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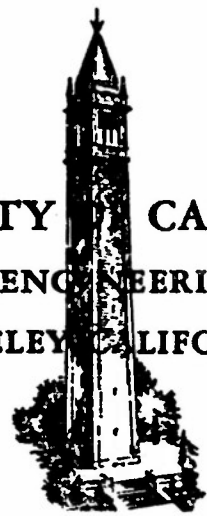
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SOME OBSERVATIONS ON GRAIN BOUNDARY SHEARING DURING CREEP

Thirty Second Technical Report

By

Bernard Fazan, Oleg D. Sherby, and John E. Dorn

22, N7-onr-295, Task Order II  
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November 15, 1953

Office of Naval Research  
Department of the Navy  
Washington 25, D. C.

ATTENTION: Mr. Julius Harwood

Dear Sir:

Attached hereto is a copy of the Thirty Second Technical Report entitled "Some Observations on Grain Boundary Shearing During Creep". This report covers the contribution of grain boundary shearing to the total creep process for high purity aluminum in the temperature range 610°K to 747°K under a stress of 250 psi. It is shown that grain boundary shearing follows the same functional relationship as the total creep strain, namely,  $\epsilon_{g.b.} = f(\Theta)$  where  $\epsilon_{g.b.}$  = creep strain due to grain boundary shearing,  $\Theta = t e^{-\Delta H_{g.b.}/RT}$  where  $t$  = time under stress  $\sigma$ ,  $\Delta H_{g.b.}$  = activation energy for grain boundary shearing,  $R$  = gas constant and  $T$  = absolute temperature. The activation energy for grain boundary shearing is essentially the same as that for the total creep process equal to about 36,000 calories per mole.

This investigation is based in part on the thesis submitted by Mr. B. Fazan for the M.S. degree in physical metallurgy. In view of its close correlation with the investigations being conducted in our O.N.R. research on the plastic properties of aluminum alloys, we wish to submit this report to the O.N.R.

The wholehearted cooperation of the Office of Naval Research in making these studies possible is sincerely appreciated.

Respectfully submitted,

*John E. Lorn*  
John E. Lorn  
Professor of Physical Metallurgy

JED:dk

SOME OBSERVATIONS ON GRAIN BOUNDARY SHEARING DURING CREEP

By

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November 15, 1953

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### ABSTRACT

Quantitative measurements of the contribution of grain boundary shearing to the creep process were made for high purity aluminum in the temperature range 610°K to 747°K under a stress of 250 psi. The relative contribution of grain boundary shearing to the total creep strain is shown to be independent of the test temperature but, as revealed by McLean, it increases as the creep stress decreases.

## INTRODUCTION

Several outstanding investigations by McLean<sup>(1-4)</sup> have demonstrated that the total creep strain in polycrystalline aluminum at 473°K arises from microscopically detectable slip, subgrain tilting and grain boundary shearing. Whereas microscopically detectable slip arises from dislocations that move out of the grains, subgrain tilting is due to entrapment of dislocations of like sign in subgrain boundaries. Consequently, creep in polycrystalline aggregates appears to be the resultant of two mechanisms, namely migration of dislocations and grain boundary shearing. This suggests that an appropriate law for creep should contain two additive terms, namely:

$$\epsilon_T = \epsilon_d + \epsilon_{g.b.} \quad (1)$$

where the total creep strain,  $\epsilon_T$ , depends on the sums of the creep strains arising from motion of dislocations,  $\epsilon_d$ , and from grain boundary shearing,  $\epsilon_{g.b.}$ .

But extensive investigations<sup>(5)</sup> over wide ranges of alloys, grain sizes, temperatures, and stresses have shown that the total creep strain is quite accurately dependent on the single term functional relationship

$$\epsilon_T = f(\Theta) \quad \sigma = \text{constant} \quad (2)$$

where  $\sigma$  = stress (constant)\*,

$$\Theta = t e^{-\Delta H/RT},$$

$t$  = time under stress,

$\Delta H$  = activation energy,

$R$  = gas constant,

and  $T$  = absolute temperature.

---

\* Eq. 2 is valid for either constant stress or constant load tests; the total creep curve, however, is different (i.e. the function,  $f$ , is different) for each test.

Since Eq. 2 was found to be valid over those ranges where appreciable grain boundary shearing is observed, it appears probable that the activation energy for grain boundary shearing might be identical with the activation energy for migration of dislocations. Therefore, under a given stress, it might be thought that

$$\epsilon_t = f_d(\theta) + f_{g.b.}(\theta) = f(\theta)$$

where  $f_d$  and  $f_{g.b.}$  represent the appropriate functions for each mechanism of creep respectively. The primary objective of this investigation was to obtain a direct check on the accuracy of this hypothesis.

#### EXPERIMENTAL PROCEDURE AND RESULTS

Rolled aluminum sheet (99.987% Al), 0.100 inches thick, was used in this investigation. Creep specimens having a 0.25 inch wide, 3 inches long gage section were so machined that their axes were in the rolling direction. All specimens were annealed for nine hours at 894°K before testing to produce a mean grain diameter of about 0.1 inch. One face of each specimen was polished electrolytically in 10 per cent fluoboric acid. In order to permit accurate measurements of grain boundary shearing, the polished surface was lightly scribed with a ruling machine accurate to  $\pm 0.0001$  inch to give a uniform grid of lines which were 0.01 inch apart. Although it was thought that surface working might modify the creep behavior in the vicinity of the scribed grid lines, this technique was nonetheless employed because such local differences in creep properties should not seriously modify the fractions of the total creep strain due to grain boundary shearing or to migration of dislocations. All creep tests were conducted under constant load with an initial stress of 250 psi. The specimen temperature, as measured by two calibrated thermocouples

attached at the two ends of the gage section, was held to within  $\pm 2^{\circ}\text{K}$  of the reported temperature. The total creep strain during the course of a test was measured by means of a simple rack and pinion extensometer sensitive to 0.01 percent for a two inch gage length. After several exploratory tests the following eight tests were made:

TABLE I  
Conditions of Test  
(Stress - 250 psi)

No.	Temp. $^{\circ}\text{K}$	Total Creep Strain $\epsilon_t$
1	610	0.037
2	747	0.037
3	610	0.070
4	747	0.070
5	610	0.110
6	640	0.110
7	700	0.110
8	747	0.110

The tests at various temperatures from  $610^{\circ}$  to  $747^{\circ}\text{K}$  were interrupted at prescribed total strains,  $\epsilon_t$ , of 0.037, 0.070, and 0.110 and the specimens were immediately removed from the creep machine for metallographic analyses and measurements of grain boundary shearing.

The technique employed for determining the amount of grain boundary shearing was that introduced by McLean<sup>(1-4)</sup>. An example of grain boundary shearing is shown in Fig. 1 and idealized in Fig. 2. The vertical displacement of a horizontal grid line at a grain boundary is designated by  $k$ . The greatest shear displacements were found on those grain boundaries that ran about  $45^{\circ}$  to the specimen axis. But wide ranges of  $k$  values



FIG. 1 SHEAR ALONG A GRAIN BOUNDARY IN HIGH  
PURITY ALUMINUM.  
CREEP STRESS = 250 PSI,  $\epsilon_t = 0.07$ ,  $T = 610^\circ\text{K}$ .  
MAGNIFICATION 100 X.



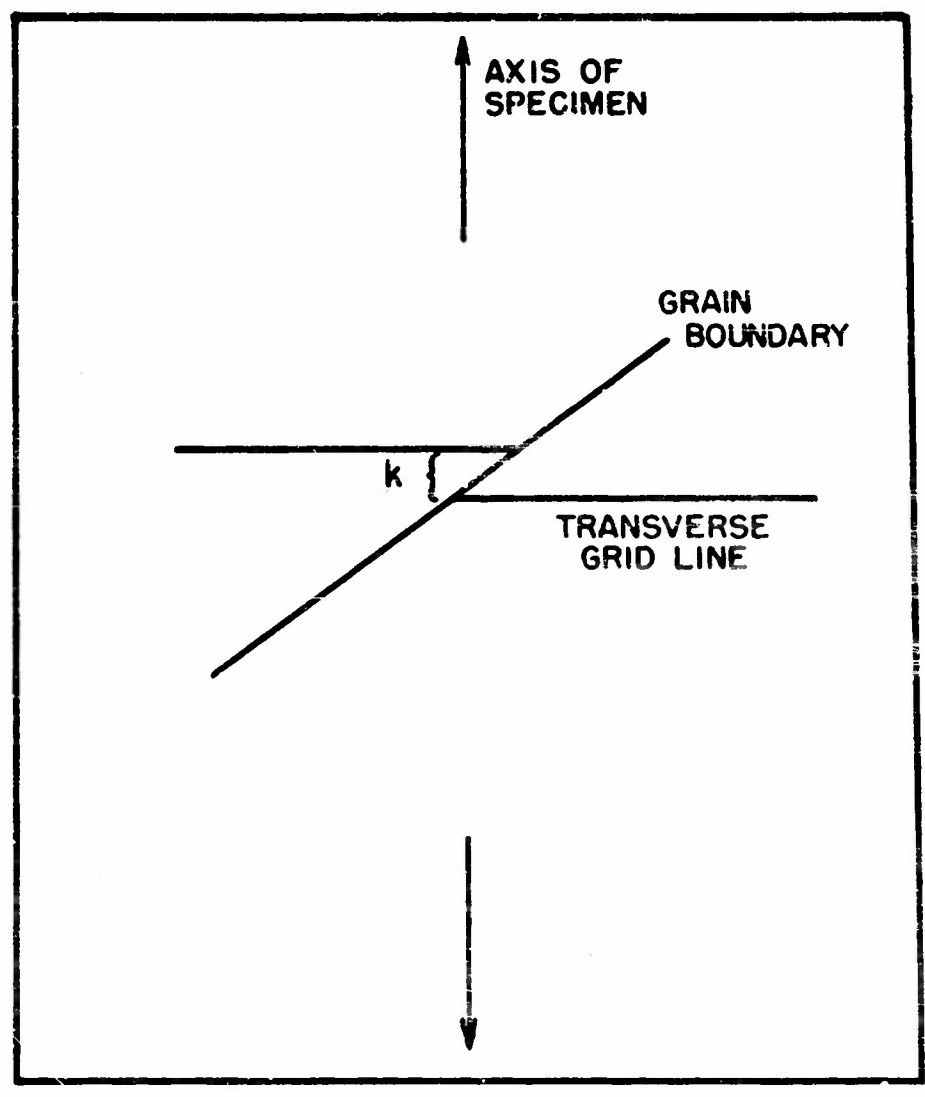


FIG. 2 IDEALIZED EXAMPLE OF GRAIN BOUNDARY SHEARING INDICATING METHOD OF MEASURING THE LONGITUDINAL SHEAR DISPLACEMENT  $k$ .

were obtained for different boundaries of the same orientation (relative to the specimen axis) suggesting that grain orientations also affect grain boundary shearing. Furthermore, in many cases the value of  $k$  varied appreciably even over a single grain boundary.

As shown in Fig. 3 at regions where grain boundary migration took place, the grid lines appear to have been bent. It was assumed that the appearance of bending was due to alternate grain boundary migration and shearing. An example of this on a coarse scale is shown in Fig. 4. Consequently, for such cases of bent grid lines  $k$  was determined by measuring the vertical displacement of the grid line over the entire region where grain boundary migration took place. An extreme example of alternate grain boundary shearing and migration on a very fine scale is illustrated in Fig. 5. It was also observed that polygonization occurred preferentially along the grain boundaries rather than in the grain cores, an example of which is shown in Fig. 6. Surface tension effects between the differently oriented sub-grains were often high enough to produce the angular grain boundaries evident in Fig. 6. Such irregular boundaries exhibited less tendency toward shearing than smooth regularly contoured boundaries.

In view of the wide scatter in the individual shear displacements,  $k$ , an attempt was made to determine an average value  $\bar{k}$ . This was done by measuring each  $k$  value along five consecutive transverse grid lines, then skipping the next ten lines and repeating the measurement for the next 5 transverse grid lines, etc. until the entire gage section had been covered. In this way an average  $\bar{k}$  was obtained over not less than 150 individual measurements per specimen. The longitudinal strain arising from grain boundary shearing is then readily determined by multiplying

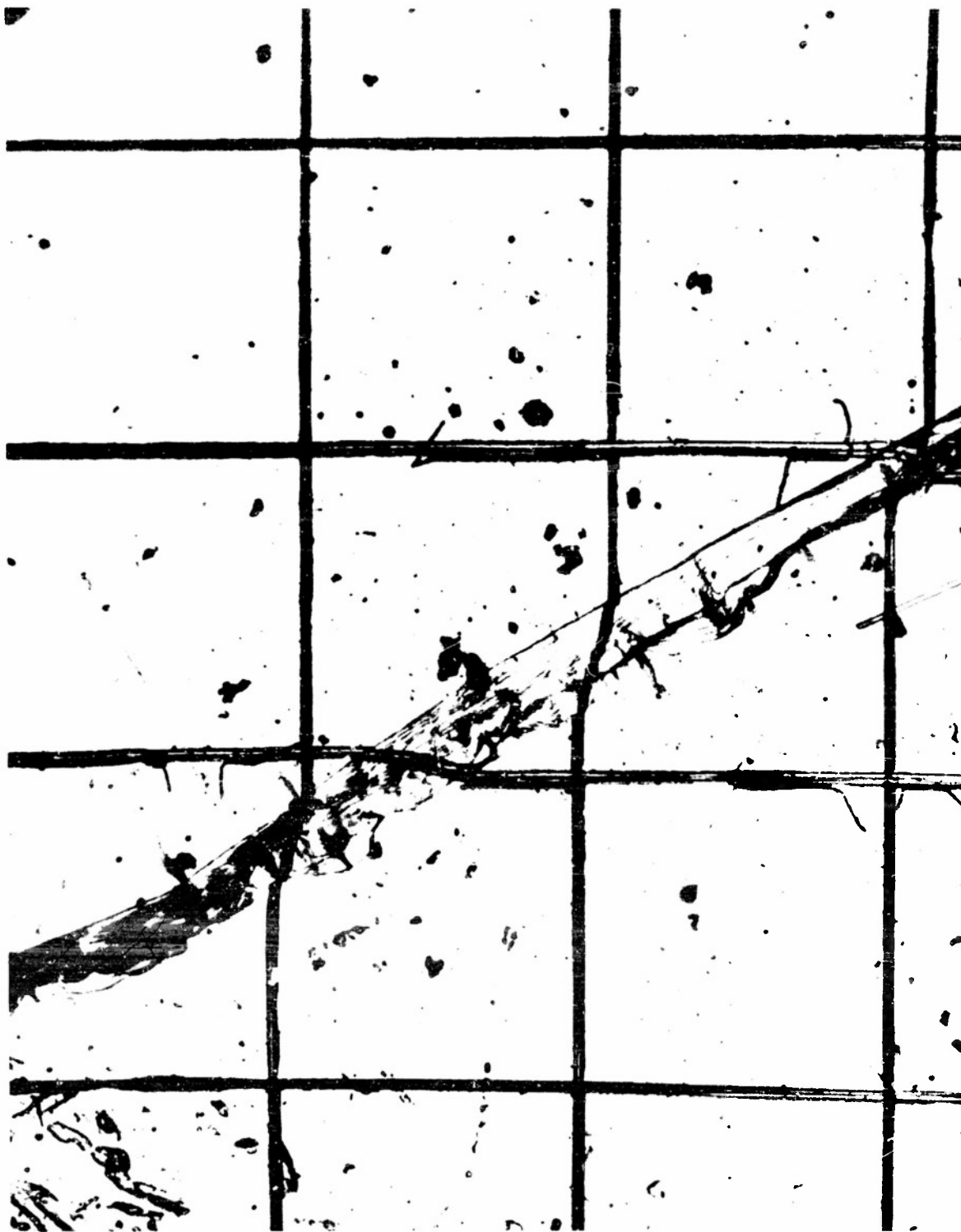


FIG 3 APPARENT BENDING OF GRID LINES NEAR THE  
GRAIN BOUNDARY AS A RESULT OF ALTERNATE  
GRAIN BOUNDARY SHEARING AND MIGRATION IN  
HIGH PURITY ALUMINUM.  
CREEP STRESS = 250 PSI,  $\epsilon_t = 0.07$ ,  $T = 747^\circ \text{K}$ , MAGNIFICATION 200X.

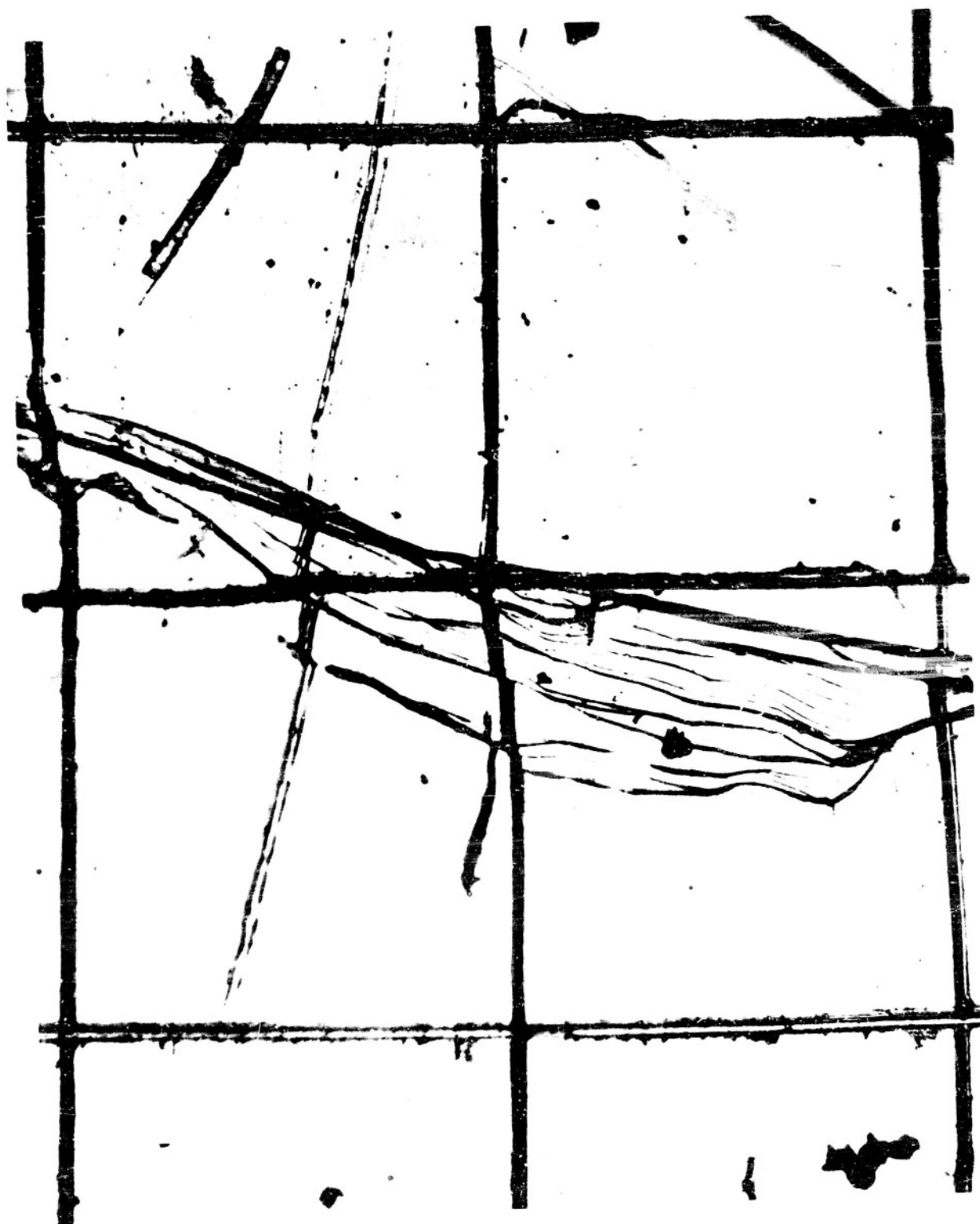


FIG. 4 MULTIPLE BOUNDARY SHEARING AND MIGRATION  
IN HIGH PURITY ALUMINUM.  
CREEP STRESS = 250 PSI,  $\epsilon_t = 0.07$ ,  $T = 747^\circ\text{K}$ ,  
MAGNIFICATION 300 X.

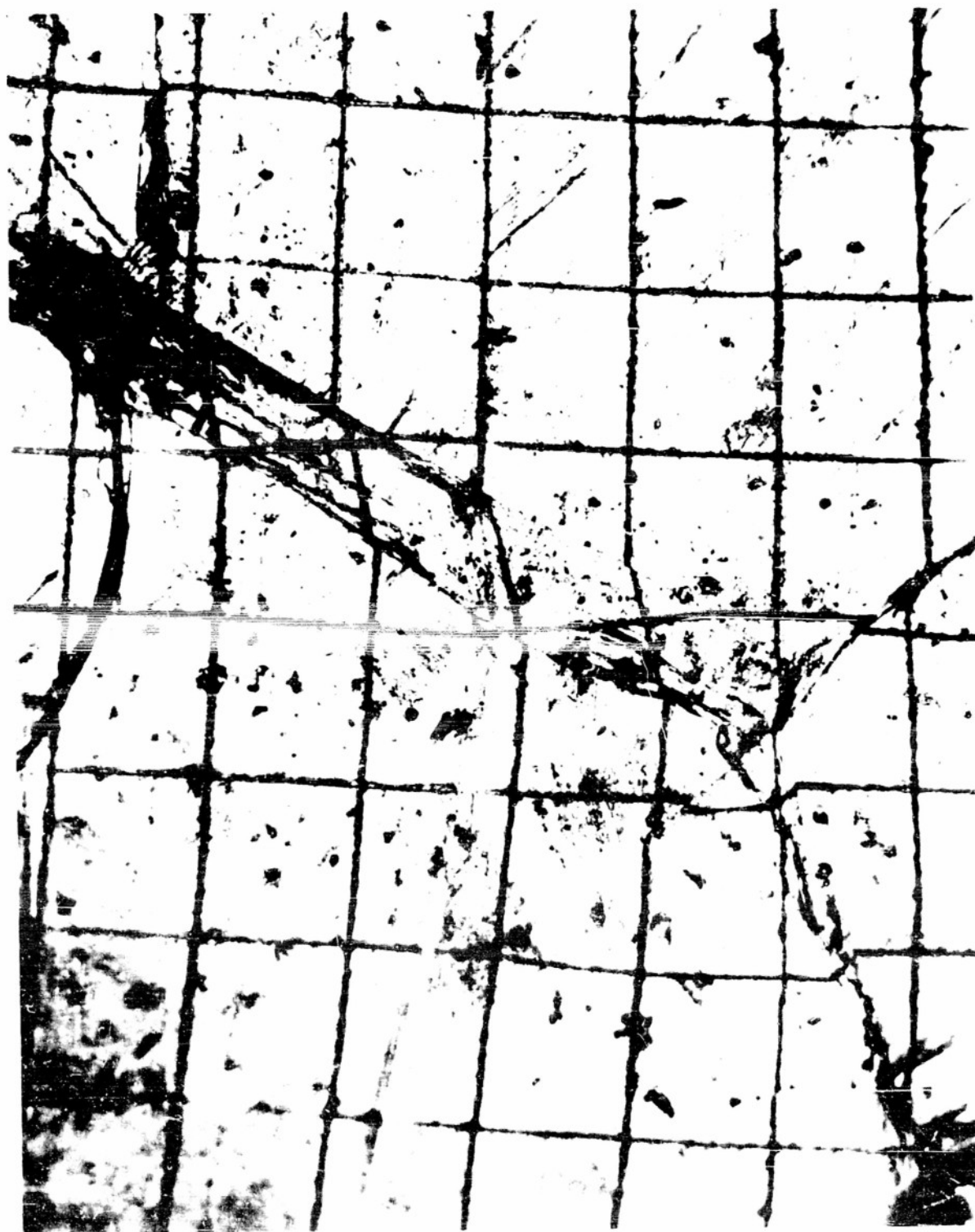


FIG. 5 COMPLEX DEFORMATION OF THE GRID NEAR A  
GRAIN BOUNDARY REGION DUE TO MULTIPLE GRAIN  
BOUNDARY SHEARING AND MIGRATION PROCESSES.  
CREEP STRESS = 250 PSI,  $\epsilon_t = 0.11$ ,  $T = 700^\circ\text{K}$ , MAGNIFICATION 100 X.



FIG. 6 OCCURRENCE OF POLYGONIZATION NEAR THE  
GRAIN BOUNDARY DURING CREEP AT 250 PSI.  
MAGNIFICATION 200 X.

longitudinal components of the average shear displacement per grain boundary,  $\bar{k}$ , by the average number,  $n$ , of grain boundaries intercepted per inch along the axis of the specimen, namely  $n = 10$ . These data are incorporated in Table II.

TABLE II  
Experimental Results

Temp. °K	Time Hours	$\epsilon_T$	$\bar{k}$ Inches	$\epsilon_{g.b.}$	$\frac{\epsilon_{g.b.}}{\epsilon_T}$	$\Delta H$ cal/mole
610	9.7	0.037	0.00035	0.0035	0.095	38,500
747	0.029	0.037	0.00031	0.0031	0.085	
610	20.4	0.070	0.00055	0.0055	0.079	35,000
747	0.105	0.070	0.00051	0.0051	0.073	
610	55.5	0.110	0.00085	0.0085	0.076	38,000
640	14.42	0.110	0.00071	0.0071	0.065	
700	1.215	0.110	0.00071	0.0071	0.065	
747	0.15	0.110	0.00075	0.0075	0.068	

#### DISCUSSION OF RESULTS

Previous investigations<sup>(5-8)</sup> have established the validity of Eq. 2 and have shown that the activation energy for creep of pure aluminum and its dilute alloys is about  $\Delta H = 36,000$  calories per mole independent of subgrain structures, cold work or grain size. According to Eq. 2 the times to achieve the same strain for two creep tests under the same stress and different temperatures are given by

$$t_1 e^{-\Delta H/RT_1} = t_2 e^{-\Delta H/RT_2} \quad (3)$$

Using the times  $t$  and temperatures  $T$  given in Table II to achieve the same total strain,  $\epsilon_T$ , the activation energies  $\Delta H$  given in the last

column of Table II were calculated. The agreement that was obtained with previous results again confirms the nominal validity of Eq. 2.

The data of Table II further reveal that essentially the same value of  $\epsilon_{g.b.}$ , is obtained for the same total creep strain,  $\epsilon_T$ , independent of the test temperature. Consequently, these data prove that

$$\epsilon_{g.b.} = f_{g.b.} (t e^{-\Delta H_{g.b.}/RT}) \quad \sigma = \text{const.} \quad (4)$$

where the activation energy  $\Delta H_{g.b.}$  for grain boundary shearing is identical with that for the total creep process, namely about 37,000 calories per mole. These correlations suggest that the rate-controlling process for grain boundary shearing is the same as that for creep as a whole since both processes exhibit the same activation energy. Since the activation energy for creep and stress-rupture<sup>(9)</sup> appears to be that for self-diffusion<sup>(5)</sup>, grain boundary shearing also appears to be dependent on a self-diffusion mechanism.

Rhines and Cochardt<sup>(10)</sup> investigated the shearing of bicrystals of high purity aluminum along their mutual grain boundary over the range from 473° to 923°K. They observed that the amount of grain boundary shearing increased with increase in difference of grain orientations. In another phase of their investigation they studied the effect of temperature on the displacement of the grain boundary under a stress of 100 psi for bicrystals whose relative crystallographic orientations were maintained constant for all samples tested. In terms of the suggestions of the current investigation their results should be correlatable by means of the  $\Theta$  parameter. Such a correlation is shown in Fig. 7, wherein an average activation energy of about 40,000 calories per mole was obtained.



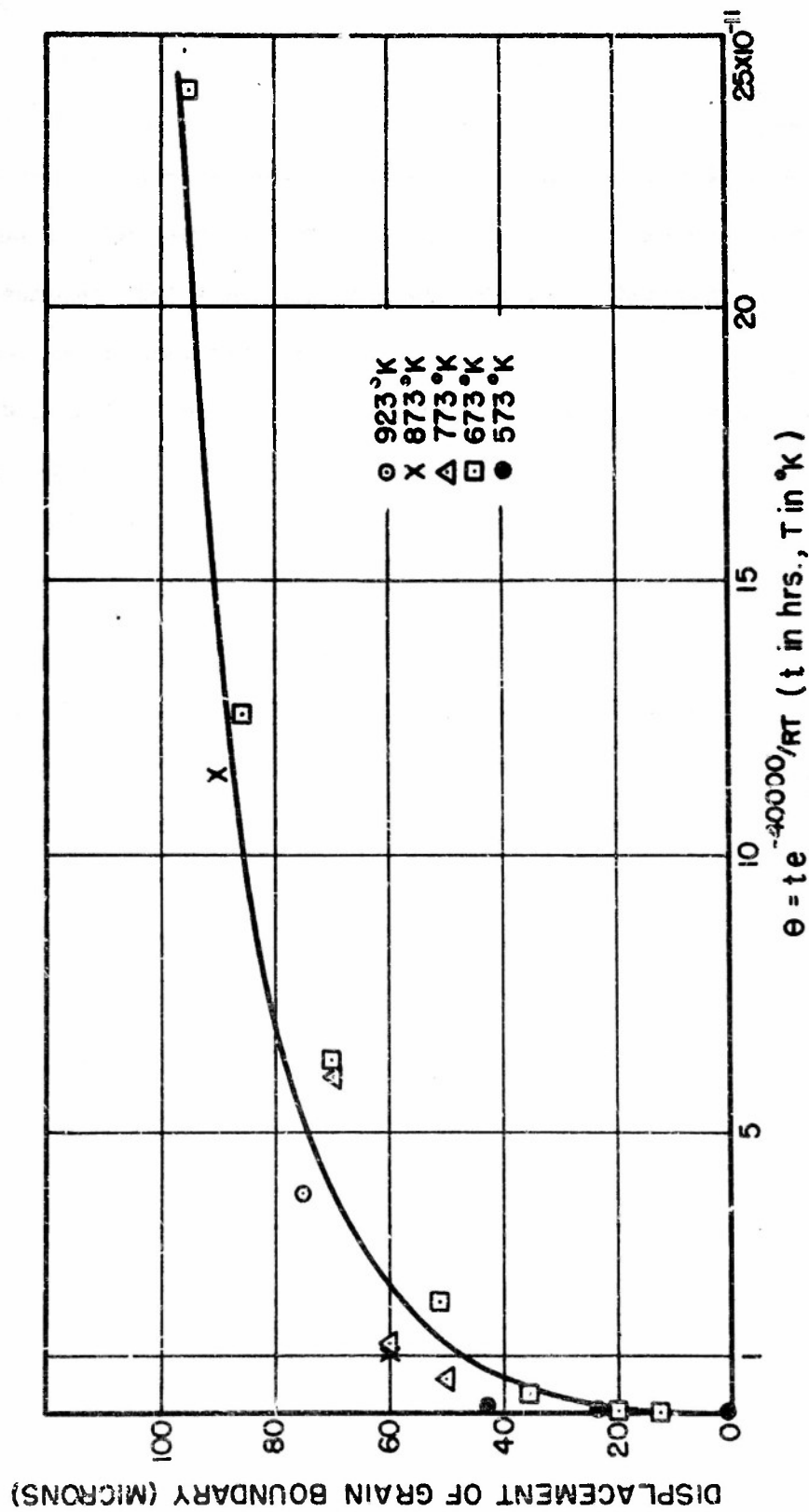


FIG. 7 GRAIN BOUNDARY DISPLACEMENT AS A FUNCTION OF  $\theta$  FOR HIGH PURITY ALUMINUM UNDER A STRESS OF 100 PSI. [DATA OF RHINES AND COCHARCT (10)]

Considering the difficulties in experimental techniques and the effect of sampling errors on this interesting investigation, the "displacement- $\Theta$ " curve appears excellent. The value of the activation energy obtained from Rhines and Cochardt's data is essentially equal to the activation energy obtained in the present investigation, and, furthermore, their results again suggest that the  $\Delta H_{g.b.}$  for grain boundary shearing is practically identical with that for creep.

McLean<sup>(1,4)</sup> has shown that the contribution of grain boundary shearing to the creep strain,  $\epsilon_{g.b.}$ , varies with the duration of the test,  $t$ , in the same way as the total creep strain,  $\epsilon_t$ , varies with time. The fact that the ratio of  $\frac{\epsilon_{g.b.}}{\epsilon_t}$  given in Table II is practically constant further substantiates McLean's original observations. Consequently, grain boundary shearing exhibits primary and secondary creep characteristics that are wholly compatible with those exhibited by the total creep strain. This is also suggested by the data given in Fig. 7 deduced from Rhines and Cochardt's investigations. Obviously, the decreasing rate of grain boundary shearing during the primary stage of creep demands that structural changes are improving the resistance to grain boundary shearing in the same way as they serve to improve the total creep resistance. This strongly suggests that grain boundary shearing is dependent on the crystallographic processes of dislocation migration in the adjacent grains on each side of the boundary. This conclusion is further substantiated by the observation that polygonization is frequently more extensive in the vicinity of a boundary than in the core of the grains.

Perhaps grain boundary shearing occurs by the following mechanism. Shear stresses are relaxed at the grain boundary. Consequently, high bending and shear stresses are concentrated on the interpenetrating

projections of each pair of grains across their mutual irregular boundary. If the stresses are relatively small, as in the case of damping capacity or shear modulus relaxation experiments, the straining occurs by shear displacements in the true grain boundary, and no permanent changes in the structure of the grains themselves occur. Under these conditions the properties of the grain boundary will be history independent and anelastic, exhibiting a viscous behavior. But under high stresses grain boundary relaxations induce sufficiently high bending and shear stresses on the grain projections in the vicinity of the grain boundary to cause these regions to undertake crystallographic mechanisms of deformation. Because of this bending, it might be expected that high polygonization would be induced in the grain boundary region as is observed experimentally. Furthermore, the accompanying strain as revealed by grain boundary shearing,  $\epsilon_{g.b.}$ , should then follow the usual history dependent laws for crystallographic mechanisms of deformation. The grain boundary itself merely serves to facilitate this crystallographic deformation process by permitting concentrations of stress on the grain projections due to stress relaxations along the true boundary.

A large number of different investigators<sup>(10-12)</sup> have reported that the contribution of grain boundary shearing to the total creep strain increases with increasing temperature. But the validity of Eq. 4 and the experimental data recorded in Table II indicate that the contribution of grain boundary shearing to the total creep strain is independent of the test temperature. In many of the previous investigations the higher temperature creep tests were conducted at lower stresses. Consequently, the increased contribution of grain boundary shearing to the total creep strain might have been ascribed to either the increased temperature of

test or the lower stress. The present investigation suggests that temperature per se may not have been the factor responsible for the previous observations. And McLean's investigations<sup>(4)</sup> have shown that the relative contribution of grain boundary shearing to the total creep strain increases as the stress is decreased in a series of constant temperature creep tests. Consequently, the previously reported apparent increase of grain boundary shearing with increase in the test temperature might have been due to the simultaneous decrease in the applied stress.

#### CONCLUSIONS

1. The contribution of grain boundary shearing to the creep strain for high purity aluminum follows the functional relationship

$$\epsilon_{g.b.} = f(\Theta) \quad \sigma = \text{constant}$$

where  $\sigma$  = creep stress,

$$\Theta = t e^{-\Delta H_{g.b.}/RT},$$

$t$  = duration of test,

$T$  = test temperature in degrees absolute,

$R$  = gas constant,

and  $\Delta H_{g.b.}$  = activation energy for grain boundary shearing.

2. a. The activation energy for grain boundary shearing is about the same as that for creep.
- b. The contribution of grain boundary shearing to the creep strain follows the same primary and secondary stages as exhibited by the total creep strain.
- c. Extensive polygonization is observed in the vicinity of grain boundaries.

These observations suggest that grain boundary shearing occurs by crystallographic mechanisms of deformation arising from bending and shearing of grain projections in the vicinity of the true relaxed grain boundary.

3. The relative contribution of grain boundary shearing to the total creep strain is independent of the test temperature, but as shown by McLean, it increases as the stress decreases.

#### ACKNOWLEDGMENTS

The authors wish to thank Mr. John Madeau who contributed much to the initial phases of this program. In addition, they wish to express their appreciation to the Aluminum Company of America for supplying the high purity aluminum.

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