UCRL-11290 Rev.

University of California

Ernest O. Lawrence Radiation Laboratory

EFFECT OF STRAIN-HARDENING ON THE LOW TEMPERATURE THERMALLY-ACTIVATED DEFORMATION MECHANISMS IN POLYCRYSTALLINE ALUMINUM

TWO-WEEK LOAN COPY

This is a Library Circulating Copy which may be borrowed for two weeks. For a personal retention copy, call Tech. Info. Division, Ext. 5545

Berkeley, California

DISCLAIMER

This document was prepared as an account of work sponsored by the United States Government. While this document is believed to contain correct information, neither the United States Government nor any agency thereof, nor the Regents of the University of California, nor any of their employees, makes any warranty, express or implied, or assumes any legal responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by its trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof, or the Regents of the University of California. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof or the Regents of the University of California.

To be published in Trans. ASM

UCRL-11290-Rev.

University of California
Lawrence Radiation Laboratory
Berkeley, California

AEC Contract No. W-7405-eng-48

EFFECT OF STRAIN-HARDENING ON THE LOW TEMPERATURE THERMALLY-ACTIVATED DEFORMATION MECHANISMS IN POLYCRYSTALLINE ALUMINUM

A. C. Nunes, Jr., A. Rosen, and J. E. Dorn

September 18, 1964

EFFECT OF STRAIN-HARDENING ON THE LOW TEMPERATURE THERMALLY-ACTIVATED DEFORMATION MECHANISM IN POLYCRYSTALLINE ALUMINUM

A. C. Nunes, Jr., A. Rosen, and J. E. Dorn

Inorganic Materials Research Division, Lawrence Radiation Laboratory, and Department of Mineral Technology, College of Engineering University of California, Berkeley, California

September 18, 1964

ABSTRACT

The low temperature thermally-activated deformation mechanisms in polycrystalline aluminum are investigated through measurements of flow stress vs temperature for various strain-hardened states.

At low temperatures, the results are in nominal agreement with the intersection mechanism. At higher temperatures a thermal recovery mechanism operates.

I. INTRODUCTION

It has now been well-established that the plastic deformation of pure F.C.C. metals at low temperatures is facilitated by thermal fluctuations. Most investigations on the thermally activated process appear to substantiate the assumption that the operative strain rate controlling mechanism is that dictated by intersection of dislocations.[1, 2, 3, 4] Although various intersection energies are involved depending on the statistical distribution of dislocations, it is now generally agreed that, on the average, the strain rate can be reasonably accurately described by[1]

$$\dot{\gamma} = Ke^{-\left(\frac{U}{kT}\right)} \tag{1}$$

where $\dot{\gamma}$ = shear strain rate,

K = a frequency factor that is dependent on the statistical distribution of intersection dislocations,

k = the Boltzmann constant

T = the absolute temperature, and

U = the energy, on the average, that must be supplied by a thermal fluctuation to complete an intersection.

The value of K is estimated to be about[1, 5, 6]

$$K = NAb\left(\frac{\nu b}{L}\right) \tag{2}$$

where N = the number of points per unit volume where intersection is imminent

A = the average area swept out per activated dislocation segment

b = the Burgers vector

ν = the Debye frequency (about $8.0 \times 10^{-12} sec^{-1}$ for aluminum)

L = the average distance between points of intersection where dislocations are temporarily arrested.

Furthermore, the value of U is a function of the applied stress, the dislocations patterns and arrays and the constriction and jog energies. For high stacking fault metals, such as high purity aluminum, where the constriction energy is small, the activation energy, U, can be given by Seeger's approximation[1]

$$U = U_i - (\tau - \tau^*) Lb^2 = U_i - (\tau - \tau^*) v \qquad (3)$$

where U_i = the total energy for intersection,

τ = the applied shear stress,

τ^* = the equivalent stress needed to overcome the athermal interactions between dislocations,

$(\tau - \tau^*)Lb^2$ = approximately the work done by the applied stress in facilitating the completion of intersections, and

v = the apparent activation volume.

Furthermore, both U_i and τ^* vary with the test temperature as does the shear modulus of elasticity[6] so that

$$U_i = U_{io} \frac{G}{G_o} \text{ and } \tau^* = \tau_o^* \frac{G}{G_o} \qquad (4)$$

where U_{io} = the total intersection energy at the absolute zero,

τ^* = the equivalent athermal stress at the absolute zero,

G = the shear modulus of elasticity at T, and

G_o = the shear modulus of elasticity at the absolute zero.

A brief reflection reveals that Eqs. 1-4 have more parameters than can be deduced from simple mechanical tests that involve only

changes in the externally adjustable variables of strain rate and temperature. Consequently, in previous discussions of the problem, certain simplifying assumptions, which were not necessarily valid, were made to facilitate analyses. A striking example of this concerns the frequently applied assumption that K is independent of the strain-hardened state.[3,4] It was the major objective of this investigation to ascertain how K, v, and τ^* actually vary with the strain hardening. It will be demonstrated that Eqs. 1-4 describe the thermally activated low-temperature strain-rate mechanism for high purity polycrystalline aluminum with good accuracy; and it will be shown that the parameters of Eqs. 1-4 agree well with those expected when the rate-controlling mechanism is that for intersection of dislocations; and K will be shown to depend on the strain-hardened state; furthermore, the variation of the apparent activation volume and the approximate variation of τ_o^* with strain-hardening will also be deduced; and it will be further demonstrated that a moderately low-temperature recovery of the strain-hardened state, which had not been previously recognized, also complicates the analysis.

II. EXPERIMENTAL METHODS AND RESULTS

High purity (99.99 wt.%) aluminum was selected for this investigation because the constriction energy of its dislocations is small and, therefore, Seeger's approximation, Eq. 3, is expected to be good. Spectrochemical analyses gave the following impurities in weight percent: 0.004 Cu, 0.002 Fe, 0.001 Si, and less than 0.001 of the remaining elements. The material was received in the form of 0.100 inch thick sheet cold rolled to the H-18 temper from which sheet tensile specimens having a 0.250 inch width and a 1.625 inch gage length were machined with the tensile axis in the rolling direction. To remove the effects of cold rolling and machining, all specimens were annealed in a potassium nitrate-nitrite bath at 680°K for 10 minutes. The resulting average grain diameter was 0.26 mm.

The testing was conducted on an Instron Tensile Testing Machine; a typical shear stress vs shear strain curve at 370°K for a shear-strain rate of $\dot{\gamma} = 3.06 \times 10^{-5}$ per second for the annealed material is shown in Fig. 1. The shear stress was taken to be the maximum shear stress or one-half of the tensile stress and the shear-strain rate at three-halves of the tensile strain rate in accordance with a Mohr's circle strain transformation for a plastic deformation with Poisson's ratio taken as one-half. Various cold-worked states designated by the flow stress at a to e in Fig. 1, were prepared by straining preliminary to determination of the subsequent yield strengths at a series of lower temperatures, and at the two shear-strain rates of 3.06×10^{-5} and 3.06×10^{-3} per second. In this way, the specimens were placed in a series of well-defined cold-worked states.

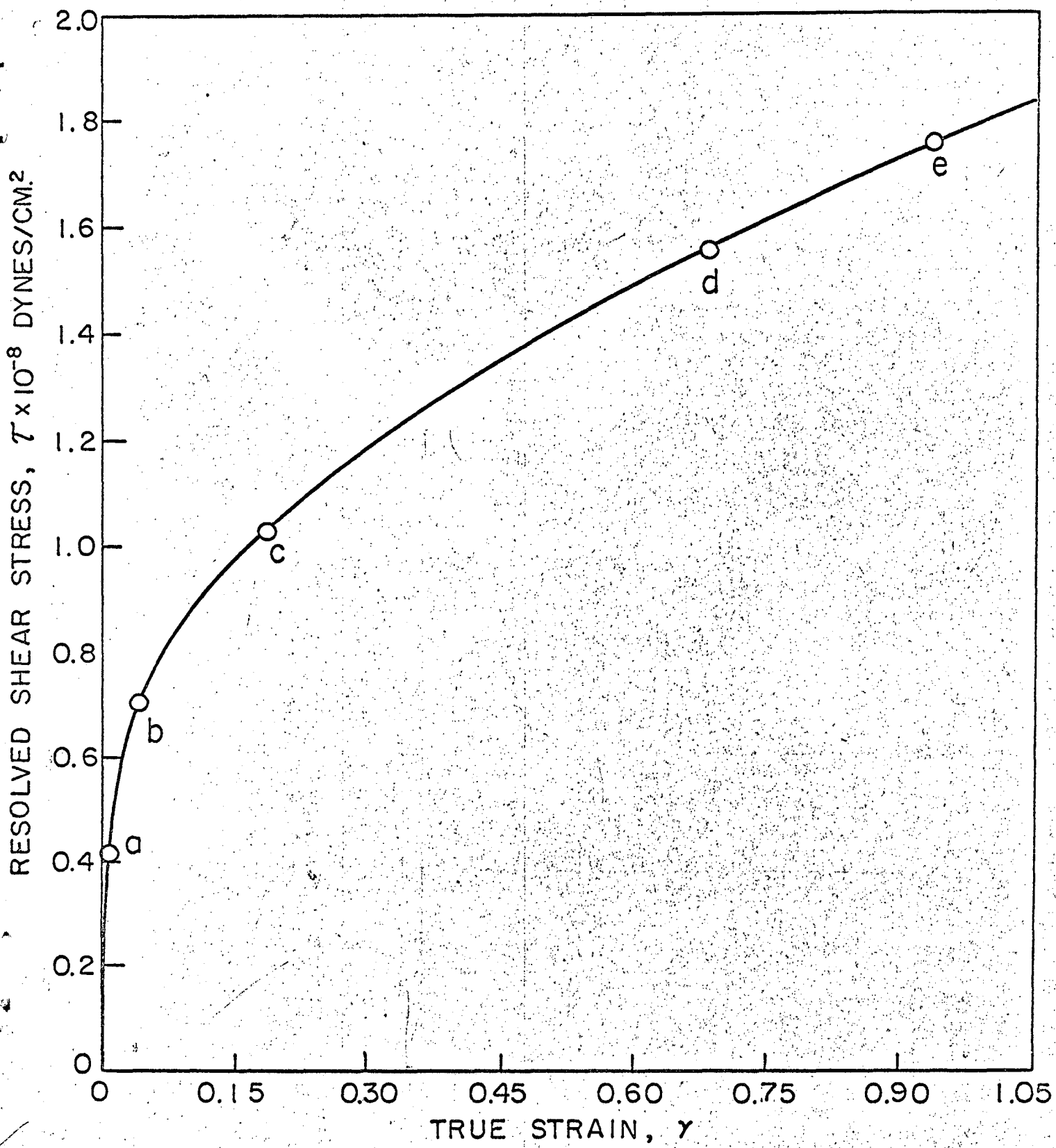

FIG. 1. RESOLVED SHEAR STRESS - TRUE STRAIN AT 370°K. SHEAR STRAIN RATE = 3.06×10^{-5} PER SECOND.

Following prestressing at a strain rate of $\dot{\gamma} = 3.02 \times 10^{-5}$ per second at 370°K, the controlled-temperature warm-water bath was removed and substituted by various constant temperature isothermal baths. The tests at liquid helium temperature were conducted with the aid of a specially designed liquid helium cryostat which was fitted to the Instron Testing Machine. After reaching thermal equilibrium at the reduced temperature, testing was resumed to determine the yield stress for the various fixed cold-worked states as a function of temperature for the two previously designated strain rates. The flow stress at yielding was determined by applying the 0.1% offset from the modulus line concept. The results of the tests are recorded in Fig. 2.

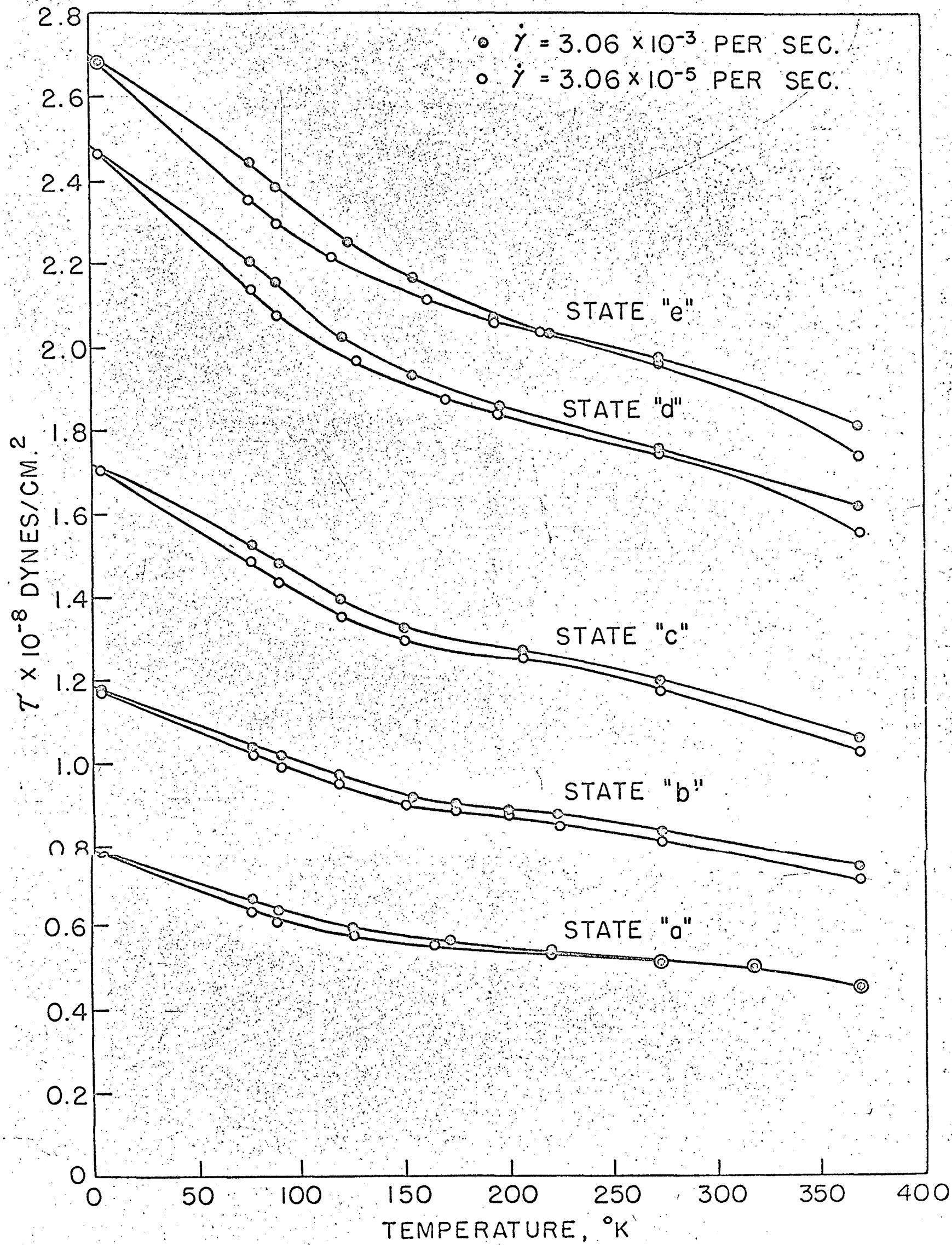

FIG. 2. FLOW STRESS vs. TEMPERATURE FOR DIFFERENT STATES.

III. DISCUSSION OF RESULTS

Eqs. 1, 3 and 4 suggests that

$$\frac{\tau G_o}{G} = \tau_o^* + \frac{U_{io}}{v} - \frac{kT}{v}\frac{G_o}{G}\ln\frac{K}{\dot{\gamma}} \quad \text{for } T < T_c \tag{5}$$

and that

$$\frac{\tau G_o}{G} = \tau_o^* \quad \text{for } T > T_c \tag{6}$$

where T_c is defined as

$$U_{io} = kT_c\frac{G_o}{G}\ln\frac{K}{\dot{\gamma}} \tag{7}$$

So that above T_c the thermal fluctuations are sufficiently frequent and great to induce intersection without requiring the mechanical aid of an overstress $\tau - \tau_o^*$. Curves of $\tau G_o/G$ vs T for the two strain rates and for the various strain-hardened states, given in Fig. 3, were deduced from the data of Fig. 2 using the data of Sutton[7] for determining the shear modulus on the (111) slip plane in the direction of the Burgers vector a/2 [$\bar{1}$01] as a function of T. Whereas the general trends of a linear decrease of $\tau G_o/G$ with T over the low temperature range and an insensitivity of $\tau G_o/G$ over the higher temperature range are in nominal agreement with the dictates of Eqs. 5 and 6, minor variations from the theoretical predictions are evident. First the values of $\tau G_o/G$ over the higher temperature range (excepting the intermediate temperature range for state e) decrease slightly with an increase in temperature; furthermore, the flow stress is slightly strain rate sensitive over the same ranges. Therefore, over the higher temperature range Eq. 6 is not wholly accurate. Since the decrease in $\tau G_o/G$ with increase in temperature appears to be more pronounced for the higher work-hardened states, a recovery mechanism appears to be operative over the higher temperature

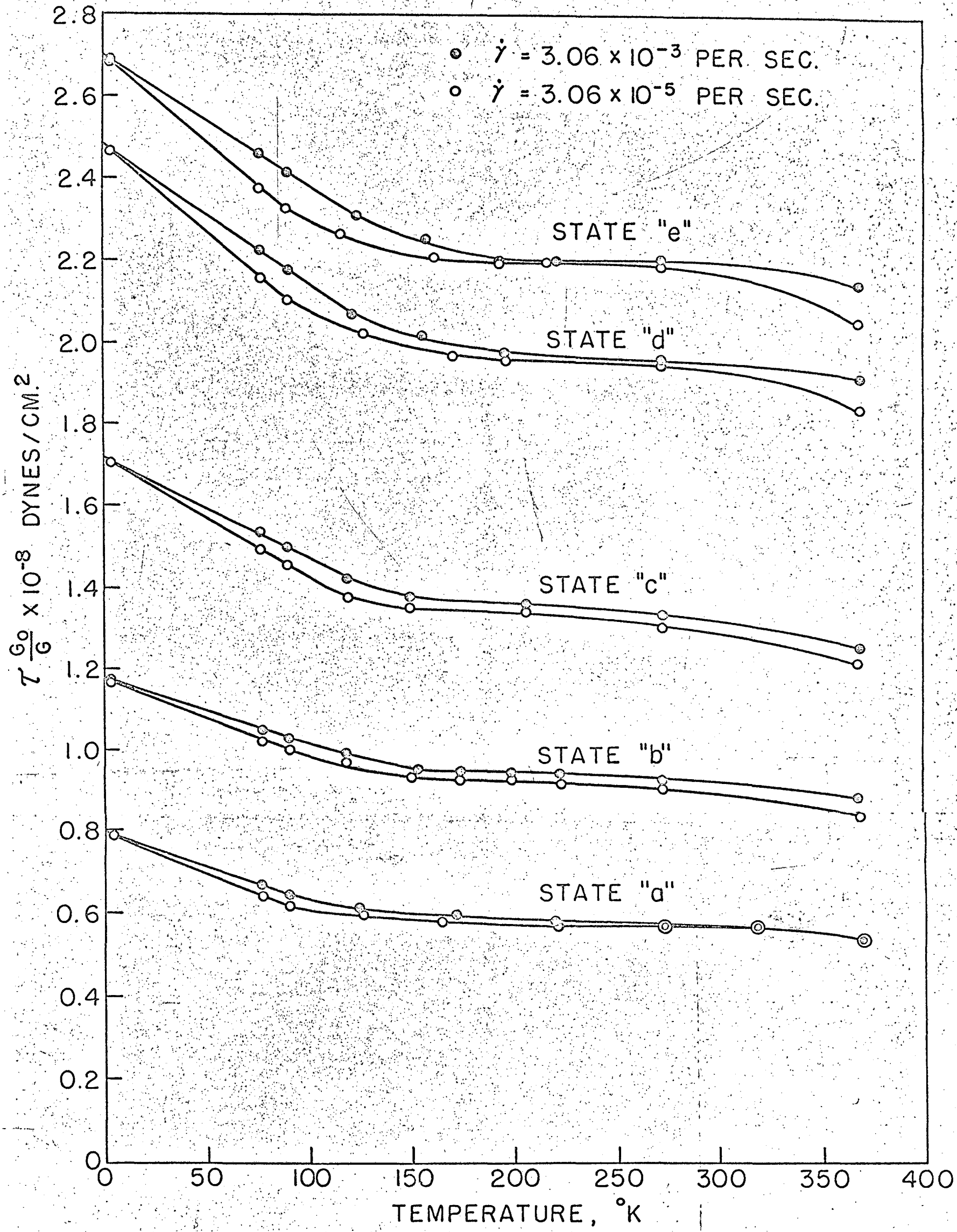

FIG. 3. FLOW STRESS vs. TEMPERATURE CORRECTED FOR THE VARIATIONS OF THE SHEAR MODULUS.

range. But the magnitude of this recovery decreases as the temperature decreases, especially as shown for the highest strain-hardened state e, suggesting that it has only a modest influence on the lower temperature flow stress data. Second, the linear portion of $\tau G_o/G$ vs T at low temperatures appears to fair into the more or less horizontal $\tau G_o/G$ vs T trends at the higher temperatures. This may arise from the effects of stress on the small constriction energy in aluminum. Equation 5, however, represents the experimental data very well over the lowest temperature region.

As shown by Eq. 5, the slopes of the $\tau G_o/G$ vs T curves at the low temperatures for the two strain rates that were employed permit the determination of K and v for the various prestressed states. Because of the introduction of recovery over the higher temperature range τ_o^* was not so easily obtained. However, the insensitivity of $\tau G_o/G$ to temperature and strain rate for state e over the intermediate range of temperatures reveals that this is also the appropriate value of τ_o^* for this state. Furthermore, it follows from Eq. 5 that for the extrapolated value of τ at the absolute zero, namely τ_o

$$U_{io} = v\,(\tau_o - \tau_o^*) \tag{8}$$

where, on the average U_{io} is a constant. Therefore for any state a

$$v_a(\tau_o - \tau_o^*)_a = v_e(\tau_o - \tau_o^*)_e \tag{9}$$

Thus since $v_e(\tau_o - \tau_o^*)_e$ is known and v_a and $\tau_{o\,a}$ are also known $\tau_{o\,a}^*$ could be estimated for each strain-hardened state a. The values of τ_o and τ_o^* deduced in this way are given as functions of the prestress τ_p in Fig. 4, were as shown $\tau_o - \tau_o^*$ is U_{io}/v. Since U_{io} is constant,

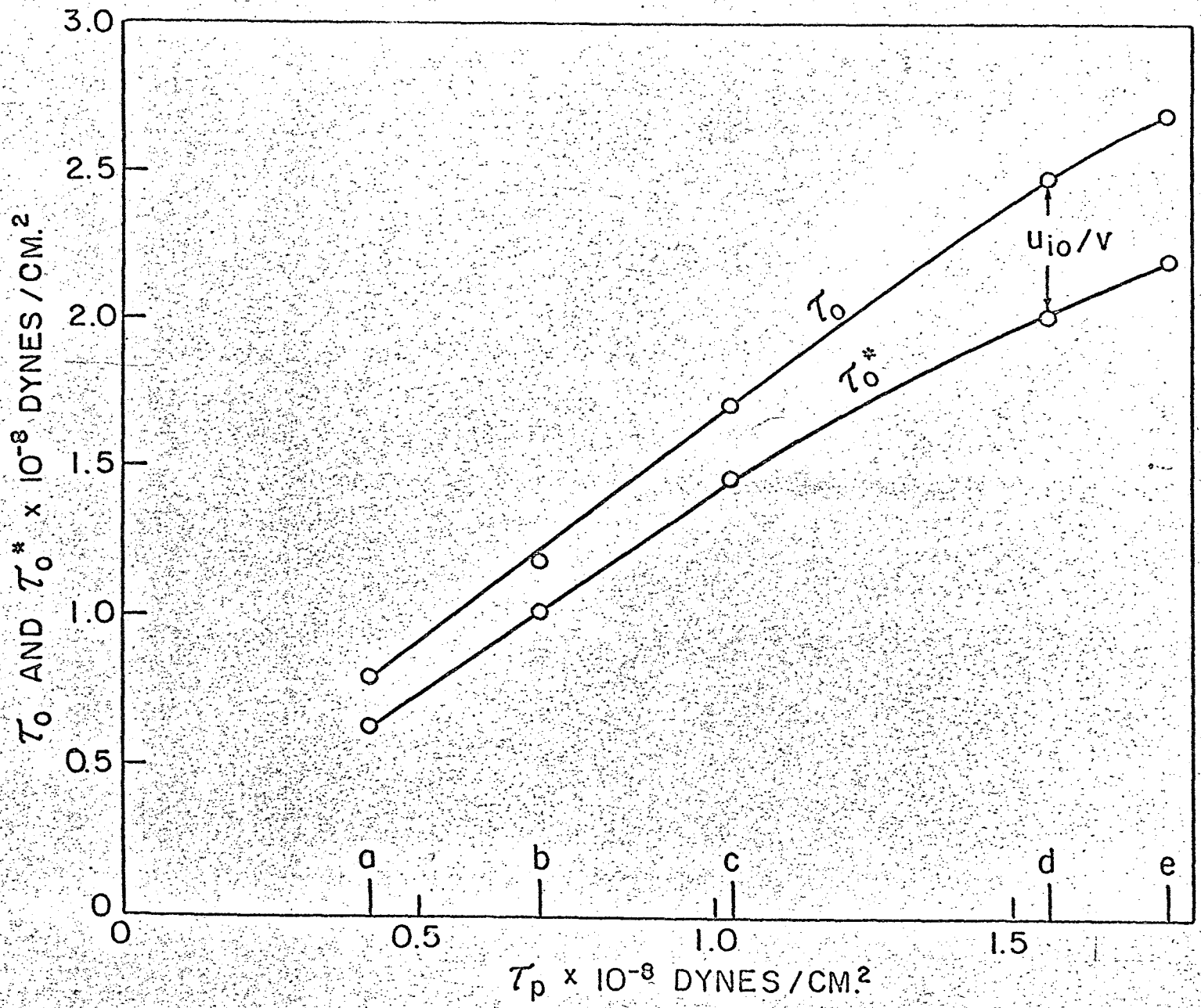

FIG. 4. LONG RANGE BACK STRESS (τ_o^*) AND FLOW STRESS (τ_o) AT ABSOLUTE ZERO vs. PRESTRESS (τ_p).

the increasing value of $\tau_o - \tau_o^*$ with τ_p arises from the fact that the apparent activation volume v decreases with strain hardening due to higher densities of dislocations. The τ_o^* vs τ_p relationship deviates slightly from the idealized expectation of a straight line. Slight additional recovery during reduction of the stress at the prestress temperature is probably responsible for this trend.

The apparent activation volume v, and the value of lnK were determined for each strain-hardened state by equating the slopes of $\tau G_o/G$ vs T curves for the two strain rates that were employed to the slopes dictated by Eq. 5. As shown in Fig. 5, the apparent activation volume remained practically unchanged over the early stages of strain hardening from (a) to (b) and then decreased rapidly with additional prestressing from (b) to (e). Consequently, the early stages of strain hardening of polycrystalline aluminum arise principally from an increase in τ_o^*, whereas, the decrease in v at higher prestrained conditions also contributes to the strain-hardening at the lower test temperatures as documented in Fig. 4 for 0°K. Contrary to the investigators' expectations, K was found to change greatly with cold work. As shown in Fig. 6 for $K\nu^{-1}$ vs τ_o^* (vide Eq. 2) K is insensitive to prestressing from state (a) to (b); but it decreases precipitously with additional prestressing from (b) to (e). For the initial prestressing conditions, it is expected that $N \simeq L^{-3}$ and $A \simeq L^2$, whence from Eq. 2 it is expected that $K\nu^{-1} \simeq \frac{b^6}{L^6} = \frac{b^6}{v^2}$. When the values of b = 2.86 Å and v for low prestresses (Fig. 5) are introduced $K\nu^{-1} \simeq -13.6$ in satisfactory agreement with the values for the lowest prestressed states (a) and (b) given in Fig. 6. Since N is

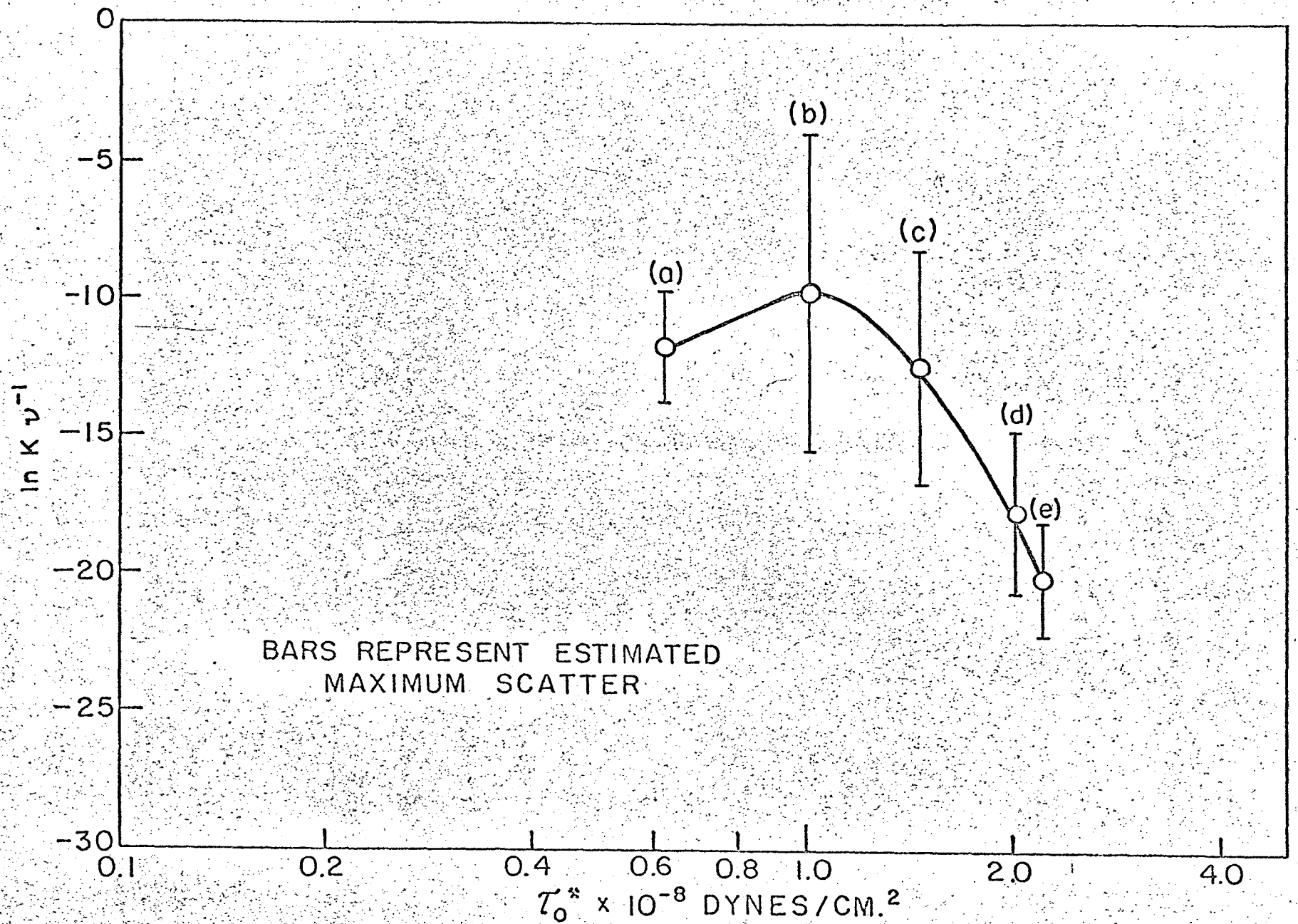

FIG. 6. NATURAL LOGARITHM OF DIMENSIONLESS FREQUENCY FACTOR ($\ln K\nu^{-1}$) vs. LONG RANGE BACK STRESS AT ABSOLUTE ZERO (τ_0^*)

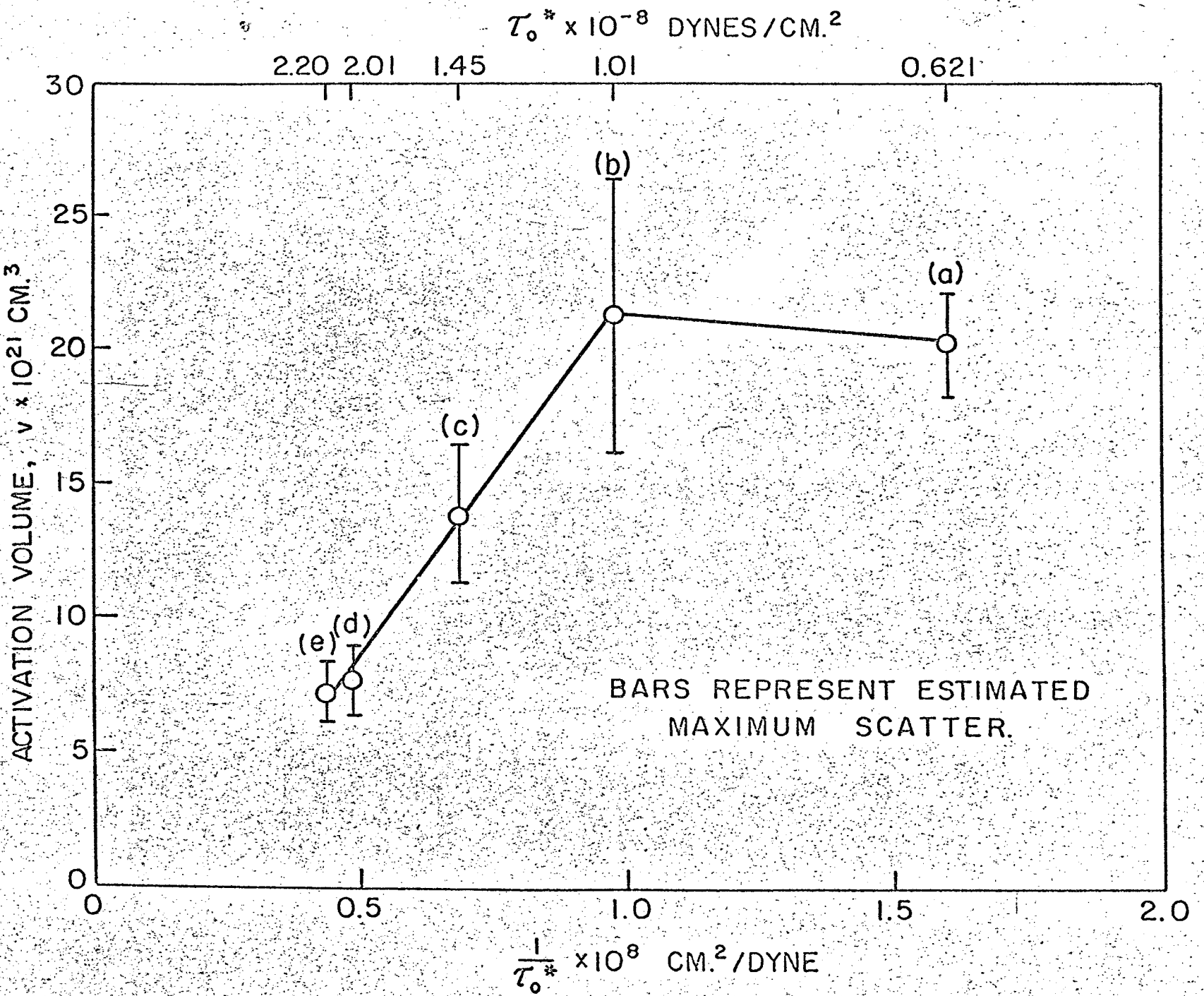

FIG. 5. ACTIVATION VOLUME vs. INVERSE τ_0^*.

expected to increase with the density of dislocations, the decrease in $K\nu^{-1}$ with prestressing from (b) to the higher prestressed states is probably attributable to a rapid decrease in A. This suggests that the area A swept out per activation is controlled by the distance between dislocations in entanglements. Thus these trends are in fair harmony with the dictates of the intersection mechanism.

An activation energy of U_o = 5110cal/mole was determined from the values of U_o/v given in Fig. 4 and the associated values of v given in Fig. 5. Thus, the average activation energy has the low value of $U_o \simeq 0.058 G_o b^3$. This result is much lower than that which would be expected for the rate-controlling mechanism of production of vacancies or interstitials of jogs in moving screw dislocations;[8, 9] it is only slightly lower than the expected value[5] of about $0.08 G_o b^3$ for the formation of a single jog in essentially undissociated dislocations. Consequently, this result also is in fair agreement with the intersection mechanism, the minor deviation from theory being accountable for the approximations in the theory and the neglect of statistical factors in formulating Eqs. 1, 2 and 3.

CONCLUSIONS

Summing up, it may be stated:

1. Two distinct regions of flow-stress temperature dependence exist in polycrystalline aluminum below 370°K.
2. The lower temperature region is thermally activated with an activation energy of about 5110cal/mole.
3. The activation volume of the lower temperature region ranges between 1000 b^3 and 250 b^3 decreasing with increasing prestress.
4. The frequency factor $\underline{K}$ varies between 10^{-4} and 10^{-10} of the Debye frequency, decreasing with higher prestresses.
5. The mechanical parameters are in nominal agreement with the intersection mechanism.
6. The flow-stress decreases slightly with temperature over the higher ranges of temperature even when corrected for temperature dependence of the elastic modulus and shows a corresponding strain rate dependence as a result of recovery.

ACKNOWLEDGMENTS

We wish to thank Dr. J. D. Mote for his help and advice in connection with the experiment and Mrs. Gloria Pelatowski for preparing the figures for the report.

This work was done under the auspices of the U.S. Atomic Energy Commission.

REFERENCES

1. A. Seeger, Z. Naturforschung., 9a, 758, 856, 870 (1954).
2. Z. S. Basinski, Phil. Mag., 4, 393 (1959).
3. S. K. Mitra, P. W. Osborne, and J. E. Dorn, Trans. Met. Soc. AIME, 221, 1206 (1961).
4. S. K. Mitra and J. E. Dorn, Trans. Met. Soc. AIME, 224, 1062 (1962).
5. J. Friedel, "Dislocation Interactions and Internal Strains," Physiques des Solides, Sorbonne, Paris, 220-262.
6. J. E. Dorn, "Energetics in Dislocation Mechanics," University of California, Lawrence Radiation Laboratory, Report UCRL-10455 (1963).
7. P. M. Sutton, Phys. Rev., 91, 816 (1953).
8. A. H. Cottrell, "Vacancies and Other Point Defects in Metals and Alloys," Institute of Metals, London, p. 1 (1958).
9. T. Broom and R. K. Ham, Ibid, p. 41 (1958).

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.