UCRL-17090

University of California

Ernest O. Lawrence Radiation Laboratory

THE MECHANISM OF BASAL CREEP
IN Mg-12 ATOMIC PERCENT Li

Berkeley, California

DISCLAIMER

UCRL-17090

UNIVERSITY OF CALIFORNIA

Lawrence Radiation Laboratory
Berkeley, California

AEC Contract No. W-7405-eng-48

THE MECHANISM OF BASAL CREEP IN Mg-12 ATOMIC PERCENT Li

Bernard Yves Chirouze
(M.S. Thesis)

September 9, 1966

THE MECHANISM OF BASAL CREEP IN Mg-12 ATOMIC PERCENT Li

Contents

THE MECHANISM OF BASAL CREEP IN Mg-12 ATOMIC PERCENT Li

Bernard Yves Chirouze

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

September 9, 1966

ABSTRACT

The effect of changes in stress and temperature on the creep rate of single hexagonal crystals of Mg-12 atomic % Li alloy, oriented for basal glide in simple shear by the {0001} $[1\bar{2}10]$ mode of slip, was determined over the temperature range of 500°K to 830°K.

In the middle of the range from 600° to about 750°K, the specimens, after a primary stage of short duration, entered secondary creep for which:

$$\dot{\gamma} = 1.6 \times 10^{-28} \tau^{4.9} e^{\frac{-37,000}{RT}} .$$

where $\dot{\gamma}$ is the shear strain rate per second, τ is the stress in dynes per square centimeter, R is the gas constant and T is the absolute temperature. These results strongly suggest that basal creep in this alloy, from 600° to 750°K, is controlled by the dislocation climb mechanism.

For temperatures less than or equal to 550°K the strain rate was found to increase very rapidly with the stress. Microscopic examination of the gage sections of the specimens showed that twinning occurred in this temperature range.

For temperatures greater than 800°K, the activation energy was found to increase substantially, indicating that some new mechanism becomes operative as the specimens approach the melting temperature.

I. INTRODUCTION

Investigations dealing with basal {0001} [$1\bar{2}10$] slip mechanisms in pure Mg have shown[1] that the strain-rate controlling mechanism for this mode at temperatures up to 350°K is that of thermally activated intersection of the glide plane dislocations with the "forest" dislocations threading through the slip plane.

Additions of Li from 8 to 12.8 atomic percent are known to modify pronouncedly the deformation characteristics of Mg at lower temperatures. In particular, over an intermediate range of temperatures, such alloys exhibit a much higher critical resolved shear stress (crss) than pure Mg. Furthermore, the flow stress for this new intermediate mechanism is insensitive to temperature. Such a region, having a crss ten times greater than that for pure Mg and extending from about 250°K to 450°K, has been found[2] for basal slip in the 12.5 atomic percent Li alloy; for temperatures above about 450°K the flow stress was shown to decrease rapidly with an increase in temperature, indicating that some thermally activated creep process again becomes rate controlling.

It was the purpose of this investigation to study the rate-controlling mechanism of basal slip in a 12.0 atomic percent Li alloy of Mg at high temperatures. It will be shown that viscous glide does not control the creep rate. However, over the temperature range from 600°K to about 750°K, creep appears to be controlled by the dislocation climb mechanism.

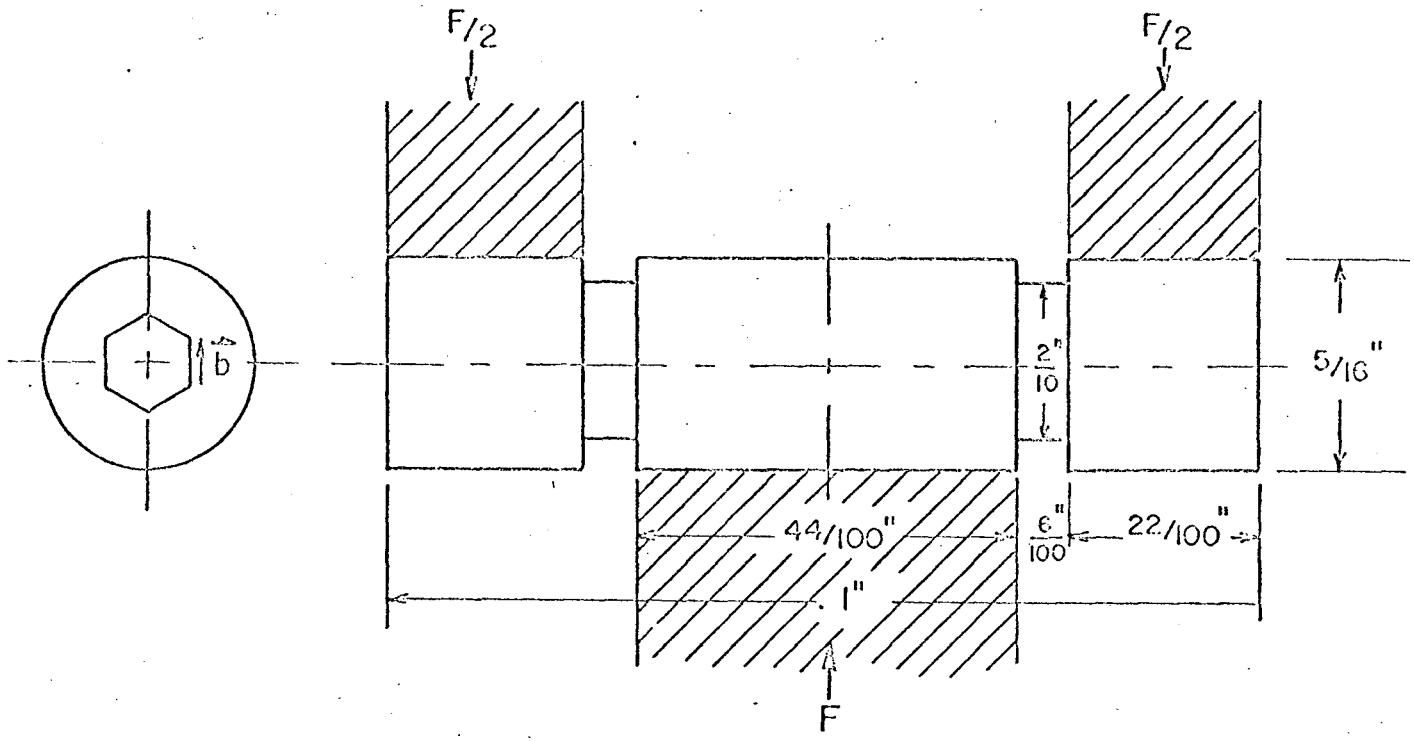

FIG. 1 GEOMETRY AND ORIENTATION OF CYLINDRICAL SHEAR CREEP SPECIMENS.

II. EXPERIMENTAL TECHNIQUE

Specimens were prepared by first growing spherical crystals in a graphite mold, orienting the spheres using the Laüe back-reflection x-ray method, machining the oriented cylindrical sphere with a spark-cutter, placing this seed at a bottom of a cylindrical steel mold, and then growing an oriented crystal rod using a modified Bridgeman technique.

The specimens contained an average 12.0 atomic percentage of Li. Because of the large composition difference between the liquidus and the solidus, and despite the fact that a large liquid metal reservoir was placed at the top of the steel mold, the single crystal rods showed a composition gradient of about .7 atomic percent per inch along their length.

Since tension creep tests for prismatic slip conducted on single crystals of this alloy have shown[3-5] that the specimens necked down immediately near the grip at which the Li content was the least, it was decided to try the double shear type of specimen shown in Fig. 1. This type of specimen appeared to be very satisfactory: (a) the shear strains obtained were uniform over each gage section and the same over both gage sections; (b) after a primary stage of creep of only a few minutes in duration, the creep rate remained uniform throughout the test up to strains of about 1.0; (c) the steady state creep rate was always found to remain unchanged after reversal of the direction of shearing.

Therefore each specimen was machined into the double shear type to the dimensions given in Fig. 1. Following that, their orientation was again checked, and their gage length and gage diameter determined with an accuracy of .0001 inches. Only specimens which were within 2° of the

desired orientation were used. Their composition is given in Table I.

All creep tests were conducted in an argon atmosphere. Furnace temperatures were held constant to ± .25°K by means of a proportional controller and specimen temperatures were determined by a pair of chromel-alumel thermocouples in direct contact with the edges of the gage sections of the specimens. One of these thermocouples was connected to the controller, and the other to a manual potentiometer. The stresses were applied with a lever arm with an accuracy of 10^4 dynes/cm^2; the strains were measured to 10^{-4} with a linear differential transformer.

The specimens entered secondary creep after strains of only .01. It was therefore possible to determine the effect of stress on the secondary creep rate by merely changing the stress during the testing of a single specimen. The same steady state creep rate was obtained for each stress regardless of the order in which the stress changes were effected. No instantaneous plastic strains were observed upon increase in stress and the transient strain upon change in stress was barely detectable and lasted less than one minute.

To determine the apparent activation energies for creep, small changes in temperature (15°K) were imposed and their effect on the steady state creep rate was considered. For such changes in temperature, the transient condition was never found to exceed 2 to 3 minutes in duration.

Table I. Composition of the specimens*

Specimen n°	wt% Li	at.% Li
#1	4.08	12.9
#2	3.33	10.8
#3	3.11	10.2
#4	3.25	10.6
#5	3.50	11.2
#6	4.07	12.8
#7	3.08	10.1
#8	4.15	13.1

* A representative figure of 12.0 has been chosen for the text.

III. EXPERIMENTAL RESULTS

Three typical creep strain-time curves are shown in Fig. 2. No initial plastic straining was observed at any temperature for any of the stresses that were applied here. As shown in the upper block of Fig. 2, a small primary creep stage of short duration occurred in all tests, but no tertiary creep was ever encountered, even though the maximum measured total creep strains were as high as 1.0. As shown in the body of Fig. 2, the creep rate was constant over most of the creep range under consideration. All further reported data refer to such a steady state creep rate.

The apparent activation energy, Q, in calories per mole, is defined by

$$Q = \frac{\partial \dot{\gamma}}{\partial(-1/RT)_{\tau}} \qquad (1)$$

where $\dot{\gamma}$ is the shear strain rate, R the gas constant, τ the applied stress, and T the test temperature in °K. Over the temperature range from 600°K to about 750°K, and over the stress range from 1.5×10^7 to 6×10^7 dynes/cm^2, a constant activation energy of about 37,000± 1000 cal/mole was obtained as shown in Fig. 3. In this range, the strain rate varied linearly with approximately the fifth power of the applied stress (vide Fig. 4). The slight differences obtained occasionally with different specimens could be attributed to differences in composition (Table I). All of the data are satisfactorily correlated by the relationship

$$\dot{\gamma} = (1.6 \pm .6) \times 10^{-28} \tau^{4.9 \pm .1} e^{\frac{-37,000 \pm 1000}{RT}} \qquad (2)$$

where $\dot{\gamma}$ is the strain rate per second, τ the applied stress in dynes/cm^2,

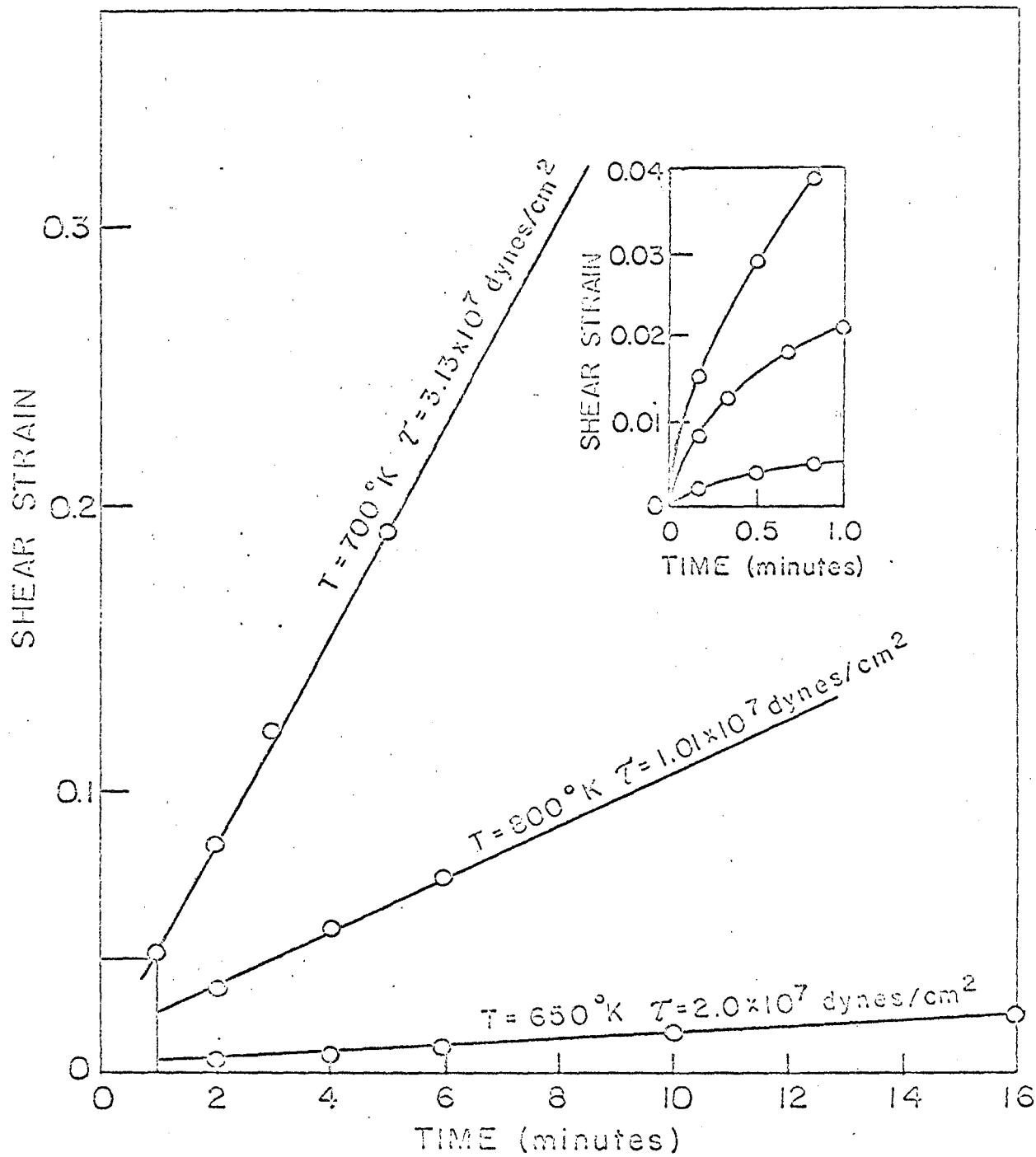

FIG. 2 TYPICAL CREEP STRAIN-TIME CURVES.

R is the gas constant in cal/deg K x mole, and T is the temperature in degrees Kelvin.

At temperatures less than or equal to 550°K and for stresses greater than 6×10^7 dynes/cm^2, the strain rate was found to depend on at least the twelfth power of the stress. However, microscopic examinations of the gage sections of the specimens after testing showed that some twinning occurred at these higher stresses and this may account for the observed extreme stress dependence of the strain-rate. No twinning could be detected for tests at the lower stresses.

At temperatures greater than or equal to 800°K, the activation energies increased rapidly, exhibiting a value of about 62,000 cal/mole at 830°K (Fig. 3), whereas the slope of the stress-strain rate curve decreased to 4.0, as shown in Fig. 4.

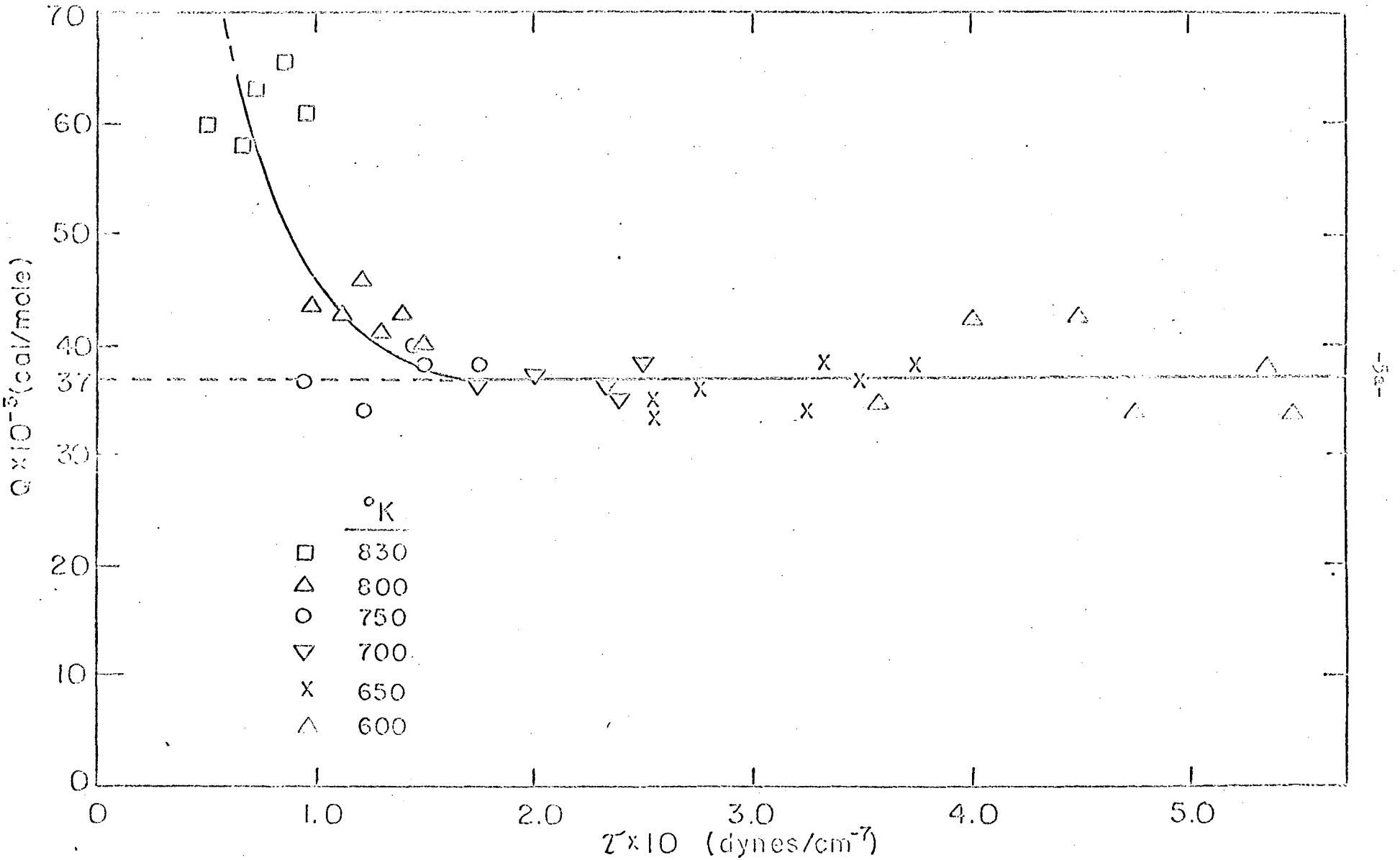

FIG. 3 EFFECT OF STRESS ON THE ACTIVATION ENERGY FOR STEADY STATE CREEP.

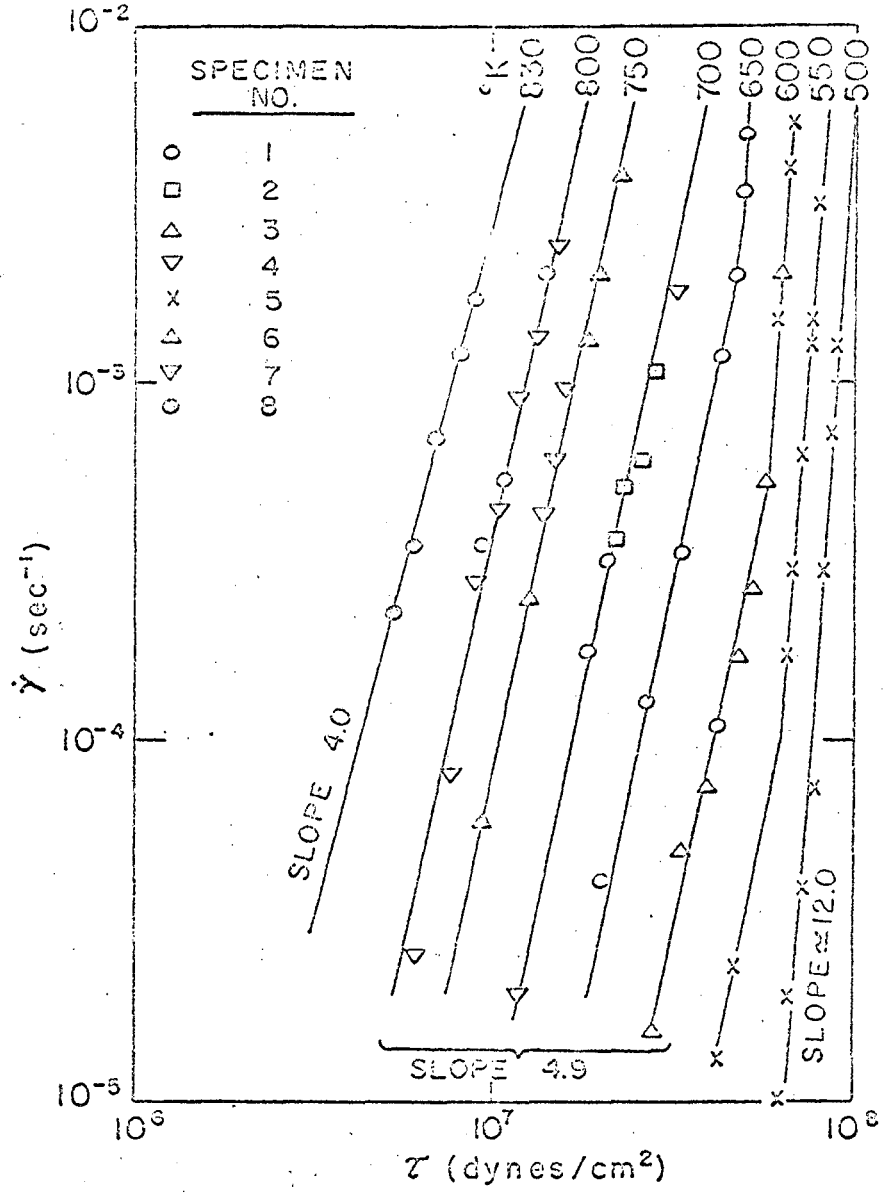

FIG. 4 EFFECT OF STRESS ON THE STEADY STATE CREEP RATE.

IV. DISCUSSION

In the temperature range from 600°K to about 750°K, as shown in Eq. (2), creep for basal slip of the 12.0 at % Li alloy of Mg is accurately described by a single activation energy term. This strongly indicates that, in this range, only one dislocation mechanism is rate-controlling. Furthermore, the activation energy is independent of the applied stress. (Fig. 3.) This fact disqualifies all possible mechanisms for which the activation energy is sensitive to the stress. As shown in Fig. 5, the athermal stress level for the 12.5 atomic percent Li alloy[2] is about 10 times higher than that for pure Mg. Therefore, if new mechanism is involved in controlling the creep rate, it must be attributed to some effect arising from alloying. Furthermore, the thermally activated mechanism of basal creep observed in the higher temperature range occurs at stresses below the extrapolated values of the athermal stress level. Consequently, when the temperature exceeds about 450°K, it seems that the dislocation barriers responsible for the athermal behavior at lower temperatures become more easily surmountable. Since 450°K is half the melting temperature, and this temperature is that at which diffusion becomes easier, a diffusion mechanism is thought to modify the nature and resistance of these barriers.

Predictions based on short range ordering considerations appear at first to be in good qualitative agreement with the general observed trends. According to Fisher[4], short range ordering results in an athermal deformation mechanism at intermediate temperatures, and Flinn[6] suggested that diffusion controlled fluctuations in local order at the

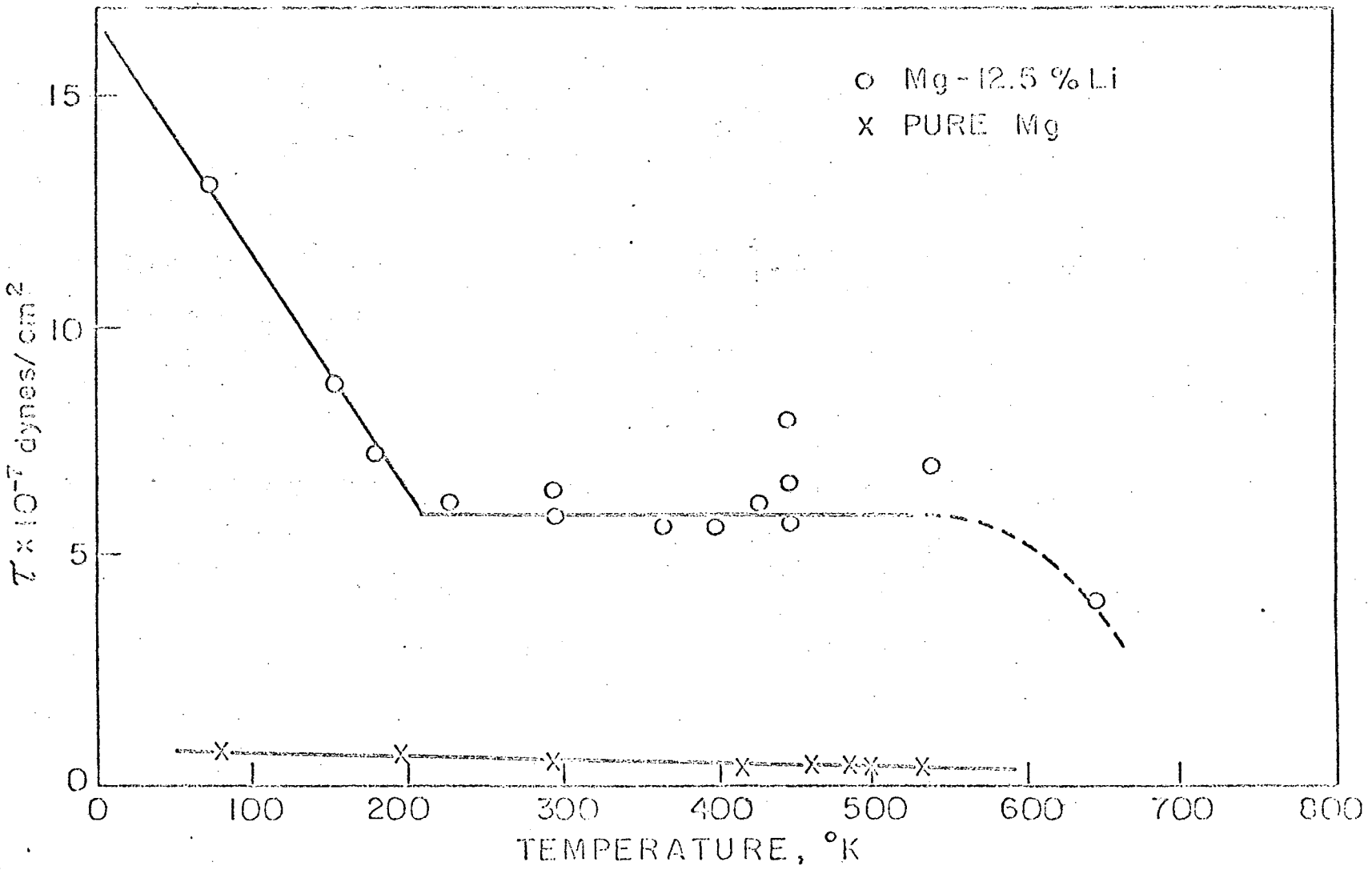

FIG. 5 CRITICAL RESOLVED SHEAR STRESS FOR BASAL SLIP vs. TEMPERATURE. (QUIMBY, MOTE AND DORN)[6]

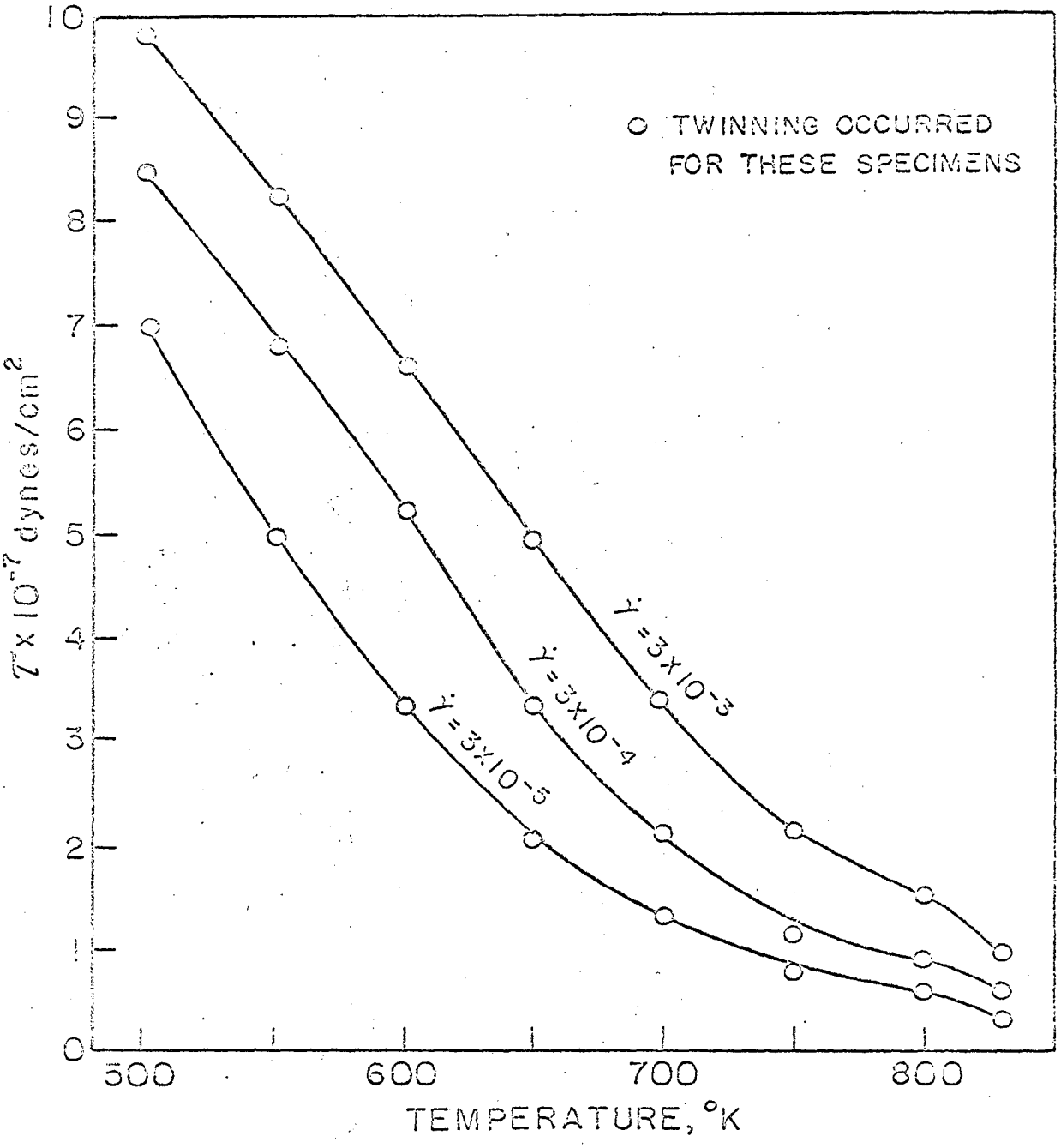

FIG. 6 STRESS VERSUS TEMPERATURE PLOT FOR BASAL CREEP OF Mg-12.0% Li ALLOY.

site of the dislocations yield a viscous type of creep in such alloys above about one-half of their melting temperature. However, Herbstein and Averbach[7] carefully investigated this alloy by diffuse x-ray scattering and found that its Cowley degree of order is practically zero. Furthermore, as will be shown later, it is not possible to rationalize the high temperature creep of the 12.0 atomic percent Li alloy of Mg in terms of a viscous mechanism; that is, a mechanism in which dislocations are considered to move in a viscous manner, the velocity of the dislocation line being proportional to the stress acting on it.[9] Thus short range ordering effects cannot account for the observed athermal strengthening of this alloy.

The athermal behavior could possibly be explained by the strain energy of interaction between solute atoms and dislocations. Effectively, Li alloying in Mg results in changes in both the a and c lattice constants and causes a substantial decrease of the c/a ratio. Li atoms in the alloy are consequently centers of tetragonal strain fields which can interact with both edge and screw dislocations. Summarizing the current status of the theory, Friedel[8] showed that an athermal stress level resulting from solid solution strengthening should increase with about the 2/3 power of the concentration of solute atoms. Since no data on basal slip are available showing the influence of the lithium concentration on the variation of the crss with the temperature, over the range 0 - 12.5% Li, it is difficult to decide whether or not this mechanism is operative. However, the results for basal slip are very similar to those previously obtained for prismatic slip[3-5], and for prismatic slip the observed athermal strengthening cannot be associated with the type

of solid solution strengthening described by Friedel; therefore it is most likely that this mechanism is also inoperative for basal slip. Furthermore, the absence of a yield point at low temperatures and of a Portevin-Lechatellier effect in the higher temperature range clearly reveal that Cottrell atmospheres are not effective in modifying the deformation. Since none of the above models can explain the obtained results, the athermal behavior of the 12.0 atomic percent Li alloy must be ascribed to other causes.

It was shown that the experimentally observed creep rate increased with the 4.9 th power of the applied stress. Consequently all mechanisms for which the creep rate increases either linearly with the stress as in Cottrell viscous drag of solute atoms, or with the cube of the stress as in Weertman's[9] viscous creep model, cannot be considered. A variation of Weertman's creep model[9] could provide a satisfying correlation with the observed stress law. When ρ is the density and v the mean velocity, of the mobile dislocations of Burgers vector b, the creep rate for this model is given by:

$$\dot{\gamma} = \rho b v \quad \text{with} \quad v = \frac{2\nu z \tau b^3 c D_o e^{-Q/kT}}{kT}$$

where $D_o e^{-Q/dT}$ is the self-diffusion coefficient, approximately the Debye frequency, z the coordination number, c a constant, and τ, C, k, T have their usual meanings. In order for this model to agree with the experimentally observed strain-rate law, it is necessary to assume that the density of mobile dislocations ρ is given by:

$$\rho = A\tau^{3.9}$$

The possible validity of this model depends on the ρ values it predicts. The values obtained by introducing the experimentally determined data into the above equations are all greater than about $10^{12}/cm^2$, which is unacceptably high. It is therefore necessary to conclude that this is not the operative mechanism.

However, one of Weertman's dislocation climb models[10] correlates well with the data. This model is based on the assumption that pairs of edge dislocations of opposite sign, moving on different parallel slip planes, are prevented from passing one another by the attractive forces acting between them. These immobile dislocations inhibit the action of the dislocation sources on their respective slip plane, thus allowing the formation of dislocation pile-ups between them and the sources. It was assumed that during steady state creep the blocked dislocations which lead the pile-ups annihilate each other by climbing. This annihilation is accompanied by movements of the pile-ups which bring new dislocations into the lead positions. According to Weertman,[10-11] the shear strain rate for this model will be:

$$\dot{\gamma} = \frac{.25\,\pi^2\,\tau^{4.5}\,D_c e^{-Q/kT}}{b^{1/2}\,N^{1/2}\,G^{3.5}\,kT} \qquad (3)$$

where N is the density of dislocation sources, G is the shear modulus and the other symbols are defined above. The derivation of Eq. (3) assumes a uniform distribution of the sources throughout the crystal and a constant value for the rate of dislocation climb. The difference between the observed stress exponent (4.9) and that predicted by Weertman (4.5) might be due to a deviation from these ideal conditions

during climb. Another possible explanation for this difference could be that the jog formation energy for basal slip is high enough, compared to the self-diffusion energy, to have to be considered. Weertman predicts[10] that in this case Eq. (3) should be modified. The exponent of the stress should be 5, and the activation energy equals the sum Q^* of the self-diffusion and the jog formation energies.

Then

$$\dot{\gamma} = \frac{0.30\ \pi^2\ \tau^5 D_o e^{\frac{-Q^*}{RT}}}{bN\ G^4\ kT} \quad . \tag{4}$$

This equation may be slightly modified in order to account for the observed dependency of the strain-rate with the 4.9^{th} power of the stress. It them becomes:

$$\dot{\gamma} = \frac{0.30\ \pi^2\ \tau^{4.9}\ D_o e^{\frac{-Q^*}{RT}}}{bN\ G^{3.9}\ kT} \tag{5}$$

Introducing the experimentally determined data into Eq. (5), (and using values for G for pure Mg[12]) the very acceptable value of $10^8/cm^2$ is found for N.

Thus, among all known rate controlling mechanisms for creep, only Weertman's dislocation climb model appears to account for the data reported here on the basal creep of the 12.0 atomic percent Li alloy of Mg. But since neither the self-diffusion energy nor the jog formation energy have been determined for this alloy, it is not known whether the value of Q = 37,000 cal/mole found for creep might be equal to the sum of these energies, as suggested in the model presented above.

For temperatures equal to or greater than about 800°K, it was shown that the activation energy increased substantially with the temperature, whereas the slope of the strain-rate stress curve decreased slightly. No present theory can account for such results. However, similar phenomena, though in a lower temperature range, have been observed by Tegart et al.[13] for polycrystalline pure Mg in which the main slip was apparently basal. It therefore appears that more than one thermally activated process might be operative in this alloy as its melting point is approached.

V. CONCLUSION

For the temperature range from 600° to 750°K:

(1) Basal creep of a 12.0 atomic percent Li alloy of Mg obeys the relationship:

$$\dot{\gamma} = 1.6 \times 10^{-28} \tau^{4.9} e^{\frac{-37,000}{RT}}$$

(2) Basal creep for the 12.0 atomic percent Li alloy occurs by a mechanism for which the activation energy is insensitive to the stress, and temperature.

(3) The experimental data are in disagreement with the various viscous creep models.

(4) Weertman's dislocation climb model[10] based on the annihilation of blocked dislocations accounts well for the observed data.

In the temperature range extending from about 800°K to the melting point, the activation energy increases substantially, indicating that more than one activated process might be operative.

For temperatures less than about 550°K, in a region where twinning has been observed, the strain-rate varies much more rapidly with the stress.

ACKNOWLEDGMENTS

The author wishes to thank Professor John E. Dorn and Mr. David M. Schwartz for their helpful suggestions and the great deal of work they put into this experiment.

The work was supported by the United States Atomic Energy Commission.

REFERENCES

1. T. A. Trozera, J. Mote, and J. E. Dorn, Trans. ASM, 53, 123 (1961).
2. H. M. Quimby, J. D. Mote and J. E. Dorn, Trans ASM 55, 149-57 (1962).
3. D. M. Schwartz, J. B. Mitchell and J. E. Dorn, UCRL Report 16901 (to be published).
4. J. C. Fisher, Acta Met., 2, 9-10 (1954).
5. Aziz Amadieh, Jack B. Mitchell and John E. Dorn, Trans AIME, 233, 1130-38 (1965).
6. P. A. Flinn, Phys. Rev., 104, 350 (1956).
7. F. H. Herbstein and B. L. Averbach, Acta Met. 4, 414, (1956).
8. J. Friedel, Dislocations, Pergamon Press Ltd., Oxford (1964).
9. J. Weertman, J. Appl. Phys., 28, 1185 (1957).
10. J. Weertman, J. Appl. Phys., 28, 362 (1957).
11. F. Garofalo, Fundamentals of Creep and Creep Rupture in Metals, MacMillan, New York (1965).
12. C. S. Roberts, Magnesium and its Alloys, John Wiley and Sons, New York (1958).
13. W. J. McG. Tegart, Acta Met., 9, 614 (1961).