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DISLOCATION SUBSTRUCTURE FORMED DURING CREEP
IN MOLYBDENUM SINGLE CRYSTALS

Dissertation
Presented in Partial Fulfillment of the Requirements for
the Degree Doctor of Philosophy in the Graduate
School of The Ohio State University

by
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* * * * * * * *

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1968

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B. A. Wilcox and A. H. Clauer, "Creep of Thoriated Nickel above and below 0.5 Tm," Trans. AIME, 236, 570 (1966).


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INTRODUCTION

Creep in metallic structures supporting loads at elevated temperatures has been observed for many years. However, with several exceptions, this area of material behavior has received only sporadic attention with regard to determining its fundamental mechanisms. In particular, the body-centered cubic metals have only recently been studied in some detail, although face-centered cubic metals have received sustained attention for the last 20 years.

Much of the present description of creep is comprised of phenomenological mechanical parameters describing creep behavior. Until quite recently, creep theories have been formulated for the most part on the basis of these mechanical parameters, e.g., the stress and temperature dependence of the creep rate. These theories have been quite useful in suggesting possible mechanisms controlling the creep rate, e.g., self diffusion. They have also shown that the dislocation substructure formed during creep must be known in order to differentiate between creep theories which arrive at similar predictions of creep behavior, but are based on different substructures.

The objective of this study was to direct attention to body-centered cubic metals, and to investigate both the mechanical creep properties and the dislocation substructure developed during creep. In this way a comparison of the observed creep behavior and creep
substructure could be made with existing creep theories. In addition, perhaps some other previously unrecognized factors influencing creep behavior could possibly be identified.
BACKGROUND

Creep in BCC Metals

Most of the effort on high temperature creep in bcc metals, i.e., creep at temperatures above one-half the melting point, $0.5 \, T_m$, has been expended in compiling engineering and design data in commercial metals and alloys. These data are not useful in achieving a better understanding of the underlying creep mechanisms, both because of the non-ideal or poorly defined composition and initial microstructure of the material and because of the absence of strain-time data obtained over a sufficient range of stress and strain. Such data are required to characterize the basic creep behavior of a metal. The high temperature creep behavior of iron, which has been a technologically important bcc metal for ages, has only recently begun to be studied in some detail\(^{(1-11)}\), with the exceptions of the work of Tapsell and Clenshaw\(^{(12)}\) in 1927 and Jenkins and Mellor\(^{(13)}\) in 1935. The other important class of metals having the body-centered cubic structure, the refractory metals, has been difficult to study until recently because their high melting points and/or susceptibility to contamination from the furnace atmosphere required special high temperature equipment and experimental technique to perform creep tests above $0.5 \, T_m$. Still more refinement in experimental technique is required in order to control and measure the experimental conditions of stress, strain, and temperature as precisely as is possible at lower temperatures.
The relatively few studies directed towards an understanding of creep in the refractory metals have been performed on Mo\(^{(14-25)}\), W\(^{(17-28)}\), Ta\(^{(29)}\), and Nb\(^{(30-32)}\). The series of references\(^{(17-25)}\) actually represent a single program in progress. Other studies which make a peripheral contribution include those on Mo\(^{(33-35)}\), W\(^{(36,37)}\), Cr\(^{(38)}\), Nb\(^{(39-41)}\), and Ta\(^{(42-44)}\).

**Creep properties**

The creep curves of the bcc metals all display one or more of the transient, steady state and tertiary stages of creep depending on the stress, temperature, and initial microstructure. There have been several instances where the creep curves did not follow the classic sequence of decelerating, constant and accelerating creep rate with increasing time. These "breaks" in the creep curves consist either of a sudden decrease in the creep rate\(^{(14)}\), an increase followed by a new higher constant creep rate\(^{(20)}\), or an increase followed by a decrease to some constant creep rate\(^{(18,22,31)}\). The change in creep rate may occur gradually\(^{(18,22)}\) or quite suddenly\(^{(31)}\). These occurrences can usually be attributed to some structural instability occurring during the test such as grain growth or recrystallization rather than a change in the creep mechanism.

A decrease in creep rate\(^{(14)}\) and increase in rupture life\(^{(35)}\) in powder metallurgy molybdenum tested in inert atmospheres at 0.88 and 0.95 \(T_m\) has been attributed to the development during creep of abnormally large grains which have consumed major portions of the gage section. Flagella\(^{(35)}\) has shown that this abnormal grain growth is
associated with a significant pickup of tungsten (up to 7.5 wt.%), from
the heating elements during the creep test. Otherwise the growth of
grains to sizes of 0.5 to 0.9 mm during testing has no transient effect
on the creep curve\(^{17,35}\).

Most of these structural instabilities observed during creep
occur because the initial structure was not stabilized by a preanneal
at a higher temperature than the creep test temperature. For example,
when the as-received stock was given a preanneal at the temperature of
the creep test, breaks in the creep curves consisting of accelerating
and decelerating creep rate occur in both Mo\(^{20,22}\) and W\(^{18}\) at about
0.65 \(T_m\). When the same material was preannealed at a temperature
higher than the creep temperature, the break in the creep curves dis-
appeared\(^{18}\). Unusual jumps in the creep curves of niobium-oxygen
alloys\(^{31}\) are probably also due to some unidentified structural
instability. The creep data on niobium and to a lesser extent tantalum
are suspect in most instances because of continuous absorption of gas
during the creep test.

For temperatures above 0.5 \(T_m\) the activation energy for creep,
\(Q_c\), is in nominal agreement with the activation energy for self
diffusion, \(Q_{SD}\), for Fe\(^{1,2}\), Mo\(^{14,24}\), W\(^{20,22,26}\), and Ta\(^{29}\),
although there are certain discrepancies. The data for the refractory
metals are summarized in Table 1. The data for Nb\(^{30,31}\) are not
included. The apparent instabilities in the creep curves and incom-
pleteness of the data make the interpretation of the results difficult.
Iron has an activation energy for creep above 0.5 \(T_m\) which is higher
than the activation energy for self diffusion\(^{1,2,45}\). However
<table>
<thead>
<tr>
<th>Metal</th>
<th>Temperature Range, T/Tₘ</th>
<th>Experiment</th>
<th>Activation Energy Q, kcal/mol</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mo</td>
<td>0.65 - 0.96</td>
<td>Creep</td>
<td>114</td>
<td>(14)</td>
</tr>
<tr>
<td></td>
<td>0.72 - 0.86</td>
<td>S.D. (a)</td>
<td>115</td>
<td>(45)</td>
</tr>
<tr>
<td></td>
<td>0.72 - 0.86</td>
<td>S.D.</td>
<td>114</td>
<td>(46)</td>
</tr>
<tr>
<td></td>
<td>0.68 - 0.75</td>
<td>S.D.</td>
<td>111</td>
<td>(47)</td>
</tr>
<tr>
<td></td>
<td>0.65 - 0.86</td>
<td>S.D.</td>
<td>100.8</td>
<td>(48)</td>
</tr>
<tr>
<td></td>
<td>0.74 - 0.91</td>
<td>S.D.</td>
<td>92.2</td>
<td>(49)</td>
</tr>
<tr>
<td></td>
<td>0.74 - 0.91</td>
<td>S.D.</td>
<td>96.9</td>
<td>(49)</td>
</tr>
<tr>
<td></td>
<td>0.84 - 0.98</td>
<td>S.D.</td>
<td>110</td>
<td>(50)</td>
</tr>
<tr>
<td>W</td>
<td>0.68 - 0.83</td>
<td>Creep</td>
<td>160</td>
<td>(26)</td>
</tr>
<tr>
<td></td>
<td>0.56 - 0.68</td>
<td>Creep</td>
<td>141</td>
<td>(27)</td>
</tr>
<tr>
<td></td>
<td>0.61 - 0.83</td>
<td>Creep</td>
<td>160</td>
<td>(20)</td>
</tr>
<tr>
<td></td>
<td>0.67 - 0.78</td>
<td>Creep</td>
<td>170</td>
<td>(22)</td>
</tr>
<tr>
<td></td>
<td>0.42 - 0.47</td>
<td>S.D.</td>
<td>135.8</td>
<td>(51)</td>
</tr>
<tr>
<td></td>
<td>0.61 - 0.81</td>
<td>S.D.</td>
<td>120.5</td>
<td>(52)</td>
</tr>
<tr>
<td></td>
<td>0.80 - 0.95</td>
<td>S.D.</td>
<td>153.1</td>
<td>(53)</td>
</tr>
<tr>
<td>Ta</td>
<td>0.60 - 0.89</td>
<td>Creep</td>
<td>114</td>
<td>(29)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>S.D.</td>
<td>110</td>
<td>(54)</td>
</tr>
</tbody>
</table>

(a) Self diffusion.
Ishida et al.\(^{(2)}\) clearly showed that the actual activation energy for creep can still be equal to the activation energy for self diffusion and the high apparent activation energy for creep was caused by the presence of the magnetic transition in the same temperature range.

In molybdenum, the value of Green et al.\(^{(14)}\) of \(Q_c = 114\) kcal/mol is higher than the average activation energy for self diffusion \(\bar{Q}_{SD} = 106\) kcal/mol. However if a correction for the temperature variation of the shear modulus is included\(^{(56)}\) the apparent activation energy for creep would be decreased slightly and give better agreement with \(\bar{Q}_{SD}\). Conway and Flagella\(^{(23)}\) achieve a good correlation of the steady state creep rate of molybdenum tested at 0.65 and 0.86 \(T_m\) using Sherby's\(^{(57)}\) method with \(Q_c = Q_{SD} = 96.9\) kcal/mol. Although they do not indicate their source of \(Q_{SD}\) it is presumably that of Askill and Tomlin\(^{(49)}\) according to Table 1.

In tungsten, knowledge of the creep behavior is more extensive. Above 0.6 \(T_m\) all the reported values of \(Q_c\) except that of Klopp et al.\(^{(27)}\) are greater than \(Q_{SD}\). None of these data have been corrected for the temperature dependence of the elastic modulus\(^{(56)}\) which would improve the agreement. Klopp, et al. show that the steady state creep rate has a continuously increasing temperature dependence from 0.5 \(T_m\) to at least 0.6 \(T_m\). An examination of their Figure 8 suggests that the temperature dependence may still be increasing slightly from 0.6 to 0.68 \(T_m\), their highest temperature. Hence, their result is not necessarily in disagreement with the others. In fact, the data of Klopp et al. are in the best agreement with the average of the reported activation energies of self diffusion.
Conway and Flagella\(^{(24)}\) again employed the Sherby method\(^{(57)}\) to correlate all their previous creep data in tungsten using

\[ Q_c = Q_{SD} = 120.5 \text{ kcal/mol}. \]

Table 1 shows that this is probably the \( Q_{SD} \) reported by Danneberg\(^{(52)}\). The correlation was fair, even though the data itself gave an activation energy for creep of \( Q_c = 170 \text{ kcal/mol} \).\(^{(22)}\) Evidently, this method of correlating creep data is not very sensitive to the choice of \( Q_{SD} \). A close comparison of creep and self diffusion activation energies must wait for more measurements of both.

From 0.4 to 0.6 \( T_m \), \( Q_c \) is much less than \( Q_{SD} \) for tungsten. Gilbert\(^{(28)}\) reports \( Q_c = 105 \text{ kcal/mol} \) for the steady state creep rate in tungsten from 0.4 to 0.6 \( T_m \). Klopp et al.\(^{(27)}\) did not observe a steady state creep rate below 0.5 \( T_m \), but at 0.5 \( T_m \) \( Q_c \) for steady state creep was on the order of 60-70 kcal/mol and increased continuously with increasing temperature up to 0.6 \( T_m \). However they obtained a constant value of \( Q_c = 106.4 \text{ kcal/mol} \) for transient creep from 0.41 to 0.60 \( T_m \). Hence, high temperature creep in tungsten does not become predominantly diffusion controlled except at temperatures above about 0.6 \( T_m \) whereas in iron creep is diffusion controlled down to 0.46 \( T_m \). The decrease in \( Q_c \) below \( Q_{SD} \) at temperatures below 0.6 \( T_m \) is too large to be accounted for by a stress dependence of the activation energy based on a self diffusion type process such as the nonconservative motion of jogged screws. However Gilbert\(^{(28)}\) suggests that nonconservative motion of jogs is rate controlling and the activation energy for creep is related to the activation energy for pipe diffusion along dislocations and for grain boundary diffusion. In support of this he
observed that an increase in grain size raised $Q_c$ from 105 kcal/mol to 130 kcal/mol. Klopp et al. suggest that the rate-controlling mechanism is recovery by cross-slip and that steady state creep appears only when the temperature is high enough, i.e., above 0.5 $T_m$, so that the edge segments can annihilate by climb.

Carvalhinhos and Argent\textsuperscript{(16)} report very unusual creep behavior in polycrystalline molybdenum tested in compression from 0.42 to 0.57 $T_m$. $Q_c$ was stress dependent varying from 61 kcal/mol at 3500 psi to 88 kcal/mol at 10,000 psi. However the stress dependence of the creep may have also been temperature dependent and this was not accounted for. These results indicate that this temperature range should be investigated more closely in molybdenum.

Although $Q_c$ appears to change as the temperature is decreased below 0.6 $T_m$, there is no evidence for a change in the stress dependence of the steady state creep rate. In tungsten above 0.6 $T_m$ the steady state creep rate, $\dot{\epsilon}_s$, is related to the applied stress, $\sigma$, by $\dot{\epsilon}_s \propto \sigma^n$ where $n = 4.2$ to 6.3\textsuperscript{(20,22,24,25-27)}. The lower value of $n = 4.2$ was obtained using Sherby's\textsuperscript{(57)} method of correlating the creep data\textsuperscript{(24)} with $Q_c = 120.5$ kcal/mol; the other methods all give $n = 5$ to 6. Below 0.6 $T_m$ there was no significant change in the stress dependence of the steady state creep rate at the same stress levels\textsuperscript{(27)}. The deviation from the power law dependence observed at lower temperatures is usually associated with higher stresses\textsuperscript{(24,27)} which in itself does not necessarily indicate a change in the creep mechanism. An identical deviation occurs in iron\textsuperscript{(1,55)} but Ishida et al.\textsuperscript{(2)} have shown that the same rate-controlling mechanism may be operating over this entire range of
temperature and stress. In addition, they demonstrated that the power
dependence can remain constant while $Q_c$ goes through wide fluctuations.
Other stress functions can be used to linearize such data over the
entire range of stress\(^{(58)}\) although there may be no theoretical justifi-
cation for doing so.

Molybdenum shows a similar stress dependence above 0.65 $T_m$
where $n = 4.5$ to $5.1\,^{(17,19,24)}$. Here the latter value was obtained by
the correlation according to Sherby\(^{(57)}\) using $Q_c = Q_{SD} =$
96.9 kcal/mol\(^{(24)}\) on data which had earlier given $n = 4.8\,^{(19)}$. Perhaps
if a higher value of $Q_{SD}$ had been used by Conway and Flagella\(^{(24)}\) for
the correlation in tungsten, a higher value of $n$ would have resulted.
Below 0.6 $T_m$, from 0.47 to 0.52 $T_m$, $n = 5.6$ in compressive creep\(^{(16)}\).
The general trend is that $n$ is higher in tungsten than in molybdenum.
The stress dependence in iron is still higher, i.e., $n = 6.9\,^{(2)}$.

The stress dependence of primary creep described by the strain
time relation of the form $\varepsilon - \varepsilon_o = bt^m$ has been determined for tungsten
and molybdenum where $\varepsilon = \text{total transient creep strain}$, $\varepsilon_o = \text{load on}$
plastic strain, $b = \text{constant}$, $t = \text{time from the start of the test}$, and
$m$ usually lies in the range of 0 to 1. Table 2 summarizes these data.
In general the stress dependence, i.e., exponent $n$, of the primary creep
strain is less than the power law stress dependence of the steady state
creep rate.

The refractory metals Cr, Mo, and W have been shown to have
identical stress rupture properties at the same stress level at the
same homologous temperature from 0.37 to 0.8 $T_m\,^{(59)}$. It is reasonable
then to assume that in this class of bcc metals the same creep
TABLE 2
STRESS DEPENDENCE OF THE TRANSIENT CREEP STRAIN

<table>
<thead>
<tr>
<th>Metal</th>
<th>Temperature Range, T/Tm</th>
<th>n</th>
<th>m</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mo</td>
<td>0.65 - 0.86</td>
<td>4.1</td>
<td>0.6 - 0.79</td>
<td>(19)</td>
</tr>
<tr>
<td></td>
<td>0.47 - 0.52</td>
<td>2.6</td>
<td>0.33</td>
<td>(16)</td>
</tr>
<tr>
<td>W</td>
<td>0.68 - 0.83</td>
<td>3.34</td>
<td>0.6</td>
<td>(18)</td>
</tr>
<tr>
<td></td>
<td>0.51 - 0.83</td>
<td>4.32</td>
<td>0.64 - 0.72</td>
<td>(19)</td>
</tr>
<tr>
<td></td>
<td>0.41 - 0.60</td>
<td>3.9</td>
<td>0.33</td>
<td>(27)</td>
</tr>
</tbody>
</table>

Mechanisms are operating at the same homologous temperature. On the basis of the activation energies for creep in tungsten, iron and molybdenum, different creep mechanisms may be operating in the vicinity of 0.4 to 0.6 Tm in refractory metals as opposed to iron. However the reasons for this difference are not known.

The stress dependence for the steady state creep rate above 0.5 Tm is similar to those observed in numerous fcc metals where n ranges between 4 and 6. This, coupled with the common observation that Qc ≈ Qsd, suggests that above 0.5 Tm the same creep mechanisms may be operative in both cubic crystal structures. This implies that the dislocation and subgrain microstructures developed during creep follow the same general pattern during creep and have a similar dependence on stress and temperature for both crystal lattices.
Mode of deformation

Several types of deformation modes are suggested by markings on the surface of a creep specimen after creep, including wavy, coarse slip bands, fine slip lines, thickening and folding of the grain boundaries, and subgrain formation. The coarse slip bands have a large displacement in each band and are wavy and forked in molybdenum single crystals and polycrystalline Ta and Nb. The spacing between these bands decreases with increasing stress and in polycrystalline tantalum, Green has shown that the coarse slip band spacing \( d \) varies with the applied stress, \( \sigma \), as \( d \propto \sigma^{-1} \). At low stresses no coarse slip bands were observed, in accordance with the findings of others in polycrystalline Fe and Nb. This is probably the result of the slip band spacing being larger than the grain diameter. Green often saw coarse slip bands only in the larger grains in tantalum and in very large grains in molybdenum tested at 0.65 \( T_m \). These coarse slip bands do not necessarily begin to form right at the beginning of the creep test since large slip bands form in grains that grow during creep in molybdenum tested at 0.86 \( T_m \).

In niobium, fine slip lines were present between the coarse slip bands whereas in tantalum no mention is made of fine slip lines. Harris did not indicate whether the fine slip lines were of the same slip system as the coarse slip bands or of an alternate slip system. Coarse slip bands from two different slip systems did occur in tantalum but their spacings differed.
In molybdenum, Conway and Flagella\textsuperscript{(21)} determined that the coarse slip bands formed in the large grains were parallel to planes of the type ((112)), i.e., ((112)), ((113)), ((114)), and ((116)). In Fe-3\%Si, Lytton et al.\textsuperscript{(9)} saw within large grains traces of etch pit bands parallel to the ((112)) or ((110)) slip plane traces having the largest resolved shear stress. Although most of the slip planes listed by them as having the highest resolved shear stress are wrong, the ones they list give a surface trace parallel to those with the highest resolved shear stress with two exceptions. Since the exceptions do not seriously change the trace angle, their conclusions are correct.

The presence of subgrains after creep has been verified metallographically in Fe-3Si\textsuperscript{(8,9)}, Fe(3,7,13), Mo(15,18), W(23,25), Cr(3), Nb(41), and Ta(29); by X-ray methods in Fe\textsuperscript{(4)}, Mo\textsuperscript{(18)}, and W(23,25); and by transmission electron microscopy in Fe\textsuperscript{(4,7)} and Fe-3Si\textsuperscript{(8)}. The most extensive study of subgrain formation has been made in Fe-3Si\textsuperscript{(8,9)} by etch pitting and to a lesser extent transmission electron microscopy. These results can be summarized as follows.

A uniform dislocation distribution was formed immediately after loading. Then, depending on the grain size, two different types of substructure formed. At larger grain sizes a banded structure formed very early in primary creep. The bands appeared to be parallel to slip plane traces but this had not been determined rigorously\textsuperscript{(8,9)}. Once formed, the spacing between the bands depended only on the stress and not on the grain size or creep strain. As the grain size decreased, an equiaxed structure evolved slowly out of the uniform dislocation
arrangement during primary creep. Steady state creep did not appear until this equiaxed subgrain structure was fully developed. The size of the equiaxed subgrains was dependent only on the stress and, once the subgrains were formed, was also independent of creep strain. Lytton et al. (9) observed that when the slip plane and slip direction were normal to a grain boundary there was a tendency for long, parallel tilt boundaries to develop normal to the slip planes and to pile up against the grain boundary. Other types of subgrain boundary-slip plane-grain boundary interactions were noted which depended on the relative orientations of each.

The observations made by others all indicate that at relatively high stresses, subgrain formation is quite prominent in Fe (3, 7, 13), Mo (18), W (23, 25), Nb (30, 41), and Ta (29). As the stress is decreased, the subgrains become coarser and are not as easy to see metallographically in polycrystalline material (41, 18, 23). The etch-pitting of coarse-grained molybdenum (18) and tungsten (23) after creep shows that in large grained material the subgrain boundaries tend to be straight and regular and to prefer certain orientations. The regularity and straightness of the sub-boundaries has also been reported in tantalum after creep at low stresses (29). Brinson and Argent (30) noted that there was a tendency for subgrains to form in bands in niobium crept at 0.5 $T_m$. Similar to other crystal structures, subgrain formation in the bcc metals is more prominent along grain boundaries (8, 9, 41); serrations in the grain boundaries have been associated with subgrains (9).

In both iron (3) and tungsten (25) the subgrain size, $d$, depends on stress as $d \propto \sigma^{-1}$. This is the same stress dependence obeyed by
the coarse slip band spacing in tantalum\(^{(29)}\). In Fe-3Si\(^{(8)}\) this relation is obeyed approximately only at low stress levels. At high stresses the variation becomes \(d \propto \sigma^{-2.4}\). At a given stress the sub-grain size in iron was independent of the degree of prior cold work or temperature.

The asterism in Laue back-reflection X-ray patterns from coarse-grained Mo\(^{(18)}\) and tungsten\(^{(23)}\) show that the rotation axis is nearly parallel to a [[110]] type direction. The asterism has broken up into a series of spots indicative of subgrains having small relative misorientations within the large grains. Generally after creep at higher temperatures the individual spots within the streak become sharper since polygonization proceeds faster. An exception to this is the observation by Brinson and Argent\(^{(30)}\) that large grains formed during creep in niobium at about 0.5 T\(_m\) showed no substructure or X-ray asterism. In iron Garofalo et al.\(^{(4)}\) used an X-ray technique to show that the sub-grain size increased during primary creep but was essentially constant during steady state creep. However, the misorientation between the subgrains appeared to increase throughout steady state creep. This is one argument offered against the hypothesis that subgrain boundaries do in fact control creep in some way rather than existing merely as a result of some independent creep mechanism. However, there has not been sufficient corroboration of the magnitude of this change in misorientation angle during creep or of a predicted influence of an increase in misorientation angle on some operative creep mechanism to substantiate the importance of subgrain boundaries as unimportant in creep.
The character and arrangement of the dislocations within the subgrain boundaries and subgrains have been investigated by etch pitting and transmission electron microscopy. The character of the subgrain boundaries has not been studied in detail and one can sum up the observations made in Fe-3%Si\(^{(8)}\), Fe\(^{(4,7,61)}\), and W\(^{(24)}\) by saying that numerous low angle dislocation network boundaries and some tilt boundaries are present after creep. Within the subgrains, dislocation loops and tangles are present in tungsten\(^{(23)}\) while in iron the individual dislocations are relatively isolated and uniformly distributed\(^{(7,8,62)}\) and may combine frequently to form nodes in a three-dimensional network\(^{(8,62)}\). The dislocations within the subgrains also appear to be aligned along several preferred directions in thin foils of iron\(^{(5,7)}\) and Fe-3%Si\(^{(8)}\) but the crystallographic directions in the foils are not given. However Dingley and Hale\(^{(61)}\) state that the character of the dislocations after creep in low alloy steels was of a random nature. These three investigations were all in approximately the same temperature range.

The free dislocation density within the subgrains decreases continuously throughout primary creep and reaches a constant level in the steady state region in Fe-3%Si\(^{(8,9)}\). The constant, steady state dislocation density is independent of grain size indicating that grain boundaries are not significant dislocation sources as proposed by Garofalo et al.\(^{(62)}\). In iron the density is either increasing very slowly or is essentially constant after primary creep but the data are incomplete\(^{(7)}\). During steady state creep the dislocation density is quite stress dependent, similar to the case of low temperature
deformation. In Fe(5,7) and an Fe-Mn-N alloy(5) the dislocation density varies with the applied stress as \( \rho \propto \sigma^2 \). However this is an approximation and Barrett(64) has shown that plotting \( \log \rho \) vs. \( \log \sigma \), where \( \rho \) is the free dislocation density and \( \sigma \) is the applied stress, can produce a range of exponents. The data on Fe-3%Si give \( \rho \propto \sigma^{2.64} \), \( \rho \propto \sigma^{1.4} \), and \( \rho \propto \sigma^{3.10} \). The exponents of 1.4 and 3 were obtained at nearly the same temperature, about 0.5 \( T_m \), and over the same stress range.

Dynamic recrystallization and grain growth during creep has been referred to earlier and no doubt plays a role in some of the creep studies mentioned here. The propensity for grain growth during creep varies widely depending on purity, prior thermal and mechanical treatment and the stress during creep. Extensive grain growth has been observed in arc melted Mo(18) and W(18) and some in Nb(30), while on the other hand the grain size remained stable in W(27) and Ta(29) creep in the same temperature range 0.5 to 0.8 \( T_m \).

In summary, in body-centered cubic metals there is some correlation of the activation energy for creep above 0.6 \( T_m \) with the activation energy for self diffusion. Below 0.6 \( T_m \) one cannot make any generalizations regarding the activation energy for creep. The motion of jogged screws and climb of edge dislocations have been proposed as rate-controlling mechanisms. However in high temperature creep observations of the substructure developed during creep have been too limited to correlate the possible mechanisms with the substructure.
Creep Theory

Creep refers to the time-dependent deformation of a material subjected to a constant load or stress. Creep can occur over a wide range of stress and temperature in any particular metal having a variety of different internal substructures. Over this spectrum of stress, temperature, and internal structure different creep mechanisms may be rate controlling within limited ranges of the variables. The following brief discussion of creep theory will necessarily be limited to creep in pure metals at temperatures above one-half the melting point (0.5 \( T_m \)), where the activation energy for creep is often equal to the activation energy for self diffusion, and to stresses greater than the yield stress, i.e., excluding microcreep.

The ultimate objective of any creep theory is to reproduce and predict the entire creep curve of total creep strain vs. time including the temperature, stress, structure, and time dependence of the creep rate. The curve of creep strain vs. time is usually made up of four distinguishable stages. These are (1) the initial instantaneous plastic strain, \( \varepsilon_0 \), that occurs as the load is applied, (2) a region of decelerating creep rate called primary creep or Stage 1, (3) a region of constant creep rate called secondary (steady state) creep or Stage 2, and (4) a period of accelerating creep rate called tertiary creep or Stage 3. Only primary and secondary creep will be discussed here. The initial plastic strain \( \varepsilon_0 \) is certainly important in the overall picture since it precedes any time-dependent creep strain. However, very responsive and sensitive strain-measuring devices are required to
properly study the initial portions of the creep curve and few studies have been made of this region.

The general form of the creep strain-time relation, excluding tertiary creep, is

\[ \varepsilon = \varepsilon_0 + b f(t) + \dot{\varepsilon}_S t \]  

where \( \varepsilon \) is the total creep strain, \( t \) is time, and \( b \) and \( \dot{\varepsilon}_S \) are constants at constant stress and temperature. Aside from relating \( \varepsilon_0 \) to stress, temperature, and structure the greatest difficulty in analyzing Equation (1) is in determining the appropriate function \( f(t) \), in the second term, which describes the transient creep component of the creep strain. The steady-state term on the right can be readily determined experimentally and can be treated separately or incorporated into \( f(t) \).

In the above form \( \dot{\varepsilon}_S \) is the steady-state creep rate. The overall relation is such that the instantaneous, transient, and steady-state contributions to the total creep strain are separate and additive. The contribution of the second term is large at relatively short times, i.e., during primary creep, where the third term is small. At longer times, during steady-state creep, the second term decays to a very small time dependent contribution and the third term dominates. Hence the steady-state mechanism and transient mechanisms are separate and they contribute to the total creep strain throughout the creep test to a greater or lesser degree. When the same creep mechanism operates over the entire creep curve it is more appropriate to combine the second and third terms into one function which changes rapidly at first and approaches a constant slope as time increases.
One can write a general empirical strain-time relation to fit any creep curve by including sufficient parameters. Many functions have been proposed and matched to a wide variety of data. These are discussed by Kennedy(65), Garofalo(66), Conway and Flagella(21,23), and Crussard(67).

Many of these functions are quite complex, giving creep strain in terms of parabolic, hyperbolic, hyperbolic sine, logarithmic, and exponential functions of time and various combinations of these functions.

Conway and Flagella(21) have matched many of the proposed time laws, both simple and complex, to the same creep curves. They all fit the experimental creep curves with varying degrees of success. Some of them give excellent fit but the complex relation of the parameters in some of these equations makes it difficult to include their dependence on stress, temperature, and structure in any reasonably simple way based on physical models for creep. Hence it would be virtually impossible to construct meaningful creep theories for many of these empirical relations.

However, it is not necessary to do so. Some of the simpler time laws do fit the curves well and others may be special cases of a more general time law. For example, Crussard(67) has shown that for many metals over a wide range of stress, temperature, and internal structure transient creep follows a power law of the form

\[ \varepsilon - \varepsilon_0 = bt^m \]  

instead of separate parabolic, logarithmic and, sometimes, hyperbolic
time laws. One reason for the multiplicity of time laws for transient creep is that curves of $\varepsilon - \varepsilon_0$ vs. the various time functions can give straight lines for more than one law because of the limited range of the data and the sensitivity of the slope on a logarithmic scale at low strains to the choice of $\varepsilon_0$. Crussard points out that this problem is easily avoided by plotting the derivative of Equation (2)

$$\frac{d\varepsilon}{dt} = b m t^{m-1} ,$$

on a log-log basis. If a power law is obeyed one then obtains a straight line of slope $(m-1)$ independent of a choice of $\varepsilon_0$. It is now clear from the derivative form that the parabolic, hyperbolic, and logarithmic laws are all special cases of the power law, Equation (2), i.e., $0 < m < 1$, parabolic; $m = 0$, logarithmic; $m < 0$, hyperbolic. Admittedly, this approach does not always work; sometimes straight lines will not be obtained on a log-log plot, and a simple power law will not suffice. The greatest limitation is that it does not apply in the limit of zero time where it would predict an infinite creep rate. However at zero time, a finite plastic strain is already imposed at the strain rate used to load the specimen so that the concept of zero time is somewhat nebulous. It is suggested that this or a similar approach can achieve a greater degree of unity between some of the time laws. They can be related by allowing $m$ to vary with stress, temperature, or structure. Hence this type of relation should be quite tractable for creep theory.

The general strain-time relation can be predicted in two ways, both of which have some experimental support. First, one may treat the
primary and steady state as separate regions of the creep curve controlled by different creep mechanisms which merely add to give the total creep strain. This was the historic approach attributable in great part to Andrade\textsuperscript{68,69}. Many other theories of transient\textsuperscript{70-79} and steady-state\textsuperscript{80-90} creep have been presented. A distinct partition between transient and steady-state creep cannot be made in some of these theories since it is implicit in them that if one or more of the physical quantities in the models are allowed to vary with strain before reaching an equilibrium state, they could conceivably apply throughout primary and steady-state creep.

The second approach is to allow the same creep mechanism(s) to operate over the entire creep curve\textsuperscript{79,91-99}. In this approach, the mechanism can be couched explicitly in terms of substructural defects as in the dislocation dynamics theories of Li\textsuperscript{93}, Webster\textsuperscript{97,98}, and Haasen\textsuperscript{99}. These theories enable one to theoretically "control" the mobile dislocation density and velocity with increasing creep strain by a suitable choice of values for the appropriate parameters. This approach can give either logarithmic or exponential time laws for transient creep\textsuperscript{93,97,98}. Raymond and Dorn\textsuperscript{94} and Barrett et al.\textsuperscript{96} have related the creep rate to the density of mobile screw dislocations in a somewhat similar manner by interpreting their theories such that the decelerating creep rate is primarily due to a decrease in the mobile screw dislocation density. The latter authors have not derived time laws.

Theories based on the concept of the creep rate being governed by a balance between work hardening and recovery\textsuperscript{79,84,95} implicitly
include both primary and steady-state creep in the theory. Work hardening occurs with increasing creep strain causing the creep rate to decrease (primary creep) until the recovery rate balances the work-hardening rate, at which time the creep rate remains constant (steady-state creep). The strain dependence of the work hardening is tied vaguely to the gradient of a stress-strain curve at room temperature or 0°K. The recovery rate is also based on some similar index, although the work hardening and recovery mechanisms sometimes are stated in terms of specific mechanisms involving substructural defects (79,95). Parabolic and logarithmic time laws are obtained.

Creep theories which contain such quantities as an empirical work-hardening rate or recovery rate as a parameter are clearly unsatisfactory as an end in understanding creep behavior. This approach was and is a necessary and useful means of relating general creep behavior to certain classes of rate-controlling mechanisms. However, ultimately one must identify these general parameters with more specific mechanisms which include elements of the substructure developed during creep and must formulate the creep curve in this more fundamental way. The theories can then be judged not only on their ability to reproduce the time, stress, and temperature dependence of the creep rate, but also on their compatibility with the creep substructure. For example, McLean (95) has recently proposed a combination of work hardening and recovery as it applies to the three-dimensional dislocation network within the subgrains in an attempt to relate this concept more closely to the observed creep substructure. The whole concept of work hardening by this approach depends on an increase of the "free" dislocation
density, i.e., those within the subgrains, with increasing creep strain until a constant dislocation density is reached in steady-state creep. However, recent studies show just the opposite structural changes. The free dislocation density decreases continuously from the time of loading until a constant lower dislocation density is achieved in the steady-state region\(^{(96)}\). Hence, McLean's theory is probably restricted to lower temperatures where no steady-state creep is observed and an increase in the free dislocation density can be the cause of the decelerating creep rate. This example illustrates the importance of including the substructural defects into the theory as quantitatively as possible so that predictions can be compared to the observed creep substructure.

The models based on the movement of jogged screws\(^{(87,91,94,96)}\) require a decreasing free dislocation density until steady state is reached. This has been observed. In addition, the dislocations must have a high screw to edge ratio. This ratio has not been determined but could be readily found by transmission electron microscopy.

The models based on dislocation dynamics\(^{(93,97-99)}\) can be made compatible with nearly any kind of creep substructure. The numerous parameters and assumptions merely need to be adjusted to provide the appropriate dislocation density-creep strain relation. The character of the moving dislocations can be identified with those which are subsequently observed. Without independent measurements of fraction of mobile dislocations, dislocation velocity-stress dependence, multiplication, and annihilation kinetics at high temperatures, however, absolute creep rates cannot be calculated. Nonetheless this approach is
probably most satisfactory in the sense that it does include all phases of motion, formation and annihilation of dislocations, and in many systems these are the principal elements contributing to creep strain. At this stage the approach is unsatisfactory in that the parameters can probably be adjusted to fit any kind of observable creep curve. It must be recast in terms of more fundamental mechanisms before it does more than present a useful physical picture of dislocation dynamics during creep.

The more specific models incorporating dislocation dynamics can be checked against creep structure. Garofalo et al. proposed a dislocation dynamics model incorporating the effect of grain size. Grain boundaries acting as sources would influence the free dislocation density and so modify the creep rate. The theory fit the experimental creep curves well over a wide range of stress, temperature, and grain size, but the dependence of dislocation density on grain size was not determined. In an Fe-3%Si alloy, the free dislocation density was independent of grain size, eliminating the above rationalization for this alloy.

It follows from the previous discussion that creep is very dependent on the microstructure. In addition it is also quite sensitive to stress and temperature. This can best be shown by rewriting Equation (1) as

\[ \varepsilon = \varepsilon_0 ((\sigma, T, st)) + b f(t) ((\sigma, T, st)) + c ((\sigma, T, st)) t , \quad (4) \]

where \( \sigma \) is the applied stress, \( T \) is the temperature, and \( st \) is a structure factor. Of these variables, the dependence on temperature is
simplest and is discussed first. Many proposals have been made describing the temperature dependence of the creep curve. These are discussed in some detail in Kennedy\(^{(65)}\) and Garofalo\(^{(66)}\). Dorn and co-workers\(^{(55)}\) have provided what is probably the most reasonable approach. They introduced a time-temperature parameter \(\theta = te^{-Q/RT}\). They have shown for a number of cases that creep curves at constant stress but at different temperatures can be superimposed when creep strain is plotted vs. \(\theta\) and that the same structure is present at the same strain independent of temperature when \(Q\) is a constant, which it often is above \(0.5 T_m\). When the entire creep curves superimpose, the same mechanism must be operative during both primary and steady-state creep. Instead of writing \(t\) in Equation (4) the temperature dependence can be included by writing \(\theta\) when \(Q\) is independent of stress and strain. Creep models covering the primary and steady-state creep regions should provide time laws for substructural changes in terms of \(\theta\). Such laws were developed in a preliminary way by Raymond and Dorn\(^{(94)}\) to demonstrate that the density of mobile screw dislocations during creep could be a function of \(\theta\). Within the limits of this discussion the activation energy for creep agrees closely with the activation energy for self diffusion. The activation energy is independent of stress and strain.

Other, more specific creep models can include a stress dependent activation energy. Such creep models are based on either movement of jogged screws\(^{(87, 91, 94, 96)}\) or on a recovery mechanism involving climb of edge dislocations\(^{(79, 85, 86)}\). These mechanisms were specifically developed to agree with the experimental \(Q_c\). Both of these mechanisms, and other mechanisms which include a stress aided, thermally activated
process usually have an additional temperature-dependent factor which need not be included in the exponential with the activation energy. At low stresses it would appear as \( \frac{\alpha}{T} \) in the pre-exponential and at higher stresses it would appear as \( e^{\frac{\alpha}{T}} \) or \( \sinh \left( \frac{\alpha}{T} \right) \) where \( \alpha \) is stress dependent. These cases are reviewed by Kennedy (65), Garofalo (66), and Hirth and Lothe (101). The temperature dependence of \( \theta \) is usually much greater than that of these added factors so that they are relatively unimportant. Perhaps this is one reason why Dorn's simple \( \Theta \) parameter has been so successful in interpreting creep, even when a more complex temperature dependence is present.

Inserting the temperature dependence of creep strain into Equation (4) was relatively easy for diffusion-controlled creep. However this is not so for creep at other temperatures or in more complex materials. More than one creep mechanism, each having a different activation energy, may be rate controlling and the temperature dependence of the creep rate, i.e., the apparent activation energy for creep, may include not only one or more true activation energies but a temperature variation of other structural variables also. To sort out this complex state of affairs one would have to apply specific creep models and include the structural parameters quantitatively in order to separate out the various components of the temperature dependence. An analogue to this situation exists in the analysis of the apparent activation energy for a chain type chemical reaction.

The stress dependence of the creep strain is not so easily included in Equation (4). Many types of empirical stress-dependence relations have been proposed (65, 66). These are essentially power laws
at low stresses, exponential laws at high stresses and hyperbolic sine
laws which include both of these cases. No stress function equivalent
to the $\Theta$ parameter has been found. There is good reason for this. It
is evident from the time dependence of the creep rate at constant stress
and temperature that the rate is very sensitive to the substructure
formed with increasing creep strain. This is the only quantity chang­
ing during creep. It has been demonstrated that the structure at a
given creep strain is quite sensitive to stress although it is rela­
tively independent of temperature. As a result the strain at the onset
of secondary creep is stress dependent which in itself eliminates the
possibility of a simple $\Theta$ function in stress. Under certain conditions
Garofalo\textsuperscript{(66)} suggests that the strain rates are equal at a constant
ratio of transient strain to total transient strain, implying identical
structures at constant values of this ratio. However, these quantities
are not easily identified from the creep curve.

There have been several detailed studies of the stress dependence
of the creep rate\textsuperscript{(102-107)} but these are quite complicated. The com­
plication results from the recognition that stress influences the creep
rate in two ways: (1) it modifies the structure, i.e., the number and
arrangement of dislocations, and (2) it modifies the rate of the con­
trolling creep mechanism. These separate effects of stress on creep
mechanism and structure have been beautifully illustrated in aluminum
by Sherby et al.\textsuperscript{(106,107)} by using stress changes at constant structure
and comparing the results to constant stress tests. Stress changes
were made at a constant creep structure by pre-creeping to a given
strain at constant stress and temperature. The stress dependence of
the strain rate immediately after the stress change is probably representative of the stress dependence of the creep mechanism. When the creep rates over entire creep curves measured at different constant stresses were normalized using this stress dependence there was a residual difference in creep rate at a constant strain associated with the stress dependence of the creep structure. More recently Raymond et al.\textsuperscript{102} have shown that the stress dependence of the creep mechanism can also be dependent on structure, i.e., creep strain, so that these parameters are interdependent over the creep curve. This analysis also suggests that transient and secondary creep are probably not two separate additive processes. The transients in the creep rate and the structural changes that take place after a stress change during the creep test should be quite useful in illuminating the true rate-controlling mechanisms. Hence creep theory must include the stress dependence of both the creep mechanism and the time-dependent creep structure.

Most studies of the stress dependence of high temperature creep have concentrated on the steady-state creep rate, $\dot{\varepsilon}_s$, in Equation (4). This is by far the easiest to study but it does include the influence of substructure. At low stresses a power law is obeyed, $\dot{\varepsilon}_s \propto \sigma^n$, with $n$ between 4 and 6. At high stresses an exponential law is observed $e^{BH}$. A hyperbolic sine law includes both these extremes. A recent careful study showed that the stress dependence of the creep rate at constant structure during secondary creep was slightly greater than the stress dependence of the secondary creep rate\textsuperscript{102}. The substructure must then contribute an inverse stress dependence to the steady-state creep
rate. This is what one would expect if the subgrain boundaries were a rate-controlling influence in addition to the density of free dislocations (from the observations that a decrease in the time and stress dependent creep rate corresponded to a decrease in the free dislocation density\(^8\)).

The theories of the secondary creep rate of Weertman\(^{85,86}\) must be regarded as tentative even though they give the correct stress dependence. They are based on edge dislocation pileups and none have been observed in creep structures with one possible exception\(^{29}\). In addition, although \(N\) dislocations in a pileup might increase the climb force on the leading dislocation by a factor \(N\), the fact that there are \(N\) dislocations in the group all tending to climb in the same direction would decrease the climb rate of the individual dislocation by about \(\frac{1}{N}\) if the climb rate was diffusion controlled. According to Li\(^{109}\) pileups of edge dislocations of opposite sign on parallel slip planes are only stable for very low \(N\) so that the use of this type of configuration is probably not warranted.

The models based on the drag of jogged screws have recently split the stress dependence into the stress activation term and a stress dependence of the density of mobile jogged screws\(^{94}\).

The dislocation dynamics theories have not yet been formulated with a temperature or stress dependence. This is perhaps not surprising. They are more or less a framework into which one can insert quantitative measurements, observations, and mechanisms as they become available for the vague parameters they now possess.
Hence, a vast number of theories have been proposed as possible mechanisms of high temperature creep. Many of them can be made to fit creep curves through a suitable choice of the parameters. Phenomenologically, the Dorn parameter, \( \theta = te^{-Q/RT} \), describes the temperature dependence of creep curves quite well at constant stress. The stress dependence of the steady-state creep rate of \( \dot{e}_s \propto \sigma^n \) has often been observed with \( n \) varying between 4 and 6. However, the proposed mechanisms either do not fit the structure very well or they require structural parameters which are not yet available.
CALCULATION OF CLIMB FORCE ON SLIP DISLOCATIONS
FOR CORNER AND EASY GLIDE ORIENTATIONS

To aid in the analysis of the data, several theoretical analyses
were necessary, as discussed in the next two chapters. First, consider
the climb force on dislocations. Together with already existing
analyses of glide forces, this enables one to predict relative climb
and glide forces on dislocations in crystals of various orientations.
The effective stress causing a dislocation in a crystal to glide is the
shear stress component acting across its glide plane in the direction
of the Burgers vector. Both the external and internal stresses must be
resolved in this manner to find the effective stress causing the dis-
location to move in its glide plane. However, at elevated temperatures,
above 0.5 $T_m$, self diffusion becomes rapid enough so that climb of the
dislocation normal to its glide plane can become an important rate-
controlling process. Since a normal stress on the edge component of
the dislocation is an important factor, this process is generally con-
sidered to be important only in finite or unrelaxed sub-boundaries or
in pile-up configurations where the internal stress field from the
nearby dislocations can be quite large. However, the smaller but
persistent climb force exerted on the dislocation by the externally
applied load as it moves through the crystal at elevated temperatures
can influence its motion in much the same way that the glide force
alone does at lower temperatures although usually to a lesser degree
because of the higher viscous resistance to climb.
However, it is somewhat misleading to adhere to a complete analogy between a normal force and a glide force acting on a dislocation loop. The glide force experienced by a dislocation because of externally applied stresses is independent of the screw-edge character of the dislocation whereas the normal force is very dependent on the dislocation character. The normal force can even change sign as the character of the dislocation changes from edge to screw. Therefore the path that an expanding dislocation loop traverses at elevated temperatures can be quite different from the path it would follow at low temperatures, even in the absence of interactions with nearby dislocations.

Hence the climb forces may be quite important at elevated temperatures where the climb mobility can become appreciable. Several authors (110-113) have pointed out the usefulness of determining the shear stresses required for dislocation glide graphically. In this section a similar method will be presented for the climb case. The method of Hartley and Hirth (110) utilizing a standard stereographic projection for the crystal system in question will be followed.

**Coordinate Systems**

Two orthogonal coordinate systems are defined, one referred to the specimen geometry and one referred to the slip systems of the specimen. The specimen is assumed to be a rod loaded uniaxially along its long axis. Figure 1 illustrates the coordinate systems referred to the specimen and Figure 2 illustrates the relationship between the coordinate systems referred to the stereographic projection of the slip
FIGURE 1. COORDINATE SYSTEMS USED TO RESOLVE STRESSES
Figure 2. Coordinate systems used to resolve stresses referred to a stereographic projection.
system. Table 3 contains the definitions of the coordinate axes. Note that \( \mathbf{e}_1, \mathbf{e}_1', \) and \( \mathbf{e}_2' \) lie in the slip plane and \( \mathbf{e}_2, \mathbf{e}_2' \) and \( \mathbf{e}_3' \) lie in a plane parallel to the specimen axis.

**TABLE 3**

**DEFINITIONS OF THE UNIT VECTORS, \( \mathbf{e}_1 \), PARALLEL TO THE COORDINATE AXES, \( x_i \)**

<table>
<thead>
<tr>
<th>Quantity</th>
<th>Definition</th>
<th>Coordinate Axis</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \mathbf{e}_3 )</td>
<td>Parallel to the axis of the specimen</td>
<td>( x_3 )</td>
</tr>
<tr>
<td>( \mathbf{e}_2 )</td>
<td>((\mathbf{e}_3 \times \mathbf{e}_1))</td>
<td>( x_2 )</td>
</tr>
<tr>
<td>( \mathbf{e}_1 )</td>
<td>((\mathbf{e}_3 \times \mathbf{e}_3')/\sin \phi)</td>
<td>( x_1 )</td>
</tr>
<tr>
<td>( \mathbf{e}_3' )</td>
<td>Parallel to the slip plane normal</td>
<td>( x_3' )</td>
</tr>
<tr>
<td>( \mathbf{e}_2' )</td>
<td>((\mathbf{e}_3' \times \mathbf{e}_1'))</td>
<td>( x_2' )</td>
</tr>
<tr>
<td>( \mathbf{e}_1' )</td>
<td>Parallel to the slip direction</td>
<td>( x_1' )</td>
</tr>
<tr>
<td>( \lambda )</td>
<td>( \sin^{-1} (\mathbf{e}_3 \times \mathbf{e}_1') ), Angle between the specimen axis and the slip direction</td>
<td></td>
</tr>
<tr>
<td>( \phi )</td>
<td>( \sin^{-1} (\mathbf{e}_3 \times \mathbf{e}_3') ), Angle between the specimen axis and the slip plane normal</td>
<td></td>
</tr>
<tr>
<td>( \theta )</td>
<td>( \sin^{-1} (-\mathbf{e}_2' \times \mathbf{e}_1) = \cos^{-1} (\cos \lambda / \sin \phi) )</td>
<td></td>
</tr>
</tbody>
</table>
Transformations between Coordinate Systems

The transformation between the \((x_1, x_2, x_3)\) and the \((x'_1, x'_2, x'_3)\) coordinate system is given by

\[ \begin{array}{c|ccc}
& x_1 & x_2 & x_3 \\
\hline
x'_1 & a_{11} & a_{12} & a_{13} \\
x'_2 & a_{21} & a_{22} & a_{23} \\
x'_3 & a_{31} & a_{32} & a_{33} \\
\end{array} \]

where \(a_{ij}\) are the cosines of the angle between \(x'_i\) and \(x_j\). These are

\[
a_{ij} = \begin{pmatrix} a_{11} & a_{12} & a_{13} \\ a_{21} & a_{22} & a_{23} \\ a_{31} & a_{32} & a_{33} \end{pmatrix} = \begin{pmatrix} \sin \theta & (\cos \theta \cos \phi) & (\sin \phi \cos \theta) \\ -\cos \theta & (\sin \theta \cos \phi) & (\sin \phi \sin \theta) \\ 0 & -\sin \phi & \cos \phi \end{pmatrix}
\]  

(5)

The stresses \(\sigma_{kl}\) referred to the specimen coordinate system \((x_1, x_2, x_3)\) may be transformed to the stresses \(\sigma'_{ij}\) referred to the slip plane coordinate system \((x'_1, x'_2, x'_3)\) by

\[
\sigma'_{ij} = a_{ik} a_{jl} \sigma_{kl}.
\]  

(6)

It is understood that the repeated suffices, e.g., \(k\) and \(l\), are to be summed over all indices, \(1\) to \(3\). Hence for uniaxial tension or compression, \(\sigma_{33}\) is the only non-zero stress and the stress resolved along the slip direction in the slip plane is given by
\[ \sigma'_{13} = a'_{13} a'_{33} \sigma_{33} \]  

(7)

A transformation in terms of the angles \( \lambda \) and \( \phi \), the angles incorporated in the Schmid factor, is presented in Appendix A.

**Force on a dislocation**

The general form for the force on a dislocation in a stress field is given by

\[ F = (\Sigma \cdot b) \times \xi \],

(8)

where \( F \) is the force vector on the dislocation, \( \Sigma \) is the stress field tensor \( (\sigma_{ij}) \), \( b \) is the Burgers vector, and \( \xi \) is the tangent to the dislocation line and gives the directional sense of the line.* The complete expression for the force on a dislocation in the \( x_1'x_2'x_3' \) coordinate system due to the stress field \( \Sigma \), is given by

\[
F = \left[ \xi_3 (b_1' \sigma'_{12} + b_2' \sigma'_{22} + b_3' \sigma'_{32}) - \xi_2 (b_1' \sigma'_{13} + b_2' \sigma'_{23} + b_3' \sigma'_{33}) \right] \mathbf{e}_1' \\
+ \left[ \xi_1 (b_1' \sigma'_{11} + b_2' \sigma'_{21} + b_3' \sigma'_{31}) - \xi_3 (b_1' \sigma'_{13} + b_2' \sigma'_{23} + b_3' \sigma'_{33}) \right] \mathbf{e}_2'
\]

(9)

\[
+ \left[ \xi_2 (b_1' \sigma'_{11} + b_2' \sigma'_{21} + b_3' \sigma'_{31}) - \xi_1 (b_1' \sigma'_{12} + b_2' \sigma'_{22} + b_3' \sigma'_{32}) \right] \mathbf{e}_3'
\]

where the components of \( \xi \) and \( b \) are also referred to the \( x_1'x_2'x_3' \) coordinates.

In order to simplify the manipulations the slip plane is chosen parallel to the \( x_1'x_2' \) plane, and \( b = (b'_1,0,0) \), \( \xi = (\xi'_1,\xi'_2,0) \). Then the climb force from Equation (9) is

\[ F_3 = [\xi'_2 (b'_1 \sigma'_{11}) - \xi'_1 (b'_1 \sigma'_{12})] \mathbf{e}_3' \]

(10)

---

*The direction of \( b \) is obtained using the right-handed convention, e.g., \( b \) and \( \xi \) point in the same direction for a right-handed screw.*
The first term in the brackets is the climb force on the edge component and the second term is the cross slip force normal to the slip plane on the screw component. If $\alpha$ is the angle between $b$ and $\xi$ measured from $b$ to $\xi$ then $\xi_1 = \cos \alpha$ and $\xi_2 = \sin \alpha$. Then for the uniaxial stress case where only $\sigma_{33} \neq 0$, we get

$$F_3 = b\sigma_{33} \sin^2 \phi \cos \Theta [\cos \Theta \sin \alpha - \sin \Theta \cos \alpha] . \quad (11)$$

Inserting the appropriate values for $\phi$, $\Theta$, and $\alpha$ one finds the force normal to the slip plane for a given slip system and orientation$^\dagger$ of the tensile axis.

If one calculates this normal force on a given slip system for all orientations of the stress axis, one can plot contours on a stereographic projection of stress axis orientations for constant normal force. This is done in Figures 3 and 4 for the edge and screw components, respectively. For a tensile stress, the climb force is positive, i.e., in the $+_a$ direction, for a "negative" edge (extra half plane extending below the glide plane) and negative, i.e., in the $-_a$ direction, for the "positive" edge dislocation. For a right-handed screw dislocation the normal glide force is in the $-_a$ direction when the tensile stress axis is in the left quadrant and is in the $+_a$ direction when the tensile stress axis is in the right quadrant. This sequence is reversed for a left-handed screw component. Figure 5 shows a graph of the direction of the normal forces on the edge and screw components with respect to

$^\dagger$Throughout the thesis "orientation" will be used to refer to the crystallographic direction parallel to the specimen and stress axis.
FIGURE 3. STEREOGRAPHIC PROJECTION OF THE CLIMB FORCE ON A PURE EDGE DISLOCATION UNDER UNIAXIAL STRESS IN UNITS OF $F_3/\sigma_{33}b$. The slip direction $e_1$ points out of the page.
FIGURE 4. STEREOGRAHIC PROJECTION OF THE CLIMB FORCE ON A PURE SCREW DISLOCATION UNDER UNIAXIAL STRESS IN UNITS OF $F_3 / \sigma_{33} b_1^\prime$.

The slip direction $e_1^\prime$ points out of the page.
FIGURE 5. THE RELATIVE DIRECTIONS OF NORMAL FORCES, (±), OUT OF THE GLIDE PLANE ON THE EDGE AND SCREW COMPONENTS OF A DISLOCATION.

The left and right quadrants refer to the stereographic projection in Figure 2.
the slip plane normal showing that the forces can either aid or oppose each other depending on the character of the dislocation.

For the corner orientations there will be sets of planes for the \((110)\)[[111]] and the \((112)\)[[111]] slip systems, respectively, which will be arranged symmetrically around the stress axis and will have the same values of \(\Theta\) and \(\Phi\). The \((110)\)[[111]] slip systems fall into two sets, one set has the maximum resolved shear stress (MRSS) while the other has no resolved shear stress. The \((112)\)[[111]] slip systems fall into 3 sets for the [[110]] and [[111]] orientations. One set has the MRSS for \((112)\)[[111]] systems, one set has one-half the MRSS, and one set has no resolved shear stress. For a [[100]] orientation there is no \((112)\)[[111]] slip system with zero resolved shear stress. The various normal and glide forces for each set of slip systems are given in Table 4 for the corner orientations. The table includes the value of \(\alpha_o\) at which the opposing forces cancel each other causing a net normal force of zero. Hence as one travels around a dislocation loop the normal force is zero and changes sign at \(\alpha_o\) and at \(\alpha_o + \pi\). However, although the distribution of the normal force over the loop varies as the stress axis changes, the net normal force over the whole loop is zero, independent of the stress axis.

It can be seen from the table that the climb force on the glide dislocations is the least for a tensile stress in the [111] direction and is greatest for a [110] tensile axis. The normal force on the screw component of the dislocations of the primary glide systems is small for all orientations, but is of the same order as the climb force.
TABLE 4

SUMMARY OF DISLOCATION CLIMB AND GLIDE FORCES FOR THE CORNER ORIENTATIONS OF THE STRESS AXIS

<table>
<thead>
<tr>
<th>Stress Axis Orientation</th>
<th>Number of Slip Systems (a)</th>
<th>Edge $F_3$</th>
<th>Screw $F_3$</th>
<th>$F_g$ (b)</th>
<th>$\alpha_0$ (c)</th>
<th>Edge $F_3$</th>
<th>Screw $F_3$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>$\sigma_{33}^b$</td>
<td>$\sigma_{33}^b$</td>
<td>$\sigma_{33}^b$</td>
<td></td>
<td>$F_3$</td>
<td>$F_3$</td>
</tr>
</tbody>
</table>

((110))[[111]] Slip Systems

<table>
<thead>
<tr>
<th>Orientation</th>
<th>Edge $F_3$</th>
<th>Screw $F_3$</th>
<th>$F_g$</th>
<th>$\alpha_0$</th>
<th>Edge $F_3$</th>
<th>Screw $F_3$</th>
</tr>
</thead>
<tbody>
<tr>
<td>[011]</td>
<td>0.667</td>
<td>0.068</td>
<td>0.408</td>
<td>5.8°</td>
<td>1.635</td>
<td>0.167</td>
</tr>
<tr>
<td>4</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>--</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>6</td>
<td>0.667</td>
<td>0.471</td>
<td>0</td>
<td>35.2°</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>2</td>
<td>0.667</td>
<td>0.471</td>
<td>0</td>
<td>54.8°</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>[001]</td>
<td>0.333</td>
<td>0.096</td>
<td>0.408</td>
<td>16.1°</td>
<td>0.816</td>
<td>0.235</td>
</tr>
<tr>
<td>8</td>
<td>0.333</td>
<td>0.471</td>
<td>0</td>
<td>54.8°</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>4</td>
<td>0.111</td>
<td>0.074</td>
<td>0.272</td>
<td>33.7°</td>
<td>0.408</td>
<td>0.272</td>
</tr>
<tr>
<td>3</td>
<td>1.0</td>
<td>0</td>
<td>0</td>
<td>0°</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>3</td>
<td>0.111</td>
<td>0.314</td>
<td>0</td>
<td>70.5°</td>
<td>--</td>
<td>--</td>
</tr>
</tbody>
</table>

((112))[[111]] Slip Systems

<table>
<thead>
<tr>
<th>Orientation</th>
<th>Edge $F_3$</th>
<th>Screw $F_3$</th>
<th>$F_g$</th>
<th>$\alpha_0$</th>
<th>Edge $F_3$</th>
<th>Screw $F_3$</th>
</tr>
</thead>
<tbody>
<tr>
<td>[011]</td>
<td>0.667</td>
<td>0</td>
<td>0.471</td>
<td>0°</td>
<td>1.416</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>0.667</td>
<td>0.408</td>
<td>0.236</td>
<td>31.7°</td>
<td>2.826</td>
<td>0</td>
</tr>
<tr>
<td>4</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>--</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>[001]</td>
<td>0.333</td>
<td>0</td>
<td>0.471</td>
<td>0°</td>
<td>0.707</td>
<td>0</td>
</tr>
<tr>
<td>8</td>
<td>0.333</td>
<td>0.408</td>
<td>0.236</td>
<td>50.8°</td>
<td>1.411</td>
<td>1.729</td>
</tr>
<tr>
<td>[111]</td>
<td>0.111</td>
<td>0</td>
<td>0.314</td>
<td>0°</td>
<td>0.354</td>
<td>0</td>
</tr>
<tr>
<td>3</td>
<td>1.0</td>
<td>0</td>
<td>0.157</td>
<td>67.8°</td>
<td>0.707</td>
<td>1.732</td>
</tr>
<tr>
<td>6</td>
<td>0.111</td>
<td>0.272</td>
<td>0.157</td>
<td>67.8°</td>
<td>0.707</td>
<td>1.732</td>
</tr>
</tbody>
</table>

(a) Positive and negative slip directions are not considered.

(b) Glide force in the slip plane.

(c) Angle between $b$ and $\xi$ at which $F_3 = 0$. 
for the [111] tensile axis. However for glide systems with one-half
the MRSS or no shear stress, the normal force on the screw component
may be larger than the climb force on the edge component. The largest
climb forces are attained in the [111] orientation on slip systems
having zero resolved shear stress. Where climb mobility is high these
systems may be active as Bardeen-Herring climb sources although they
are inactive in glide.

A comparison of the ratio of the normal force to the glide
forces as a function of orientation shows that the dislocations in the
primary slip systems will have a much greater tendency to climb than
glide for the [110] orientation, while for the other orientations the
climb force is less than the glide force. It is interesting to note
that there is no normal (cross-slip) force on the screw dislocations
of the primary slip systems of the ((112))[[111]] type for any of the
corner orientations, but there is a normal force on screws of the
primary ((110))[[111]] type slip systems. Also the fact that the
resolved shear stress is larger on the ((112)) systems compared to the
((110)) systems could cause a greater tendency to cross-slip out of
((110)) slip planes than to cross-slip out of ((112)) slip planes.

The ratios of the normal to the glide forces in Table 4 suggest
that the best orientation to try to sort out glide and climb is the
[011] orientation. Perhaps one could compare the stress and temperature
dependence of the creep behavior of the [011] orientation and an
orientation with a much lower climb to glide force ratio.
GENERAL ANALYSIS OF DISLOCATION REACTIONS
IN THE BCC STRUCTURE

Burgers Vector and Slip Plane Notation
for ((110)[[111]] Slip Systems

To study the dislocation interactions in the bcc crystal lattice it is useful to have the slip planes and directions designated by letters analogous to the Thompson tetrahedron\(^{(115)}\) to avoid handling the unwieldy Miller indices. One manner of doing this is shown in Figure 6 for ((110)[[111]] slip systems.* The slip planes are identified by Greek letters and the Burgers vectors of the dislocations are identified by a pair of Roman letters. The sequence of the letters indicates the direction of the vector when referred to Figure 6. The common slip vectors all contain the letter "0" and are of the type \(\frac{a}{2}[[111]]\). They will be referred to as 0 vectors. One may list from this figure all the possible slip plane-slip direction combinations for a given type of slip system, e.g., ((110)[[111]]. This is done in Table 5. The table lists two sets of notation for identical sets of Burgers vectors and slip planes. In set I all 0 vectors have 0 as the first letter of the pair, in set II all 0 vectors have 0 as the second letter of the pair, and each set contains both positive and negative

*After this notation was developed it was found that Amelinckx and Dekeyser\(^{(115)}\) had developed a similar notation.
Notation of \{110\} \{111\} systems.

Notation of \{112\} directions.

FIGURE 6. NOTATION OF THE CUBE
TABLE 5
LIST OF THE [[011]] TYPE SLIP PLANES AND THEIR $\frac{a}{2}$[[111]] TYPE SLIP VECTORS

Set I is identical to set II(a)

<table>
<thead>
<tr>
<th>Slip Plane</th>
<th>Burgers Vectors</th>
</tr>
</thead>
<tbody>
<tr>
<td>I $\alpha$</td>
<td>A'O</td>
</tr>
<tr>
<td>$\beta$</td>
<td>BO</td>
</tr>
<tr>
<td>$\gamma$</td>
<td>CO</td>
</tr>
<tr>
<td>$\delta$</td>
<td>DO</td>
</tr>
<tr>
<td>$\varepsilon$</td>
<td>A'O</td>
</tr>
<tr>
<td>$\rho$</td>
<td>BO</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>II $\alpha$</th>
<th>OC'</th>
<th>OD'</th>
<th>OA</th>
<th>OB</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\beta$</td>
<td>OD'</td>
<td>OA'</td>
<td>OB</td>
<td>OC</td>
</tr>
<tr>
<td>$\gamma$</td>
<td>OA'</td>
<td>OB'</td>
<td>OC</td>
<td>OD</td>
</tr>
<tr>
<td>$\delta$</td>
<td>OB'</td>
<td>OC'</td>
<td>OD</td>
<td>OA</td>
</tr>
<tr>
<td>$\varepsilon$</td>
<td>OC</td>
<td>OC'</td>
<td>OA'</td>
<td>OA</td>
</tr>
<tr>
<td>$\rho$</td>
<td>OD'</td>
<td>OD</td>
<td>OB</td>
<td>OB'</td>
</tr>
</tbody>
</table>

(a) The notation refers to Figure 6.
slip vectors as is easily verified by referring to Figure 6. To aid in visualizing a particular slip plane combination, pairs of planes are illustrated in Figure 7.

It is easiest to interrelate the vectors by referring to the lettering sequence ABCDAB ... or A'B'C'D'A'B' ... . The different types of Burgers vectors may then be described in the following way:

1. \( b = \frac{a}{2}[[111]] \)
   a. All 0 vectors are \( \frac{a}{2}[[111]] \) vectors.
   b. If one 0 vector has the 0 second in the pair, the parallel 0 vector may be obtained by placing the 0 first and replacing the leading letter with its alternate (nonsuccessive) letter in the sequence and priming it, e.g., \( AO = OC' \), \( BO = OD' \).
   c. One can obtain the negative vectors by retaining 0 in the same position but replacing the other letter with its alternate letter and priming it. This is done in the sets I and II in Table 5, e.g., \( AO = -C'O \), \( BO = -D'O \).

2. \( b = a[[100]] \)
   a. The letters are nearest neighbors in the sequence and are either both primed or both unprimed, e.g., \( AB, D'A' \).
   b. The letters are identical with one primed and one unprimed, e.g., \( BB' \).

3. \( b = a[[110]] \)
   a. The letters are alternate letters in the sequence and are either both primed or both unprimed.
FIGURE 7. PAIRS OF ((110))[[111]] SLIP SYSTEMS
b. The letters are nearest neighbors in the sequence with one primed and one unprimed.

The various reactions may be studied by reacting one dislocation of each type with all others having a common letter. From Figure 6 it is obvious that all reactions involving 0 vectors may be obtained by taking a vector such as \( A0_\alpha \) and reacting it with all the 0 vectors in set II. For reactions involving \( b = a[[100]] \) type dislocations, the reactions between one \( b = \frac{a}{2}[[111]] \) type dislocation and one \( b = a[[100]] \) dislocation are of greatest interest. This is most easily accomplished by considering each of a set of four parallel edges of the cube in order and reacting them with the 0 vectors connecting the ends of the vector to the center of the cube. In this way all positive and negative 0 vectors are included. The resultant vector of such a reaction is either an 0 vector or a vector of the type \( \frac{a}{2}[[311]] \).

Dislocations of the type \( b = a[[110]] \) have been reported in iron\(^{(63)}\), but the technique used to identify them was not unequivocal due to a possible influence of elastic anisotropy on electron diffraction contrast\(^{(117,118)}\). Therefore, reactions involving them will not be considered here nor will reactions involving two dislocations of the type \( b = a[[100]] \) since these create a resultant dislocation of \( b = a[[110]] \).

**Dislocation Reactions**

One can list all the possible reactions involving two dislocations for the \(((110))[[111]]\) slip systems in the bcc crystal structure by choosing one Burgers vector and slip plane and reacting it with all
others in either set I or set II of Table 5. The two sets of notation facilitate the determination of interactions by allowing a choice of the set compatible with the interacting dislocation chosen.

To begin, we will choose a dislocation, \( \mathbf{A}_0 \alpha \), having a Burgers vector \( \mathbf{A}_0 \) lying in the \( \alpha \) plane. It will then be reacted with all the dislocations of set II. Table 6 lists the description of the resultant interactions. In the table the line direction, \( \xi_p \), of the product dislocation is parallel to the line of intersection of the glide planes of the reacting dislocations. If the product dislocation is sessile it would remain in this position. The plane of the product dislocation is normal to \( (\mathbf{b}_p \times \xi_p) \), i.e., the plane of a dislocation by definition contains both the Burgers vector and the core of the dislocation. From the table one can immediately pick out the reactions of interest either in terms of the pairs of reacting dislocations or in the type of resultant dislocation.

In a crystal where more than one slip system is active, any of the other systems may be superimposed on \( \mathbf{A}_0 \alpha \) by a suitable choice of rotations of the cube around the cube axes. Of course, the product dislocation from any reaction is rotated through the same sequence of rotations. For crystals having the stress axis parallel to the corner orientations, \([\{100\}], \([\{110\}], \) and \([\{111\}], \) the symmetry makes this quite simple. As Table 4 shows, there are at most three dissimilar sets of slip systems.
### TABLE 6
DESCRIPTION OF DISLOCATION REACTIONS IN THE BCC STRUCTURE FOR 
\((110)[111]\) SLIP SYSTEMS

<table>
<thead>
<tr>
<th>Reaction Number</th>
<th>Glide Planes</th>
<th>Reaction</th>
<th>Result of Cutting without Combination (a)</th>
<th>(b_p) (b)</th>
<th>(\xi_p) (c)</th>
<th>Plane of the Product Dislocation ((b_p \times \xi_p))</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>(\alpha)</td>
<td>(\alpha)</td>
<td>(AO \alpha + OC\alpha = AC'\alpha)</td>
<td>(a[[11]])</td>
<td>--</td>
<td>(\alpha)</td>
</tr>
<tr>
<td>2</td>
<td>(\alpha)</td>
<td>(\alpha)</td>
<td>(AO \alpha + OD\alpha = AD\alpha)</td>
<td>(a[[110]])</td>
<td>--</td>
<td>(\alpha)</td>
</tr>
<tr>
<td>3</td>
<td>(\alpha)</td>
<td>(\alpha)</td>
<td>(AO \alpha + OA\alpha = 0) Annihilate</td>
<td>--</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>4</td>
<td>(\alpha)</td>
<td>(\alpha)</td>
<td>(AO \alpha + OB\alpha = AB\alpha) Combine (?)</td>
<td>(a[[100]])</td>
<td>--</td>
<td>(\alpha)</td>
</tr>
<tr>
<td>5</td>
<td>(\alpha \beta)</td>
<td>(\alpha \beta)</td>
<td>(AO \alpha + OA\beta = AA\rho) Jog, jog</td>
<td>(a[[110]])</td>
<td>(D'B[[111]])</td>
<td>(\alpha)</td>
</tr>
<tr>
<td>6</td>
<td>(\alpha \beta)</td>
<td>(\alpha \beta)</td>
<td>(AO \alpha + OB\beta = AB\alpha) Kink, jog</td>
<td>(a[[100]])</td>
<td>Ditto</td>
<td>(\alpha)</td>
</tr>
<tr>
<td>7</td>
<td>(\alpha \beta)</td>
<td>(\alpha \beta)</td>
<td>(AO \alpha + OC\beta = AC) Jog, jog</td>
<td>(a[[110]])</td>
<td>Ditto</td>
<td>((112))</td>
</tr>
<tr>
<td>8</td>
<td>(\alpha \beta)</td>
<td>(\alpha \beta)</td>
<td>(AO \alpha + OC\gamma = AA\gamma) Jog, jog</td>
<td>(a[[100]])</td>
<td>(AB[[100]])</td>
<td>(ABA'B'[[100]])</td>
</tr>
<tr>
<td>9</td>
<td>(\alpha \gamma)</td>
<td>(\alpha \gamma)</td>
<td>(AO \alpha + OA\gamma = AA'\gamma) Jog, jog</td>
<td>(a[[110]])</td>
<td>Ditto</td>
<td>(ABA'B'[[100]])</td>
</tr>
<tr>
<td>10</td>
<td>(\alpha \gamma)</td>
<td>(\alpha \gamma)</td>
<td>(AO \alpha + OB\gamma = AB\gamma) Jog, jog</td>
<td>(a[[100]])</td>
<td>Ditto</td>
<td>(ABCD[[100]])</td>
</tr>
<tr>
<td>11</td>
<td>(\alpha \gamma)</td>
<td>(\alpha \gamma)</td>
<td>(AO \alpha + OC\gamma = AC) Jog, jog</td>
<td>(a[[110]])</td>
<td>Ditto</td>
<td>(ABCD[[100]])</td>
</tr>
<tr>
<td>12</td>
<td>(\alpha \gamma)</td>
<td>(\alpha \gamma)</td>
<td>(AO \alpha + OD\gamma = AD) Jog, jog</td>
<td>(a[[100]])</td>
<td>Ditto</td>
<td>(ABCD[[100]])</td>
</tr>
<tr>
<td>Reaction Number</td>
<td>Glide Planes</td>
<td>Reaction</td>
<td>Result of Cutting without Combination</td>
<td>$b_p$</td>
<td>$\xi_p$</td>
<td>Plane of the Product Dislocation ($b_p \times \xi_p$)</td>
</tr>
<tr>
<td>-----------------</td>
<td>--------------</td>
<td>----------</td>
<td>---------------------------------------</td>
<td>-------</td>
<td>--------</td>
<td>-----------------------------------------------</td>
</tr>
<tr>
<td>13</td>
<td>$\alpha \delta$</td>
<td>$AO_\alpha + OB'_\delta = AB'$</td>
<td>Jog, kink</td>
<td>a[[110]]</td>
<td>AC'[[110]]</td>
<td>$\delta$</td>
</tr>
<tr>
<td>14</td>
<td>$\alpha \delta$</td>
<td>$AO_\alpha + OC'_\delta = AC'$</td>
<td>Kink, kink</td>
<td>a[[111]]</td>
<td>AC'[[110]]</td>
<td>$\alpha, \delta, \epsilon$</td>
</tr>
<tr>
<td>15</td>
<td>$\alpha \delta$</td>
<td>$AO_\alpha + OD_\delta = AD$</td>
<td>Jog, kink</td>
<td>a[[100]]</td>
<td>Ditto</td>
<td>$\delta$</td>
</tr>
<tr>
<td>16</td>
<td>$\alpha \delta$</td>
<td>$AO_\alpha + OA_\delta = 0$</td>
<td>Kink, kink annihilate at intersection</td>
<td>--</td>
<td>Ditto</td>
<td>--</td>
</tr>
<tr>
<td>17</td>
<td>$\alpha \epsilon$</td>
<td>$AO_\alpha + OC_\epsilon = AC_\epsilon$</td>
<td>Jog, kink</td>
<td>a[[110]]</td>
<td>Ditto</td>
<td>$\epsilon$</td>
</tr>
<tr>
<td>18</td>
<td>$\alpha \epsilon$</td>
<td>$AO_\alpha + OC'_\epsilon = AC'$</td>
<td>Kink, kink repel at intersection $AO = OC'$</td>
<td>a[[111]]</td>
<td>Ditto</td>
<td>$\alpha, \epsilon, \delta$</td>
</tr>
<tr>
<td>19</td>
<td>$\alpha \epsilon$</td>
<td>$AO_\alpha + OA'_\epsilon = AA'$</td>
<td>Jog, kink</td>
<td>a[[100]]</td>
<td>Ditto</td>
<td>$\epsilon$</td>
</tr>
<tr>
<td>20</td>
<td>$\alpha \epsilon$</td>
<td>$AO_\alpha + OA_\epsilon = 0$</td>
<td>Kink, kink annihilate at intersection</td>
<td>--</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>21</td>
<td>$\alpha \rho$</td>
<td>$AO_\alpha + OD'_\rho = AD'$</td>
<td>Jog</td>
<td>a[[110]]</td>
<td>D'B[[111]]</td>
<td>$\alpha$</td>
</tr>
</tbody>
</table>
### TABLE 6 (Contd.)

<table>
<thead>
<tr>
<th>Reaction Number</th>
<th>Glide Planes</th>
<th>Reaction</th>
<th>Result of Cutting without Combination</th>
<th>Plane of the Product Dislocation $(\mathbf{b}_p \times \mathbf{f}_p)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>22</td>
<td>$\alpha \rho$</td>
<td>$A_0\alpha + O\rho = AD$</td>
<td>Jog, jog</td>
<td>a[[100]] Ditto $\beta$</td>
</tr>
<tr>
<td>23</td>
<td>$\alpha \rho$</td>
<td>$A_0\alpha + OB\rho = AB$</td>
<td>Kink, jog</td>
<td>a[[100]] Ditto $\alpha$</td>
</tr>
<tr>
<td>24</td>
<td>$\alpha \rho$</td>
<td>$A_0\alpha + OB'\rho = AB'$</td>
<td>Jog, jog</td>
<td>a[[110]] Ditto ((112))</td>
</tr>
</tbody>
</table>

(a) The first word refers to the reacting dislocation $A_0\alpha$, the second to the other reacting dislocation. "Jog" refers to an offset of the dislocation having a component normal to the dislocation line. "Kink" refers to an offset lying within the glide plane of the dislocation.

(b) Gives the total Burgers vector of the product dislocation if combination takes place at the line of intersection of the slip planes.

(c) The line direction of the product dislocation. This corresponds to the line of intersection of the glide planes of the reacting dislocation.
**Formation of \( b = a[[001]] \) Dislocations during Tensile Deformation**

Since dislocations having a Burgers vector of \( a[[001]] \) have been observed in dislocation networks \((60,117)\) and in tilt boundaries formed in bcc metals during high-temperature creep (this study), it is obvious that they may play a significant role in the recovery process occurring during creep. In this light it is of interest to determine the modes of formation of dislocations having this Burgers vector and also the magnitude of the climb and glide forces on them.

This is done for each of the corner orientations and the orientation giving maximum resolved shear stress on the \((110)][[111]]\) system. Only combinations of the active slip systems are considered. The grown-in dislocations and those formed locally on macroscopically inactive systems are presumed to be much rarer than dislocations from the active systems and, therefore, to contribute little to the creep properties or dislocation substructure.

From Table 6 it can be seen that there are nine reactions which may lead to a \( b = a[[001]] \) dislocation. In one case, 4, the reacting dislocations share the same glide plane, in all the others the combination can occur at the line of intersection of the slip planes perhaps creating an attractive intersection.* In seven instances, 4, 6, 7, 15, 19, 22, and 23, the plane of the product dislocation is of the \((110))\)

*For parallel dislocations it is energetically favorable for two appropriate \( b = a/2[[111]] \) dislocations to combine to give one \( b = a[[100]] \) dislocation.

+The plane of a dislocation must contain both \( b \) and \( \xi \). Therefore the normal to the plane of the dislocation is parallel to \( b \times \xi \).
type. For the other two reactions, 9 and 12, the plane of the dislocation is of the \((001)\) type. In the following climb force calculations, the climb force refers to the plane of the product dislocation and is calculated from Equation (11).

[[001]] stress axis

Assume the stress is applied along the AA' direction of the cube in Figure 6, the eight active slip systems are shown below.

<table>
<thead>
<tr>
<th>Slip Plane</th>
<th>Slip Directions</th>
</tr>
</thead>
<tbody>
<tr>
<td>(\alpha)</td>
<td>(OB_\alpha, OD_\alpha; OA_\alpha, OC_\alpha)</td>
</tr>
<tr>
<td>(\beta)</td>
<td>(OB_\beta, OD_\beta; OC_\beta, OA_\beta)</td>
</tr>
<tr>
<td>(\gamma)</td>
<td>(OD_\gamma, OB_\gamma; OC_\gamma, OA_\gamma)</td>
</tr>
<tr>
<td>(\delta)</td>
<td>(OD_\delta, OB_\delta; OA_\delta, OC_\delta)</td>
</tr>
</tbody>
</table>

\(AO_\alpha\) is an active dislocation and Table 6 shows that of the nine reactions given earlier that can result in a \(b = a[100]\) dislocation, 4, 6, 7, 9, 12, and 15 involve the active systems listed above.

Only reactions 6 and 9 contribute dislocations which are aided in climb by the external stress, but they do not experience any glide force in their glide plane. Further, none of the other \(b = a[100]\) dislocations have any force exerted on them by the tensile stress.

[[110]] stress axis

The stress axis is placed along \(AB'\) in the cube. The four active slip systems are shown below.
Slip Plane | Slip Directions
--- | ---
\(\alpha\) | \(OA_{\alpha}, OC_{\alpha}'\)
\(\gamma\) | \(OD_{\gamma}, OB_{\gamma}'\)
\(\epsilon\) | \(OA_{\epsilon}, OC_{\epsilon}'\)
\(\rho\) | \(OD_{\rho}, OB_{\rho}'\)

\(AO_{\alpha}\) is again an active Burgers vector and an inspection of Table 6 shows that reactions 12 and 22 give a \(b = a[[100]]\) resultant dislocation. The reactions are summarized in Table 7. The \(a[[100]]\) Burgers vectors from both reactions are perpendicular to the tensile stress so that no normal stress is exerted on the edge component by the external stress.

[[lll]] stress axis

The stress axis is chosen along \(A'C\). The six active slip systems are--

Slip Plane | Slip Directions
--- | ---
\(\alpha\) | \(OA_{\alpha}, OC_{\alpha}'\); \(OB_{\alpha}, OD_{\alpha}'\)
\(\delta\) | \(OA_{\delta}, OC_{\delta}'\); \(OD_{\delta}, OB_{\delta}'\)
\(\rho\) | \(OD_{\rho}, OB_{\rho}'\); \(OD_{\rho}, OB_{\rho}'\)

Choosing \(AO_{\alpha}\) as an active slip dislocation, Table 6 shows that the reactions 4, 15, 22, and 23 can create \(b = a[[100]]\) dislocations. The reactions are summarized in Table 7.

The [[lll]] orientation is the only orientation for which all the reactions produce a \(b = a[[100]]\) dislocation having a climb force. It also contains (1) the only case where a glide force on the screw component may aid or restrict climb out of the plane of the dislocation,
<table>
<thead>
<tr>
<th>Stress Axis</th>
<th>Reaction</th>
<th>$b_p$</th>
<th>$\xi_p$</th>
<th>Plane of Dislocation ($\frac{b_p \times \xi_p}{|}$)</th>
<th>$\alpha$</th>
<th>$\frac{F_3}{\sigma_{33}^b}$</th>
<th>$\frac{F_3}{\sigma_{33}^b}$</th>
<th>$\frac{F_g(a)}{\sigma_{33}^b}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>001</td>
<td>4</td>
<td>AB</td>
<td>--</td>
<td>$\alpha$</td>
<td>0-180°</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>001</td>
<td>6</td>
<td>AA'</td>
<td>D'B</td>
<td>$\rho$</td>
<td>54°44'</td>
<td>0.816</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>001</td>
<td>7</td>
<td>AB</td>
<td>D'B</td>
<td>$\alpha$</td>
<td>54°44'</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>001</td>
<td>9</td>
<td>AA'</td>
<td>AB</td>
<td>$\rho$</td>
<td>54°44'</td>
<td>0.816</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>001</td>
<td>12</td>
<td>AD</td>
<td>AB</td>
<td>$\rho$</td>
<td>54°44'</td>
<td>0.816</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>001</td>
<td>15</td>
<td>AD</td>
<td>AC'</td>
<td>$\delta$</td>
<td>54°44'</td>
<td>0.816</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>110</td>
<td>12</td>
<td>AD</td>
<td>AB</td>
<td>$\rho$</td>
<td>54°44'</td>
<td>0.816</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>110</td>
<td>22</td>
<td>AD</td>
<td>D'B</td>
<td>$\rho$</td>
<td>54°44'</td>
<td>0.816</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>111</td>
<td>4</td>
<td>AB</td>
<td>--</td>
<td>$\alpha$</td>
<td>0-180°</td>
<td>0.334</td>
<td>0</td>
<td>0.472</td>
</tr>
<tr>
<td>111</td>
<td>15</td>
<td>AD</td>
<td>AC'</td>
<td>$\delta$</td>
<td>54°44'</td>
<td>0.272</td>
<td>0</td>
<td>0.472</td>
</tr>
<tr>
<td>111</td>
<td>22</td>
<td>AD</td>
<td>D'B</td>
<td>$\rho$</td>
<td>54°44'</td>
<td>0.272</td>
<td>0.272</td>
<td>0</td>
</tr>
<tr>
<td>111</td>
<td>23</td>
<td>AB</td>
<td>D'B</td>
<td>$\alpha$</td>
<td>54°44'</td>
<td>0.272</td>
<td>0.272</td>
<td>0</td>
</tr>
</tbody>
</table>

$F_g(a)$ is the glide force in the "glide plane" of the dislocation.
and (2) the cases where there is a glide force in the possible glide plane of the dislocation.

Discussion

The product \(a[[100]]\) dislocation in the \([[011]]\) orientation may move either by climb or glide only through interactions with other dislocations. They would not contribute actively to creep by themselves and in fact may act as a hindrance to the active dislocations. Because of this inactivity they may not multiply or annihilate independent of reactions between or with \(\frac{a}{2}[[111]]\) dislocations.

In the \([[111]]\) orientation the product \(a[[100]]\) dislocations can all climb and possibly glide under the applied stress. This may make them a more important factor in the creep structure since they may now multiply by climb or glide independent of the reactions between \(\frac{a}{2}[[111]]\) dislocations.

The product dislocations in the \([[001]]\) orientation may multiply as Bardeen-Herring climb sources, but would be inactive in glide.

Burgers Vector and Slip Plane Notation for \(((112))[[111]]\) Slip Systems

The use of a lettered geometrical construction becomes quite unwieldy when extended to \(((112))[[111]]\) slip systems. When studying specific reactions it is probably easier to go to a stereographic projection to get the desired geometrical information. However the generalized system is still quite useful for cataloguing the types of reactions and geometries one can expect for a given set of active slip systems, and, as will be pointed out later, Birth's \((120)\) rule for dissociation of dislocations can be easily adapted to this notation.
The simplest way to proceed with the cubic figure introduced earlier is to add the $[[112]]$ directions and notation as shown in Figure 6. The $(112)$ planes themselves are not represented, but they are visualized as being normal to the $[[112]]$ directions. Note that all the $[[112]]$ directions lie in a $(110)$ type plane. The symbol for a $(112)$ plane will be two letters enclosed in parentheses, e.g., $(1s)$, one of the pair is upper case and one is lower case and they must either be adjacent letters in the alphabet or be the same letter with one primed and one unprimed.

The whole notation system becomes more complex when $[[112]]$ directions are added to the cube and there is often more than one way to represent some types of vectors. However, the letter pairs representing the vectors or planes do fall into certain general patterns so that instead of listing specifically all the members of a family it is possible to adopt a general notation and represent all the vectors or planes of a given type by giving the patterns in their notation. This will become clearer as the general rule is explained.

The generalized notation will be written in letter pairs from the general alphabetical sequences FQRST or P'Q'R'S'T' (called the P system), which will represent specific pairs of letters from the notation in Figure 6, i.e., from the sequences ABCDAB ... or A'B'C'D'A'B' ... (called the A system). The nearest neighbor, next-nearest neighbor, and sequential relationship between the letters in the P system are significant and must be preserved when substituting in letters for specific vectors or planes from the A system. Lower case letters in the P system represent lower case letters in the A system and the
nearest neighbor, next-nearest neighbor, and sequential relationship between the lower case and upper case letters must be preserved in going to specific notation. The letters within a pair of letters may be interchanged (reversing the direction of the vector) but all other relationships must remain unchanged. For example, the general pattern $P'_1$ represents the specific vectors $A'b'$, $Bc'$, $Da'$, $a'D$, $d'C$, etc., and $R't$ represents the specific vectors $A'c$, $B'd$, $C'a$, $dB'$, etc. The letter $G$ will be used to denote a Greek letter. If necessary the letters $X$ and $x$ will denote random letters.

Most vectors occur twice on the cube. By using only the bottom corners and the $A'$ and $B'$ corners of the cube and the $G$ planes one can represent all the vectors in unprimed and partially primed pairs of letters. Using the top corners and the $D$ and $C$ corners along with the $G'$ planes, one obtains a set of partially and completely primed pairs of letters. Throughout this discussion only the bottom half and the back half of the cube (as drawn in Figure 6) containing the $G$ planes will be used.

Table 8 gives the general relations between the notation and type of vector for all the vectors drawn on the cube. Other vectors such as $\frac{a}{2}[[110]]$ have not been included since this would add complexity to an already complex situation. If these vectors are required for the study of additional dislocation reactions one could represent an $\frac{a}{2}[[110]]$ as $RS/0$ (where $RS/0$ is the midpoint of the $RS$ vector) and an $\frac{a}{2}[[100]]$ as $\frac{1}{2}RS$, or one might insert upper case Greek letters at the midpoints of the cube edges. An alternative method would be to shrink the cube so that the edges are of a length $\frac{a}{2}$ instead of $a$ and all the
vectors in Table 8 are shortened by half. Many of the dislocation dissociations suggested in the literature\(^{(121-128)}\) could then be handled by modifying the present notation.

**TABLE 8**

**GENERAL NOTATION OF THE VECTORS REPRESENTED ON THE CUBE**

<table>
<thead>
<tr>
<th>Notation</th>
<th>Lattice Vector</th>
<th>Notation</th>
<th>Lattice Vector</th>
</tr>
</thead>
<tbody>
<tr>
<td>RO</td>
<td>(\frac{a}{2}(111))</td>
<td>RG</td>
<td>(\frac{a}{4}(112))</td>
</tr>
<tr>
<td>R'O</td>
<td>(\frac{a}{2}(111))</td>
<td>R'G</td>
<td>(\frac{a}{4}(112))</td>
</tr>
<tr>
<td>RS</td>
<td>(a(100))</td>
<td>R'G'</td>
<td>(\frac{a}{12}(112))</td>
</tr>
<tr>
<td>RR'</td>
<td>(a(100))</td>
<td>Gr</td>
<td>(\frac{a}{12}(112))</td>
</tr>
<tr>
<td>R'S'</td>
<td>(a(110))</td>
<td>G'r'</td>
<td>(\frac{a}{12}(112))</td>
</tr>
<tr>
<td>RT</td>
<td>(a(110))</td>
<td>Gr</td>
<td>(\frac{a}{12}(112))</td>
</tr>
<tr>
<td>R'S</td>
<td>(a(110))</td>
<td>R'r</td>
<td>(\frac{a}{3}(111))</td>
</tr>
<tr>
<td>Rs</td>
<td>(\frac{a}{3}(112))</td>
<td>r'0</td>
<td>(\frac{a}{6}(111))</td>
</tr>
<tr>
<td>R'b</td>
<td>(\frac{a}{3}(112))</td>
<td>r'O</td>
<td>(\frac{a}{6}(111))</td>
</tr>
<tr>
<td>R't'</td>
<td>(\frac{a}{3}(112))</td>
<td>G'O</td>
<td>(\frac{a}{4}(110))</td>
</tr>
</tbody>
</table>

**Dislocation Reactions**

Before looking at specific reactions, several geometrical relations are worth noting:

1. Each \((112)\) plane contains only one \([111]\) direction. Therefore a \((112)\) plane, e.g., \((Sr)\), contains only one vector RO. The lower case letter in the plane notation identifies the upper case letter in the 0 vector.
2. There are three \((112)\) planes in a \([111]\) zone. An \(\mathbf{R}_0\) vector lies in three \((112)\) planes \((Qr), (Sr), (R'r)\).

3. If a \([112]\) direction, \(\mathbf{R}_p\), is normal to the stress axis, the slip plane is inactive since it has zero resolved shear stress.

4. If an \(\mathbf{R}_0\) vector is normal or parallel to the stress axis all \((112)\) planes in its zone are inactive, i.e., have zero resolved shear stress.

5. A dislocation of a given \(\mathbf{R}_0\) vector will create kink-kink intersections only when cutting dislocations in the \((112)\) planes of its zone and will create jog-jog intersections when cutting dislocations lying in any of the other \((112)\) planes. The magnitude of the jog component will depend on the slip planes involved.

Since each \((112)\) glide plane has only one \([111]\) Burgers vector the possible reactions are easily identified. The reactions between pairs of \(\frac{\mathbf{a}}{2}[111]\) glide dislocations would, of course, result either in \(\mathbf{a}[100]\) or \(\mathbf{a}[110]\) dislocations if they combined. In the absence of combination, long-range attractive or repulsive forces would originate from elastic interactions depending on the relative sign of the dislocations and short-range forces would originate from jog or kink formation when the dislocations cut through each other. The sign of the jog created in a dislocation by intersection is important in modifying the dislocation's mobility. Larger jogs would be expected to exert a greater drag on a dislocation while kinks cause no drag at all.

Table 9 lists the relative length of jog normal to the glide plane for all \((112)][111]\) glide dislocations intersected by a \((\bar{2}11)[111]\) dislocation. Both the letter notation and Miller indices are listed.
TABLE 9
SUMMARY OF THE REACTIONS BETWEEN DISLOCATIONS OF THE ((112))[(111)] SLIP SYSTEMS AND THE NORMAL FORCES ON a[[100]] PRODUCT DISLOCATIONS

The intersecting dislocation is \( b = \frac{a}{2}[111] \) on the (211) glide plane, (aD)AO

<table>
<thead>
<tr>
<th>Intersected Dislocation</th>
<th>Glide Plane</th>
<th>Burgers Vector</th>
<th>( \xi_p )</th>
<th>( b_p )</th>
<th>Plane of the Product Dislocation ( (b_p \times \xi_p) )</th>
<th>Normal and Glide Forces on the Product Dislocations with Stress Axis Parallel to [011]</th>
</tr>
</thead>
<tbody>
<tr>
<td>(211)(dA)</td>
<td>(111) DO</td>
<td>4</td>
<td>4</td>
<td>[011]</td>
<td>a[100] AD</td>
<td>(011) 90.0° 0 0 0 0</td>
</tr>
<tr>
<td>(121)(dC)</td>
<td>&quot;</td>
<td>2</td>
<td>4</td>
<td>[315]</td>
<td>&quot;</td>
<td>(015) 59.5° 0 0 0 --</td>
</tr>
<tr>
<td>(112)(dD')</td>
<td>&quot;</td>
<td>2</td>
<td>4</td>
<td>[351]</td>
<td>&quot;</td>
<td>(015) 59.5° 0 0 0 --</td>
</tr>
<tr>
<td>(112)(cC')(Aa')</td>
<td>(111) CO</td>
<td>4</td>
<td>2</td>
<td>[153]</td>
<td>a[001] AA'</td>
<td>(510) 59.5° 0.431 0.249 --</td>
</tr>
<tr>
<td>(121)(cD)</td>
<td>&quot;</td>
<td>2</td>
<td>2</td>
<td>[113]</td>
<td>&quot;</td>
<td>(110) 25.2° 0.213 0.320 0.354 --</td>
</tr>
<tr>
<td>(211)(cB)</td>
<td>&quot;</td>
<td>2</td>
<td>2</td>
<td>[120]</td>
<td>&quot;</td>
<td>(210) 90.0° 0.500 0 0 --</td>
</tr>
<tr>
<td>(121)(bA)</td>
<td>(111) BO</td>
<td>4</td>
<td>2</td>
<td>[135]</td>
<td>a[010] AB</td>
<td>(501) 59.5° 0.431 0.249 --</td>
</tr>
<tr>
<td>(112)(bB')</td>
<td>&quot;</td>
<td>2</td>
<td>2</td>
<td>[131]</td>
<td>&quot;</td>
<td>(101) 25.2° 0.213 0.320 0.354 --</td>
</tr>
<tr>
<td>(211)(bC)</td>
<td>&quot;</td>
<td>2</td>
<td>2</td>
<td>[102]</td>
<td>&quot;</td>
<td>(201) 90.0° 0.500 0 0 --</td>
</tr>
<tr>
<td>Intersected Dislocation</td>
<td>Relative Magnitude of Jog</td>
<td>Plane of the Product Dislocation</td>
<td>Normal and Glide Forces on the Product Dislocations with Stress Axis Parallel to ([011])</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>-------------------------</td>
<td>--------------------------</td>
<td>----------------------------------</td>
<td>---------------------------------</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Glide Plane</td>
<td>Burgers Vector</td>
<td>(\xi_p)</td>
<td>(\frac{b_p}{b})</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Intersected</td>
<td>Intersecting</td>
<td>Edge (\alpha) (F_3)</td>
<td>Screw (\sigma_{33}^b) (F_3) (F_g)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Dislocation</td>
<td>Dislocation</td>
<td>(\sigma_{33}^b) (\sigma_{33}^b) (\sigma_{33}^b)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>((\bar{2}1\bar{1})(aD))</td>
<td>([11\bar{1}]) (A^0)</td>
<td>0 0 --</td>
<td>(\frac{a}{2\sqrt{6}}) times the relative magnitude.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>((1\bar{1}\bar{2})(aA'))</td>
<td>&quot;</td>
<td>0 0 ([11\bar{1}])</td>
<td>(\frac{a}{2\sqrt{6}}) times the relative magnitude.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>((1\bar{2}\bar{1})(aB))</td>
<td>&quot;</td>
<td>0 0 ([11\bar{1}])</td>
<td>(\frac{a}{2\sqrt{6}}) times the relative magnitude.</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

(a) The actual length of jog normal to the glide plane of the dislocation is \(\frac{a}{2\sqrt{6}}\) times the relative magnitude.

(b) The line direction of the product dislocation. This corresponds to the line of intersection of the glide planes of the reacting dislocations.

(c) Burgers vector of the product dislocation.

(d) \(\alpha\) is the angle between \(b\) and \(\xi\).
There is a definite relation between the letter notation and the reaction. All systems are reacted with the system \((aD)A0\). Then one finds that all intersections with \(DO\) dislocations, on \((d_\perp)\) slip planes create a full jog \((2\sqrt{2})\) on the intersecting dislocation, intersections with \(AO\) dislocations create no jog, and with \(BO\) and \(CO\) dislocations, half jogs \((\sqrt{2})\) are created. On the intersected dislocation, full jogs are created on dislocations on \((A)\) planes, no jogs on dislocations in \((a)\) planes and half jogs on dislocations in the remaining planes. The line of intersection of the glide planes follows a similar pattern. A model of the cube in Figure 6 allows one to quickly identify the letters in the blank spaces.

Individual reactions leading to \(a[100]\) or \(a[110]\) dislocations are, of course, possible for each pair of slip plane and slip directions by changing the sign of one of the reacting dislocations. Only the reactions resulting in \(a[100]\) dislocations will be considered. In Table 9 one of the \((211)[111]\) slip systems active in the \([011]\) orientation is reacted with all the other \((211)[111]\) slip systems. The predominant reaction will be with the other active slip vector, \(\frac{a}{2}[\overline{1}11]\). All the other slip systems should be relatively inactive.

Only a few of the product \(a[100]\) dislocations lie on \((110)\) planes in which they might have a reasonable glide mobility. The other product dislocations lie on higher index planes and would be expected to be relatively immobile. The \(a[100]\) dislocations formed by the reaction of the active slip vectors in the \([011]\) orientation again have no normal forces imposed on them by the applied stress. Hence they are dependent on interactions with other dislocations in order to move by
climb or glide. For \((112)(111)\) slip systems as in \((110)(111)\) slip systems the \([100]\) dislocations probably act as a hindrance to the motion of the active dislocations.

**Dissociation of Dislocations**

A potentially important mode of dissociation of screw dislocations in bcc crystals is the reaction:

\[
\frac{a}{2} [111] = \frac{a}{6} [111] + \frac{a}{6} [111] + \frac{a}{6} [111] .
\]  

(12)

Each of these partials moves out on one of the \((112)\) planes in the zone of the Burgers vector. The direction which they move from the core of the screw dislocation may be important in creating an anisotropy in the critical resolved shear stress.\(^{128,129}\)

The rule for dissociation presented by Hirth\(^{119}\) can be easily adapted to the cube notation described in a previous section. The rule is repeated below modified slightly to accommodate the notation in Figure 6.

"Choose the sense vector \(\xi\) to point out of the cube along an \(0\) vector, and adopt the convention that \(b\) is coincident with \(\xi\) for a right-handed screw. Then if \(b\) points out of the cube, the intrinsic fault is produced by moving a right-handed screw in the direction

\[
s = Rs \times OS = Rp \times b
\]

(13)

a left-handed screw in the \(-s\) direction."
EXPERIMENTAL PROCEDURES

Materials

The starting material was arc-melted Climelt (R) Mo received as 1/4-inch diameter bar stock from the Climax Molybdenum Company. The producer gives an analysis for the stock of (in weight percent) > 99.9 Mo, 0.018 C, < 0.0004 O₂, < 0.0001 H₂, < 0.0001 N₂, < 0.001 Fe, < 0.001 Ni, < 0.001 Si.

This material was given a purification treatment for removal of carbon which consisted of two steps: (1) annealing 2 to 4 hours at 2100°C in a flowing wet hydrogen atmosphere (dew point greater than 21°C), followed by (2) a 2 to 4 hour anneal at 2100°C in a flowing dry hydrogen atmosphere (dew point -23° to -36°C). This procedure decreased the carbon to a range of 3 to 15 ppm carbon with most analyses giving 4 to 8 ppm carbon, 3 to 7 ppm O₂, and < 1 ppm N₂. The micro-combustion method was used for the carbon analysis and the vacuum fusion method was used for oxygen and nitrogen. The particular hydrogen anneal procedure outlined above is not critical to achieve this level of purification, and it was found that after only an hour or so at lower temperatures and at dew points less extreme than those listed above, the analysis for carbon and oxygen did not vary from the range given above.
Crystal Growing

Single crystals were grown in an electron beam zone melting apparatus. Since purification was not a factor (because of the hydrogen annealing treatments), the crystals were grown in vacuums of $1 \times 10^{-4}$ to $1 \times 10^{-5}$ torr. A slow traverse rate of 2 mm/min was chosen to minimize the grown-in dislocation density (15).

The single crystals were seeded and grown from the purified bar stock. It was not necessary to purify the stock first in order to seed crystals having a $[[100]]$ crystallographic direction along the rod axis. However, in order to successfully seed and grow crystals with a $[[111]]$ or $[[110]]$ axis the carbon had to be decreased to a low level. The critical composition was not determined, but the only discernible difference between stock which regularly yielded long $[[110]]$ and $[[111]]$ crystals and that which did not was a carbon content of 3 to 15 ppm and about 180 ppm respectively.

Preparation of Creep Specimens

The creep tensile specimens were shaped from the single crystal rods by electrical spark-discharge machining on a Servomet spark machine. Two to four tensile specimens were made from each as-grown crystal by latheing with a tungsten tool. The rod was held gently in a rotating chuck and a contoured tool was passed by the rod until it assumed the shape of the tool. The tool did not touch the specimen. Initial roughing cuts were made on the Range 1 setting (high energy spark discharge) and the spark energy was decreased as the final gage diameter was approached until the finishing cut was made on Range 4.
The damaged layer was probably less than 0.002 inch thick (see Appendix B).

If single crystals having the high carbon content of the as-received material (180 ppm) were machined, the surface was severely cracked by the spark discharge even at low energy settings. Raising the bath temperature to 120°C did not decrease this tendency for cracking. However, crystals grown from the purified stock with low levels of carbon were far less prone to cracking even when machined at room temperature. It appears that the carbide particles in the higher carbon crystals serve as crack nucleation sites and enhance the tendency to form surface cracks.

After machining, approximately 5 to 10 mils were removed from the specimen diameter by electropolishing in a solution of 1 part H$_2$SO$_4$ : 6 parts ethanol by volume.

The polished specimens were given a final anneal at 2100°C for four hours in a flowing dry hydrogen atmosphere to remove any deformation introduced in handling and to provide a uniform pre-creep condition. The analysis for carbon and oxygen after this final hydrogen anneal remained in the range quoted earlier after the first hydrogen anneal.

**Equipment**

**Furnace**

Creep tests were performed in a high temperature vacuum furnace capable of 2000°C and $3 \times 10^{-5}$ to $5 \times 10^{-6}$ torr vacuum at temperature. It had a cylindrical tantalum heating element and was surrounded on the
top, bottom, and sides by molybdenum radiation shields. These are shown in Figure 8. The temperature gradient over the specimen gage length was adjusted by raising and lowering the top and bottom radiation shields and by tapering the element itself.

The temperature was measured by mounting a thermocouple bead in a small indentation in the side of the bottom grip. This ensured that it was in exactly the same position each time. The specimen temperature was calibrated to this thermocouple in the following manner: A dummy specimen identical to the actual creep specimens was made with small indentations in the center and at each end of the gage length just below the shoulder. After mounting in the furnace thermocouples were fastened at these points. Those at the end of the gage length had to pass through holes in the grips in order to make contact. The furnace was then heated to a series of temperatures, waiting one to two hours at each point to reach thermal equilibrium. The temperature gradients were determined and after cooling down the top and bottom baffles were readjusted. This procedure was continued until the lowest possible gradient was achieved. For given baffle positions the gradient was different at different temperatures, necessitating some calibration over different temperature ranges. The center of the specimen was invariably hotter than the ends of the specimen even when shielded by several layers of tantalum sheet. In the lowest gradient obtainable the center was 2° to 4°C hotter than the ends giving an overall gradient of ± 2°C.
FIGURE 8. SCHEMATIC OF THE LOADING TRAIN

A. Load Cell
B. Specimen
C. Pulley on knife-edge
D. Contoured lever arm and pulley on knife-edge
E. Weight and weight pan
F. Fingers for placing and removing weights
G. Driving system for raising and lowering weights through a sliding seal
H. Heating elements and baffles
I. To vacuum pump
The specimens tested in this gradient deformed uniformly over the whole gage length up to 45% tensile strain. This implies a small gradient since the creep rate in molybdenum is very temperature dependent, small temperature differences could conceivably cause nonuniform deformation along the specimen gage length. When the apparent gradient was improved to ± 0.5°C by extensively baffling the exposed gage length from the heating element, the specimens were invariably necked down at the ends of the gage length, indicating "hot spots" at these points. These results suggest that the apparent temperature gradient of ± 1° to 2°C was probably better than this due to some fixed measurement error in either the center or end thermocouples giving an apparent temperature difference of 2° to 4°C from the actual temperature.

The temperature control cycle was adjusted to ± 0.2°C at best. However for given control settings this, too, varied over the test temperature range, causing the usual control cycle to be ± 0.5°C.

In the temperature change experiments the time to reach within 0.5°C of the desired temperature varied with the magnitude and direction of the temperature change. For a temperature change of 5°C the time was less than 1/2 minute. For a temperature change of 20°C it was 5 to 10 minutes at the most and was usually less than 5 minutes.

**Loading System**

Constant stress throughout the creep test was maintained with a contoured constant stress lever arm. The lever arm was mounted in a vacuum chamber attached to the creep furnace so that the entire loading system was enclosed within the vacuum. The loading arrangement is
shown in Figure 8. An advantage of this arrangement is that the load is transmitted to the specimen through knife edges, so that at the low required loads no errors are introduced through sliding seals or bearings.

The load was applied smoothly and quickly to the weight pan at a constant rate corresponding to a deformation rate of 0.44 in/min which imposed a maximum initial strain rate in the specimen of 0.44 min⁻¹. The load was recorded continuously during the creep test and confirmed that the stress was constant within ±1%.

Stress changes during a creep test are accomplished by lifting weights off the pan and/or placing them on it by engaging long pegs extending from the sides of the weights in grooves cut in fingers attached to the loading device. By staggering the pegs on successive weights, the load could be changed in various increments during the creep test.

Strain Measurement

The creep strain was measured optically. A 20-power filar microscope with one fixed and one movable cross hair was used to measure the relative motion of two molybdenum rods. The rods were threaded into the faces of the top and bottom grips respectively so that only the specimen elongation between the shoulders was measured. The rods each had a flat face that was grooved at regular intervals and were stained with spots from zirconia powder. Through a careful choice of filters the markers were visible up to at least 1900°C and displacements of 100μ inches could be reproducibly measured.
**Testing Procedure**

The specimen grips were of the split-shell type machined from Mo-TZM alloy (Mo-0.5 Ti-0.1 Zn-0.02 C). Two split halves of the grip were fitted over the buttonhead of the specimen and a pullrod. An open-ended cylindrical sleeve was then slid down over the two split halves and locked them together. For tests above 1200°C it was found necessary to dust the mating surfaces of the grip with zirconia powder. This effectively prevented the surfaces from welding together even after hours under load at 1900°C.

A loading jig was used to hold the pullrod assembly in rigid alignment without stress on the specimen while the assembly was inserted in the furnace. After the assembly was threaded into the load train within the furnace a light aligning load of less than 100 psi was applied and the jig was removed. Several threaded joints in the load train could then be adjusted to bring the lever arm to the initial position indicated on a dial gage monitoring the initial lever arm movements. The dial gage was not in contact with the lever arm during the test. This initial position had to make allowances for the subsequent elastic elongation of the load train when the weight was applied and the thermal elongation of the load train when heated to the test temperature.

After placing the necessary weight on the loading fingers and a baffle of tantalum foil around the specimen the furnace was closed up and pumped down. The specimen was held at temperature for at least two hours to allow the temperature to equilibrate completely. The load was then applied by lowering weights at a steady rate onto the loading pan via the motorized gear train. Strain measurements were made at the
instant the load was completely applied (as seen from the recording chart) and at 20 second intervals thereafter until the creep rate decreased sufficiently to allow readings to be taken at longer intervals. At load-on the strain measuring rods were moving rapidly relative to each other causing the load-on strain reading to be only an approximation. Thus, an accurate initial strain rate could not be determined.

One of the strain measuring rods passed out of view at tensile strains of 43-46% so the tests were terminated at this point if not sooner and the specimens were cooled under load to preserve the creep structure.

**Optical Metallography**

For the etch pitting studies it was necessary to slice the crept specimens parallel to certain crystallographic planes. The specimen was glued to a brass disc using a mixture of 50 volume percent powdered graphite and 50% model airplane dope. The brass disc was mounted on the worktable of the spark machine goniometer and a back reflection Laue pattern was taken of the specimen while it was mounted on the goniometer. At low strains Laue patterns were taken on both shoulders and at the center and ends of the gage length to check the uniformity of deformation, determine the stability of the tensile axis, verify the monocrystallinity of the specimen after creep, and orient the crystal for slicing. At high creep strains the asterism on the patterns made from the deformed crystal was so pronounced that they could not be used for crystal orientation. Then Laue's taken on the
undeformed ends of the specimen were used for orienting the specimen for slicing. This was a reasonable procedure for specimens with a (110) tensile axis because this orientation was stable during tensile deformation.

The specimens were then sliced parallel to the three (100) planes on a Servomet spark machine using Range 4 and 28 mil copper wire for slicing. The cut specimens were carefully mounted in "Kold-weld" such that an electrical contact was made between the specimen and a wire lead entering the back of the mount. This enabled the cut surface to be metallographically prepared and then electropolished in a 1:6 by volume solution of H₂SO₄ and ethanol to remove all mechanical damage before etch pitting. Approximately 5 to 10 mils were removed by electropolishing prior to etch pitting. This was deemed to be sufficient in view of the low dislocation (etch pit) density observed within many of the subgrains formed during creep, and the low dislocation (etch pit) densities, $2 \times 10^6$ cm⁻², in the undeformed buttonhead regions of the specimens. These observations support the contention that the damaged surface was removed prior to etch pitting of the specimens.

After electropolishing, the orientation of the specimen surface was checked by the Laue back reflection method. Except for heavily deformed specimens, there was sufficient information on the diffraction pattern to determine the plane of the etched surface. This orientation was subsequently used in all the trace analyses.
The etch pitting reagent was a dilute Murakami's reagent suggested by Prekel and Lawley\(^\text{(130)}\). Its composition is:

\[
\begin{align*}
7 \text{ gm NaOH} \\
10 \text{ gm } K_3Fe(CN)_6 \\
100 \text{ cc } H_2O
\end{align*}
\]

diluted 30:1 with water. At room temperature an etching time of 30 to 45 seconds was required.

**Preparation of Foils for Transmission Electron Microscopy**

The creep specimens which were to have foils for transmission electron microscopy prepared from them were brought through to the slicing operation by the same procedure used for the optical metallography specimens. However for foils, sections 0.020 inch thick were cut from the creep specimens parallel to the desired crystallographic plane. The sections were cut with 28 mil copper wire on Range 4 of the spark machine. Since the damaged layer should extend less than 5 mils from the cut surface, the center of the disc should be free of damage. This was verified by examining as-grown single crystals prepared in the same manner and by etch pitting as mentioned earlier. The dislocation density was quite low in both studies. In addition most of the few dislocations observed in the foils had small precipitates on them, indicating they were grown-in dislocations and that no dislocations were introduced by the cutting and etching techniques.

The 20 mil thick slices are mounted on a brass disc with the airplane dope-graphite mixture and discs of 2.3 mm diameter (required by the specimen holder in the electron microscope) were spark-trepanned
from the slices. The light grooves left by wire cutting were removed from each face of the discs on the spark machine by passing a tungsten tool over the faces, using Range 6.

The discs were cleaned in acetone and jet indented in the arrangement shown in Figure 9 with a solution of 15% HNO₃ in H₂O, using 35 to 40 volts at a current of 90-100 ma. They were indented from each side, stopping just short of perforation, leaving a web about 3 mils thick in the center. The indented disc was mounted in a stainless steel tweezer which had had its jaws bent to receive the disc and exert a light pressure required to maintain electrical contact. The tweezers were then painted with a chemical resistant insulating coating (Lacomit) to prevent polishing of the covered areas. The mounted specimen was electrolytically thinned in an arrangement suggested by Stickler and Engler (131) shown schematically in Figure 10 in an electrolyte of 1:6 by volume solution of H₂SO₄ and ethanol at room temperature. The polishing voltage was 14 volts during most of the thinning period but was slowly decreased down to about 5 volts just before perforation. This point is guessed at and served to decrease the polishing rate to the point where the hole did not grow appreciably before it was detected and the voltage was cut off. This procedure required some experience to attain a feel for the time involved but small holes of 10-50 µ diameter were achieved, thereby increasing the thinned area available for observation.

Increasing the diameter of the holes in the cathodes shown in Figure 10 caused the outer area of the disc to polish at a faster rate
FIGURE 9. ARRANGEMENT FOR INDENTING THE DISCS BEFORE THINNING
FIGURE 10. ARRANGEMENT FOR THINNING INDENTED SPECIMENS
The specimen is held between the holes in the two cathodes on the line of the light source and microscope.
than the thin web in the center because of the higher current concentration around the periphery. This resulted in a very shallow "dish" in the thinned specimen and a relatively large thinned area in the center when a hole did appear.

After thinning the specimen was immediately rinsed through three rinses of methyl alcohol and was subsequently stored in air in a desiccator.
RESULTS

It was originally planned to creep specimens having orientations \([\langle 100\rangle], [\langle 111\rangle],\) and \([\langle 110\rangle]\) to study the influence of the number of active slip systems and various types of dislocation interactions on the dislocation substructures developed during creep. However, in the initial stages of the investigation several characteristics became evident in crystals having a \([\langle 100\rangle]\) or \([\langle 111\rangle]\) orientation that made them undesirable for this study. These characteristics are presented in the next section. Consequently, the studies of creep properties and creep structures were conducted only on crystals having the \([011]\) orientation.

Mode of Creep Deformation

Rotation of tensile axis

It was known that a tensile axis parallel to the \([\langle 100\rangle]\) and \([\langle 111\rangle]\) directions is in a metastable position during tensile deformation. If the stress remains directed along the \([\langle 100\rangle]\) and \([\langle 111\rangle]\) directions, the favored slip systems are symmetrically disposed around the stress axis, have an equal resolved shear stress, and would be expected to slip equal amounts. This would not cause any net change in crystal orientation and the tensile axis would remain parallel to its original crystallographic direction. However any small deviation of the stress axis from the \([\langle 100\rangle]\) or \([\langle 111\rangle]\) directions immediately
raises the resolved shear stress on some of the operating systems and decreases it on others. Further slip on the favored system(s) continuously raises the resolved shear stress on it so that very quickly it can become the primary slip system. This results in a progressive movement of the tensile axis from its initial orientation towards the active slip direction or along a path representative of the average influence of all contributing slip directions.

It was thought that this instability might not occur since Keh\textsuperscript{132} observed that the tensile axis did not deviate from the [[100]] direction and deviated less than 2° from the [[111]] direction when iron crystals were tested at 298°C up to a tensile strain of 12%. However the instability did appear in this investigation and introduced two undesirable effects. The rotation of the stress axis caused the resolved shear stress on the slip systems to change during creep so that constant shear stress could not be maintained. A more severe limitation was that the deformation was usually not uniform along the gage length. Evidently one slip system would operate more abundantly locally causing a local deviation of the stress axis. Once this happened geometrical softening set in and the same system continued to operate almost exclusively. Since this could happen anywhere along the gage length several necked-down regions would form and eventually a wedge-type fracture would occur in one of them. Figure 11 shows the nonuniform deformation and wedge-type fractures representative of the [[111]] and [[100]] orientations, while Figure 12 shows the uniform deformation observed in the [[110]] crystals. [[110]] crystals also failed by a wedge-type fracture.
FIGURE 11. [[111]] ORIENTED CRYSTAL TESTED AT 3900 psi AND 1270 C.

FIGURE 12. [[011]] ORIENTED CRYSTAL TESTED AT 5750 psi AND 1254 C.
The [[110]] orientation is stable during tensile deformation. Any deviation of the stress axis from the [[110]] lowers the resolved shear stress on the slip system(s) responsible for rotating the stress axis off the [[110]] and raises it on the system(s) which would rotate the stress axis back to [[110]]. Hence in the tests of the [[110]] orientation all the slip systems maintained their initial alignment with the stress axis during creep and constant shear stress could be maintained with the contoured lever arm.

The directions of rotation of the tensile axis from the initial [[100]] and [[111]] tensile orientations are shown in Figure 13. The orientations were determined after creep by the Laue back reflection method, taking patterns from the buttonhead and at various points along the gage length. The arrows indicate the direction of rotation but since the deformation was not uniform along the gage length, the amount of rotation cannot be related to the relative shear strain contributions of any of the slip systems. The direction of rotation of the [001] tensile axis indicates that this orientation deformed primarily by slip on the (112)[111] system and not by ((110))[[111]] slip. In the latter instance the tensile axis would have moved towards the [011] direction. Figure 14 is an (001) stereographic projection which will be referred to when specific crystallographic planes or directions are mentioned. The movement of the [111] tensile axis towards [011] is consistent with the operation of the (211)[111] slip system, but the same rotation could also be produced by favored ((110))[[111]] systems, e.g., (110) and (101)[111].
FIGURE 13. ROTATION OF THE TENSILE AXIS DURING CREEP

Crystal 38-1 was tested at 1350 °C and 3800 psi.
Crystal 63-4 was tested at 1190 °C and 4250 psi.
Crystal 64-2 was tested at 1250 °C and 5050 psi.
FIGURE 14. (001) STEREOGRAPHIC PROJECTION

The poles discussed most often in the text have solid symbols. The tensile axes are parallel to [001], [111], and [011].
Surface slip band observations

The schematic drawing in Figure 15 shows the general appearance of the slip bands formed on a crystal of the [011] orientation during creep. The offsets of the slip bands indicate that only the [111] and [111] slip directions are operative. No slip bands having offsets corresponding to the [111] or [111] directions perpendicular to the tensile axis were observed, although they would have been easily detected on the [011] side of the crystals if they had operated extensively. In addition there was no dimensional change parallel to [011] with \( \pm 1\% \) of the original diameter. All dimensional changes were parallel to [100].

The slip band traces on the (011) side of the crystals were parallel to the favored ((112)) and ((110)) slip planes, especially at low creep strains. The traces of these systems are parallel on this surface at 35° to the tensile axis and cannot be differentiated. On the (100) face the primary ((112)) slip traces are at 90° and the primary ((110)) slip traces are at 45° to the tensile axis. However the actual slip band traces were wavy and had segments parallel to both ((110) and ((112)) slip planes. Therefore they did not resolve the question of which slip planes were operating. At high creep strains the coarse slip bands tended towards an angle of 45° to the tensile axis in the (011) view and 90° to the tensile axis in the (100) view.

More detailed observations of slip bands on [011] crystals are presented later.

The slip bands on the [111] and [001] crystals resembled those of the [[110]] orientation. It appears that for the [111] orientation,
FIGURE 15. SCHEMATIC DRAWING OF THE SLIP BANDS FORMED DURING CREEP

The slip bands in the (100) view should be wavy.
for example, either one ((112))[[111]] system, e.g., the (211)[111] system, or a pair of ((110))[[111]] systems, e.g., [111] on (110) and (101), predominated under the influence of geometrical softening. This gave them the general appearance of one set of slip bands in Figure 15 for small strains. However, after the tensile axis rotated sufficiently close to [011] the other system(s), i.e., (211)[111], or (110) and (101)[111], began to operate and they subsequently operated simultaneously. This was particularly true in the vicinity of the wedge fracture where the strain was quite high and a back reflection Laue pattern taken near the fracture of Figure 11 showed that the local stress axis was within 8° of [011].

Laue X-ray observations

Asterism in back reflection Laue patterns can also provide information regarding the operative slip systems. The asterism is an elongation or streaking of the diffraction spots due to bending of the reflecting lattice planes. The bending axis, and the axis of the asterism, is the crystallographic direction lying in the glide plane perpendicular to the slip direction. By determining the crystallographic direction of the asterism and knowing the slip direction the slip plane can be deduced.

For the [011] orientation the Laue spots showed relatively little broadening after 1 and 2% creep strain at 900 psi and 1650°C. After 5% strain the Laue spots had broadened into an arc of about 2.5° around the [011] direction and had broken up into small individual spots. Neighboring "sub-spots" within the major Laue spots were less
than a half a degree apart. After 22\% strain the asterism had spread over an arc of 5° to 8° around the [0\overline{1}1] direction with very little broadening in any other direction and the greater elongation of the spots made it difficult to identify the individual diffraction spots in some instances. Neighboring sub-spots within an elongated diffraction spot were now up to a degree apart. At 42\% creep strain little had changed except for a somewhat greater elongation of the diffraction spots.

The Laue patterns in Figure 16 illustrate the asterism caused by large creep strains, clearly showing the [0\overline{1}1] axis of the asterism. Laue patterns taken at different points along the gage length showed that the crystal had rotated locally up to ± 5° from the original orientation (compared to the unstrained buttonhead) as can be seen in Figure 16 by comparing the patterns having the X-ray beam directed along [0\overline{1}1].

The diffraction spots from crystals tested at high stresses at 1150° to 1200°C had a greater tendency for streaking rather than for breaking up into spots. This suggests either that the subgrains were too fine to give resolvable "sub-spots" or that recovery by polygonization and subgrain formation was unable to keep abreast of the deformation rate.

The asterism of the diffraction spots from crept crystals of the [[100]] or [[111]] orientations was quite generally around the [[110]] direction normal to the slip directions. That is, the crystals usually attained an elliptical cross-section and the major axis of the ellipse was parallel to the [[110]] direction corresponding to the axis
FIGURE 16. ASTERISM IN LAUE BACK REFLECTION SPOTS
FROM A CREPT CRYSTAL

The crystal was tested at 1569 C at 850 psi to 36% strain.
of the asterism. The edge of the wedge-type fractures were also parallel to this [[110]] direction. Infrequently the diffraction spots also had short streaks around some other axis but they were too short to identify the axis. There was little broadening of the spots normal to the axis of the asterism.

The repeated observation that the asterism occurs around a [[110]] direction indicates that during creep slip occurs (1) on the ((112))[[111]] system favored by the maximum resolved shear stress or (2) by alternate cross-slip on a pair of ((110)) slip planes in their common [[111]] direction on such a fine scale that the average slip plane within the dimensions of the X-ray beam was a ((112)) plane or (3) by a combination of (1) and (2).

Creep Properties

The tensile creep properties were studied on crystals of the [011] orientation. Tensile stresses, and tensile strains rather than shear strains are used throughout the paper because of the uncertainty of the operative glide planes. In addition the deformation is so inhomogeneous that an average shear strain would not be any more meaningful than tensile strain.

Creep curves

A notable feature of the creep behavior of the molybdenum single crystals tested here was the absence of "steady state" creep up to at least 45% tensile strain. Steady state creep is commonly reported in materials crept at temperatures above one-half the melting temperature
(0.5 T_m). Figure 17 shows the creep curves plotted as true strain vs. time on linear coordinates. Figure 18 shows the creep curves plotted on a log-log plot. One could easily make the error of ascribing a constant creep rate to the late portions of the creep curves in Figure 17. However, Figure 19 shows that even in high creep strains the creep rate is still slowly decreasing but is approaching a constant creep rate. Steady state creep was never observed in a single crystal showing uniform strain within the gage length over the whole range of temperature and stress covered in this program. Several crystals that did show an apparent steady state had necked down at both ends of the gage length. This was apparently caused by a nonuniform temperature gradient with high temperatures at the ends of the gage length. The uniformity of the strain over the gage length in the other specimens implies that the measured temperature gradient was close to the true gradient.

The creep strains were quite difficult to measure accurately within the first several minutes, depending on the overall rate of the creep test. This part of the creep curve cannot be analyzed quantitatively to determine the load on strain or the initial creep rates. However several interesting facts were noted. During loading there often was a small inflection in the load-time curve at a large fraction of the load, although the load was being applied at a constant deflection rate. In some of the tests, immediately after load-on the creep rate was very high for the first few seconds followed by a rapid slow-down during the first 1 to 2 minutes after the test was begun (between 0.01 to 0.02 creep strain). The creep rate then decreased smoothly
FIGURE 17. TYPICAL CREEP CURVES FOR MOLYBDENUM SINGLE CRYSTALS OF THE [011] ORIENTATION
FIGURE 18. TYPICAL CREEP CURVES PLOTTED LOGARITHMICALLY

The experimental $\epsilon_o$ is subtracted from the total creep strain.
FIGURE 19. STRAIN DEPENDENCE OF THE CREEP RATE
with creep strain. The extremes in the creep rate at very low creep strains in Figure 19 illustrates this. The high initial creep rates represent a test which could be fit smoothly without a break from the beginning of the test. The lower creep rate is from a test which had an inflection at one minute. At higher creep strains the curves are identical. These effects could not be examined in detail and are not well documented because of the difficulty of following the creep strain accurately at low creep strains where the creep rates could be quite large. For this reason the above observations are mentioned only for the sake of completeness and are not intended to be conclusive observations.

The strain dependence of the creep rate is quite dependent on both stress and temperature. An example of the influence of temperature on the strain dependence of the creep rate is shown in Figure 20 for low creep strains and the time dependence of the creep rate is shown in Figure 21 for these same specimens. The time law is parabolic, 
\[(\varepsilon - \varepsilon_0) \propto t^m, \dot{\varepsilon} \propto t^{m-1}\]
only over small regions of the creep curve, if at all. Although one cannot compare the strain rate dependence over the same range of strain early in primary creep, there is a common region between about 0.02 and 0.05 true strain where the time dependence of the creep rate can be approximated by a straight line in Figure 21. If one uses a parabolic time law as a crude comparison of the influence of temperature on primary creep behavior one sees that \(m\) increases from \(m = 0.37\) at 1688°C to \(m = 0.7\) at 1917°C. If one uses the method of de Lacombe\(^{(133)}\) to fit the early portions of the creep curve to a
FIGURE 20. STRAIN DEPENDENCE OF THE PRIMARY CREEP RATE
FIGURE 21. TIME DEPENDENCE OF THE PRIMARY CREEP RATE

The slope of the lines is m-1.
parabolic time law the agreement with the m values in Figure 21 is good as shown in Table 10. Both of these methods give m independent of \( \varepsilon_0 \). The results indicate a larger increase in m between 1751° and 1852°C than in any other interval. This is also evident in Figures 20 and 21.

**TABLE 10**

VALUES OF m AND \( \varepsilon_0 \)

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Temperature °C</th>
<th>m</th>
<th>Graphical</th>
<th>deLacombe</th>
<th>( \varepsilon_0 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>93-4</td>
<td>1550</td>
<td>--</td>
<td>0.47</td>
<td></td>
<td>0.003</td>
</tr>
<tr>
<td>93-1</td>
<td>1588</td>
<td>0.37</td>
<td>0.48</td>
<td>--</td>
<td>0.012</td>
</tr>
<tr>
<td>90-4</td>
<td>1751</td>
<td>0.45</td>
<td>0.47</td>
<td>0.012</td>
<td>0.012</td>
</tr>
<tr>
<td>93-2</td>
<td>1852</td>
<td>0.67</td>
<td>0.69</td>
<td>0.011</td>
<td>0.017</td>
</tr>
<tr>
<td>93-3</td>
<td>1917</td>
<td>0.70</td>
<td>0.71</td>
<td>0.016</td>
<td>0.021</td>
</tr>
</tbody>
</table>

(a) This experimentally measured value is an estimate at best and is probably low.

(b) From Crussard’s method (67).

As shown by Crussard (67) one can estimate \( \varepsilon_0 \) by making the slope in a log true strain vs. log time plot in primary creep conform to the m given in Table 10. The strain one must subtract from the total creep strain to accomplish this is \( \varepsilon_0 \). This enables one to determine an apparent \( \varepsilon_0 \) in spite of the difficulty of measuring \( \varepsilon_0 \) exactly.

Table 10 gives the values of the experimental estimate of \( \varepsilon_0 \) and the \( \varepsilon_0 \) obtained from plotting the data according to Crussard’s suggestion. The experimental values of \( \varepsilon_0 \) will be lower than the true values so the
differences are in the proper direction. \( \varepsilon_0 \) increases with increasing temperature as expected.

Whereas Figure 18 shows a log creep strain vs. log time plot using the experimentally estimated values of \( \varepsilon_0 \), Figure 22 shows a log creep strain vs. log \( \theta \) plot where the initial portions of the creep curve were drawn to conform to slopes of \( m \). The \( Q_c \) in \( \theta \) was obtained from creep data discussed in the following section. A different average value of \( m \) was used below 1751°C and above 1852°C. The difference between the low and high temperature tests at low strains is quite evident, although at higher creep strains the curves are the same.

To determine whether the test results were unique to single crystals, several creep tests on polycrystalline specimens were conducted. A coarse-grained, hydrogen-annealed specimen tested at 1800°C and 1400 psi crept just like the single crystals. However two specimens made from the as-received stock tested at 1650°C and 1700 psi entered the third stage of creep (accelerating creep rate) at about 15% strain. From 9 to 15% strain one could measure a constant strain rate but it is more properly described as a minimum creep rate at the inflection of the creep curve.

**Activation energy for creep**

Even though a steady state creep rate was not observed the activation energy (temperature dependence) of creep can be obtained from the data by applying the temperature compensated time concept of Dorn and co-workers\(^{(55)}\). They have shown that for many materials one can superimpose the creep curves obtained at different temperatures but
FIGURE 22. LOG CREEP STRAIN VERSUS LOG $\theta$ WHERE $\theta = t_e \frac{-108,000}{RT}$

The low temperature curves use $m = 0.50$ and the high temperature curves use $m = 0.70$. 
at the same stress, by plotting true strain vs. \( \frac{Q_c}{RT} \) where \( t \) is the actual time, \( T \) is the temperature of the test, \( R \) is the gas constant, and \( Q_c \) is the activation energy for creep. It follows, then, that if one plots the logarithm of the time to reach a given true strain vs. \( \frac{1}{T} \) the slope of the line will be \( \frac{Q_c}{R} \). This was done for the times to reach true strains of 0.1, 0.2, and 0.3. All the lines were parallel, indicating that the creep curves would superimpose throughout the creep test confirming that only the temperature was influencing the creep rates at a given strain. Figure 23 shows the results for the two stresses, 900 and 2050 psi, plotting time to reach 0.2 true strain. The activation energy for creep was found to be 105 to 108 kcal/mol with no discernible stress dependence within the limits of the data. The value of \( Q_c \) lies within the range of activation energies for self diffusion, \( Q_{SD} \), in molybdenum reported in the literature, and summarized in Table 1.

The time, \( t \), is not corrected for the temperature dependence of Young's modulus as suggested by Sherby\(^{(134)}\) since the stress dependence of the creep behavior could not be accurately measured. This is not a serious problem since the modulus correction often causes only a minor decrease in the uncorrected values of \( Q_c \). This correction can be estimated using the form of the correction term given by Ardell, et al.\(^{(135)}\),

\[
Q_c = Q_{SD} - n R \left( \frac{T^2}{E} \right) \frac{dE}{dT}
\]  

(14)

where \( E \) is the Young's modulus and the other factors have been previously identified. No temperature dependence of the shear modulus is available
FIGURE 23. ACTIVATION ENERGY FOR CREEP AT TWO STRESSES
but Armstrong and Brown (136) have reported the Young's modulus in polycrystalline molybdenum up to 2000°C. Above 1100°C the modulus decreases rapidly because of grain boundary relaxation. However, a reasonable approximation of \( \frac{dE}{dT} \) can be obtained by extrapolating the curve from below 1100°C to 1900°C parallel to the reported curve for tungsten plotted on a homologous temperature scale. When this is done the correction is \(-1.86\) kcal/mol from 1400°C to 1900°C. For the usual range of \( 3 < n < 6 \) this is only 5 to 10 kcal/mol.

Values of \( Q_c \) can also be measured by making an abrupt change in temperature during a creep test at constant stress, and measuring the creep rate just before and just after the temperature change (2). This must be done quite carefully, particularly when one is not performing the experiment under conditions of steady state creep where there is a well-defined constant creep rate before the temperature change and again when thermal equilibrium is achieved after the temperature change. When, as in this investigation, the creep rate is continuously changing, the complete creep curve of \( \dot{\delta} \) vs. \( t \) must be graphically differentiated and the true creep rate \( \dot{\delta} = \frac{\Delta\delta}{\Delta t} \) plotted against true strain, \( \delta \). The resulting curves are extrapolated to the instant the temperature change is made and \( Q_c \) is calculated from

\[
Q_c = \frac{R \ln \frac{\dot{\delta}_1}{\dot{\delta}_2}}{\Delta \frac{1}{T}}. \tag{15}
\]

Equation (15) assumes that \( Q_c \) is the only temperature dependent factor influencing the creep rate.
In this investigation the creep rates were accurately measured within the sensitivity of the strain measurement by graphically differentiating short sections of creep curves plotted so that the slopes made approximately a 45° angle with the coordinate axes. As noted earlier, the temperature equilibrated within ten minutes at the most after a change and was probably very close to the final temperature in two to five minutes. The creep rates were always measured at times greater than ten minutes after the temperature change and extrapolated back through this period of equilibration to the instant the temperature change was made. Figure 24 shows the results of a temperature change experiment.

The results of the temperature change experiments on crystals of [011] and one [001] orientation are given in Table 11. The crystal having the [001] orientation is included since it deformed relatively uniformly along its gage length. There is no noticeable dependence of $Q_c$ on the direction of the temperature change, the orientation, stress, or temperature. The data in Table 11 give an average value of $Q_c = 95 \text{ kcal/mol}$ for all temperatures, stresses, and orientations used. With the exception of crystal 89-1 the individual specimens are in good agreement with this average.

In specimens 90-1 and 89-1 the first temperature change was made while the strain rate was still rather high and rapidly decreasing. This introduced an error into the calculated value of $Q_c$ due to the uncertainty in extrapolating the creep rate-creep strain curves to the strain at which the temperature change was made. The values in the table, 96 and 98 kcal/mol respectively, represent a good fit to the data.
FIGURE 24. VARIATION OF CREEP RATE WITH CREEP STRAIN DURING A TEMPERATURE CYCLING EXPERIMENT
### TABLE 11

**ACTIVATION ENERGY FOR CREEP IN MOLYBDENUM SINGLE CRYSTALS MEASURED BY TEMPERATURE CHANGES**

<table>
<thead>
<tr>
<th>Specimen Number</th>
<th>Tensile Axis</th>
<th>Stress, psi</th>
<th>Average Temperature, C</th>
<th>$Q_c$ kcal/mole</th>
</tr>
</thead>
<tbody>
<tr>
<td>38-1(^{(a)})</td>
<td>[001]</td>
<td>3800</td>
<td>1335</td>
<td>89</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>77</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>98</td>
</tr>
<tr>
<td>90-1</td>
<td>[011]</td>
<td>2200</td>
<td>1566</td>
<td>96</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>91</td>
</tr>
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<td></td>
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<td></td>
<td></td>
<td>92</td>
</tr>
<tr>
<td>89-1</td>
<td>[011]</td>
<td>4600</td>
<td>1310</td>
<td>98</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>113</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>117</td>
</tr>
<tr>
<td>59-1</td>
<td>[011]</td>
<td>5700</td>
<td>1183</td>
<td>90</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>89</td>
</tr>
</tbody>
</table>

\(^{(a)}\) Strain was relatively uniform over the gage length.

The error in the rest of the data is probably about $\pm 10\%$. The rate of reaching thermal equilibrium is quite good under the circumstances and the calibration of the specimen temperature indicates that the temperature is known to at least $\pm 2^\circ$, although experience would indicate that it is more like $\pm 1^\circ$. For a temperature change of 20 to 30$^\circ$ we would have at most an error in $\Delta T$ of $\pm 10\%$. Estimating the instantaneous change in strain rate is the other source of error in Equation (15). However this error is minimized by averaging the trace of the strain rate vs. strain curves over the whole creep test so that the uncertainty in the individual values of $\Delta \dot{\varepsilon}$ for each temperature change are smoothed out over all the temperature changes.
Although the average $Q_c$ determined by the temperature change experiments is lower than that determined under constant stress and temperature conditions, a consideration of the uncertainty in the two values of $Q_c$ indicates that they are in reasonable agreement with each other and both lie within the range of the reported activation energies for self diffusion presented in Table 1.

**Stress dependence**

The stress dependence of the creep rate during primary creep is a difficult quantity to determine. It is best measured by differential stress experiments at a constant structure. A specimen is crept at some constant stress and temperature to a given creep strain and the stress is decreased. This is done for several different stress decrements. If structure is a factor, transients in the creep rate will occur after the stress decrement and the strain rate *immediately* after the stress decrease is the desired strain rate at constant creep substructure.

Several stress decrement experiments were carried out but the decrements were too large, causing the strain rates after the decrement to be too small to measure. Once these decrements had been made, the creep structure was no longer the desired structure and the test had to be discontinued. These experiments were not continued because of insufficient available experimental run time.

However, creep tests at constant stress were conducted at 1410°C from 1800 to 3000 psi to provide baseline data of the time dependent creep rate at different stresses to compare to the stress
differential experiments. One could then separate the stress dependence of the creep mechanism from the stress dependence contributed by the substructure. Although this could not be done because no stress differential test results were obtained, the stress dependence of the primary creep strain can be compared to those reported by Conway and Flagella for Mo(19) and W(18,19) and by Klopp et al. (27) for W as shown in Table 2. Instead of graphically differentiating the curves to get the creep rates and then m as was done in Figure 21, the primary creep stage is analyzed by the method of de Lacombe (133), with the use of the relations given by Conway and Flagella (19). If it is assumed that the initial portion of the creep curve follows the parabolic time law, 

\[ \epsilon = bt^m \]

as in Figure 21, then the exponent m is obtained independent of the choice of \( \epsilon_0 \) as discussed earlier. The results are shown in Table 12.

For all stresses \( 0.45 < m < 0.62 \) with an average value \( \bar{m} = 0.53 \). This is slightly higher than the values of \( m \) below 1751°C in Figure 21. Also included in Table 12 are the measured and calculated values of \( \epsilon_0 \) and calculated values of b. The value of b is the true creep strain at unit time. In Figure 25 log b is plotted against log \( \sigma \) using an average m of 0.55. The slope gives \( b \propto \sigma^{3.9} \). This stress dependence is not very sensitive to the choice of m. For \( m = 0.50 \), slightly higher than that observed for the lower temperature creep curves in Figure 21, the slope gave \( b \propto \sigma^{4.0} \).

This stress dependence is in good agreement with those listed in Table 2. This suggests that the creep mechanism in these single
Figure 25. Stress dependence of the primary creep strain parameter $b$.
crystals is similar to that in the polycrystalline material, which is reasonable since the polycrystalline material had a coarse grain size.

**TABLE 12**

STRESS DEPENDENCE OF VARIOUS PARAMETERS AT 1410°C

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Stress, psi</th>
<th>$\varepsilon_0$ (a)</th>
<th>$\varepsilon_0$ (b)</th>
<th>m</th>
<th>b(b)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Experimental</td>
<td>Calculated</td>
<td></td>
<td></td>
</tr>
<tr>
<td>C-8</td>
<td>1800</td>
<td>--</td>
<td>0.039</td>
<td>0.59</td>
<td>0.0025</td>
</tr>
<tr>
<td>97-4</td>
<td>2000</td>
<td>0.027</td>
<td>0.037</td>
<td>0.50</td>
<td>0.0045</td>
</tr>
<tr>
<td>98-2</td>
<td>2000</td>
<td>0.027</td>
<td>0.041</td>
<td>0.62</td>
<td>0.0037</td>
</tr>
<tr>
<td>C-3</td>
<td>2300</td>
<td>0.044</td>
<td>0.054</td>
<td>0.50</td>
<td>0.0071</td>
</tr>
<tr>
<td>C-4</td>
<td>2300</td>
<td>0.037</td>
<td>0.045</td>
<td>0.45</td>
<td>0.0081</td>
</tr>
<tr>
<td>C-6</td>
<td>2300</td>
<td>0.025</td>
<td>0.054</td>
<td>0.59</td>
<td>0.0095</td>
</tr>
<tr>
<td>C-2</td>
<td>2700</td>
<td>0.050</td>
<td>0.061</td>
<td>0.56</td>
<td>0.0127</td>
</tr>
<tr>
<td>99-2</td>
<td>3000</td>
<td>0.058</td>
<td>0.063</td>
<td>0.50</td>
<td>0.0182</td>
</tr>
<tr>
<td>97-2</td>
<td>3000</td>
<td>0.058</td>
<td>0.068</td>
<td>0.48</td>
<td>0.012</td>
</tr>
</tbody>
</table>

(a) The experimental values of $\varepsilon_0$ are estimates and are probably too low.

(b) Calculated using the calculated values of m and $\varepsilon_0$.

**Slip Band Studies**

The term "slip band" indicates a group or "packet" of closely spaced slip lines which together form a band separated by some distance from a similar neighboring band. In this section only the slip bands on [[110]] specimens will be discussed. Temperature did not have any
discernible effect on the slip band formation. The slip band appearance and spacing were the same after creep over a wide range of temperature, 1550° to 1917°C and 1403° to 1630°C, at the same stress. The scatter in the measurements of the slip band spacings were fairly large as will be seen later, so there may be a small undetectable temperature dependence of the spacing within the scatter.

**Effect of stress**

While temperature did not appear to have a noticeable effect on the slip line character and spacing, the stress had a marked effect on both. Figures 26 and 27 show the distribution of slip bands along part of the gage length after creep at a low and intermediate stress, the two stresses used for the activation energy determination, the etch pitting and the transmission electron microscopy studies. The same views at both stresses are included in the same figure to facilitate comparison. Figure 28 shows the slip band appearance at a much higher stress. This test was made just above 0.5 \( T_m \) and lasted only two minutes, setting itself somewhat apart from a "creep" test, but the slip band appearance and the transmission electron microscopy studies of this specimen did not show any departures, except in scale, from what was observed after longer creep tests.

The change in the slip bands with increasing stress is quite evident in the \((0\overline{1}1)\) view in Figures 28, 29, and 30. At the low stress, in Figure 29, most of the deformation appears to be confined to quite narrow bands having large displacements with very little detectable slip on the same system between these bands. There is some slip on the
After creep at 900 psi and 1917°C to 43.5% strain

After creep at 2050 psi and 1427°C to 40.6% strain

FIGURE 26. SLIP BAND DISTRIBUTION ALONG SPECIMENS CREPT AT A LOW AND ON INTERMEDIATE STRESS. (011) view
After creep at 900 psi and 1917 C to 43.5% strain

After creep at 2050 psi and 1427 C to 40.6% strain

FIGURE 27. SLIP BAND DISTRIBUTION ALONG SPECIMENS CREPT AT A LOW AND INTERMEDIATE STRESS. (100) view
FIGURE 28. SLIP BANDS FORMED AT A HIGH STRESS

After creep at 5750 psi and 1254 C to ~ 25% strain.
FIGURE 29. SLIP BANDS AFTER CREEP AT 900 psi AND 1917 C TO 43.5% STRAIN

(011) view
FIGURE 30. SLIP BANDS AFTER CREEP AT 2050 psi and 1410 C TO 40.6% STRAIN

(011) view
alternate system, but on a much finer scale, varying from barely visible slip lines in the upper photomicrograph to heavier slip lines in the lower one. It appears from the displacements of the latter slip lines across the heavy slip bands in the lower photomicrograph that the shear strain within the heavy slip bands is quite large, being approximately 3 in this case. The slip bands are 20 to 25 μ wide in this view. Using this figure one can arrive at the crude estimate that about one-half of the total creep strain is contributed by slip within the heavy slip bands. The curvature of the slip bands of the alternate slip system crossing between the heavier bands indicates that there is also some fine slip on the principle slip system in the region between the heavy bands. The stability of the tensile axis during creep requires that the rest of the creep strain must be contributed by the alternate slip system. This slip must occur on a very fine scale since few slip lines of that slip system are visible.

The heavy slip bands formed at a higher stress, shown in Figure 30, are not as sharply defined as those formed at the lower stress. This is so because the slip bands are more closely spaced and because the displacement per slip band is not as large as at lower stresses. In view of the absence of reliable reference lines across the slip bands, the displacements were not estimated. Slip on the alternate slip system can also be observed, but the top photomicrograph in Figure 30 shows that it is very fine.

The "patchy" surface markings visible in both Figures 29 and 30, which are obviously not slip bands, are probably caused by thermal
etching. This may be thermal etching of subgrain boundaries since the markings show a definite preferred orientation normal to and at a slight angle to the slip bands. However, this hypothesis cannot be proved because the \(((011))\) planes do not etch pit.

Two sets of slip lines also formed at the highest stress as shown in Figure 28. Again there is a coarser set and a finer, more closely spaced set, but the finer set is much more evident here than it was at the lower stresses. The total creep strain is not quite as large, so it is possible that the difference in displacement between the coarse and fine slip bands may increase as the creep strain increases; this could occur for example if the coarser slip bands are most active after the fine set has formed. This is suggested by the observations at lower stresses. The wavy character of slip in the \((100)\) view is very evident in this figure. It is also evident that there is a concentration of slip in regions which are very rumpled in appearance. These regions are separated by wider regions containing only a few slip bands. This is probably also true at the lower stresses but the larger scale and depth of the irregularities makes it difficult to see. This "banding" in the \((100)\) views would conform to the lamellar nature of the creep strain observed in the \((011)\) views at all stresses.

The stress dependence of the slip band spacing is shown in Figure 31 and the data are presented in Table 13. The spacing refers only to the heavy slip bands and not to the finer slip frequently observed. It was measured parallel to the tensile axis and resolved normal to the \(((112))\) slip plane which is taken to be the average trace
FIGURE 31. STRESS DEPENDENCE OF THE SLIP BAND SPACING

The double circles represent 2 points.
### TABLE 13
STRESS DEPENDENCE OF SLIP BAND SPACING

<table>
<thead>
<tr>
<th>Specimen Number</th>
<th>Tensile Stress, psi</th>
<th>Temperature, °C</th>
<th>Slip Band Spacing, (a) mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>90-2</td>
<td>900</td>
<td>1644</td>
<td>0.27</td>
</tr>
<tr>
<td>90-4</td>
<td>900</td>
<td>1751</td>
<td>0.42</td>
</tr>
<tr>
<td>92-3</td>
<td>900</td>
<td>1650</td>
<td>0.20</td>
</tr>
<tr>
<td>93-3</td>
<td>900</td>
<td>1917</td>
<td>0.34</td>
</tr>
<tr>
<td>93-4</td>
<td>900</td>
<td>1550</td>
<td>0.31</td>
</tr>
<tr>
<td>96-1</td>
<td>900</td>
<td>1649</td>
<td>0.27</td>
</tr>
<tr>
<td>91-2</td>
<td>2050</td>
<td>1550</td>
<td>0.09</td>
</tr>
<tr>
<td>91-3</td>
<td>2050</td>
<td>1601</td>
<td>0.13</td>
</tr>
<tr>
<td>94-2</td>
<td>2050</td>
<td>1403</td>
<td>0.10</td>
</tr>
<tr>
<td>94-3</td>
<td>2050</td>
<td>1624</td>
<td>0.12</td>
</tr>
<tr>
<td>95-1</td>
<td>2050</td>
<td>1451</td>
<td>0.10</td>
</tr>
<tr>
<td>95-3</td>
<td>2050</td>
<td>1625</td>
<td>0.11</td>
</tr>
<tr>
<td>C-2</td>
<td>2700</td>
<td>1410</td>
<td>0.10</td>
</tr>
<tr>
<td>99-2</td>
<td>2900</td>
<td>1410</td>
<td>0.07</td>
</tr>
<tr>
<td>58-1</td>
<td>5750</td>
<td>1254</td>
<td>0.003</td>
</tr>
</tbody>
</table>

(a) Taking the average trace of the slip bands parallel to (112) planes.
of the slip bands. This is somewhat arbitrary since \((110)\) slip planes are active as well, but any difference in the resolution factor is small compared to the scatter in the measurements. The stress dependence of the slip band spacing follows a \(\sigma^{-1}\) dependence reasonably well.

Although data from 1254° to 1917°C is included in Figure 31, an inspection of Table 13 shows that there is no observable temperature dependence of slip band spacing in specimens crept at the same stress and different temperatures. Therefore temperature exerts at most only a secondary influence on the slip band spacing.

**Strain dependence**

The development of the slip bands with increasing creep strain at the low stress (900 psi) is shown in Figures 32 and 33. On the \((0\overline{1}1)\) side of the specimen, parallel to the slip directions, there were no visible slip lines at 1\% strain, but at 2\% strain, fine, well-defined slip lines were observed. At 5\% strain the widely spaced "coarse" slip bands were beginning to form at a regular spacing. These slip bands continued to develop as creep proceeded.

On the \((100)\) side of the specimens the slip directions made their highest angle with the surface and fine wavy slip lines were visible after only 1\% strain. At 1\% strain the slip lines were fine and wavy, and tended to be clustered together. At 2 and 5\% strain the slip continued to be relatively concentrated and the evolution of the heavy slip bands started. At higher strains the \((100)\) side assumed the rumpled appearance visible at 22\% strain, having heavy corrugations
Figure 32. The development of slip bands with increasing creep strain.

The creep strain is given below each figure and the traces of the possible slip planes are indicated. Tested at 900 psi and 1650 C (011) view.
FIGURE 33. THE DEVELOPMENT OF SLIP BANDS WITH INCREASING CREEP STRAIN

The creep strain is given below each figure and the traces of the possible slip planes are indicated. Tested at 900 psi and 1650 C (100 view)
parallel to \(((112)\) traces representative of large displacements within relatively short distances along the gage length.

**Slip band orientation**

It is clear that the slip bands were not confined to either \(((110)\) or \(((112)\) planes but that the wavy slip bands had an average orientation between two or more contributing slip planes. In the \((0\overline{1}1)\) view of the crept specimen in which the slip directions were parallel to the surface, the slip bands were straight, while in the \((100)\) view the slip bands were wavy and irregular. This is expected in the presence of cross-slip since edge dislocations intersect the \((0\overline{1}1)\) surface and screw dislocations intersect the \((100)\) surface. Hence, a definite determination of the slip band orientation could not be made, even at low strains, because of their wavy and diffuse character. The large offsets in the coarse slip bands at high creep strains were also difficult to follow accurately around the specimen for any appreciable distance because of the severe rumpling of the surface. However an estimate of the favored slip band orientations can be made from the \((100)\) and \((0\overline{1}1)\) views of the specimen surface by determining the average trace of the slip bands in these views.

In the \((0\overline{1}1)\) views the traces of predicted primary \(((110)\) and \(((112)\) slip planes coincided as has been shown in Figure 32. Although no distinction could be made between the two possible types of operative slip planes, it can be seen that the observed slip band traces are nearly parallel to the trace of these possible slip planes. In the \((100)\) views the traces of the possible primary \(((110)\) and \(((112)\) slip
planes are at an angle of 45° making it easy to differentiate between them. While in many instances, particularly at low strains, the observed traces were between the ((112)) and ((110)) traces, there was a definite tendency for the observed slip bands to be closer to ((112)) traces than to ((110)) traces at all strains. While there were many instances where a slip trace was parallel to a ((112)) trace, only one example of a slip band parallel to a ((110)) trace was found and this is shown in Figure 34. In addition this trace is only a short segment of a band that is predominantly parallel to a ((112)) trace. The usual appearance of slip bands at this magnification is also shown in Figure 34. There are characteristically large ridges in the surface running parallel to ((112)) traces.

Many times heavy slip bands in one direction only were observed in a given section of the gage length, although there could be evidence of slip on a finer scale in the other direction. Heavy slip bands in both directions in the same region usually occur in a transition zone between a region having heavy slip bands in one direction and a region having heavy slip bands in the other direction. However, these regions can be quite large. This is illustrated in Figures 15 and 26.

**Etch Pitting Studies**

The etch pitting studies were made on crystals crept at the low stress, 900 psi (0.63 kg/mm²), to form a creep structure sufficiently coarse that the details could be observed metallographically. The ((100)) planes were used for the etch pitting study because only planes up to about 25° from ((100)) will etch pit. This enabled grains formed
FIGURE 34. THE ONLY SLIP BAND FOUND PARALLEL TO A (110) SLIP PLANE TRACE IS SHOWN IN THE TOP FIGURE.

The bottom figure shows the usual appearance of slip bands in this view. Tested 900 psi and 1650 C. (100) view
during creep to etch pit in spite of some misorientation from the original crystal orientation. Prekel and Lawley \(^{130}\) report that to get a one to one correspondence of etch pit to dislocation one must etch a \(([421])\) plane. Here this plane was found to be very near the limit of the etch pitting orientations and it was found necessary to have any deviation of the surface normal, \([[[421]]]\), be towards the nearest \([[[001]]]\) or there would be undesirable etching effects. This would lead to a very uneven etch pitting response on a plane cut from a crept crystal causing uncertainties in the dislocation density measurements and eliminating the only advantage of this surface in studying the dislocation substructures.

The initial dislocation substructure is shown in Figure 35. The subgrains are on the order of 0.7 mm in diameter and are elongated in the growth direction, along the axis of the crystal. The very wide spacing of the dislocations in the grown-in subgrain boundaries indicates that these are very low angle boundaries. They have not been observed in specimens crept beyond 1% strain. The dislocation density was estimated by counting the etch pit density on a \(([100])\) plane parallel to the growth axis of a crystal which had been given the same annealing treatments the creep specimens receive. The initial dislocation density is \(2 \times 10^6 \text{ cm}^{-2}\), which coincides with Prekel and Lawley's \(^{130}\) values for hydrogen annealed, electron beam zoned molybdenum single crystals.

**Strain dependence**

Dislocation substructures formed during creep were studied at load-on strain, i.e., about 1%, and at 2, 5, 22, and 42% tensile creep
FIGURE 35. MICROSTRUCTURE BEFORE CREEP
strain. These are the same crystals whose slip line development is shown in Figures 32 and 33. The term substructure or dislocation substructure as used here denotes the arrangement of the dislocations and subgrain boundaries within the specimen. The orientation of the \((100)\) etch pit planes is given in Figure 36. The \((100)\) plane is parallel to the tensile axis and the \((100)\) view in Figure 15, and the \((010)\) and \((001)\) planes are at \(45^\circ\) to the tensile axis and parallel to the narrow dimension. They would be seen edge on at \(45^\circ\) to the tensile axis in the \((100)\) view of Figure 15.

At 1\% strain the substructure is made up of apparently diffuse bands having a high dislocation density separated by wide bands relatively free of substructure. Figures 37 and 38 show montages of the \((010)\) and \((001)\) planes, and Figures 39, 40 and 41 show the substructure in more detail. The narrow bands of high dislocation density actually consist of several closely spaced rows of etch pits instead of a random distribution. The average trace of the narrow bands lies between the \(((112))\) and \(((110))\) plane traces in Figures 37 and 38; Figures 39 and 41 in the areas marked "A" show that the rows are made up of short rows of etch pits of alternating direction. Hence the apparent trace of the band is some average trace of the shorter segments. Where the boundaries are not segmented such as at B in Figure 41 they appear to be parallel to a \(((111))\) tilt boundary or a \(((110))\) slip plane trace.

The appearance of the segmented boundaries could be interpreted as alternate slip on pairs of \(((110))\) slip planes, e.g., \((110)\) and \((101)\) in Figures 40 and 41, and one must look at the etch pit pattern on a \((100)\) plane to resolve this question. Unfortunately, the \((100)\) surface
FIGURE 36. THE PLANES FOR THE ETCH PITTING STUDY ARE PARALLEL TO THE DASHED ((100)) TYPE PLANES IN THE SCHEMATIC
FIGURE 37. CREEP SUBSTRUCTURE AT LOAD-ON STRAIN, \( \sim 1\% \), ON AN (010) PLANE
FIGURE 38. CREEP SUBSTRUCTURE AT LOAD-ON STRAIN, ~ 1%, ON AN (001) PLANE

0.5 MM
FIGURE 39. CREEP SUBSTRUCTURE AT LOAD-ON STRAIN, ~ 1%
FIGURE 40. CREEP SUBSTRUCTURE AT LOAD-ON STRAIN, ~ 1%
FIGURE 41. CREEP SUBSTRUCTURE AT LOAD-ON STRAIN, ~ 1%
for this strain was accidentally damaged and cannot be presented. If the segmented etch pit traces on the (010) and (001) planes are actually the traces of slip on pairs of ((110)) slip planes, then on a (100) plane the traces should also be segmented on about the same scale and the directions of the individual segments should be at 45° to the tensile axis and at 90° to each other. The average direction of the segmented etch pit line should be nearly parallel to [011]. However on (100) planes for higher creep strains, to be shown later, no such traces are observed.

The etch pit traces crossing the relatively clear areas between the narrow bands usually occur as widely spaced single traces and have two generally preferred directions. One set is approximately normal to the narrow bands and has traces aligned in successive clear areas over large distances. Inspection of the figures suggests that these boundaries are moving down the "clear" bands and trail rows of etch pits behind them into the narrow bands. The other set is at about 45° to the narrow bands and has traces which are quite irregular.

Closely spaced rows of etch pits also appear within a subgrain or in the region between the narrow bands. Examples of these are marked by a "C" in Figures 40 and 41.

After 2% strain Figures 42, 43, and 44 again show the banded creep structure comprised of narrow bands of closely spaced rows of etch pits separated by wide areas having widely spaced single traces of etch pits extending completely across the area. However the etch pit traces in Figures 45, 46, and 47 are no longer arranged in irregular rows, but have assumed an arrangement into what appear to be well
FIGURE 42. CREEP SUBSTRUCTURE AT 2% STRAIN ON THE (001) PLANE
FIGURE 43. CREEP SUBSTRUCTURE AT 2% STRAIN ON THE (010) PLANE
FIGURE 44. CREEP SUBSTRUCTURE AT 2% STRAIN ON THE (100) PLANE
FIGURE 45. CREEP SUBSTRUCTURE AT 2% STRAIN
FIGURE 46. CREEP SUBSTRUCTURE AT 2% STRAIN
FIGURE 47. CREEP SUBSTRUCTURE AT 2% STRAIN
defined sub-boundaries. It would be difficult to rationalize most of the etch pit traces as slip trace pile-ups on slip planes because of the curved, continuous nature of the rows.

A three plane trace analysis of the various sets of sub-boundaries and the narrow bands was made on the original montages in Figures 42, 43, and 44 at 100X magnification. The results shown on the standard stereographic projection in Figure 48 indicate that the intersections of the traces fall in the area around the poles of the (211) and (211) slip planes and the (111) and (111) planes. The traces marked A are the average traces of the narrow bands of subgrains and those marked B are the traces of unsegmented single boundaries. Before going further the method used in making and interpreting the three plane trace analysis should be discussed. Since the planes were not part of the same cube, the individual traces could not be followed on all three planes. However the relative orientations of the cube faces were maintained, i.e., (100) as opposed to (100) and (010) as opposed to (001), throughout the preparation phase, even though this would not matter in many instances. This was done so that the traces could be plotted as accurately as possible. Moreover the (010) and (001) surfaces were cut from adjacent positions in the creep specimen as shown in Figure 36 so that the most prominent slip system operating in that region of the gage length would be common to both sections, although nearly all the sections show some shorter lengths of narrow bands formed by dislocations from the alternate slip system.
Figure 48. The traces of the normals to the sub-boundaries at 2% strain.

The traces from each of the (100) planes are dashed differently.
The boundaries parallel to the \((\overline{1}11)\) planes correspond to tilt boundaries formed by dislocations having the primary Burgers vectors \(\frac{a}{2}[\overline{1}11]\) and \(\frac{a}{2}[\overline{1}11]\). Hence, the long closely spaced boundaries within the narrow bands are all \((\overline{1}11)\) or \((\overline{1}11)\) tilt boundaries and the single boundaries nearly normal to these narrow bands are the other \((\overline{1}11)\) tilt boundaries, either \((\overline{1}11)\) or \((111)\). The trace of the normal to the vertical boundaries crossing the wide "clear" areas in Figure 44 passes through the \((0\overline{1}1)\) pole in Figure 48 and if the average traces of the normals to the boundaries at approximately \(45^\circ\) to the narrow bands in Figures 42 and 43 were placed on the stereographic projection they would intersect the other normal trace near the \((0\overline{1}1)\) pole. This was not done at this strain since the boundary directions are not well defined in these views and an average direction would be very qualitative. A three plane trace analysis for the structure after 22% creep strain which will be discussed later gives a less ambiguous analysis of this boundary.

The banded appearance of the creep structure can also be seen on the \((100)\) plane parallel to the tensile axis shown in Figure 44. The surface is about \(5^\circ\) off of the \((100)\) plane so that the bands of each system, which appear to lie nearly parallel to the \((112)\) slip planes or \((111)\) tilt boundaries, are at a slight angle to each other.

As mentioned earlier Figures 44 and 47 show few, if any, etch pit traces made up of short segments at \(\pm 45^\circ\) to the tensile axis, indicating that the etch pit traces within the narrow bands are not slip lines made up of a pair of \((110)\) slip planes. Some of the etch pit configurations in this view could be interpreted as alternate slip on a pair of \((112)\)
and \((110)\) slip planes by successive cross slip, except that the configuration of the etch pits on the transverse \((001)\) planes does not appear to support this interpretation.

After 5% creep strain, the banded structure was not quite so pronounced and the subgrains between the narrow bands were growing larger, as shown in Figures 49 and 50. This coarser structure appears to have formed at the expense of some of the narrow bands of dislocations still present at 2% strain and some of the long, narrow subgrains also. The banded pattern of the creep structure is quite evident on the \((100)\) plane in Figure 50 where there are narrow bands of long narrow subgrains alternating with wider bands of relatively large equi-axed subgrains. It is quite clear that the narrow bands are parallel to the \((112)\) or \((111)\) planes as mentioned earlier. Figures 51, 52, and 53 illustrate the structure within the narrow bands in more detail and show that many of the boundaries have become quite straight and parallel, aligned quite closely with the traces of the \((111)\) and \((\bar{1}11)\) planes.

The montages in Figures 54, 55, and 56 show that further coarsening of the structure occurred after 22% creep strain. Whereas after 1% creep strain an estimate of the average spacing between the narrow bands on the \((010)\) and \((001)\) planes was 0.12 mm, at increasing creep strains this became 0.12 mm at 2%, 0.18 mm at 5%, and 0.3 mm at 22% creep strain. After 42% strain the appropriate band spacing is difficult to identify.

The sub-boundaries in Figures 57, 58, and 59 have a distinct preferred orientation and several interesting configurations are
FIGURE 49. CREEP SUBSTRUCTURE AT 5% STRAIN ON THE (010) PLANE
FIGURE 50. CREEP SUBSTRUCTURE AT 5% STRAIN ON THE (100) PLANE
FIGURE 51. CREEP SUBSTRUCTURE AT 5% STRAIN
FIGURE 52. CREEP SUBSTRUCTURE AT 5% STRAIN
FIGURE 53. CREEP SUBSTRUCTURE AFTER 5% STRAIN
FIGURE 54. CREEP SUBSTRUCTURE AT 22% STRAIN ON THE (001) PLANE
FIGURE 55. CREEP SUBSTRUCTURE AFTER 22% STRAIN ON THE (010) PLANE
FIGURE 56. CREEP SUBSTRUCTURE AFTER 22% STRAIN ON THE (100) PLANE
FIGURE 57. CREEP SUBSTRUCTURE AFTER 22% STRAIN
FIGURE 58. CREEP SUBSTRUCTURE AFTER 22% STRAIN
FIGURE 59. CREEP SUBSTRUCTURE AFTER 22% STRAIN
noticeable. At A in Figure 58 there are junctions of boundaries forming a sharp corner, not rounded as one might expect when there is no third boundary at the junction to stabilize a sub-boundary "edge."

There are many places where the (111) tilt boundaries contain jogs which are all parallel and very regular in appearance. At B in Figure 58, a (111) and (111) boundary meet with a third boundary parallel to the previously mentioned jogs in the (111) boundaries.

Another trace analysis of the sub-boundary orientations was made on the original 100X magnification montages in Figures 54, 55, and 56 and the results are given on the stereographic projection in Figure 60. The greater regularity of the creep structure at this strain is evident from the close grouping of the boundary normal trace intersections around the specific poles. It is again stressed that the identification of the poles is not unambiguous because a single trace could not be identified on all three surfaces. However by matching possible orientations of sub-boundaries made up primarily of dislocations having the $\frac{a}{2}[111]$ and $\frac{a}{2}[\overline{1}11]$ Burgers vector to the choice of planes given by the trace analysis, the poles shown in Figure 60 are the most reasonable choices.

The significance of the ((111)) boundaries was described earlier but the additional pole identified here, the (011) plane, is the plane of a boundary corresponding formally to two sets of intersecting screw dislocations of equal density having Burgers vectors $\frac{a}{2}[111]$ and $\frac{a}{2}[\overline{1}11]$. Of course local relaxations are expected in such a crossed grid of dislocations. The short segments in the tilt walls mentioned earlier
FIGURE 60. THE TRACES OF THE normals to the sub-boundaries at 22% strain

The traces from each of the (100) planes are dashed differently.
cannot be unambiguously identified either, they may be parallel to any of the four possible ((110)) slip planes or the (100) plane. However, if one analyzes the situation it will be noted that there are few short segments on the ((111)) boundaries in the (100) plane, many less than the number observed in the (010) and (100) planes. The sectioning angle between the planes cannot account for this discrepancy if the jogs are the ((110)) planes. Further, relatively few of the offsets in the ((111)) boundaries are at $45^\circ$ to the tensile axis in spite of the regularity of the structure in the (010) and (001) views. However, since the (100) plane is nearly parallel to the etch pit surface, few of the jogs in the ((111)) boundaries, if parallel to the (100) plane, would be intersected by the etch pit surface. Those that were could make any angle to the tensile axis depending on the direction of deviation of the etch pit surface from the (100) plane. Therefore it seems likely that most of the jogs in the ((111)) boundaries are parallel to the (100) plane.

Finally, at 42% creep strain the structure is still quite inhomogeneous. The banded structure is still evident and it appears in Figures 61, 62, and 63 as if the large subgrains are propagating the banded structure by forming sub-subgrains. The periodicity of the substructure is still quite obvious in Figure 63. Figures 64, 65, and 66 show that the essential details of the structure are unchanged, but a nonuniformity in etch pitting response is quite obvious. This may be caused by greater absorption of impurities by the boundaries if they remained in the same position for long periods during the creep test or it may reflect differences in misorientation angle across the boundaries.
FIGURE 61. CREEP SUBSTRUCTURE AFTER 42% STRAIN ON THE (010) PLANE
FIGURE 62. CREEP SUBSTRUCTURE AFTER 42% STRAIN ON THE (001) PLANE
FIGURE 63. CREEP SUBSTRUCTURE AFTER 42% STRAIN ON THE (100) PLANE
FIGURE 64. CREEP SUBSTRUCTURE AFTER 42% STRAIN
FIGURE 65. - CREEP SUBSTRUCTURE AFTER 42% STRAIN
If one is prepared to accept the hypothesis that the etch pit density is proportional to the dislocation density even though there is not a one to one correspondence, then one can measure the relative change in the dislocation density with creep strain by counting the etch pit density on the \((001)\) planes. This was done, counting only the etch pits within the subgrains and excluding the subgrain boundaries. The results are plotted in Figure 67. There is a rapid decrease initially followed by a more gentle decrease in dislocation density. The densities at each strain were averages of three sets of data from counts made on all three of the \((100)\) surfaces except for the \((001)\) surface at 22\% creep strain. This specimen had a very high density of etch pits which indicated that not all the grinding damage had been removed by electropolishing before etch pitting. This omission was necessary since fresh dislocations do cause etch pits in molybdenum. The error bands represent the range of the average densities on the individual \((100)\) planes. Beyond 45\% creep strain the etch pit density within the subgrains is approaching the density in the uncrept crystals, \(2 \times 10^6 \text{ cm}^{-2}\).

**Temperature dependence**

Temperature has no obvious influence on the creep structure. The structure developed by creep at 1917°C in Figures 68 and 69 is similar to that in Figures 61, 62, and 63 formed at 1650°C, although the structure in Figures 61 and 62 was formed in 46.3 hours and that in Figure 68 was formed in 1.13 hours. The banded structure is much more evident in Figure 69 than in Figure 63 but since the structures in
FIGURE 67. VARIATION OF ETCH PIT DENSITY ON ((001)) PLANES WITH CREEP STRAIN FOR CREEP AT 900 PSI AND 1650 C

The error bars enclose the range in the densities from the individual ((001)) planes at each strain.
FIGURE 68. CREEP SUBSTRUCTURE ON AN (010) PLANE AT A HIGHER TEMPERATURE

Tested at 900 psi and 1917 C to 43.5% strain.
FIGURE 69. CREEP SUBSTRUCTURE ON THE (100) PLANE AT A HIGHER TEMPERATURE

Tested at 900 psi and 1917 C to 43.5% strain
the (010) views are quite similar this is possibly caused by the non-uniformity of the slip along the gage length.

**Stress dependence**

The effect of stress on the size of the substructure is seen by comparing Figures 70 and 71 to Figures 61-63, 68 and 69. The higher stress drastically decreased the size of the structure but the pattern and arrangement of the subgrain boundaries remained the same. However, Figures 70 and 71 show that the alternating bands of narrow, long subgrains and blocky equiaxed subgrains were probably more prevalent at this strain and stress than they were after creep at a lower stress.

The average spacing between the bands of narrow subgrains in Figures 69 and 71 agreed quite closely with the average spacing of the slip bands measured on the same specimen in an (011) view. This strongly suggests that the narrow bands of subgrains and the narrow slip bands coincide. It was quite difficult to get direct verification of this postulate because of rounding of the edges of the specimen during electropolishing. Electropolishing is necessary to provide a damage-free surface for etch pitting.

**Transmission Electron Microscopy**

**As-grown crystals**

As-grown crystals made from both purified and as-received stock were studied briefly in addition to the detailed examination of the crept specimens. The crystals grown from as-received stock containing approximately 180 ppm carbon contained numerous precipitate particles
FIGURE 70. CREEP SUBSTRUCTURE ON (010) PLANE AT A HIGH STRESS

Tested at 2050 psi at 1601 C to 41.5% strain.
FIGURE 71. CREEP SUBSTRUCTURE ON THE (100) PLANE AT A HIGH STRESS

Tested at 2050 psi and 1601 C to 41.5% strain.
with a relatively high dislocation density around them, like those shown in Figure 72. The particles are similar to those observed by Lawley and Geigher\(^{(137)}\) and Prekel and Lawley\(^{(130)}\).

Numerous faults were seen in some of the particles as shown in Figure 72, while others did not appear to have any, even after tilting. While attempting to perform contrast experiments on the faults they faded from view. They did this so rapidly, in a matter of a few minutes, that they could not be studied in any detail. The fading may be caused by a decrease in transmission caused by contamination of the surface, a composition or phase change caused by beam heating, or some other cause.

The particles were identified tentatively as Mo\(_2\)C from interplanar spacings calculated from electron diffraction patterns taken in the electron microscope. A diffraction pattern of the molybdenum matrix adjacent to the particle was taken to calibrate the camera constant for each diffraction pattern of the particle. The average lattice spacings from ten determinations were \(a_0 = 3.084\ \text{Å}\) and \(c_0 = 4.728\ \text{Å}\). The values quoted in the literature\(^{(138-140)}\) range from \(a_0 = 2.994\) to \(3.012\ \text{Å}\) and \(c_0 = 4.722\) to \(4.735\ \text{Å}\). Particles were not observed in the hydrogen purified single crystals, whose only major difference from the unpurified crystals was a much lower carbon level.

Only a few dislocations were visible in the purified single crystals and these were decorated with a fine discontinuous precipitate. It is assumed that this too was a carbide precipitate. The low frequency of observation of dislocations in the purified crystals corresponds to the low, \(2 \times 10^6\ \text{cm}^{-2}\), etch pit density measured in
FIGURE 72. PARTICLES IN UNPURIFIED MOLYBDENUM SINGLE CRYSTALS

The single particles were most common.
crystals prepared in the same manner. In addition, the low dislocation density and the absence of "clean" dislocations demonstrates that the foil preparation technique does not introduce dislocations into the foils.

**Creep structure**

The general features of the creep structure at increasing strain will be presented first followed by observations of specific configurations that are typical of the creep substructures.

Folks parallel to the (211) slip plane were cut only from some of the etch pit specimens crept at 900 psi and 1650°C. Folks from specimens with a very small creep strain contained long groups of straight dislocations lying in the plane of the foil as shown in Figure 73. A Burgers vector determination showed they were edge dislocations with \( b = \frac{a}{2}[\overline{1}11] \), one of the active slip directions identified from the slip line and Laue back reflection studies. The groups of dislocations were up to several microns wide and much longer, in many cases completely crossing the thinned area so that a length could not be measured. The groups also contained some shorter segments of dislocations at an angle to the foil plane. These are possibly edge segments on (110) type slip planes left behind by cross-slipping screw dislocations. Sub-boundaries had already begun to form at this low strain. The boundaries were normal to the foil plane, making it difficult to resolve the individual dislocations in the boundary. However tilting the foils made it clear that the dislocations were in the plane of the foil; in addition the boundary went out of contrast for a \( g = [0\overline{1}1] \).
FIGURE 73. GROUPS OF EDGE DISLOCATIONS LYING IN THE (211) GLIDE PLANE

1% strain at 900 psi and 1650 C
parallel to the dislocation lines. Therefore it appears that these sub-boundaries were \((\overline{111})\) tilt boundaries and the dislocations moved by glide on the \((211)\) plane.

In order to get more information from the individual foils the bulk of the transmission electron microscopy was performed on crystals crept at higher stresses to refine the structure to the point where the configurations in the thinned areas of the foils could be related directly to the configurations observed in the etch pitting studies. As mentioned earlier, this increase in stress does not appear to change the character of the creep substructure but only the relative scale. Most of the transmission electron microscopy was performed on crystals crept at 2000 psi and at 1410°C, and the figures presenting micrographs of foils from these specimens do not have the creep conditions noted. If the creep conditions vary from the above stress and temperature they are noted in the figure. The orientations of the foils sliced from the crept specimens are shown in Figure 74.

At low creep strains the sub-boundaries are still in the process of forming and they are rather loosely knit compared to their appearance at high creep strains. Figure 75 shows a montage of a group of long parallel boundaries forming at 5% creep strain. They are composed primarily of evenly spaced parallel dislocations and their trace in the foil is nearly parallel with the trace of the \((111)\) plane in the \((105)\)
FIGURE 74. ORIENTATION OF THE TEM FOILS SLICED FROM THE CRYSTALS AFTER CREEP
FIGURE 75. A GROUP OF LONG PARALLEL BOUNDARIES FORMING AT 5% STRAIN
foil plane drawn in the figure. Hence the boundaries are quite likely to be tilt boundaries forming from the primary slip dislocations.

At low strains there are always a large number of dislocations near the sub-boundaries as in Figure 75 but relatively few within the subgrains. This was often observed in the etch pitted substructures also. The branch of the montage going away from the boundary at the lower right illustrates how rapidly the dislocation density falls off away from the sub-boundaries. The possible slip planes for the $\frac{3}{2}[111]$ dislocations which form the boundaries are the $(1\overline{1}0)$ whose normal is shown, the $(\overline{1}01)$ which is about 58° to the foil plane and whose trace is perpendicular to the [100] direction, and the $(\overline{2}11)$ which makes an angle of 76° with the foil plane and whose trace is about 20° clockwise from the [110] direction. One cannot identify any group of dislocations moving on a single glide plane, but one can visualize the bowed dislocations in the loose arrays forming into tilt boundaries by glide on the $(\overline{2}11)$ plane. If the loose arrays are assembling themselves into $(111)$ tilt boundaries then perhaps they glide together in this configuration rather than remaining on the glide plane of the source. The edge dislocations in Figure 73 may well be a section through one of these arrays.

The general appearance of the boundaries indicates that there was little activity on slip systems other than those having the $\frac{3}{2}[111]$

*The various planes in Figure 75 could also be indexed as $(\overline{1}11)$ tilt planes, i.e., $g = [0\overline{1}0]$ could have been chosen, but the traces do not fit the structural features as well.
Burgers vector. However near the top of the montage the presence of stranger dislocations is probably the cause for the tilt boundaries bending away from the (111) plane. There are reactions between individual dislocations away from the sub-boundaries. This indicates that slip on a system with a different Burgers vector had occurred in that area, but it was not possible to perform a Burgers vector analysis.

The development of a sub-boundary from several arrays of dislocations is shown in Figure 76. There appears to have been some interaction between two of the boundaries to create the boundary segment parallel to the (100) plane.

At strains of 10% and greater most dislocations are in sub-boundaries. The sub-boundaries are usually quite regular in appearance and orientation and can range from very low angle boundaries having a dislocation spacing on the order of 500 Å to much higher angle boundaries. However, most of the sub-boundaries at 20% creep strain still have less than 2° orientation difference across them. This corresponds to a spacing of about 80 Å between dislocations in a tilt boundary. This spacing is approaching the limit for resolution of the dislocations within the boundaries.

A typical arrangement of sub-boundaries at high creep strain is shown in Figure 77. There are long closely spaced boundaries parallel to a (111) plane and numerous segments of boundary parallel to a (100) plane. The foil plane is (011) so the tilt planes (111), (111), and (100) are seen edge on. Hexagonal networks composed of 2[111] and 2[111] dislocations are parallel to the foil plane and several are visible. Outside this area there are large subgrains much larger and
FIGURE 76. THE DEVELOPMENT OF A SUB-BOUNDARY
FIGURE 77. LONG NARROW SUBGRAINS BOUNDED BY PARALLEL (111) TILT BOUNDARIES
more equiaxed than these. This corresponds to the etch pit structures where long narrow subgrains were separated by large relatively equiaxed subgrains. There are relatively few dislocations within the subgrains.

The development of the creep structure at increasing creep strain is the same in general outline whether observed by TEM or by etch pitting. However, transmission electron microscopy allows us to examine in detail the arrangement of the dislocations within the sub-boundaries and the arrangement of the sub-boundaries themselves.

A common feature is the impingement of two tilt boundaries. Figure 78 shows a group of long parallel (111) tilt boundaries meeting a (111) tilt boundary. Many times there is little reaction between the boundaries to distort the intersection. However, networks do sometimes develop at an intersection and examples of both configurations are present in Figure 78. The angle across the sub-boundaries could not be measured directly* but the stepwise change in contrast is in the same direction as one crosses the successive boundaries indicating that they

*The best method for determining the angular misorientations across low angle boundaries is to measure the relative distance and direction moved by Kikuchi lines in electron diffraction patterns taken on either side of the boundary[11]. However this method requires a component of the rotation axis parallel to the photographic plate so that the Kukuchi lines move some resolvable distance normal to themselves. Roughly 1 cm on the plate corresponds to 1° rotation of the crystal lattice around an axis parallel to the plate for the conditions used in this study. If the rotation is wholly around an axis normal to the plate then the Kikuchi lines merely rotate around the plate center. Since there is an error on the order of 1 to 2° in inserting the plate in the cassette and in determining line directions on the diffraction pattern, small misorientation angles around the plate normal cannot be easily measured. The misorientations across (111), (111), and (100) tilt boundaries are usually around the [011] axis so that the misorientations could not be measured in (011) foils.
FIGURE.78. LONG PARALLEL (111) TILT BOUNDARIES MEETING A (111) TILT BOUNDARY
are probably all the same sign. The inability to consistently measure the misorientation angles of the tilt boundaries in (011) foils is because the line directions of the edge dislocations in the boundaries are usually normal to the foil, i.e., parallel to the [011] direction.

An interesting configuration is the meeting of two tilt boundaries in an "L" junction without apparently requiring a third boundary to stabilize the edge. The intersection may be either somewhat distorted as in Figure 78 or quite sharp as in Figure 79. A direct identification of the type of boundaries making up the configuration by doing a Burgers vector analysis is difficult in a foil parallel to the (011) plane because the dislocations are predominantly normal to the foil. However this same type of junction has been observed in (111) foils where the dislocations are parallel to the plane of the foil and a Burgers vector analysis is easily performed. Figure 80 shows this intersection. The light gray dislocations lie in a tilt boundary nearly parallel to the foil plane and the dark dislocations lie in a plane which is at an angle to the foil. Part of the junction is quite sharp since the dislocations in both boundaries are parallel to [011] and the boundaries intersect along this line. Note also a part of the boundary where the light and dark dislocations alternate. Another view of the same boundary is shown in Figure 81. The flatness of some sections of the tilt boundary in both figures is remarkable.

The boundaries go completely out of contrast for \( g = [011] \) giving \( g \cdot b = 0 \) for the Burgers vectors \( \frac{a}{2}[111], \frac{a}{2}[\overline{1}11], \) and \( a[100] \). Since the Burgers vectors are normal to [011] and [011] is parallel to the dislocation lines, the boundaries are composed of edge dislocations
FIGURE 79. INTERSECTION OF ($\overline{1}11$) AND (111) TILT BOUNDARIES

The 'matte' appearance is due to etching of the foil surface. Tested at 5750 psi and 1254 C to 35% strain.
FIGURE 80. JUNCTION OF (111) AND (111) TILT BOUNDARIES IN THE PLANE OF THE FOIL
FIGURE 81. TILT BOUNDARY CONTAINING STRANGER DISLOCATIONS PARALLEL TO THE BOUNDARY DISLOCATIONS
and are indeed tilt boundaries. It remains to be determined whether the boundary lying at an angle to the foil plane is a \( \overline{111} \) or \( 100 \) tilt boundary. The best that one can do is to determine which is the most probable since it was not possible to differentiate between \( b // [\overline{1}11] \) and \( b // [100] \) with the choice of reflections used. However, one can estimate the foil thickness from the projection of the boundaries, compare the result to the relative intensity of transmission observed through the foil in that area and see which estimate is most reasonable. If this is done, one gets \( t = 3500 \) Å for a \( \overline{111} \) plane and \( t = 1700 \) Å for a \( 100 \) plane. Since transmission through this area of the foil was weak, the thickness was probably closer to \( 3500 \) Å than to \( 1700 \) Å so that the boundary is probably a \( \overline{111} \) tilt boundary. An analysis of the reactions with the stranger dislocations visible for \( g = [0\overline{1}1] \) indicates that it is also possible that the boundary is a \( 100 \) tilt boundary. However reactions between dislocations are quite sensitive to their arrangement, particularly within a sub-boundary containing several sets of dislocations having different Burgers vectors. Hence the foil thickness is probably the better criterion. Both kinds of tilt boundaries are present in the creep structure and the general appearance of the "L" junctions is probably much the same for either type. A definite example of a \( 100):(111) \) "L" junction will be shown later and the following analysis in terms of a \( \overline{111}):(111) \) "L" junction is applicable to it as well, as will be shown.

If one now describes the configuration in Figure 80 as an "L" junction between a \( 111 \) and \( \overline{111} \) tilt boundary it illustrates many of the features observed within tilt boundaries and at tilt boundary
intersections. This is particularly helpful when looking at foils cutting the tilt boundaries at a large angle to the dislocation line direction.

One can reason that the dislocations in the two tilt boundaries do not react parallel to the line of intersection since the reaction would be

$$\frac{a}{2}[111] + \frac{a}{2}[\bar{1}11] = a[011],$$

(16)

which does not decrease the elastic energy. However by "wrapping" themselves around into screw orientation the dislocations along the line of intersection can react

$$\frac{a}{2}[\bar{1}11] + \frac{a}{2}[111] = a[100].$$

(17)

A schematic of the reactions is shown in Figure 82. Using this scheme of reacting the boundary dislocations, a schematic of the boundary in Figure 80 can be drawn as shown in Figure 83. Several features are of interest. At A an $\frac{a}{2}[111]$ dislocation has "wrapped around" into the $(\bar{1}11)$ boundary creating a ledge in the boundary and at B an $\frac{a}{2}[\bar{1}11]$ dislocation has done the same thing in the $(111)$ boundary. Figure 84 shows an example of a reaction like that drawn at B. These ledges are frequently observed in tilt boundaries and can be identified in many of the micrographs included here. At C a group of ledges has piled up in the $(\bar{1}11)$ boundary creating a facet of the boundary which would appear as a hexagonal network if projected onto the $(0\bar{1}1)$ plane. This is also evident in Figure 80. If the facet grows sufficiently large it would
FIGURE 82. THE REACTION BETWEEN PARALLEL $\frac{a}{2}[111]$ AND $\frac{a}{2}[\overline{1}11]$ DISLOCATIONS AT A TILT BOUNDARY INTERSECTION
FIGURE 83. SCHEMATIC DRAWING OF THE DISLOCATION REACTIONS AT AN 'L' JUNCTION OF ((111)) TILT BOUNDARIES

The Burgers vectors refer to a positive line sense along the [011] direction.
FIGURE 84. A (111) TILT BOUNDARY CONTAINING LEDGES PARALLEL TO THE FOIL PLANE

The stranger dislocation has \( b = \pm \frac{8}{2}[11\bar{1}] \).
probably rotate parallel to the (011) plane. The junction at D is not sharp and as the boundary becomes a mixture of $\frac{a}{2}[\overline{1}11]$ and $\frac{a}{2}[111]$ dislocations the outward normal to the tangent of the boundary rotates in the acute angle between [111] and [111]. For example, by using the development of Amelinckx and Dekeyser (114) regarding the plane of boundaries consisting of two sets of edge dislocations, it can be shown that a facet composed of evenly spaced alternating $\frac{a}{2}[111]$ and $\frac{a}{2}[\overline{1}11]$ dislocations would be parallel to the (011) plane. A low angle section of such a boundary composed of a mixture of $\frac{a}{2}[111]$ and $\frac{a}{2}[\overline{1}11]$ dislocations is shown in Figure 85. The "L" junctions in Figures 78 and 79 can be rationalized as sections through D and E respectively in Figure 83.

The "stranger" dislocations visible in the preceding figures when $g = [011]$ have a Burgers vector different from the Burgers vector(s) of the other dislocations in the boundary and create singularities in the regular pattern of the other dislocations. The Burgers vector of these dislocations can be identified directly if long lengths of $b = a[010]$ and $b = a[001]$ dislocations are excluded. The creation of a long length of dislocation of $b = a[[100]]$ seems to require special conditions which are highly improbable for a[010] and a[001] dislocations under the present circumstances. These conditions are discussed in the Discussion section. With this condition, analysis shows that the stranger dislocations in Figures 80 and 81 have $b = \pm \frac{a}{2}[1\overline{1}1]$ and in Figures 84 and 85 have $b = \pm \frac{a}{2}[11\overline{1}]$. Usually the stranger dislocations wander erratically within the boundary but in some instances as in
FIGURE 85. A (111) TILT BOUNDARY AND A MIXED TILT BOUNDARY

The stranger dislocation has \( b = \pm \frac{a}{2} [111] \).
Figure 81 they are aligned parallel to the dislocations comprising the tilt boundary over long distances.

An advanced stage of the "wrap-around" of tilt boundaries to form a network boundary is shown in Figure 86. Here again the edge dislocations in the (111) tilt wall in the plane of the foil show residual contrast for all \( g \)'s except when \( g \cdot b \times \vec{r} = 0 \) is satisfied(141), i.e., \( g \) is parallel to \( \vec{r} \), the dislocation line. The two tilt boundaries have combined to form a network boundary extending to the right. In Figure 87 the two vertical boundaries are predominantly \((1\overline{1}1)\) tilt boundaries. They appear to be moving to the left and their dislocations "knit" into the network boundary behind them. These two tilt boundaries are "following" the \((\overline{1}11)\) boundary in Figure 86. All three tilt boundaries have a misorientation angle around the \([0\overline{1}1]\) direction in the same direction, showing that they are composed of dislocations of the same sign. This is also shown by the fact that the dislocations from all three tilt boundaries react in the same way with the dislocations in the network boundary, i.e., the reacting dislocations all have the same relative position for a given reaction as drawn in Figure 82.

The passage of the tilt boundaries then progressively increases the angle across the network sub-boundary by increasing the density of dislocations of the same sign within the boundary. The misorientation angle across the network boundary to the right of the tilt boundary in Figure 87 is 1.1° around the \([0\overline{1}1]\) direction. The sum of the misorientation angles across the three tilt boundaries is the same (within the error of measurement of \(\pm 0.2°\)) but in the opposite direction. This is required by conservation of the Burgers vectors, i.e., the lattice
The boundary in the plane of the foil is a (111) tilt boundary, the tilt boundary on the left is a (111) tilt boundary and the network boundary would project as a hexagonal network on the (011) plane.
FIGURE 87. (111) TILT BOUNDARIES KNITTING INTO THE NETWORK BOUNDARY

The boundaries appear to be moving to the left, increasing the misorientation across the network boundary as they move along.
orientation to the left of the boundary in Figure 86 is the same as the lattice orientation below the network boundary in Figure 87 since one can trace a path between these two points without intersecting a boundary. The change in the relative densities of the two sets of dislocations in the network boundary also modifies the orientation of the network boundary plane (118). When there are equal numbers of \( \frac{a}{2}[111] \) and \( \frac{a}{2}[\overline{1}11] \) dislocations the boundary plane is parallel to the (0\( \overline{1} \)1) plane. However when the relative densities of the two sets of dislocations are not equal, the boundary plane rotates away from (0\( \overline{1} \)1). Therefore as the tilt boundary passes down the network boundary, the network boundary plane modifies its orientation immediately behind it. However part of the change in orientation could be caused by movement of the network boundary and partial pinning at the tilt boundary intersections creating a "bowing out" effect on the network boundary sections between the tilt boundary intersections.

In the etch pitting studies there were numerous sub-boundary traces parallel to the traces of (100) or several possible ((110)) planes. Transmission electron microscopy of thin foils shows that these segments are indeed parallel to (100) planes. No planes parallel to the possible ((110)) planes have been observed. A good example which relates directly to the arrangement of the structure observed on the etch pitted surfaces is shown in Figure 88 in a specimen which has a considerably refined creep structure after testing at 5750 psi and 1254°C. The structure of the boundaries in Figure 88 shows that they are composed of parallel, straight dislocations suggesting that they are tilt boundaries, but the Burgers vector analysis is difficult
The boundaries are observed edge-on on the right and tilted, showing the dislocations in the boundary, on the left.
because the dislocations are nearly normal to the foil plane. The (100) segments at "A" are right at the junction of two planes parallel to (111) and (111). At the upper segment the group of dislocations at "B" appears to have been a (111) boundary but has broken up and shifted from its probable former (111) orientation. The boundary at C is also nearly parallel to (111) and may have formerly intersected the lower (100) segment before moving on towards the upper right.

In a foil cut parallel to the (104) plane the boundaries can be identified more easily. Figure 89 illustrates a Burgers vector analysis on a (100) tilt boundary at "A" at the intersection of a (111) and a (111) tilt boundary. The dislocations having \( \mathbf{b} = \pm a[100] \) in the (100) tilt boundary at A are out of contrast at the upper left, appear as the closely spaced dislocations at the upper right and as the black region at the lower right (the images overlap due to the steep angle of the boundary to the foil). The few dislocations visible in this boundary at the upper left have \( \mathbf{b} = \frac{4a}{2}[111] \) and are out of contrast at the lower right. The dislocations having \( \mathbf{b} = \pm \frac{a}{2}[111] \) in the (111) tilt boundary at B have only residual contrast at the upper right and those having \( \mathbf{b} = \pm \frac{a}{2}[111] \) in the (111) boundary at C are out of contrast at the lower right. The stranger dislocations in boundary B have \( \mathbf{b} = \pm \frac{a}{2}[111] \) and there are also several \( \mathbf{b} = a[100] \) dislocations in the (111) boundary at C.

The dislocations having \( \mathbf{b} = \pm a[100] \) are unambiguously identified if \( \mathbf{b} = a[[110]] \) are excluded. However there are two choices for each of the \( \mathbf{b} = \frac{a}{2}[[111]] \) dislocations. The \( \mathbf{b} = \pm \frac{a}{2}[111] \) and \( \pm \frac{a}{2}[111] \)
FIGURE 89. FORMATION OF a[100] DISLOCATIONS AT THE INTERSECTION OF (111) AND (111). TILT BOUNDARIES
were chosen since these are consistent with the observed slip directions and the existence of the $b = a[100]$ dislocations.

The $a[100]$ dislocations are evidently formed by the direct combination of the dislocations at the intersection of the $(111)$ tilt boundaries. The dislocations in all three boundaries have parallel projections in the foil plane and from the observations in other foil planes the line directions of the dislocations in all three boundaries are nearly parallel to the $[0\overline{1}1]$ direction. Therefore the reacting and product dislocation lines are parallel to each other and to the line of intersection of the tilt planes, and react directly along their length as

$$\frac{a}{2}[111] + \frac{a}{2}[1\overline{1}1] = a[100].$$

The $a[100]$ dislocations then climb away from the intersection in the $(100)$ tilt plane. The stranger dislocations in the $(\overline{1}11)$ tilt boundary must have $b = \frac{a}{2}[\overline{1}11]$ and react to form short screw segments of $a[100]$ dislocations. At the intersection of the boundaries the $\frac{a}{2}[111]$ dislocations in the $(111)$ tilt boundary "c" and the $\frac{a}{2}[1\overline{1}1]$ dislocation in the $(\overline{1}11)$ tilt boundary "b" annihilate and the excess $\frac{a}{2}[111]$ dislocations enter the $(100)$ boundary. These $\frac{a}{2}[111]$ dislocations in the $(100)$ boundaries can be identified by comparing the micrographs for $g = [010]$ and $g = [\overline{1}10]$.

In the etch pitting investigation, many short segments of boundary parallel to $(100)$ plane traces were observed in $(111)$ boundary traces. Isolated segments of $(100)$ tilt boundary within a $(111)$ tilt boundary have also been frequently observed in thin foils.
Figure 90 shows a Burgers vector analysis on one such segment at "A."
The dislocations having $\mathbf{b} = a[100]$ in the (100) tilt boundary segment
are out of contrast at the upper left for $\mathbf{g} = [010]$ but are in contrast
for all the other $\mathbf{g}$'s. The dislocations in the boundary to either side
of the (100) segment are out of contrast for $\mathbf{g} = [\overline{1}10]$ and have
$\mathbf{b} = \frac{a}{2}[111]$.

As mentioned earlier a foil plane cut parallel to the (100)
plane contained a boundary nearly in the plane of the foil. The dis-
locations in this boundary displayed some residual contrast for all $\mathbf{g}$'s
except when $\mathbf{g} \cdot \mathbf{b} \times \mathbf{g} = 0$, as shown for a small segment of this boundary
in Figure 91. However the contrast was usually absent, light or very
uneven for all $\mathbf{g}$'s making it quite certain that these dislocations have
$\mathbf{b} = \pm a[100]$, are primarily edge type, and the boundary parallel to the
foil plane is a (100) tilt boundary. An intersection between the (100)
tilt boundary and a (111) or (1\overline{1}1) tilt boundary is shown in Figure 92.
All the dislocations go out of contrast for $\mathbf{g} = [0\overline{1}1]$ as before (not
shown). The $a[100]$ dislocations show light contrast and the $\frac{a}{2}[111]$ dislocations show dark contrast; note particularly the alternating
contrast in the parallel dislocations in the dark region at the bottom
of the upper figure. It appears that dislocations from the (100)
boundary are joining the ((111)) boundary and in some places have
reacted to form configurations similar to those seen earlier at the
intersections of (111) and (1\overline{1}1) boundaries. The reaction is of the
type

$$a[100] + \frac{a}{2}[\overline{1}11] = \frac{a}{2}[111],$$

(18)
FIGURE 90. A SEGMENT OF (100) TILT BOUNDARY IN A (111) TILT BOUNDARY
FIGURE 91. VARIABLE CONTRAST DISPLAYED BY DISLOCATIONS IN THE PLANE OF THE FOIL WITH $b = a[100]$ NORMAL TO THE FOIL

This is a part of a (100) tilt boundary lying nearly in the plane of the foil containing stranger dislocations of $b = \frac{a}{2}[111]$. 

0.5 µ.
The (100) boundary is almost in the plane of the foil.
giving a configuration similar to that drawn in Figures 82 and 83. The dislocations don't combine directly along their length parallel to the line of intersection of the tilt planes since the reaction would be

$$a[100] + \frac{a}{2}[111] = \frac{a}{2}[311]$$  \hspace{1cm} (19)

which is an improbable reaction.

Several of the a[100] and \(\frac{a}{2}[\overline{1}11]\) dislocations can be traced into both boundaries and illustrate the continuity of the dislocations across the blunted "L" type intersection between the boundaries.

Figure 93 is another region of the same set of boundaries and again the dislocations showing light contrast have \(b = \pm a[100]\) and the dislocations showing dark contrast have \(b = \pm \frac{a}{2}[111]\) or \(\pm \frac{a}{2}[\overline{1}11]\). It is quite clear after a comparison with the previous figures that the intersecting (100) and ((111)) tilt boundaries can interact in a similar manner to that of intersecting ((111)) tilt boundaries.

Numerous examples of the coalescence of subgrains are observed in the foils cut from crept specimens. These are shown in Figure 94 in foils cut parallel to two different planes through the boundaries. In the top micrograph a higher angle tilt boundary is forming to the right from the coalescence of two tilt boundaries of the same sign as predicted by Li (142). Note the disturbance where one dislocation has glided only part way into the coalesced boundary. If the dislocations were of opposite sign they would combine to annihilate at the intersection and create a lower angle boundary to the right. The dislocation density in the product boundary would equal the difference in the
FIGURE 93. A SUB-BOUNDARY CONTAINING a[100] AND $\frac{a}{2}[111]$ DISLOCATIONS

The photo on the left was taken after the specimen was removed and reinserted in the microscope.
FIGURE 94. SUBGRAINS COALEScing BY TILT BOUNDARIES KNITTING TOGETHER
dislocation densities of the reacting boundaries. The coalescing boundary is not responsible for the bending of the longer boundary up to the left since there are also bends in the boundary in the region not yet combined. There should be no bending on the left caused by the coalescing boundary since the boundary on the right has remained straight and uniform after the coalescing subgrain's boundary has passed. In the lower micrograph, the dislocations in the boundaries lie nearly parallel to the plane of the foil. Also the predominant edge character of the dislocations in the creep structure outside the sub-boundaries is obvious.

Although tilt boundaries were by far the most commonly observed sub-boundaries, boundaries composed of large numbers of two sets of dislocations forming dislocation network boundaries were also observed. An example of the reaction between network boundaries and tilt boundaries is shown in Figure 95. This same area appears on the left in Figure 77. The tilt boundaries are parallel to the (111) planes and the contrast is low for \( g = [211] \) so they are probably (111) tilt boundaries. The network boundaries project as hexagonal networks in the plane of the photograph which is parallel to the (011) plane. The combining of the tilt boundary and the large mesh network boundary creates a network boundary with a much smaller mesh and a different boundary plane. This configuration has been observed several times but it is not clear just how it forms, or what the relative motion of the boundaries may be.

Other examples of network boundaries are shown in Figures 96 and 97. Figure 97 shows a fairly well developed hexagonal network and
FIGURE 95. INTERSECTIONS BETWEEN NETWORK BOUNDARIES AND TILT BOUNDARIES
FIGURE 96. NETWORK BOUNDARY

Tested at 5750 psi, 1254 C to 35% strain

FIGURE 97. HEXAGONAL NETWORK

The a[100] segments are out of contrast. Tested at 5740 psi and 1254 C to 35% strain.
Figure 96 shows an extensive network boundary which changes mesh size and boundary orientation where it joins with other boundaries. The planes of the other boundaries indicate that they have predominantly tilt character.

Summary of experimental results

1. The dislocations in the creep substructure were predominantly edge dislocations.

2. The etch pit (free dislocation) density decreased continuously with creep strain but approaches a constant density at high creep strains.

3. Large numbers of a[100] edge dislocations were formed at ((111)) tilt wall intersections (transmission electron microscopy) and perhaps within the subgrains (etch pitting).

4. The line directions of the $\frac{a}{2}$(111) edge dislocations from both slip systems, and the a[100] dislocations, were nearly parallel to [011] because of slip on (211) and (211) slip planes. There was some deviation from this direction because of the edge dislocations formed as a result of cross slip and because of climb relaxation.

5. It has not been determined whether or not the a[100] dislocations move by glide on the (011) plane.

6. A given type of tilt wall, whether ((111)) or ((100)), can contain parallel edge dislocations of the other Burgers vectors in it.

7. Tilt walls of various lengths formed from the dislocations of the two $\frac{a}{2}$(111) Burgers vectors and the a[100] dislocations. These
boundaries joined in various configurations as they moved relative to one another, i.e., they formed "Y," "L" and "T" intersections.

8. There often was a higher density of dislocations near the sub-boundaries than the average density within the subgrains. Sometimes this high density was concentrated on only one side of the boundary.

9. In a given volume of crystal, part of the slip was concentrated into bands of high slip displacement, and, by inference from the stability of the tensile axis and estimate of slip in the coarse slip bands, there was a nearly equal amount of uniform or finely dispersed slip on the alternate system.

10. At two stresses the average spacing of the narrow bands of subgrains in the (100) view coincided with the coarse slip band spacing.

11. The substructure underwent recovery with increasing creep strain.

12. The total misorientation within the diameter of the x-ray beam increased up to at least ±5° at 43% strain. It is not clear how this misorientation was distributed across successive subgrains. However, the transmission electron microscopy showed examples where the misorientation increases across successive boundaries. Qualitatively from etching characteristics of the sub-boundaries and from transmission microscopy it appeared that the average misorientation was greater at higher creep strains compared to lower creep strains. This has not been definitely established.

13. Based on the etch pitting response, high angle grain boundaries (10° or more) may be present at high creep strains.
14. The coarse slip band spacing decreases with increasing applied stress.

15. Primary creep persisted up to large creep strains.

16. The tensile axis does not rotate more than $\pm 5^\circ$ from [011] during creep. This slight misorientation appears to vary locally within the gage length.

17. The activation energy for creep ($Q_c = 105$ to $108$ kcal/mol) is approximately equal to the activation energy for self-diffusion in molybdenum ($Q_{SD} = 93$ to $115$ kcal/mol).
DISCUSSION

Creep Properties

The agreement between the activation energy for creep, $Q_c$, and the activation energy for self diffusion, $Q_{SD}$ verifies that creep is diffusion controlled. The temperature change experiments gave a $Q_c$ about 10 percent lower than the constant temperature tests. This has also been reported in tungsten $(20,22)$ and in a comparison of Dorn's results in aluminum by Garofalo $(66)$. It is not clear whether this is a significant difference. It may be related to a slight contribution from a recovery process occurring over the entire creep curve as compared to the temperature dependence of the creep mechanism alone. The temperature dependence of the creep mechanism alone would be measured by temperature change experiments, assuming the structure does not change immediately after the temperature change. This should be examined in more detail and may illuminate some aspects of the separate temperature-dependent contributions of the creep substructure and the creep mechanism. These two factors are intimately related and their relationship remains one of the oldest unsolved problems in creep.

The stress dependence of the primary creep strain was in good agreement with values reported in polycrystalline molybdenum $(19)$ and tungsten $(17,19,27)$. This indicates that nothing significantly different occurred during creep in the single crystals compared to polycrystalline molybdenum during primary creep.
The time dependence of the strain rate, or the time law of creep strain, during early primary creep was quite complex. As mentioned earlier there appeared to be several interesting effects associated with loading that could not be accurately observed. The inflection in the loading cycle and immediately after loading may possibly be related to the rounded yield point observed at lower temperatures.

Except over a limited region of creep strain, the time dependence of the primary creep rate is not a simple power law, but has a more complex time dependence. This applies not only to the possible transition region between transient and steady state creep, but to the creep rate immediately following the completion of loading as well. For comparison's sake and to avoid some other more complex, equally empirical time law, the early portion of the creep curves were approximated by a power law. According to Crussard (67) one might expect a gradual increase in m with increasing temperature as observed in many other metals. This is in agreement with the present results except for the rather abrupt increase in m between 1751° and 1852°C or just above 0.7 \( T_m \). This behavior appears to be limited to the relatively low creep strains (less than 0.04 creep strain). At creep strains greater than 0.06 the creep curves obey a \( \Theta \) parameter relation over this whole temperature range using the experimentally determined \( Q_c = 108 \text{ kcal/mol} \). Although \( m \) is temperature dependent it appears to be relatively independent of stress, at least below 0.7 \( T_m \). It is possible that there is a slight change in the creep or recovery mechanism during loading or immediately thereafter above 0.7 \( T_m \), but this change does not persist to very large creep strains. However, without a more detailed knowledge
of the early part of the creep curve and creep structure above and below 0.7 Tm no more can be said at this point.

**Mode of Deformation**

The combined observations of slip bands, Laue back-reflection patterns, transmission electron microscopy and the rotation of the tensile axis in [110] oriented crystals all indicate the primary mode of slip is \(((112))[[111]]\). The wavy nature of the slip bands, the line directions of some of the dislocations in thin foils, and some Laue back-reflection patterns show that there is also a small amount of slip on \(((110))\) slip planes. This is probably associated with the presence of cross-slip between parallel \(((112))\) planes. Table 4 shows that the resolved shear stress \(\frac{\tau}{b} \) is nearly twice as large on the \(((110))\) cross-slip planes compared to the \(((112))\) cross-slip planes.

Chen and Maddin\(^{(143)}\) performed high temperature tensile tests on randomly oriented molybdenum single crystals. They concentrated on the asterism in Laue back-reflection patterns and the rotation of the tensile axis with tensile strain and concluded that slip occurred on \(((110))\) slip planes. However they did not investigate orientations near [011] or [001] where \(((112))[[111]]\) slip would be favored. At lower temperatures, Guieu and Pratt\(^{(144)}\) could find no clear evidence of \(((110))\) slip in [011] oriented crystals deformed in tension. In one case they were able to identify slip traces with the (211) and \((\overline{2}11)\) planes. Above 353 K the asterism in Laue back-reflection x-ray patterns indicated slip always took place on \(((112))\) planes. At higher temperatures \(((112))\) slip becomes more prominent in numerous bcc metals.
and would be expected to do so in molybdenum as well. Hence there is substantial agreement between this study and Guiu and Pratt's observations.

In this study the \([111]\) and \([001]\) corner orientations were quite unstable and the tensile axis must have begun rotating at very low strain. At lower temperatures in iron (\(132\)), the tensile axis at the corner orientations \([100]\) and \([111]\) was stable, i.e., it did not deviate significantly from the initial crystallographic direction during tensile deformation. However Guiu and Pratt\(^{(144)}\) found at 353 and 413 K that the \([100]\) tensile axis was stable until localized necking occurred, associated with single slip on a \((112)\)\([111]\) system. At this point single slip continued with the neck propagating throughout the gage section until very high uniform elongations were attained. This may be attributable to differences in work hardening on the active and latent slip systems. In order for the tensile axis to remain parallel to the \([100]\) or \([111]\) orientations the work-hardening rate on the active system must be greater than the latent hardening rate on the inactive system plus the geometrical softening factor. If it is not, then an active system will continue to operate once started. At high temperatures especially, the work-hardening rate will be quite small and the corner orientations will not be stable during tensile deformation.

The coarse, heavy slip bands have been commonly observed in both single crystals and coarse-grained polycrystalline metals. They become more prominent as the stress decreases and their spacing is inversely proportional to the applied stress. Since only about half or less of the creep strain could be accounted for by the coarse slip bands and
the tensile axis remains parallel to [011] during creep, there must be an equal amount of slip on the alternate slip system. This slip is evidently so uniformly distributed within the crystal that any slip lines are very fine. This may be equivalent to the observations in polycrystalline metals after significant creep at low stresses. As the creep stress is decreased in polycrystalline metals the coarse slip bands disappear (presumably because their spacing is larger than the grain size) and only very fine slip lines are visible \( (29,145,146) \).

There are then two types of slip contributing to the creep strain: (1) a very concentrated shear on one slip system having local strains on the order of \( \gamma = 3 \) and (2) a uniformly distributed, homogeneous, slip on the same and alternate slip system(s). In addition, there are some areas which have relatively heavy slip bands from both slip systems present in the same area. This is in a transition region and the slip structure will be somewhat more complicated in this region. It is not clear in most instances whether both systems are operating simultaneously or whether one is in the process of superseding the other.

The transmission microscopy studies showed that the line directions of the edge dislocations were usually parallel to the [011] direction from slip on the \( (\overline{2}11) \) and \( (211) \) planes. Therefore most of the discussion will consider only those dislocation interactions and geometries originating from slip on the \( (\overline{2}11)[111] \) and \( (211)[\overline{1}11] \) slip systems.
Creep Substructure

The various methods used to study the creep substructure will not be discussed separately, but rather the results of each of them will be discussed and correlated with the others. The etch-pitting studies give the best illustration of the overall development of subgrains and dislocation distribution while the transmission electron microscopy brings out the details of the structure. The slip line studies serve to link the external appearance of creep deformation, often studied in the past, to the internal structure. The Laue back-reflection study supplements these observations by showing how creep deformation disrupts the continuity of the crystal.

Much of the creep structure is composed of tilt walls. These are both very long tilt walls joined to others in a manner in which the long-range stresses are probably quite low and short tilt walls which probably have a significant long-range stress field. Hence dislocation-tilt wall and tilt wall-tilt wall interactions may be exerting an important influence on the creep behavior. Some aspects of these interactions are discussed prior to a discussion of the creep structures.

Li(147,148) has calculated the resistance of infinite and finite tilt walls to penetration by individual parallel edge dislocations having parallel or nonparallel Burgers vectors. He considered both glide and climb penetration. Since this is exactly the geometry present in the creep substructures, a summary of these results is presented:

1. The approaching dislocation and the dislocations in the tilt wall have parallel Burgers vectors.
a. Infinite tilt wall fully pinned

(1) An infinite tilt wall of evenly spaced dislocations has alternating "layers" of positive and negative shear stresses parallel to the slip planes of the dislocations in the wall. These "layers" extend large distances from the wall and each has a thickness of one-half the dislocation spacing in the wall.

(2) The long-range interaction with a dislocation of the same sign is always less than that of an isolated, pinned dislocation on the same slip plane at the position of the wall.

(3) This long-range attraction or repulsion becomes weaker as the dislocation spacing, D, in the wall decreases.

(4) The maximum force exerted by the tilt wall on a dislocation passing through the wall midway between two dislocations in the wall increases as D decreases. The maximum stress \( \sigma_{xy} \approx 0.45 \frac{\mu b}{2 (1-v) D} \), where \( \mu \) is the shear modulus, \( b \) is the Burgers vector, \( v \) is Poisson's ratio and \( D \) is the dislocation spacing in the tilt wall.

(5) A smaller maximum stress \( \sigma_{xy} \approx 0.22 \frac{\mu b}{2 (1-v) D} \) is required to push a free dislocation through at \( D/2 \) by a second approaching dislocation.

b. Infinite tilt wall partially pinned

(1) If only the dislocation in the slip plane of the approaching dislocation is free to move in the tilt wall, the maximum stress to penetrate the wall (or
knock the free dislocation out) is approximately one-tenth of that in l.a. (3), i.e., \( \sigma_{yx_{\text{max}}} = 0.04 \frac{\mu b}{2(1-\nu)D} \).

(2) Piling up against a tilt wall containing free dislocations is unlikely.

c. Finite tilt wall

(1) A finite tilt wall has a long-range stress field which exerts a maximum shear stress \( \sigma_{yx_{\text{max}}} = \frac{\mu b}{2\pi (1-\nu) D} \) at a point a distance \( \frac{\ell}{2} \) from the wall where \( \ell \) is the length of the tilt wall.

(2) The long-range stress field resembles that of a single dislocation of strength \( Nb \) at large distances, where \( N \) is the number of dislocations in the wall.

(3) The resistance to penetration of the wall at short range, within a distance \( D \), is similar to the infinite tilt wall.

(4) The attractive region is limited to a distance of about \( D \) from the tilt wall in the middle of the tilt wall.

2. The approaching dislocation's Burgers vector is at an angle to the Burgers vector of the tilt wall dislocation.

a. Infinite tilt wall fully pinned

(1) The minimum shear stress for penetration by slip increases as the angle \( \psi \) increases for \( \psi \gg 20^\circ \). For \( \psi = 70.5^\circ \), \( \sigma_{yx_{\text{min}}} = 1.6 \frac{\mu b}{2 (1-\nu) D} \) at \( D/2 \).

(2) The minimum normal stress for penetration by climb decreases as \( \psi \) increases. At \( \psi = 70.5^\circ \), \( \sigma_{n_{\text{min}}} \approx 0.4 \frac{\mu b}{2 (1-\nu) D} \) at \( D/2 \).
(3) The long-range interactions at distances greater than 
D from the tilt wall are negligible.

b. Finite tilt wall

(1) The short-range resistance to penetration within a 
distance D of the tilt wall is similar to the infinite 
tilt wall.

(2) The long-range resistance to a slipping dislocation is 
relatively independent of Ψ. \( \sigma_{\text{xy max}} = \frac{\mu b}{2\pi (1-\nu) D} \) 
but the relative position at which this maximum occurs 
does vary with Ψ.

(3) The long-range resistance to a climbing dislocation is 
decreased slightly as Ψ increases.

(4) Rearrangement of a given number of randomly distributed 
dislocations into a finite tilt wall produces only a 
slight strengthening.

The angle Ψ = 70.5° is the approximate angle between the primary slip 
vectors observed in this study.

Sub-boundary junctions

In addition to the interactions between individual dislocations 
and tilt walls, the interactions between the tilt walls themselves are 
also important. The regular arrangement of the sub-boundaries in the 
creep sub-structures, particularly at high creep strain creates specific 
types of sub-boundary intersections and arrangements. Two of the types 
of intersections that were prominent both in the etch-pitted structures 
and in the TEM foils were (1) the "L" junction in which two different
((111)) tilt boundaries or a ((111)) and ((100)) tilt boundary joined along a line parallel to the dislocations in the tilt boundaries and

(2) a "Y" junction in which two different ((111)) tilt boundaries and a ((100)) tilt boundary joined together along a line parallel to the dislocations in the boundaries. The dislocations in the individual boundaries may not always be strictly parallel to each other and the line of intersection, but they are very nearly so.

These types of tilt boundary intersections may be explained by examining first the types of ((111)) tilt boundary configurations that can occur involving only two Burgers vectors in both the positive and negative sense. For our example we choose the observed slip vectors $\pm \frac{a}{2}[111]$ and $\pm \frac{a}{2}[\overline{1}1\overline{1}]$. The two possible arrangements with the line direction of the boundary dislocations parallel to the line of intersection is shown in Figures 98 and 99. The lengths $l_1$ and $l_2$ are the lengths of the finite tilt walls and $D_1$ and $D_2$ are the spacings between the dislocations in the respective tilt walls. Tilt wall 1 is placed along the y axis and tilt wall 2 is placed along the x axis. The dislocations are presumed to be evenly spaced within each tilt wall. For simplicity the tilt walls are placed at 90 degrees, hence their glide planes are also at 90 degrees. This should be a reasonable approximation for ((111)) planes meeting at 109.5 degrees.

The procedure will be to use the components of the stress tensor for a finite tilt wall $(142,149)$ and superimpose the stress fields of the perpendicular tilt walls. The original expressions for the various components of the stress field of a finite tilt wall were given with the origin of the x,y coordinate system in the center of the tile wall.
FIGURE 98. INTERSECTION OF TWO PERPENDICULAR TILT BOUNDARIES

FIGURE 99. INTERSECTION OF TWO PERPENDICULAR TILT BOUNDARIES
One can easily derive the stress tensor with the origin at the top of the tilt wall by assuming the tilt wall is a continuum of dislocations as shown by Hirth and Lothe\textsuperscript{101}. For the configuration of wall 1 in Figure 98, the stress field components are

\begin{align}
\sigma_{xx} &= -\frac{K}{D} \left[ \frac{x^2}{x^2 + y^2} - \frac{x^2}{x^2 + (y+l)^2} + \frac{1}{2} \ln \frac{x^2 + (y+l)^2}{x^2 + y^2} \right] \tag{20} \\
\sigma_{yy} &= -\frac{K}{D} \left[ -\frac{x^2}{x^2 + y^2} + \frac{x^2}{x^2 + (y+l)^2} + \frac{1}{2} \ln \frac{x^2 + (y+l)^2}{x^2 + y^2} \right] \tag{21} \\
\sigma_{xy} &= -\frac{K}{D} \left[ \frac{xy}{x^2 + y^2} - \frac{x(y+l)}{x^2 + (y+l)^2} \right] \tag{22}
\end{align}

where $K = \frac{\mu b}{2\pi (1-\nu)}$ and $\mu$ = shear modulus, $\nu$ = Poisson's ratio and $b$ is the Burgers vector. The wall of length $l$ is located at $0 \geq y \geq l$ and contains evenly spaced dislocations a distance $D$ apart. These equations are identical with those given by Li\textsuperscript{142,148} derived by the method of Cottrell\textsuperscript{149} if one substitutes $y' + \frac{l}{2} = y$ and $\frac{l}{2} = L$ in Li's\textsuperscript{148} Equations (16) to (18).

These equations are good approximations at distances greater than $D$ from the tilt wall. At distances smaller than $D$ the stress field of the nearest dislocation in the wall becomes significant and the continuum approximation breaks down\textsuperscript{142}. Within the finite tilt wall, away from its ends, Li\textsuperscript{142} states that the local stress field is the same as if the dislocations were in an infinite tilt wall. However for our purposes we are interested in the general trends of the stress field over a much larger region and we will use Equations (20) to (22).
The stress components will refer to the coordinate system in Figure 98. For wall 1, Equations (20) to (22) give the stress components directly by substituting \( l = l_1 \) and \( D = D_1 \), but for wall 2 the coordinates of wall 2 must be transformed to those of wall 1. This is done by using the Equations (5) with \( a_{12} = -1, a_{21} = 1 \), all others \( a_{ij} = 0 \). This is accomplished by replacing \( x \) and \( y \) in Equations (20) to (22) with \( y \) and \( -x \) respectively.

The stress field components for wall 2 referred to the coordinate system in Figure 98 are

\[
\sigma_{xx} = - \frac{K_2}{D_2} \left[ - \frac{y^2}{x^2 + y^2} + \frac{y^2}{(l_2-x)^2 + y^2} + \frac{1}{2} \ln \left( \frac{(l_2-x)^2 + y^2}{x^2 + y^2} \right) \right] \\
\sigma_{yy} = - \frac{K_2}{D_2} \left[ \frac{y^2}{x^2 + y^2} - \frac{y^2}{(l_2-x)^2 + y^2} + \frac{1}{2} \ln \left( \frac{(l_2-x)^2 + y^2}{x^2 + y^2} \right) \right] \\
\sigma_{xy} = - \frac{K_2}{D_2} \left[ \frac{xy}{x^2 + y^2} + \frac{X}{(l_2-x)^2 + y^2} \right].
\]

The constant \( K \) contains the Burgers vector of the specific tilt wall and is therefore indexed also. The local force on each tilt wall is the result of the stress field of the tilt wall itself and that of the other tilt wall. The external stress is not included.

If the normal stresses are evaluated at the position of the tilt wall, then the climb forces on wall 1 are

\[
F_{c,1} + F_{y,1} = - b_1 (\sigma_{xx,1} + \sigma_{xx,2})
\]
at \( x = 0 \), where the subscripts 1 and 2 refer to tilt walls 1 and 2 respectively, and

\[
F_{c,2} = F_{x,2} = b_2 (\sigma_{yy,1} + \sigma_{yy,2})
\]  

(27)

at \( y = 0 \). Substituting into Equations (26) and (27) from Equations (20) to (25) we get

\[
F_{c,1} = b_1 \left\{ \frac{K_1}{2D_1} \ln \left( \frac{y + \kappa_1}{y^2} \right) + \frac{K_2}{D_2} \left[ -1 + \frac{y^2}{y^2 + \kappa_2^2} + \frac{1}{2} \ln \left( \frac{y^2 + \kappa_2^2}{y^2} \right) \right] \right\}
\]

(28)

and

\[
F_{c,2} = -b_2 \left\{ \frac{K_1}{D_1} \left[ -1 + \frac{x^2}{x^2 + \kappa_1^2} + \frac{1}{2} \ln \left( \frac{x^2 + \kappa_1^2}{x^2} \right) \right] + \frac{K_2}{2D_2} \ln \left( \frac{\kappa_2 - x}{x^2} \right) \right\}
\]

(29)

At the point of interest, near the junction of the tilt boundaries, it is evident that as \( y \) nears zero in Equation (28) and \( x \) nears zero in Equation (29), the logarithmic terms get very large, increasing in the same direction. Hence the climb forces act to drive the dislocations near the origin out of the tilt walls toward the intersection of the boundaries. Therefore this configuration is not stable with regard to climb. With regard to glide the respective glide forces along the tilt walls 1 and 2 are

\[
F_{g,1} = F_{x,1} = b_1 (\sigma_{xy,1} + \sigma_{xy,2})
\]

(30)

at \( x = 0 \), and

\[
F_{g,2} = F_{y,2} = -b_2 (\sigma_{xy,1} + \sigma_{xy,2})
\]

(31)
**FIGURE 100.** FORMATION OF A (100) TILT WALL AT THE INTERSECTION OF TWO ((111)) TILT WALLS

This is a 'Y' intersection.

**FIGURE 101.** 'STABLE' INTERSECTION OF TWO ((111)) TILT WALLS

This is an 'L' intersection.
at \( y = 0 \). The complete expressions are

\[
F_{g,1} = - \frac{b_1 K_2}{D_2} \frac{y \xi_2}{y_2 + \xi_2^2} \\
F_{g,2} = - \frac{b_2 K_1}{D_1} \frac{x \xi_1}{x^2 + \xi_1^2}
\]  

(32)  

(33)

These glide forces act to move wall 1 to the right and wall 2 down, but near the intersection the glide forces approach zero. The net result is that at the intersection the dislocations from each wall are brought together by both the climb and glide forces. If we now assume that the walls are 109.5 degrees apart and assign them \( \frac{a}{2}[(111)] \) Burgers vectors, e.g., \( b_1 = \frac{a}{2}[\overline{1}11] \), \( b_2 = \frac{a}{2}[\overline{1}11] \), which are crystallographically consistent with the FS/RH rule.* The dislocations meeting at the intersection can then combine along the line of intersection by the reaction

\[
\frac{a}{2}[\overline{1}11] + \frac{a}{2}[\overline{1}11] = a[100] .
\]

These \( a[100] \) dislocations can then climb away from the line of intersection under the same stress field exerted on the \( \frac{a}{2}[(111)] \) dislocations and thereby create a (100) tilt plane. This is shown in Figure 100. The normal stresses of both walls are compressive at this point. Although the calculations have not been made, the \( a[100] \) dislocations would probably continue to climb away from the intersection as they were

* The direction of \( b \) is obtained using the right handed convention, e.g., \( b \) and \( \xi \) point in the same direction for a right handed screw.
formed until the length of the (100) tilt wall was some large fraction of the length of either or both of the ((111)) tilt walls. The symmetry of the arrangement shows that the glide force on the (100) tilt wall is small or zero depending on the relative length of the walls and the spacing of the dislocations within them.

The alternative arrangement of dislocations at a tilt wall intersection is shown in Figure 99. The extra half planes point in the same direction as one travels along the boundary around the intersection. One might expect this to be a lower energy configuration than the previous one. The change in $b_2$ changes the sign of the stress components of tilt wall 2 in Equations (23) to (25) and the climb force relation, Equation (27), but those of tilt wall 1 remain unchanged.

The climb forces are again given by Equations (28) and (29).

\[
F_{c,1} = b_1 \left\{ \frac{K_1}{2D_1} \ln \left( \frac{y + \ell_1}{y^2} \right) - \frac{K_2}{D_2} \left[ -1 + \frac{y^2}{y^2 + \ell_1^2} + \frac{1}{2} \ln \frac{y^2 + \ell_1^2}{y^2} \right] \right\}
\]

\[
F_{c,2} = b_2 \left\{ \frac{K_1}{D_1} \left[ -1 + \frac{x^2}{x^2 + \ell_1^2} + \frac{1}{2} \ln \frac{x^2 + \ell_1^2}{x^2} \right] - \frac{K_2}{2D_2} \frac{(\ell_2 - x)^2}{x^2} \right\}
\]

Now near the intersections, as $y$ and $x$ approach zero, the logarithmic terms largely cancel each other. If one makes the simplifying assumption that $D_1 = D_2$, $b_1 = b_2$ then as $x$ and $y$ go to zero

\[
F_{c,1} = \frac{bK}{D} \left( 1 + \ln \frac{\ell_1}{\ell_2} \right) = \frac{bK}{D} \ln \frac{\ell_1}{\ell_2}
\]

\[
F_{c,2} = \frac{bK}{D} \left( -1 + \ln \frac{\ell_1}{\ell_2} \right) = \frac{bK}{D} \ln \frac{\ell_1}{\ell_2}
\]
The climb forces near the boundary intersection may now be away from the intersection, equal to zero or greater than zero depending on the relative lengths of the walls. As mentioned earlier this calculation does not give the forces within a distance of about D of the wall. However, when the forces are very large as a result of the stress concentration at the tip of the wall, the calculation should give a reasonable approximation to the forces on the dislocations at the intersection. When the forces are low, then as the dislocations approach within a distance D, e.g., at the intersection, the interactions will be closer to two dislocation interactions. Inspection of the stress field of a single dislocation shows that two parallel edge dislocations having perpendicular Burgers vectors are at equilibrium with respect to climb and glide when they lie in a plane at 45 degrees to their respective slip planes. From the continuum approach one can only show the general behavior at the intersection due to the stress fields of the walls themselves. Near the intersection, (1) the climb forces are away from the intersection in the wall which is much shorter than the other, (2) the climb forces are very small or zero if the walls are of the same order of length, and (3) the climb forces are towards the intersection in the wall which is much longer than the other.

If one assigns Burgers vectors to the dislocations in the tilt walls, a comparison with the first case shows that the reaction at the intersection is

\[ \frac{a}{2}(111) + \frac{a}{2}(111) = a(011) \]

which is energetically unfavorable.
This unfavorable energy change would cause a local repulsion between the dislocations near the intersection which could balance the climb forces in Equations (34) and (35) if they aren't too large. The intersection is then stable with respect to climb.

The glide forces on the respective tilt walls are

\[ F_{g1} = \frac{b_1K_2}{D_2} \frac{y \ell_2}{\ell_2^2 + y^2} \]  \hspace{1cm} (38)

and

\[ F_{g2} = \frac{b_2K_1}{D_1} \frac{x \ell_1}{x^2 + \ell_1^2} \]  \hspace{1cm} (39)

These forces tend to move the tilt walls apart, but near the intersection they become very small. The equilibrium position of the leading dislocations may be on a plane nearly parallel to \( x = y \) as mentioned earlier.

Although most of the tilt wall has a slip stress on it from the stress field of the other tilt wall, the tilt walls appear to be quite straight and quite parallel to the appropriate \((111)\) plane. The stress field of the tilt wall must be strong enough to maintain its own dislocations in the proper alignment. Li\(^{(148)}\) has shown that a distortion of the tilt boundary out of the pure tilt alignment immediately creates a long-range stress field which is a higher energy configuration. Therefore this configuration probably represents the "L" junction noted in the substructure and is shown in Figure 101 as the intersection between two \((111)\) planes.
However, this "stable" configuration is not limited to "L" junctions between (hkl) type tilt planes. It also applied to the (100)-(111) tilt wall junctions created when a segment of (100) tilt wall is isolated within a (111) tilt boundary. The dislocation spacing in the (100) tilt wall can adjust itself to make up for the differences in the length of the Burgers vectors and to maintain a constant mis-orientation angle across the combined sub-boundary. The formation of these isolated segments of (100) tilt wall in a (111) tilt wall suggests that they were formed by the temporary intersection of two (hkl) tilt walls. This would originate from an arrangement like that illustrated in Figure 102, called a "T" junction.

In this case the position of tilt wall 2 is at y'. The expressions for the climb and glide forces along the walls due to the combined stress fields of both walls are formed in the same way as they were earlier using Equations (20) to (25) except for substituting $y - y' = y$ into Equations (23) to (25). Therefore the climb and glide forces are

$$
F_{c,1} = b_1 \left\{ \frac{K_1}{2D_1} \ln \frac{(y+y_1)^2}{y^2} + \frac{K_2}{D_2} \left[ 1 - \frac{(y-y')^2}{\xi_2^2 + (y-y')^2} + \frac{1}{2} \ln \frac{\xi_2^2 + (y-y')^2}{(y-y')^2} \right] \right\}
$$

$$
F_{g,1} = -\frac{b_1K_2}{D_2} \frac{(y-y') \xi_2}{\xi_2^2 + (y-y')^2}
$$

at $x = 0$, and
FIGURE 102. TWO TILT WALLS MEETING IN A 'T' JUNCTION
\[ F_{c,2} = -b_2 \left\{ \frac{K_1}{D_1} \left[ - \frac{x^2}{x^2 + y_2^2} - \frac{x^2}{x^2 + (y'+l_1)^2} + \frac{1}{2} \ln \frac{x^2 + (y'+l_1)^2}{x^2 + y_2^2} \right] \\
+ \frac{K_2}{2D_2} \ln \frac{(l_2-x)^2}{x^2} \right\} \] (42)

\[ F_{g,2} = \frac{b_2K_1}{D_1} \left[ \frac{x y'}{x^2 + y_2^2} - \frac{x (y'+l_1)}{x^2 + (y'+l_1)^2} \right] \] (43)

at \( y = y' \). At \( y' = 0 \) and \( y' = -l_1 \) the relations reduce to Equations (28) to (31) and (34) to (37). It is clear from Equation (43) and the fact that \( 0 \leq y' \leq -l_1 \), that wall 1 produces a glide pressure on wall 2 that is always in the same direction, e.g., in the -y direction in Figure 102. This is consistent with the direction of the glide force on individual dislocations of the same Burgers vector calculated by Li (142).

The climb force on the dislocations near the intersection in wall 2 will be quite large except when \( y' \) is nearly equal to \(-l_1\). Hence there will be a tendency to force the dislocations in the two walls together and form a [100] dislocations at the intersection. This type of reaction will create the configuration shown in Figure 103. It was shown earlier that the glide forces will tend to maintain the lower half of tilt wall 1 and tilt wall 2 in the relative positions shown.

After creating a segment of (100) tilt wall in the (111) tilt wall, the (111) tilt wall can move on again by glide, leaving the (100) tilt wall segment behind.
FIGURE 103. FORMATION OF A (100) TILT WALL SEGMENT IN A (111) TILT WALL.

The (111) tilt wall can break away from the intersection and move down by glide.
Although the actual tilt walls are at an angle of 109.5° and the Burgers vectors are at an angle of 70.5° instead of 90° the general conclusions should be the same. The Burgers vectors and tilt walls are only 19.5° from the perpendicular and since all that is involved is a resolution of the stress tensor through this small rotation the differences in the stress fields will not be large, and the directions of the forces will not be reversed. Carrying through the resolution factors makes the expressions more unwieldy and the general trend is not as clear. Of course, for an accurate determination the calculation should include the proper angular relationships. For example, it is equally possible geometrically to have tilt walls meet at a Y or L junction at 71° instead of 109° but this has not been observed. This arrangement is possibly unstable with respect to glide when the true angular relationships are included.

The detailed calculations for these configurations should now be made taking into account the actual angular relationships and anisotropic elasticity. This is necessary to determine whether the conclusions reached with the approximations used here for the various configurations still stand after a more rigorous treatment. In addition it would allow some estimate to be made of the relative importance of the interactions between the various tilt walls and individual dislocations. For example, what influence does the length of the (100) tilt wall segments have on allowing the intersecting tilt wall to move on; how do they interact with gliding individual dislocations, etc.?
Strain dependence of the substructure

At load-on strain the etch-pitted surfaces have a banded structure made up of diffuse rows of etch pits extending across most of the cross-section of the crystal. The reason for this alternation of light and dark etch pit bands is not clear. It is an etching effect that may be related to the sign of the Burgers vector, positive or negative, the type of Burgers vector, $[[111]]$ or $[[100]]$, or the character of the dislocation, screw or edge. A comparison of the two bands in Figures 40 and 46 indicates that the darker pits are related to the prominence of segments aligned approximately parallel to the $(100)$ trace. This suggests that the dark pits are related to a $(100)$ dislocations in this area. This aspect of the etch pitting technique warrants further investigation since the etch pitting technique would then provide a great deal more information.

Although one cannot describe the load-on structure in detail one can make several inferences. At high strains the long, closely spaced boundaries in the bands have been identified as $(111)$ tilt boundaries containing $(100)$ tilt boundary segments. It would be difficult to picture these bands as evolving from anything except the diffuse bands that are present from the beginning of the creep test. This suggests that the diffuse bands of dislocations are diffuse "tilt bands." In addition, tilt walls have been observed after load-on strain in thin foils. The manner in which they form at the regular spacing is not clear. Perhaps rather uniform slip on one system interacts with regularly spaced slip bands on the alternate system. This would create numerous a $(100)$ dislocations which could act as obstacles to further
glide and initiate the formation of tilt walls. As Li\(^{(142)}\) has pointed out, the growth of tilt walls can be autocatalytic, particularly under conditions of an external stress and a uniform distribution of slip dislocations. There is a volume of crystal at the end of the tilt wall in which the wall creates a shear stress "pulling" glide dislocations of the same sign into alignment at the end of the wall. This volume of crystal increases as the wall grows longer\(^{(142)}\). Thus, if a minimum of climb is required the accumulation of dislocations into loose tilt wall configurations aligned across the cross-section of the crystal could occur very rapidly.

The tilt walls of opposite sign are prevented from spontaneously gliding together and annihilating by the pinning effect of the a[100] dislocations. In order to move in the direction of glide of the ((111)) tilt walls, the a[100] dislocations have to move by a process of climb and glide\(^{(150)}\). Tilt walls of the same sign then form and pile up behind each other under the influence of the applied stress but repel each other at small distances by their own stress fields, forming the closely spaced rows of etch pits. The alternating bands may be tilt walls of opposite sign formed to relieve long-range bending stresses.

Alternatively a close examination of Figures 37, 39 and 40 suggests that the load-on substructure is composed of finite boundaries sweeping down the channels between the bands. The nature of these boundaries has not been established but their apparent ease of movement and the direction of their trace suggests that they are predominantly ((111)) tilt walls. As these tilt walls move down the channels they
appear to sweep them clear of dislocations and trail out rows of dislocations from each end. These trailing rows add to the thickness of the bands behind the moving boundaries. Another common feature of the moving tilt boundaries is that they appear to have segments parallel to (100) on one end but do not react in the same way on the other end. There is the possibility that the boundary alignment tends toward the (011) plane at the opposite end.

The rows of etch pits behind the moving boundaries suggest that these dislocations are associated with the moving boundaries. One type of association could originate by the long-range stress field of the passing boundaries causing localized dislocation multiplication and/or rearrangement on the alternate slip system. These dislocations move in this long-range stress field to either side of the boundary depending on the sense of their Burgers vector. The stress field of an isolated finite tilt wall can be as large as \( \sigma_{xy} = \frac{\mu b}{2(1-\nu)D} \) at a distance \( \lambda/2 \) on either side of the tilt wall\(^{(142,148)}\). This stress field will cause the individual dislocations on the alternate slip system to move in the same direction as tilt wall 2 of the same Burgers vector in Figure 102. For comparison, an isolated tilt wall with a misorientation angle of 1° has \( D = 156 \times 10^{-8} \) cm and \( \sigma_{xy \text{ max}} = \frac{\mu}{240} \) whereas a tensile stress of 900 psi at 1650°C causes a resolved shear stress on the (211)[\(\overline{1}11\)] and (211)[\(\overline{1}11\)] systems of \( \tau_a = \frac{\mu}{3.5 \times 10^4} \). The value of shear modulus was taken to be one-half the Young's modulus at 1650°C reported by Armstrong and Brown\(^{(136)}\). The misorientation angle for a sub-boundary with a maximum long-range stress equal to the applied stress is \( \Theta = 6.9 \times 10^{-3} \) degrees and \( D = 2.26 \times 10^{-4} \) cm. It is clear that most of the "finite"
sub-boundaries will have a long-range stress field larger than the applied stress provided that it is not completely relieved by other local sub-boundaries. At this early stage in creep it appears that the moving boundaries are finite to a degree and their arrangement is not so regular that their long-range stress field would be neutralized.

One can construct numerous geometrical models of the structure resulting from a moving finite tilt wall causing multiplication of dislocations within its long-range stress field. Some of these dislocations can be allowed to react with the dislocations in the tilt wall through a combination of climb and glide to create (100) segments at one end of the tilt wall and/or a segment parallel to the (011) plane made up of alternating (111) edge dislocations at the other end. These are the configurations observed in the substructures. It is assumed that the boundaries are moving in the direction in which they are bowed. This is the shape of a tilt wall under an applied stress if it is pinned at both ends. The motion of the finite tilt wall trailing dislocations is probably similar to a tilt wall pinned at the ends according to the earlier calculations of tilt boundary intersections.

Using these assumptions only two models provide an arrangement similar to the observed arrangement. Idealized schematic diagrams are presented in Figures 104 and 105. In Figure 104 the multiplication and/or rearrangement occurs behind the moving boundary rather than in front of it. This is consistent with the trails forming behind the boundary and the arrangement of the (100) boundary segments experimentally observed. If the multiplication or rearrangement of existing
FIGURE 104. DISLOCATION REARRANGEMENT AND MULTIPLICATION BEHIND MOVING TILT WALLS
FIGURE 105. DISLOCATION REARRANGEMENT AND MULTIPLICATION AT THE ENDS AND BEHIND MOVING TILT WALLS
dislocations occurs ahead of the boundary, the dislocations of a given Burgers vector glide to opposite ends of the boundary. An inspection of the drawing shows that the (100) segments will form at the opposite end of the moving boundary and form an acute angle with it. This is seldom observed.

One interesting feature of the model in Figure 104 is that the dislocations on the alternate slip system are moving against the applied stress, i.e., shortening the crystal. This may not be unreasonable since the local stress can be much greater than the applied stress as shown earlier. However there are large numbers of these dislocations in the narrow bands. This requires a large amount of slip on the other system to sustain a high tensile strain rate. It would also cause lattice rotation. The trails of dislocations from neighboring channels are of opposite sign on opposite sides of the narrow bands, tending to reduce the possibility of the accumulation of these dislocations through mutual annihilation. This can be visualized by picturing a succession of the configurations shown in Figure 104 placed side by side.

To avoid the disturbing feature of having so many dislocations moving against the applied stress one can also visualize dislocation multiplication and rearrangement occurring in the shear stress field at the end of the tilt wall. There the long-range stress field aids the applied stress but it is not as large as the stress field beside the tilt wall. This approach gives the arrangement shown in Figure 105. The dislocations move up to a position at the end of the tilt wall similar to the arrangements shown in Figure 104. In this case it appears that only one trailing boundary will remain no matter how many
sub-boundaries pass, since dislocations of opposite sign will move into the trailing boundary of the preceding moving sub-boundary and annihilate it.

If the dislocations multiply and rearrange in the stress field at the ends of the moving boundary ahead of the boundary, they will be of opposite sign. Again the arrangement of the (100) segments will not coincide with the most frequently observed arrangement.

It is evident from the above discussion that although the dislocation substructure has a definite pattern, this pattern cannot be easily rationalized at this point. However some interesting implications of the various suggested modes of deformation have been suggested by the load-on substructure. To clarify this issue further, study of the substructure developed during loading is required to establish the structure of the narrow bands, the moving sub-boundaries, and the relationship between the two.

Frequently there is a high density of dislocations immediately adjacent to a sub-boundary. At times this is accompanied by a very low dislocation density behind the boundary. These dislocations do not appear to be a part of the boundary. This was observed both in the etch-pitted structures as in Figure 46 and in the thin foils as in Figure 75. The implication is that sub-boundaries can be barriers to gliding and climbing dislocations even at high temperatures. A review of Li's calculations presented earlier suggests that they can present a barrier to the dislocations of both slip systems. For a fully pinned dislocation wall the minimum stress for penetration by individual dislocations is similar for both systems.
For penetration of a tilt wall by dislocations of the alternate slip system, the minimum resistance for glide is \( \sigma_{xy, \text{min}} = \frac{\mu}{48} \) as given in 2.a.(1) and for climb is \( \sigma_{n, \text{min}} = \frac{\mu}{193} \) as given in 2.a.(2) for a tilt wall having \( \theta = 1^\circ \) and \( D = 156 \times 10^{-8} \text{ cm} \). The applied shear stress and normal stress on edge dislocations of the \((211)[111]\) and \((211)[\overline{1}11]\) systems are \( \tau_a = \frac{\mu}{3.5 \times 10^4} \) and \( \sigma_n, a = \frac{\mu}{2.5 \times 10^4} \) respectively at 900 psi and 1650°C. However, in view of the mobility of the sub-boundaries the dislocations are not fully pinned. Probably the only pinning effect would originate from a random a[100] dislocation in the \((111)\) tilt wall. Dislocations of the same system as the moving sub-boundary are probably either absorbed or annihilated in it by a combination of climb and glide. Although unpinned tilt walls are much weaker barriers to dislocations than fully pinned walls\(^{(147,148)}\) more specific estimates of the dependence of the strength of the wall on the number of unpinned dislocations and the angle between the Burgers vectors of dislocations of other slip systems have not been made. However, the very fact that the tilt wall is a "stable" configuration implies that it will impose a resistance to a dislocation attempting to pass through it.

The pronounced edge character of the dislocations both within the subgrains and of course in the sub-boundaries raises the question, "What has happened to the screw components?" One can offer a possible rationalization as follows. From the wavy nature of the slip lines it is clear that cross slip is quite easy. The frequent cross slip of the screw segments would inhibit the operation of planar sources whereby large numbers of dislocations are generated within a narrow band.
Instead, dislocation multiplication would occur primarily by the Koehler\textsuperscript{151} double cross-slip mechanism which would tend to create a uniform distribution of slip dislocations throughout the crystal. This is what is observed here, and by Guiru and Pratt\textsuperscript{144} at lower temperatures. Apparently the screw components readily annihilate each other by cross-slip or move much more rapidly than edges. The propensity for cross-slip thus prevents large numbers of screw dislocations from accumulating in the slip plane so that boundaries parallel to the slip planes with a twist component, which are observed at low temperatures\textsuperscript{152,153}, do not form. In addition the improbability of two or more dislocations moving on the same slip plane against a tilt wall increases the apparent strength of the tilt wall.

Above 5% creep strain the sub-boundaries had begun to take on a well-defined appearance. The bands of subgrains are composed of long narrow subgrains separated by wide bands of large subgrains. As the strain increases and the structure coarsens there seems to be two concurrent processes modifying the substructure. The continued growth of the larger subgrains is accompanied by a widening of the bands of narrow subgrains. At 42% strain new long subgrains continue to form within the larger subgrains, presumably from the pile-up of tilt boundaries against the subgrain boundaries.

By comparing the montages of the \((001)\) planes at \(45^\circ\) to the tensile axis it appears that the coarsening of the structure with increasing creep strain may occur in the following sequence. Alternate bands of the bands of narrow subgrains present at low creep strain
collapse slowly, due to the drag of the a[100] dislocations, to form a single sub-boundary. These sub-boundaries then move to the nearest remaining band. This process is going on to some extent at all creep strains but the collapse of the narrow bands is most noticeable from 2 to 22% strain and the migration of the remaining boundaries is most noticeable from 5 to 42% strain. This correlates with the slip band observations in Figure 32. Below 5% strain the slip bands are relatively fine and close together, but at 5% strain the coarser slip band spacing is beginning to develop. This slip band spacing does not change noticeably at higher creep strains although one cannot be quantitative. The spacing is not exact over the gage length and not all the slip steps have the same displacement, making it difficult to determine whether a particular slip band has operated for some period of time and then stopped. However it appears that the coarse slip band spacing begins to establish itself at about 5% creep strain and these slip bands sustain very high displacements as creep progresses. It is difficult to describe the structure after 42% creep strain as alternating bands of narrow and coarse subgrains. However the heavily etched boundaries in the (100), longitudinal, view indicates that the same periodicity persists as creep strain increases. On the transverse ((001)) planes such a spacing is consistent with the substructure, although it is somewhat obscured by the inhomogeneity of the substructure.

It was not possible to verify directly that the coarse slip bands coincide with the bands of narrow subgrains. However the only substructural feature which one can associate with narrow, widely spaced
slip lines are the bands of narrow subgrains. The observation that the average spacing of the narrow bands of subgrains on the longitudinal (100) sections was similar to the average spacing of the coarse slip bands supports this suggestion.

At these high creep strains it is possible (see the etch pitting results) that high-angle (of the order of 10 to 12°) grain boundaries have developed. These could also help to concentrate slip into narrow bands, to produce apparently coarse slip bands by a grain boundary sliding type process. Some heavily etched boundaries become noticeable in the (100) views after only a 5% strain. However Laue back reflection patterns don't support the existence of misorientations larger than about 2.5° at this strain. After 42% strain there is a definite possibility on the basis of both the heavily etched appearance of some of the boundaries and the Laue back reflection patterns that large angle boundaries can be present. Grain boundary sliding may then contribute to the creep strain at high strains. However, this contribution, if present, must be quite small since the creep rate is still decreasing slightly at high creep strains. Significant grain boundary sliding would be expected to somewhat compensate for a slowly decreasing creep rate within the grains.

It would be meaningful to determine whether the concentration of slip into narrow, heavy slip bands can be rationalized in terms of the bands of narrow subgrains with which they appear to be associated. An idealized schematic of the creep structure after a relatively large creep strain is presented in Figure 106. It incorporates most of the
FIGURE 106. AN IDEALIZED SCHEMATIC DRAWING OF THE ARRANGEMENT OF THE SUBGRAINS INTO BANDS DURING CREEP

The bands have been drawn parallel only to (111) but in many instances there are segments of bands parallel to (111) and (111). The tensile axis is parallel to [011].
features observed in the substructure studies. The bands of narrow subgrains have been drawn parallel to the $(111)$ plane and the cross walls parallel to the $(\overline{111})$ plane only for simplicity. In reality the narrow bands form parallel to both planes and do interpenetrate to some extent. The vertical boundaries in the $(100)$ view are $(0\overline{1}1)$ network boundaries. These are parallel to the $(011)$ view and are not shown in this view. The heavy slip bands are presumed to coincide with the narrow bands of subgrains.

An enlarged schematic of the suggested structure in terms of tilt walls and dislocation geometry is shown in Figure 107. In this figure the tensile axis is horizontal and the structure viewed in this way resembles the arrangement on the $45^\circ$ transverse sections. In the bands of large subgrains, the dislocations of the $(2\overline{2}1)[1\overline{1}1]$ system move under the applied stress into the tilt walls along the narrow bands, or form new tilt walls parallel to the existing ones. Dislocations of opposite sign move to opposite sides of the wide band. The long-range stress fields of the nearest tilt wall segments in the narrow bands would tend to oppose this motion, but the proximity of the closely spaced tilt walls of opposite sign within the narrow bands would significantly reduce any long-range stress field extending out into the coarse subgrains. The distribution of the $\frac{a}{2}[1\overline{1}1]$ dislocations gliding across the coarse subgrains would be relatively uniform because of cross-slip, as discussed earlier. Although not shown in the drawing for the sake of clarity, dislocations of the $(211)[\overline{1}11]$ system could also be moving within the wide band. These could interact with the
FIGURE 107. A POSSIBLE ARRANGEMENT OF THE DISLOCATIONS IN THE SUBGRAIN BOUNDARIES AND THEIR MOTION DURING CREEP
dislocations in the tilt walls to form a(100) dislocations. These would slow the glide movement of the tilt walls, postpone their mutual annihilation, and allow the formation of a succession of tilt walls. An inspection of the drawing verifies that they would be of the appropriate sign to do this. The (111) cross walls would also contribute an internal stress which would alternately aid and oppose the applied stress. However at higher strains many of these sub-boundaries appear to have a high misorientation angle and would therefore contribute less to the internal stress.

Inside the narrow bands where the (111) tilt wall segments of opposite sign are close together the long-range stress fields of the tilt walls reinforce each other and the externally applied stress. Of course these long-range stress fields will not be nearly as large as those calculated for isolated tilt walls, but the structure is not regular and it seems quite reasonable to assume that they do introduce an internal stress field. In addition it need not retain a large fraction of its ideal value in order to be significant in comparison with the applied stress. This localized, enhanced stress field will tend to channel the motion of dislocations of the alternate, (211)[111] system down the length of the long subgrains. The velocity and perhaps the density of the mobile dislocations will be larger in this region than in other parts of the crystal. Hence, a great deal of slip on the one system will be concentrated within the narrow bands.

An inspection of the etch pitting micrographs in Figures 58 and 65 suggests that not only individual dislocations move down these channels, but that short tilt boundaries form across the narrow
subgrains, and these also move along the channel by a process of climb and glide. The geometry is clear in Figure 107. It can be seen that they can stop for a short time and react at one end at the "T" junction (see Figure 103), forming a segment of (100) tilt wall to relieve the local stress concentrations. After some period of time it may be released from this intersection and move on down the channel until it again comes to rest and reacts. It may annihilate by meeting a tilt wall of opposite sign or dissipate itself by using up most of its dislocations in forming (100) tilt walls. This arrangement is quite evident in Figures 58 and 59.

These segments of (100) tilt wall may also inhibit the motion of other sub-boundaries or dislocations. As shown in Table 9 there is no pressure exerted on the boundary by the applied stress. Therefore any motion of these tilt walls or the individual a[[100]] dislocations must be caused by interactions with other dislocations. Penetration of the (100) tilt walls may also be more difficult because of this. Li\(^{(147)}\) showed that penetration of a tilt wall by individual dislocations is easier if the tilt wall dislocations also move slightly under the applied stress.

The numerous (100) segments in the (111) tilt walls creates an average trace of the bands lying between (111) and (211) which is experimentally observed. Some of the waviness in the slip bands may not result from cross slip, but may be a result of neighboring segments of advancing edge dislocations climbing different heights as they move down the channels and interact with the changing subgrain structure. The preferential channeling of slip on one system and the uniform
distribution of slip on the alternate system would explain the absence or presence of fine slip lines only on the alternate system in a section of crystal where heavy slip bands on the other system are present and little lattice rotation has occurred.

It should be pointed out that the shear stress on the $\{\overline{2}11\}[\overline{1}11]$ system is also enhanced in the center of the narrow bands. Dislocation movement in this region would annihilate the successive tilt walls in these bands from the inside out. What probably happens is a continuous annihilation and reformation of the long tilt walls in the narrow bands. The etch pitted figures at high strain show that in some regions along the length of a band annihilation may be nearly complete. At other points, new segments of bands are forming parallel to the existing bands or parallel to the alternate system, creating an interpenetration of these bands of narrow subgrains. As the creep strain increases one should observe a continuous initiation of heavy slip bands parallel to both systems in the same region of the crystal.

The reasons for the establishment of a coarse slip band spacing during high temperature creep is still not known. Figure 31 shows that at low stresses this spacing can be on the order of a millimeter or slightly smaller. It would not appear to be related to long-range stress field interactions because of these relatively long distances. It may have some connection with a balance established between the diffusion distances and climb forces on the dislocations under a given applied stress or a critical distance-time relationship between dislocation glide and a recovery process.
It is clear that internal stress fields will persist to some degree in the structure throughout creep. As shown in Figure 107 the continuing formation and motion of tilt walls will create configurations similar to Figures 101 and 103. These arrangements do contain finite tilt walls having long-range stress fields before they are partially relieved by further climb and glide.

*Rate-controlling mechanisms*

It is not possible to definitely specify the rate-controlling mechanism during creep in molybdenum single crystals. Mechanisms could be constructed which would be compatible with the structure and activation energy for self diffusion, but there would be no way to differentiate between them at this time and little would be gained. The creep substructures suggest that it is highly probable that several processes are contributing to the creep rate in varying degrees. These will be discussed briefly later. Although no models will be presented for a creep mechanism it is possible to point out certain current creep theories which do not apply to molybdenum single crystals and perhaps do not apply to some annealed, unalloyed polycrystalline metals either.

The high edge character of the dislocations in the creep substructure precludes the motion of jogged screws \(^{80,91,94}\) as a rate-controlling mechanism. If the motion of jogged screws controlled the rate, there would be a much higher proportion of screw dislocations in the creep substructure. The observed directionality of the dislocations favors edge dislocations. Transmission electron microscopy of thin foils of crept polycrystalline bcc metals have also shown a noticeable
preferred alignment of dislocation line directions, but no determination of the character of these dislocations was made\(^{(7,8)}\).

The absence of any sign of glide pile-ups in the substructure, or, for that matter, the absence of any sign of slip bands in the etch-pitted structure suggests that the climb of edge dislocations out of dislocation pile-ups\(^{(85,86)}\) is not applicable either. This is in accord with the noticeable ease of cross slip which inhibits the operation of coplanar sources and therefore drastically reduces the formation of coplanar arrays of edge dislocations and edge dislocation pile-ups. One of the original creep theories still used to describe the strain dependence of the creep rate during high temperature creep is the concept of having both work hardening and recovery taking place concurrently during creep. The relative rate of either process may be dependent on creep strain\(^{(78,79)}\). The theory depends on the slope of a tensile stress-strain curve to obtain the work hardening parameter. This requires that the dislocation density be increasing with increasing strain since the increase in the flow stress with increasing strain has been related to an associated increase in the dislocation density\(^{(154,155)}\). In fact McLean\(^{(95)}\) specifically relates the decrease in creep rate during primary creep to a decrease in the mesh size of the three-dimensional dislocation network. This requires that the free dislocation density increase with creep strain.

In contrast, the free dislocation density decreases continuously with creep strain. The subgrains coarsen and the subgrain boundaries take on a more recovered aspect with increasing creep strain. The total amount of any work hardening appears to occur only during loading.
Immediately after loading only the extent of recovery increases with increasing creep strain. Hence work hardening does not cause the decreasing creep rate during primary creep.

The actual rate-controlling mechanism can be a combination of many different dislocation-dislocation interactions, dislocation-point defect interactions and sub-boundary-dislocation or sub-boundary point defect interactions. The substructural dependence on temperature and stress is still not known quantitatively, and the more important of the above types of mechanisms cannot be determined. Some possibilities will be discussed briefly.

Figures 19 and 67 suggest that the decreasing creep rate with increasing creep strain may be related to the decreasing free dislocation density. If the mobile dislocation density were a relatively constant fraction of the free dislocation density and factors such as the dislocation velocity were relatively insensitive to creep strain, then there may be a similarity between strain dependence of the etch pit density and the strain rate\(^{(8)}\). Figure 108 shows this comparison by normalizing the strain rate and etch pit density to their values at high creep strain where they aren't changing very rapidly. Their relative strain dependence is quite different. Although this eliminated a direct dependence, it is quite possible that a part of the strain dependence of the creep rate is related to the decrease in the free dislocation density.

This decrease in free dislocation density as the creep strain increases is puzzling. Evidently the multiplication rate fell below
FIGURE 108. A COMPARISON OF THE RELATIVE DEPENDENCE ON THE CREEP RATE AND ETCH PIT DENSITY ON CREEP STRAIN
the annihilation rate as soon as loading was complete. Perhaps the
ease of cross slip frequently causes the screw segments to annihilate
each other. In doing so, they effectively remove themselves as
Koehler\(^{(151)}\) sources. This may occur at a sufficiently high rate in
the absence of such constraints as increasing load or constant strain
rate such that there is a continuous decrease in the free dislocation
density until it reaches a certain low level. Under conditions of
enforced deformation such as a rising load or constant strain rate,
the crystal would be forced to provide sufficient mobile dislocations
to match these constraints.

Possible rate-controlling mechanisms may be related to the
diffusion of vacancies between intersections of opposite sign like the
intersection in Figure 103, or perhaps to the distance between (100)
segments of opposite sign on opposite sides of the narrow bands in
Figure 107. Each of those intersections requires a net flux of vacan-
cies either to or away from it in order to relieve the stress concen-
trations by climb. The climb of a[100] dislocations in (111) tilt
walls may control the rate of glide of the tilt walls or vice versa if
the stress is not sufficient for the tilt wall to pull free of the
pinning dislocations\(^{(148)}\). These types of approach appear to be most
attractive at present. The success of the Dorn \(\Theta\) parameter in normal-
izing the creep curves at a constant stress level indicates that the
structure is identical at the same strain independent of the tempera-
ture. This follows on the assumption that this parameter has the same
meaning in these molybdenum single crystals as it does in aluminum\(^{(156)}\).
It also indicates that the strain rates at any given strain differ only by the temperature dependence of the $\theta$ parameter. The temperature dependence of the $\theta$ parameter is equal to the activation energy for self diffusion which suggests that the temperature dependence of the creep rate may well be a result only of the temperature dependence of the flux of vacancies between structural features a constant distance apart.

It is also clear from the structure that one must not only consider the climb of isolated edge dislocations within the subgrains, but one must also consider the climb of groups of edge dislocations, for example, a tilt boundary relaxing by climb. In these configurations the climbing dislocations probably cannot be treated as isolated sources and sinks but must be treated as overlapping sources and sinks. In addition, creep theory should not include the substructure as a uniform distribution of defects. It is quite nonuniform and the conditions for developing and maintaining this nonuniformity should be included in the theory since it is possibly quite important in determining the creep behavior or absolute creep rate.
CONCLUSIONS

1. \(((112))[[111]]\) slip is the common mode of deformation at elevated temperatures in molybdenum single crystals of the [011] orientation.

2. The activation energy for creep is \(Q_c = 107\) kcal/mole. This indicates that the creep rate is diffusion controlled.

3. Edge dislocations predominate in the creep substructure. This observation excludes the movement of jogged screws as a rate controlling mechanism.

4. Many long edge dislocations having \(b = a[100]\) on a \((\overline{1}11)\) "glide" plane are present after creep at elevated temperatures. It is not known whether they are glissile. Therefore it is not certain whether all of them have been formed by reactions between \(a/2 [[111]]\) dislocations or whether there is some independent multiplication of the \(a [[100]]\) dislocations.

5. The \(a[100]\) dislocations formed by the reactions of the primary \(a/2 [[111]]\) dislocations have no forces imposed on them from the external stress. They can move only through interactions with other dislocations.

6. It is inferred that \(((111))\) tilt walls move by glide at elevated temperatures. Therefore gliding tilt walls do contribute to the creep strain.
7. Sub-boundaries react to relieve long-range stresses.
   a. (100) tilt walls are formed at the intersection of a
certain configuration of ((111)) tilt walls.
   b. One can have a "stable" "L" intersections between two
((111)) tilt walls or between a ((111)) and a ((100))
tilt wall.

8. The presence of long-range interactions between individual
dislocations and tilt walls may be quite important during creep. This
has the implication that much of the dislocation movement occurs in
favorable regions of the structure and an "averaging" of the structure
for theoretical purposes may be misleading.

9. The edge dislocations on both the primary ((112))[[111]]
and ((110))[[111]] slip systems have a high normal to glide force ratio
when the stress axis is parallel to a [[110]] direction. At elevated
temperatures where the climb mobility may be high, climb of mobile edge
dislocations out of the glide plane may be important.

10. The creep strain is quite nonuniform. Both very localized
concentrated slip and uniformly distributed fine slip occurs. This is
related to the dislocation substructure formed during creep.

11. Strain dependent work hardening does not cause the decrease
in creep rate during primary creep. The creep rate decreases as the
substructure "recovers."

12. Steady state creep is not observed up to high creep strains.
This may be due to the fact that the creep structure had not yet
reached an equilibrium arrangement.
13. The [100] and [111] orientations are unstable during tensile deformation at elevated temperatures and they form necked regions along the gage length during creep.
RECOMMENDATIONS FOR FUTURE WORK

1. The dislocation substructure, the stress dependence, and the activation energy of the creep rate should be studied over a wide temperature range extending below 0.5 Tm. The appearance of the unique structural features developed during creep could then be correlated directly with the creep properties. This is a temperature region in which the creep behavior of bcc refractory metals does appear to be changing.

2. The stress dependence of the creep rate must be investigated in detail. This should be done by
   a. Determining the strain dependence of the creep rate at different stresses.
   b. Measuring the stress dependence of the creep rate by the stress change technique. Do this at different creep strains.
   c. Characterizing the dependence of the dislocation substructure on stress.
   d. Learning the manner in which the substructure established at one stress changes to that representative of another stress after a stress change.

3. A careful study of the relative angular misorientations of neighboring subgrains and the relation of the misorientations to the banded substructures must be made and related to the strain dependence
of the creep rate. This would aid in assessing the influence of the sub-boundaries themselves on the creep rate.

4. It should be determined whether the a[100] dislocations are glissile.

5. The influence of a pre-strain on the subsequent creep behavior and substructure should be examined to provide more information on the relation between the strain dependence of the creep rate and the substructure.

6. The grain size dependence of the creep behavior and substructure in polycrystalline molybdenum must be investigated. In this way the spectrum of creep properties and substructure from single crystal to fine grained polycrystals may be characterized. This could provide very useful information regarding the operative creep mechanisms and their relation to the substructure.
APPENDIX A

COORDINATE TRANSFORMATION IN TERMS OF $\lambda$ AND $\phi$

The transformation $a_{ij}$ directly in terms of $\lambda$ and $\phi$, the usual angles given for a slip system referred to the stress axis of a specimen, is more unwieldy than that in terms of $\theta$ and $\phi$. It is presented below.

$$
a_{ij} = 
\begin{bmatrix}
  a_{11} & a_{12} & a_{13} \\
  a_{21} & a_{22} & a_{23} \\
  a_{31} & a_{32} & a_{33}
\end{bmatrix}
$$

\[
\begin{bmatrix}
  (1 - \frac{\cos^2 \lambda}{\cos^2 \phi})^{1/2} & \frac{\cos^2 \lambda}{\sin \phi} & \cos \lambda \\
  -\frac{\cos \lambda}{\sin \phi} & \cos \phi \left(1 - \frac{\cos^2 \lambda}{\sin^2 \phi}\right)^{1/2} & \sin \phi \left(1 - \frac{\cos^2 \lambda}{\sin^2 \phi}\right)^{1/2} \\
  0 & -\sin \phi & \cos \phi
\end{bmatrix}
\]

(44)
APPENDIX B

SPARK MACHINING DAMAGE

In many cases the damage introduced into the surface of a piece being machined by the spark-discharge method is considerable even though the tool does not touch the work piece. This is an important point since spark machining has been widely adopted for cutting and shaping single crystals for fundamental studies. However, since the tool does not touch the specimen, the deformation is not propagated through the specimen by bending or compression forces exerted by the machining tool and the damage is confined to a surface layer. This simplifies the preparation of "strain free" single crystals. If the depth of the surface damage is recognized and this damaged surface layer is completely removed then one can produce good single crystal specimens shaped by spark machining. The critical factor is the determination of the depth of damage. For soft metals such as copper and silver this can be as high as 1.5 mm (157-159) under severe machining conditions while for harder metals such as the refractory metals, it would probably be much less.

The depth and type of damage produced during spark latheing molybdenum single crystals was determined for each range of spark energy on the Servomet, a commercially manufactured spark machine. The settings varied from Range 1, which gives the highest machining rate and
creates a heavily pitted surface, to Range 7 which gives the lowest machining rate and produces a surface having a satin finish of about 10 \( \mu \) roughness. A tungsten tool was used and the bath was mineral oil at a temperature of 87 ± 3°C. The crystal was made from purified stock and had a low carbon level of the order of that used in the creep studies.

The photograph on the right in Figure 109 shows a single crystal rod of \([100]\) orientation with successive increments along its length lathed on progressively higher range settings. The strip photographs to the left correspond to sections cut through the lathed surfaces at about 65° to the rod axis.* Note the numerous surface cracks caused by the latheing operation. A detailed determination of the plane of these cracks was not made, but their traces are consistent with the \((100)\) cleavage plane as observed by Beardmore and Hull\(^\text{159}\). As the energy of the spark or the machining rate is decreased the propensity for crack formation decreases accordingly. Figure 110 shows the variation of the crack depth in Figure 109 with the range setting. A small correction for the angle of the sectioned surface is included so that the depths are those normal to the machined surface.

Although a direct measurement of the depth of penetration of spark-impact induced dislocations was not possible, an estimate of this depth can be made from etch-pitting surfaces containing cracks.

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*This was done in the event some way could be discovered to preserve the edges of the surface during electropolishing so that the true depth of damage could be assessed by etch pitting fresh dislocations on the \((421)\) plane. This effort was abandoned after several unsuccessful attempts.
FIGURE 109. THE APPEARANCE OF THE SPARK LATHED SURFACE FOR DIFFERENT MACHINING RATES IN A MOLYBDENUM SINGLE CRYSTAL

The numbers indicate the range setting on the Servomet. The machined surface in the sectional view was given a copper strike and nickel plate to preserve the edge during preparation.
FIGURE 110. MAXIMUM DEPTH OF CRACKS PRODUCED IN MOLYBDENUM SINGLE CRYSTALS BY SPARK LATHEING AT INCREASING RANGE SETTINGS ON THE SERVOMET.

The bath was mineral oil at 87 C and a tungsten tool was used.
Figure 111 shows the damage associated with the tips of cracks on the surface of a sphere after latheing, electropolishing, and etch pitting. The cracks were introduced on Range 1, the severest condition, after which lighter cuts were used to smooth the surface and 2 to 5 mils were removed by electropolishing. Hence the surface is near the maximum crack depth and the dislocation arrays seen emanating from the crack tips at the surface are probably representative of the arrays extending from the bottom of the crack down into the crystal. The distance the dislocations have moved from the crack tips can only be approximated and a conservative estimate is that the maximum distance is 5 to 7 μ (2-3 mils) at the most. For less severe machining conditions such as Ranges 4 and 5, this distance is probably much less, perhaps on the order of the ratio of the crack propagation depths. This would suggest a maximum damaged layer (including cracks and fresh dislocations) less than 6 μ (2.5 mils) deep for Range 4 and less than 2 (≈1 mil) deep for Range 5. Although the bath liquid and temperature is different from that used in machining the creep specimens (kerosene at room temperature), it is probable that there is little difference between the two conditions. There was no visible surface cracking on the creep specimens after latheing on Range 5 as one would predict from Figures 110 and 111 and the electropolished surfaces did not show any indentations which would be related to surface cracks.
FIGURE 111. DISLOCATION ARRAYS FORMED AT CRACK TIPS BENEATH THE LATHED SURFACE OF A CRYSTAL
It is often difficult to establish a basis of comparison between the specimens one is using for a given experimental program and that of other investigators. Chemical analysis for interstitial content is somewhat uncertain and the comparative perfection of single crystals is difficult to assess. To this end the yield stress in tension at room temperature was measured. The stress-strain curves exhibited the rounded upper yield point often observed in [110] oriented bcc single crystals and is shown in Figure 112. Table 14 lists the proportional limit and upper yield stress for the three specimens tested here and other results on [011] oriented single crystals taken from the literature. Our crystals compare quite favorably with the crystals used by other investigators.
FIGURE 112. TENSILE STRESS-STRAIN CURVE AT ROOM TEMPERATURE


<table>
<thead>
<tr>
<th>Temperature °K</th>
<th>Proportional(a) Limit, psi</th>
<th>Upper Yield Stress psi</th>
<th>Lower Yield Stress psi</th>
<th>Strain Rate (sec)^{-1}</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>300</td>
<td>37,622</td>
<td>41,446</td>
<td>40,336</td>
<td>1.6 x 10^{-4}</td>
<td>This investi-</td>
</tr>
<tr>
<td></td>
<td>40,168</td>
<td>44,247</td>
<td>--</td>
<td>1.6 x 10^{-4}</td>
<td>gation</td>
</tr>
<tr>
<td></td>
<td>43,662</td>
<td>46,573</td>
<td>43,265</td>
<td>1.6 x 10^{-4}</td>
<td></td>
</tr>
<tr>
<td>300</td>
<td>46,000</td>
<td>--</td>
<td>--</td>
<td>4.2 x 10^{-4}</td>
<td>(162)</td>
</tr>
<tr>
<td>293</td>
<td>--</td>
<td>32,000</td>
<td>--</td>
<td>6.5 x 10^{-5}</td>
<td>(144)</td>
</tr>
<tr>
<td>293</td>
<td>--</td>
<td>49,000</td>
<td>47,000</td>
<td>6.5 x 10^{-3}</td>
<td>(144)</td>
</tr>
</tbody>
</table>

(a) Stresses are given as tensile stresses.
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