (6) it follows that

$$\epsilon = f(\theta, \sigma_c) \tag{4}$$

$$\theta = t\, e^{-Q/RT} \tag{5}$$

σ_c = initial stress at constant load (6)

the creep rate is given by equation (7) as the initial stress

$$\dot{\epsilon} = \frac{\partial f}{\partial \theta}\frac{\partial \theta}{\partial t} = f'(\theta)\, e^{-Q/RT} \tag{7}$$

is independent of time.[7] Furthermore experimental results have shown that when $\dot{\epsilon}$ as taken as minimum creep rate, the following expression is obtained:

$$\dot{\epsilon}_{MIN} = C\, e^{-Q/RT} \tag{8}$$

Thus if the log. of minimum creep rate is plotted versus the reciprocal creep temperature at the same load in a region where Q remains constant, a straight line will result.[9, 10] The negative slope of this line would equal the activation energy as shown in equation (9).

$$-Q = \frac{R \ln (\dot{\epsilon}_1/\dot{\epsilon}_2)}{\left\{\frac{1}{T_2} - \frac{1}{T_1}\right\}} \tag{9}$$

where $\dot{\epsilon}_1$ and $\dot{\epsilon}_2$ are minimum creep rates.

Fig. 15 shows the minimum creep rate versus reciprocal creep temperature curves for 10,000 psi., 5,000 psi., and 2,500 psi. initial stresses. From these curves the activation energy for creep was calculated to equal

a constant value of 82,000 cal/mole between temperatures of 550°C to 750°C. Below 550°C the value falls off rapidly to a value of 28,000 cal/mole at 500°C.

The second method for obtaining activation energy is from time to rupture data. If it is assumed that the strain damage leading to rupture at a given load is dependent only upon the strain to rupture, the value of θ at rupture should be the same for all tests. Sherby and Dorn have proven the validity of this by experimental data.[10], [11] Thus equation (10) follows. The plot of $\log_e t_R$ versus $1/T$ for constant loads will be a straight line where Q is constant as given by the relationship (11). The slope of this curve

$$\theta = t\, e^{-Q/RT} = F(\sigma_c) \tag{10}$$

$$Q = \frac{R \ln (t_{R1}/t_{R2})}{\left(\frac{1}{T_1} - \frac{1}{T_2}\right)} \tag{11}$$

will be equal to the activation energy.

A plot of time to rupture versus reciprocal creep temperature is shown in Fig. 16. Activation energy is calculated to be 79,000 cal/mole between temperatures of 750°C and 550°C. Below 550°C the value falls off rapidly to 31,000 cal/mole at 500°C.

The above two methods led to average values of Q equal to 80,000 cal/mole between temperatures of 550°C to 750°C and 30,000 cal/mole below 500°C. Between 500°C and 550°C the activation energy changes too rapidly to obtain an accurate value. In other words the slope of the curves in Fig. 15 and Fig. 16 between 500°C to 550°C is inaccurate. Sherby, Lytton, and Dorn have shown the inaccuracies of experimental values for activation energy at points where there is a rapid change occuring.[10]

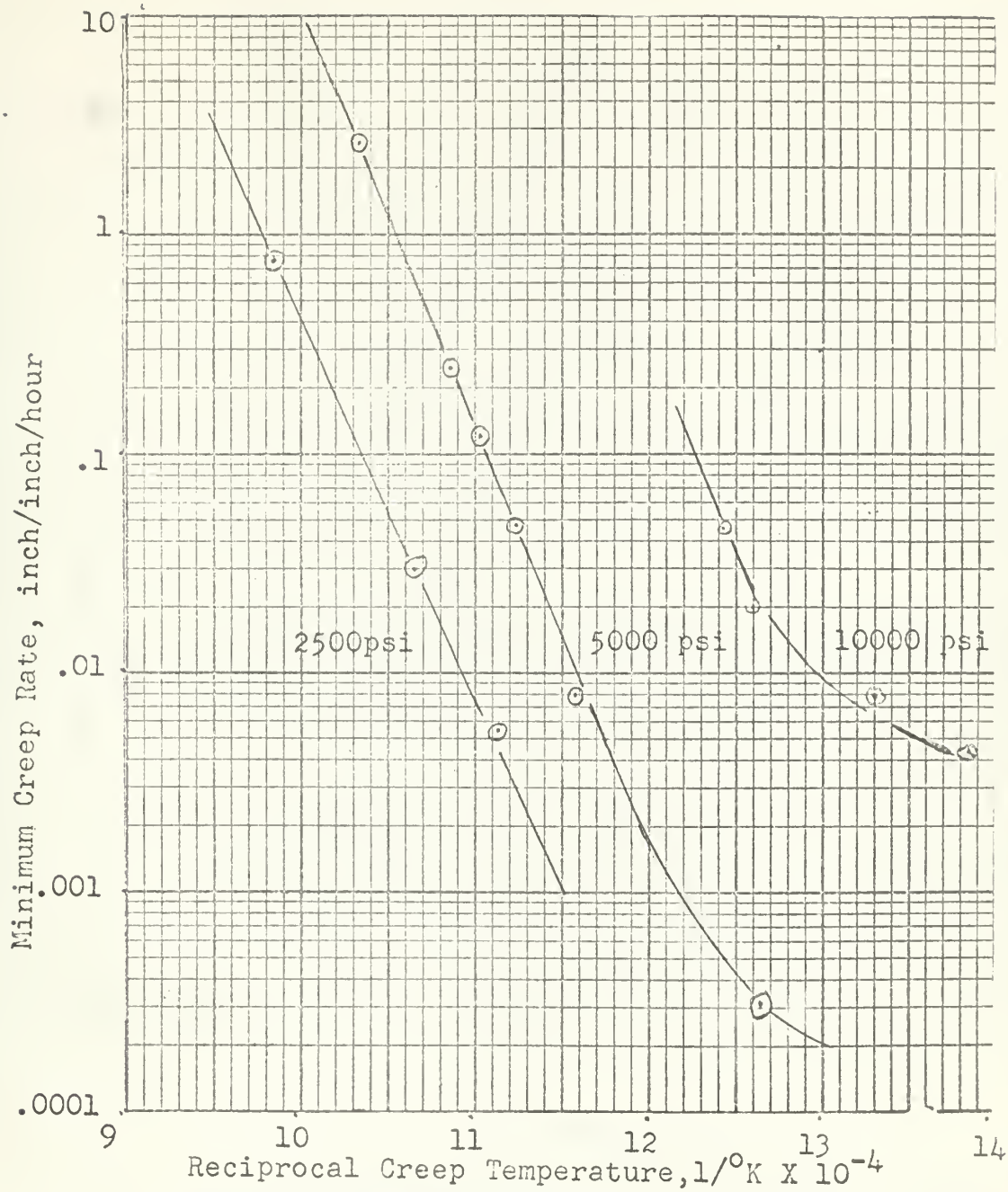

FIG. 15 MINIMUM CREEP RATE VS. RECIPROCAL CREEP TEMPERATURE CURVES.

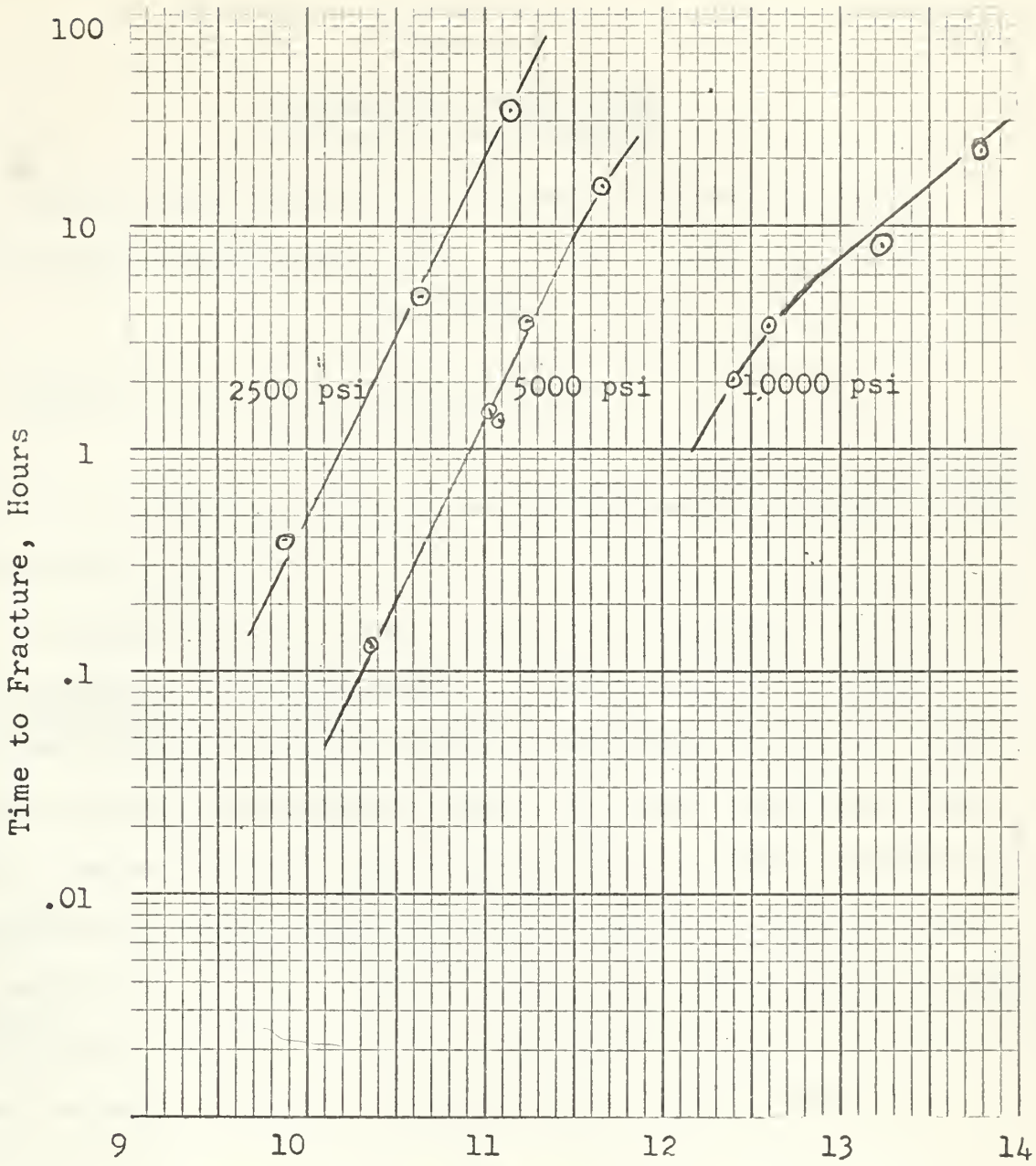

FIG. 16 TIME TO RUPTURE VS. RECIPROCAL CREEP TEMPERATURE CURVES.

APPENDIX IV

CALCULATION OF TEMPERATURE -- COMPENSATED TIME PARAMETER, θ.

The temperature-compensated time parameter, θ, can be evaluated from the following expression:

$$\theta = \int_o^t e^{-Q/RT} dt \tag{8}$$

For isothermal tests this expression becomes:

$$\theta = t\, e^{-Q/RT} \tag{9}$$

Thus for an isothermal test the value of θ is equal to a constant ($e^{-Q/RT}$) multiplied by time.

For varying temperature tests θ was found by graphical solution. Fig. 17, 18, and 19 show the temperature cycles and $e^{-Q/RT}$ cycles of each test. Values of $e^{-Q/RT}$ are computed mathematically for the corresponding points on the temperature curve. The entire temperature cycle is not shown as the cycle repeated itself after the first cycle which was altered as the packing around the furnace openings was slightly altered upon application of load. From (8) it can be seen that the area under the $e^{-Q/RT}$ versus time curve at any time will be equal to the value of θ for that point. Values of Q for each isothermal test is found in Table III.

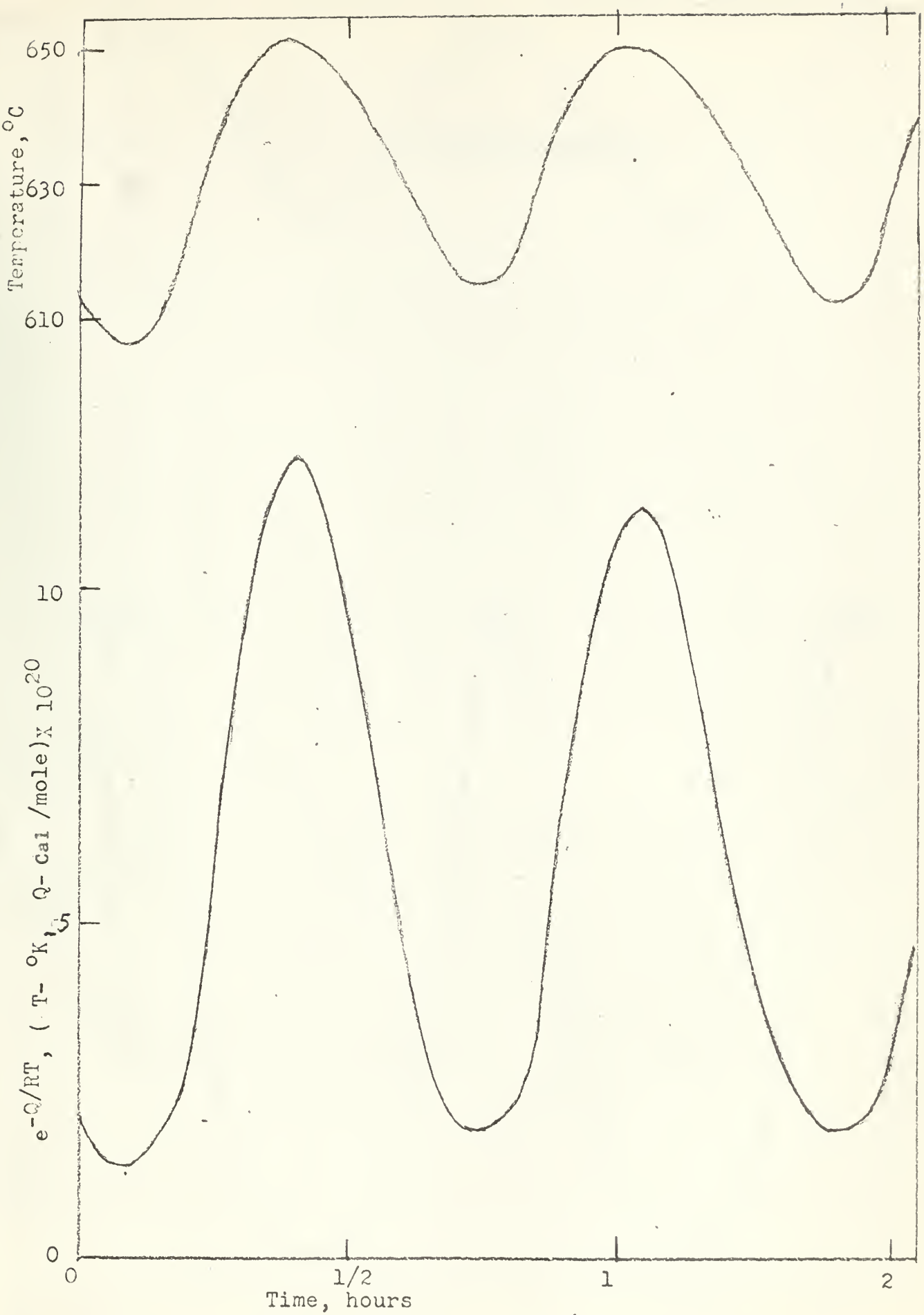

FIG. 17 TEMPERATURE AND CORRESPONDING $e^{-Q/RT}$ CYCLES FOR THE 2500psi. INITIAL STRESS CYCLIC TEMPERATURE TEST.

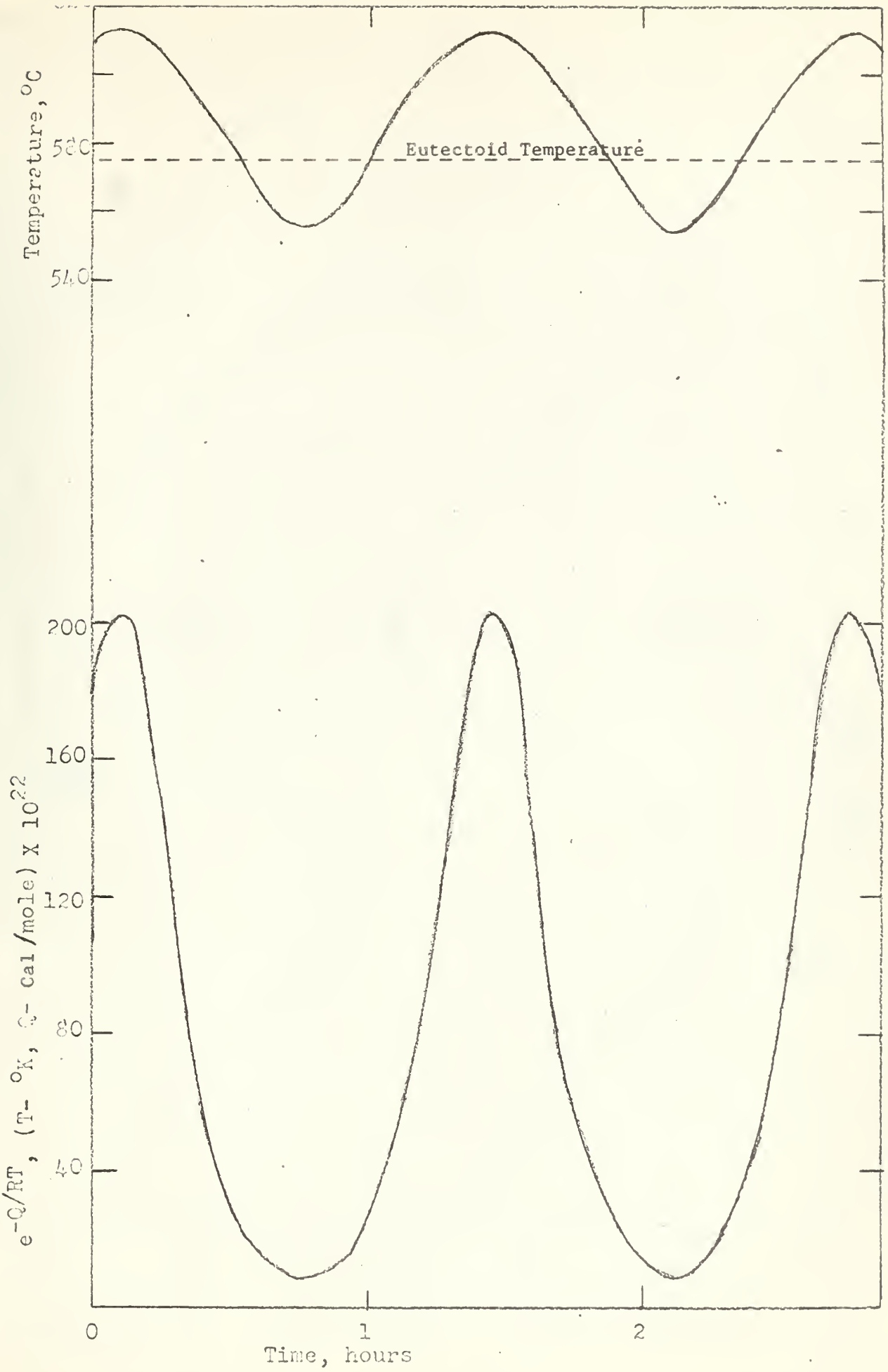

FIG. 18 TEMPERATURE CYCLE AND CORRESPONDING $e^{-Q/RT}$ CYCLE FOR THE 5000 psi. INITIAL STRESS CYCLIC TEST.

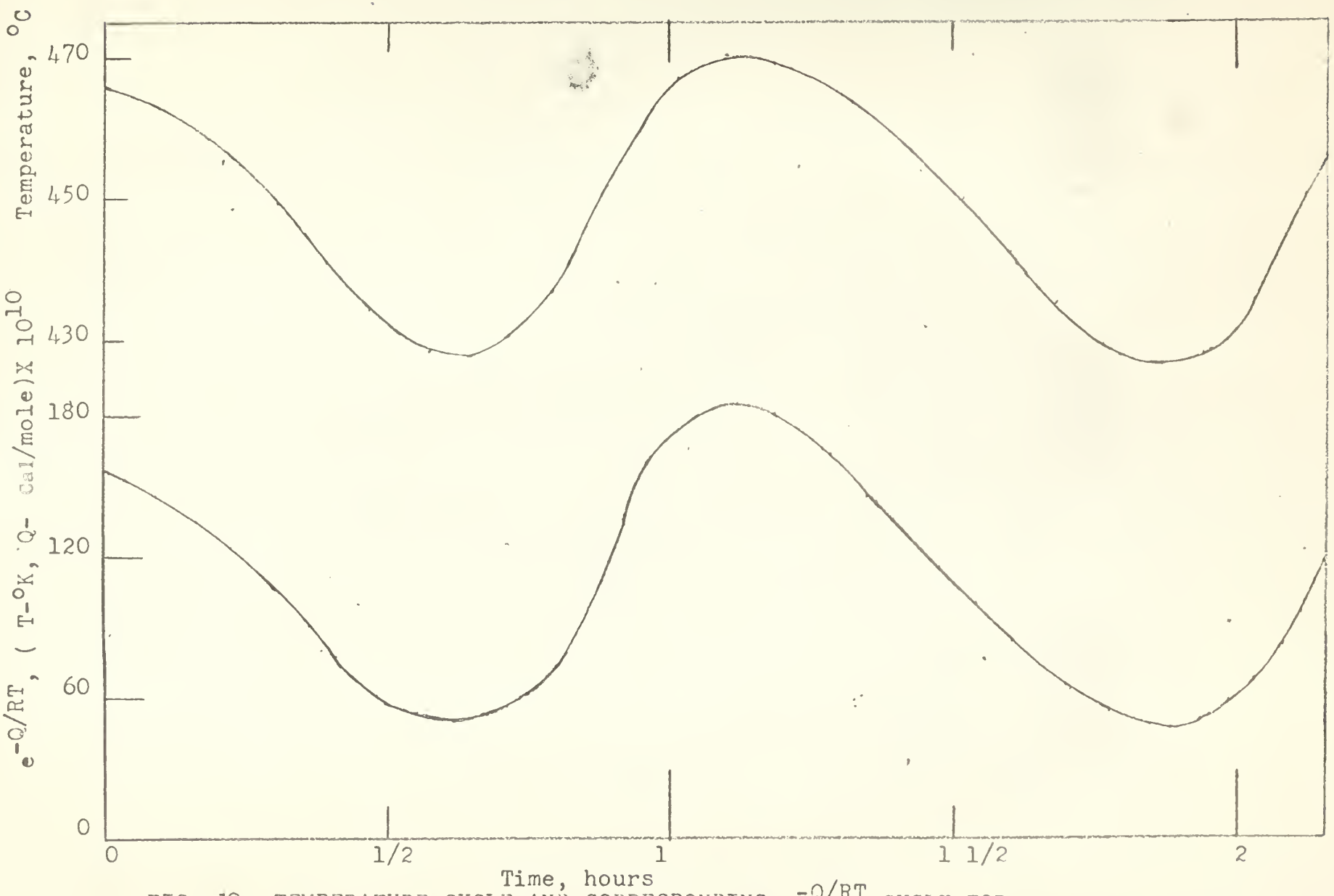

FIG. 19 TEMPERATURE CYCLE AND CORRESPONDING $e^{-Q/RT}$ CYCLE FOR 10,000psi. INITIAL STRESS CYCLIC TEST.