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August 1967

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ABSTRACT

The effect of changes in temperature and stress on the creep behavior of Fe - 4% Si was investigated over the temperature range from 650° to 1175°K. The shear strain rate, $\dot{\gamma}$, obeys the relationship

$$\dot{\gamma} = K \frac{\tau^{5.75}}{TG^{4.75}} \exp\left(-\frac{g}{RT}\right)$$

where K is a constant, τ the applied shear stress, G the shear modulus, g the free activation energy for diffusion, R the gas constant and T is the absolute temperature. The results strongly suggest that the creep in this alloy is controlled by a dislocation climb mechanism.

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I. INTRODUCTION

In a recent investigation, Ishida, Cheng and Dorn[1] observed that the apparent activation energy for creep of α-Fe from 480° to 775°K corresponded to that for self-diffusion. Over the magnetic transformation range (775° to 1045°K), the apparent activation energy for creep increased steeply with increasing temperature in agreement with the known decrease in the free activation energy for self-diffusion. Passing through the Curie temperature, the apparent activation energy decreased abruptly following which it then increased mildly as the temperature further increased.

Inasmuch as the apparent activation energy for creep of α-Fe below the Curie transformation range of temperatures appeared to decrease with increasing values of the stress, the data were analyzed in terms of a model based on the nonconservative motion of jogged screw dislocations. But since the observed creep rate was much higher than that which could be accounted for on the basis of usual models, the authors found it necessary to assume that pipe diffusion became significant over the lower temperature range. Furthermore, the abnormally high activation energy for creep above the Curie temperature was attributed to grain boundary migration which became prevalent in this temperature range.

It was thought that a number of the interesting issues that arose in the creep of α-Fe might be resolved by investigating the creep of Fe - 4% Si alloy: This alloy has about the same Curie range and therefore should give creep results that closely parallel those for α-Fe and in this way provide comparable data for analysis of creep mechanisms.

Furthermore, it can be annealed at higher temperatures which could provide some stabilization against grain boundary migration. In addition, it might serve to illustrate the possible effects of short range ordering on creep.

II. MATERIALS AND TECHNIQUES

The material used in this investigation was supplied by the Westinghouse Electric Corporation having the following composition by weight percent: Fe - 96.02, Si - 3.92, C - 0.014, N - 0.001, O - 0.07, S - 0.002 and P < 0.002. The as received 3/8 in x 3/4 in bars were rolled to 0.200 in thickness at 473°K to a final thickness of 0.063 in. Flat tensile specimens were machined from the sheets, having a gage section 0.250 in wide and 1.70 in long. After recrystallization at 1248°K under argon for 10 minutes, the specimens exhibited a uniform equiaxed grain size of ASTM No. 3.

Tests were conducted in machines fitted with Andrade-Chalmers lever arms so contoured as to maintain a constant stress within ± 0.2% of the reported values. Strains were measured by a linear differential transformer having a sensitivity of $\pm 5 \times 10^{-5}$. During tests the specimens were contained in an argon-filled chamber which was immersed in a temperature-controlled molten tin bath. The specimen temperature was determined by means of thermocouples attached directly to the gage section and was maintained to within ± 1°K of the reported values. For activation-energy determinations, rapid changes in temperature were obtained within 30 seconds by the direct self-resistance heating of the specimen, and maintained to ± 2°K of the reported values. Activation energies were

calculated from the instantaneous creep rates just preceeding and immediately following abrupt changes in temperature of about 12°K.

Since the Fe - Si alloy exhibits long range or short range order,[2-9] depending on the composition, x-ray analysis of this material was done using filtered Cobalt radiation. No superlattice lines were apparent and it was therefore concluded that only short range order was present.

III. EXPERIMENTAL RESULTS

Creep controlled by a single thermally activated process, may frequently be represented by the relationship

$$\dot{\gamma} = A\, e^{-\frac{g}{RT}} \tag{1}$$

where $\dot{\gamma}$ is the shear strain rate, A is a constant, g is the free energy of activation, R is the gas constant and T is the absolute temperature. Both A and g might depend on the instantaneous values of the stress, temperature and the structure. Following Eq. (1), the experimentally determinable apparent activation energy is defined as

$$Q = \frac{\partial \ln \dot{\gamma}}{\partial\left(-\frac{1}{RT}\right)} \simeq \frac{\Delta \ln \dot{\gamma}}{\Delta\left(-\frac{1}{RT}\right)} = \frac{R \ln \frac{\dot{\gamma}_2}{\dot{\gamma}_1}}{\frac{1}{T_1} - \frac{1}{T_2}} \tag{2}$$

A typical example of a cyclic-temperature creep curve is shown in Fig. 1, together with the calculated values of the apparent activation energies. The apparent activation energies obtained in this way were independent of strain, and, for any given temperature and stress, were

the same in both the primary and secondary stages of creep. The mean values of the apparent activation energies for various temperatures and applied shear stresses are summarized in Table I; the applied shear stress τ is taken as $\tau = \sigma/2$, where σ is the applied tensile stress.

In Fig. 2 the apparent activation energies, with their scatter bands identified, are plotted as a function of temperature regardless of the stress used. This figure clearly indicates the presence of four distinct regions:

Region I: Below about 925°K the apparent activation energy increased mildly and linearly with temperature. Although the higher temperature tests were conducted at lower stresses, the observed trend, as will be shown later, is due to the effect of temperature and not stress on the apparent activation energy.

Region II: From 925°K to 1035°K (Curie temperature ≈ 1000°K) the apparent activation energy, Q, increased steeply with temperature and reached a maximum value of ∿ 147.9 kcal/mole at 1000°K. Above the Curie point, however, Q decreased abruptly with temperature to 82 kcal/mole; the decrease was not as sharp as in the case of α-Fe. This difference may be due to the short range ordering existing in this alloy.

Region III: Above the Curie transformation range, and up to about 1135°K, the apparent activation energy remained almost constant at 82 kcal/mole.

Region IV: Beyond 1135°K, Q increased mildly with temperature. Although the cause of this increase was not subjected to a detailed study, it was believed, as in the case of α-Fe, to be due to grain and subgrain

boundary migration.

In order to ascertain whether the stress enters Eq. (1) exclusively as a result of its presence in the preexponential term, A, or whether stress also affects the free activation energy, g, a series of tests were conducted by rapid change in stress at a given temperature. A typical example of these tests is shown in Fig. 3. The effects of shear stress on the shear strain rate, $\dot{\gamma}$, are summarized in Fig. 4 where $\dot{\gamma} = 3/4\ \dot{\varepsilon}$ and $\dot{\varepsilon}$ is the tensile strain rate. These results clearly demand that

$$\dot{\gamma} = B\ \tau^{5.75} \tag{3}$$

where B is a constant.

IV. DISCUSSION

Since the creep mechanism in α-Fe was previously interpreted to be the nonconservative motion of jogged screw dislocations for temperatures below the Curie point it is natural to see whether the same mechanism is operative in Fe - 4% Si. Moreover, Barrett and Nix[10] have recently reported this mechanism to be operative in the creep behavior of a Fe - 3% Si alloy. Accordingly the results will first be analyzed in terms of this mechanism.

Following the recent treatment of Raymond and Dorn,[11] the shear strain rate derived on the basis of nonconservative motion of jogged screw dislocations can be expressed as

$$\dot{\gamma} = 2\rho b^2 \nu (z-1)\, e^{-\frac{g(\alpha)+N\ell_j b \frac{N_b}{A_s}\bar{\varepsilon}(\alpha)}{RT}} \sinh \frac{N\tau\ell_j b^2}{RT} \qquad (4)$$

where ρ = the mobile dislocation density

$g(\alpha)$ = the free energy of activation for diffusion per mole in an ordered alloy with a degree of short range order of α

ℓ_j = the average distance between jogs

N_b = the number of bonds broken in sweeping out an area A_s

A_s = the area of slip plane

$\bar{\varepsilon}(\alpha)$ = the average increase in energy per bond that is broken in an ordered alloy with a degree of short range order of α

z = the coordination number

ν = the Debye frequency

N = Avogadro's number

b = the Burgers vector

The second term in the exponential is due to the solid solution strengthening obtained as a result of short range ordering. This subject has recently been reviewed by Dorn and Mote.[12] It is to be noted that this contribution to strengthening increases with increasing degrees of short range order. However, for the alloy studied this contribution was found to be of the order of about 1 kcal/mole even at the lowest temperature investigated ($T \approx 650°K$ where $\alpha \approx -0.082$). Therefore, it will be neglected hereafter in the analysis.

Thus we can rewrite Eq. (4) as

$$\dot{\gamma} = 2\rho b^2 \nu (z-1)\, e^{-\frac{g(\alpha)}{RT}} \sinh \frac{N\tau\ell_j b^2}{RT} \qquad (5)$$

where $g(\alpha)$ increases with decreasing temperature. Using Eq. (5) the apparent activation energy, Q, is given by

$$Q = \frac{\partial \ln \dot{\gamma}}{\partial\left(-\frac{1}{RT}\right)} = \frac{\partial \ln \rho}{\partial\left(-\frac{1}{RT}\right)} + g(\alpha) - T\,\frac{\partial g(\alpha)}{\partial T} - N\tau\ell_j b^2 \coth \frac{N\tau\ell_j b^2}{RT} \qquad (6)$$

We will assume an empirical relation that

$$\rho = C(\gamma)\,\tau^m \qquad (7)$$

where $C(\gamma)$ is a function of strain only and decreases during the primary stage to reach a steady-state value over the secondary stage of creep. Thus, noting that $h(\alpha) = g(\alpha) - T\,\frac{\partial g(\alpha)}{\partial T}$, Eq. (6) becomes

$$Q = h(\alpha) - N\tau\ell_j b^2 \coth \frac{N\tau\ell_j b^2}{RT} \qquad (8)$$

Over the lower temperature side of Region I, since high stresses were used in this range, $\coth \frac{N\tau\ell_j b^2}{RT} = \coth \frac{\tau V}{RT} \simeq 1$ (where $V = N\ell_j b^2$ = activation volume). Then Eq. (8) assumes the form

$$Q = h(\alpha) - \tau V \qquad (9)$$

This relationship was indeed observed over Region I, as shown in Fig. 5. The value of V was found to be 0.4 cal/mole/psi, giving a value for ℓ_j of 26b; from the figure, $h(\alpha)$ was deduced to be 84,500 cal/mole.

Instead of Eq. (7), if ρ assumes an empirical relation of the form $\rho = C'(\gamma)(\tau/G)^m$, then we obtain a value of ℓ_j of 3.45b which is unusually small. Thus, as shown above, we will assume that the mobile dislocation density, ρ, is independent of shear modulus.

At higher temperatures, since lower stresses were used in the creep tests, $\sinh \frac{N\tau \ell_j b^2}{RT} \simeq \frac{N\tau \ell_j b^2}{RT} = \frac{\tau V}{RT}$. Incorporating this equality with Eq. (7) in Eq. (5), together with the experimentally obtained stress law from Fig. 4, we obtain a value for m of 4.75.

There are three significant points in determining the rate-controlling mechanism for creep for the alloy in question. These are the activation energy, the stress law, and the preexponential factor which includes the dislocation density. Although the model for nonconservative motion of jogged screw dislocations appears to correlate rather well with the observed apparent activation energy and stress law, it nevertheless gives an unacceptably high value for the density of mobile dislocations. For example, at T = 648°K, $\dot{\gamma} = 2.3 \times 10^{-6}$ sec^{-1} and τ = 32,850 psi, Eq. (5), with experimentally deduced $\ell_j = 26b$ and $g(\alpha)$ which can be deduced as shown in Reference 1, predicts $\rho \simeq 1.2 \times 10^{16}$ cm^{-2}. Even at the higher temperature where the stresses were lower, it still gives exceptionally high values of ρ (e.g. $\rho \simeq 5 \times 10^{14}$ cm^{-2} at T = 790°K, $\dot{\gamma} = 6.2 \times 10^{-6}$ sec^{-1} and τ = 11,000 psi). Therefore it must be concluded that the nonconservative motion of jogged screw dislocations is not responsible for the creep behavior of Fe - 4% Si.

An alternative rate-controlling mechanism for high temperature creep of metals and alloys is that of dislocation climb.[13-16] This model also suggests that the apparent activation energy for creep corresponds to that for diffusion. Furthermore, it also predicts a power stress law. Unfortunately there is at present no diffusion data available for this alloy, but it is thought that the activation energy for diffusion

must be in the vicinity of that for α-Fe. The generally accepted value of the activation energy for diffusion for pure α-Fe is of the order of 60 kcal/mole.[1] Over Region II of Reference 1 (corresponding to Region I in this investigation), where the effect of the magnetic transformation on creep was thought to be negligible, the apparent activation energy for creep for α-Fe was found to have the same value as that for diffusion. In Region I of this investigation the apparent activation energy for Fe - 4% Si was found to be of the order of 70 kcal/mole. Recently, Davies[8] also observed an apparent activation energy for creep of about 72 kcal/mole for the same alloy in the corresponding temperature range, and Karashima et al.,[17] using a Fe - 4.52 at.% Si alloy, reported Q = 81.5 ± 3.5 kcal/mole in the ferromagnetic region and Q = 74.4 ± 0.6 kcal/mole in the paramagnetic temperature range. Although these values are slightly higher than that for α-Fe they nevertheless must be quite close to that for diffusion for the alloy studied. It is well known from diffusion experiments[18] that the activation energy for diffusion in the long range ordered state is higher than that for the disordered state. No diffusion data for short range ordered alloys is available at present but a recent calculation on the equilibrium vacancy concentration in short range ordered binary alloys[19] indicates that the free energy of formation of a vacancy increases with increasing degree of short range order. If the migration energy of a vacancy also increases in the same manner, which is likely, then the activation energy for diffusion will be higher in the ordered state than in the disordered one provided that diffusion is taking place by a vacancy mechanism. In this event, one would expect a higher activation

energy for diffusion in Fe - 4% Si than in α-Fe.

Although the existing climb theories[13-16] for high temperature creep differ in their prediction of the power in the stress law, depending on the details assumed by each model, they nevertheless have the following general form

$$\dot{\gamma} = K \frac{\tau^n}{TG^{n-1}} e^{-\frac{g}{RT}} \tag{10}$$

where K is independent of the temperature. Weertman's model[13,14] based on the climb of piled-up edge dislocations gives n = 3 to 4.5, whereas Chang's model[16] based on climb due to the dissolution of dislocation trails predicts n = 4. The present experimental results, however, suggest that n = 5.75.

In order to see the effect of temperature on the activation energy, Eq. (10) may be substituted into Eq. (2) to give the apparent activation energy

$$Q = \frac{\partial \ln \dot{\gamma}}{\partial \left(-\frac{1}{RT}\right)} = g - T \frac{\partial g}{\partial T} - RT - (n-1) \frac{RT^2}{G} \frac{dG}{dT}$$

$$= h - RT - (n-1) \frac{RT^2}{G} \frac{dG}{dT} \tag{11}$$

The third term beyond the last equality sign represents the contribution due to the decrease of shear modulus with increasing temperature; this always has a positive value.

Recently, Lytton[20] has measured the Young's modulus of Fe - 3.1% Si as a function of temperature up to 1473°K. Using these results and

following Eq. (11) when $h = Q + RT + \frac{(n-1)RT^2}{G}\frac{dG}{dT}$ is plotted against temperature and shear stress, as shown in Figs. 6 and 7 respectively, it is evident that h is constant over Region I. Therefore, it is concluded that the slight increase of Q with increasing temperature over this region is due to effect of temperature on the shear modulus of elasticity rather than the effect of stress. This is in contrast with the trend previously suggested for α-Fe.[1] From either Fig. 6 or Fig. 7, the activation energies for diffusion in the ferromagnetic and paramagnetic region were deduced to be 71.4 and 69.6 kcal/mole respectively. Both values are considerably higher than those reported for α-Fe by Buffington et al.[21] (h = 60 and 57.2 kcal/mole for ferromagnetic and paramagnetic iron respectively). A possible explanation of this difference could again be short range ordering.

The anomalous changes in Q over Region II, as will be shown in the following, can be attributed to the way in which g in Eq. (11) depends on the temperature over the magnetic transformation. In order to see how g/T might vary throughout the Region II we rewrite Eq. (11) as

$$g - T\frac{\partial g}{\partial T} = h = Q + RT + (n-1)\frac{RT^2}{G}\frac{dG}{dT} \tag{12}$$

Integrating Eq. (12) we obtain

$$\frac{g}{T} = \frac{g_1}{T_1} + \int_{\frac{1}{T_1}}^{\frac{1}{T}} h \, d\left(\frac{1}{T}\right) \tag{13a}$$

The diffusivity is given by

$$D = D_o' \, e^{-g/RT} = D_o \, e^{-h/RT}$$

where

$$D_o = D_o' \, e^{S/R} = f\delta\lambda^2\nu \, e^{S/R} = 0.72 \times \frac{4b^2}{3} \nu \, e^{S/R}$$

$$\simeq 8.06 \times 10^{-3} \, e^{S/R} \quad cm^2/sec \qquad (14)$$

f being the correlation factor, δ the jump factor, and λ the jump distance which are given for the b.c.c. lattice. We will assume D_o is the same as in iron, and equal to 0.5 cm^2/sec as reported by Lazarus.[22] Thus, we get the entropy s = 8.2 cal/mole °K. Moreover, in Eq. (13a) taking T_1 = 648°K, h_1 = 71.4 kcal/mole (from Fig. 6) and the above calculated entropy, we find that g_1/T_1 = 102 cal/mole °K. Thus Eq. (13a) becomes

$$\frac{g}{T} = 102 + \int_{\frac{1}{648}}^{\frac{1}{T}} h \, d\left(\frac{1}{T}\right) \qquad (13b)$$

By graphical integration the values of g/T can be evaluated and plotted against $\frac{1}{T}$, as shown in Fig. 8. It is readily seen that the slope, which is actually the enthalpy of activation, h, is higher in the ferromagnetic range than in the paramagnetic range, as expected from the diffusion data of α-Fe.

Zener,[23] in a model later elaborated by LeClaire,[24] proposed that the free energy of activation for diffusion, g, could be related to

the Young's modulus, E, in the following way:

$$g \approx CE \tag{15}$$

where C is a constant and virtually independent of temperature. In order to see this correlation, E/T, obtained from Lytton's results,[20] is also plotted in Fig. 8. The agreement is indeed very good; furthermore, it is clear that the anomalous changes in the apparent activation energy in the Curie transformation range are essentially due to the decrease in the free activation energy for diffusion.

The dislocation climb model as represented by Eq. (10) also gives the correct stress law demanded by the results shown in Fig. 4. Unfortunately, there is not sufficient data available at present in order to further evaluate the constant K in Eq. (10). Nevertheless the trends observed in this investigation suggest that Eq. (10) is in good agreement with the experimental results, although none of the existing models predicts a power of n = 5.75. However, a number of assumptions are made in Weertman's theory, and an analysis of these suggest that this value of n may be reasonable at high stress levels. Furthermore, high values of n (>7) have previously been reported for Fe containing Si additions of up to 10.7 at.%.[8] Thus we can only tentatively say that the rate-controlling mechanism for high temperature creep of Fe - 4% Si appears to be dislocation climb rather than the nonconservative motion of jogged screw dislocations as reported by Barrett and Nix[10] for Fe - 3% Si.

V. CONCLUSIONS

1. The creep behavior of Fe - 4% Si from 650° to 1100°K obeys the relationship

$$\dot{\gamma} = K \frac{\tau^{5.75}}{TG^{4.75}} \exp -\left(\frac{g}{RT}\right)$$

2. The apparent activation energy for creep is insensitive to stress and strain, and increases slightly with increasing temperature from 650° to 925°K. Over the magnetic transformation range, 925° to 1035°K, the apparent activation energy increases steeply with increasing temperature and reaches a maximum at the Curie temperature. It then decreases sharply to a lower value, and increases slightly with increasing temperature from 1035° to 1135°K. Beyond 1135°K, the apparent activation energy again increases sharply.

3. The anomalous increase and then decrease in activation energy with increasing temperature over the range of magnetic transformation is in good agreement with the known decrease in the free energy of activation for diffusion.

4. The activation energies for diffusion in the ferromagnetic and paramagnetic temperature range were deduced to be about 71.4 and 69.6 kcal/mole respectively.

5. Over the entire temperature range investigated the creep behavior was found to be in agreement with the dislocation climb mechanism rather than the mechanism of nonconservative motion ofjogged screw dislocations as previously reported for α-Fe.

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Table 1

$\bar{T} = \frac{2\,T_1\,T_2}{T_1 + T_2}$ °K	Shear Stress τ psi	Apparent Activation Energy Q kcal/mole
648	32,850	71.8
690	25,700	74.8
790	11,000	74.5
850	6,600	81.8
925	3,245	86.0
970	1,790	103.2
985	1,660	116.8
1000	1,465	147.9
1020	1,040	95.0
1025	1,015	88.7
1035	995	82.1
1070	680	80.8
1133	475	85.4
1155	415	91.2
1164	390	98.5

FIGURE CAPTIONS

Figure 1 Typical creep rate-extension curve under cyclic temperature condition for use in the evaluation of Q.

Figure 2 Apparent activation energies for creep as a function of absolute temperature.

Figure 3 Typical creep rate-extension curve under cyclic stress condition for use in the evaluation of stress dependence.

Figure 4 Effect of stress on the creep rate.

Figure 5 Apparent activation energy for creep, Q kcal/mole.

Figure 6 Apparent activation energies corrected for changes of shear modulus as a function of temperature.

Figure 7 Apparent activation energies corrected for changes of shear modulus as a function of applied shear stress.

Figure 8 Temperature dependences of free energy of activation for diffusion and of elastic modulus.

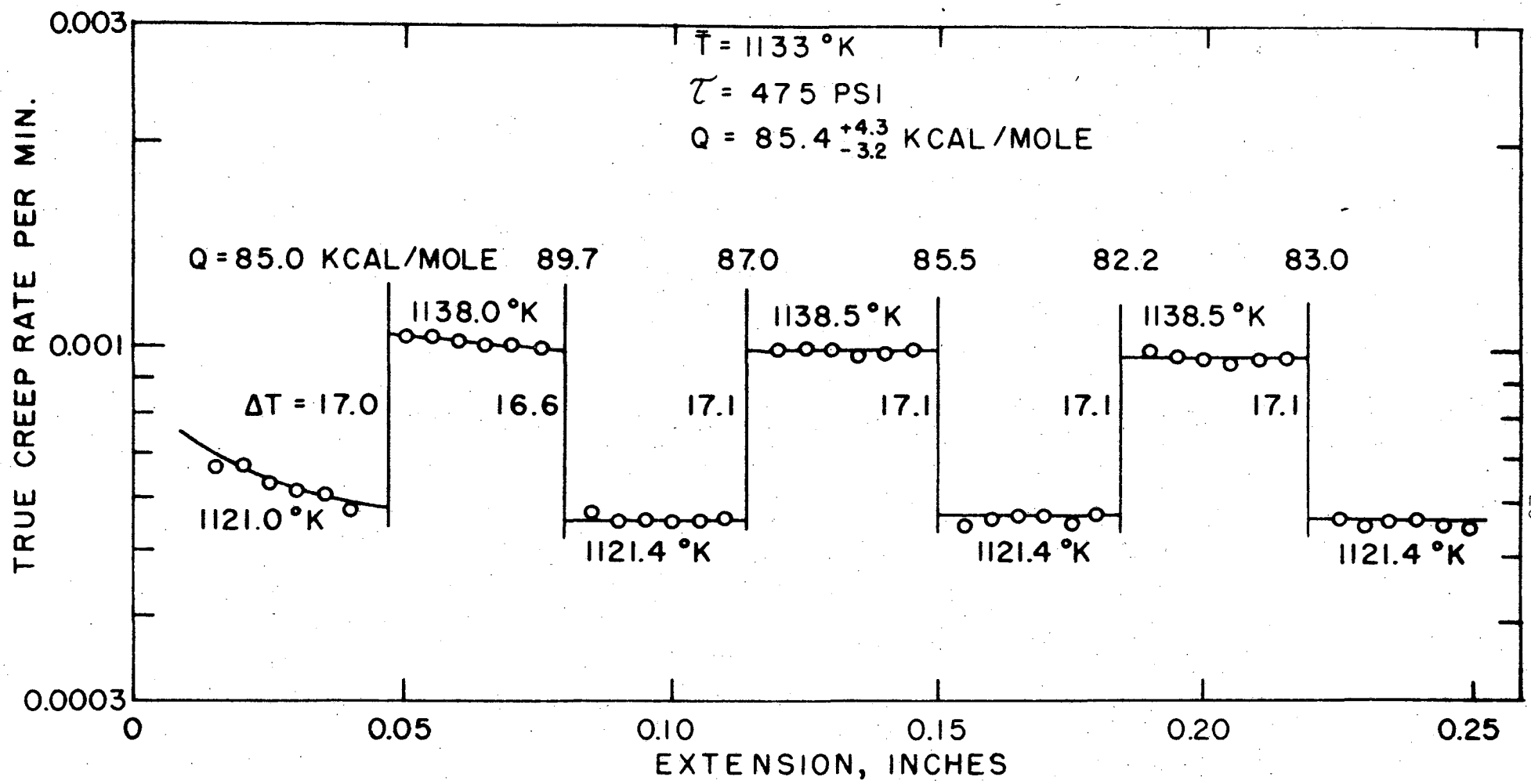
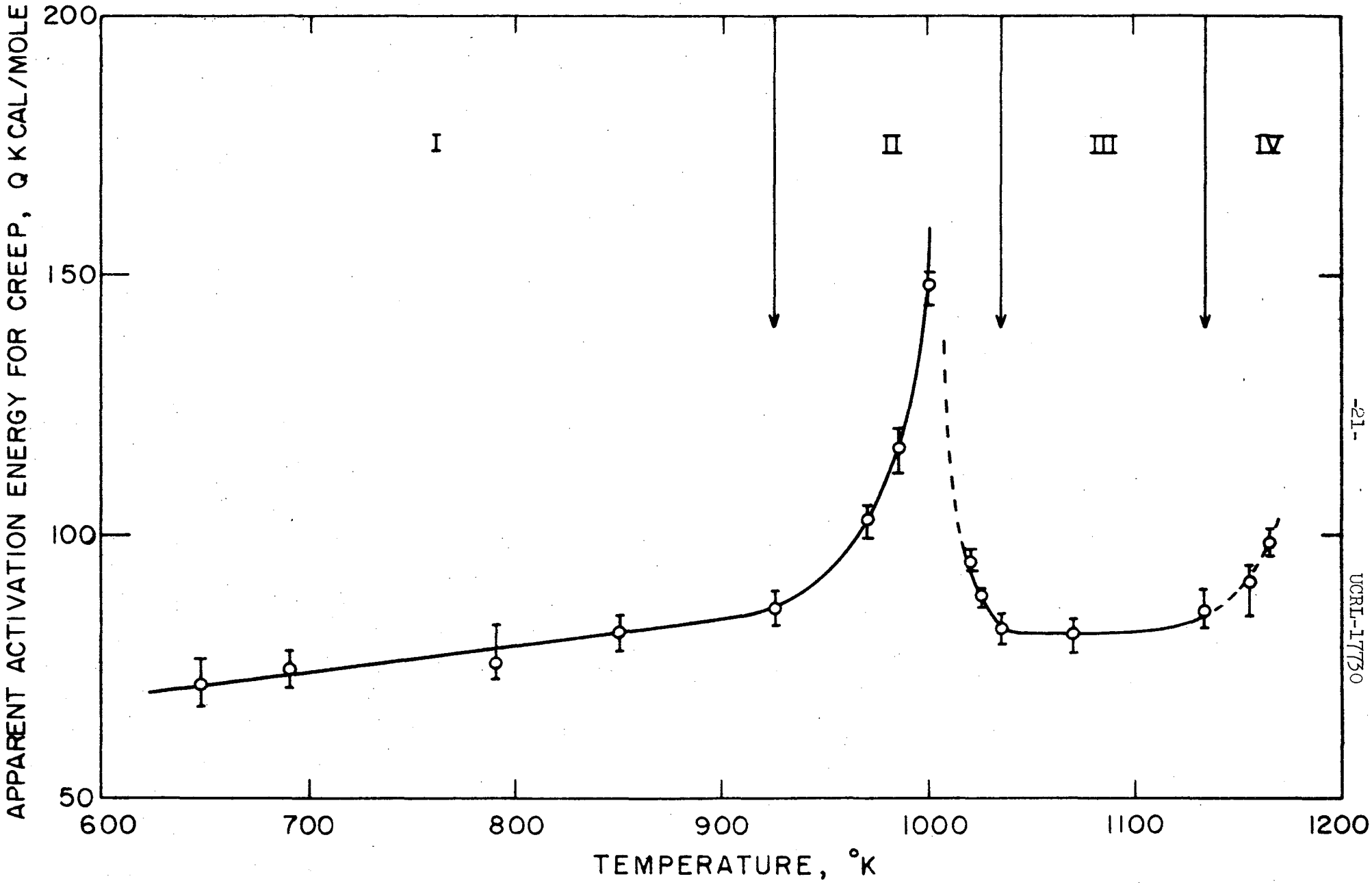

0.003
0.001
0.0003
TRUE CREEP RATE PER MIN.
T̄ = 1133 °K
τ = 475 PSI
Q = 85.4 +4.3 -3.2 KCAL/MOLE
Q = 85.0 KCAL/MOLE
89.7
87.0
85.5
82.2
83.0
1138.0 °K
1138.5 °K
1138.5 °K
ΔT = 17.0
16.6
17.1
17.1
17.1
17.1
1121.0 °K
1121.4 °K
1121.4 °K
1121.4 °K
0
0.05
0.10
0.15
0.20
0.25
EXTENSION, INCHES

FIG 1


APPARENT ACTIVATION ENERGY FOR CREEP, Q KCAL/MOLE
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100
50
600
700
800
900
1000
1100
1200
TEMPERATURE, °K
I
II
III
IV

FIG. 2

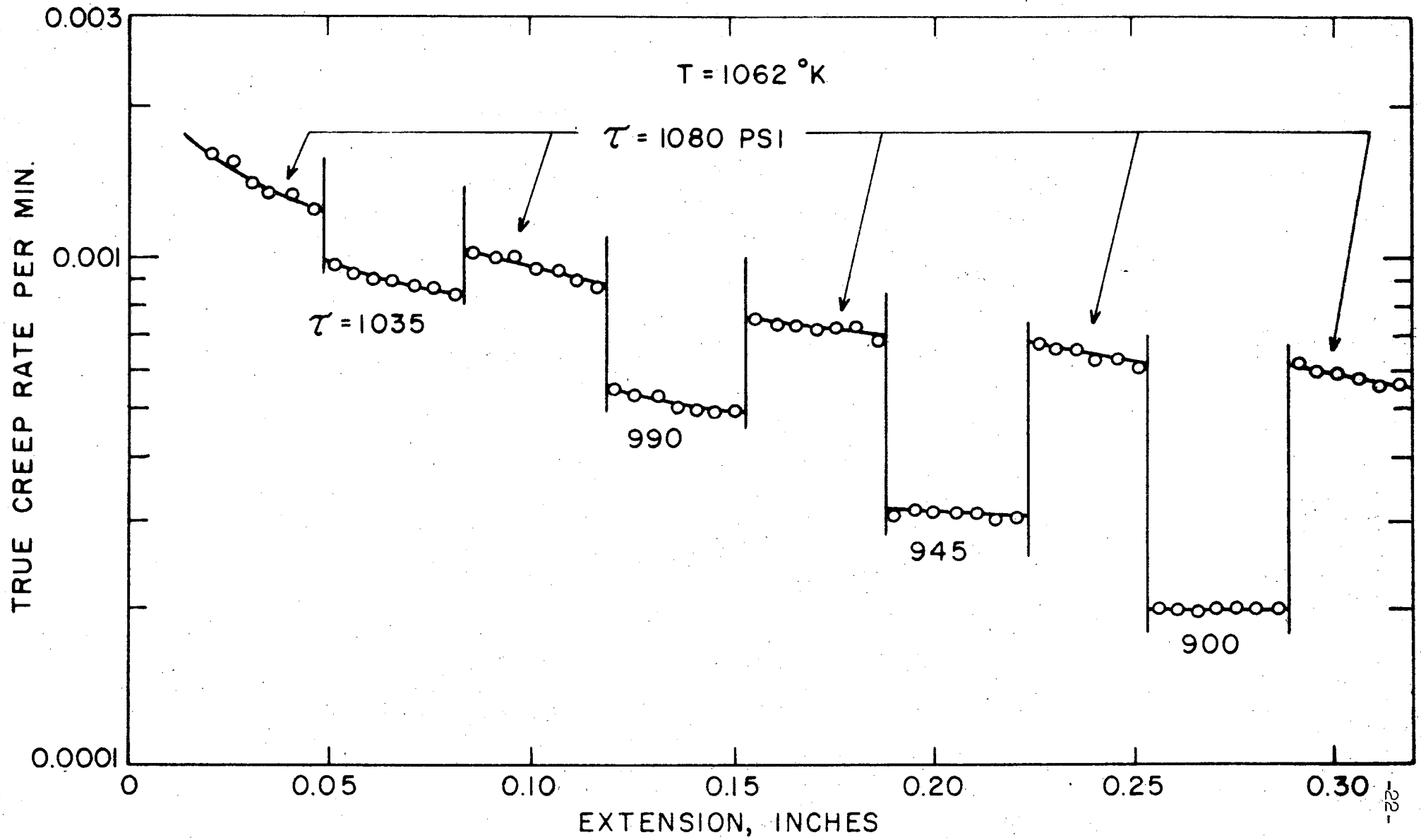
T = 1062 °K
τ = 1080 PSI
τ = 1035
990
945
900
0.003
0.001
0.0001
TRUE CREEP RATE PER MIN.
0
0.05
0.10
0.15
0.20
0.25
0.30
EXTENSION, INCHES

FIG. 3

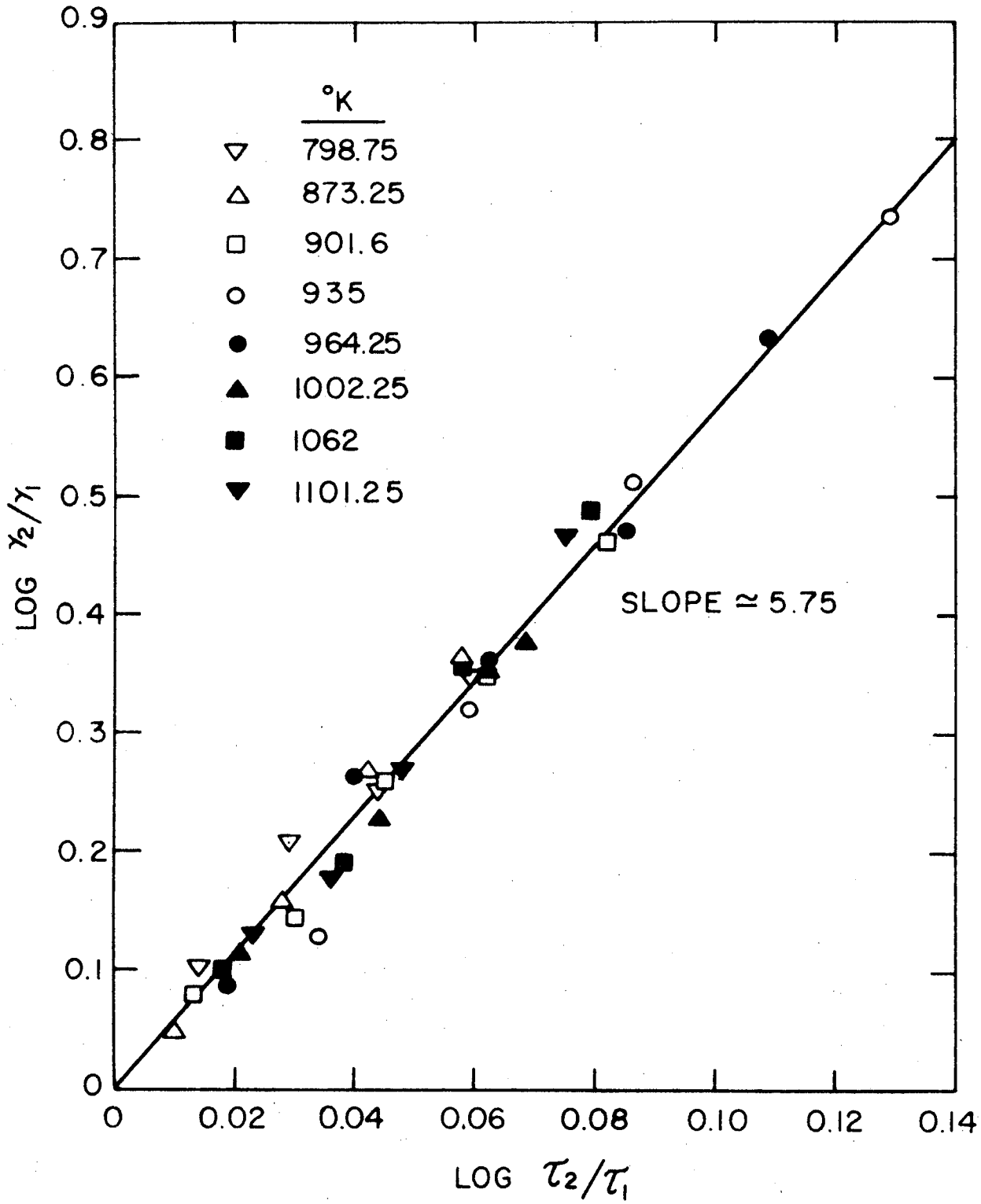
°K
798.75
873.25
901.6
935
964.25
1002.25
1062
1101.25
SLOPE ≃ 5.75
LOG γ_2/γ_1
LOG τ_2/τ_1
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
0.02
0.04
0.06
0.08
0.10
0.12
0.14

FIG. 4

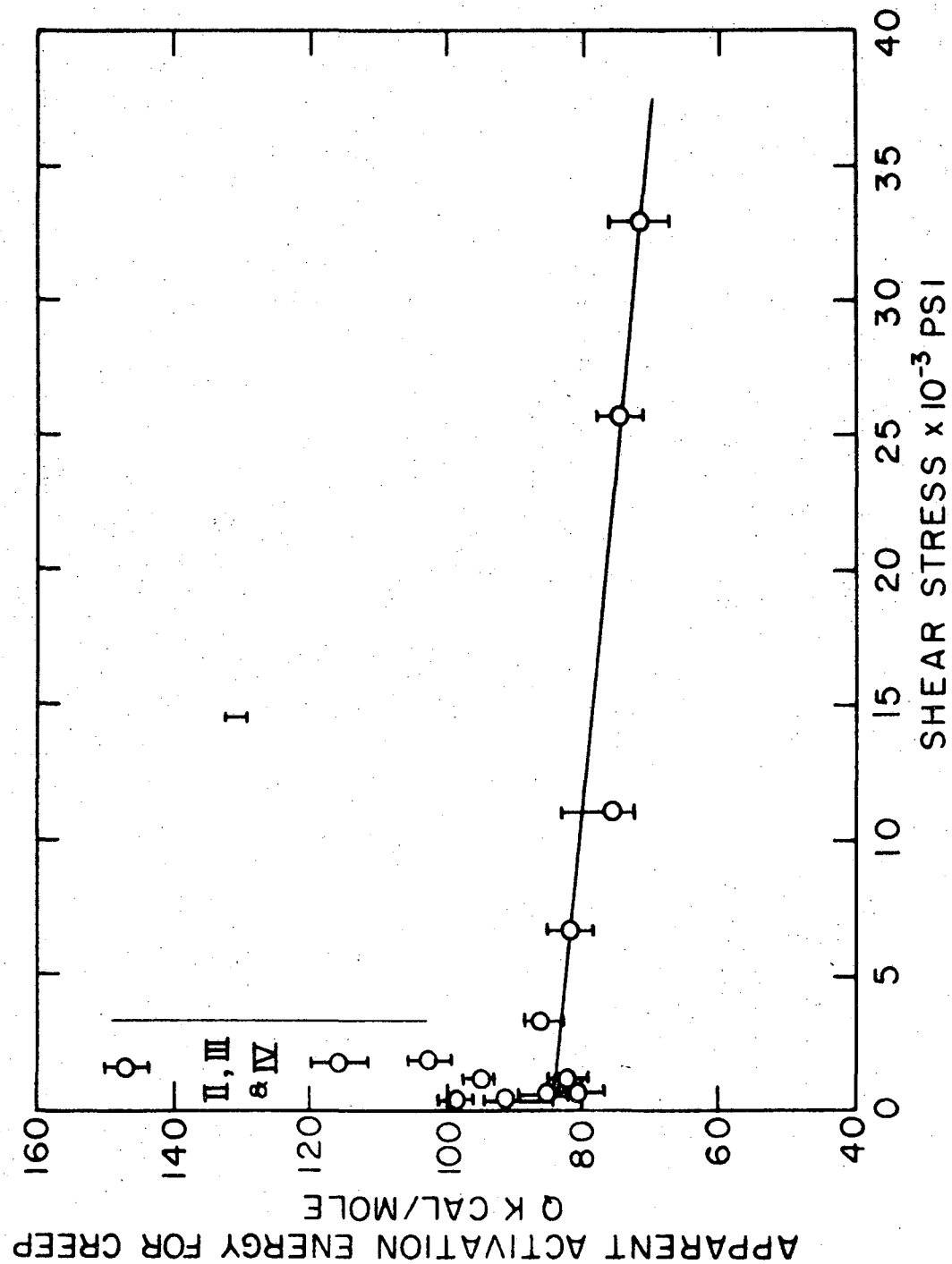
APPARENT ACTIVATION ENERGY FOR CREEP
Q K CAL/MOLE
160
140
120
100
80
60
40
0
5
10
15
20
25
30
35
40
SHEAR STRESS x 10^-3 PSI
I
II, III
& IV

FIG. 5

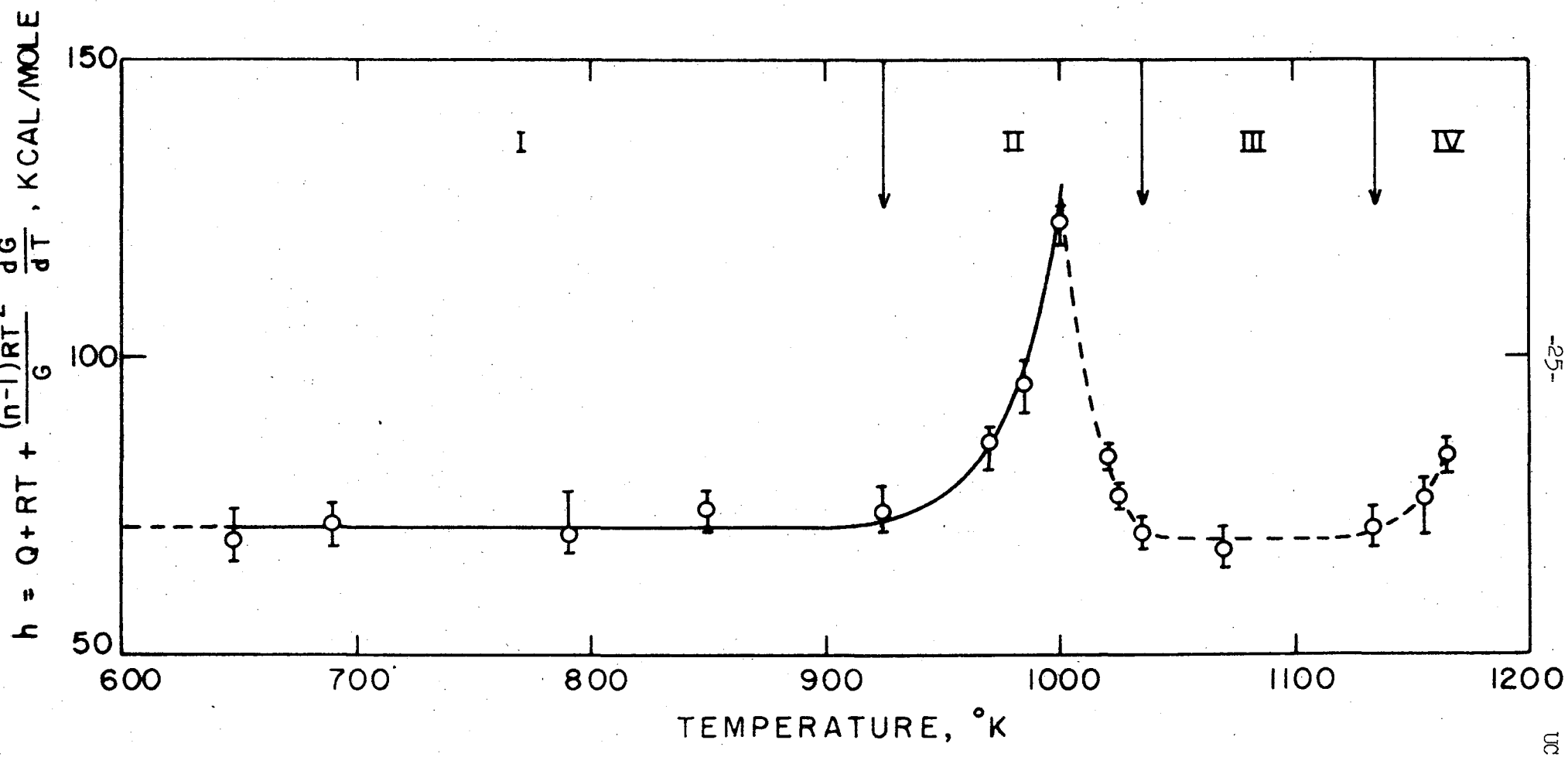
$h = Q + RT + \frac{(n-1)RT^2}{G} \frac{dG}{dT}$, KCAL/MOLE
150
100
50
600
700
800
900
1000
1100
1200
TEMPERATURE, °K
I
II
III
IV

FIG. 6

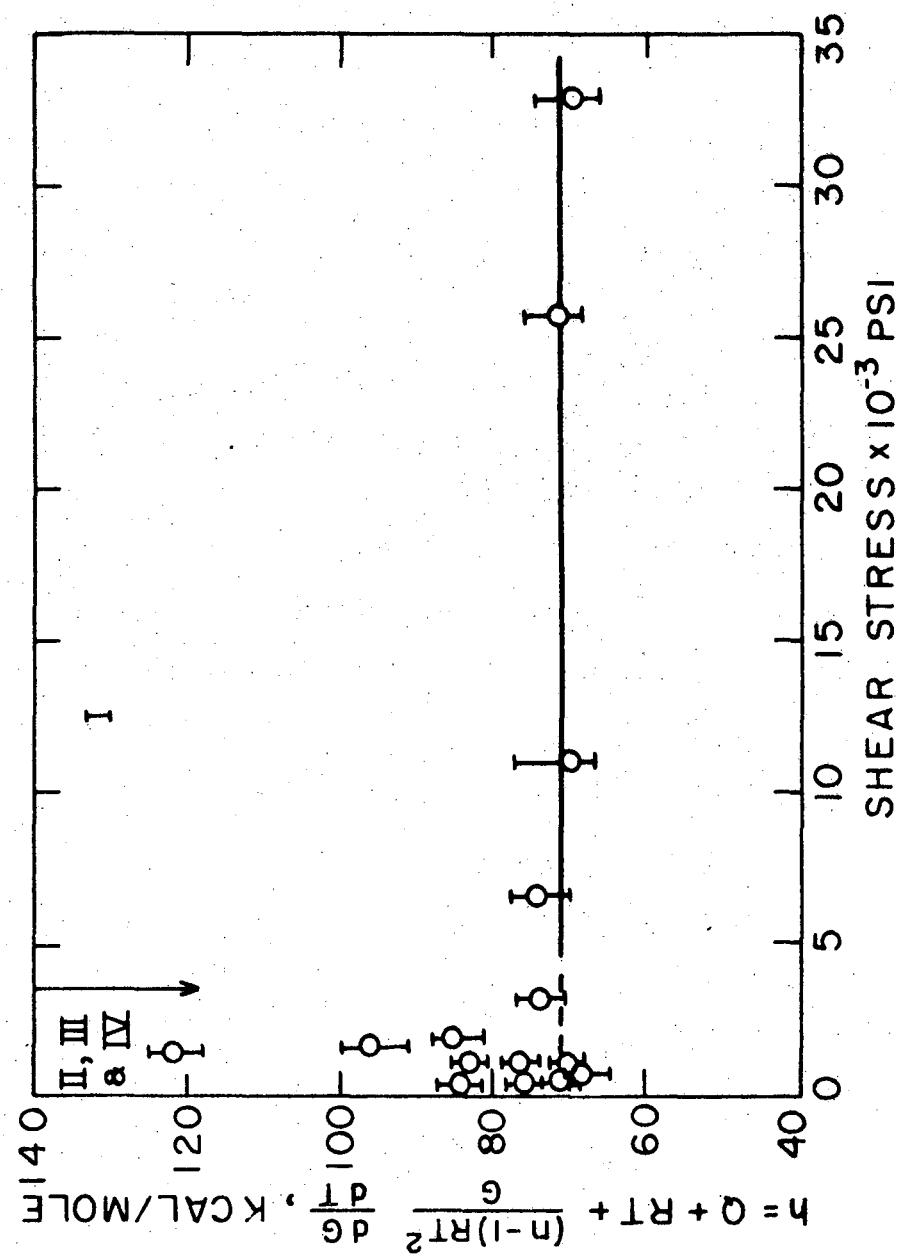
h = Q + RT + (n-1)RT²/G dG/dT , KCAL/MOLE
140
120
100
80
60
40
II, III & IV
I
0
5
10
15
20
25
30
35
SHEAR STRESS x 10⁻³ PSI

FIG. 7

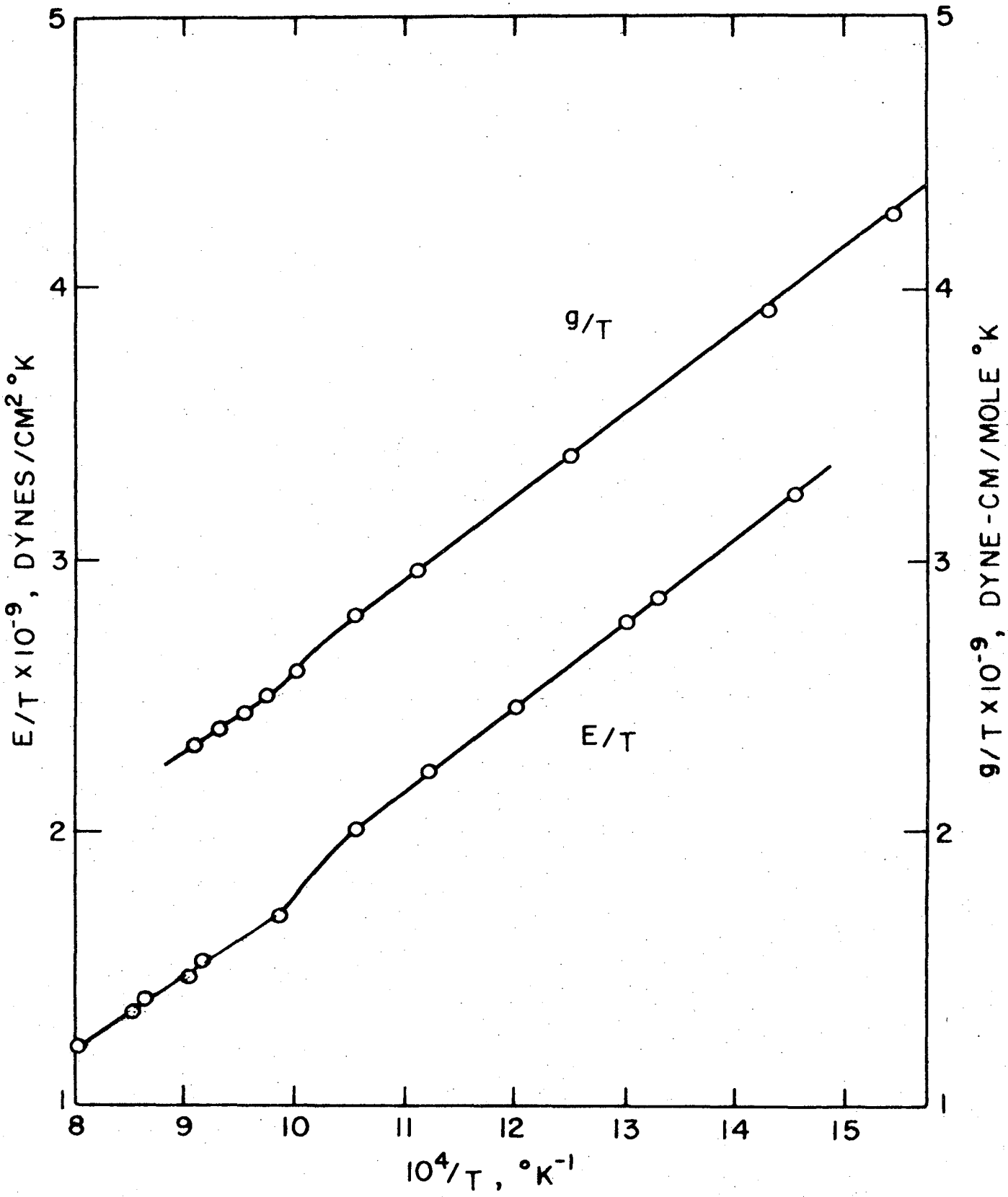
5
4
3
2
1
8
9
10
11
12
13
14
15
g/T
E/T
E/T X 10^-9, DYNES/CM^2 °K
g/T X 10^-9, DYNE-CM/MOLE °K
10^4/T, °K^-1

FIG. 8

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