FULMER RESEARCH INSTITUTE LTD.

R.167/4/November, 1961.

THE SPACING OF SLIP LINES IN METALS

by

J.T.Barnby, B.Sc., Ph.D.

343 6 10

The research reported in this document has been sponsored in part by the Office, Chief of Research and Development, U.S. Department of Army, through its European Office under contract number DA-91-591-EUC-1625.

FULMER RESEARCH INSTITUTE LTD.

THE SPACING OF SLIP LINES IN METALS

Contract Number:- DA-91-591-EUC-1625.

Annual Technical Status Report No.1.

Period: November 1960 to October 1961.

The research reported in this document has been sponsored in part by the Office, Chief of Research and Development, U.S. Department of Army, through its European Office, under contract number DA-91-591-EUC-1625.

ABSTRACT

Yield stresses of polycrystalline brass tensile specimens have been measured at 25°C and -196°C. The grain sizes of the specimens were varied, and the yield strength was found to obey the relation

$$\sigma_y = \sigma_i + k_y d^{-\frac{1}{2}}$$

where d is the average grain diameter and σ_i and k_y are constants for a given test temperature and composition. Both temperature and composition have been varied, and the dependence of σ_i and k_y on these parameters has been studied. The results are interpreted as being consistent with the theories, of Petch[7] and Cottrell[8] on the grain size dependence of yield stress. Since k_y is practically independent of temperature it is suggested that the dislocation locking, which is theoretically proportional to k_y, is of the Suzuki[4] type (i.e. segregation of solute to stacking faults). The decrease in stacking fault energy with zinc concentration is thought to have an important bearing on operation of Suzuki locking. Values of σ_i are found to decrease initially with increasing zinc content of the alloys. This is thought to be a result of a tendency towards easy glide in brass polycrystals, which may be connected with the wider separation between partial dislocations and the decrease in stacking fault energy.

The behaviour of σ_i and k_y is compared with work on the spacing of slip lines, and it is concluded that the slip line distribution changes as a direct result of the operation of the solution-hardening mechanism which dictates the values of k_y.

1. INTRODUCTION

Deformation of pure metals such as copper has been shown by Kuhlmann-Wilsdorf[1] to result in the production of fine slip lines on the surface of a tensile specimen, with a regular spacing of 200-300Å. In α-brass deformation is commonly observed to result in deep, randomly and widely spaced slip lines. Thus there is a tendency for a given shear in a pure metal to be accomplished by small shears on a large number of slip planes; in fact on all available slip planes for a given system since Kuhlmann-Wilsdorf[2] calculates that the spacing 200-300Å is the minimum spacing at which dislocations may pass each other. In alloys like α-brass, however, a comparable shear takes place by large shears on a few randomly-spaced slip planes. The change in spacing of slip lines with zinc content of brass has been studied in detail by electron-micrographs of slip lines, and a short summary of that work is included in this report.

This change in slip line spacing and depth is thought to be a direct result of the control of the yield stress of the alloy by some mechanism(s) of solid solution-hardening. The mechanisms which could be operative in α-brass are: Cottrell locking[3], Suzuki locking[4], Short-range-order effects as described by Fisher[5], and long-range-order[6]. These hardening mechanisms depend on the parameters of composition and temperature in different ways. Moreover, some of these mechanisms act as frictional forces on dislocations, and others as initial locking forces. Fortunately it is possible to separate these two types of hardening using a theory developed by Petch[7] and by Cottrell[8] to describe the grain size dependence of the yield stress of an alloy. The measurements described below are of the yield stresses of specimens of different grain sizes, which allows separation of frictional and locking forces. Concentrations of zinc and temperature are also varied, which is expected to help to differentiate between the types of locking forces and so decide which is operative.

2. EXPERIMENTAL PROCEDURE

Alloys were cast from O.F.H.C. copper and 99.99% pure zinc to obtain the nominal compositions shown in table I. Analyses for copper were made, and the zinc content, obtained by difference, showed that the compositions were close to those required.

TABLE I

Nominal composition $^{w}/_{o}$ zinc	Analysis figure for copper	Zn% by difference
1	98.8	1.2
2	97.6	2.4
3	96.7	3.3
4	95.3	4.7
10	89.2	10.8
15	84.3	15.7
30	69.5	30.5
37	62.7	37.3
O.F.H.C. copper	99.9	-

The brasses were cast as 4 in. diameter billets, forged at 800°C to slabs 6¼ in. x 1½ in. thick and rolled at 800°C to slabs 1 in. thick. This hot working was sufficient to break down the cast structure. The slabs were cold-rolled to give a thickness reduction of 40%, then sliced into bars and turned to round rod suitable to act as blanks for cylindrical tensile specimens of the standard Hounsfield no.14 design, i.e. gauge length 1 in., gauge diameter ¼ in.

To obtain a suitable range of grain sizes the cold-worked blanks were sealed in evacuated silica tubes and annealed in a closely controlled electric furnace. Since the volume was restricted, dezincification was almost non-existent, and as a further precaution the gauge length was machined into the blank after the anneal so that no dezincified surface layer could affect the tensile tests. Fine grain sizes were obtained by anneals from 20 to 60 minutes at 575°C, and other grain sizes by 20 minutes to 2 weeks at 700°C. The very short anneals at 700°C were not capable of producing a suitable fine grain size.

After the annealing treatment, gauge-lengths were machined into the blanks and the gauge lengths were ground and polished to a high finish in order to remove any work-hardened surface layer. Grain size measurements were made on the polished end of each specimen, by the line intercept method. Twin boundaries were included in the counts since there is only a 1 in 4 chance of any particular twin boundary being the operative slip plane, and so not acting as a barrier to dislocation movement comparable to a grain boundary.

Tensile testing was carried out in a hard-beam Polanyi type machine, stress being measured by strain-gauges inside a proving ring. A continuous record of load versus time was obtained and the yield stress determined as the point where the load-time curve deviated from a straight line. No difficulty was experienced in assigning a value to this point. In a few cases where an upper and lower yield stress was recorded, the lower yield stress has been plotted. Tests at room temperature (25°C) were undertaken with no special control of temperature. Tests at -196°C were carried out with the specimen and part of the tensile machine immersed in liquid nitrogen. In the latter case, specimens were placed under a small load at room temperature in order to obtain axial loading. The specimens were then immersed in liquid nitrogen and left until rapid boiling ceased, before the test was started. Pre-loading at room temperature prevents non-axial loading which can arise due to icing-up in the ball-and-socket joints of the machine, which are also immersed in liquid nitrogen. All tensile tests were carried out with a constant crosshead speed of 6.92×10^{-3} cm/min on a gauge length of 2.50 cm.

3. RESULTS

The measurements are summarized in figure 1. There it is seen that for the pure metal, O.F.H.C. copper, there is no measurable dependence of the yield stress on grain size; moreover the temperature dependence of the yield stress is small since there is little difference in stress between the results at 25°C and -196°C. There is little significant difference between the copper and an alloy containing 1% zinc, though there is a suggestion of a little grain size dependence of the yield stress, and of a decrease in the intercept at $d^{-\frac{1}{2}} = 0$. Figure 1c shows a significant

departure from the behaviour of copper in that the slope of the line has definitely increased, and the intercept decreased. In figures 1d, 1e and 1f this trend continues and confirms the results of figures 1b and 1c. In figure 2a the intercepts at $d^{-\frac{1}{2}} = 0$ (i.e. σ_i) are plotted against the zinc contents of the alloys, and figure 2b shows a similar plot for the slope of the grain size dependence, k_y. The surprising features of these results are that σ_i decreases initially at 25°C and reaches a constant value at around 10% zinc; at -196°C there is again an initial decrease of σ_i, with increasing zinc concentration, followed by an increase; there is very little temperature dependence of k_y since the values at 25°C are very close to those at -196°C.

4. DISCUSSION OF RESULTS

Petch[7] and Cottrell[8] have described the grain-size dependence of the yield stress of b.c.c. metals by the equation:

$$\sigma_y = \sigma_i + k_y d^{-\frac{1}{2}}$$

where σ_y is the lower yield stress, σ_i interpreted as a frictional force opposing the movement of dislocations, k_y is related to the stress concentration near a piled-up group of dislocations, and d is the average grain diameter. The basic model involved is that propagation of slip from one grain to the next must take place by the formation of a piled-up group of dislocations in grain A, which concentrates the applied force sufficiently to unpin a dislocation source, in grain B, from its atmosphere of solute atoms. This process allows plastic deformation to spread across grain boundaries which act as major barriers to dislocation movement. The equation developed for yielding in b.c.c. metals should therefore apply to any metal where yielding takes place in this way. The experiments of Cottrell and Ardley[9] have shown that yielding in α-brass is similar to yielding in a steel; moreover there is considerable evidence for the existence of piled-up groups of dislocations in α-brass.[10,11].

The behaviour of k_y

Cottrell[8] writes:

$$k_y = \sigma_d l^{\frac{1}{2}}$$

where σ_d is the force necessary to unlock a dislocation from a solute atmosphere, and l is the average length of a dislocation in the network. Thus the slope k_y should be directly proportional to the unlocking force. Table II shows the values of k_y measured for the brasses in comparison with values for other metals. It is interesting to see that copper irradiated with a low dose of neutrons has a value of k_y very similar to a 3% zinc brass.

TABLE II

Metal	k_y cgs.	T °C	σ_i 10^3psi.	Reference
O.F.H.C. copper	-	25		present work
	-	-196		
copper with low neutron dose	0.08×10^8	20		12
" " " " "	0.08×10^8	-196		12
copper with high neutron dose	0.16×10^8	-196		12
1% zinc brass	-	25	6.2	present work
" "	-	-196	7.0	" "
3% " "	0.09×10^8	25	3.5	" "
" "	0.12×10^8	-196	5.0	" "
10% " "	0.21×10^8	25	2.8	" "
" "	0.23×10^8	-196	5.4	" "
15% " "	0.29×10^8	25	2.1	" "
" "	0.31×10^8	-196	5.6	" "
30% " "	0.42×10^8	25	2.0	" "
" "	0.44×10^8	-196	8.0	" "
zinc (99.995%)	0.57×10^8	-196		13
iron	2.1×10^8			14
magnesium	0.85×10^8			14
molybdenum	2.6×10^8			14

Figures 1 and 2b would therefore suggest that the locking force increases rapidly in the brasses from 1% up to 5% zinc, and thereafter it continues to rise linearly with the concentration of zinc. This behaviour is consistent with the work of Cottrell and Ardley[9] who found initial yield drops in α-brass single crystals containing 1% or more zinc. The size of the yield-drop was found to increase with zinc concentration up to 30% zinc in the work of those authors, and this may be taken as showing in a qualitative way that the size of the locking force is increasing. However, it is interesting to compare this behaviour with the concentration dependence of the Suzuki effect as shown in figure 11 of Suzuki's article[4]. The agreement between his curves and the concentration dependence of k_y is quite good. Cottrell and Ardley[9] explain their yield drops in terms of segregation of zinc to dislocations; but this effect would predict a steep temperature dependence of k_y. Although the results of Jamison and Sherrill[15] suggest that Cottrell locking becomes important in α-brass below -100°C, the small difference between the values of k_y measured here at 25°C and -196°C suggest that it is at -196°C that the Cottrell atmospheres are first becoming important. The temperature independence of k_y may be reconciled with Cottrell's interpretation of k_y if the locking mechanism can predict a temperature independent σ_d, and this condition is fulfilled by Suzuki's theory for segregation of zinc to stacking faults in brass. The size of k_y is in reasonable agreement with both the Suzuki and Cottrell theories since with $l = 10^{-4}$cm. $\sigma_d = 10^9$ dynes. Also, as mentioned above, the concentration dependence of k_y is in reasonable agreement with the Suzuki mechanism. The hardening obtainable by both long-range and short-range order would not be expected to give the initial rapid rise in k_y with zinc content. Though it seems probable that Suzuki locking controls the yield stresses of brass polycrystals over most of the temperature range 25°C to -196°C, it seems unlikely that this mechanism controls the flow stress in the region of jerky flow observed by Cottrell and Ardley[9], and noticed in some of these polycrystalline specimens. It is difficult to envisage the rapid segregation of the large number of zinc atoms necessary for Suzuki locking, and so the serrated stress-strain curve seems attributable to Cottrell atmospheres.

It is suggested that the concentration dependence and the temperature independence of k_y is evidence of the control of the initial yielding of brasses with more than 1% zinc, in the temperature range 25°C to -196°C, by Suzuki locking. Evidence for this has also been presented by Hibbard and co-workers.[16,17].

The behaviour of σ_i

A surprising feature of figures 1 and 2a is that σ_i decreases initially with increasing zinc content. The σ_i value for copper is due to dislocation-dislocation interactions, impurity precipitates and the Peierls force, if the models of Petch or Cottrell are applied. The change in σ_i with zinc content must be due to dislocation-dislocation interactions, or the Peierls[3] force, and the former seems more likely. Since figure 1 shows the decrease in σ_i to be large and significant, it cannot be neglected. Addition of zinc to copper results in a rapid decrease in the stacking-fault energy for small zinc concentrations[18], and this, in turn, must increase the area of stacking fault between partial dislocations. At the same low zinc concentrations the extent of easy-glide in single crystals is considerably extended;[19] the phenomenon of overshoot[22] on the most favourable slip system occurs, and k_y increases steeply. The first two of these effects have their counterpart in polycrystalline behaviour since Hibbard[17] interprets a low rate of work-hardening in brass polycrystals as evidence of easy glide. In the present work it was noticeable that where brass specimens with a large grain size yielded at a lower stress than a copper specimen of comparable grain size, the initial work-hardening rate was considerably smaller in the brass.

In a polycrystal it is the complicated slip behaviour near the grain boundary which determines the yielding and also the initial work-hardening rate. In the copper specimen it appears that slip on at least two slip systems immediately occurs in the neighbourhood of the grain boundary on yielding; whereas in the brass specimen there is evidence that slip occurs predominantly first on a single slip system. If some dislocation multiplication occurs near grain boundaries before macroscopic yielding is observed,[20] the decrease in σ_i with zinc content can be a result of a smaller density of forest dislocations in a brass specimen.

It is interesting to note that the temperature dependence of σ_i seems to increase with increasing zinc content(see figure 1). Further data on this would be most desirable; a possible explanation could be that intersection of dislocations becomes more difficult as the separation between partial dislocations increases. The higher activation energy associated with constricting the stacking fault before intersection no doubt leads to a greater temperature dependence of the stress necessary for intersection.

Correlation between mechanical properties and slip-line-spacing.

Figure 3 shows the results of past work on slip line spacings, in the form of ogive plots of the summed number of slip line spacings up to any value s; versus the spacing s. The figure shows that for the copper specimen most of the lines occurred at a spacing of about 300Å, corresponding to the "elementary structure" of Kuhlmann-Wilsdorf[1]. Whereas there are still many slip lines with a 300Å spacing, there are also many lines at higher spacings, and this trend continues as the zinc content increases. It is implicit in figure 3 that as well as the slip lines at higher zinc content being more widely spaced, there are fewer lines. Since the specimens have undergone comparable strains, this means that each line has contributed more shear and is deeper than the copper slip lines. This is clearly visible on electron micrographs of slip lines in the brasses. The growth of such slip lines has been studied in detail in an 80/20 brass by Fourie and Wilsdorf.[21]

To summarise the slip line spacing results, the fine slip in copper is replaced in brass by fewer slip lines with much greater spacing and depth, and with a more random spacing distribution as the zinc concentration increases. Taking the half height of the ogive curves as a measure of the change in slip line structure, about 1/3 of the change has taken place in a 5% zinc alloy and more than 2/3 of the total change has taken place in a 15% zinc alloy. Comparing this with the change in mechanical properties, one sees that the rapid increase in k_y and decrease in σ_i occurs also in the first 5 to 10% of zinc in solid solution. This is also the concentration range where the stacking fault energy is changing rapidly, and this effect could be strongly related to an increased Suzuki locking (increased k_y) and a decrease in σ_i as explained above.

The break-up of the "elementary structure" seen in the slip line spacing work could therefore be ascribed to the same solid solution hardening mechanisms which change σ_i and k_y, with the stacking fault energy playing an important part in the hardening. The picture which emerges is one where segregation of zinc to stacking faults in zinc gives a locking mechanism with a weak temperature dependence. Stacking faults become larger, both because of a decrease in the stacking fault energy and because solute segregates to them. Smaller numbers of dislocation sources are likely to operate as the locking increases and an unlocked source produces large numbers of dislocations and therefore a deep slip step. Where a large internal stress concentration does exist, it would be likely to unlock a group of sources on closely spaced slip planes, and thus the appearance of clusters of slip lines (a slip band) in the brasses, is not surprising.

5. CONCLUSIONS

The temperature independence of k_y points to the conclusion that Suzuki locking controls the initial yielding of brasses containing more than 1 $^w/_o$ zinc over most of the temperature range 25°C to -196°C. A tentative explanation of the initial decrease in σ_i with increasing zinc content is that the tendency towards easy glide, as the zinc content is increased in brass polycrystals, effectively reduces the density of forest dislocations cut by glide dislocations. The major change in spacing of slip lines with the zinc content of a brass is attributed to the same solid solution hardening mechanism as is responsible for the increase in k_y.

6. RECOMMENDATIONS FOR FURTHER WORK

It would be valuable to confirm the present results on mechanical properties by testing alloys of intermediate compositions. These alloys are already in a suitable form for testing. Further, it would be revealing to make measurements of k_y and σ_i at temperatures greater than 25°C. At sufficiently high temperatures dislocation locking mechanisms become ineffective. The way in which this strength is lost at high temperature could provide further evidence on the type of solid solution hardening which occurs at low temperatures, and at the same time,

data could be collected to elucidate the solute hardening mechanism at high temperature. Suggested future work has been given in detail in a proposal for renewal of Contract DA-91-591-EUC-1625, dated 28th August, 1961.

PERSONNEL

The investigation has been carried out by Dr. J.T.Barnby assisted in the experimental work by Mr. M.W.H.Gillham, under the general supervision of Mr. G.B.Brook.

SUMMARY OF COSTS

The approximate man-hours expended on this contract were 1256.

The cost of materials expended was £195.7s.4d.

No important items of equipment were obtained at direct contract expense.

JTB/SL
27.11.61.

REFERENCES

1. D.Kuhlmann-Wilsdorf & H.Wilsdorf. Acta Met. 1953, 1, 394.
2. D.Kuhlmann-Wilsdorf, D.Van der Merwe & H.Wilsdorf. Phil. Mag.1952, 43, 632.
3. A.H.Cottrell. Dislocations and Plastic Flow in Crystals. Oxford 1953.
4. H.Suzuki. Dislocations and Mechanical Properties of Crystals, eds. Fisher et al. Wiley 1957, p.361.
5. J.C.Fisher. Acta Met. 1954, 2, 9.
6. R.Feder, A.S.Nowick & D.B.Rosenblatt. J. Appl. Physics, 1958, 29, 984.
7. N.J.Petch. J.I.S.I. 1953, 174, 25.
8. A.H.Cottrell. Trans A.I.M.E. 1958, 212, 192.
9. G.W.Ardley & A.H.Cottrell. Proc. Roy. Soc. 1953, A219, 328.
10. J.D.Meakin & H.Wilsdorf. AFOSR-TN 60-4 1960.
11. B.J.Takamura & S.Miura. J.Phys.Soc.of Japan 1958, 13, 1421.
12. R.E.Smallman & K.H.Westmacott. AERE report M/R 2699 1958.
13. J.T.Barnby. Thesis Birmingham University 1958.
14. A.N.Stroh. Advances in Physics 1957, 6, 418.
15. R.E.Jamison & F.A.Sherrill. Acta Met. 1956, 4, 197.
16. N.G.Ainslie, R.W.Guard & W.R.Hibbard. Trans A.I.M.E. 1959, 215, 42.
17. W.R.Hibbard. Trans A.I.M.E. 1958, 212, 1.
18. J.Nutting & J.M.Arrowsmith. Joint Symposium on Structural Processes in Creep - Inst. of Metals and Iron and Steel Inst. 1961.
19. J.Garstone & R.W.K.Honeycombe (see reference 4 p.391).
20. D.A.Thomas & B.L.Averbach. Acta Met. 1959, 7, 69.
21. J.T.Fourie. Acta Met. 1960, 8, 88.
22. Von Göler & Sachs. Z.Physik 1929, 55, 581.

The following reports have been issued:-

CONTRACT NO. DA-91-591-EUC-1625

Q.T.S.R. No.1		
R.167/1/February 1961	Further Work on Spacing of Slip Bands	J.T.Barnby, B.Sc., Ph.D.
Q.T.S.R. No.2		
R.167/2/May 1961	-do-	J.T.Barnby, B.Sc., Ph.D.
Q.T.S.R. No.3		
R.167/3/August 1961	-do-	J.T.Barnby, B.Sc., Ph.D.

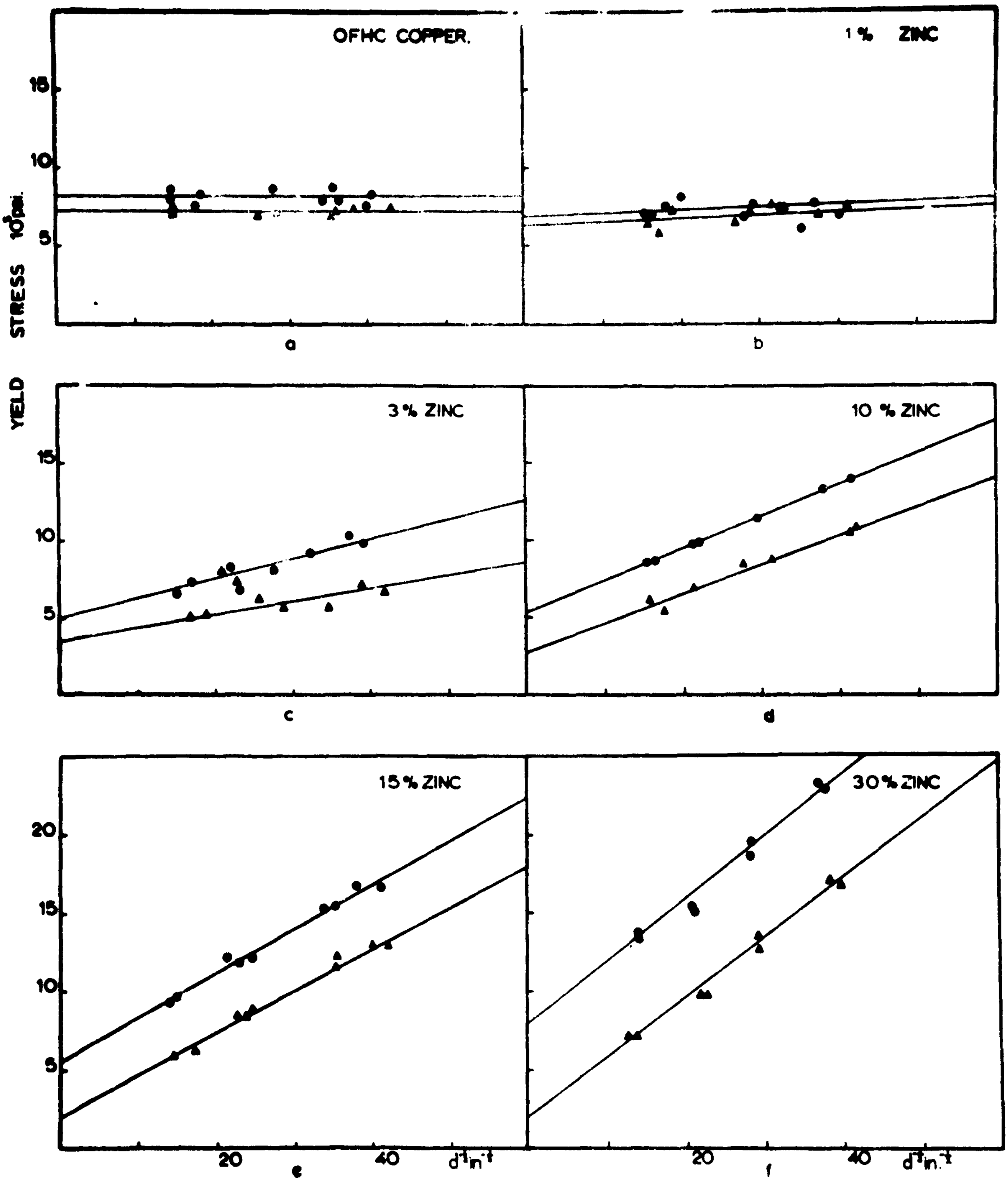

FIGURE 1.
YIELD STRESS VS. $d^{-\frac{1}{2}}$ FOR A SERIES OF BRASSES. TESTS AT 25°C ▲, TESTS AT -196°C ●.
(d = AVERAGE GRAIN DIAMETER.)

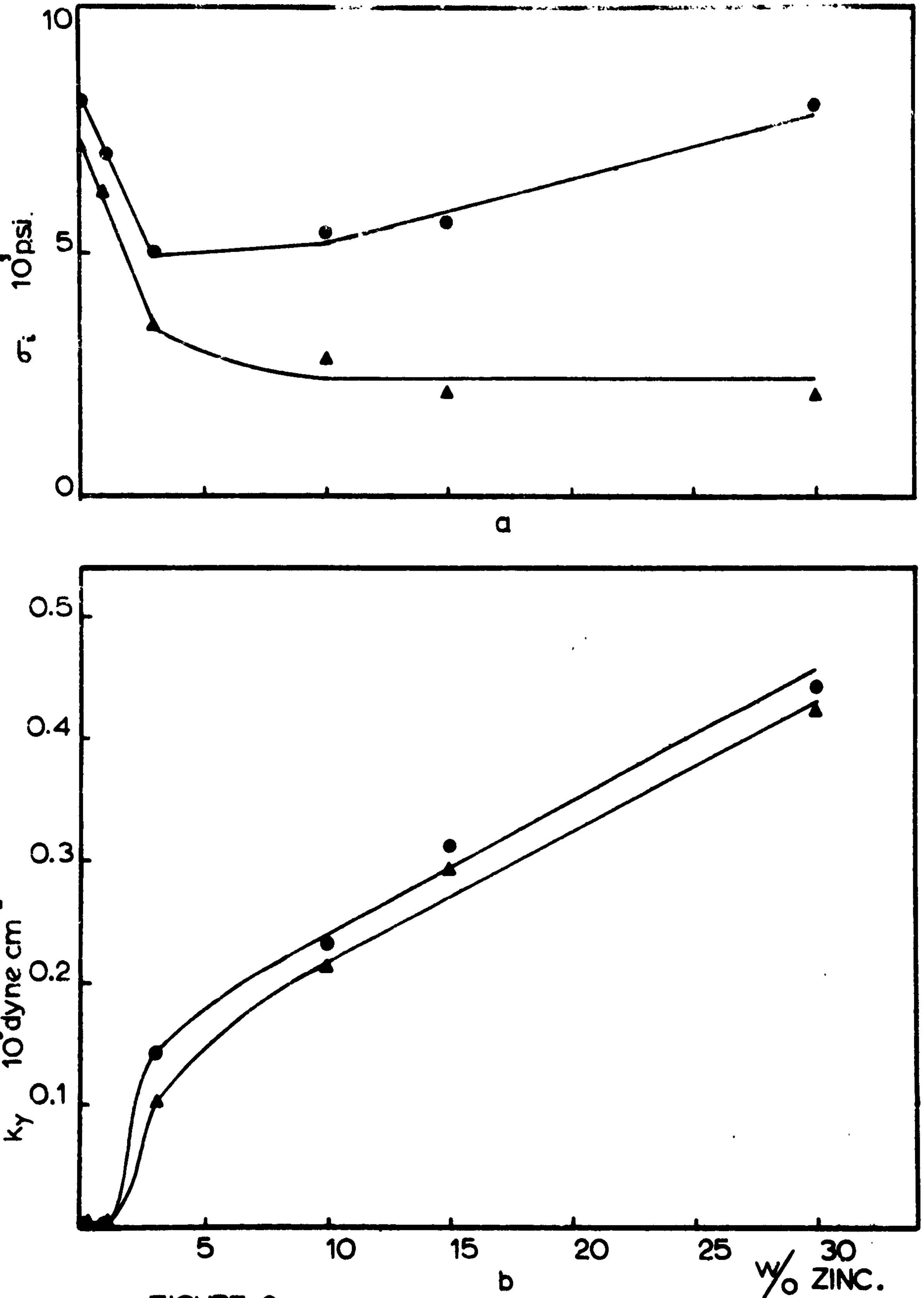

FIGURE 2.
TESTS AT 25°C ▲. TESTS AT -196°C ●.

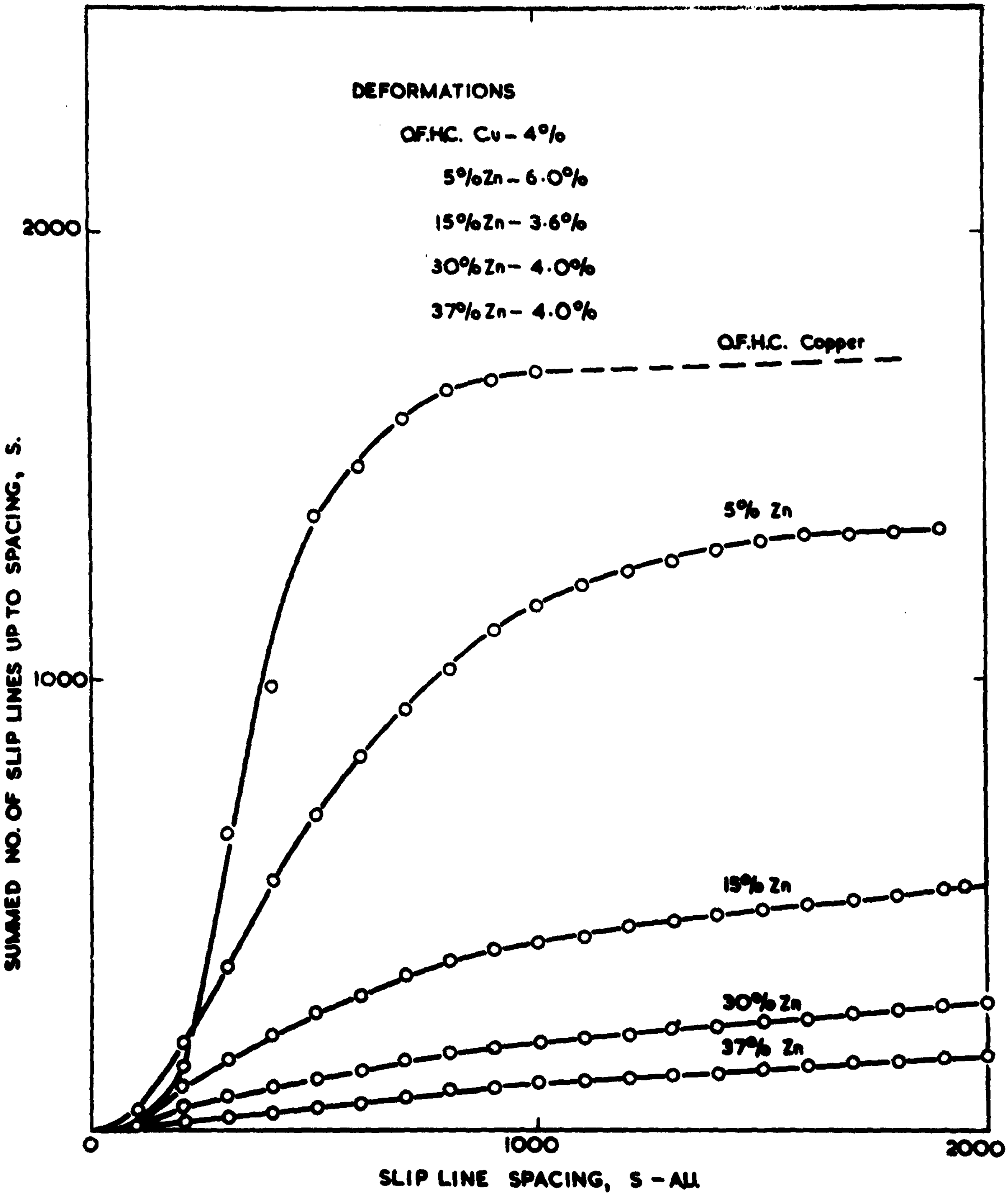
DEFORMATIONS
O.F.H.C. Cu – 4%
5% Zn – 6.0%
15% Zn – 3.6%
30% Zn – 4.0%
37% Zn – 4.0%
O.F.H.C. Copper
5% Zn
15% Zn
30% Zn
37% Zn
2000
1000
0
1000
2000
SUMMED NO. OF SLIP LINES UP TO SPACING, S.
SLIP LINE SPACING, S – AU.

FIG. 3.