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THE EFFECT OF IMPURITY AND FOREST DISLOCATIONS ON THE FLOW STRESS OF COPPER

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THE EFFECT OF IMPURITY AND FOREST DISLOCATIONS ON THE FLOW STRESS OF COPPER

by

P. C. J. Gallagher*

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*Inorganic Materials Research Division of the Lawrence Radiation Laboratory, University of California, Berkeley and formerly at Cavendish Laboratory, Cambridge.

I. INTRODUCTION

The relative importance of several possible factors in determining the flow stress throughout the stress-strain curve of face centered cubic metals is still rather uncertain. In the present work an investigation has been made of the relative magnitudes of τ_s, the short range, thermally activable part of the flow stress, and of τ_g, the long range stress, for copper single crystals at room temperature. In an attempt to perform definitive experiments so as to identify the magnitudes of τ_s and τ_g characteristic of particular interactions, known impurity contents and dislocation distributions have been introduced to the crystals. By identifying the values of τ_s/τ_g characteristic of impurity-dislocation, and of forest-primary dislocation interactions it is possible to establish the regions of the stress-strain in which these reactions play an important role.

Evidence exists for the effect of impurity on both τ_s and the total flow stress at low stresses. In Haasen's (1958) temperature cycling experiments on nickel crystals considerably larger values of τ_s were found for specimens of only commercial purity. Comparison of the critical resolved shear stress, τ_o, for copper of 99.999% and 99.98% purity (Schüle et al, 1962) shows that impurity affects the onset of macroscopic flow considerably. Observations by the present author (unpublished work) suggest that the initial flow stress may in addition be affected by the distribution of impurity. A copper single crystal annealed close to the melting point for a number of hours showed a lower initial flow stress when tested subsequently at room temperature

if, prior to cooling, it was deformed in tension to 1 or 2 percent strain. More quantitative results reflecting what is thought to be the effect of oxygen on the magnitude of τ_s are shown in this paper. To investigate τ_s/τ_g as a function of purity, strain rate change tests have been performed on copper single crystals of easy glide orientation whose impurity level ranged from 5 to 200 p.p.m.

Basinski and Basinski (1964) have shown the sensitivity of the total stress to forest dislocations in an initial flow stress experiment on copper single crystals. The etch-pit counts made in their experiment showed that torsional deformation applied to one of the two specimens tested increased its forest dislocation density considerably, leaving the density in the principal slip plane unaltered; furthermore, the increased flow stress could be correlated with the increase in forest dislocation density. As will be discussed later, however, the increase in flow stress from 128 gms/mm^2 to 295 gms/mm^2 due to the forest dislocation density (N_f) increasing from $5.6.10^5/cm^2$ to $3.3.10^6/cm^2$ suggests that Saada's relationship $\tau_g(f) = 1/5\ Gb(N_f)^{1/2}$ must be modified. Paxton and Cottrell (1954), Rebstock (1957) and Holt (1959) have also investigated the effect of incremental torsional deformation interrupting a tensile test, and have shown that the total flow stress is increased. Seeger et al (1957) measured both τ_s and τ_g in stage II after performing tests intermittently in tension and torsion. These workers found that τ_s/τ_g remained unchanged following an increase in forest dislocation density arising from the torsional strain, and interpret this as resulting from an increase in the density of Lomer-Cottrell barriers, rather than from

primary-forest dislocation interactions. Seeger et al (1957) interpret τ_s/τ_g in stage I as characteristic of forest dislocation interactions, while other workers (Young 1962, Basinski 1959, Basinski and Basinski 1964) consider that impurity effects make a substantial contribution.

In the present work a strain rate change by a factor of 10 led to a stress increment $\Delta\tau_s$, which is proportional to τ_s. Since a knowledge of this proportionality factor involves a specific flow stress theory, and a considerable part of the interpretation of the present results depends only on the relative magnitudes of τ_s/τ_g, the values of $\Delta\tau_s/\tau$ ($\propto\tau_s/\tau$) are tabulated. To determine τ_s/τ_g characteristic of forest-primary interactions τ and $\Delta\tau_s$ have been measured by strain rate changes both before and after an increment of torsional deformation was introduced during a tensile test in the easy glide region. As Paxton and Cottrell discussed in their paper, and as Basinski and Basinski have shown by etch-pit measurements, for a single crystal of easy glide orientation the torsional stress causes an increase in dislocation density only on the secondary planes leading to forest dislocations intersecting the principal tensile glide plane. The function $\delta(\Delta\tau_s)/\delta(\tau)$ (where $\delta(\Delta\tau_s) = (\Delta\tau_s$ (after torsion) $- \Delta\tau_s$ (before), for x10 strain rate changes, and $\delta(\tau)$ is the total tensile flow stress difference caused by the increment of torsional deformation.) is thus a measure of the relative increase in τ_s and τ_g due to forest dislocations interacting with primary dislocations. The ratio $\delta(\Delta\tau_s)/\delta(\tau)_{(forest)}$ may be compared with $\Delta\tau_s/\tau$ for copper crystals of various purities in the easy glide region, and also with the ratio $\Delta\tau_s/\tau$ obtained from a x10 strain rate change during stage II of the stress-

strain curve. It is thus possible to decide whether

1) $\Delta\tau_s/\tau_{(\text{easy glide})}$ is due to forest-primary interactions in which case $\delta(\Delta\tau_s)/\delta(\tau)_{(\text{forest})}$ should equal $\Delta\tau_s/\tau_{(\text{easy glide})}$.
2) $\Delta\tau_s/\tau_{(\text{stage II})}$ is due to forest-primary interactions in which case $\delta(\Delta\tau_s)/\delta(\tau)_{(\text{forest})}$ should equal $\Delta\tau_s/\tau_{(\text{stage II})}$.
3) $\Delta\tau_s/\tau_{(\text{easy glide})}$ is impurity dependent, for in this case markedly different ratios will be obtained as the impurity level is altered.

In addition to the information derived specifically to answer the above questions, the present results afford detailed data on the variation of $V\tau$ with stress (V is the apparent activation volume), and the applicability of the Cottrell-Stokes Law.

II. RESULTS OF STRAIN RATE TESTS FOR SPECIMENS OF DIFFERENT PURITY

Unless otherwise indicated the results reported are for specimens which were deformed at 295°K. Using extension rates of 0.01 cm/min and 0.1 cm/min for the 5 cm long specimens, and a fast chart speed, more than 20 strain rate cycles could be completed in the easy glide region which corresponded to 10% tensile shear strain. The impurity was introduced to the specimens during an anneal close to the melting point in an evacuated chamber containing a vapour pressure of the impurity to be introduced. During this procedure the specimens were held in the tensile gripping system which was to be used so that no extraneous deformation resulted prior to test. The initial flow stresses of the specimens ranged from 76 gms/mm^2 to 137 gms/mm^2, but no correlation has been attempted between τ_o and impurity content since the specimens underwent different annealing treatments.

Figures 1 and 2 are for copper crystals both of which contained 150 p.p.m. impurity as determined subsequently by spectographic analysis. The Cottrell-Stokes Law may be expressed by saying that it is strictly applicable in those regions for which $V\tau$ is constant. We see, therefore, that only for stresses $\gtrsim$ 2.5 kg/mm^2 is this condition met (corresponding to a shear strain $\gtrsim$ 30%). The constant value of $V\tau$ which is reached at high stresses is 7.0 ± 0.2 e.v., corresponding to $\Delta\tau_B/\tau$ = (8.4 ± 0.3)/1000 for a x10 strain-rate change. By contrast, Fig. 3 shows the results of a similar experiment on a silver single crystal, for which the Cottrell-Stokes Law is obeyed for $\tau \gtrsim$ 0.75 kg/mm^2, at a shear-strain $\gtrsim$ 15%. The mean value of $V\tau$ = 6.54 ± 0.05 e.v., and thus for a x10 strain-rate

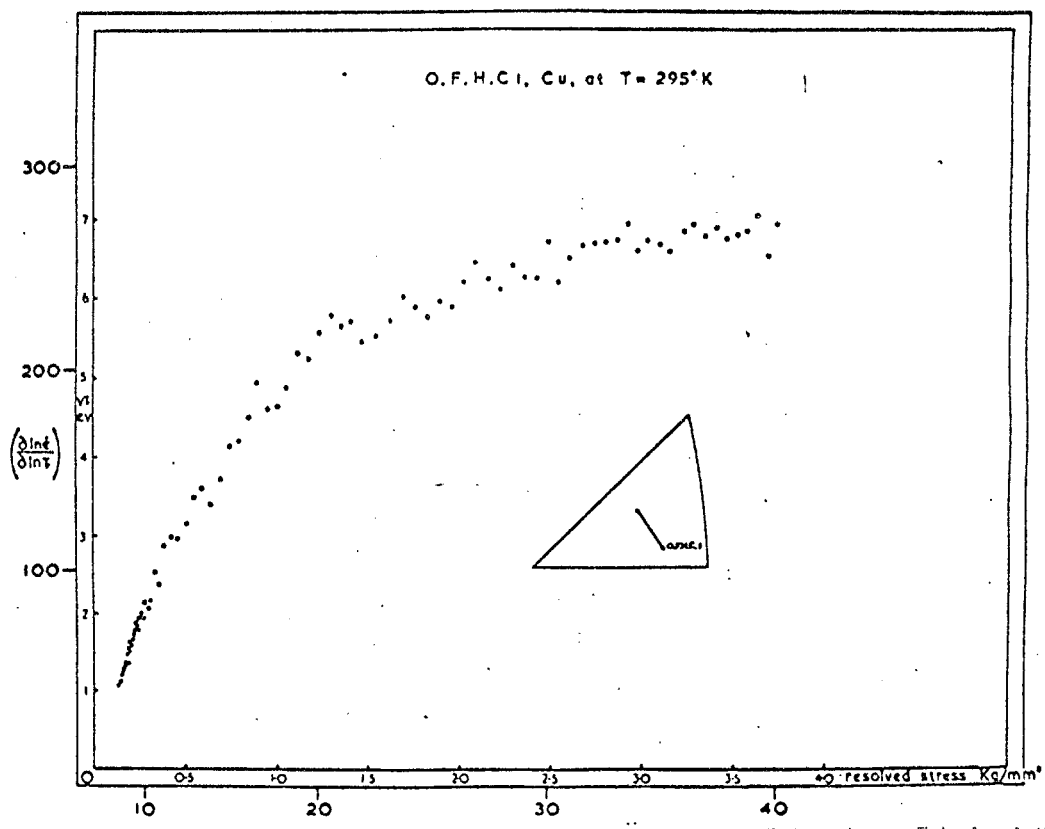
O.F.H.C I, Cu, at T = 295°K
300
200
100
$\left(\frac{\partial \ln \dot{\varepsilon}}{\partial \ln \tau}\right)$
0·5
1·0
1·5
2·0
2·5
3·0
3·5
4·0 resolved stress Kg/mm²
10
20
30
40

Fig 1

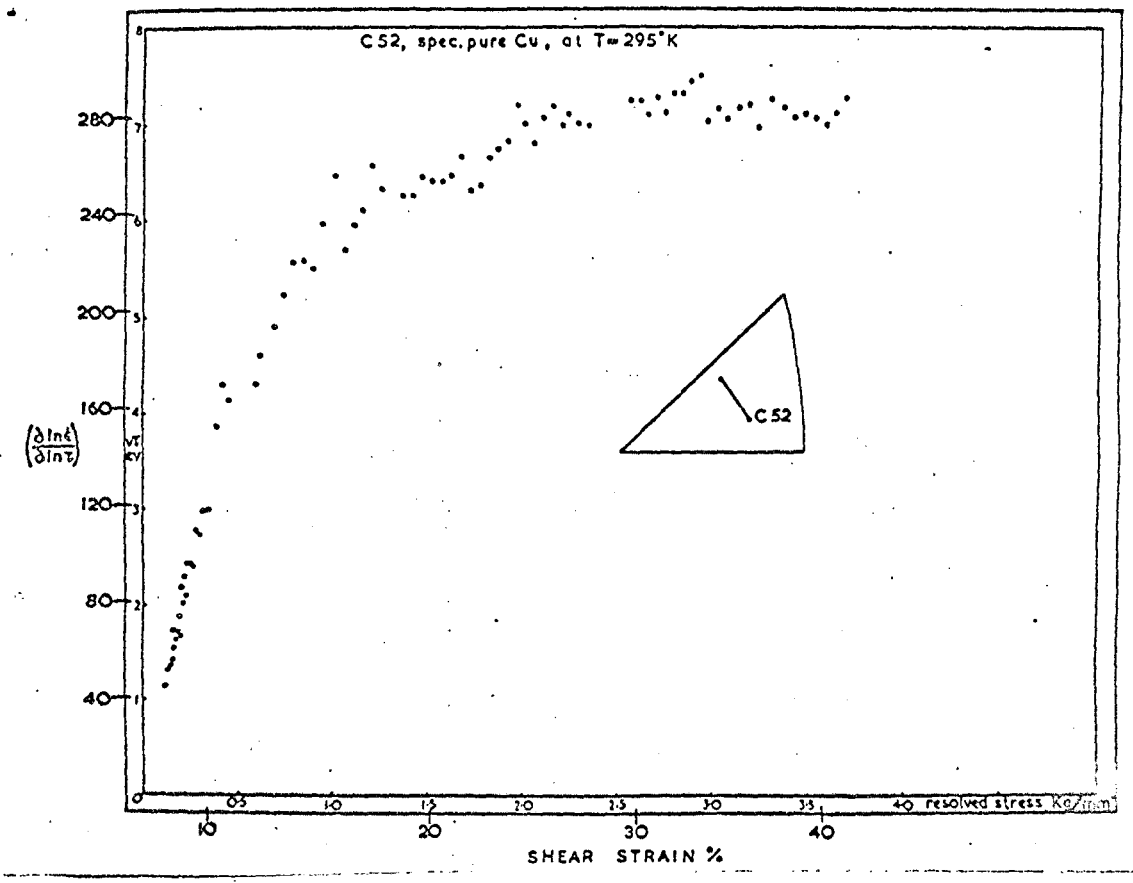
C52, spec. pure Cu, at T=295°K
280
240
200
160
120
80
40
$\left(\frac{\partial \ln \dot{\epsilon}}{\partial \ln \tau}\right)$
VT
KV
C52
0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0 resolved stress Kg/mm²
10
20
30
40
SHEAR STRAIN %

Fig 2

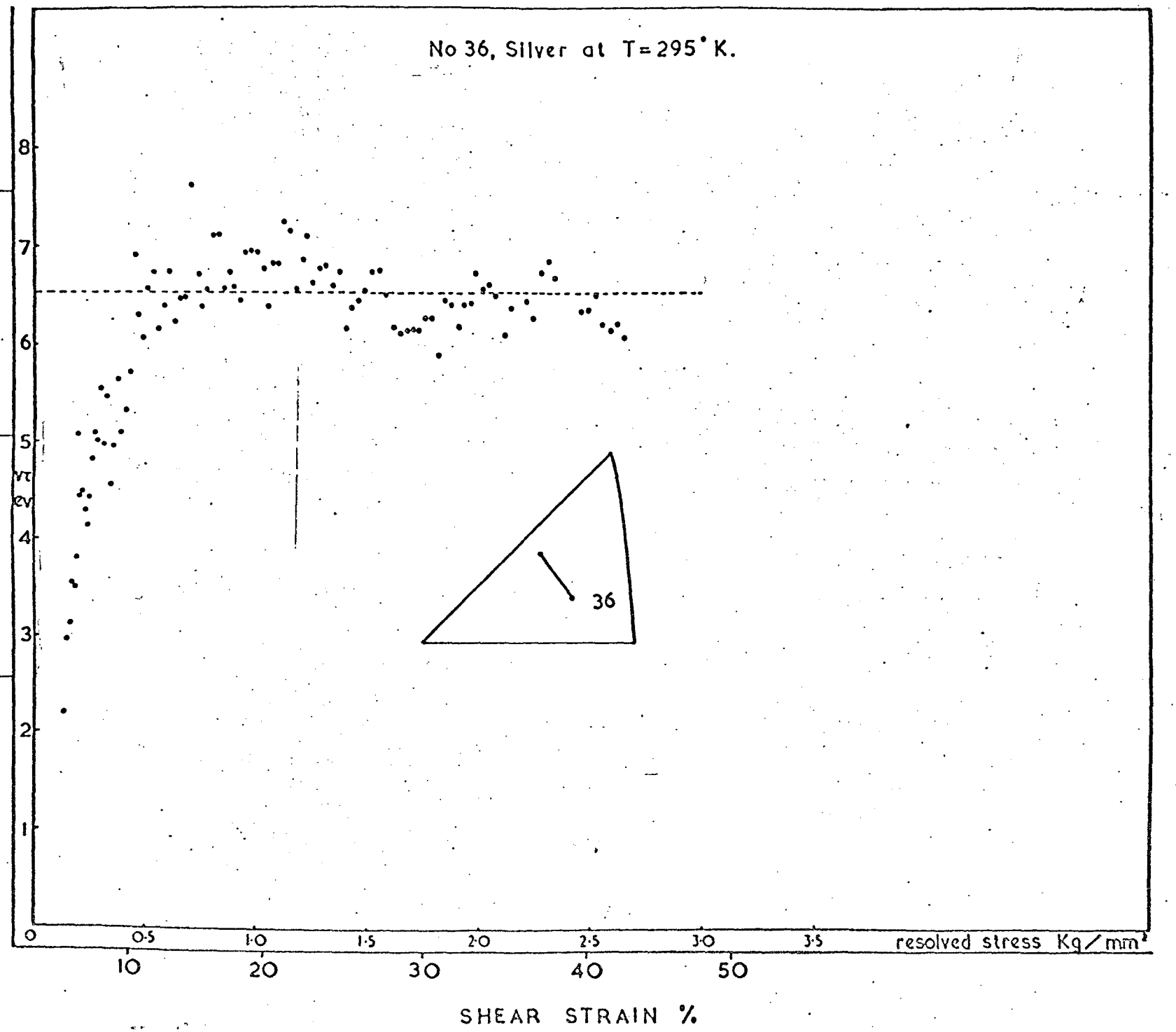
No 36, Silver at T=295° K.
300
200
100
8
7
6
5
4
3
2
1
$\left(\frac{\partial \ln \dot{\varepsilon}}{\partial \ln \tau}\right)$
36
0
0·5
1·0
1·5
2·0
2·5
3·0
3·5
resolved stress Kg/mm²
10
20
30
40
50
SHEAR STRAIN %

Fig 3

change at high stresses $\Delta\tau_s/\tau = 7.8/1000$. The point at which the law is obeyed does not appear to correlate with a prominent feature in the stress-strain curve. This is perhaps indicative of stage II becoming gradually dependent upon one major flow stress determining factor, while at lower stresses, even well into stage II, numerous other reactions make contributions whose magnitudes depend upon the internal structure of the particular crystal. Although the present results on copper show that the stress at which the law is obeyed is closely the same as τ_3, the possibility of this being a general rule is invalidated since for silver the law holds for stresses considerably less than τ_3.

A striking feature of the results is that in stage I the magnitude of $\Delta\tau_s$ (and therefore, τ_s) is constant irrespective of the specimen's impurity content in the range investigated. This result is at variance with the findings of Thornton, Mitchell and Hirsch (1962) who found that $\Delta\tau_s/\tau$ was constant, i. e. Cottrell-Stokes Law was obeyed. Seeger (1963), however, using an incremental loading method found that the activation volume was constant during easy glide in agreement with the present results. Figure 4 shows the results of the present work, together with those of Seeger on a crystal of unreported purity (from Seeger's values of V, the activation volume, derived from incremental loading the value of $\Delta\tau_s$ has been calculated which would have been measured if the results had been derived from x10 strain rate changes). The discrepancies in the published data on the Cottrell-Stokes Law probably reflect the sensitivity of τ, and τ_s particularly, to inhomogeneities in dislocation distribution, general crystal perfection, and the nature, content, and

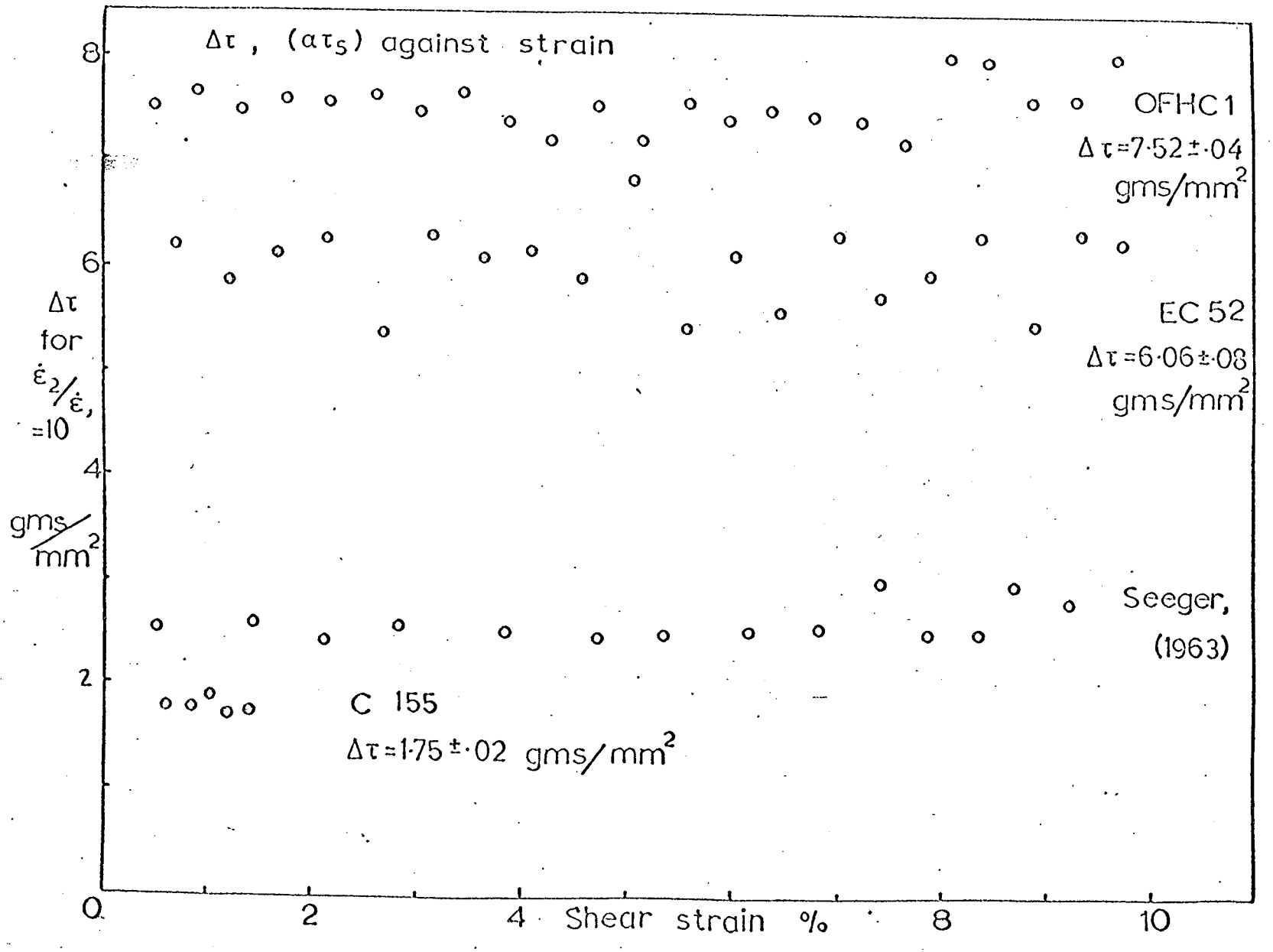
Δτ , (ατ5) against strain
OFHC 1
Δτ=7·52±·04
gms/mm²
EC 52
Δτ=6·06±·08
gms/mm²
Seeger,
(1963)
C 155
Δτ=1·75±·02 gms/mm²
Δτ
for
ε̇2/ε̇1
=10
gms/mm²
0
2
4
6
8
10
Shear strain %

Figure 4

distribution of impurities.

The other striking feature in Fig. 4 is the strong correlation between the magnitude of $\Delta\tau_s$ and the total impurity content. Since $\Delta\tau_s$ has been found to be constant during easy glide, $\Delta\tau_s/\tau$ decreases as the stress increases. Table 1 gives, for specimens of different purity, the mean value of $\Delta\tau_s$, and the range of values of $\Delta\tau_s/\tau$ (proportional to $\tau_s/(\tau_s + \tau_g)$) from the onset of flow to the end of stage I.

The value of $\Delta\tau_s$ increases by a factor of 4 for an impurity level change from 5 p.p.m. to $\sim$ 200 p.p.m. The last column in Table 1 shows how the enhanced value of τ_s/τ in easy glide arises only for the specimens with the higher impurity contents. In the present experiments (and from the previous results of many workers, for example, Basinski 1959, Thornton et al 1962) the characteristic value of $\Delta\tau_s/\tau$ in stage II for a x10 strain rate change is $\approx$ 8/1000, and is only slightly sensitive to impurity content. It is significant, therefore, that the stage I values of $\Delta\tau_s/\tau$ in the purest specimens in the present work were of this same order.

TABLE I

$\overline{\Delta\tau_s}(\propto \tau_s)$ and $\Delta\tau_s/\tau$ for $\dot{\varepsilon}_2/\dot{\varepsilon}_1 = 10$, for Easy Glide Copper at 295°K, as a Function of Impurity Content

Total Impurity p.p.m.	$\overline{\Delta\tau_s}$ gms/mm^2	$\Delta\tau_s/\tau$ Stage I
5	1.49 ± .1	(11 → 6)/1000
8	1.75 ± .02	(15 → 12.7)/1000
53	2.25 ± .1	(20 → 17)/1000
150	6.4 ± .3	(40 → 30)/1000
150	6.06 ± .08	(51 → 24)/1000
150	7.52 ± .04	(54 → 34)/1000
220	4.98 ± .02	(49 → 30)/1000

3. RESULTS FOR CRYSTALS DEFORMED IN TENSION AND TORSION

(a) Easy Glide Region

The experimental procedure was to make four changes in tensile strain rate prior to the first increment of torsional strain so that $\Delta\tau_s$ characteristic of the normal easy glide region could be determined with accuracy. The torsional deformation was introduced by hand with levers attached to the lower tensile grip, and was of such a magnitude as to enable the small change $\delta(\Delta\tau_s)$ to be readily determined without raising the stress level above that normally associated with stage I. No attempt was made to measure the increment of torsional strain since for the rod crystals employed this will vary across the specimen section, while even for tubular crystals there is a longitudinal variation due to the effect of the grips.

The torsional deformation caused an increase in τ and $\Delta\tau_s$, and a slight increase of the subsequent work-hardening rate in the tensile deformation. A particularly useful feature of the results is that the values of $\Delta\tau_s$ were constant for the first few strain rate changes after the torsional deformation, enabling $\delta(\Delta\tau_s)$ to be defined with accuracy. Figures 5 and 6 illustrate typical results. The specimen whose behaviour is illustrated in Fig. 5 contained 8 p.p.m. impurity, while that in Fig. 6 contained 53 p.p.m. Only one increment of torsional deformation was possible for each specimen in the easy glide region, and, using the notation of Fig. 5 the results obtained are shown in Table 2. The striking feature is that the torsional deformation has caused interactions, which, irrespective of the specimens' impurity content, lead to $\Delta(\Delta\tau_s)/\delta(\tau)$

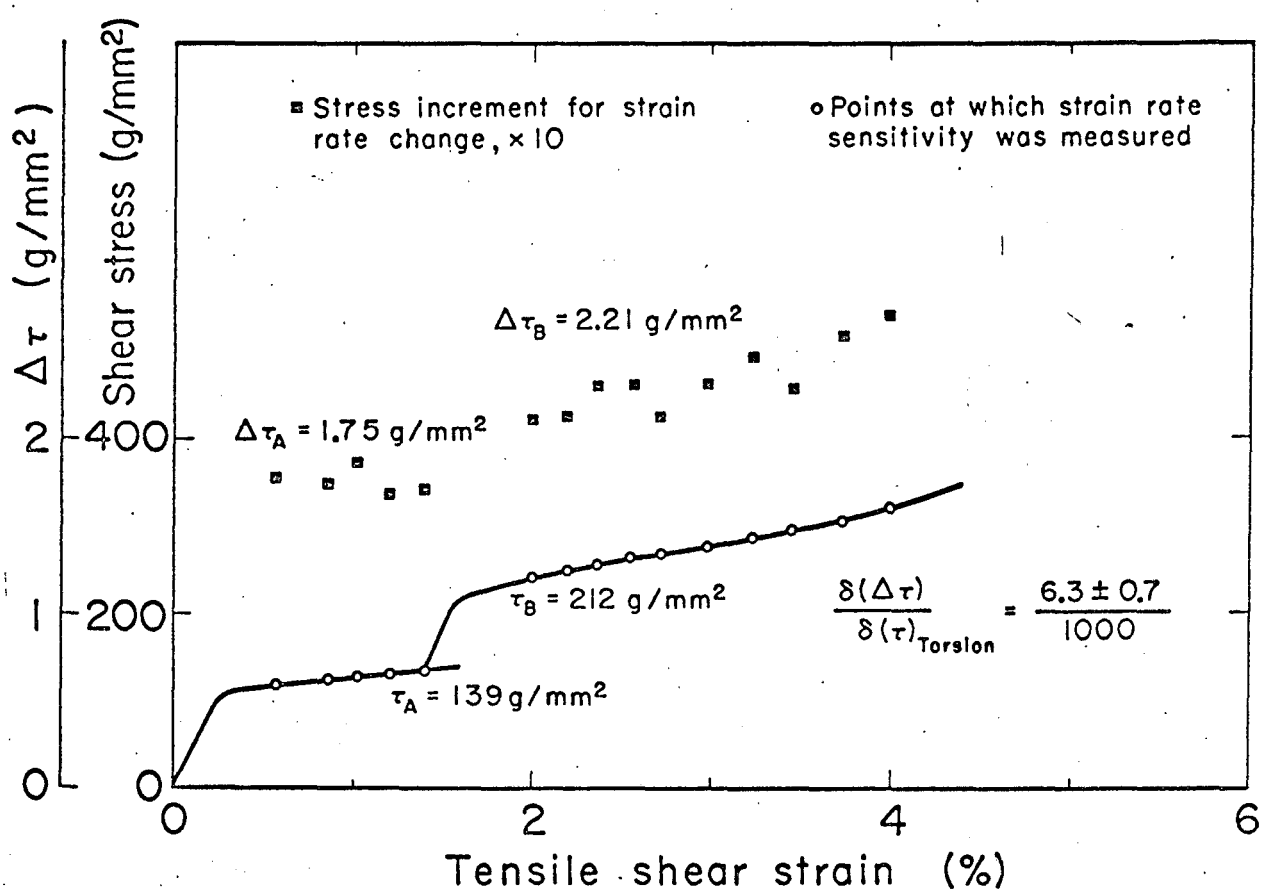

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Figure 5 Typical Results in a Stage I Tensile and Torsional Experiment

Copper 153, Deformed in Tension and Torsion

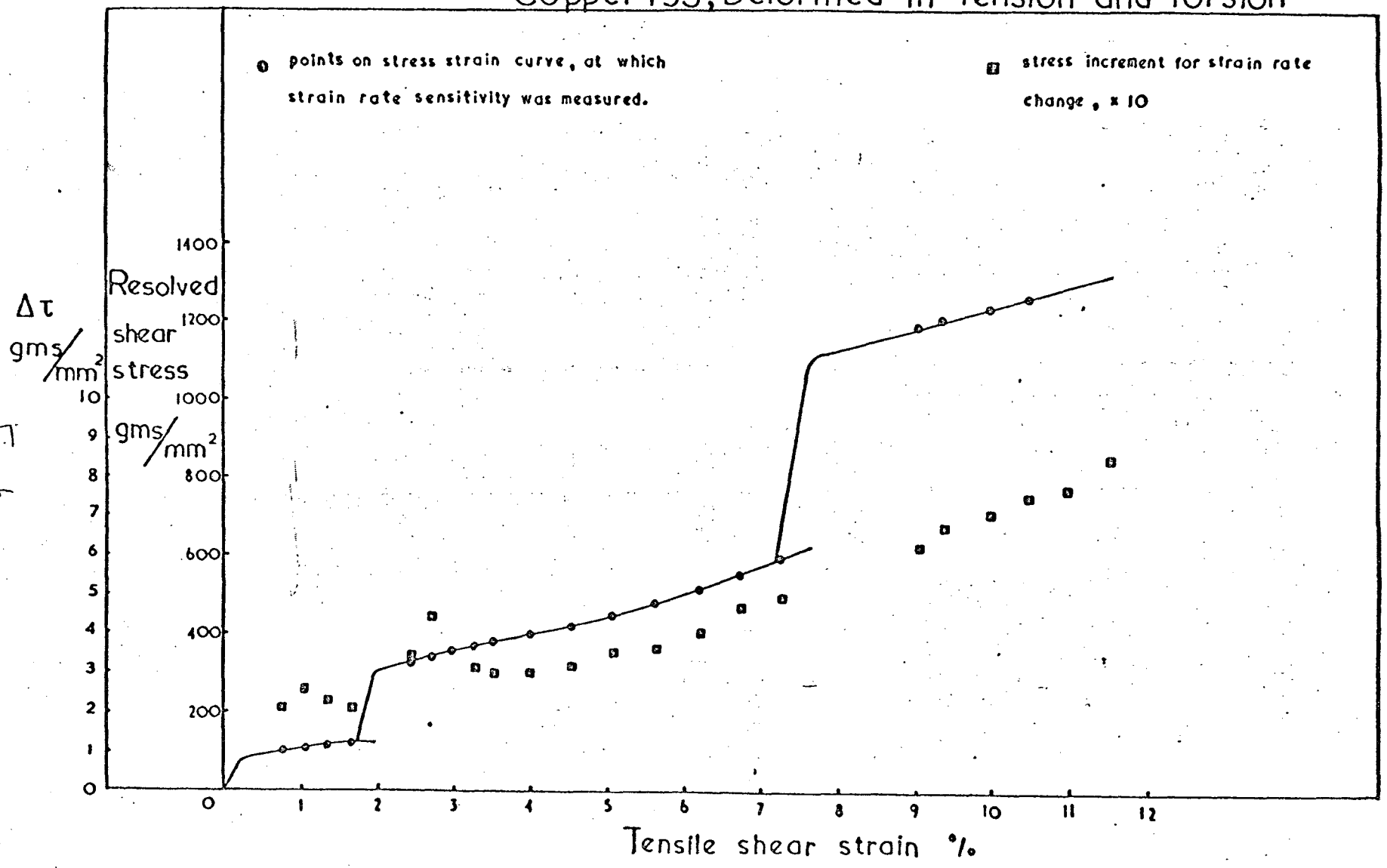

Fig 6

TABLE II

$\delta(\Delta\tau_s)/\delta(\tau)$ Due to Deformation in Torsion During Easy Glide

$\Delta\tau_A$ gms/mm^2	$\Delta\tau_B$ gms/mm^2	$\tau_B - \tau_A$ gms/mm^2	$\delta(\Delta\tau_s)/\delta(\tau)$ (forest)	$\Delta\tau_A/\tau_A$	Impurity p.p.m.
1.49 ± .1	2.17 ± .1	77.9	(8.7 ± 1.7)/1000	10/1000	5
1.75 ± .02	2.21 ± .03	73.2	(6.3 ± .7)/1000	13/1000	8
2.25 ± .1	3.31 ± .1	173.5	(6.1 ± 1.0)/1000	17/1000	53
4.98 ± .02	5.61 ± .1	79.4	(7.9 ± .8)/1000	30/1000	220
*5.68 ± .03	7.76 ± .1	228.	(9.1 ± .5)/1000	32/1000	200

*Deformed at 78°K

from 6 → 9/1000. Seeger et al (1961) have shown that the behaviour of the primary dislocations in stage I is best thought of in terms of a number of individual dislocations rather than groups or pile-ups. The straightforward and most reasonable result of the torsional deformation is, therefore, that forest dislocations have been introduced which have interacted with individual dislocations in the primary plane. The incremental changes $\delta(\Delta\tau_s)/\delta(\tau)$ are accordingly a measure of the proportions of short to long range stress characteristic of forest-primary dislocation interactions.

We find, therefore, that τ_s/τ which is characteristic of forest dislocations interacting with the primary network leads to a value of $\Delta\tau_s/\tau$ in a strain rate change test which is identical within the experimental error with that which is obtained in stage II. It is also clear from the absence of correlation between $\delta(\Delta\tau_s)/\delta(\tau)$ and $\Delta\tau_A/\tau_A$ with differing impurity levels that the enhanced values of $\Delta\tau_A/\tau_A$ are not due to forest dislocations, but to the impurity level.

From this section and from section 2 we draw the following conclusions concerning the points enumerated earlier.

1) For specimens with only 5 p.p.m. impurity, $\Delta\tau_s/\tau_{(\text{easy glide})}$ is of the same order and possible rather larger than $\delta(\Delta\tau_s)/\delta(\tau)_{(\text{forest})}$. Enhanced values of $\Delta\tau_s/\tau$ are obtained for crystals with greater impurity levels, indicating a strong dependence of the short-range part of the flow stress on impurity.

2) The stage II ratio, $\Delta\tau_s/\tau$ is equal to $\delta(\Delta\tau_s)/\delta(\tau)_{(forest)}$ and both these terms are relatively insensitive to impurity. This is a strong indication that primary-forest dislocation interactions control in large measure the stage II flow stress.

Because of their relevance to theories of the flow stress and work-hardening the above conclusions are considered the major results of the present investigation, but a number of further results will be briefly mentioned as similar events do not appear to have been previously reported.

A most unusual occurence following torsional deformation is illustrated in Fig. 7. This specimen contained $\sim$ 150 p.p.m. impurity but was annealed in a poor vacuum prior to test, becoming contaminated with oxygen to such an extent that a slight oxide film could be seen on its surface. Prior to the first increment of torsional deformation the strain-rate change tests gave $\Delta\bar{\tau}_s = 4.07 \pm .05$ gms/mm^2 which is consistent with the variation of $\Delta\tau_s$ with impurity content as determined from the other experiments in the present work. Following a moderate torsional strain, which raised the flow stress from 117 gms/mm^2 to 185 gms/mm^2, the first strain rate change by a factor of 10 led to $\Delta\tau_s = 8.6$ gms/mm^2, while after a further small amount of tensile strain $\Delta\tau$ increased to 17.2 gms/mm^2. Thus, τ_s was raised by a factor $\sim$ 4, apparently due to a redistribution of the gas content caused by the torsional deformation, and only at a shear stress of 0.75 kg/mm^2 did the specimen return to the expected τ_s/τ ratio. This result makes it

Copper EC 72, Deformed in tension and torsion at 295°K

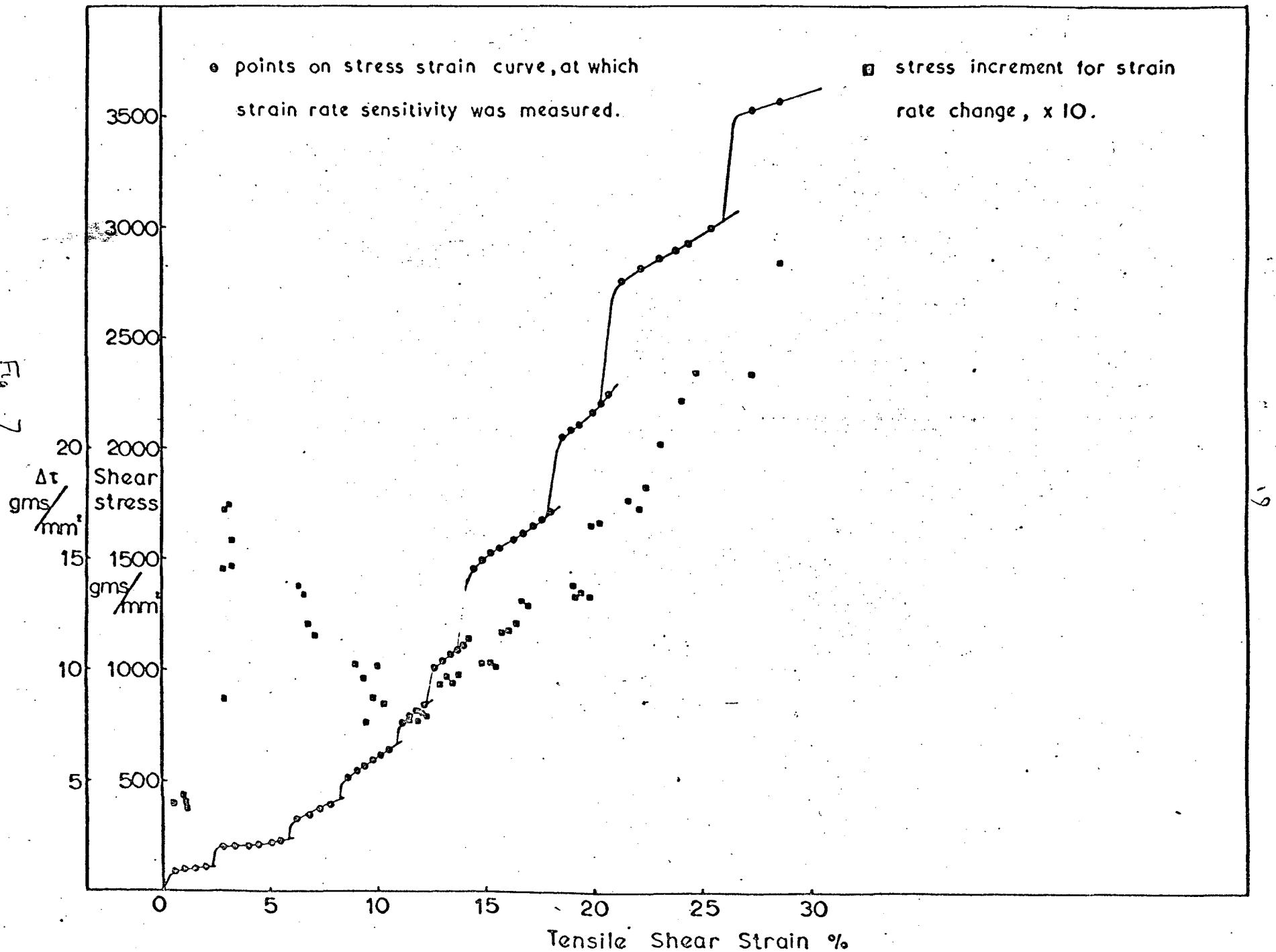

Fig 7

clear that a detailed interpretation of τ_s in stage I must take into consideration both the impurity level and its distribution. Since the specimen's oxygen content appears to be the only factor which distinguishes it from the other crystals it is perhaps worthwhile to mention other recent instances in which oxygen has been observed to affect crystal properties. The present author has found from extended node measurements in a Ag-In alloy (e/a $\sim$ 1.10) that oxygen raised the stacking fault energy from 15.7 ± 1 ergs/cm^2 to 23 ± 1.2 ergs/cm^2 (to be published). In addition, Haasen (private communication) has shown from τ_3 measurements that thorough de-oxygenation of silver reduces the stacking fault energy by a factor $\sim$ 2, leading to $\gamma_{Ag} \sim 15$ ergs/cm^2 at room temperature. This is further confirmation to the conclusion drawn by the present author (Ph.D. thesis, Cambridge, 1964) that $\gamma_{Ag} < 1/2$ (γ_{Cu} and γ_{Au}), since earlier τ_3 measurements had led to rather high values of γ_{Ag} ($\sim$ 40 ergs/cm^2, rather than the extended node result, $\gamma_{Ag} \sim 20$ ergs/cm^2 (Loretto et al 1964, Gallagher 1964 (thesis)).

(b) Stage II and Stage III

Since the present results have indicated that the major contribution to the flow stress in stage II is forest-primary interactions one expects that a twist experiment in stage II will lead to $\delta(\Delta\tau_s)/\delta(\tau) = \Delta\tau_s/\tau_{(\text{stage II})}$. This result has in fact been obtained by Seeger et al (1957) as mentioned earlier. In the present work, however, although $\delta(\Delta\tau_s)/\delta(\tau)_{(\text{stage II})}$ rarely exceeded 8/1000, the value characteristic of a x10 change in strain-rate in stage II, it was in many cases lower, and was frequently negative, i. e. the effect of torsional deformation

was to reduce the short range flow stress. The results are complicated in stage II by unloading effects, but allowance has been made for the Haasen-Kelly effect (1957) in determining $\delta(\Delta\tau_s)/\delta(\tau)$. Figures 8 and 9 illustrate the fact that in general the increase in τ caused by relaxing and twisting contains a smaller component of τ_s than if the stress had increased a similar amount in the course of tensile deformation. Attempts were made to avoid relaxing the load during the increment of torsional deformation, but some relaxation is unavoidable due to the torsional strain increasing the specimen length. A further interesting result was obtained on a specimen in which from $\tau = 5.5$ kg/mm^2 up to $\tau = 8$ kg/mm^2, the highest stress at which it was tested, a twist reduced both the value of $\Delta\tau_s$, and the total stress.

A reasonable explanation for these results appears to be that the dislocation movement on secondary systems is effective in reducing the stress concentrations in tangles which existed in the specimen deformed in tension. Except at very high stresses the overall increase in the dislocation density will lead to an increase in τ_g, but τ_s is much more structure sensitive, and depends on the detailed nature of the local dislocation distribution. At very high stresses it is possible that the introduction of slip on other systems will enable the high internal stress to be reduced by annihilation and cross slip leading to a decrease in the total stress as well as its temperature dependent part.

Thus, in the present work the extension of the experiment to high stresses has not led to as pleasing a substantiation of the conclusions drawn as was hoped. Nevertheless, the results at low stresses, where the

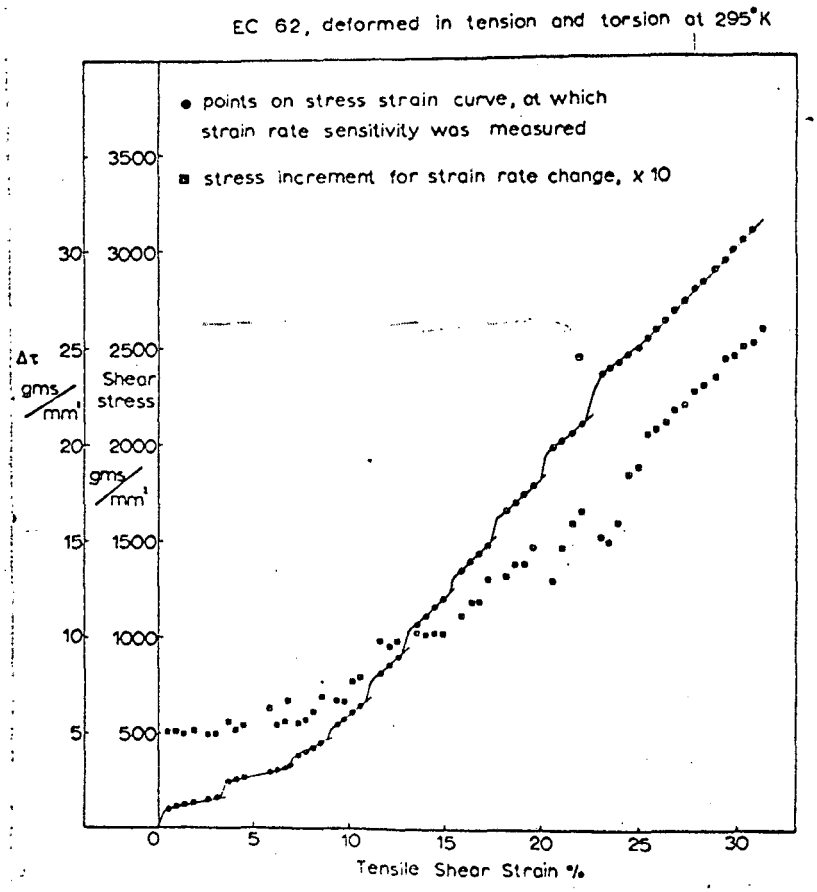
EC 62, deformed in tension and torsion at 295°K
• points on stress strain curve, at which strain rate sensitivity was measured
▪ stress increment for strain rate change, x 10
Δτ gms/mm²
Shear stress gms/mm²
3500
3000
2500
2000
1500
1000
500
30
25
20
15
10
5
0
5
10
15
20
25
30
Tensile Shear Strain %

Fig 8

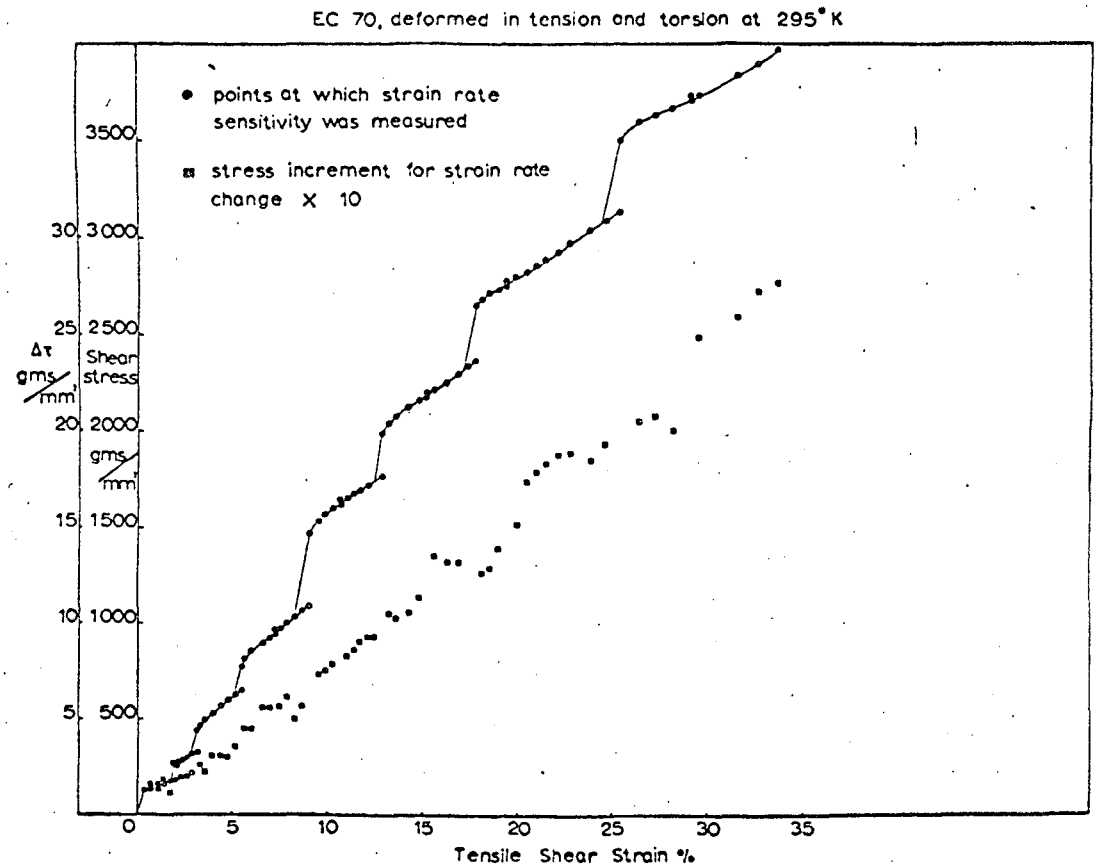
EC 70, deformed in tension and torsion at 295° K
points at which strain rate sensitivity was measured
stress increment for strain rate change X 10
Δτ gms/mm²
Shear stress gms/mm²
3500
3000
2500
2000
1500
1000
500
30
25
20
15
10
5
0
5
10
15
20
25
30
35
Tensile Shear Strain %

Fig 9

experiments are uncomplicated by the effects described, are felt fully to justify the conclusions enumerated earlier.

IV. DISCUSSION

A combination of the present results with earlier work, notably by Young (1962), Hordon (1962), Basinski and Basinski (1964), and Tinder and Washburn (1964) enables a much clearer picture of stage I and the pre-yield region to be obtained. A number of conclusions are drawn below, and are followed by the results which substantiate them.

(1) At very low stresses in the pre-yield region the flow stress is due to impurity-dislocation interactions. The flow stress is very sensitive to impurity-dislocation interactions until the pre-yield multiplication stress ($\sim$ 20 gms/mm^2) is reached.

(2) Thereafter the internal stress is sufficiently large to enable the dislocations to penetrate the impurity obstacles more readily. The glide process becomes increasingly dependent upon forest-primary dislocation interactions, and the dislocation mean free path is an exceptionally strong function of the stress. The yield stress (τ_o) is merely defined by the point at which $\Delta\varepsilon/\Delta\tau$ approximates to its maximum value.

(3) The magnitude of τ_g in stage I is due to forest dislocations interacting with the primary glide dislocations.

(4) The magnitude of τ_s in stage I is due almost entirely to impurity-dislocation interactions. For crystals with $\sim$ 5 p.p.m. impurity τ_s in stage I is small and largely attributable to

forest-primary interactions.

The comments concerning the pre-yield region and the initiation of plastic flow are applicable to specimens of very low dislocation density, and will be modified below for cases where the initial dislocation density in the specimen is greater than $\sim 10^5/cm^2$.

Concerning points (1) and (2) the work of Tinder and Washburn (1964) showed that dislocations moved irreversibly at resolved stresses as low as ~ 4 gm/mm^2. In addition, on relaxing the load and re-pulling, the initiation of flow was accompanied by discontinuous yielding. Furthermore, in this region dilute alloys of copper required a larger stress to produce the same pre-yield strain. An interesting result was that the introduction of fresh dislocations to a crystal eliminated the discontinuous yielding and resulted in a larger amount of pre-yield strain for a given flow stress, together with dislocation movement at sensibly zero applied stress. There is thus a correlation between this behaviour and the result mentioned earlier, that some deformation at high temperature following an anneal is effective in lowering the yield stress subsequently measured after cooling the specimen. It is clear that the result depends upon the provision in the crystal of dislocations which are less efficiently pinned by impurities.

Hordon (1962) found a residual flow stress ~ 15 to 20 gms/mm^2 (depending on the strain rate) which he attributed to impurity effects. His crystals were of a nominal 99.999% Cu. Young (1962) suggests that for his crystals of 99.999% purity the yield stress in a low dislocation density crystal is largely attributable to impurity-dislocation interactions.

The basis for this idea is that the initial dislocation density (N) of his samples was irrelevant in determining τ_o (for $5.10^3 < N < 2.10^5$), whereas a good correlation was obtained between the values of τ_o for specimens prepared from one large crystal and which presumably had the same impurity content. It is surprising, should this be the case, that a stronger temperature dependence of τ_o has not been observed, and that Young should not have measured a stronger dependence of τ_o upon strain rate. In addition, at only slightly higher stress levels in the present work the value of τ_s associated with impurity in a crystal of 99.995% Cu has been shown to be relatively small.

Young does in fact attribute the yield stress of a crystal with $N = 10^6/cm^2$ to dislocation interactions, and shows that its initial flow stress, $\tau_o = 94$ gms/mm^2, is about equal to the stress necessary for multiplication, $Gb(N)^{1/2}$. As Basinski and Basinski (1964) point out further valuable information concerning the yield process is revealed by the flow properties of the above crystal, contained in Fig. 6, Young (1962). The important point is that by the time other crystals which had flowed at a lower stress reach $\tau = 94$ gm/mm^2, they have acquired a <u>higher dislocation density on the primary glide plane</u> than is contained in the crystal which has $\tau_o = 94$ gm/mm^2. This indicates a lack of correlation between the dislocation density in the primary slip plane, and the yield stress. Only guarded conclusions should be drawn, however, since for the higher dislocation density crystal a greater fraction of the dislocations were in sub grain boundaries.

That the yield stress does in fact correlate with the <u>forest dislocation density</u> was shown by Basinski and Basinski (1964), by testing a virgin

crystal, and in addition one which was twisted prior to tensile testing. The flow stress increased as did the forest dislocation density while the density of primary dislocations remained unchanged. As mentioned earlier a strong increase in τ was caused by the increase of N_f, the forest dislocation density and caluclations suggest $\tau = \phi Gb(N_f)^{1/2}$ with $\phi \sim 1$, rather than $\sim 1/5$ as proposed by Saada (1961). It is felt, therefore, that the two major contributions to the yield stress in a crystal are impurity-dislocation pinning effects, and forest-primary dislocation interactions. The relative magnitude of these effects will clearly depend upon the impurity content (and distribution) and the dislocation density in both the primary and secondary glide planes.

The clearly defined macroscopic yield stress found in crystals whose tensile axis is such that one slip system is primarily operative appears to be a result of conditions particularly favourable to a rapid variation with stress of L, the mean free path of a dislocation. Clear evidence for the effect of forest dislocations in defining $\Delta L/\Delta\tau$ is the observation that corner orientation crystals generally have a less well defined yield stress, implying a more limited dependence of L on τ. The many observations that L is constant during easy glide (e. g. Young 1962, Basinski and Basinski 1964) indicate that the dislocation mean free path at the yield stress is not only of such a magnitude as to give a readily measureable strain, but happens also to be the maximum value which the dislocations attain.

The evidence in corroboration of points (3) and (4) follows principally from the present work, and from the results of Basinski and Basinski (1964) and others who have measured the variation of dislocation density for both primary and forest dislocations. As the above authors point out

the flow stress does correlate better with the variation in forest density, and demonstrated this more forcefully by the initial yield stress experiment already described.

It seems that no other conclusion can be drawn except that forest dislocations are the flow stress determining factor in stage I. In the present work the most positive results concern τ_s, which has been shown to be particularly dependent upon impurity. They have also shown, however, that for impurity levels $\sim$ 5 p.p.m. that the magnitude of τ_s is only of the same order as that due to forest dislocations interacting with primary glide dislocations. There is no evidence for any further contribution to τ_s.

We may write, therefore, for stage I,

$$\tau_s = \tau_{(f)s} + \tau_{(i)s} \qquad (1)$$

where $\tau_{(f)s}$ is the short range stress normally associated with the forest-primary interaction process, and $\tau_{(i)s}$ is due to impurities, and appears to be almost zero for solute contents as low as $\sim$ 5 p.p.m. when the dislocation density is $\sim 10^6/cm^2$. This term is also sensitive to the distribution of the impurity as might be expected, since segregation of impurity to dislocations will clearly effect the locking stress. It would be useful to have an idea of the absolute magnitudes of $\tau_{(f)s}$ and $\tau_{(i)s}$, which involves determining the factor α, where $\alpha\Delta\tau_s = \tau_s$. By developing a theory of the flow stress Seeger (1963) found $\tau_s = 28$ gm/mm^2 in stage I for the particular crystal which he used. Examination of his results indicates that for a x10 strain-rate change the appropriate value

of α is of the order of 10.

For the moment we shall use this as an order of magnitude estimate, even though the description of stage I presented here is at considerable variance with that developed by Seeger et al (1961). Thus, on this basis using Table I for the purest crystal used, τ_s was $\sim$ 15 gm/mm^2 in stage I, while for a specimen containing $\sim$ 200 p.p.m. impurity, $\tau_s \sim 65$ gms/mm^2, while in each case the contribution from $\tau_{(f)s}$ to τ_s was equal to $\sim$ 10 gms/mm^2. What is, of course, more important to the present results is that α should be reasonably constant despite variations in the magnitude of τ_s, and insensitive to the factors contributing to τ_s and to the stress at which the strain-rate change is performed. If one considers the result of a strain rate increase to be slightly higher potential barriers due to the effective decrease in temperature, the fraction by which τ_s is increased appears to depend only upon the magnitude of the strain rate change. The concentration of the barriers, their absolute height, and the nature of the interaction causing them appear to be irrelevant.

There is, in fact, evidence from the present work and from the literature that α is less than the value $\sim$ 10 adopted in the previous paragraph. The forest dislocation density has been shown to increase during stage I (see, for instance, Basinski and Basinski, 1964) and yet for the range of impurities used in the present work $\Delta\tau_s$ was constant within close limits throughout stage I. For example, in Fig. 4, for the three sets of results which extend to 10% strain in no case is there an increase in $\Delta\tau_s$ of more than $\sim$ 0.5 gm/mm^2 in stage I, while the total

stress has increased from ~ 120 gm/mm^2 to ~ 240 gm/mm^2. Using $\alpha = 10$ one would expect an increase in $\Delta\tau_{(f)s} \sim 1.2$ gm/mm^2. In addition, the variation of the work-hardening rate in stage II from 90°K to 400°K has been shown to be due only to the change in elastic modulus (Diehl and Berner, 1960). Thus, since the present work has shown that the flow stress in stage II is determined by forest-primary interactions, $\tau_s/\tau_{g(forest)}$ must be very small indeed.

One would expect, however, that for forest dislocations intersecting dislocations in the glide plane τ_s/τ_g would be large, since thermal activation is expected to aid the process considerably. Earlier estimates have indicated that $\tau_s/\tau_{g(forest\text{-}primary\ intersection)}$ is $\sim 1/5$ (Saada, 1960) or ~ 1 (Seeger, 1963). The constancy of $\Delta\tau_s$ in the present results throughout state I irrespective of specimen purity, coupled with the results of Diehl and Berner suggest that for <u>the forest-primary interactions which determine the flow stress</u>, $\tau_s/\tau_{g(forest)}$ is certainly $< 1/10$, and is more likely to be $\sim 1/20$. It appears that forest-primary <u>intersections</u> do not determine the flow stress, but that their interaction is the important factor.

For $\tau_s/\tau_{g(forest)} = 1/20$, we find that $\alpha = 6$ and τ_s in stage I is reduced to 60% of the values given in the previous paragraph. It is felt that the figures thus obtained represent much more closely the total contribution of impurity to the flow stress.

In conclusion, a speculation is made concerning the major contributory factor to the end of stage I for crystals of easy glide orientation. A striking feature about τ_2, the stress at the beginning of stage II, is

that it is generally $\sim 2\tau_o$. When the primary slip system has a resolved stress of τ_2, the secondary systems have a resolved stress $\sim \tau_o$, and it is suggested that the principal property of the stress τ_o is that the sensitive parameter, L (the dislocation mean free path) reaches a particularly large value. It is suggested therefore that the "increased activity on the secondary plane" at the end of stage I is due to precisely the same cause as leads to τ_o being so readily defineable. Of course, by the time the crystal has reached the end of stage I the dislocation structure is sufficiently different from that at the yield stress as to limit the slip distance of the secondary dislocations. Nevertheless, their increased mobility due to the resolved stress reaching $\sim \tau_o$, will, it is suggested, lead to a rapid increase in the number of forest-primary dislocation interactions, and thus to a change in the work-hardening behaviour almost as dramatic as that which occurs at the macroscopic yield stress.

The rationalizations proposed in the present paper have been made possible by a positive identification of the important factors in determining the flow stress. While a detailed theory has not been established, it is felt that the present experimental results coupled with those existing in the literature have enabled a more consistent description to be evolved.

FIGURE HEADINGS

Figure 1 V_τ plotted against stress and strain (%), copper.

Figure 2 V_τ plotted against stress and strain (%), copper.

Figure 3 V_τ plotted against stress and strain, silver.

Figure 4 $\Delta\tau_s$ in easy glide, illustrating the constancy of τ_s with strain, and the effect of impurity.

Figure 5 Deformation in torsion and tension, illustrating the measurements made and the notation used.

Figure 6 Deformation in torsion and tension in the easy glide region.

Figure 7 The anomalous variation of τ_s following torsional deformation in stage I.

Figure 8 Torsional and tensile deformation in stage I and stage II, illustrating decrease in τ_s due to a twist.

Figure 9 Torsional and tensile deformation in stage I and stage II, illustrating decrease in τ_s due to a twist.

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