TEMPERATURE DEPENDENCE
OF THE ACTIVATION VOLUME AND THE FLOW STRESS
OF CADMIUM SINGLE CRYSTALS

by

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ABSTRACT

Cadmium single crystals of 99.994% purity were tested by the strain rate change method to determine the activation volume over the temperature range from 77 K to 508 K. The temperature dependence of the flow stress and of the work hardening coefficient over the same range was also investigated. All three parameters exhibited an anomalous behavior at a temperature of about 275 K from that predicted by a single mechanism model as discussed by Seeger (26) for the same temperature range.

Theories are discussed to explain the observed results for cadmium as well as to be consistent with previously reported results on magnesium and zinc. It is concluded that the Seeger approach explaining these deformation parameters is correct with some extension and modification. It is believed that the deformation behavior is of a different nature below the temperature of about 275 K from that above. Specific models for these differences are discussed.
INTRODUCTION

The plastic deformation of metal single crystals can be divided into three stages, as given by the schematic stress-strain curve shown in Figure 1.

The first stage is called "easy glide." This is believed to be caused by slip on only one slip system. This stage is observed in f.c.c and c.p.h. single crystals, but it can extend to a much greater strain for the latter, approximately 100%. Stage I terminates by a change in the work hardening to a larger value, i.e. stage II. The slope of the curve during Stage II is constant. This portion is the result of duplex slip in f.c.c. metals and twin formation in c.p.h. crystals. Stage III sets in at a strain which increases with decreasing temperature and is believed to be associated with the operation of extensive cross slip.

A large number of investigations have been carried out on the plastic deformation of cadmium. These include studies of the flow stress dependence on temperature, strain-rate, surface conditions, impurities, and the creep behavior of cadmium crystals.

The only available flow stress data on cadmium known to the author is that of Schmid and Boas and Roscoe. Schmid and Boas' work was done in the 1930's. It is felt that their procedures were probably excellent for the times, but fall short of the standards set now. They did
not have the facilities to get very high purity specimens. Research over the last ten years has shown that reproducibility of the critical resolved shear stress for two different specimens of seemingly identical prior history cannot be realized accurately. Consequently comparison of results of different specimens is not felt to be reliable.

The work done by Glen on the creep of cadmium at low temperatures (1.2 - 90°K) is considered the most reliable information of its type on this metal. This work partly eliminates the above objections. Glen found that measurable creep occurs at 1.2 and 4.2°K and suggests that it is due to a quantum mechanical tunneling, allowing dislocations to penetrate barriers that cannot be overcome by the applied stress alone.

From 1935 to 1960 many investigators have shown the effects of surface films on cadmium crystals. The general effect presented in these papers is that the film keeps the dislocations from flowing out of the crystal and the indicated flow stress is definitely increased.

A yield point phenomenon, as described in the theory by Cottrell and Bilby, occurs in cadmium. Smith and Cottrell and Gibbons reported on this effect. The latter authors suggest that the effect might be caused by dissolved nitrogen, since the effect was found to be absent in specimens grown under an argon atmosphere. To the author's knowledge no follow up studies have been carried out in this area to conclusively prove that this "yield phenomenon" is due to the interaction of a dislocation and a nitrogen atom.

Seeger has developed a theory of the flow stress which he applies to all closed-packed structures. The basic concept is that the rate of straining, $\dot{\varepsilon}$, as prescribed by the experimental conditions determines a certain applied stress which will be necessary to move large
numbers of dislocations over energy barriers which occur in the glide plane. In general these barriers can be overcome with the aid of thermal activation. Therefore the strain rate can be described by ordinary reaction rate theory, but the stress dependence of the activation energy, \( u(x) \), must be specified. The strain rate is given by:

\[
\dot{\varepsilon} = N A b \lambda_0 \exp \left[ -\frac{u(x)}{kT} \right]
\]

(1)

where \( N \) - the number per unit volume of sites at which activation takes place,

\( A \) - the average area swept out by each dislocation segment in going from one activation site to the next,

\( b \) - the dislocation strength or Burger's vector,

\( \lambda_0 \) - the frequency factor describing the number of attempts per second the dislocation makes to overcome the barrier, which depends on the type and spacing of the obstacles,

\( u(x) \) - the activation energy, a function of stress.

The activation energy is assumed to be linearly dependent upon the stress.

\[
u(x) = \mu_0 - T_{\text{eff}} \nu
\]

(2)

The effective stress is the applied stress minus the internal stress.

\[
T_{\text{eff}} = T_A - U_G
\]

\( U_G \) is that energy barrier which must be overcome without thermal assistance. The energy term which aids in crossing the barrier is the product of the total force, which is the effective stress times the strength of the dislocation times the dislocation segment length, and the activation distance, which is the width of the barrier. The three length terms are combined and called the activation volume.
\[ \gamma = b d l_0 \]  

where \( b \) - the strength of the dislocation

\( d \) - the activation distance,

\( l_0 \) - the length of dislocation between obstacles.

Equation 1 may be solved for the applied stress \( \tau \)

\[ \tau = \tau_0 + \frac{\mu_0 - kT \ln (NAb \lambda_0 / \dot{\varepsilon})}{V} \]  

\( \tau \leq \tau_o \) \hspace{1cm} (4)

and

\[ \tau = \tau_0 \]  

\( \tau > \tau_o \) \hspace{1cm} (5)

where \( \tau_o \) is given by:

\[ kT_0 = \frac{\mu_0}{\ln (NAb \lambda_0 / \dot{\varepsilon})} \]  

\hspace{1cm} (6)

If all terms except the strain rate and the applied stress are taken as constants for one test, the change in flow stress due to a change in strain rate can be written as:

\[ V (\tau - \tau_o) = kT \ln \frac{\dot{\varepsilon}}{\dot{\varepsilon}_o} \]  

\hspace{1cm} (7)

Conrad and Robertson\(^5\) investigated magnesium single crystals over the temperature range of 78 to 368°K. Using the equation

\[ \frac{V}{kT} = \ln \frac{\dot{\gamma}_{n+1}}{\dot{\gamma}_n} \frac{1}{\Delta t} \]  

\hspace{1cm} (8)

they found that \( V/kT \) was constant from 78 to 203°K. At higher temperatures (from 203 to 368°K) a strain effect seemed to give higher values. The tests were stress increment creep type where the strain rate was measured before and after the incremental change in stress.

Thornton and Hirsch\(^2\) conducted tests on copper and gold polycrys-
tals and found that $\nu \tau$ was insensitive to stress, prestrain, and the size of the stress increment. However, $\nu \tau$ was larger for the higher temperature. The stress increment creep tests were run at 80 and 300°K. The relationship they used is

$$\nu \tau = - k T \Delta (\ln \Delta t) \sigma / \Delta \sigma$$

where

$$\Delta = \frac{N A b \lambda}{C}$$

and $C$ is the slope of a plot of $\epsilon$ as a function of $\ln t$. The change in $\ln \Delta t$ is obtained from a plot of strain versus $\ln$ time on which the curve subsequent to the increment is moved along the axis of $\ln t$ until its slope is equal to that of the initial curve. They could not get an absolute value of $\nu$ since they did not know the shear stress on the glide plane.

Adams and Cottrell\(^1\), in studying the flow stress in copper crystals, found that the change in flow stress due to a sudden change in temperature was proportional to the flow stress except in the strain region of less than 5%. Cottrell and Stokes\(^9\) examined the reversible change of the flow stress with temperature in aluminum. They concluded that the temperature dependent part of the flow stress is proportional to the total flow stress.

Hirsch, Mitchell, and Thornton\(^{13}\) found for copper single crystals that $\nu \tau$ is small and insensitive to strain initially, then increases with strain until it is again independent of strain. This was believed to be due to the change in the dislocation network with strain. The rate change method of testing was employed. The relation used for the interpretation of their data is:
where $\sigma$ is the normal or tensile flow stress measured.

Seeger analyzed some results obtained by Boas and Schmid for cadmium single crystals. From the temperature dependence of the flow stress at two strain rates (Figure 17a), Seeger determined the activation energy by:

$$
\ln \left( \frac{\dot{\varepsilon}_1}{\dot{\varepsilon}_2} \right) = \ln \left( \frac{T_2}{T_1} \right) e^{\frac{kT_1}{\Delta \sigma}}
$$

where $\dot{\varepsilon}_i$ is the strain rate at temperature $T_i$.

For strain rates of $2 \times 10^{-3}$ and $2 \times 10^{-1}$, the temperatures were 470 and 560°K. These values give an activation energy of 27 kcal/mole.

By letting $d$ and $b$ be $2.97 \times 10^{-8}$ cm, a value of $3.4 \times 10^{-8}$ cm was calculated for $l_o$, the average length of dislocation between obstacles. Equations 3 and 4 are used for this calculation, with $T = 0$.

By using the results found by Roscoe for room temperature deformation of cadmium single crystals given in Figure 17b, Seeger calculated $l_o$ and found exact agreement with the previously computed value ($l_o = 3.4 \times 10^{-8}$ cm). Figure 17b is a plot of the flow stress as a function of the logarithm of the strain rate.

Seeger gives another plot of cadmium data from the work of Schmid and Boas. Figure 17c shows the temperature dependence of the flow stress for cadmium single crystals. Comments on this data were given earlier in this section.

This investigation was an attempt to show the validity of the equa-
tion of state, equation 1, through the correlation between experimental results and existing theories.
EXPERIMENTAL PROCEDURE

Specimen Preparation

High purity (99.994%) cadmium was purchased from the American Smelting and Refining Company, South Plainfield, N. J. From this bulk material 0.5 inch diameter single crystals were grown under an argon atmosphere by the Bridgman technique. After growth the specimens were spectrographically analyzed and found not to have gained additional impurities. Table 1 gives the results of the spectrographic analysis before and after growth. The higher purity after growth is probably due to the fact that the tip or end to freeze first of the crystal was analyzed only. This portion has actually been zone-refined by one pass.

An etching technique was developed to reveal if grown specimens were single crystals. A solution of 95 ml CH₃OH, 1 ml H₂SO₄, and 5 ml HNO₃ caused distinct reflections from different grains. Since a film forms quite readily on cadmium, the crystals were washed with alcohol, then water, and dried under a cool air blast.

For this work knowledge of the orientation of the basal plane and the expected slip direction with respect to the tensile axis of the specimen was essential. The crystal orientation was obtained by the Laue back-reflection technique as described in Barrett³. Table 2 gives the orientations of the crystals tested.

For all the orientation work on undeformed specimens, x-rays from a chromium target energized at 30 kv and 2½ ma for 2½ hours were used with a 0.025 inch pin hole collimator. A change in x-ray procedure was made in order to extend the life of the target. For determining the change in orientation due to straining, x-rays came from a silver target energized at 30 kv and 27 ma for 45 minutes with the same collimator. For
all x-ray studies standard x-ray film and developed techniques were used.

A solution of $10 \, g \text{CrO}_3$, $3 \, ml \text{H}_2\text{SO}_4$, and $100 \, ml \text{H}_2\text{O}$ was used to polish the crystals. The normal procedure of removing the chromate film with a $2\% \text{H}_2\text{SO}_4$ solution was used before washing and drying the polished specimens. This polished surface was essential in making observations of the slip bands formed on the surface of a deformed specimen.

Aluminum grips were designed with threads tapped in one end and with the internal diameter of the other end slightly greater than the diameter of the specimen. These grips were attached to the specimen with Shell Epon VI adhesive. The threads were used to attach the specimen to the tensile machine by the use of universal joints which assisted in aligning the specimen for axial tension.

The specimens were annealed for one hour at $115^\circ C$ to allow the Epon to cure. Additional annealing at $200^\circ C$ and $250^\circ C$ was used to reduce any accidental straining due to handling.

Testing

The measurement of the applied load was read directly on the load-time recorder of an Instron tensile machine. The load was measured through a load cell which employs a highly sensitive electronic weighing system of bonded-wire strain gages for detecting and recording. The recording of the load was done through an A-C bridge circuit into the recorder pen circuit. The load measurements were accurate to $\pm 0.5\%$ of the full scale deflection which was either 20 or 50 pounds.

Cross-head speeds of $2 \times 10^{-4}$ and $2 \times 10^{-2}$ inches per minute were used in the strain rate change method of testing. The chart was driven at 0.2 and 20 inches per minute so that the initial slopes of the curves,
i.e. the elastic region, would be the same for each test. The strain was calculated directly from the speed of the cross-head and the chart, and the known gage length of the specimen. An intermediate cross-head speed of $2 \times 10^{-3}$ inches per minute and a chart speed of 2 inches per minute were employed to obtain the data on the dependence of the flow stress with temperature.

The measured load had to be resolved into a stress acting on the slip plane in the slip direction for an exact interpretation of the results. To accomplish this resolution, use was made of the orientation factors. These factors are the cosines of the angles between the tensile axis and the normal to the basal plane and the slip direction. When the crystal is deformed in tension the basal plane tends to rotate toward the tensile axis and the slip direction moves in a manner as to align itself with the tensile axis. The result of these movements is a change in the area and in the orientation factors. A useful means of calculating the change in the cosine factor for each test is to use a proportional change with the amount of strain for that test. The relationships for the change in angle with strain are:

$$\frac{\ell}{\ell_0} = \frac{\sin \psi}{\sin \psi_0}$$
$$\frac{\ell}{\ell_0} = \frac{\cos \theta}{\cos \theta_0}$$

where $\ell$ = length of specimen,

$\psi$ = angle between tensile axis and nearest slip direction,

$\theta$ = angle between tensile axis and the normal to the basal plane,

$\ell_0, \psi_0, \theta_0$ = original values for the undeformed specimen.

The cross-sectional area for each test can be determined from the calculated change in the angle between the basal plane and the specimen axis.
Temperature equilibrium was obtained by immersing the specimen in a controlled temperature bath (Figure 2). The lowest temperature employed was that of boiling liquid nitrogen, 77°K. A dewar flask with isopentane and immersion copper tubing carrying liquid nitrogen was used for attaining temperatures between 150 and 295°K. The same dewar with an additional heating coil and an oil bath in place of the isopentane was employed for attaining temperatures above room temperature. Three iron-constantan thermocouples were placed near the specimen and a Wheelco proportional controller was operated by a fourth thermocouple in the bath. The bath was stabilized by the controller and the temperature at the specimen was checked by the other three thermocouples. The temperature in the bath did not vary more than \( \pm 1^\circ \) over the length of the specimen.

To run a strain rate change test, the load was increased at the slow rate of straining until a definite plastic flow was observed. The load was taken off at the fast rate and then reapplied at the fast rate. The change in flow stress was measured between the stress level at which flow was stopped at the slow rate and that stress at which the crystal began to flow at the fast rate. The time required for a change in strain rates was about 2 seconds.

The flow stress at the faster strain rate was taken as the intercept of a straight line extension of the easy glide region with the elastic portion of the curve. This procedure was used for medium and low temperatures. This neglected the yield point effects and any irregularities due to pen response. The value of the flow stress at liquid nitrogen temperature after measuring the flow stress at a higher temperature was taken as the first definite deviation from linearity. The reason for the change in procedure is that a film formed on the crystal
during the changing of the baths which raised the measured flow stress. Thus by using the stress level that indicated dislocations were beginning to move out the surface, results would be obtained that were comparable to the results on activation volume. A similar effect due to an oil film was observed during the rate change tests at the high temperatures (greater than 400°K) and fast strain rates. The same corrective measures were applied.

It was observed that several flow stress ratio points were determined when a flat easy glide region existed for both the high temperature and the liquid nitrogen temperature yield. Temperatures at which the ratio points exist for this type of result are 222, 263, 294, 348, and 372°K.

Several series of strain-rate change tests were run going down in temperature and coming back to the starting temperature. Additional randomness was achieved in the data by performing the liquid nitrogen and room temperature tests before the series was run in the isopentane bath.

The temperature dependence of the flow stress was determined by obtaining a flow stress at one temperature, then changing as soon as possible to a liquid nitrogen bath and measuring the new flow stress at the lower temperature. Many difficulties were encountered in these tests. It was not possible to make an instantaneous change to the low temperature since the surrounding liquid of the elevated temperature bath would freeze in the liquid nitrogen, and the crushing of ice or frozen oil would be measured instead of the flow of the crystal. A time lapse of 3 to 8 minutes between the changing of the baths was generally observed. During this time the essential gripping devices were wiped clean to re-
duce the previously mentioned problem. Finally a suitable technique of cleaning and raising the bath was devised by conducting standard tests on a steel specimen. The maximum curvature due to any ice or oil film was measured and taken into consideration for the interpretation of the experimental results.
RESULTS

The activation volume was determined through a series of rate change tests over a wide range of temperature. The results of these tests are shown in Figures 3 - 7. The 'hump' in these curves around 275°K was the first indication that something unusual was occurring. In order to confirm these results, the temperature dependence of the normalized flow stress was found (Figure 8). This plot shows again the change in mechanism at a temperature near 275°K.

By normalization it is meant that the temperature dependence of the elastic constants is removed from the values of the flow stress by dividing each value by the value of the shear modulus at the temperature the flow stress was obtained. The temperature dependence of the shear modulus was obtained from the adiabatic measurements reported by Garland and Silverman. Their results covered the temperature range 0 to 300°K. A straight line was used to extrapolate for values of the modulus at higher temperatures.

The work hardening coefficient remained less than \(100 \times 10^6\) dynes per centimeter squared over the entire temperature range, indicating continuation of "easy glide". The temperature dependence of the normalized work hardening also indicates a change in mechanism at the same temperature as the others (Figure 9). Table 3 gives the values of the normalized work hardening coefficients \(\frac{\Theta}{T^2/G_0}\) for four crystals.

The strain dependence of the activation volume is given in Figure 10. This shows that over a wide range of strains there is not a sufficient variation to believe that the temperature dependence of the activation volume would be drastically affected by the difference in strains between tests. The strain for a series of tests was about 10%, which is
less than the maximum strain of 15% shown in Figure 10.

The strain dependence of the flow stress at 297°K with respect to the flow stress at 77°K remained reasonably constant over a large strain range (Figure 11). Therefore it was decided that by normalizing each flow stress to that at 77°K, the flow stress could be determined without considering the strain. To insure that recovery effects did not affect the normalized flow stress values, a test was run at 308°K with a long duration between initial deformation and testing the crystal again at liquid nitrogen, i.e. approximately 10 minutes. This point was compared to values at the same temperature for shorter transition times. If recovery was affecting the results, the flow at nitrogen should have been lower and the ratio would be larger. The point was found to be lower than those of the prior runs for that temperature with shorter recovery times.

Figure 12 is a plot of stress versus strain for a series of rate change tests. The incremental change in stress, the temperature, and the rates of straining determine the activation volume. Figure 13 shows a typical temperature dependence of the slope of the plastic region of the stress-strain curve.

Figure 16 shows a typical set of glide lamellae at 50x. Repeatedly throughout the work the surface of the specimens was investigated for slip bands and the results reported apply to specimens deformed on one glide plane. These metallographic observations occasionally showed a twin, but these were most infrequent and not of sufficient number or magnitude to affect any of the reported results.

Usually during initial loading of a new crystal at all temperatures, or for the deformation at the slow rate above 350°K, some jerky flow was
exhibited. This jerkiness settled down after a small pre-strain. In
the cases where jerkiness was observed, the crystals were pre-strained
beyond the jerky region before making strain rate or flow stress change
tests.

A test was run at 273°K to check the recovery during unloading and
reloading at the fast strain rate. This resulted in no appreciable
effect as the crystal began to flow at the same level as that where flow
was stopped. That is the flat region representing easy glide was an ex-
tension of the prior easy glide region exactly, although some rounding
around below this level was detected.
DISCUSSION

Work Hardening

We consider two sources of hardening during easy glide in hexagonal closed-packed metals. To describe these, we make use of the following model.

There exists a dislocation forest threading the basal plane which is composed of primarily screw dislocations. The number of forest dislocations per unit area will be denoted as $N''$. We assume each potential dislocation source has the area $l_0^2$. Therefore the density of sources will be $1/l_0^2$. The distance between forest dislocations is also $l_0$.

Since this theory deals with macroscopic yielding, we can say that all sources operate at about the same stress level.

Dislocations are given off by these sources, but they do not overcome the forest barriers. Therefore a certain number ($n$) of dislocations pile up in each area $l_0^2$. The strain generated by these dislocations is

$$
\varepsilon_i = n \ N'' \ b \ L
$$

where $L$ is the average distance moved by a dislocation.

There is an elastic interaction since the next dislocation must overcome the stress field of the pile-up. This stress is given by

$$
\tau_G = \alpha \ n \ G \ b \sqrt{N''}
$$

where $\alpha$ = constant depending on orientation;

and the work hardening will be given by

$$
\frac{d\tau_G}{d\varepsilon_i} = \theta_E = \frac{\alpha \ G}{L \sqrt{N''}}
$$
Equation 12 gives the first contribution to the hardening. \( N'' \) and \( L \) are constants, so this term is mildly temperature dependent as \( G \) decreases with increasing temperature.

A second contribution to the strain hardening arises from the consideration that after a few dislocations have piled up, the leader breaks through the forest. Once the first dislocation breaks through there is a catastrophic break-through of edge dislocations in succeeding groups. The net effect is a movement of an edge dislocation front through the crystal. We assume that only the edge dislocations are able to move at low temperatures and only the screws at high temperatures.

The additional strain, \( \varepsilon_2 \), for loops becoming longer than \( L_0 \) is given by

\[
\varepsilon_2 = \frac{N \sigma \beta \delta}{a} \tag{13}
\]

where

- \( a \) = the mean spacing between active slip lines,
- \( N \) = the density of moving dislocations on the basal plane, number per unit area,
- \( R \) = the distance the edge moves.

Now as the dislocations cut through the forest they acquire jogs. A jog will be generated at each intersection of the gliding dislocation and a forest dislocation. Therefore the number of jogs accumulated by the cutting of the forest dislocations will be \( N''L_0R \). The number of deformation jogs per unit length of dislocation \( (n_j) \) is equal to \( N''R \).

We assume that all jogs move conservatively in edge dislocations. The conformity plane of the jog will be defined by the Burger's vector of the gliding dislocation and the Burger's vector of the cut dislocation. In general the jogs will not be in the basal plane and will therefore take an additional amount of stress to move them. The additional force
per unit length is

\[ \Delta F = (T_{\text{eff}} - T_s) b \]  

(14)

where \( T_s \) is the critical stress to move a jog on its glide plane.

This force must act over the length of the jog. So that

\[ \Delta T = T_s b \frac{\Delta l}{l} \]

i.e. the force necessary to move the jog must come from an additional increment of the applied stress.

\[ \Delta T = T_s \frac{b}{l} \]

where \( l \) is the new equilibrium spacing of jogs on edge dislocations.

The length per jog is \( 1/N^r \). This gives

\[ \Delta T = T_s b N^r \]  

(15)

From equation 13

\[ R = \frac{\varepsilon_2 a}{N b l_0} \]

then

\[ \Delta T = T_s N^r \varepsilon_2 a \]

and

\[ \frac{d T_s}{d \varepsilon_2} = \theta_2 = \frac{T_s N^r a}{l_0 N} \]  

(16)

where we assume \( (N/N^r \varepsilon_2) = 0 \).

This contribution to the work hardening has as yet an unknown temperature dependence through \( T_s \) and the elastic constants. There is also a weak strain dependence over the first \( 5\% \) deformation due to the variation of \( N \) with strain. That is the contribution to work hardening from jogs can be considered from two view-points. The first is to assume \( T_s \) only temperature dependent through the elastic constants and is independent of the dislocation spacing between partials. A temperature dependence to the work hardening can then arise through interaction of the split disloc-
tion and point defects. The second temperature dependent hardening can arise quite easily if \( \tau_s \) is assumed dependent upon the spacing of the partials and by applying the assumption that the stacking fault energy decreases with increasing temperature as suggested by Thornton and Hirsch. This model will be discussed later.

As edge dislocations sweep out more area in the slip plane they will encounter vacancies randomly spaced in the lattice. The forming of diffusional jogs by vacancies that are picked up will add another term to this part of the work hardening.

We assume that the energy of formation of vacancies is less than the energy of migration of vacancies. The thermal equilibrium concentration of vacancies is given by

\[
C_v = C_0 \exp \left( -\frac{u_f}{kT} \right)
\]

where \( C_0 \) - the number of atoms per cm\(^3\),

\( u_f \) - the formation energy of a vacancy.

There will be a finite probability of the vacancy becoming attached to the moving dislocation. This probability can be given by

\[
\rho \propto \exp \left( -\frac{(u_f - U_i)}{kT} \right)
\]

where \( U_s \) - the energy of attaching the vacancy to an edge dislocation, independent of width of the dislocation,

\( U_i \) - the interaction energy of the vacancy and the edge dislocation, both electrical and elastic interaction.

Now the net probability will be

\[
C = C_0 \exp \left\{ -\frac{(u_f - U_i + U_s)}{kT} \right\}
\]

where \( C \) is now the concentration of vacancies per unit volume which attach themselves to the moving dislocations and form diffusional jogs.

The number of diffusional jogs per unit length will be
so that the length per jog is
\[ \frac{1}{C^{1/3}} R \]
and the necessary stress increment is
\[ \Delta T' = T_j \cdot C^{1/3} R \]
where \( T_j \) is the stress to move the diffusional jog.

The portion of the work hardening due to the moving of the dislocations through the crystal is
\[ \left( \frac{dT}{d\varepsilon} \right)_\varepsilon = \Theta_j = \frac{a}{N \ell_0} \left\{ \frac{T_s N''}{\ell_1} + \frac{T_j}{C^{1/3}} C^{1/3} \frac{dN}{d\varepsilon} \right\} \]
and the overall work hardening will be given by
\[ \Theta = \Theta_j + \Theta_2 = \frac{aG}{L' \sqrt{N''}} + \frac{a}{N \ell_0} \left\{ \frac{T_s N''}{\ell_1} + \frac{T_j}{C^{1/3}} C^{1/3} \frac{dN}{d\varepsilon} \right\} \]
but since it is assumed \( \frac{dN}{d\varepsilon} = 0 \), and \( 1_o \) is approximately \( a \), the work hardening normalized for the temperature dependence of the elastic constants will be
\[ \frac{\Theta}{\Theta_0} = \frac{a}{L' \sqrt{N''}} + \frac{1}{\Theta_0 G \ell_1} \left\{ \frac{T_s N''}{\ell_1} + \frac{T_j}{C^{1/3}} C^{1/3} \left[ \exp \left( \frac{u_s - u_1 + u_3}{kT} \right) \right]^{2/3} \right\} \]

We now impose a large restriction on the theory: we believe that dislocation pile-ups are unimportant at low temperatures and the elastic interaction is weak. So that the work hardening is determined by
\[ \frac{\Theta}{\Theta_0} = \frac{T_s N''}{C \ell_1} + \frac{T_j}{C \ell_1} \left[ \exp \left( \frac{u_s}{kT} \right) \right]^{2/3} \]
where \( U_0 \) is given by

\[
U_0 = U_f - U_I + U_s
\]

At intermediate temperatures the pile-ups become important due to
the blockage by polygonization walls (subgrain formation) and the work
hardening is determined by equation 20.

At high temperatures dislocation climb is so easy that pile-ups do
not offer any further resistance and \( L \) approaches the dimensions of the
crystal making the first contribution to the hardening approach zero.
The rate of annihilation of jogs on the dislocation line increases and
an equilibrium is reached between those created and those annihilated.
Since no additional stress will be necessary to move the dislocations,
this part of the hardening will also go to zero.

The activation energy for the vacancies to attach themselves to the
moving edge dislocations and create jogs can be found from the value of
the slope of the \( \ln \left( \frac{\theta}{\theta_0} - \frac{T_{s}' N''}{G N} \right) \) as a function of \( 1/T \).

\[
\frac{d \ln \left( \frac{\theta}{\theta_0} - \frac{T_{s}' N''}{G N} \right)}{d \left( \frac{1}{T} \right)} = -\frac{2}{3} \frac{U_0}{k}
\]

From Figure 14 the activation energy is 0.13 ev. By assuming
reasonable values for \( U_f \) (0.3 ev) and \( U_I \) (0.45 ev), a value of \( U_s \) is
found: \( U_s \) is 0.3 ev. To obtain this curve a reasonable value of \( \frac{T_{s} N''}{G N} \)
had to be assumed, which for CS 61 was \( 8 \times 10^{-6} \).

A qualitative explanation was sought for the mechanism where \( T_s \)
is assumed dependent upon partial dislocation separation. The normalized
work hardening coefficient from the results appeared logarithmic with
temperature. It seemed natural that the hardening increase may be di-
rectly related to the excess stress necessary to move jogs in extended
dislocations; the separation of the latter being temperature dependent.

The work hardening coefficient can be written as

\[ \beta = \frac{a}{l_0} \frac{N''}{N} \frac{l_5}{l} \]

from equation 16.

Since it is postulated that a change in the width of extended dislocations exists with temperature, it remains to be shown that there is a stress increment necessary to move the constricted portion of a dislocation; the stress increment being greater for more extended dislocations.

From dislocation theory it can be shown that the mutual repulsion force between extended dislocations is proportional to \( 1/x \), where \( x \) is the spacing of the two partial dislocations. Therefore every point close to the constriction will have a greater force of repulsion than that given by the force at \( x_0 \), i.e. the equilibrium spacing. Therefore some stress increment, which we will call \( l_5 \), is required to move the jogged extended dislocation through the lattice.

We would expect \( l_5 \) to depend linearly on the length over which the constriction occurs, or about linearly with \( d \) so that

\[ l_5 = KD = Ke^{u_0T} \]

where \( k \) and \( u_0 \) are constants for each crystal.

Now

\[ \frac{\beta}{G} = \frac{N''}{2N} Ke^{u_0T} \]

where \( l_0 = a \).

The experimental data for \( \ln \left( \frac{\beta}{G} \right) \) versus temperature is a straight line with a slope of \( W \beta/G \).
The values of $W_d$ and $W_{sf}$ for four crystals are given in Table 5. These should be the same for the assumptions made. The ratio of the two slopes is found to be approximately one for each crystal and the average of the four tests is unity.

Flow Stress

The flow stress has two separate regions that meet at the same temperature that the anomaly in work hardening occurs. The low temperature process is assumed to be the edge dislocations cutting through the forest of screw dislocations. At high temperatures the screw dislocations become more mobile and they control the flow stress.

For the low temperatures the activation energy and the activation volume increase with temperature. So from equation 4 we see that for a proper balance of these terms the flow stress can remain constant as shown by the experimental results.

When the gliding screw dislocations control the flow stress, the activation energy becomes constant. The observed result of this is a linear decline in the flow stress. The data demands that the separation of partials become temperature independent for the gliding screw dislocations in the high temperature region, i.e. $T > 275^\circ K$.

The temperature dependence of the constriction energy for both processes can be calculated in the following manner. An empirical relationship is determined from theoretically defined values of the stacking fault energy and the constriction energy given by Schock and Seeger.\(^2\)

$$E_c^e = Gd00 / \gamma^2$$  \hspace{1cm} (23)

where $E_c^e$ - the constriction energy for edge dislocations in ev,

$\gamma$ - the stacking fault energy in ergs/cm$^2$
From the experimental results given in Figure 15, the equation for the temperature dependence of the activation volume is determined.

\[ V = 2 \times 10^{-17} e^{\frac{W_d}{T}} \]  \hspace{1cm} (24)

where \( W_d \) is a constant for each crystal, tabulated in Table 5.

Since \( d \) is the only parameter in the volume that varies significantly with temperature, we can rewrite equation 24 as

\[ d = 3.3 \times 10^{-8} e^{\frac{W_d}{T}} \]  \hspace{1cm} (25)

where \( l_0 = 2 \times 10^{-4} \) cm

\( b = 3 \times 10^{-8} \) cm

From isotropic elasticity

\[ d = \frac{Gb^2}{\pi(1-\nu)} \left[ 1 - \frac{2\nu}{2-\nu} \cos \frac{\alpha}{2} \right] \]  \hspace{1cm} (26)

where \( \nu \) = Poisson's ratio (0.33 for cadmium)

\[ \alpha \] = angle between total Burger's vector and line of axis of partial dislocation, 90° for an edge dislocation.

So that by combining equations 23, 25, and 26;

\[ \left( E_c e \right)^{\frac{1}{2}} = \frac{6400 \pi}{Gb^2} \left[ \frac{1-\nu}{2+\nu} \right] 3.3 \times 10^{-8} e^{\frac{W_d}{T}} \]  \hspace{1cm} (27)

and for screw dislocations where \( \alpha \) equals 0°,

\[ E_c^s = \frac{256 \pi}{Gb^2} \left[ \frac{1-\nu}{2-3\nu} \right] 3.3 \times 10^{-8} e^{\frac{W_d}{T}} \]

The empirical relation for the constriction energy for screw dislocations is

\[ E_c^s = 32/\gamma \]

The approximate energy relationships for the processes are given in Table 4 for \( T = 275^\circ K \). Since it is assumed here that edges control
the flow stress at low temperatures and the splitting of partials in edges is predominantly controlling the temperature dependence of \( \nu \); especially in the light of a prior assumption that screw dislocations at higher temperatures cannot show a marked temperature dependence of the partial separation distance. The significance of Table 4 is that it indicates that at temperatures below 275\( ^* \)K the energy to constrict an edge dislocation decreases with temperature and hence it is the rate controlling mechanism of the flow stress. That is that the high temperature and low temperature mechanisms are considered independent and the one with the lowest activation energy controls the flow stress.

From the slope of the line through the data points the intercept of the normalized flow stress at \( T = 0^* \)K was obtained. This gave a value of the activation energy for the high temperature mechanism to be 1.85 ev from

\[
U_0 = \frac{E}{\nu}
\]

where \( \nu = 4 \times 10^{-19} \text{ cm}^3 \), and the absolute zero flow stress \( T_0 \) is 107.4 psi when \( \frac{E}{\nu} = 60 \text{ psi} \) (see Figure 8).

This value agrees very well with the 1.8 ev for the type of process assumed to occur. Using the value of \( U_0 \) and equation 6, a value of 706\( ^* \)K was calculated for \( T_0 \). These values were used in this calculation:

\[
N = 4 \times 10^{12} \text{ cm}^{-3}
\]
\[
A = 4 \times 10^{-8} \text{ cm}^2
\]
\[
b = 3 \times 10^{-8} \text{ cm}
\]
\[
\varepsilon = 1.67 \times 10^{-5} \text{ sec}^{-1}
\]
\[
\lambda = 5 \times 10^{10} \text{ sec}^{-1}
\]

If \( \lambda \) is changed to \( 5 \times 10^{12} \text{ sec}^{-1} \), the value of \( T_0 \) becomes 613\( ^* \)K.

These are both higher than any point taken in the experiments.
The theory of the flow stress discussed above has been basically credited to Seeger as it uses a model having a uniform forest density. Basinski used a model for thermally activated glide similar to that of Wyatt for creep. This idea suggests that the forest is non-uniform and the gliding dislocations are held by obstacles with 'soft spots'. By thermal activation the gliding dislocations cut through these soft spots. A relation for the activation energy is given as

\[ U_0 = kT \ln \frac{N \lambda b kT \alpha_0}{\dot{\varepsilon}} \]

where all terms are the same as previously defined except N, which is the number of soft spots per unit volume per unit energy.

The logarithmic term can be calculated from the following

\[ \ln \frac{N \lambda b kT \alpha_0}{\dot{\varepsilon}} = \frac{1}{1 + \frac{\sigma/M}{\alpha_0} \frac{1}{T} \left( \frac{3 \ln M}{\alpha_0 \ln \dot{\varepsilon}} \right)} \]

\[ \frac{1}{1 - T \frac{\sigma}{\alpha_0} \frac{1}{T} \ln M} \]

Basinski obtained a value of 13 for this logarithmic term, whereas Seeger's reported value for a similar term and metal was 32.

The value of the logarithmic term was calculated from the results of this work. For temperature below 275°K, the value was less than one (0.94 - 0.87). However, in this region a reasonable value of the frequency factor, \( \lambda_0 \), was found by using the values of the constants that had been used to calculate \( T_0 \) (see page 26). \( \lambda_0 \) varies from 2 to \( 8 \times 10^{10} \) sec\(^{-1} \) for the low temperature range.

For the high temperature range, where the activation volume had reached equilibrium, agreement was found with Seeger's value of the
logarithmic term. Values from 37 to 23 were calculated, decreasing with increasing temperature.

The value of the constants are

\[
\begin{align*}
\left[ \frac{\partial \left( \frac{\sigma}{\mu} \right)}{\partial T} \right] & = 0 \quad T < 275^\circ K \\
& = -3 \times 10^{-3} \quad T > 275^\circ K \\
\frac{\partial \ln \mu}{\partial T} & = -5.4 \times 10^{-4} \quad T > 0^\circ K
\end{align*}
\]

The value of \( \frac{\sigma}{\mu} \) was taken from Figure 8. By assuming that \( \ln \sigma \) vs \( \ln \dot{\varepsilon} \) was linear, the value of \( \left( \frac{\partial \mu}{\partial \ln \dot{\varepsilon}} \right)_T \) was found from the two flow stresses during a strain rate change test.

Basinski's model leads to rather small activation energies at 0^\circ K which are not deemed reasonable; although it is not correct for the author to consider 77^\circ K as similar in behavior to 0^\circ K as is necessary for the comparison.

**Activation Volume**

The activation volume increases with temperature due to the decrease in the stacking fault energy. This causes the activation distance, \( d \), to increase with increasing temperature as seen from values of \( v \) and equation 26. The other parameters, \( b \) and \( l_0 \), are constant with temperature in the low temperature range. No appreciable increase in the volume could be obtained from the thermal expansion of the lattice.

At the temperature where the change in flow stress mechanism occurs, a sharp drop in the activation volume occurs. This is due to the dislocation lines becoming more joggy which means that \( l_0 \) decreased. The activation distance, \( d \), gets smaller by a factor of 2 since we are now
considering screw dislocations which have a smaller separation distance. An equilibrium value of the activation volume is reached when the number of jogs being produced by deformation and point defects is equal to the number lost by jog annihilation. Motion of jogged screw dislocations is known to require point defect generation. The high temperature mechanism is therefore believed to be the thermally activated glide of screw dislocations with interstitial defect generation. This process is believed to require more energy than the screws cutting the forest dislocations.

Agreement with certain of these experimental results has been shown in other investigations. Conrad showed that in magnesium the activation volume increases linearly with temperature in the low temperature region. He performed creep experiments on single crystals.

An increase in the number of jogs on dislocation lines at high temperatures was shown in zinc by Rinnovatore and Brown. They showed that at -72°C the Baushlinger effect was present and that the flow stress increased. This effect was attributed to the increased number of jogs due to vacancies and the forming of dislocation loops from jogged dislocations.

A. Deruyttere, et al. found that zinc showed an increase in the work hardening at -72°C from that at -186°C. They gave no explanation for this anomaly. They felt it did not occur in cadmium, but we believe that they failed to study the correct temperature range.

From the low temperature creep experiments performed by Glen, a value of $\alpha$, the slope of the logarithmic creep curve, is reported. By using the relation that

$$\alpha = \frac{kT}{\nu \theta}$$
it is possible to calculate a corresponding value of $\kappa$ from the present results. For $v = 3.2 \times 10^{-19} \text{ cm}^3$ and $\theta = 361 \text{ psi}$, the result was $130.5 \times 10^{-5}$ compared with Glen's value of $37 \times 10^{-5}$ for the temperature $77^\circ\text{K}$. This agreement is believed to be fairly good since $v$ is a sensitive function of a particular specimen through $l_o$.

This discussion has been based on a temperature dependence of the stacking fault energy to account for the temperature dependence of $v$. Another possible method of interpretation is to assume that $v$ is a function of $(\tau - \tau_0)^2$. The temperature dependence of $\tau_0$ or the normalized flow stress should affect $v$. The present results show $v$ constant where the flow stress is markedly temperature dependent and $v$ to be varying where the normalized flow stress ratio is relatively constant. In the light of this and the work hardening results, this approach $^{29}$ was not considered. It is important to note, however, that the flow stress in the low temperature region is believed to be controlled by the first motion of dislocations through the forest. The hardening is a consequence of continued motion of these dislocations and jog formation.
SUMMARY

1. The activation volume of cadmium single crystals was found to increase logarithmically with temperature from 77 to 275°K and then suddenly decrease to a constant value up to 508°K.

2. The work hardening coefficient for the same specimens was found to increase with temperature from 77 to 275°K and then rapidly decrease to zero above 275°K, for a strain rate of $2 \times 10^{-3}$ min $^{-1}$.

3. The normalized flow stress was found to be constant over the temperature range 77 to 275°K, above which it seemed to decrease almost linearly with increasing temperature to 508°K.

4. The results can be explained by postulating a decrease in stacking fault energy with increasing temperature below 275°K. Jog formation in gliding dislocations is considered to give rise to the increased work hardening coefficient with temperature. This is most likely due to a dependence on the excess stress to move a jog in an extended dislocation, upon the separation distance of partial dislocations.

5. The high temperature deformation, $T>275°K$, is believed to be controlled by the generation of interstitials by gliding jogged screw dislocations.

6. The investigation is not yet complete enough to conclude that the previous explanation is correct. Further studies on the strain rate dependence of $\nu$, the normalized flow stress, and the work hardening coefficient are needed.
BIBLIOGRAPHY


ACKNOWLEDGMENT

The author gratefully acknowledges the guidance, assistance, and encouragement given by Dr. John M. Roberts, who suggested the problem.

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FIGURES AND TABLES
## TABLE 1

**SPECTROGRAPHIC ANALYSIS**

by American Smelting and Refining Company, South Plainfield, N. J.

<table>
<thead>
<tr>
<th>Element</th>
<th>Before Growth</th>
<th>After Growth*</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mg</td>
<td>$&lt; 0.0001$</td>
<td>ND</td>
</tr>
<tr>
<td>Pb</td>
<td>$0.006\pm$</td>
<td>$0.004\pm$</td>
</tr>
<tr>
<td>Fe</td>
<td>$0.0001$</td>
<td>$0.0001\pm$</td>
</tr>
<tr>
<td>Bi</td>
<td>$&lt; 0.0001$</td>
<td>ND</td>
</tr>
<tr>
<td>Cu</td>
<td>$0.0005\pm$</td>
<td>$0.0001\pm$</td>
</tr>
<tr>
<td>Ag</td>
<td>$&lt; 0.001$</td>
<td>$0.0001$</td>
</tr>
<tr>
<td>Cd</td>
<td>$99.99%$</td>
<td>$99.99%$</td>
</tr>
</tbody>
</table>

ND = none detected
Cd = per cent by difference

* Two specimens analyzed from material at the tip of the crystals. The results are the same for both.*
<table>
<thead>
<tr>
<th>Specimen</th>
<th>$\theta_0$</th>
<th>$\phi_0$</th>
<th>$\gamma_0$</th>
</tr>
</thead>
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<tr>
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<td>60.5°</td>
<td>39.5°</td>
<td>45.0°</td>
</tr>
<tr>
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<td>72.0</td>
<td>19.0</td>
<td>55.0</td>
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<tr>
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<td>68.0</td>
<td>29.0</td>
<td>48.0</td>
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<tr>
<td>CS 61</td>
<td>19.0</td>
<td>71.0</td>
<td>78.0</td>
</tr>
<tr>
<td>CS 62</td>
<td>19.0</td>
<td>71.0</td>
<td>78.0</td>
</tr>
<tr>
<td>CS 71</td>
<td>59.0</td>
<td>31.5</td>
<td>59.0</td>
</tr>
<tr>
<td>CS 72</td>
<td>59.0</td>
<td>31.5</td>
<td>59.0</td>
</tr>
<tr>
<td>CS 81</td>
<td>71.0</td>
<td>20.0</td>
<td>54.0</td>
</tr>
</tbody>
</table>

$\theta_0$ - Angle between tensile axis and the normal to the basal plane.

$\phi_0$ - Angle between the tensile axis and the expected slip direction.

$\gamma_0$ - Angle between the tensile axis and the second nearest slip direction.
<table>
<thead>
<tr>
<th>Crystal</th>
<th>Temperature, °K</th>
<th>$\frac{\theta T}{\alpha T} \times 10^5$</th>
</tr>
</thead>
<tbody>
<tr>
<td>CS 3</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>263</td>
<td>22.75</td>
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<tr>
<td></td>
<td>223</td>
<td>9.72</td>
</tr>
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<td></td>
<td>172</td>
<td>5.31</td>
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<tr>
<td></td>
<td>121</td>
<td>4.27</td>
</tr>
<tr>
<td>CS 61</td>
<td></td>
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</tr>
<tr>
<td></td>
<td>272</td>
<td>9.79</td>
</tr>
<tr>
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<td>6.83</td>
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<tr>
<td></td>
<td>173</td>
<td>2.34</td>
</tr>
<tr>
<td></td>
<td>121</td>
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<td>11.24</td>
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<tr>
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<td>4.83</td>
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<td></td>
<td>173</td>
<td>10.96</td>
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<tr>
<td></td>
<td>148</td>
<td>12.34</td>
</tr>
<tr>
<td></td>
<td>120</td>
<td>10.62</td>
</tr>
</tbody>
</table>

* Took $\Theta_2$ values for highest energy glide region.
<table>
<thead>
<tr>
<th>Low Temperature Mechanism (T = 275°K)</th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Energy to constrict an edge dislocation</td>
<td>≈ 1.2</td>
<td></td>
</tr>
<tr>
<td>Energy to constrict a forest screw dislocation</td>
<td>≈ 0.3</td>
<td></td>
</tr>
<tr>
<td>Energy to jog an edge dislocation</td>
<td>≈ 0.6</td>
<td></td>
</tr>
<tr>
<td>Energy to jog a screw dislocation</td>
<td>≈ 0.45</td>
<td></td>
</tr>
<tr>
<td><strong>Total energy</strong></td>
<td>≈ 2.55 ev</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>High Temperature Mechanism</th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Energy for formation of an interstitial defect</td>
<td>≈ 1.8 ev</td>
<td></td>
</tr>
<tr>
<td>Crystal</td>
<td>$W_d$</td>
<td>$W_{e/G}$</td>
</tr>
<tr>
<td>---------</td>
<td>---------</td>
<td>-----------</td>
</tr>
<tr>
<td>CS 3</td>
<td>$1.12 \times 10^{-2}$</td>
<td>$1.24 \times 10^{-2}$</td>
</tr>
<tr>
<td>CS 61</td>
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<tr>
<td>CS 81</td>
<td>1.0</td>
<td>.945</td>
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</table>
TEMPERATURE CONTROL APPARATUS

A Dewar
B Controller Thermocouple
C Universal Joints
D Specimen
E Grips
F Copper Tubing
G Thermocouples

Figure 2
Figure 6

Activation Volume vs Temperature

Decreasing Temperature
Increasing Temperature

Temp. °K

CS 62

Vact x 10 cm^3
Flow Stress vs Temperature

Strain Rate
- CS 62: $2 \times 10^{-3}$/min
- CS 61: "
- CS 42: "
- CS 3: $1 \times 10^{-3}$/min

Temperature °K

Figure 8
Figure 9

Work Hardening Coefficient vs Temperature °K
Strain Rate $2 \times 10^{-2}$ /min.
NORMALIZED FLOW STRESS vs STRAIN

CS 71
Temperature 297ºK
Strain Rate $2 \times 10^{-3}$ /min

- before anneal
- after 18 hour anneal

Figure 11
Figure 12

STRESS vs STRAIN

CS 1 Temperature 77 °K

\[ a = 2 \times 10^{-4} \text{ min}^{-1} \] (Strain Rate)

\[ b = 2 \times 10^{-2} \text{ min}^{-1} \] ("")

\( \tau \text{ psi} \)

68.7

58.9

49.1

0

5.68

4.26

2.84 \times 10^3

1.42

\( Y \times 10^3 \)
SLOPES of EASY GLIDE FLOW
C S 81

\[ \tau \text{ psi} \]

\[ 77^\circ K \quad 148^\circ K \quad 198^\circ K \quad 263^\circ K \quad 280^\circ K \quad 305^\circ K \]

\[ \gamma \]

\[ \gamma \]

\[ 56.03 \quad 77.34 \quad 66.60 \quad 55.04 \quad 51.43 \quad 50.78 \text{ psi} \]

\[ 1 \times 10^{-3} \]

NOTE: The absolute value of the yield point at each temperature has been shifted to a common value. Strain Rate = \( 2 \times 10^{-2} / \text{min} \)

Figure 13
\[ \left( \frac{\theta}{G} - \frac{\tau_s N''}{GN} \right) \text{ vs } \frac{1}{T} \]

CS 61

Figure 14
Figure 15

ACTIVATION VOLUME vs TEMPERATURE

CS 3

$V \times 10^{18} \text{ cm}^3$

$T \degree K$

Figure 15
GLIDE LAMELLAE FOR CADMIUM SINGLE CRYSTAL

50x

FIGURE 16
DATA FROM SEEGER on CADMIUM SINGLE CRYSTALS

(a) Two Strain Rates

(b) Room Temperature

(c) Figure 17