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DISLOCATION MOBILITY IN PURE COPPER AT 4.2°K*

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ABSTRACT

A dislocation damping coefficient equal to 1.0×10^{-5} dyne sec/cm² is deduced from etch pit measurements of fast-moving ($\sim 2 \times 10^4$ cm/sec) edge-oriented dislocations in copper single crystals at 4.2°K. Comparison of the results with previous mobility studies in copper indicates that the damping coefficient decreases monotonically with decreasing temperature. The present results are consistent with theoretical predictions concerning the magnitude of electronic damping of dislocation motion at very low temperatures.

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DISLOCATION MOBILITY IN PURE COPPER AT 4.2°K

I. INTRODUCTION

There exists in the literature conflicting experimental evidence in regard to electronic damping of dislocation motion in metals. Several investigators have reported that electronic damping increases with decreasing temperature (e.g. Mason and Rosenberg¹ in aluminum, Mason and MacDonald² in niobium, and Parameswaran and Weertman³ in lead) and at the lowest temperatures exceeds the magnitude of the total dislocation damping at room temperature. Other investigators (e.g. Alers and Thompson⁴ and Fate⁵ in copper) have reported only a small residual electronic damping component in the liquid helium temperature range.

In both cases, the majority of the results were derived from an internal friction experiment in which the attenuation of an ultrasonic stress pulse propagated through a single crystal was interpreted in terms of the Granato-Lucke theory⁶ (one notable exception is the work of Parameswaran and Weertman³ in which a stress pulse technique was employed). Unfortunately, this approach has a major weakness in that the distribution of dislocation loop lengths and total dislocation density, parameters that are important in the theory, cannot be accurately measured.

Hikata and Elbaum⁷ employed Rogers'⁸ extension of the Granato-Lucke theory to develop an internal friction technique whereby the dislocation damping coefficient of superconducting metals in the normal state was determined in the vicinity of their transition temperatures without prior knowledge of the characteristics of the dislocation network. Hikata and Elbaum derived from their measurements on lead single crystals a small residual damping equal to 8.6×10^{-5} dyne cm/cm², that was independent of

temperature between 4.2°K and 11°K .

In a later work, Hikata, Johnson, and Elbaum⁹ reported the development of a bias stress technique for use in internal friction experiments and here again the damping coefficient could be measured without knowledge of the test specimen's dislocation density (although assumptions were made concerning the distribution of the dislocation loop lengths). This technique was applied to an aluminum single crystal where a small temperature independent damping coefficient equal to 1.4×10^{-5} dyne sec/cm² was found below 50°K .

All of the internal friction measurements are made at low stress levels so that dislocation velocities do not exceed approximately 10 cm/sec or about 10^{-4} of the lowest sonic velocity. For dislocation velocities much less than the lowest sonic velocity, the theory of the conduction electron-dislocation interaction is well understood,^{10, 11, 12} and gives a temperature-independent damping coefficient of the order of 10^{-5} to 10^{-4} dynes cm/sec² for metals such as copper, aluminum and lead, in good agreement with the experiments of Hikata and Elbaum⁷ and Hikata, Johnson, and Elbaum⁹. It was pointed out¹³ that for the reasonably large dislocation velocities (10^2 to 10^4 cm/sec) obtainable in stress-pulse mobility experiments, the theoretical treatment applicable in the low velocity limit remains valid. To the authors' knowledge the only experimental measurement of the electronic damping coefficient in the high velocity region is the work in lead³, where a large temperature-dependent electronic damping coefficient was reported in the liquid helium temperature range.

With the aim of providing further evidence for the behavior of the damping coefficient in the high velocity region, the present investigators

carried out a set of dislocation mobility experiments with very high purity copper single crystals. Torsional stress pulses of microsecond duration were applied to cylindrical crystals on which the displacement of selectively introduced dislocations was measured by an etch pit technique.

2. EXPERIMENTAL TECHNIQUES

2.1 Outline of Experimental Procedure

The experimental procedure can be outlined as follows:

1. A copper single crystal is electrolytically machined into a right circular cylinder with the cylindrical axis in a $\langle 110 \rangle$ direction. Two narrow plane surfaces, parallel to a set of $\{111\}$ -oriented planes which contain the cylindrical axis, are electrolytically machined on opposite lateral surfaces of the crystal (Fig. 1).
2. These surfaces are scratched with a thin needle to produce edge dislocations on intersecting $\{111\}$ planes.
3. The positions of these dislocations are determined by etching the two lateral plane surfaces.
4. One $\{110\}$ end surface of the crystal is bonded to a torsion machine. The system is thereupon cooled to liquid helium temperature, following which a torsional stress pulse is applied to the crystal.
5. The positions of displaced dislocations are established by re-etching the two scratched surfaces.

2.2 Test Specimens

A copper single crystal, 3.2 cm in diameter, and with an orientation near a $\langle 111 \rangle$ direction was grown from 99.999 percent pure material by a modified Bridgman technique. This parent crystal was acid-sliced and electrolytically machined into cylindrical test crystals 1.3 cm in diameter, about 2.5 cm long, and with the cylindrical axis within 0.25° of a $\langle 110 \rangle$ direction. Two plane $\{111\}$ -type surfaces, approximately 2 mm wide, were electrolytically machined on opposite lateral sides of each test crystal, as shown in Fig. 1.

The test crystals were annealed for two weeks at 1020°C in a purified argon atmosphere. Following this, the crystals were electrolytically polished at -50°C to remove traces of evaporation pits. Etching the $\{111\}$ surfaces of annealed crystals revealed between 10^2 and 10^3 dislocation etch pits per cm^2 . The crystals were generally free of substructure.

2.3 The Selectively-Introduced Dislocations

Three sets of $\{111\}$ slip planes intersect each lateral plane surface. Their traces in that surface are given by three $\langle 110 \rangle$ Burgers vector directions, one parallel to the cylindrical axis of the crystal, the remaining two at angles of 60° . The lateral surface was scratched parallel to one of the latter two directions by means of a thin needle supported in a torsion device. Dislocations were produced on the slip planes whose traces on the surface were the remaining two Burgers vectors. Only the movement of edge dislocations lying in the vertical set of slip planes (with normal direction x_1 in Fig. 1) were studied in this experiment. These were quite easily recognizable because their shear stress resolving factor for the torsion stress was twice that of any other type of dislocation intersecting the lateral surfaces, and they moved considerably farther during the torsion test.

2.4 Observation of Dislocations

A standard double-etch technique was used to reveal the positions of dislocations on the $\{111\}$ lateral surfaces both before and after displacement by the applied torsion stress. After the torsion test, but before the final etch, each test crystal was electropolished for several seconds at -50°C to remove any tarnish that appeared on the crystal during contact with the atmosphere.

2.5 The Torsion Test

The torsion-testing machine used in previous mobility studies in copper^{14, 15} was employed in this experiment. In brief: One end surface of a test crystal is bonded to a circular steel rod by means of an organic solvent (liquid propane and isopentane in a volumetric ratio of 10:1) which hardens at 44°K but does not plastically deform the specimen to any significant extent when cooled to 4.2°K. A torsional shear stress is applied to the test crystal by the propagation of a torsional stress wave down the circular torsion rod and into the crystal. The stress wave reverses sign and direction upon reaching the bottom, free end of the crystal, propagates upwards and reduces the applied stress to zero. Hence, the stress state at any horizontal cross-section of the crystal consists of a loading torsional wave followed later by an equivalent unloading wave. The time between the arrival of the loading wave and that of the unloading wave is proportional to the distance from the cross-section to the free end, and is given by twice that distance divided by the torsional shear wave velocity.

The state of torsion is monitored by a set of strain gages attached to the torsion rod several cm above the test crystal, and is recorded on an oscilloscope.

3. EXPERIMENTAL RESULTS

3.1 Torsional Stresses in the Test Crystal and Resolved Shear Stress on Dislocations.

The shape of the applied stress pulse in this experiment was essentially the same as that reported in previous mobility studies in copper.¹⁴ In Appendix A of Ref. 14, it was explained that the state of torque at the surface of the test crystal bonded to the torsion rod is calculated from the oscilloscope record of strain gage output, with knowledge of the acoustic impedances of the torsion rod, bonding agent, and test crystal. In Appendix B of Ref. 14, it was shown how the anisotropic elastic properties of the test crystal and a shear modulus defect due to the presence of grown-in dislocations distort somewhat the torsional stress state in the crystal. The anisotropic effects in the particular case of a $\langle 111 \rangle$ cylindrical axis were discussed and found to be unimportant. In the Appendix of this paper, we show that for the $[110]$ -oriented crystals used in this investigation, the stress state is not distorted from that of pure torsion along the $[\bar{1}1\bar{1}]$ direction.

To obtain the stress-time history for an individual dislocation produced by the scratching procedure and displaced by the torsional stress, the following procedure was adopted: From the strain-gage record, the state of torque is calculated as a function of time at the top surface of the test crystal. This torque-time history is separated into its two components, loading and unloading wavefronts. The average distance of the dislocation in question from the free end of the crystal is then measured (equal to the distance from the point midway between initial and final etch pits to the free end). The loading and unloading wavefronts are recombined into a

torque-time profile where the time between arrival of the initial and reflected waves is reduced with respect to that at the top surface of the crystal by the ratio of the average distance to the length of the crystal.

The resolved shear stress $\tau_R(t)$ on a dislocation of edge orientation at the lateral surface (Fig. 1) is given in terms of the torque at that cross-section $M(t)$, by

$$\tau_R(t) = \frac{0.94 r_o}{I} M(t) , \quad (1)$$

where 0.94 is the shear stress resolving factor, r_o is the radius of the test crystal equal to 0.635 cm, I is the moment of inertia about the cylindrical axis, equal to 0.255 cm^4 , and t is time.

3.2 Dislocation Displacements and the Measurement of Dislocation Mobility.

Four crystals were tested in the torsion machine, at three different values of applied torque (equal to 1.2×10^6 dyne cm for two tests, 2.4×10^6 dyne cm for one test, and 3.1×10^6 dyne cm for one test). For the two tests at the lowest applied torque, detectable dislocation displacement from the scratches occurred only in a few instances, indicating that the resolved shear stress at the lateral surface ($\sim 2.8 \times 10^6$ dyne/cm²) was generally not sufficiently large to cause dislocation motion. At the highest applied torque, dislocation displacement from the scratches was uniform (see Fig. 2).

The displacements and average distances from the free end of the crystal were measured for the dislocations moving farthest from each scratch. The time integral of stress for each of these dislocations, $\int \tau_R(t) dt$, was

determined as indicated in Section 3.1. Dislocation displacement is plotted as a function of $\int \tau_R(t)dt$ in Fig. 3. Noting that the time duration of stress is of the order of $10 \mu\text{sec}$,¹⁴ we determine from Fig. 2 that dislocation velocities of the order of $2 \times 10^4 \text{ cm/sec}$ were realized.

The relation between dislocation displacement, d , and dislocation velocity, v , is.

$$d = \int v(t)dt, \quad (2)$$

while from Fig. 3

$$d = K \int \tau_R(t)dt, \quad (3)$$

where $K = 2.58 \times 10^{-3} \text{ cm}^3/\text{dyne sec}$ is the maximum slope through the experimental points. Comparing Eq. 2 with Eq. 3 we find

$$v = K \tau_R. \quad (4)$$

The dislocation drag coefficient, B , (defined by $Bv = \tau_R b$, where b is the dislocation Burgers vector) is then given by.

$$B = \frac{b}{K} \quad (5)$$

$$= 1.0 \times 10^{-5} \text{ dyne/sec/cm}^2,$$

where $b = 2.55 \times 10^{-8} \text{ cm}$. In Fig. 4, we have plotted the temperature dependence of B as determined in this experiment and in previous investigations.^{14, 15}

4. DISCUSSION OF RESULTS

4.1 Characteristics of Dislocation Motion

Our experimental results (Section 3.2) indicate the existence of a resolved shear stress τ_o ($\sim 2.8 \times 10^6$ dynes/cm²) below which the dislocations introduced by scratching remain immobile. Two possible origins of this effect are postulated; (i) the dislocations are strongly pinned during the scratching procedure and, on the average, the critical stress necessary to break the dislocations free is τ_o , or (ii) the dislocations introduced by the scratching procedure contain a density of sessile jogs that exert a constant retarding force approximately equal to $\tau_o b$ per unit length on a moving dislocation. Condition (i) will have a strong retarding influence on dislocation mobility for those applied stresses τ_R that are just slightly greater than τ_o ¹⁶, but is unimportant for applied stresses significantly larger in magnitude¹⁷. Condition (ii) alters the form of the velocity-stress relation to

$$Bv = (\tau_R - \tau_o)b, \quad \tau_R > \tau_o, \quad (6)$$

and in terms of the dislocation displacement d ,

$$d = \frac{b}{B} \left[\int \tau_R(t)dt - \int \tau_o dt \right]. \quad (7)$$

We note from Fig. 3 that Eq. 3 is deduced from the data points of test 4, so at least in this case $\int \tau_o dt \ll \int \tau_R(t)dt$ and the retarding effects of sessile jogs is unimportant. Hence dislocation motion away from the scratch in test 4 is characterized by two steps: (i) Rapid expansion of a pinned dislocation loop until breakaway takes place, followed by (ii) expansion of the freed loop at a high dislocation velocity.

4.2 Comparison with Previous Work and with Theory

An interesting aspect of the experimental results reported herein and in previous work by the same authors^{14, 15} is the monotonically decreasing magnitude of the damping coefficient with decreasing temperature (Fig. 4), a result which is at variance with the direct mobility work in lead single crystals³.

The damping coefficient at 4.2°K reported in this investigation is of the same order of magnitude as that of lead⁷ and aluminum⁹ determined by modified internal friction techniques, although three orders of magnitude of dislocation velocity separate the present work from the internal friction experiments. According to a theoretical calculation¹³, which includes some small corrections to the theory of the conduction electron-dislocation interaction, the edge dislocation damping coefficient is given by the following expression;

$$B_{\text{edge}} = \left[\frac{1}{6\pi} + \frac{1}{96} \left(\frac{1 - 2\nu}{1 - \nu} \right)^2 \right] n_0 m v_F b^2 q_{\text{max}}, \quad (8)$$

where n_0 and m are the free electron density and mass, v_F is the Fermi velocity, ν is Poisson's ratio, and q_{max} is the maximum allowed wavevector in the Fourier decomposition of the velocity of the moving dislocation.

The wavevector q_{max} has two limiting values: If it is assumed that the conduction electrons do not interact directly with the dislocation core, then $q_{\text{max}} \sim 1/d$, where d is the width of the core region. On the other hand, if it is assumed that the core region can support the motion of phonons, then q_{max} is larger and equal to q_D , where q_D is the Debye radius. It is expected that the correct value of q_{max} lies somewhere between the two limits.

The material constants for copper are $n_0 = 8.5 \times 10^{22}$ electrons/cm³, $m = 9.1 \times 10^{-28}$ gm, $v_F = 1.6 \times 10^8$ cm/sec, and $v \approx 0.32$. The Peierls calculation for the core width¹⁸ gives $d = h/(1-v)$, where h is the distance between slip planes equal to $\sqrt{\frac{3}{2}} b$. The Debye radius is $q_D = 1.7 \times 10^8$ cm⁻¹. Substituting these values into Eq. 8, we find

$$0.5 \times 10^{-5} \text{ dyne sec/cm}^2 < B_{\text{edge}} < 2.6 \times 10^{-5} \text{ dyne sec/cm}^2,$$

which is in agreement with our experimental value of 1.0×10^{-5} dyne sec/cm².

APPENDIX

EFFECT OF ELASTIC ANISOTROPY ON THE TORSIONAL STRESS STATE

Pure torsional stresses and displacements cannot be simultaneously realized about a $\langle 110 \rangle$ axis in a cubic crystal lattice. Hence, a purely torsional wave cannot be propagated without dispersion in a $\langle 110 \rangle$ direction in a copper single crystal. The differential equations of motion concerning this behavior are extremely complex, involving elastic stiffnesses that depend on orientation about the $\langle 110 \rangle$ torsion axis, and an exact solution has not been determined. It is reasonable to assume, however, that after the arrival of a torsional wavefront at a $\{110\}$ surface of a crystal, for times comparable in magnitude to the width of a cross section divided by a shear wave velocity, interaction between elements of a cross section will be limited (i. e., plane sections will remain plane, and displacements will not differ significantly from those of pure torsion). On the other extreme, the steady-state solution would be expected to be close to that of pure torsional stress, with attendant warping of the cross section,¹⁹ because in this case the requirement of stress-free lateral boundaries is identically satisfied.

Distortion of the torsional stress state alters the resolved shear stress on mobile dislocations, and reduces the accuracy with which the dislocation drag coefficient B is determined, for B is calculated on the basis of a pure torsional stress state (Sections 3.1 and 3.2). To deduce the error limits for B , the maximum possible distortion of this stress must be calculated. This occurs when the crystal displacements are purely torsional.

As it will be simplest to express the elastic quantities in circular

cylindrical coordinates, we define a convenient coordinate system with the $[110]$ direction of Fig. 1 equal to the cylindrical z -axis or x_3 . We let the x_1 and x_2 axes (r, ϕ coordinates) be in the (110) plane and θ is the angle between x_1 and the $[\bar{1}10]$ direction in that plane. Then the stiffness matrix for the (x_1, x_2, x_3) coordinate system is given by

$$\begin{bmatrix} C_{11} & C_{12} & C_{13} & 0 & 0 & C_{16} \\ C_{12} & C_{22} & C_{23} & 0 & 0 & C_{26} \\ C_{13} & C_{23} & C_{33} & 0 & 0 & C_{36} \\ 0 & 0 & 0 & C_{44} & C_{45} & 0 \\ 0 & 0 & 0 & C_{45} & C_{55} & 0 \\ C_{16} & C_{26} & C_{36} & 0 & 0 & C_{66} \end{bmatrix}$$

The stiffness components we shall need are C_{44} and C_{55} . In terms of the three independent stiffnesses ($C'_{11}, C'_{12}, C'_{44}$) with coordinate axes in the cubic directions, they are:

$$\begin{aligned} C_{44} &= \frac{1}{4}[C - C' \cos 2\theta] \\ C_{45} &= -\frac{1}{4}C' \sin 2\theta, \end{aligned} \tag{A-1}$$

where C is equal to $C'_{11} - C'_{12} + 2C'_{44}$ and C' is the anisotropy factor, given by $C'_{11} - C'_{12} - 2C'_{44}$.

In circular cylindrical coordinates, the only nonzero displacement is

$$u_{\phi} = \alpha r z, \tag{A-2}$$

where α is the angle of twist per unit length. The only nonzero strain is

$$\epsilon_{z\phi} = \frac{1}{2}\alpha r. \tag{A-3}$$

Using the stiffness matrix given above, we find that the nonzero stresses are

$$\tau_{z\phi} = 2C_{44} \epsilon_{z\phi}$$

and

A-4

$$\tau_{zr} = 2C_{45} \epsilon_{z\phi} .$$

If $2r_0$ is the diameter of the crystal, then the torque, M , about the cylindrical axis is given in terms of $\tau_{z\phi}$ by

$$\begin{aligned} M &= \int_{r=0}^{r_0} \int_{\theta=0}^{2\pi} \tau_{z\phi} r^2 dr d\theta \\ &= \frac{\alpha CI}{4} , \end{aligned}$$

A-5

where we have used Eq. A-1 and I is the moment of inertia about the cylindrical axis.

In the $[\bar{1}\bar{1}\bar{1}]$ direction on the (110) surface, $\theta = \cos^{-1} \left(-\sqrt{\frac{2}{3}} \right)$ and the nonzero stresses are given by

$$\tau_{z\phi} = \frac{3C - C'}{6} \epsilon_{z\phi}$$

and

A-6

$$\tau_{zr} = \frac{\sqrt{2}C'}{3} \epsilon_{z\phi} .$$

Finally, $\tau_{z\phi}$ and τ_{zr} are resolved onto the $(\bar{1}11)$ slip plane in the $[110]$ Burgers vector direction to give the resolved shear stress, τ_R , on an edge dislocation at the outer radius of the crystal (Fig. 1). We find

$$\tau_R = 0.94 \tau_{z\phi} + 0.33 \tau_{zr} .$$

A-7

Using Eqs. A-3, A-5 and A-6, Eq. A-7 reduces to

$$\tau_R = \frac{0.94r_o M}{I} . \quad (A-8)$$

This is the same resolved shear stress as found when the distortion due to anisotropy is ignored (Section 3, Eq. 1). Hence along the $[\bar{1}1\bar{1}]$ direction, the two nontorsional components of stress (in Eq. A-6) cancel one another when resolved in the direction of the Burgers vector of the mobile dislocations considered in this paper.

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Fig. 1

The test specimen orientation used in the mobility experiment. The lateral $(\bar{1}\bar{1}\bar{1})$ surface is shown with scratches parallel to a Burgers vector direction at intervals of about 4mm. A dislocation loop produced by the scratching process and lying in the $(\bar{1}\bar{1}\bar{1})$ slip plane with Burgers vector in the $[110]$ direction is shown schematically both before and following expansion by the applied torsion stress. The dislocation intersects the lateral $(\bar{1}\bar{1}\bar{1})$ surface in an edge orientation.

Fig. 2

A scratched area on a lateral ($\bar{1}\bar{1}\bar{1}$) surface of a test crystal after testing in the torsion machine at 4.2°K. The few large etch pits indicate the low background dislocation density before the torsional stress was applied. The long vertical slip bands trace the movement of individual dislocations from the scratch at the bottom of the photograph. The scratch contained a high density of dislocation sources which induced the considerable amount of dislocation generation in the form of slip bands. From Test No. 4.

Fig. 3

Dislocation displacement plotted as a function of the time integral of resolved shear stress.

Fig. 4

Dislocation damping coefficient plotted as a function of temperature for edge dislocations in copper. (B at 4.2°K from present investigation. Other values after Jassby and Vreeland.^{14, 15})