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ALLOYS AND COMPOSITE MATERIALS BRANCH

**EFFECT OF TEMPERATURE ON THE
CREEP OF POLYCRYSTALLINE ALUMINUM
BY THE CROSS-SLIP MECHANISM**

N. Jaffe and J. E. Dorn

University of California

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JUNE 1960

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University of California, Berkeley, Calif. EFFECT OF TEMPERATURE ON THE CREEP OF POLYCRYSTALLINE ALUMINUM BY THE CROSS-SLIP MECHANISM, by N. Jaffe and J. E. Dorn, March 1960. 17p. incl. illus. 8 refs. (Project 7360; Task 73604) (WADD TR 60-53) (Contract AF 33(616)-3860) Unclassified report The apparent activation energy for creep of polycrystalline aluminum was determined over the range of 273° to 350°K by the effect of small abrupt changes in temperature on the creep rate. A constant activation energy of 27,400 ± 1000 cal/mole was obtained over strains of 0.003 to 0.23, stresses ranging from 2250 to 6000 p.s.i. and strain rates varying from 0.1145 x 10⁻⁵ (over)	UNCLASSIFIED	UNCLASSIFIED
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CREEP OF POLYCRYSTALLINE ALUMINUM
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N. Jaffe and J. E. Dorn

University of California

JUNE 1960

Materials Central
Contract No. AF 33(616)-3860
Project No. 7360

WRIGHT AIR DEVELOPMENT DIVISION
AIR RESEARCH AND DEVELOPMENT COMMAND
UNITED STATES AIR FORCE
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

FOREWORD

This report was prepared by the Institute of Engineering Research, University of California, Berkeley, California, under USAF Contract No. AF 33(616)-3860. The contract was conducted under Project No. 7360, "Materials Analysis and Evaluation Techniques," Task No. 73604, "Fatigue and Creep of Materials." The work was administered under the direction of Materials Central, Directorate of Advanced Systems Technology, Wright Air Development Division, with Lt. A. E. Riesen acting as project engineer.

This report was prepared during the contract period of 1 April 1959 to 31 March 1960.

The authors wish to acknowledge the support of the Wright Air Development Division in making this investigation possible, and for the technical aid and suggestions contributed by their metallurgical staff. They also wish to thank the Aluminum Company of America for supplying the high purity aluminum used in this investigation. They are also indebted to Mrs. Erik Ellentuck for preparing the manuscript.

ABSTRACT

The apparent activation energy for creep of polycrystalline aluminum was determined over the range of 273° to 350° K by the effect of small abrupt changes in temperature on the creep rate. A constant activation energy of $27,400 \pm 1000$ cal/mole was obtained over strains of 0.003 to 0.23, stresses ranging from 2250 to 6000 p.s.i. and strain rates varying from 0.1145×10^{-5} to 29.5×10^{-5} per minute. Metallographic studies and comparison with theory suggested that creep in this range is controlled by the rate of cross-slip of dislocations.

Both X-ray diffraction analyses and room temperature tensile stress-strain data following precreep revealed that the substructure produced in this range by creep under a given stress depended only on the total creep strain being independent of the actual test temperature. As a result it was deduced that the total strain ϵ during creep under a given stress should be a function of temperature compensated time $\Theta = te^{-\frac{Q}{RT}}$, where t is the duration of the test, Q the apparent activation energy, R the gas constant and T the absolute temperature. A number of creep tests conducted at two different temperatures verified the validity of this conclusion.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:



Chief, Strength and Dynamics Branch
Metals and Ceramics Laboratory
Materials Central

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EFFECT OF TEMPERATURE ON THE CREEP OF POLYCRYSTALLINE ALUMINUM BY THE CROSS-SLIP MECHANISM

INTRODUCTION

Previous investigations⁽¹⁾ have revealed that the apparent activation energies for creep of high purity polycrystalline aluminum are insensitive to the applied stress, strain and strain rate. On the other hand, the apparent activation energy is dependent on the temperature^(1, 2) as shown by the data reproduced in Fig. 1. Each plateau of this curve is associated with a unique rate controlling dislocation mechanism for thermally activated creep. It is now very well-established that over the high temperature range, where the activation energy is about 35,500 cal/mole, the creep rate is controlled by the dislocation climb mechanism.⁽³⁾ Over the intermediate range of temperatures, from about 260° to 370°K, another unique activation energy, namely 28,000 cal/mole, is obtained. Current evidence strongly supports the contention that cross slip is the rate controlling mechanism for creep in this case:

1. Crystal recovery can occur by a number of mechanisms prominent among which are climb and cross slip. Astrom,⁽⁴⁾ using the Borelius microcalorimeter, reported rapid crystal recovery of cold-worked polycrystalline Al at 340° - 370°K giving an activation energy of 28,000 cal/mole and additional recovery at 455°K - 478°K giving an activation energy of 36,000 cal/mole. Whereas the latter recovery process was clearly associated with the dislocation climb mechanism, the former, 28,000 cal/mole process could have arisen as a result of the cross-slip mechanism.
2. Single crystals of high purity Al favorably oriented for octahedral glide exhibit an activation energy for creep of about 3,400 cal/mole over the range from 0° to 400°K and that of about 28,000 cal/mole over the range from 600° to 775°K.⁽⁵⁾ Whereas the lower activation energy process is accompanied by the development of sharp slip-band traces, the 28,000 cal/mole process was uniquely characterized by extensive cross-slip.
3. Theoretical calculations by Seeger and Schoeck⁽⁶⁾ suggest an apparent activation energy for cross-slip in Al of about 24,000 cal/mole and another theoretical estimate by Friedel⁽⁷⁾ gives the observed 28,000 cal/mole as the theoretical activation energy.

It is therefore consistent with this evidence to, at least tentatively, ascribe the 28,000 cal/mole creep process in Al to the operation of cross slip as the rate controlling mechanism.

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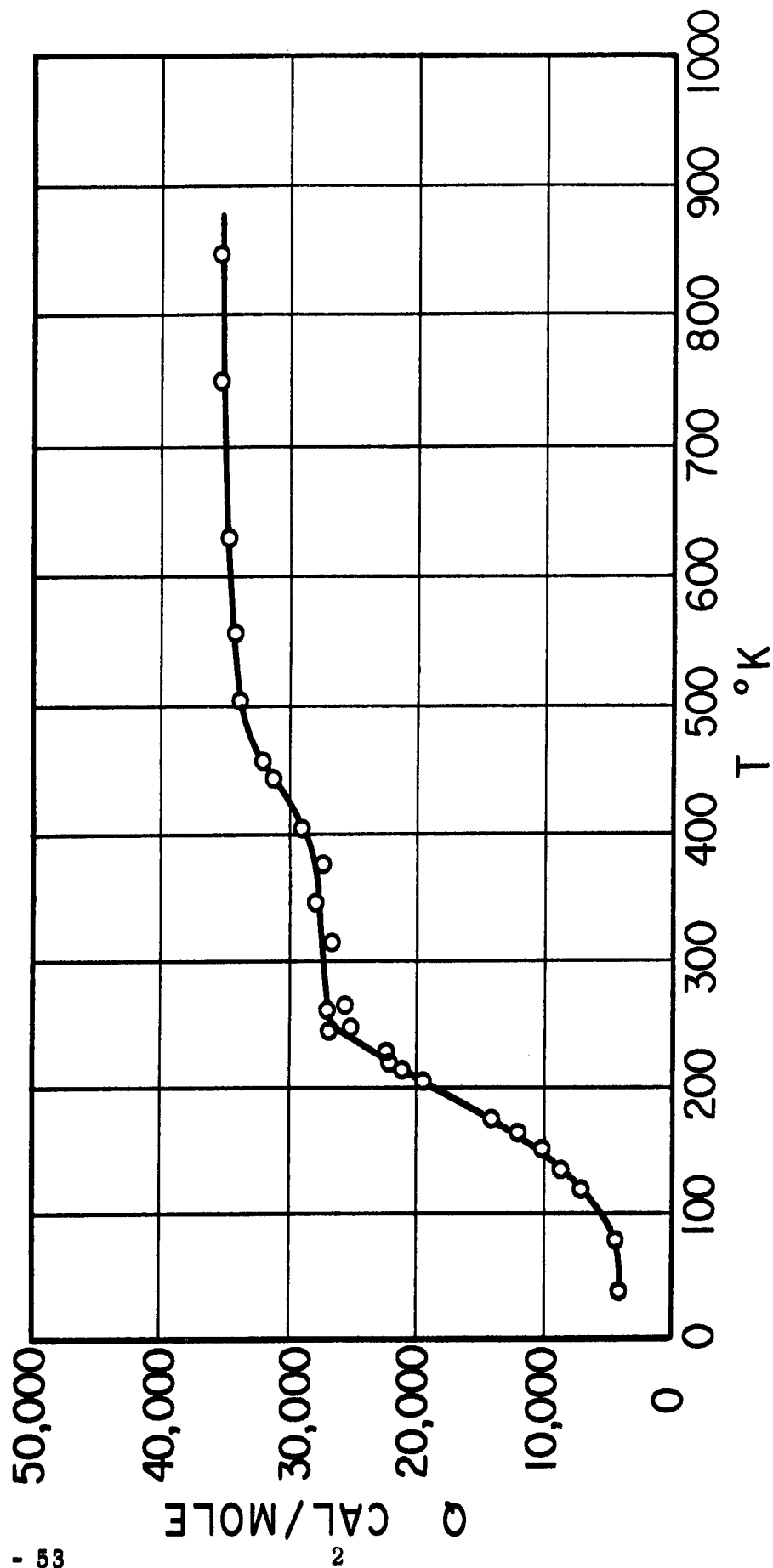


FIG.1 ACTIVATION ENERGIES FOR CREEP OF PURE ALUMINUM AS A
FUNCTION OF TEMPERATURE

Although creep has been rather well-explored in the dislocation climb range, very little, excepting perhaps the activation energy, is known about creep in the cross-slip range. This investigation was undertaken, therefore, to reconfirm the activation energy for creep in polycrystalline Al in the intermediate temperature range and to study the strain-time relationship for creep at a constant stress in the cross-slip region.

EXPERIMENTAL PROCEDURE AND TECHNIQUE

The three high purity Al alloys described in Table I were used in this investigation

Table I

Impurities, Heat Treatment and Grain Size

Alloy	Cu	Fe	Si	W	others	Temp °C	Time hrs.	Grain diam. in.
A	0.002	0.002	0.001	0.000	0.000	412	1.0	0.023
B	0.004	0.002	0.000	0.000	0.000	384	1.5	0.0031
C	0.000	0.002	0.000	0.004	0.000	384	1.5	0.0019

Specimens were milled from 0.100 in. thick cold-rolled sheets with their tensile axes in the rolling direction following which they were given a recrystallization and grain growth heat treatment at temperatures well-above those which were subsequently employed in the creep tests.

True stresses were maintained constant throughout each creep test by the use of contoured Andrade-Chalmers types of levers. The strain-time curves were autographically recorded, the strain sensitivity of the linear variable differential transformer system being about 10^{-6} .

The apparent activation energies for creep, Q , were determined by the effect of a rapid change in temperature of about 5°K from T_1 to T_2 (See Fig. 2) on the corresponding rates $\dot{\epsilon}_1$ and $\dot{\epsilon}_2$ according to:

$$\dot{\epsilon}_1 / \dot{\epsilon}_2 = e^{-\frac{Q}{RT_1}} / e^{-\frac{Q}{RT_2}} \quad (1)$$

Such small changes in temperature were effected by exchanging the constant temperature bath about the specimen with another bath controlled at the second temperature; it took the specimen about 3.5 minutes to reach thermal equilibrium at the new temperature. The creep rate just before a change in temperature was determined directly from the strain-time chart by measurement with a derivimeter. The creep rate just following a change in temperature was also measured in the same way after the creep curve was extrapolated through the transient temperature interval.

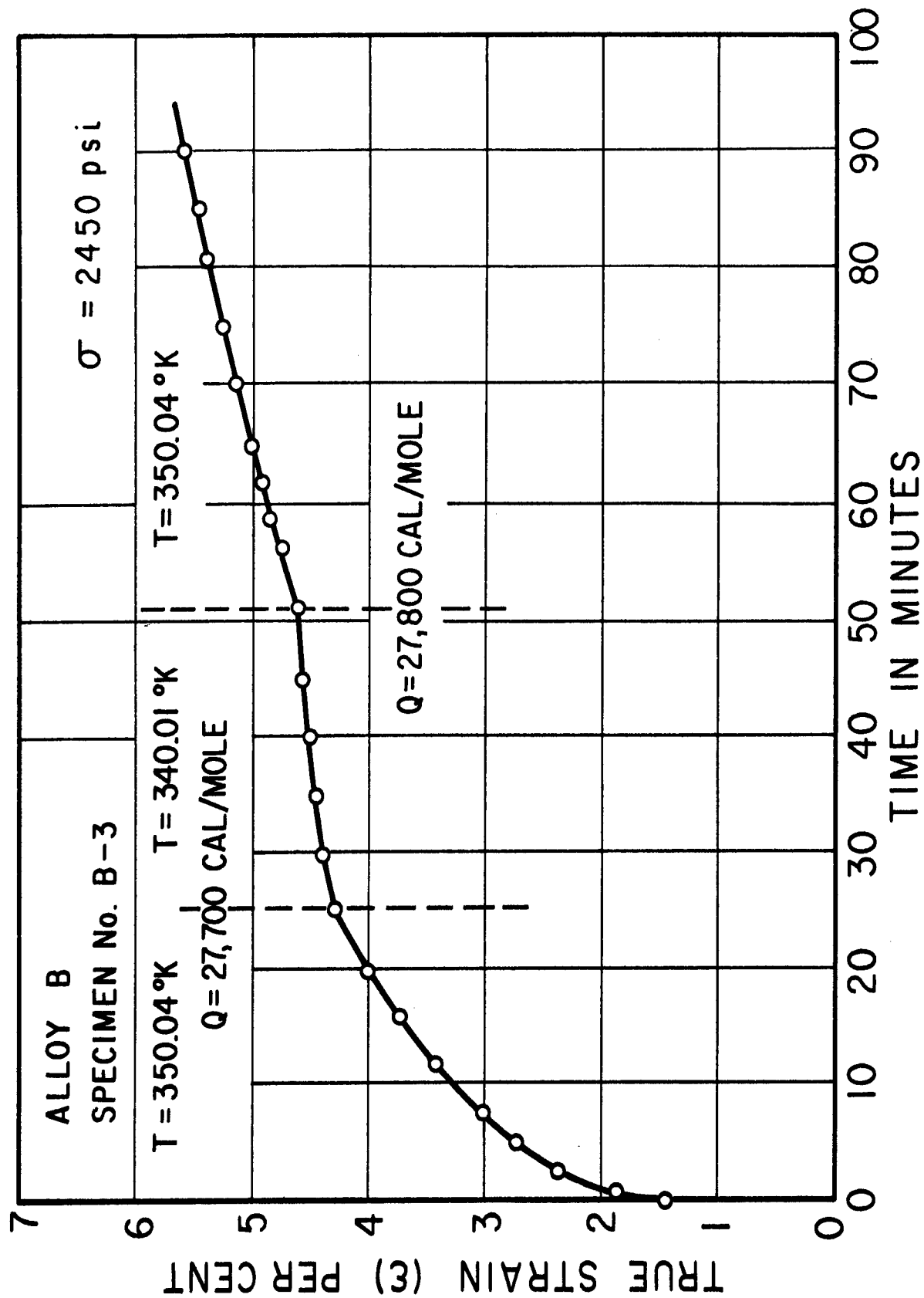


FIG.2 TYPICAL CREEP CURVE FOR CHANGE IN TEMPERATURE TEST

RESULTS AND DISCUSSION

In general creep by the cross-slip mechanism is given by

$$\dot{\epsilon} = 2NAb\sqrt{v}e^{-\frac{Q}{RT}} \quad (2)$$

where	$\dot{\epsilon}$	=	the tensile strain rate or twice the shear strain rate.
	N	=	the number of active points for cross slip per unit volume.
	A	=	the average area swept out per dislocation upon cross slipping.
	b	=	the Burger's vector.
	$\sqrt{v}$	=	the frequency of vibration of the dislocation segment that undergoes cross slip.
	R	=	the gas constant
	T	=	the absolute temperature.
	Q	=	the apparent activation energy for cross slip.

Since all of the factors affecting the creep rate, excepting Q, are insensitive to the temperature, the temperature dependence of creep in the cross-slip region is exclusively determined by the exponential term of Eqn. 2. The activation energy Q, however, depends on the local stress and the temperature. But since Q is not a sensitive function of the temperature the apparent activation energies obtained by application of Eqn. 1 will agree closely with the true activation energies for cross slip.

The apparent activation energies as obtained by the rapid change in temperature technique and recorded in Table II, give a mean value of $27,400 \pm 1,000$ cal/mole over temperatures ranging from 273° to 350° K, stresses ranging from 2,250 to 6,000 psi, strains from 0.003 to 0.23, and mean strain rates of 0.145×10^{-5} to 29.5×10^{-5} per min. These data confirm previous observations⁽¹⁾ that the activation energy for cross-slip in aluminum is insensitive to strain, stress, and temperature over the range of strain rates that were investigated.

The failure to detect an effect of the applied stress on the activation energy, however, must be attributed to the insensitivity of the technique used in this investigation. First the localized stress that influences cross-slip is equal to the applied stress minus the long range back stress field. As

Table II
Apparent Activation Energies for Creep of Aluminum

Specimen No.	Applied Tensile Stress psi	Strain $\epsilon = \ln l/l_0$	T ₁ °K	T ₂ °K	$\dot{\epsilon}_1 \times 10^5$ per min.	$\dot{\epsilon}_2 \times 10^5$ per min.	$\dot{\epsilon}_{app} \times 10^5$ per min.	Q cal./mole
A-1	3200	0.00241	278.25	273.00	0.250	0.095	0.154	27,800
	3600	0.00298	273.00	278.25	0.281	0.723	0.451	27,100
	3500	0.00305	278.25	273.00	0.568	0.224	0.357	26,800
	3500	0.00311	273.00	278.25	0.211	0.553	0.342	27,700
A-2	4000	0.0036	278.25	273.00	31.8	12.5	19.9	26,700
	4000	0.00381	273.00	278.25	2.55	6.53	4.08	27,000
	4000	0.01416	278.25	273.00	3.30	1.30	2.07	26,800
	4000	0.02392	273.00	278.25	1.00	2.55	1.60	26,900
	4000	0.04018	278.25	273.00	2.55	0.97	1.57	27,800
	4000	0.04989	273.00	278.25	0.61	1.65	1.003	28,500
A-3	3000	0.00254	278.25	273.00	0.22	0.086	0.137	27,000
A-4	2500	0.01845	273.00	278.25	0.071	0.185	0.114	27,600
A-5	2250	0.00946	273.00	278.25	0.078	0.199	0.124	26,900
A-6	3800	0.01421	278.25	273.00	16.0	6.20	9.96	27,300
	3800	0.01541	273.00	278.25	1.97	5.01	3.14	26,800
B-1	4500	0.1435	278.25	273.00	3.38	1.32	2.11	26,900
	5741	0.2194	273.00	278.25	2.01	5.12	3.21	27,000
	6000	0.2345	278.25	273.00	5.98	2.31	3.72	27,400

Table II (cont.)

$$Q(\text{average}) = 27,400 \text{ cal./mole}$$

the applied stress is increased, additional dislocations which increase the back stress are generated. Thus the change in the local stress might indeed be quite negligible even for an appreciable increase in the applied stress. Furthermore, if the difference in the extreme strain rates $\dot{\epsilon}_{low} = 0.1145 \times 10^{-5}$ and $\dot{\epsilon}_{high} = 29.5 \times 10^{-5}$ per minute used in this investigation were exclusively attributed to a change in activation energy at a mean temperature of 309° K as a result of the applied stress, the change in activation energy would be about 3,000 cal/mole which is only slightly greater than the scatter range. Therefore a much greater range in strain rates, beyond that readily achievable experimentally, would have to be used to detect the effect of stress on the activation energy for cross slip. A more sensitive alternate technique of studying the stress law for cross slip is now being used to rationalize this issue.

The fact that the activation energies reported in Table II agree well with those observed by Astrom⁽⁴⁾ for recovery of cold worked Al and those predicted by Schoeck and Seeger⁽⁶⁾ and by Friedel⁽⁷⁾ suggests that creep of Al over the range of conditions studied here occurs by the cross slip mechanism. The photomicrograph of Fig. 3 showing extensive cross slip further confirms the concept that cross slip is a prominent feature in the range of creep studied here.

It has been demonstrated that above 527° K, where creep of polycrystalline Al is controlled by the dislocation climb mechanism, the total strain ϵ obtained at time t under a constant stress σ is dependent on a temperature compensated Θ ⁽³⁾ where

$$\epsilon = f(\Theta) \quad \text{for } \sigma = \text{const.} \quad (3)$$

and

$$\Theta = \int_0^t e^{-\frac{Q}{RT}} dt \quad (4)$$

The question, therefore, arises as to whether this useful relationship might not also apply to the cross-slip region of creep. In order to test this point the various creep data shown in Figs. 4A, 4B, and 4C, were obtained, which confirm the validity of Eqn. 4 for the cross-slip region. Whereas each stress and each alloy has its own unique $\epsilon - \Theta$ curve, the $\epsilon - \Theta$ curves for a given stress are independent of the test temperature, at least within the cross-slip range of temperatures.

The validity of Eqn. 3 for the cross-slip range suggest that the same dislocation substructure is obtained at the same value of Θ for a given creep stress, independent of the actual test temperature. This conclusion is readily illustrated by differentiating Eqn. 3 with respect to the time which gives

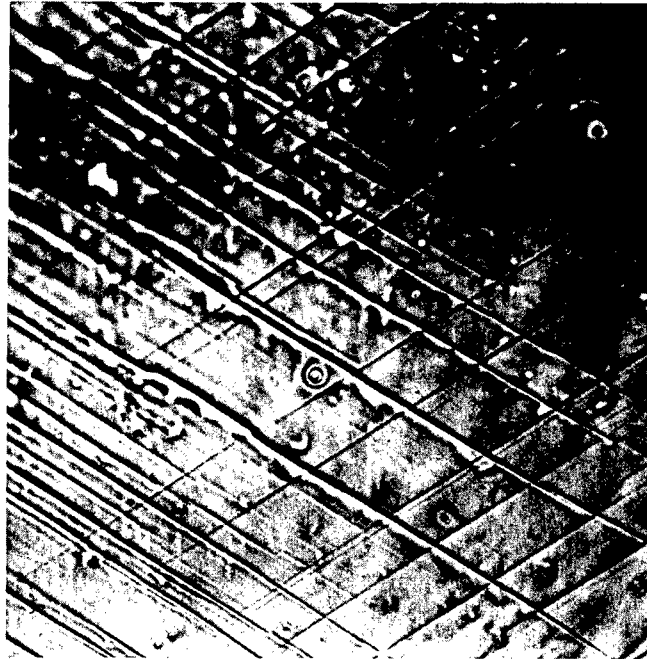


FIG. 3 PHOTOMICROGRAPH FOLLOWING CREEP
AT 308 °K, 5000 PSI TO A STRAIN
OF 0.2309

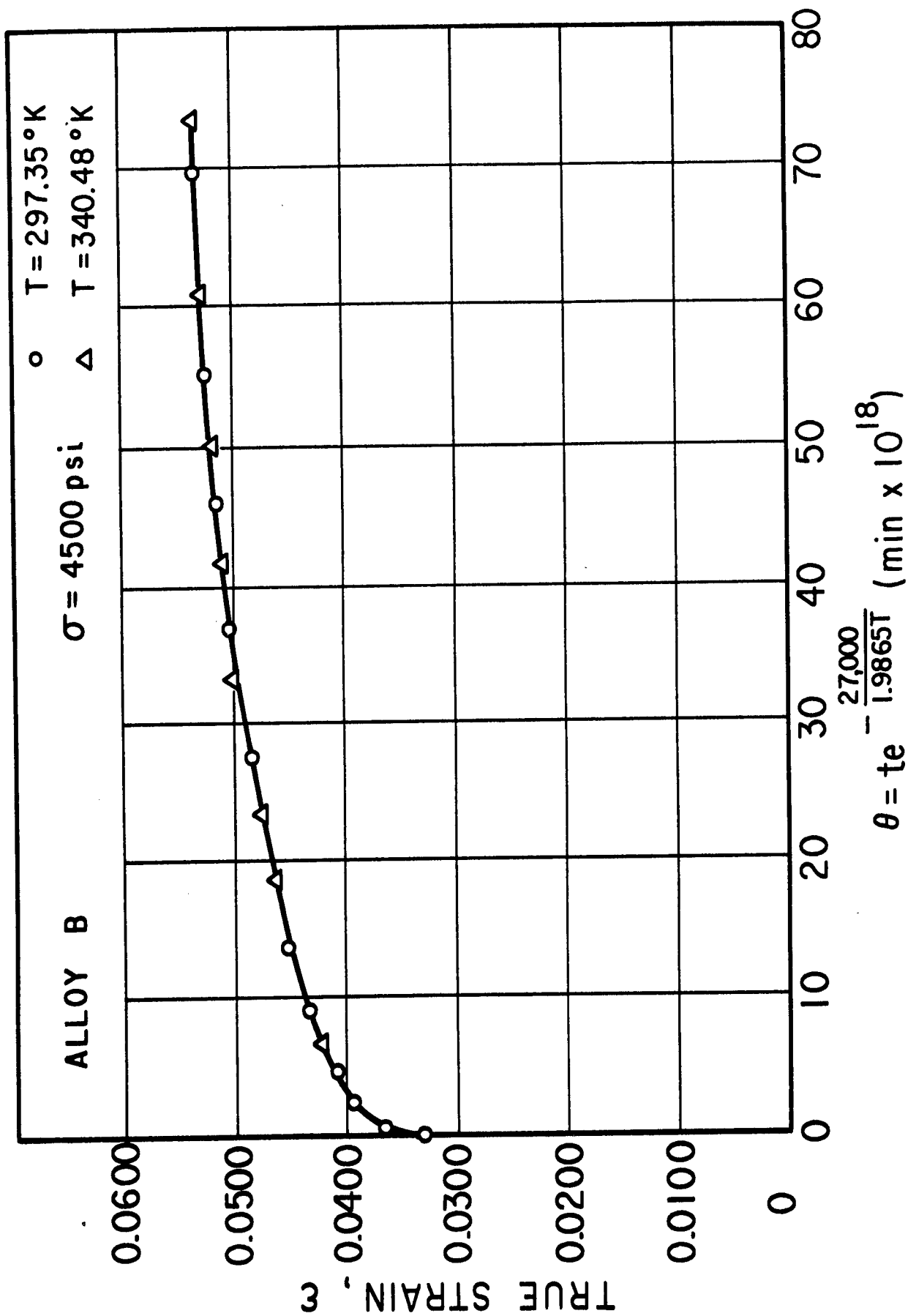


FIG. 4a CORRELATION OF CREEP STRAIN AS A FUNCTION OF A TEMPERATURE COMPENSATED TIME

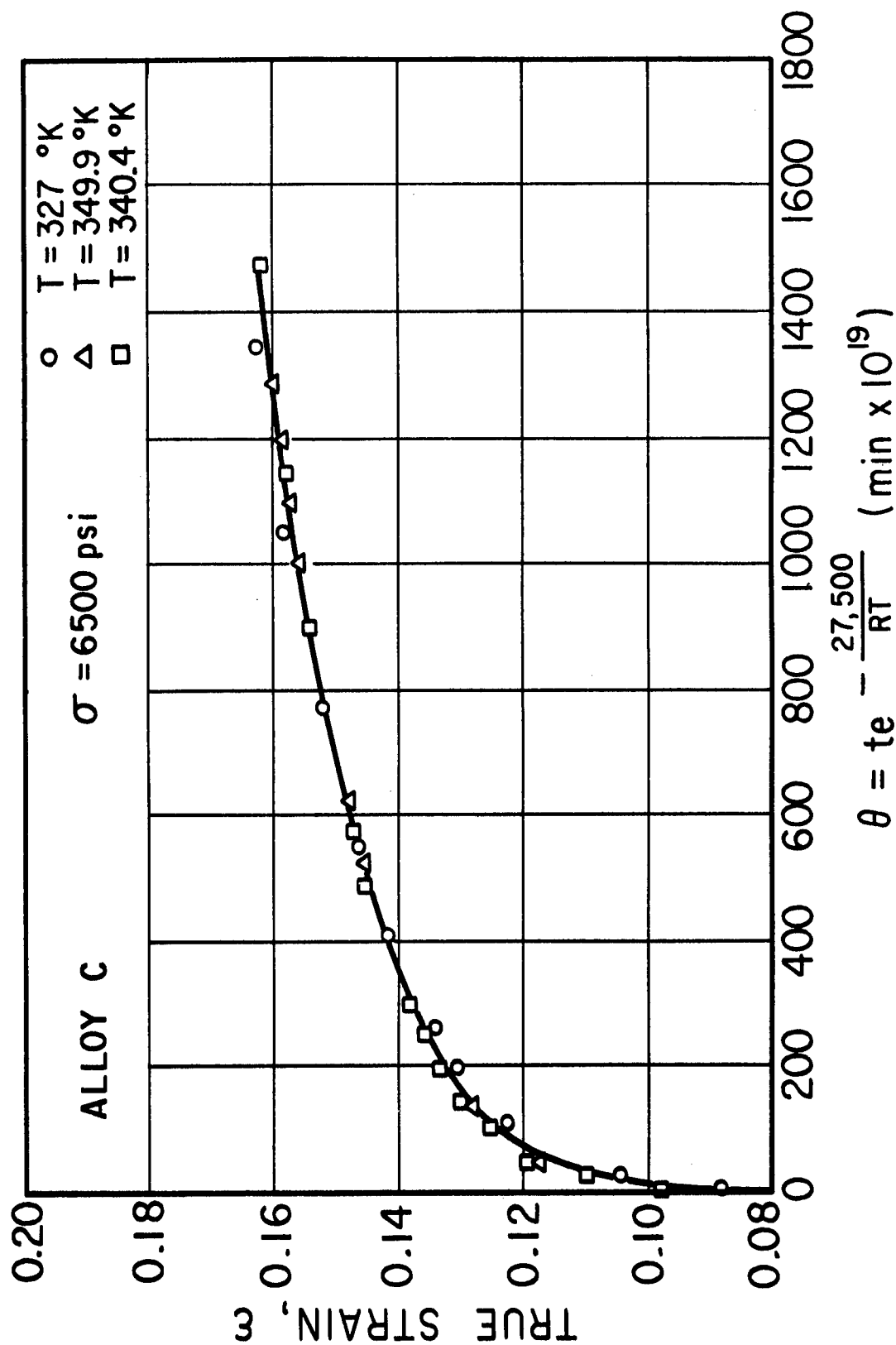


FIG. 4b CORRELATION OF CREEP STRAIN AS A FUNCTION OF A TEMPERATURE COMPENSATED TIME

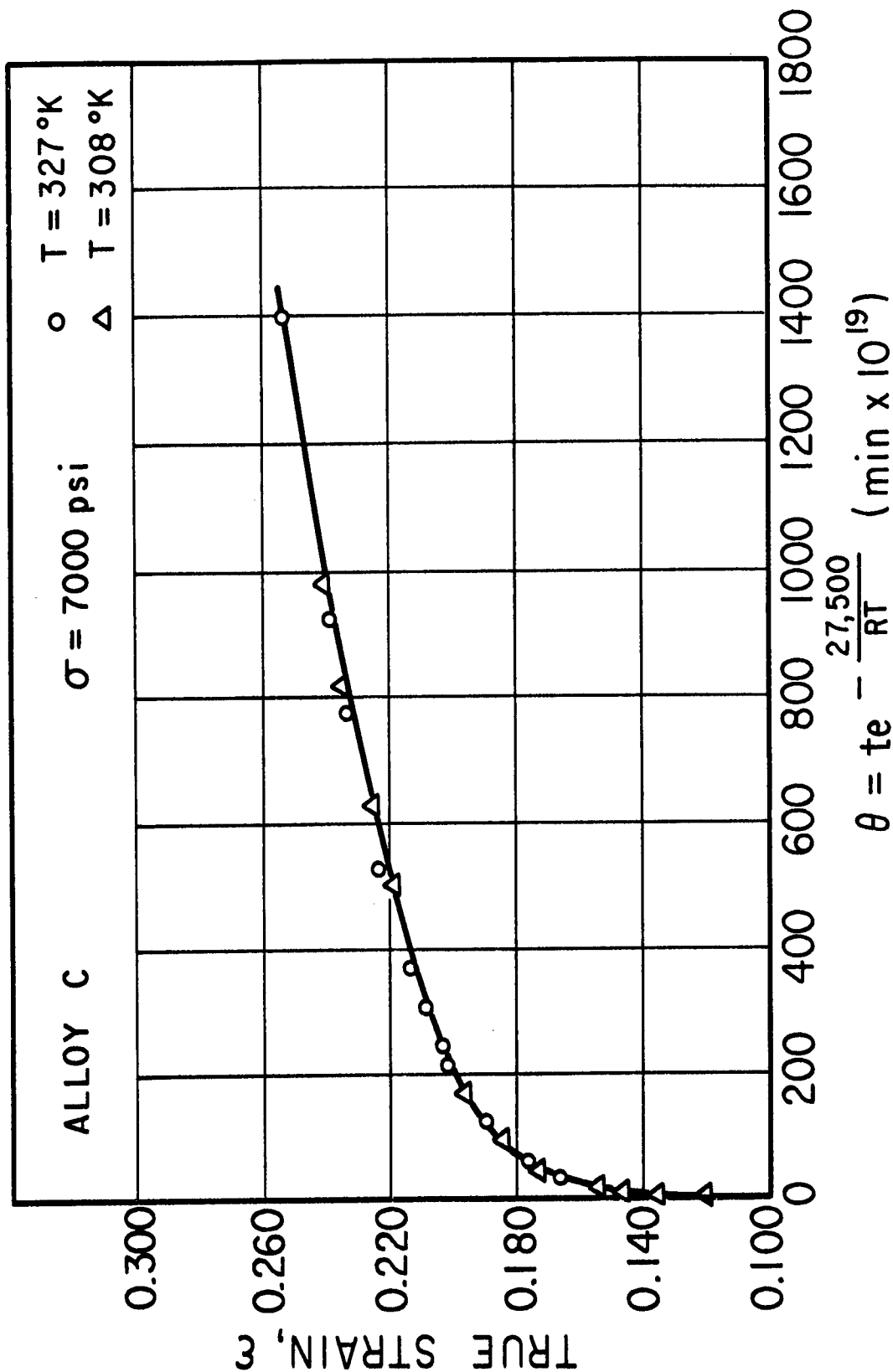


FIG. 4c CORRELATION OF CREEP STRAIN AS A FUNCTION OF A TEMPERATURE COMPENSATED TIME

$$\dot{\epsilon} = f'(\theta) e^{-\frac{Q}{RT}} \quad (5)$$

$$\text{where } f'(\theta) = df/d\theta$$

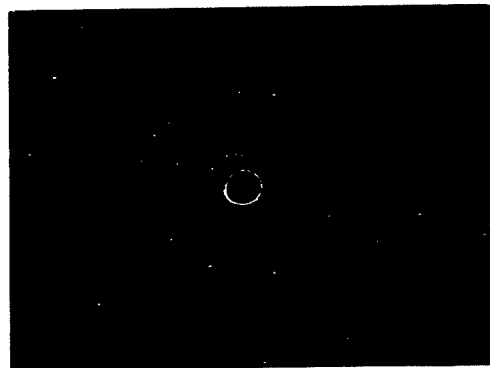
and comparing the result with Eqn. 2. Consequently the pre-exponential terms of Eqn. 2 which depend on the substructure must equal $f'(\theta)$.

Conversely if the substructure generated during creep under a given stress is dependent only on θ , the pre-exponential terms of Eqn. 2 can be represented by the function $f'(\theta)$, as shown in Eqn. 5, which upon integration gives Eqn. 3. Therefore the same substructure should be developed for a given creep stress at the same strain, independent of the test temperature within the range where cross-slip is the rate controlling mechanism. Two types of tests were employed to confirm this correlation.

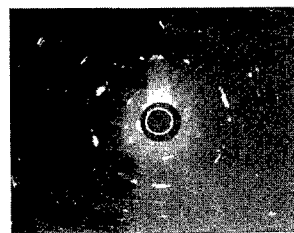
The back reflection Debye-Sherrer radiographs shown in Fig. 5 were taken following creep at different temperatures. Since the pinhole was small and the original grain size relatively large only a few grains exposed to the beam were oriented so as to satisfy Bragg's Law. Consequently only a few spots appear on the Debye-Sherrer circles for the annealed specimen. Following creep, however, the individual grains became distorted causing diffraction over a segment of the Debye-Sherrer circle. And within the sampling scatter, approximately the same type of Debye-Sherrer pattern was obtained for creep under the same stress to the same strain for both test temperatures.

The diffuse character of the Debye-Sherrer arcs from a single grain are distinctly different from the previously reported spotty arcs (8) obtained as a result of polygonization during creep in the dislocation climb range of temperatures suggesting that the arcing in the cross-slip range of temperatures arises not from polygonization but perhaps from more uniform bending of the deformed crystals due to piled up dislocation arrays.

A more definitive result was obtained by using tensile tests at room temperature as a sensitive measure of the substructure generated during creep. In Fig. 6 are shown a series of stress strain curves from specimens that were crept under the same stress to a series of identical strains at two different temperatures. As creep continued, the flow stress in subsequent tension increased substantially. The increase in the tensile flow stress at room temperature with increasing straining in the dislocation cross-slip region of creep is entirely consistent with the attendant increased arcing of the Debye-Sherrer spots from a single grain. Both observations suggest that the creep rate in the cross-slip region decreases with time as a result of the introduction of additional barriers to dislocation motion during the course of creep.



ANNEALED UNSTRAINED
SPECIMEN



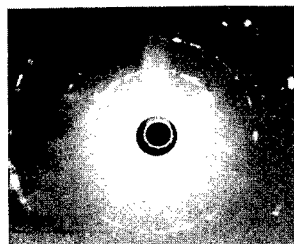
$\epsilon = 0.0408$
 $T = 308^{\circ}\text{K}$



$\epsilon = 0.0410$
 $T = 327^{\circ}\text{K}$



$\epsilon = 0.0870$
 $T = 308^{\circ}\text{K}$



$\epsilon = 0.0903$
 $T = 327^{\circ}\text{K}$



$\epsilon = 0.1959$
 $T = 308^{\circ}\text{K}$



$\epsilon = 0.1961$
 $T = 327^{\circ}\text{K}$

FIG. 5 X-RAYS OF SPECIMENS CREPT TO CORRESPONDING STRAINS AT
308°K AND 327°K UNDER A STRESS OF 5000 PSI.

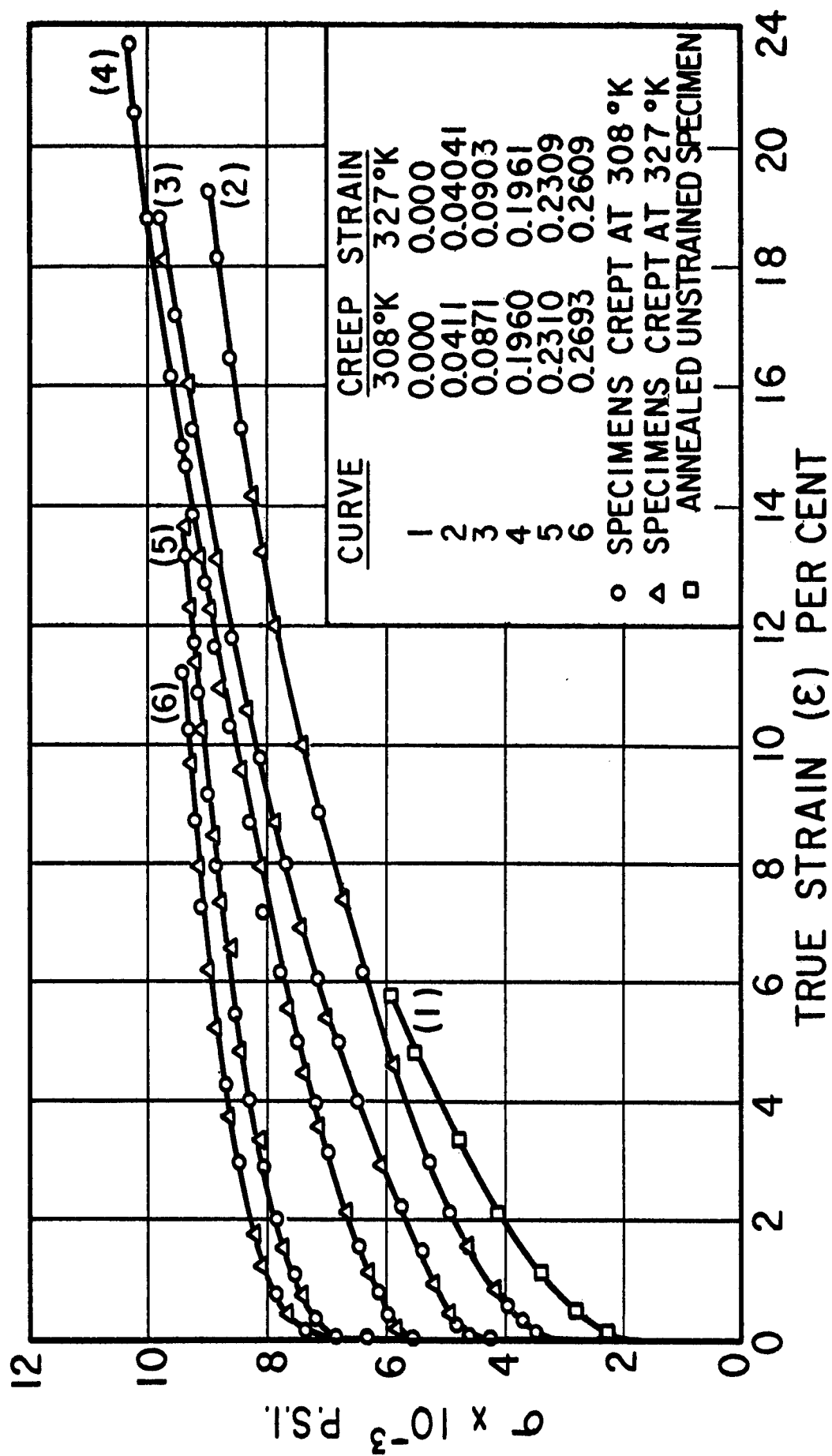


FIG. 6 STRESS STRAIN CURVES OF CREPT SPECIMENS

Within the experimental scatter the same stress strain curves were obtained following the creep at a given stress to the same strain for each of the two different temperatures. These data not only reveal that the substructure generated during creep in the cross-slip region depends on the creep strain under a given stress but they also confirm the validity of Eqn. 3.

CONCLUSIONS

1. The apparent activation energy for creep of polycrystalline Al over the range from 275° to 345°K is 27,400 cal/mole. Metallographic observations and comparison with theoretical calculations on the activation energy suggest that the creep rate is controlled by the cross-slip mechanism in this range.

2. The substructure generated during creep in the cross-slip region under a given stress is independent of the test temperature, being dependent only on the strain.

3. The total strain ϵ following creep in the cross-slip region under a given stress is related to a temperature compensated time $\Theta = t e^{-\frac{Q}{RT}}$ where t is the duration of creep, Q the activation energy, R the gas constant and T , the absolute temperature.

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