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Publication Date

1969-03-01

Submitted to Acta Metallurgica

UCRL-18803
Preprint

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March 1969

AEC Contract No. W-7405-eng-48

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CREEP OF A DISPERSION STRENGTHENED STEEL

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ABSTRACT

March, 1969

The creep behavior of a quenched and tempered Fe-Mo-C steel was investigated over the range of the magnetic Curie transformation from 875° to 1075°K. The apparent activation energy for creep was insensitive to the applied stress and had the same value over the primary and secondary stages. It exhibited the same type of hump at the Curie transformation temperature as that observed for the apparent activation energy for creep pf α - Fe, which it exceeded by about 11,000 cal/mole. The secondary creep rate of this alloy increased with the 6.8 power of the applied stress in complete agreement with similar trends for α - Fe. The secondary creep rate, however, was about 10^{-3} that of α - Fe due principally to its higher activation energy for creep.

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This work was done under the auspices of the United States Atomic Energy Commission.

I. INTRODUCTION

Creep of most metals and some solid solution alloys at high-temperatures follows the relationship$^{(1)}$

$$\frac{\dot{\gamma}s\ kT}{DGb} = A \left(\tau/_G\right)^n \qquad (1)$$

where $\dot{\gamma}s$ is the secondary shear strain rate, kT the Boltzmann constant times the absolute temperature, D the diffusivity, G the shear modulus of elasticity, and τ is the applied shear stress. Where the dislocation climb mechanism prevails A is about 3×10^6 or greater and n varies with metal and alloy from 4.2 to about 7.0 dependent on stacking fault energy, impurities and other as yet unknown factors.

In contrast to studies on pure metals and solid solutions there have been very few systematic investigations on the creep behavior of dispersion strengthened alloys despite the more intense technological interest in their higher creep resistance: The creep of a few dispersion strengthened alloys follow somewhat closely the relationship given by Eq. 1. The creep of other dispersion strengthened metals and alloys, however, exhibit significant deviations from the tenets of Eq. 1. For example, the power n for thoriated Ni$^{(2,3)}$ is about 40, and that for SAP$^{(6)}$ is approximately 10; furthermore, the apparent activation energy for creep of thoriated Ni$^{(2,3)}$ is about 190,000 cal./mole, and that for SAP$^{(4,5,6)}$ ranges from 150,000 - 400,000 cal./mole, suggesting that creep might not be diffusion controlled in these systems. It is indeed difficult to determine whether or not such high activation energies for creep might in one way or another be related to that for diffusion.

Recent investigations(7) have shown that the apparent activation energy for creep in α-Fe shows a pronounced hump at the magnetic Curie transformation that is directly dependent on that for self-diffusion. If the apparent activation energy for creep of a dispersion strengthened steel exhibited an analogous hump at the Curie transformation it would prove, regardless of its magnitude, that the creep was at least in part dependent on diffusion. The following investigation was initiated to examine this possibility.

II. MATERIALS AND TECHNIQUE

An ideal steel for this investigation would be one in which the dispersed phase remains unaltered during the high-temperature creep tests. To approximate this idealization a Mo-C steel was selected and heat treated so as to obtain a fairly stable dispersion of Mo_2C in the steel matrix. The analysis in weight percent of the steel used was as follows: Mo-4.00, C-0.19, Mn-0.50, Si-0.08, O-0.029. The 2-3/8 in. diameter by 10 in. high ingot was quench-cast in a Cu mold and homogenized under argon for 7 days at 1100°C. Following removal of the surface by turning, it was forged to 5/8 in. in diameter at 1100°C and swaged in four steps at about 1000°C to 0.34 in. in diameter. Between each swaging step the bars were surface cleaned.

The bars were austenitized under argon at 1200°C for two hours, quenched in ice brine, and thereafter cooled in liquid nitrogen to provide fairly complete transformation of the austenite to martensite. Each bar was tempered and given a spheroidization anneal under argon at 800°C for 24 hours and then quenched in water. Samples from the various bars exhibited the same uniform grain size and dispersion of carbides. Honeycomb(8) has shown that, in a steel of about the same composition and heat treatment, the dispersed phase consists of Mo_2C particles which are quite stable. The creep-testing temperatures were somewhat below the spheroidizing temperature and should not have introduced any major alteration in the microstructure. Typical examples of the microstructure before and after creep-testing are shown in Figs. 1a and b respectively. No changes in the microstructure during the course of creep at the highest test temperature could be detected.

Double-shear types of specimens, previously used(9) with high success, were adopted for this investigation. This type of specimen exhibited uniform shear over the gage section and the same shear strain on each gage section. Duplicate tests gave identical results.

All creep tests were conducted in an argon atmosphere. Furnace temperatures were held constant by a proportioning controller that was activated by two chromel-alumel thermocouples placed on the specimen adjacent to the two gage sections. Specimen temperatures were held constant to better than ± 1°C throughout each constant temperature test. Loads were applied through a lever arm and the stresses were determined to an accuracy of ± 0.15 psi. Shear displacements of the specimen were determined by means of a linear differential transformer from which strains accurate to 10^{-4} were calculated; gage section dimensions were measured to the nearest 0.0002 inches.

The apparent activation energies for creep were determined from the effect of small changes in temperature, ± 14° to 16°C, on the secondary creep rate. The fixtures and specimens had sufficiently low heat capacity to permit the temperature to equilibrate and the new creep rate to be obtained in about three minutes. The new creep rates were determined following the three minute transient period.

In a few cases the effect of stress on the steady-state creep rate was obtained directly from creep curves at constant stress. Most of the data on correlations between stress and the steady-state creep rate, however, were obtained from constant-temperature creep tests in which

III. EXPERIMENTAL RESULTS

a. Effect of Stress on the Steady-State Creep Rate

A typical creep curve is shown in Fig. 2. It reveals initial strain upon loading, followed by primary (I), secondary (II), and tertiary(III), creep as are obtained for high-temperature creep of annealed metals. The fact that a brief primary stage is followed by an extensive steady-state condition of secondary creep reflects the fact that a rather stable microstructure had been obtained and that it remained substantially unaltered over the entire steady-state condition.

Typical examples of the steady-state creep rate, obtained when the stress was periodically changed, are illustrated in Fig. 3. A rather brief transient in the creep rate was observed upon an increase or a decrease in the stress level. As in the case of pure metals, this was followed by a steady-state creep rate that was independent of the previous stress history and depended only on the acting stress. The same steady-state creep rates were obtained from the constant and the periodically varied stress tests.

The effect of stress on the secondary creep rates for the series of temperatures that were investigated is shown in Fig. 4. The blocked symbols refer to data obtained from constant-stress creep tests, the open symbols refer to data obtained by the periodically varied stress tests in creep, and the cross symbols refer to data obtained from the Instron tests. The three sets of data agree well with each other. As shown in Fig. 4, the effect of stress on the secondary creep rate is insensitive to the test temperature, and can be represented by the stress was changed periodically during creep. Additional data were obtained from the steady-state shear stress obtained from tests conducted in an Instron machine at a constant strain rate. It was observed that following a brief transient upon change in stress or strain rate a new steady-state appropriate to the new condition was established. The same steady-state creep rate was obtained regardless of the differences in the three test procedures that were employed.

IV. DISCUSSION

The apparent activation energy for creep of the Mo_2C dispersion strengthened steel is invariant over the primary and secondary stages of creep and is insensitive to variations in the stress. As shown in Fig. 6, it exhibits a hump over the temperature range of the magnetic Curie transformation that is wholly analogous to that for the apparent activation energy for creep(7), and also self-diffusion in α-Fe. Since no alternate explanation for the hump in the apparent activation energy for creep of this Mo_2C dispersion strengthened steel appears possible, it is concluded that its creep rate is determined by a diffusion controlled mechanism. Furthermore, as shown in Fig. 4, the secondary creep rate increases with the same 6.8th power of the applied stress for all temperatures covering the range of the magnetic Curie transformation, in complete agreement with the corresponding relationship for creep in α-Fe. These observations lead to the inevitable conclusion that the creep of this dispersion strengthened steel is controlled by the dislocation climb mechanism.

The sole point of departure between the creep of this dispersion strengthened steel and that for α-Fe arises from the fact that its apparent activation energy is about 11,000 cal./mole higher than that for α-Fe. To illustrate this point, Eq. 1 will be adjusted to read:

$$\frac{\dot{\gamma}s\ kT}{DGb\,e(-Hc/RT)} = Ac\left(\tau/G\right)^{n} \qquad (2)$$

where D refers to the self-diffusivity in α-Fe and Hc is an additional enthalpy of activation term for the dispersion strengthened steel.

$$\dot{\gamma}_s = K^{'} \{T\} \tau^{6.8} \qquad (2)$$

where $K^{'} \{T\}$ is a function of the temperature.

b. Effect of Temperature on the Secondary Creep Rate.

Creep must necessarily occur as a result of a thermally activated mechanism. Consequently the major influence of temperature on the creep rate must arise from an exponential term involving minus the free energy of activation per mole over the gas constant, R, times the absolute temperature, T. Since the temperature can also enter the analysis in other less pronounced ways it is convenient to define an apparent activation energy for creep by

$$Qc = \left\{\frac{\Delta \ln \dot{\gamma}}{\Delta (- 1/RT)}\right\}_{\tau} = R \frac{\ln \dot{\gamma}_2/\dot{\gamma}_1}{\frac{1}{T_1} - \frac{1}{T_2}} \qquad (3)$$

where $\dot{\gamma}_2$ and $\dot{\gamma}_1$ are the creep rates following and preceeding small changes in temperature from T_1 and T_2. Typical examples of cyclic temperature creep curves are shown in Fig. 5. Following a very short temperature transient they invariably give a limiting creep rate. Most of the data on the apparent activation energies for creep were obtained for the steady-state. However, as in the case of metals, the apparent activation energy for creep of this dispersion hardened alloy was also found to be independent of the stage of creep and of the applied stress. The apparent activation energies for creep and their scatter bands are shown in curve "a" of Fig. 6 as a function of temperature.

Thus, the apparent activation energy for creep of the dispersion strengthened steel is given by

$$Qc = \left\{\frac{\partial \ \ln \dot{\gamma}}{\partial -1/_{RT}}\right\}_{\tau} = H_D + H_c - RT - \frac{(n-1)\ RT^2}{G}\left(\frac{\partial G}{\partial T}\right) \qquad (3)$$

where H_D is the enthalpy for self-diffusion in α-Fe, namely $(\partial \ln D)/\partial/(-^1/RT)$. An estimate of the magnitude of each term of Eq. 3 is given in Fig. 6. H_D was deduced directly from the data of Lai and Borg(10,11) on tracer self-diffusion in α-Fe covering the range of the magnetic Curie transformation. No data are available on the variation of the moduli of elasticity with temperature for the matrix of the dispersion strengthened steel under study here. A fair estimate of the modulus effect, however, was deduced from data on α-Fe by Köster(12) and the corresponding date on an Fe - 4 wt % Mo steel by Fuchs(13). The trends of the moduli for α-Fe and the Fe - 4 wt % Mo alloy were reasonably similar and their mean value was adopted in the present analysis for evaluating $-(n-1)\ \frac{RT^2}{G}\ \partial G/_{\partial T}$. The value of $H_D - RT - \frac{(n-1)}{G}\ RT^2\ \frac{\partial G}{\partial T}$, shown in Fig. 6, agrees well with the previously reported activation energy for creep in α-Fe(7). In contrast, the apparent activation energy, Qc, for the dispersion strengthened steel, vide Fig. 6, appears to be uniformly higher by about 11,000 cal./mole. It is possible that this difference arises from experimental error which is quite high for both H_D and Qc. Several factors, however, suggest that the effect is real. (a) The good agreement obtained between Qc for α-Fe and $H_D - RT - (n-1)\ \frac{RT^2}{G}\ \frac{\partial G}{\partial T}$, suggests that the latter expression is reasonably correct. In addition Qc for the dispersion strengthened steel was determined to an accuracy of about ± 3,000 cal./mole. and it scattered approximately 4,000 cal./mole. from the mean values. Consequently the difference of 11,000 calories per mole is judged to be real. (b) The secondary creep rates of the dispersion strengthened steel can be summarized in accord with Eq. 2 as shown in Fig. 7 using Hc as 11,000 cal./mole. Thus

$$\frac{\dot{\gamma}s\ \{disp\}\ kT}{DGb\ e^{-11,000/RT}} = 6 \times 10^{15} \left(\tau/_G\right)^{6.8} \qquad (4)$$

where as similar data for α-Fe(7), also shown in Fig. 8, reveals that

$$\frac{\dot{\gamma}s\{\alpha\text{-Fe}\}kT}{DGb} = 2 \times 10^{17} \left(\tau/_G\right)^{6.8} \qquad (5)$$

The good coincidence between the secondary creep rate data for the dispersion strengthened steel and that for α-Fe, as shown in Fig. 7, enhances the concept that Hc is real, and about 11,000 cal./mole.

The secondary creep rates of the dispersion strengthened steel, $\dot{\gamma}s$ {disp.}, are less than those for α-Fe, namely $\dot{\gamma}s$ {α-Fe} by a factor f given by

$$\dot{\gamma}s\ \{disp\} = f\ \dot{\gamma}s\ \{\alpha\text{-Fe}\} = 0.03\ e^{-11,000/RT}\ \dot{\gamma}s\{\alpha\text{-Fe}\} \qquad (6)$$

which reveals that the extra enthapy of activation for creep, Hc, constitutes a strengthening factor.

The question concerning the origin of Hc has not yet been answered. It might arise from one or more of a number of possibilities such as (a) effects of alloying on the diffusivity of vacancies, (b) effects of solute atom drag on the climbing dislocations, and (c) effects of substructural changes induced by the dispersed phase in restraining activation for the climb of dislocations.

V. CONCLUSIONS

1. The creep of a tempered Fe - 4% Mo -0.20%C - 0.50% Mn steel over the range of temperatures covering the magnetic Curie transformation form 875° to 1075° is largely diffusion controlled. The apparent activation energy for creep has the same hump at the Curie temperatures as does the activation energy for diffusion in α-Fe. The apparent activation energy for creep of this dispersion strengthened steel, however, is about 11,000 cal./mole higher than that for creep of α-Fe.

2. The secondary creep rate $\dot{\gamma}s$ for this dispersion strengthened steel was found to obey the relationship

$$\frac{\dot{\gamma}s\ kT}{DGb\, e^{-Hc/RT}} = A\left(\tau/G\right)^{6.8}$$

where the symbols have their usual meaning as described in the text of this report. Whereas, $Hc \simeq 11{,}000$ cal./mole and $A = 6 \times 10^{15}$ for the dispersion strengthened steel, $Hc = 0$ and $A = 2 \times 10^{17}$ for α-Fe.

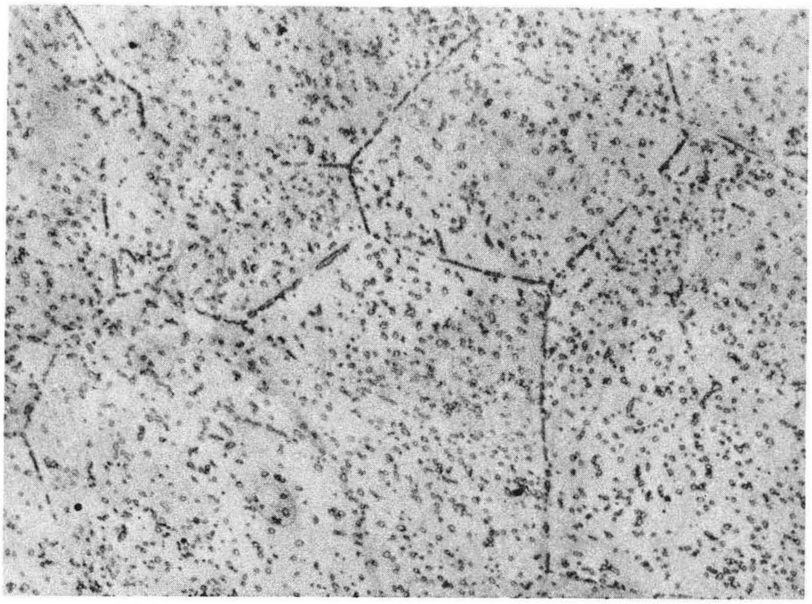
3. The increased creep resistance of the dispersion strengthened steel over that for α-Fe arises principally from the value of Hc. The physical origin of this term, however, is not known.

ACKNOWLEDGEMENT

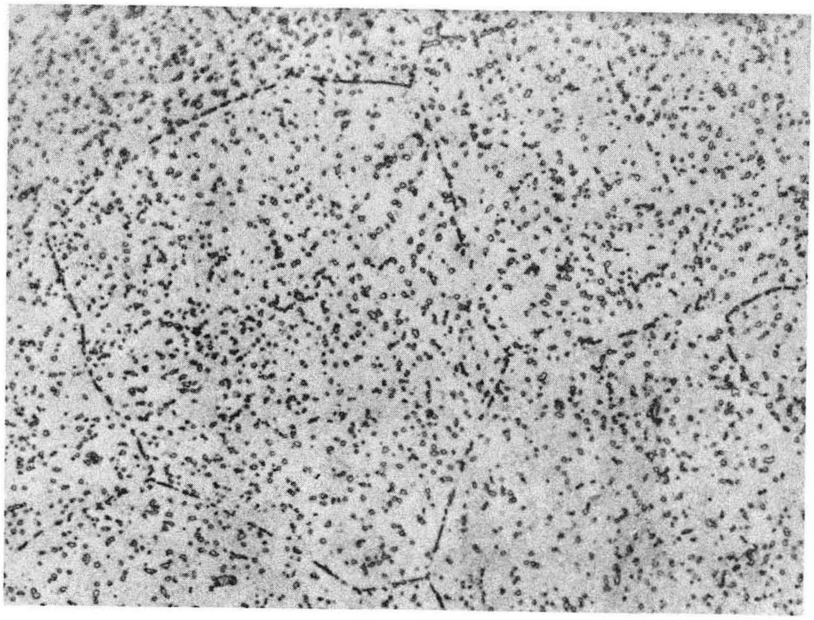
The authors would like to express their appreciation to the United States Atomic Energy Commission for their support of this research through the Inorganic Materials Research Division of the Lawrence Radiation Laboratory of the University of California. Thanks are also due to Dr. Maximo Victoria for his kind help on the experimental phases, and Mr. James E. Bird for his invaluable discussions during the preparation of this manuscript.

REFERENCE

1. A. K. Mukherjee, J. E. Bird, and J. E. Dorn, "Experimental Correlations for High-Temperature Creep", UCRL Report 18526, (October, 1968).
2. B. A. Wilcox and A. H. Clauer, "High-Temperature Creep of Ni-ThO_2 Alloys", in "Oxide Dispersion Strengthening" G. S. Ansell, T. D. Cooper, and F. V. Lenel, eds. New York, Gordon and Breach, 1968.
3. B. A. Wilcox and A. H. Clauer, Trans. AIME, 236 570 (1966).
4. P. Guyot, Acta Met., 12, 665 (1962).
5. G. Ansell and J. Westman, Trans. AIME, 215, 838 (1959).
6. C. L. Meyers, J. C. Shyne and O. D. Sherby, J. Australian Inst. of Metals, 8, 171 (1963).
7. Y. Ishida, C. Y. Cheng, and J. E. Dorn, Trans. AIME, 236, 964 (1966).
8. R. W. K. Honeycombe, H. J. Harding, and J. J. Irani in "High Strength Materials" V. Zackay ed., P. 213 (1966).
9. D. M. Schwartz, J. R. Mitchell and J. E. Dorn, Acta Met. 15, 485 (1967).
10. D. Y. F. Lai and R. J. Borg, Trans. AIME 233, 1973 (1965).
11. D. Y. F. Lai and R. J. Borg "The Diffusion of Iron in Various Ferrous Alloys" Lawrence Radiation Laboratory, Livermore Dept. UCRL-50314 (1967).
12. W. Köster, Z. Metall Kunde 39, 1 (1948).
13. Axel Fuchs, Dissertation "Das Kriech Verhalten von Eisen Molybdan Misch Kristallen in Temperatur Bereich von 500° bis 1000°C," Der Jechnischen FaKultat der Universitat, Erlangen, Nurnburg, Erlangen (1968).



1-a. Before



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1-b. After

Fig. 1 Microstructure of the alloy
a. Before creep test
b. After creep test at 952°K

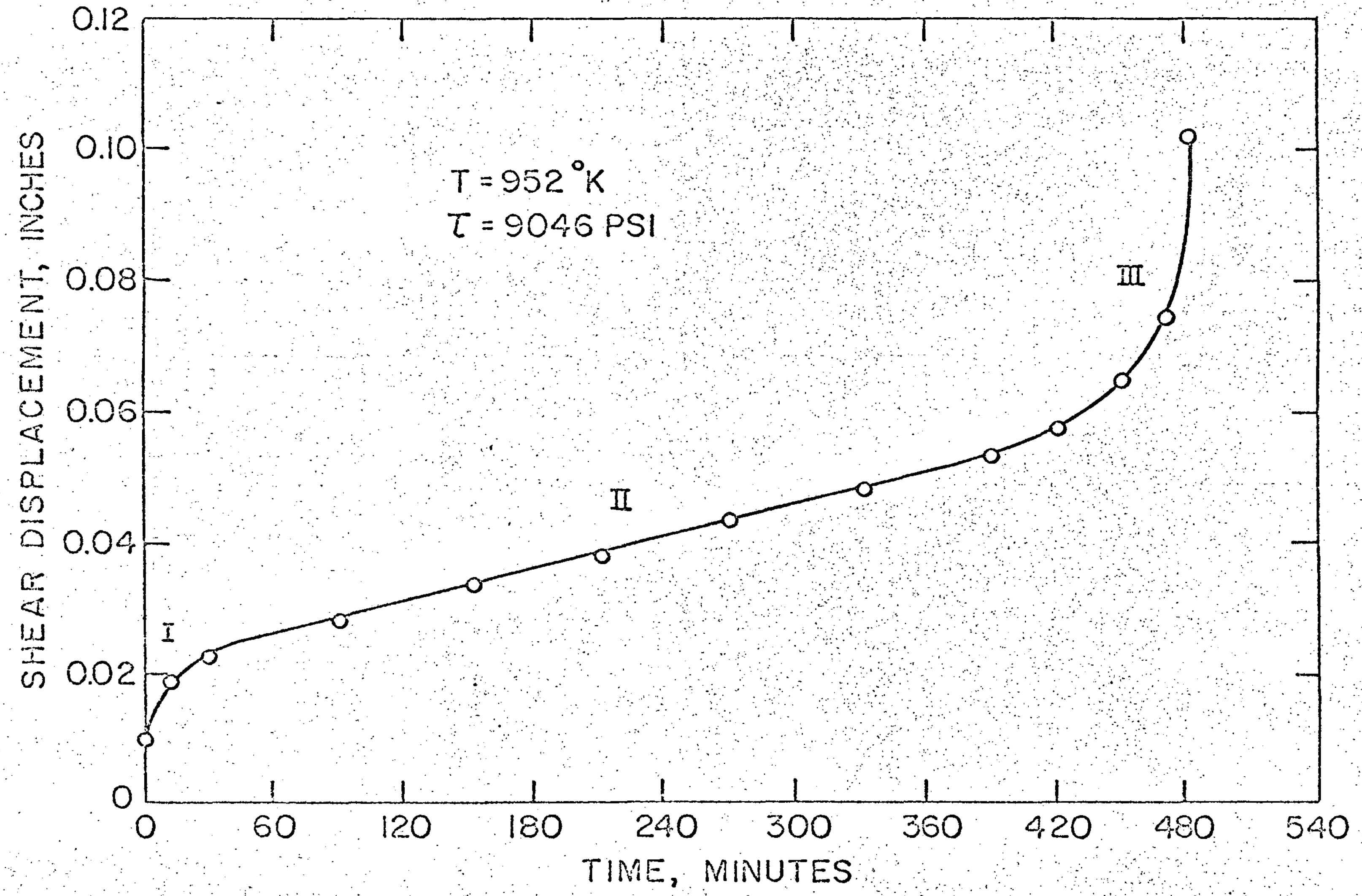

FIG. 2 TYPICAL CREEP STRAIN VERSUS TIME.

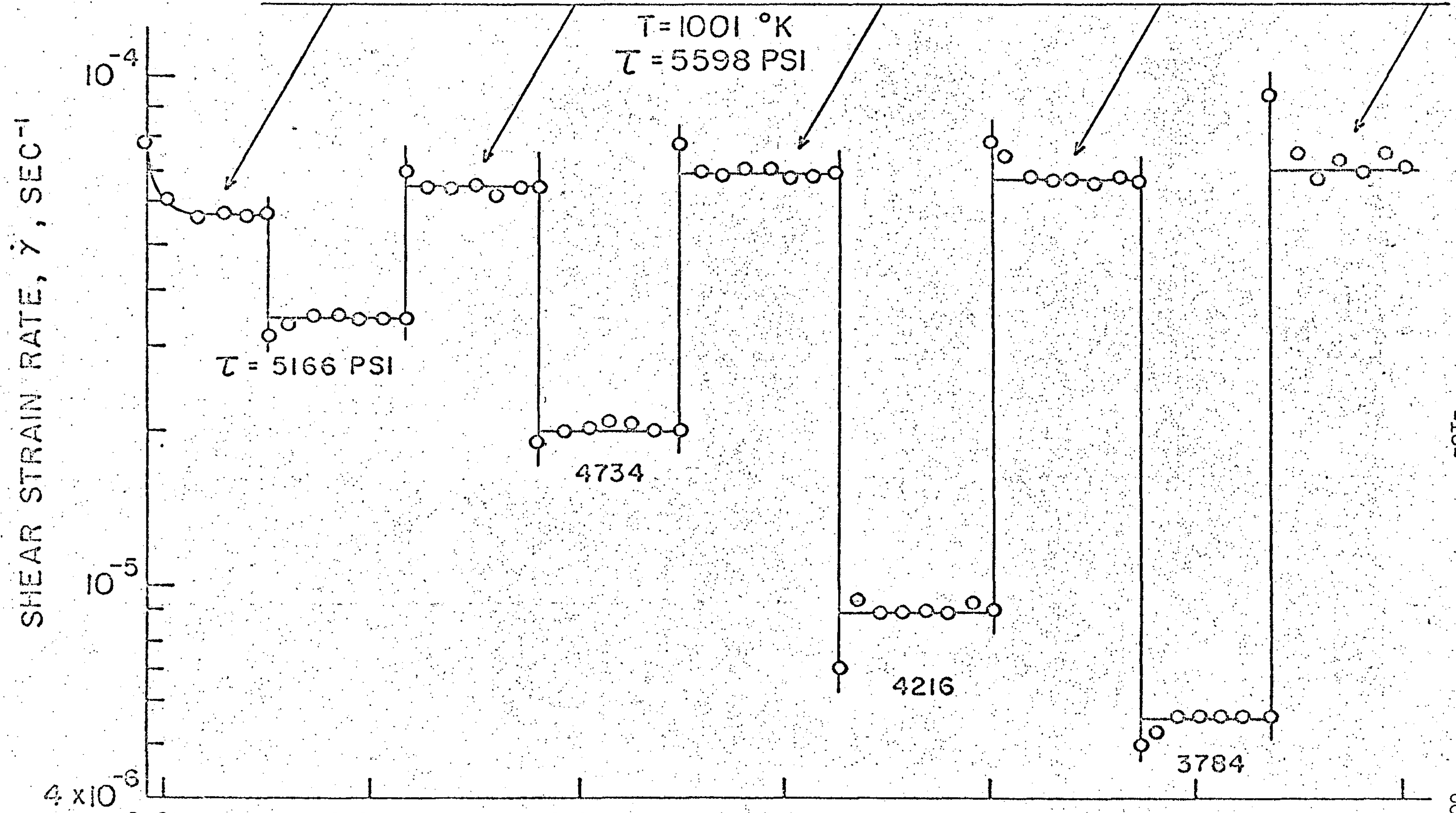

FIG. 3 TYPICAL CREEP RATE - EXTENSION DATA FOR CYCLIC STRESS TESTS.

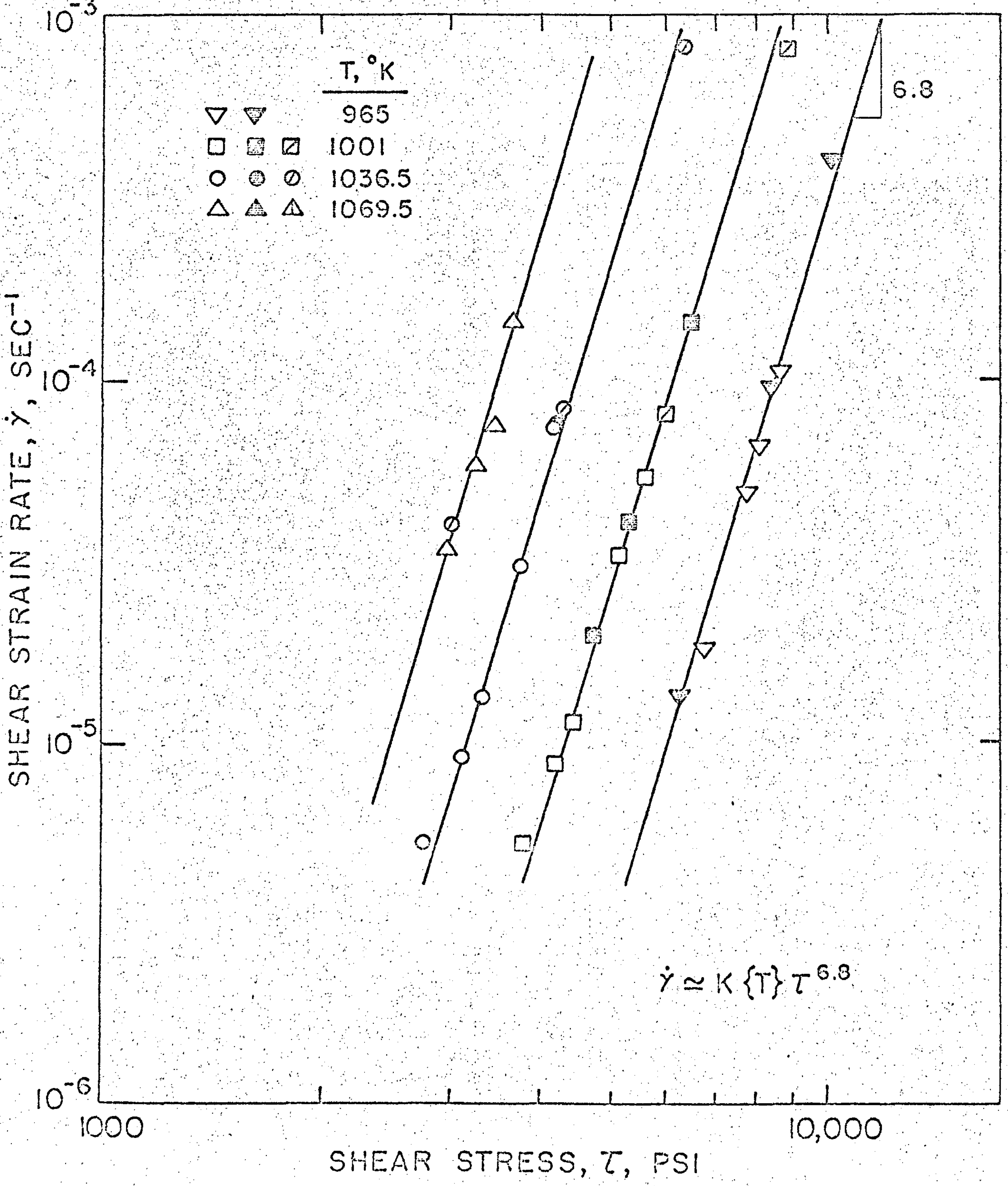

FIG. 4 EFFECT OF STRESS ON THE SECONDARY CREEP RATE.

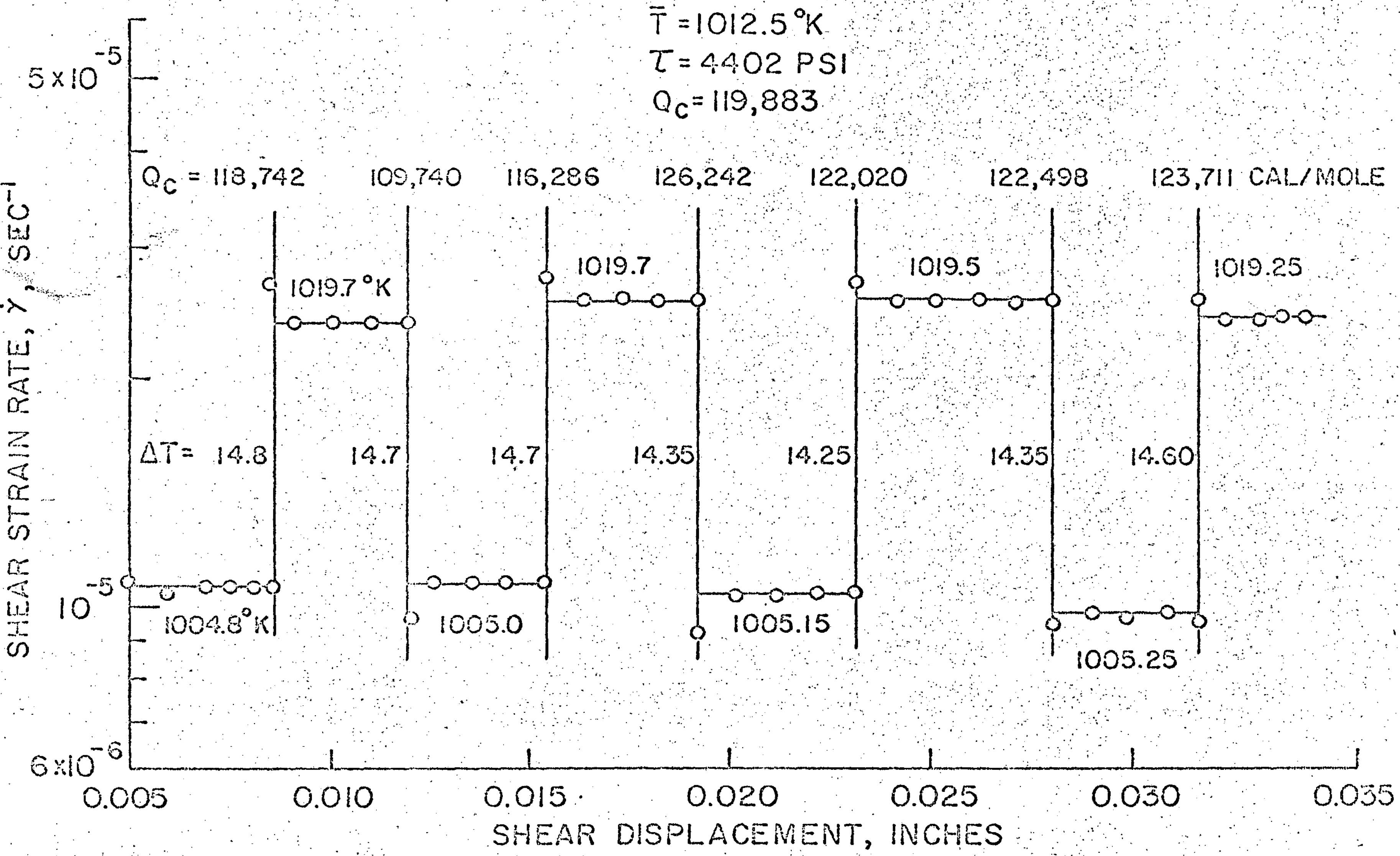

FIG. 5 TYPICAL CREEP RATE-EXTENSION CURVES FOR CYCLIC TEMPERATURE TESTS.

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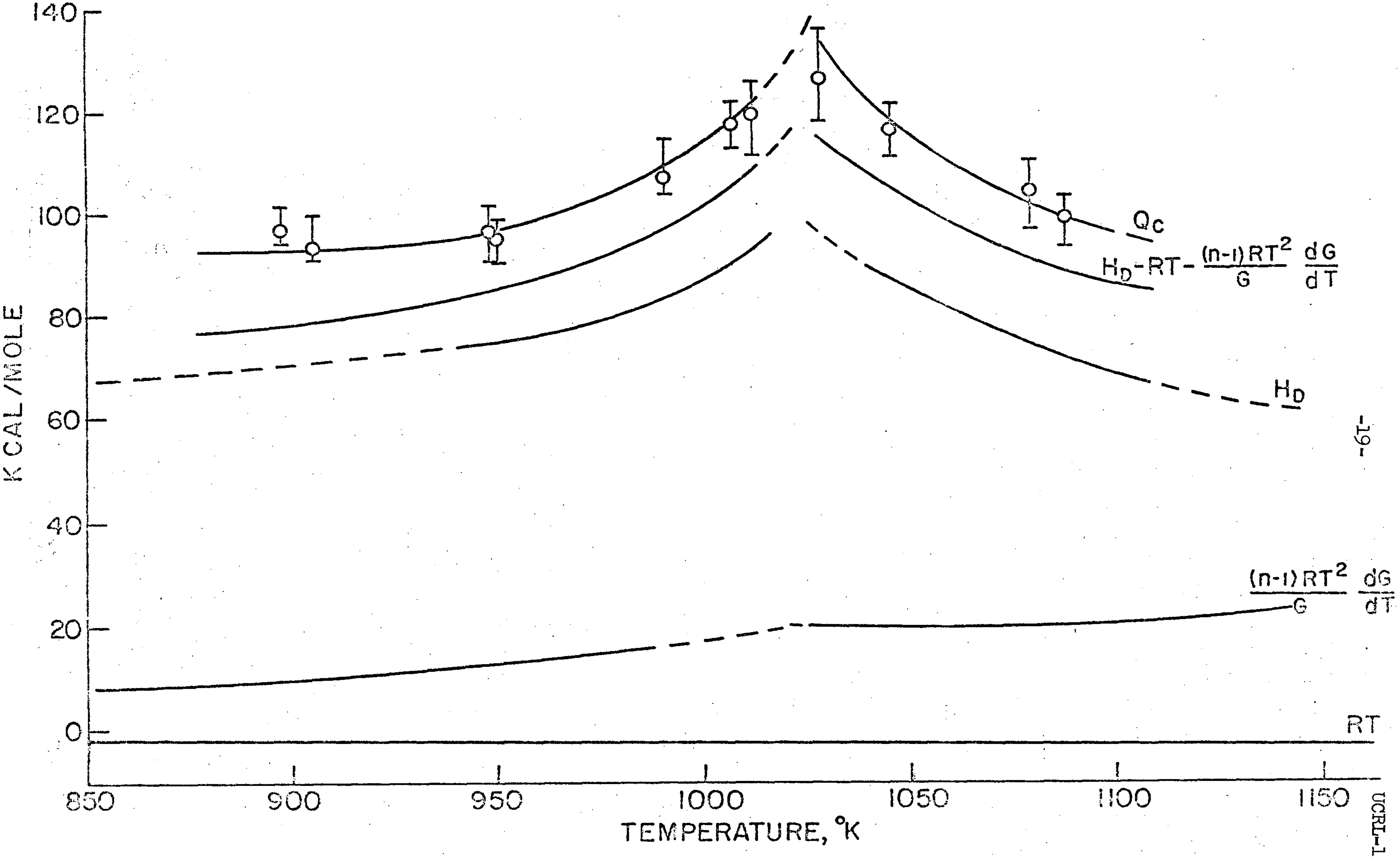

FIG. 6 TEMPERATURE DEPENDENCE OF Q_C OF Mo-STEEL, H_D AND G OF α-Fe.

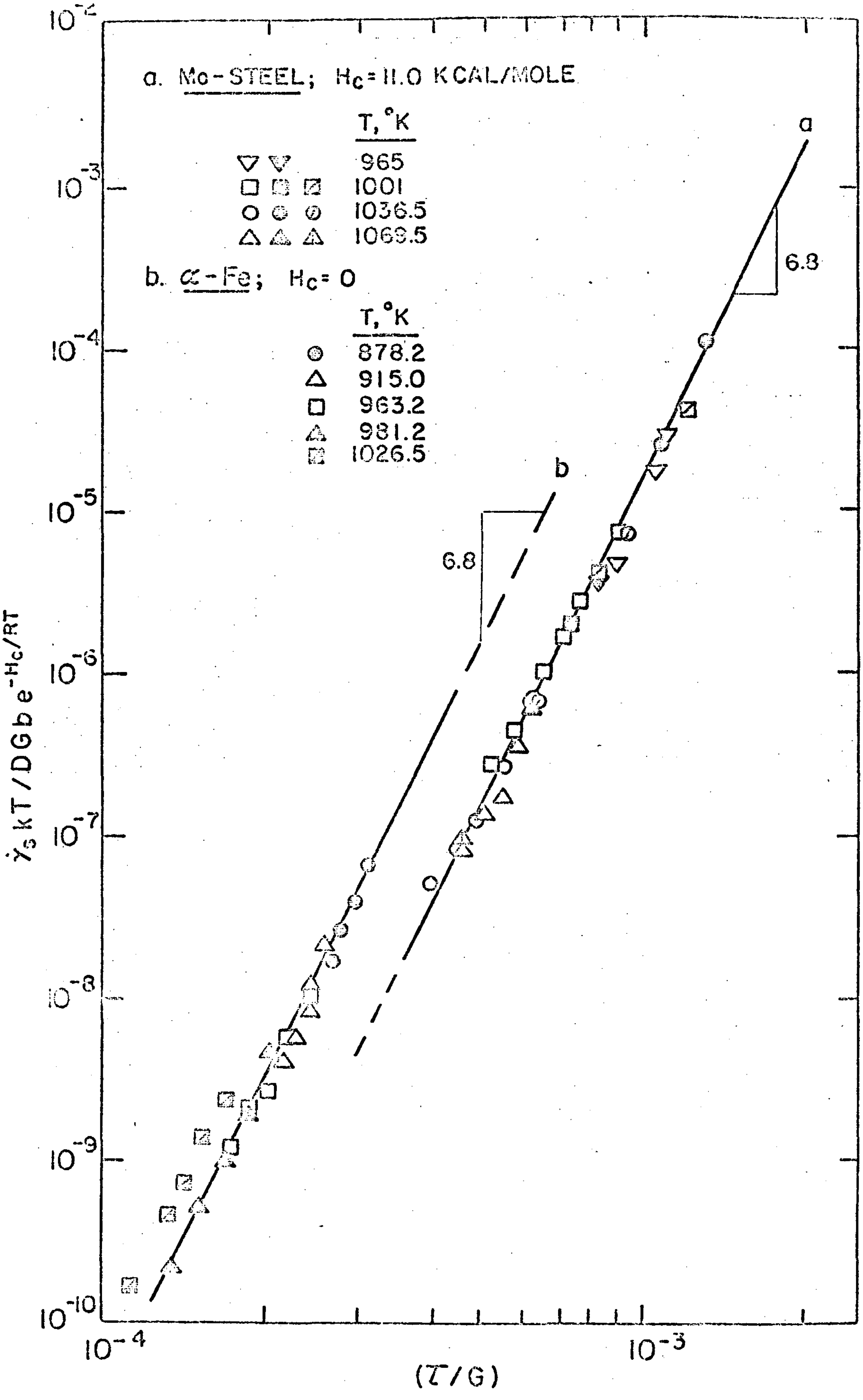

FIG. 7 EFFECT OF G, D, T AND b ON THE SECONDARY CREEP RATE OF Mo-STEEL AND α-Fe.

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