

Effect of Strain Induced Dislocations on the Resistivity of Copper

M. J. FERNANDO* AND M. A. V. DEVANATHAN†

Institute of Scientific and Industrial Research, Colombo 7, Sri Lanka.

(Paper accepted : 4 November 1975)

Abstract : An experimental technique is described for obtaining electrical resistivity - strain curves and stress - strain curves for copper at uniform rates of strain in the range of 4.7 to 91.0% strain per minute. Plots of resistivity corrected for dimensional changes with (true strain)^{1/2} gave two linear sections with the higher gradient at high strain. The gradients increase with increase in rate of strain. Corresponding stress vs (true strain)^{1/2} plots also showed two linear sections but with the second gradient slightly lower than the first.

An expression taking into account the annealing rate constant (h) and the rate of strain is derived. For a selected value of h , a plot of corrected resistivity with (true strain)^{1/2} shows two linear sections for all rates of strain. Further, the graph of corrected stress vs (true strain)^{1/2} at $h = 0.05$ fall into one straight line for all rates of strain.

The value of dislocation density calculated on our electrical resistivity measurements is in excellent agreement with that obtained by transmission electron microscopy.

1. Introduction

It is well known that the resistivity of a metal is due to electron scattering by phonons, impurities and structural defects. At constant temperature, scattering of electrons by phonons and impurities would be expected to be constant. However, a metal subject to strain, would contain more structural defects than that of the unstrained.

A major structural defect which controls the metal behaviour under strain is the density of dislocations. It has been demonstrated¹ that a metal under strain undergoes a multiplication of its dislocations such that the dislocation density is proportional to the extent of strain.

It would therefore appear that any change in resistivity of a metal under strain could only arise from the increase in scattering of the electrons brought about by an increase in density of dislocations. In order to verify this experimentally, we have carried out measurement of variation of resistivity of copper with strain for which independent data of density of dislocation as a function of strain are available.

2. Experimental

Soft drawn copper wire (purity 99.90%) of length 50 cm and diameter 0.162 cm was strained at predetermined rates on a Mitchell Halifax metal working lathe suitably modified for the purpose.² The wire was held between two chucks, one of which was

*Head, Chemistry Division, Research and Development Office, Highways Department, Ratmalana.

†Director, Tea Research Institute, Talawakelle.

fixed to an immovable post of the lathe, whereas the other chuck was bolted with an adaptor to the movable carriage aligned in position for tension with insulation to isolate the wire from the metal parts of the lathe.

The variation of resistance with strain at two points 10 cm apart in the central portion of the copper wire was monitored, using the 4 point probe method by means of a 'Keithley' model 503 milliohmmeter, designed to measure low value resistances from 0.001 ohm full scale to 1000 ohm full scale. The recorder output of the milliohmmeter was fed to a 'Sargent' Recorder Model MR. The resistance was recorded continuously on straining the wire to the breaking point.

Stress-strain curves for the same copper wire were obtained under the same conditions of dynamic strain by attaching a proving ring (maximum load 226.5 kg, with a resolution of 0.455 kg per division on a dial gauge) to the immovable post of the lathe. The dial readings of the proving ring are recorded at regular intervals of time using a stop-watch, during continuous strain till the wire snapped.

3. Results

The variation of resistivity (ρ) uncorrected for dimensional changes and annealing of dislocations with true strain (ϵ) for different rates of strain ($\dot{\epsilon}$) — 0.0470 (26 rpm), 0.1084 (59 rpm), 0.2118 (116 rpm), and 0.3989 (220 rpm) strain per minute respectively is shown in Figure I.

Figures II and III illustrate the variation of ρ corrected and true stress with $\epsilon^{\frac{1}{2}}$ respectively for different rates of strain.

The reproducibility of these results is within $\pm 0.5\%$. Fully tabulated results are given elsewhere.²

4. Discussion

4.1 Strain and Density of Dislocations

The relationship between shear strain (ϵ) and the density of dislocations (N) is well established.¹¹ For polycrystalline extruded rods or drawn copper wire it could be⁹ shown that

$$N = A\epsilon \quad (1)$$

where A is constant viz 2.8×10^8

It was shown by Young¹¹ that the relationship given by equation (1) is valid over the entire elastic and plastic range for metals such as Cu, Ag, Ni, and their alloys.

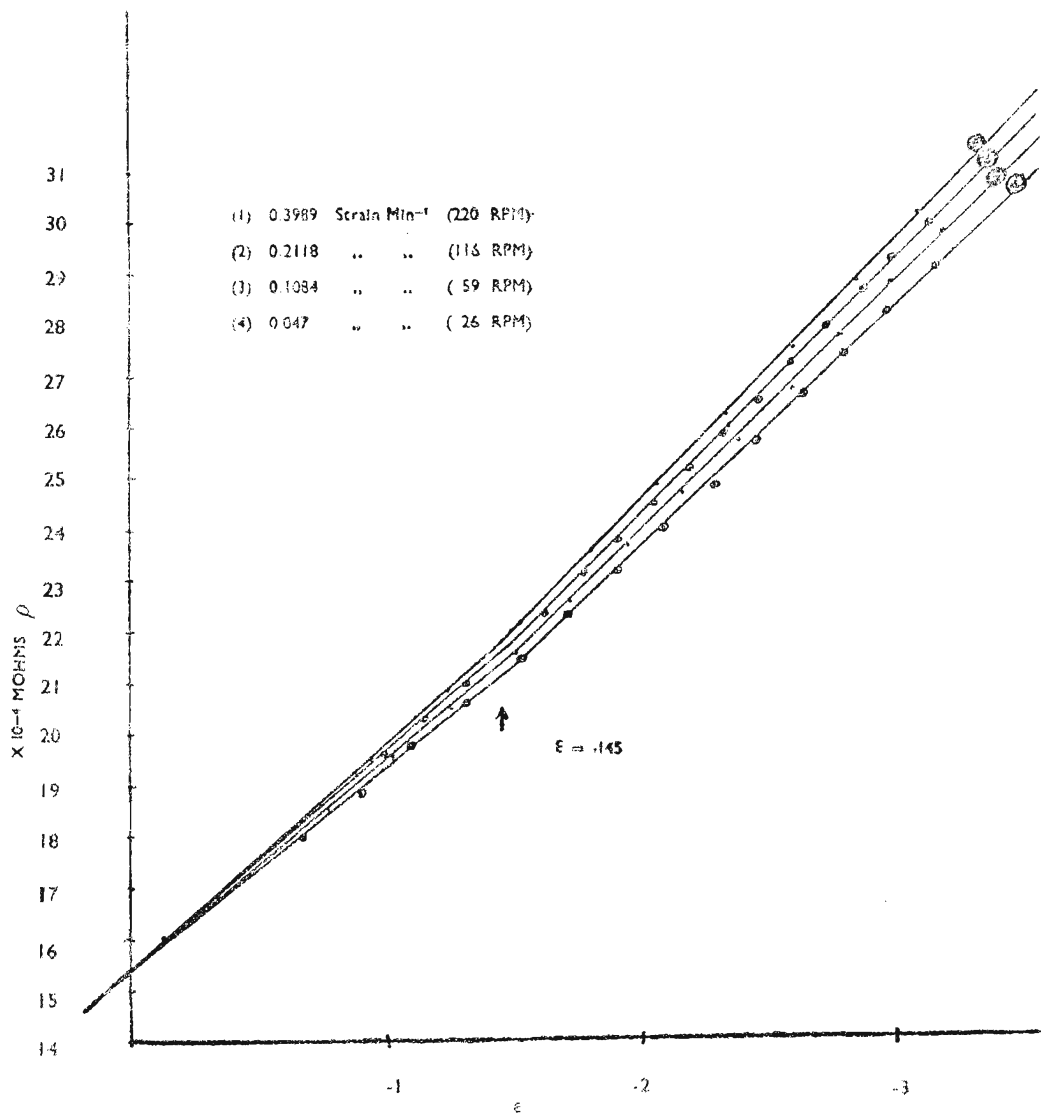


Figure 1 Uncorrected resistivity (ρ) vs true strain (ϵ) for different rates of strain

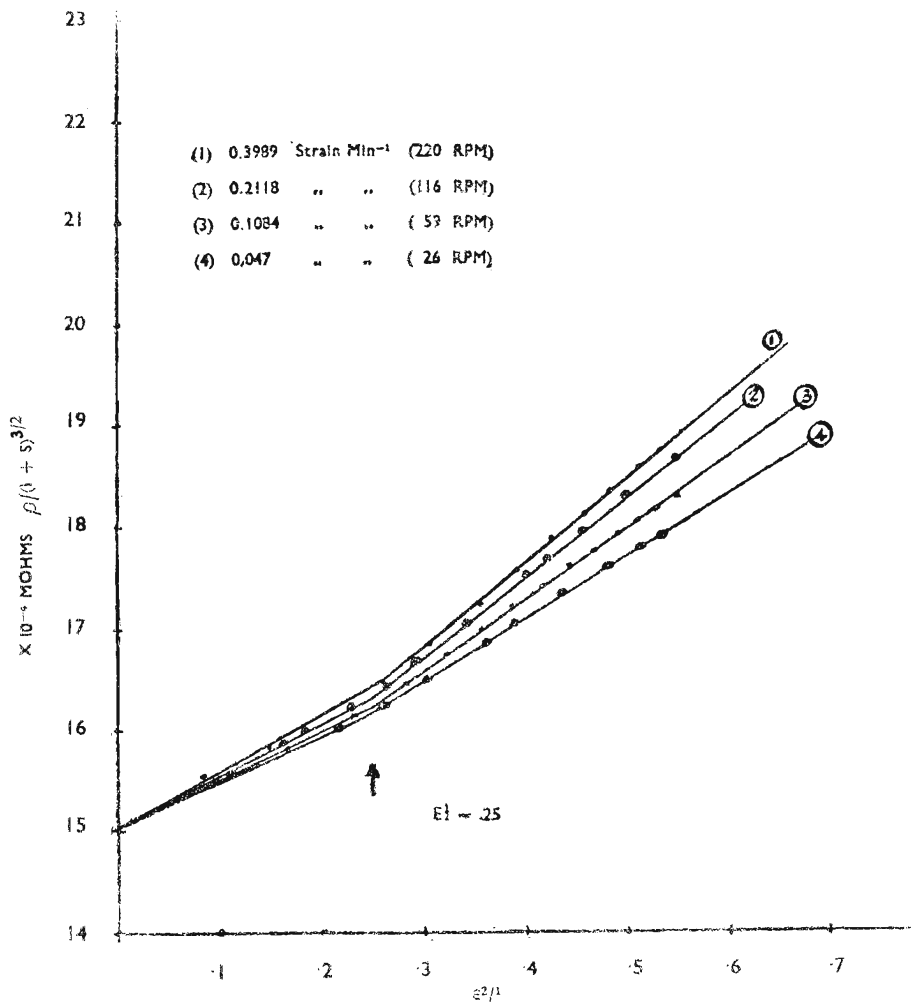


Figure. II Corrected resistivity (ξ) vs (true strain)^{1/2} for different rates of strain

100-1007

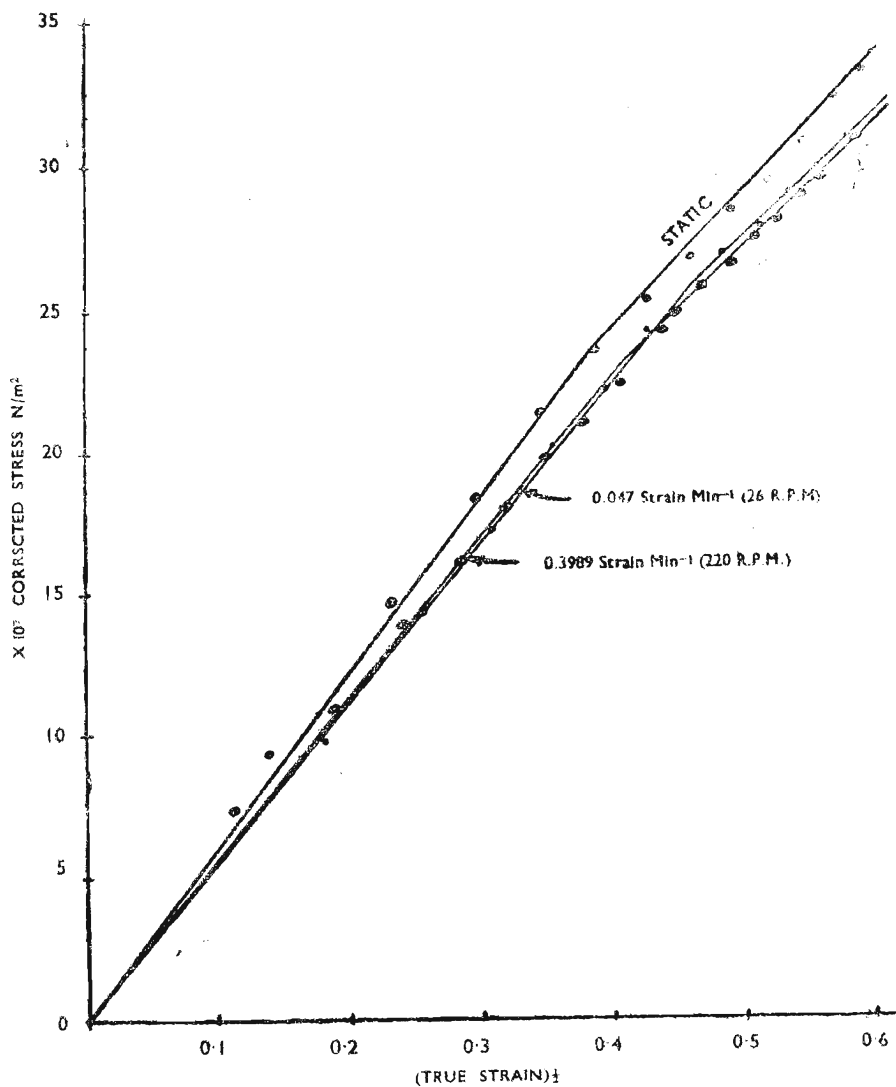


Figure. III Corrected stress vs (true strain)^{1/2} for different rates of strain

4.2. Dynamic Strain and Density of Dislocations

Static stress-strain relations are often influenced by concomitant relaxation effects that often prevail during testing. When a crystal is strained, a certain density of dislocations is produced according to equation (1). But due to thermal agitation a certain fraction of these would get annealed. Consequently instantaneous density of dislocations would depend also on the rate of strain and this density would drop as the crystal relaxes. It is therefore necessary to derive an expression taking into account this relaxation effect. The rate of annealing of the dislocations may be assumed to be proportional to the density of dislocations. Hence the differential equation for the rate of change of dislocations with time under dynamic strain may be written as

$$\frac{dN}{dt} = \frac{Ad\varepsilon}{dt} - hN \quad (2)$$

where h is the rate constant for the assumed first order annealing reaction. h is expected to be characteristic of the metal and dependent only on the temperature, provided the shear rate is not so large as to produce an appreciable generation of heat in the specimen.

The solution of the differential equation may be written as

$$N = A\varepsilon \left[1 - \frac{S^2 h}{2\varepsilon r} \right] \quad (3)$$

where S is simple strain given by $S = rt$ in which r is a constant determined by the speed set for the operation and t is the time.

4.3. Resistivity and True Strain

It is well known that the resistivity of a metal (ρ) is the sum of resistivities due to scattering of electrons by phonons, impurities and structural deformations, otherwise known as Mathiessen Rule.

$$\rho = \rho_{\text{thermal}} + \rho_{\text{impurities}} + \rho_{\text{deformations}} \quad (4)$$

The electron scattering, and resistivity of a metal are related by the equation

$$\rho = \frac{2 m U}{nq^2 \lambda} \quad (5)$$

where m is the effective mass of an electron, n is the number of free electrons per unit volume, q is the electronic charge, U is the average velocity of the electron and λ is the mean free path of the electron. Therefore, equation (4) can be written as

$$\rho = \frac{2 m U}{nq^2} \left[\frac{1}{\lambda_{\text{thermal}}} + \frac{1}{\lambda_{\text{imp}}} + \frac{1}{\lambda_{\text{deformation}}} \right] \quad (6)$$

When a metal is strained at constant temperature, the contributions to the electrical resistivity by phonon scattering and impurities scattering may be regarded as constant. Therefore the increase in resistivity of a metal under strain would largely depend on deformation scattering which in turn would vary with the density of dislocations produced.

For pure copper, the equation (6) can be written as

$$\rho = \frac{2 m U}{nq^2} \frac{1}{\lambda_0} + \frac{2 m U}{nq^2} \frac{1}{\lambda} \text{ deformations} \quad (7)$$

where λ_0 is the mean free path of the electron for phonon and impurities scattering when the strain (ϵ) is zero. If λ is regarded as the mean distance between two dislocation lines⁴ we have

$$\lambda = \frac{1}{2 N^{\frac{1}{2}}} \quad (8)$$

$$\therefore \rho_{\epsilon} = \rho_{\epsilon=0} + \frac{4 m U}{nq^2} N^{\frac{1}{2}} \quad (9)$$

It is well known that $N = A\epsilon$, where A is a constant, termed as the density of dislocation per unit true strain.

\therefore Equation (9) can be rewritten as

$$\rho_{\epsilon} = \rho_{\epsilon=0} + \frac{4 m U}{nq^2} \cdot A^{\frac{1}{2}} \epsilon^{\frac{1}{2}} \quad (10)$$

According to this equation, ρ_{ϵ} is proportional to $\epsilon^{\frac{1}{2}}$. Figure II shows that ρ corrected for dimensional changes is proportional to $\epsilon^{\frac{1}{2}}$ for various rates of strain. For any particular rate of strain, it is found that the gradient changes at $\epsilon^{\frac{1}{2}} = 0.25$ (or $\epsilon = 0.0625$) showing an increase in the constant of proportionality (A).

Under dynamic strain conditions, a certain number of dislocations would get annealed according to equation (3). If this value of N is substituted in equation (9), we have

$$\rho_{\epsilon} = \rho_{\epsilon=0} + \frac{4 m U}{nq^2} \cdot A^{\frac{1}{2}} \epsilon^{\frac{1}{2}} \left[1 - \frac{S^2 h}{2\epsilon r} \right]^{\frac{1}{2}} \quad (11)$$

The above equation can be rewritten as

$$\rho'_{\epsilon} = \frac{\rho_{\epsilon}}{\left[1 - \frac{S^2 h}{2\epsilon r} \right]^{\frac{1}{2}}} = \rho'_{\epsilon=0} + \frac{4 m U}{nq^2} \cdot A^{\frac{1}{2}} \epsilon^{\frac{1}{2}} \quad (12)$$

where ρ'_{ϵ} refers to the resistivity corrected for dimensional changes and healing or annealing of dislocations due to thermal agitations.

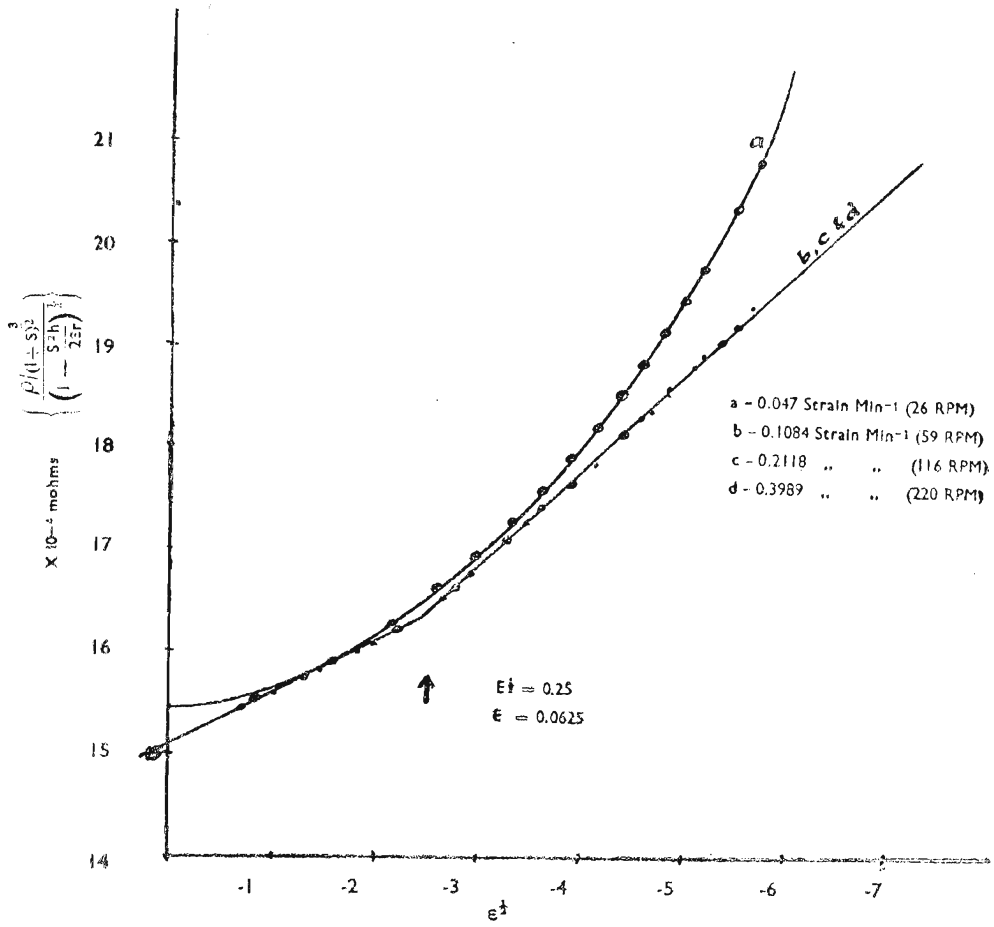


Figure IV. Resistivity corrected for dimensional changes and healing of dislocations vs (true strain)_t at h=0.05

The Figure IV shows a plot of ρ'_ϵ vs $\epsilon^{\frac{1}{2}}$ for the value of $b = 0.05$ for different rates of strain. The graphs corresponding to 0.1084 (59 rpm), 0.2118 (116 rpm) and 0.3989 (220 rpm) strain per minute emerge into one graph showing the validity of equation (12). The common graph takes the form of two straight lines with different gradients. The gradient changes to a higher value corresponding to a strain of $\epsilon = 0.0625$.

It was shown by Hoar³ and others and confirmed by Devanathan and Fernando⁴ that below a critical straining rate of 5%, anomalous results are obtained as observed for 0.0470 (26 rpm) strain per minute.

4.4 Calculation of A by Resistivity

The gradient of the graph ρ'_ϵ vs $\epsilon^{\frac{1}{2}}$ is given by $\frac{4mU}{nq^2} A^{\frac{1}{2}}$. Further we know that

$\rho_{\epsilon=0}$ is given by $\frac{2mU}{nq^2} \frac{1}{\lambda}$. By experiment it is found that $\rho_{\epsilon=0}$ is 1.515×10^{-6}

ohm-cm (Vide Fig. IV). In the case of copper, assuming the standard values of 1.578×10^6 for U and 8.426×10^{-8} for λ , the relative value of $\frac{m}{nq^2}$ is found to be 4.03×10^{-20} . The common graph of resistivity corrected for dimensional changes and healing vs $\epsilon^{\frac{1}{2}}$ takes the form of two straight lines (Vide Fig. IV). Taking the first gradient, A is found to be 3.92×10^{12} whereas for the second A is 1.56×10^{13} .

4.5 Taylor --- Mott Equation

Of the many theories of work hardening, the Taylor-Mott theory^{6,7,10} of dislocation interaction, gives by far the best overall explanation. According to this theory, the stress should be linearly proportional to (strain) ^{$\frac{1}{2}$} over the entire range. Experimental stress-strain curves obtained under static conditions show that this relationship is a good first approximation. The belief that this relationship is not exactly valid has led to the plotting of stress vs strain curves rather than those of stress vs (strain) ^{$\frac{1}{2}$} . The former graphs suggest a linear work hardening region followed by a straight line with a lower gradient.

For the understanding of the strength of metals, it appears that the establishment of a law applicable to work hardening region is an essential pre-requisite for further progress. It is now usual to plot stress vs strain relations obtained at pre-determined rates of strain. However, even during strain at a pre-determined rate, specimen tends to relax and this effect distorts the stress-strain relationships.

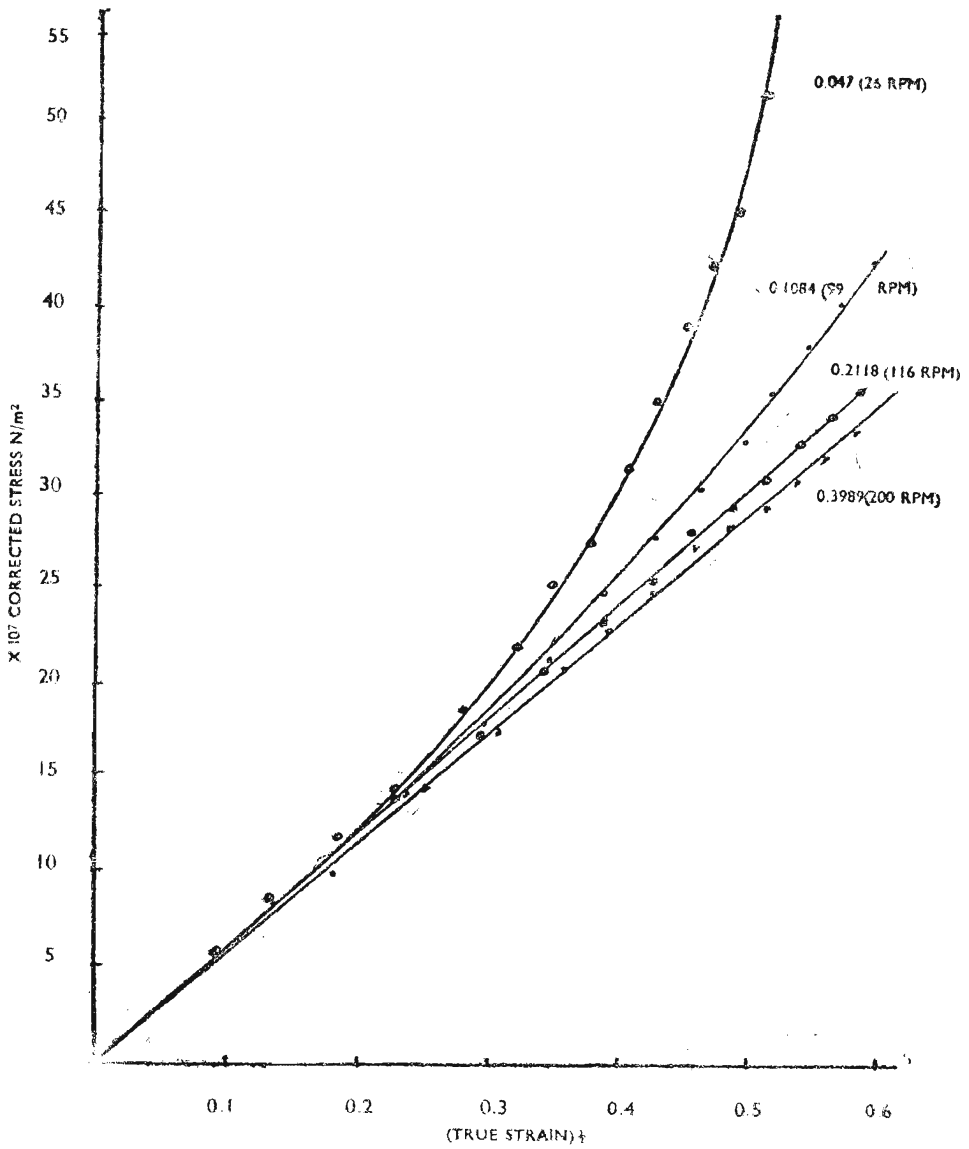


Figure V. Corrected stress vs (true strain) $^{\frac{1}{2}}$ at $h=0.1566$

From the well known theory of Taylor-Mott, it can be shown that for copper, the stress (τ) is given by

$$\tau = \frac{1}{2} b G N^{\frac{1}{2}} \quad (13)$$

Replacing N by $A\varepsilon$ equation (13) becomes

$$\tau = \frac{1}{2} b G A^{\frac{1}{2}} \varepsilon^{\frac{1}{2}} \quad (14)$$

According to this equation, τ vs $\varepsilon^{\frac{1}{2}}$ should be a straight line. Since the dynamic stress-strain relations are more reliable than the static in understanding metal behaviour, it is better to plot τ vs $\varepsilon^{\frac{1}{2}}$ under dynamic conditions as shown in Figure III. A straight line is obtained up to $\varepsilon = 0.15$ after which the points deviate from it. The deviating points appear to fall on another straight line with a somewhat smaller gradient. The Taylor-Mott theory does not provide any explanation for this deviation from linearity at high values of ε .

4.6. Validity of Taylor-Mott Expression

As pointed out in Section 5, the equation (13) needs a modification to account for the deviation from linearity at high values of ε , taking into consideration the relaxation effect.

Substituting for N from equation (3) in equation (13), we obtain,

$$\tau = \frac{1}{2} b G A^{\frac{1}{2}} \varepsilon^{\frac{1}{2}} \left[1 - \frac{S^2 h}{2\varepsilon r} \right]^{\frac{1}{2}} \quad (15)$$

If τ_c is defined as $\tau / \left[1 - \frac{S^2 h}{2\varepsilon r} \right]^{\frac{1}{2}}$, equation (15) becomes

$$\tau_c = \frac{1}{2} b G A^{\frac{1}{2}} \varepsilon^{\frac{1}{2}} \quad (16)$$

If the correction factor involved in the term $\left[1 - \frac{S^2 h}{2\varepsilon r} \right]^{\frac{1}{2}}$, adequately represents the healing rate, τ_c vs $\varepsilon^{\frac{1}{2}}$ must be a straight line for all strains and all rates of strain.

Figure V shows a plot τ_c vs $\varepsilon^{\frac{1}{2}}$ for $h = 0.1566$ obtained from the electrolytic studies. Since the electrolytic experiments would be biased towards surface effects, it is reasonable to expect that h would be too large a factor, in other words there would be an over correction as seen in Figure V. Since there is no a priori method of calculating h , we may select suitable values for h generally less than 0.1566 which would give adequate correction. Some values of h tested in this way are $h = 0.10$ and $h = 0.05$. Figure VI shows a plot of τ_c vs $\varepsilon^{\frac{1}{2}}$ for $h = 0.05$.

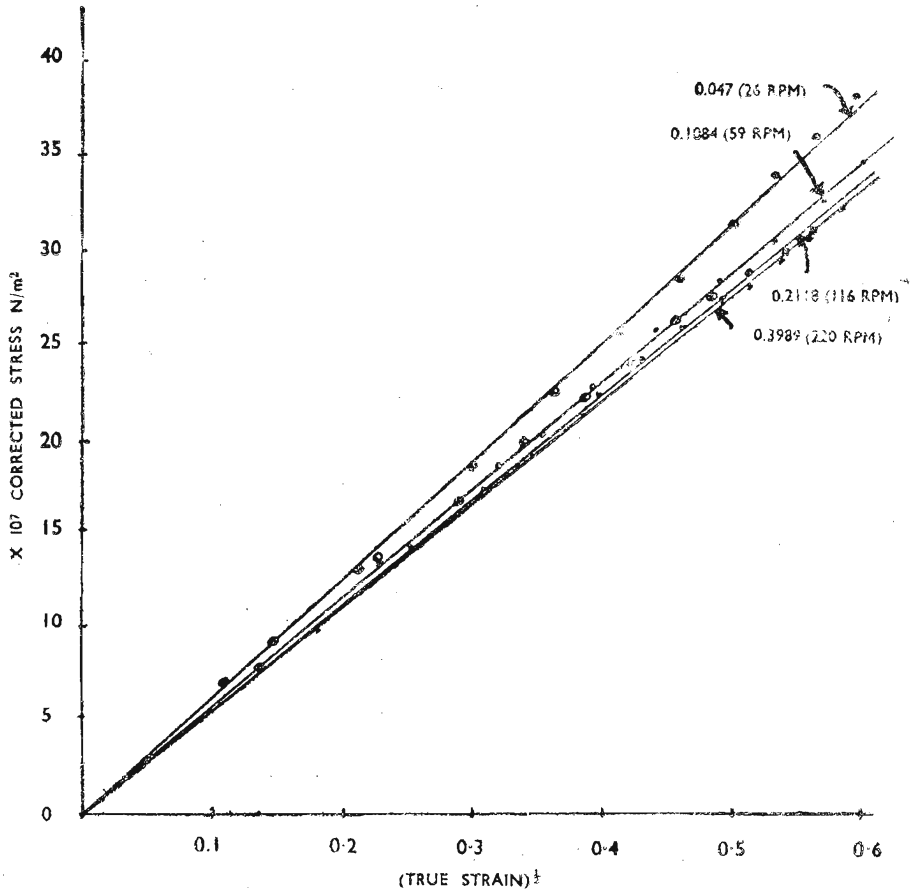


Figure VI. Corrected stress vs (true strain)^{1/2} at h = 0.05

With a value of 0.05 for h , all graphs for different rates of strain are straight lines but with slightly varying gradients depending on the rate of strain with the slowest rate having a higher gradient than the larger rate.

The fact that straight lines without kinks are obtained confirms the validity of the expression given by the equation (15). The graphs for speeds corresponding to strain/min 0.1084 (59 rpm), 0.2116 (116 rpm) and 0.3989 (220 rpm) nearly coincide with only one speed corresponding to 0.0470 strain/min (26 rpm) slightly higher. Slight variations of the gradients in the order of $59 > 116 > 220$ rpm may be attributed to the effective temperature being slightly higher during work hardening at increasing rates of strain. Hoar and West³ have shown that dynamic strain behaviour falls into a uniform pattern only if the strain rate exceeds a certain critical value 0.05 strain/min. The 0.0470 strain/min is just below the critical value. It is possible that the reason for this small deviation may lie in a variation of the rate constant for the healing process when time is large.

The validity of equation (13) shows that the Taylor-Mott expression for work hardening is basically correct. The apparent failure of the equation at high strains is due to the incorrect value of N used. When N is corrected for relaxation effects, the Taylor-Mott expression is found to be applicable throughout the entire strain range and for all rates of strain.

4.7. Calculation of A by Stress-strain Curves

As shown in equation (16), a value for A can be determined from the gradient of τ_c vs $\epsilon^{\frac{1}{2}}$ curve. A is found to be 7.24×10^{10} as obtained from the gradient of Figure VI. A obtained from etch pit counts and from our experiments on anodic dissolution¹ is 3×10^8 .

From these values it would appear that either one in 240 dislocations is effective for anodic dissolution or on the average, 240 dislocations cluster to give rise to one etch pit or dissolution site.

4.8. Significance of A Values

The value of A calculated as above refer to the density of dislocations at unit ϵ . However, since the rupture point of copper is at 0.3ϵ , the maximum number of dislocations at rupture point is $0.3A$.

For copper, the density of dislocations from transmission electron microscopy is 10^{11} to 10^{12} . This may be regarded as the maximum value possible for the density at its rupture point. From the value of A obtained by the measurements of resistivity in our experiments, the density of dislocations is in the range of 10^{12} to 5×10^{12} . Since both methods are based on the scattering of electrons by dislocations, the agreement is not unexpected.

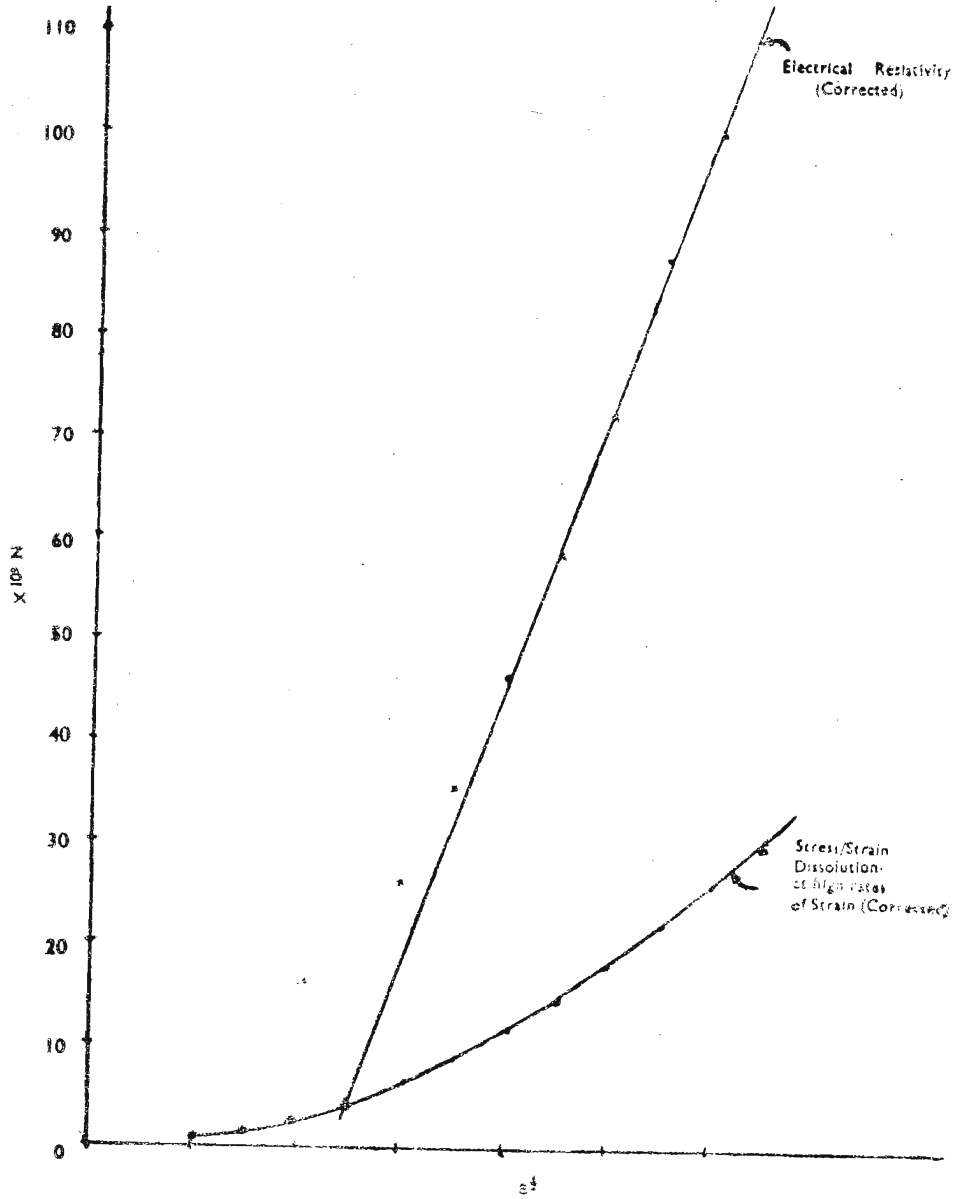


Figure VII. Absolute number of dislocations vs (true strain)^{1/2}

On the other hand, as mentioned in Section 7, the A values obtained by etch pit counts and anodic dissolution are in excellent agreement since both techniques are based on preferential dissolution of surface sites to which dislocations converge. At the rupture point of 0.3ϵ , the density of dislocations as calculated by these methods would correspond to 10^8 . This value is 10^4 times smaller than that obtained by electron scattering. This clearly indicates that not all dislocations are effective in producing sites for etching or anodic dissolution.

From the stress-strain curves, the value of A obtained is 7.24×10^{10} corresponding to a value of 2.4×10^{10} for the density of dislocations at rupture point. This shows that more dislocations are effective in enhancing the mechanical behaviour.

The density of dislocations calculated by anodic dissolution, stress-strain curves, and electrical resistivity is in the ratio of $1 : 10^2 : 10^4$ approximately. Therefore, it is clear that the density of dislocation largely depends on what method is adopted to 'see' the dislocations.

The Figure VII shows a plot of the absolute number of dislocations (N) with ϵ^3 for the three different methods under consideration. The anodic dissolution curve and the electrical resistivity curve have been reduced to the same scale as the stress strain curve by normalizing the 'A' factor by g_1 and g_2 (where $g_1 = 2.41 \times 10^2$ and $g_2 = 1.86 \times 10^{-2}$) respectively.

The fact that the graph of the dislocation density vs ϵ^3 for anodic dissolution coincides with that for stress-strain behaviour shows that for all strains,

$$\frac{N \text{ (anodic dissolution)}}{N \text{ (stress-strain)}} = \text{constant} \left(\frac{1}{g_1} \right)$$

and

$$\frac{N \text{ (electrical resistivity)}}{N \text{ (stress-strain)}} = \text{constant} \left(\frac{1}{g_2} \right) \text{ up to}$$

$$\epsilon^3 = 0.25.$$

But beyond $\epsilon^3 = 0.25$, more dislocations are created although not all are effective in enhancing the work-hardening. The existence of these cannot therefore be inferred from the stress-strain or the anodic dissolution data, but the electrical resistivity measurements are sensitive enough to detect them.

Unlike the electron-transmission microscopy techniques which cannot be applied to these specimens under strain in situ, the electrical resistivity method appears to be more useful.

It is interesting to calculate the mean free path for electron scattering due to dislocations under strain. By electrical resistivity measurements, it can be shown that mean free path gradually changes from 300 Å° at zero strain to 25 Å° at the rupture point corresponding to $\epsilon = 0.3$.

At the characteristic turning point in the density of dislocations, corresponding to $\epsilon = 0.0625$ (Vide Figure IV) the mean free path for electron scattering is found to be 100 Å°.

The measurement of electrical resistivity is a useful method for monitoring dislocations in metals.

Acknowledgements

We wish to thank Mr. S. F. Laurentius, Director of the Ceylon Institute of Scientific and Industrial Research, Colombo 7, Ceylon for providing laboratory facilities and for his interest.

We also thank Dr. M. H. C. Wijetunge for helpful discussions.

References

1. DEVANATHAN, M. A. V. & FERNANDO, M. J., (1970) *Electro Chim. Acta*, **15** : 1623—1636. Oxford : Pergamon Press.
2. FERNANDO, M. J. (1969), Ph.D. Thesis, University of Ceylon, Colombo.
3. HOAR, T. P. & WEST, J. M. (1962), *Proc. Roy. Soc. Sec. A.*, **268** : 304—315.
4. KITA, H., BOCKRIS, J. O' M. & ENYO, M. (1961), *Can. J. Chem.*, **39** : 1670.
5. MARIN, J. (1962), *Mechanical Behaviour of Engineering Materials*, New York : Prentice Hall.
6. MOTT, N. F. (1952), *Phil. Mag.*, **43** : 1151.
7. MOTT, N. F. (1960), *Trans. Am. Inst. Min. Metall. Engrs.*, **218** : 962.
8. MOTT, N. F. & JONES, H., (1958), *The Theory of the Properties of Metals and Alloys*, New York: Dover Publications.
9. SCHMIDT, E. & WASSERMANN, G. (1927). *Z. Phys.* **42** : 779.
10. TAYLOR, G. I., (1964). *Proc. Roy. Soc. Sec. A.* **145** : 362.
11. YOUNG, F. W., (1962), *J. Appl. Phys.* **33** : 963.