Dislocation Mobility in Germanium*

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The stress and temperature dependence of the glide velocity of individual dislocations in pure germanium has been measured by means of an etch-pit technique. The motion may be described by a thermal activation energy which, for 60° dislocations, varies from 1.49 eV at 8.0 kg/mm^2 to 2.25 eV at 0.8 kg/mm^2. For screws, it is 1.47 eV independent of stress. At constant temperature, the velocity varies with stress in a complex manner which is not adequately accounted for by previous theories alone.

I. INTRODUCTION

UNTIL recently, concepts of the atomic processes involved in dislocation glide found their experimental basis largely in the bulk mechanical behavior of crystals. These macroscopic experiments have, on the whole, proved incapable of answering detailed questions of dislocation dynamics involving, for example, the energy dissipation mechanisms of a moving dislocation.

Several years ago, Gilman and Johnston[1] developed an etch-pit technique for measuring on a microscopic scale the average velocities of individual dislocations in LiF crystals under an applied stress. Their subsequent work has yielded detailed and valuable information concerning dislocation mobility, multiplication, origins, interaction, etc. Using these data on the properties of individual dislocations, it has proved possible to describe satisfactorily many aspects of the bulk mechanical behavior of crystals.

The present experiment was directed toward the more limited goal of measuring the stress and temperature dependence of the velocity of an individual dislocation in a pure crystal having certain properties desirable from the theoretical viewpoint. Due to its high chemical purity, its adaptability to experimental techniques and its supposed high Peierls stress, germanium was chosen for this investigation. Employing techniques related to those of Gilman and Johnston, Stein and Low have measured the velocity of advance of slip bands in 3% Si-Fe alloy single crystals.[2] The same type of information has been obtained for several semiconductors, including germanium, by Chaudhuri, Patel, and Rubin.[3] The Gilman and Johnston method was utilized for the present work, however, in order to free gliding dislocations from possible effects of internal stresses present in slip bands. With this approach it was possible to move a given dislocation several times at different stresses and temperatures and to differentiate between screw and composite 60° dislocations.

The data presented here are discussed in the following paper.[4] There a model is developed which seems reasonably well adapted to germanium.

II. EXPERIMENTAL PROCEDURES

A. General Method

A given measurement proceeded as follows: Crystals containing a number of chemically clean, isolated dislocations were etched by a chemical which leaves small, sharp pits at points where dislocations intersect the surface. A known stress was then applied for a time sufficient to move the dislocation several tens of microns. After removal of the stress, a second etch revealed the new location of the dislocations while at the same time enlarging and flattening the vacated pits. Using a microscope, the distance moved during the stress was measured; from this, an average velocity over the distance travelled was easily computed. Figure 1 shows a dislocation half-loop, the two ends of which emerge through a {111} surface. From its initial location at the two central pits, the half-loop has been moved and etched twice. In practice, it was often possible to move a given dislocation as many as eight times at different stresses and temperatures. The data appeared not to depend upon previous test conditions or upon the distance moved.

B. Apparatus

The three-point bending jig used to apply stress to the crystal is illustrated schematically in Fig. 2. Total plastic flow was always so slight that the stress on the dislocations could be taken as the stress at the surface of an elastic beam. Dead loading was employed for times ranging between 1 sec and 10 h.

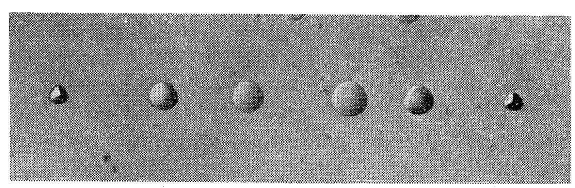

FIG. 1. A typical half-loop after two moves; ×400.

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[1] For a review, see J. J. Gilman and W. G. Johnston, in *Solid State Physics*, edited by F. Seitz and D. Turnbull (Academic Press Inc., New York, 1962), Vol. 13, p. 147.

[2] D. F. Stein and J. Low, J. Appl. Phys. **31**, 362 (1960).

[3] A. R. Chaudhuri, J. R. Patel, and L. G. Rubin, J. Appl. Phys. **33**, 2736 (1962).

[4] V. Celli, M. Kabler, T. Ninomiya, and R. Thomson, following paper, Phys. Rev. **130**, 58 (1963).

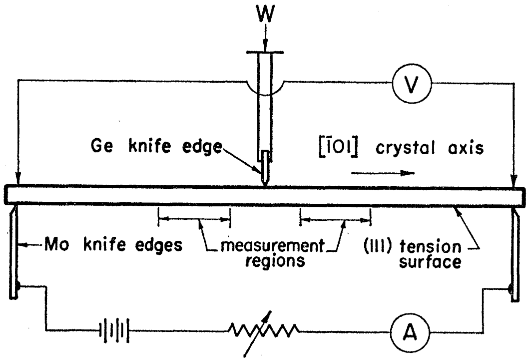

FIG. 2. Three-point bending jig for applying stress to the crystal.

Appropriate test temperatures were attained by Joule heating via a direct current through the crystal. Test temperatures always fell in the intrinsic conductivity range, where the crystal resistivity may be used as a measure of average temperature.[5] For this purpose, the voltage across the crystal was measured through molybdenum pressure contacts at the ends. By applying auxiliary voltage probes and by monitoring thermal radiation from a crystal, it was determined that, for a given test, temperatures were uniform within about 6°K over the surface areas where motion was followed. The temperature reproducibility from test to test and among different samples was also estimated at roughly 6°K.[6] It is difficult to assess the systematic error affecting the tests as a group; however, we believe that it could hardly have amounted to more than 15°K.

To avoid oxidation of the Ge, tests were performed in a vacuum jar at air pressures $<10^{-5}$ mm Hg. The apparatus as a whole was conceived to minimize chemical contamination of the hot crystals.

C. Material and Preparation

All data presented here were derived from a 40 Ω cm *n*-type germanium ingot pulled from the melt in a hydrogen atmosphere and containing fewer than 100/cm² dislocations.[7] A number of tests on commercial zone-leveled material were, on the whole, consistent with these results.

Rectangular samples approximately $0.9\times1.5\times33$ mm³ were prepared, and microscopically smooth surfaces were achieved with a standard chemical polish.[8] Crystals were oriented as in Fig. 2, the largest surfaces being (111); all measurements were made on these surfaces. To forestall chemical contamination, crystals were always rinsed in concentrated HNO_3 and aqueous KCN prior to testing, as were all parts of the apparatus which contacted them.

D. Dislocation Injection

Fresh dislocation half-loops were introduced into the crystal by scratching the surface lightly with a sharp carbide scriber and applying stress for a short time in the apparatus at around 730°K. The scratch and most of the half-loops issuing from it were then polished off, leaving a smooth surface containing a suitable number of adequately isolated dislocations. The diameters of the half-loops were always small compared to the crystal dimensions.

III. BURGERS VECTORS

In germanium and silicon, as in the face-centered cubic metals, slip occurs in ⟨110⟩ directions on {111} planes. Glissile dislocations which have not been severely annealed show a strong tendency to lie along ⟨110⟩ lattice directions.[9] In the present work, this situation was illustrated in two ways. First, half-loop shapes were mapped by repeatedly polishing away thin layers from the crystal surface and following the dislocation lines via etch pits. This procedure was carried out for a number of half-loops at various stages of expansion. Second, it was found that, if a crystal was sliced so as to expose a surface coincident with an active slip plane, the present etch would reveal dislocations as shallow etch grooves along the surface. Dash[10] has, with identical results, employed this technique to observe dislocations in silicon. Using these two approaches, we have concluded that the overwhelming majority of the dislocations used for our velocity measurements assumed the half-hexagon shapes illustrated schematically in Fig. 3. That is, screw and 60° types predominated.

For small dislocation half-loops emerging through the (111) surfaces, the applied stress distribution may be regarded as uniaxial along the $[\bar{1}01]$ crystal axis. Thus, the $(11\bar{1})$ and $(\bar{1}11)$ slip planes will experience a nonzero shear stress. These planes are equivalent, and to be specific we discuss the $(11\bar{1})$ slip plane. Here, the

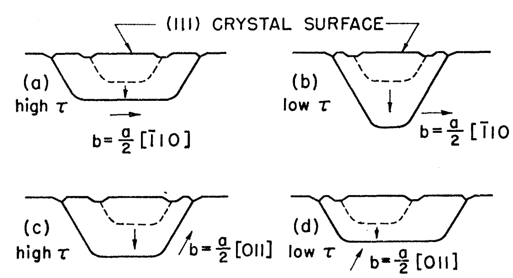

FIG. 3. Typical shapes and movements of half-loops having the two possible Burgers vectors, at high and low applied stress.

[5] F. J. Morin and J. P. Maita, Phys. Rev. **94**, 1525 (1954).
[6] Piezoresistance effects were found to be negligible for the experimental conditions employed.
[7] This crystal was kindly donated by R. Buschert of Purdue University.
[8] Chemical polish: Equal parts by volume conc. HF, conc. HNO_3 and H_2O, used boiling for several sec. Etch: By weight, 3 parts KOH, 2 parts $K_3Fe(CN)_6$ in 25 parts H_2O, used boiling for 15 sec.
[9] G. L. Pearson and F. L. Vogel, in *Progress in Semiconductors*, edited by A. F. Gibson (John Wiley & Sons, Inc., New York, 1962), Vol. 6.
[10] W. C. Dash, J. Appl. Phys. **29**, 705 (1958).

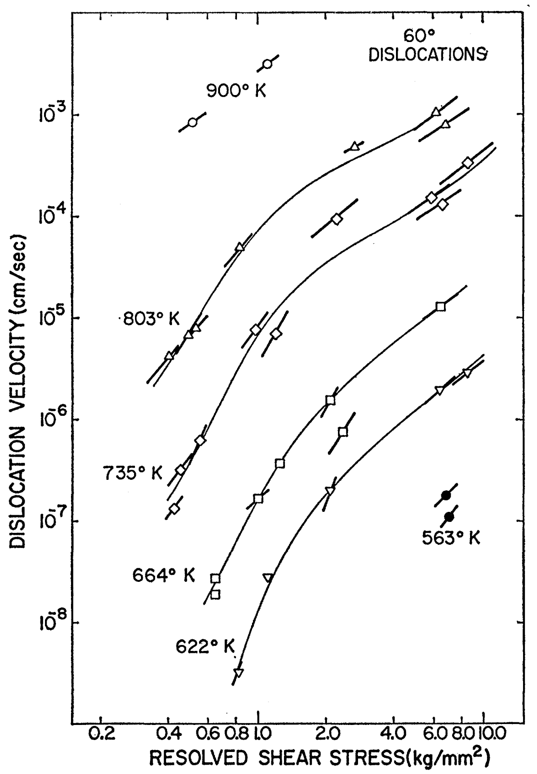

FIG. 4. Velocity vs applied stress for 60° dislocations in Ge. The line segments through the points represent the extent of the data gained from a number of dislocations in a particular test. Their slopes reflect the shape of the over-all curve.

Burgers vectors $\frac{1}{2}a[\bar{1}10]$ and $\frac{1}{2}a[011]$ will undergo a nonzero resolved shear stress. There will be no stress parallel to $\frac{1}{2}a[101]$, which will thus be inoperative.

The plane of Fig. 3 is the $(11\bar{1})$ slip plane. The dashed lines are the initial and the solid lines the final positions of half-loops having the two possible Burgers vectors and gliding in typical tests at high (≈ 10 kg/mm²) and low (≈ 0.5 kg/mm²) resolved shear stresses τ. If $b=\pm\frac{1}{2}a[\bar{1}10]$ for a half-loop whose motion is being followed, then the emerging segments will both be of 60° character. Thus, under all imposed conditions of temperature and stress, both ends of the half-loop will move at the 60° velocity. This case is illustrated in Fig. 3(a) and (b). On the other hand, if $b=\pm\frac{1}{2}a[011]$ as in Fig. 3(c) and (d), then one end of the half-hexagon will be 60° and the other screw. These will, in general, glide at different velocities. Therefore, for a crystal containing both types of half-loops, a single test is generally adequate to establish the Burgers vector of each. Half-loops which move symmetrically about their center will have $b=\pm\frac{1}{2}a[\bar{1}10]$; those which move nonsymmetrically will have $b=\pm\frac{1}{2}a[011]$.

Though the behavior just described was dominant in the present experiments, the situation illustrated in Fig. 3(d) sometimes exhibited an anomaly. That is, the left-hand (60°) end of the half-loop was observed to turn back into a screw orientation and cross-glide on the unstressed $(1\bar{1}1)$ plane.[11] This case is of interest here as a further confirmation of Burgers vector assignments; its detailed discussion will be given elsewhere.

A further incidental substantiation of Burgers vector determinations was accomplished by expanding a large number of half-loops from a source characterized by Fig. 3(c) and (d). As expected, a slip step was observed at the crystal surface.

For a given type of dislocation, one would expect to observe the same velocity on the compression and tension surfaces in a bent sample. Such was the case at all stresses for half-loops with b parallel to the surfaces and at high stresses when b intersected the surface, as in Fig. 3(a) and (c). However, when b intersected the surface and when the applied stress was low, the velocity became several times higher on the tension surface than on the compression surface. We believe that this effect was caused by thermal radiation from the crystal surface giving rise to temperature and stress gradients perpendicular to the surface. These stress gradients influence only those dislocations whose Burgers vectors have a component perpendicular to the surface. At a given temperature the thermal stress is a constant which adds to the applied stress on the tension surface and subtracts from it on the compression surface. A rough calculation of the temperature gradient necessary to account for the observed thermal stress yields results which are not unreasonable.

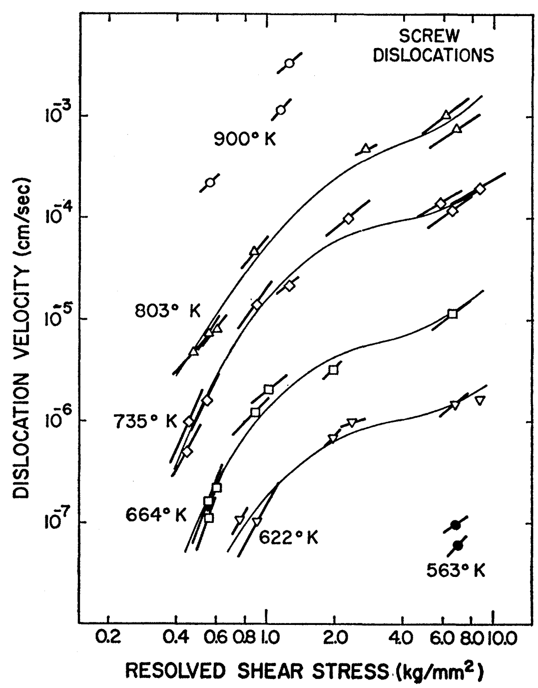

FIG. 5. Velocity vs applied stress for screw dislocations in Ge.

[11] M. N. Kabler, Bull. Am. Phys. Soc. 7, 65 (1962).

A large majority of the present data was taken from the tension surface. However, enough compression surface data was taken to make possible an average over those dislocations having Burgers vectors intersecting the surface. The velocity vs applied stress data was plotted and the magnitude of the thermal stress necessary to bring the curves for the compression and tension surfaces into coincidence was determined at each temperature. These thermal stresses were then used to correct all the screw data. A typical correction amounted to approximately 0.05 kg/mm^2 at 664°K and was relatively insensitive to crystal temperature.

IV. RESULTS

The data obtained for 60° and screw dislocations are displayed in Figs. 4 and 5, respectively. The logarithm of velocity is plotted against the logarithm of the applied resolved shear stress at various test temperatures. The previously mentioned slight stress correction has been applied to the screw data. Each point represents a particular test after which the velocities of at least 40 and usually over 100 dislocations were measured and averaged. The short line segment through most of the points shows the variation of velocity with stress along the crystal due to the three-point bending arrangement. These line segments are important reflections of the slopes of the over-all velocity vs stress curves, particularly at the higher stress levels. The curves are therefore drawn to fit both the slopes and magnitudes of the data points.

Concerning the relative velocities of 60° and screw dislocations, the following features may be noted: In the vicinity of 6 kg/mm^2, screw and 60° dislocations move at the same velocity, independent of temperature. At higher stresses (up to the fracture stress, approximately 12 kg/mm^2) there are strong indications that the 60° dislocations move faster. At a temperature in the vicinity of 770°K the velocities become nearly equal at all stresses. As stress and temperature are decreased below about 5 kg/mm^2 and 770°K, the 60° velocity decreases faster than the screw velocity. However, at low stresses and at temperatures above 770°K, screw dislocations move slower than 60°. This latter observation is consistent with work by Tyler and Dash,[12] who found that decorated hexagonal dislocation loops in germanium deformed by twisting at 823°K were asymmetrical in such a way as to indicate a screw velocity roughly seven tenths that of a 60° dislocation.

Considering the differences in experimental methods, the present results agree satisfactorily with those of Chaudhuri *et al.*[3] within their range of overlap. Those investigators worked at temperatures above 710°K, which are out of the region where the difference between screw and 60° velocities is greatest. Thus, the fact that they did not differentiate between 60° and screw dislocations does not seriously hamper comparison with the present data. Their velocities tend to be somewhat higher than ours. This is likely due to additional stresses exerted by dislocations in an active slip band upon the leading dislocation of that band. In the present work, groups of dislocations gliding together as a slip band have been observed to move up to several times as fast as isolated dislocations of like character. This enhancement diminished at high-applied stress.

V. DISCUSSION

Some general remarks may be made concerning the applicability to the present data of several empirical and theoretical formulas which have been used previously.

On the basis of a number of physical models, a stress dependence of the form $v \propto e^{A\tau}$ or $v \propto \sinh A\tau$ has been derived, where v is the dislocation velocity, τ is the applied stress, and A is a constant which may depend weakly on temperature.[13] The fact that this stress dependence may arise as a first approximation to simple kink models for a dislocation moving in a lattice where the Peierls force[14] is dominant makes it very attractive for germanium. However, it appears to be inapplicable by itself, since it demands that curves of Fig. 4 and Fig. 5 be concave upward throughout their range. Nevertheless, the data are consistent with this model in the high stress range.

The stress dependence of dislocation velocity in LiF and 3% Si-Fe alloy has been found to be describable by $v \propto e^{-\tau_0/\tau}$, where τ_0 is a constant which depends upon temperature.[1,2] This type of stress dependence is also an adequate representation of the present data except at high stress. However, there are conceptual difficulties with the available theoretical treatment.[15] In the following paper,[4] an alternative theoretical basis for this relationship will be constructed, based on a model combining a large Peierls stress and discrete dislocation dragging points. This model also accounts for the high-stress behavior and for the observed temperature dependence.

Mention should be made of an empirical relationship, $v \propto \tau^n$ with n possibly depending on temperature, which may be fitted to data on all materials referred to so far, at least over restricted ranges of stress. No theoretical account has been given for this form, and it appears inadequate to describe the present experiments.

Regarding the temperature dependence, it is possible to characterize dislocation glide as a simple thermally activated process with a temperature-independent activation energy. This energy is 1.47 eV for screws, independent of stress. For 60° dislocations, it varies from 1.49 eV at 8.0 kg/mm^2 to 2.25 eV at 0.8 kg/mm^2. Under the analysis presented in the following paper,

[12] W. W. Tyler and W. C. Dash, J. Appl. Phys. **28**, 1221 (1957).

[13] H. G. van Bueren, *Imperfections in Crystals* (North-Holland Publishing Company, Amsterdam, 1960).

[14] J. Weertman, J. Appl. Phys. **28**, 1185 (1957); J. Lothe and J. P. Hirth, Phys. Rev. **115**, 543 (1959).

[15] J. J. Gilman, Australian J. Phys. **13**, 327 (1960).

these activation energies take on a somewhat more involved interpretation.

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Theory of Dislocation Mobility in Semiconductors*

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A new model for dislocation motion appropriate for crystals having high Peierls stress is presented. The model makes use of dragging points on the dislocation which restrict the free motion of kinks on the line. It predicts a dislocation velocity with an exponential dependence inverse in the stress and an activation energy appropriate to kink nucleation. In extensive Appendixes, kink theory is used to develop explicit formulas for the stress dependence of the kink nucleation energy and to work out the statistical details of the nucleation rate. Also, a detailed theory of kink collisions is developed when the kink population is high. Finally, the experiments of Kabler described in the previous paper of this journal are interpreted in terms of the model with appropriate parameters for Ge.

I. INTRODUCTION

THE mobility of individual dislocations as a function of stress and temperature has been measured by etch-pit techniques for several nonmetals.[1-3] In all cases the mobility increases rapidly with temperature, consistent with the idea that the dislocation motion is thermally activated. If the resistance to motion were simply due to the interaction of the dislocation strain field with thermal vibrations, the mobility should decrease with temperature.[4] Thus, the dislocation does not behave as if it were moving in a homogeneous elastic continuum, and other possible obstacles to motion must be considered.[5] These can be due to the discrete nature of the crystal medium (Peierls barrier), or to the interaction of the dislocations with other defects or impurities present in the material. Furthermore, it is well known that jogs on screw dislocations cannot glide conservatively, at least when they are several Burgers vectors high. Thus, the drag on the dislocation due to debris formation must also be considered.

The various mechanisms mentioned above are not mutually exclusive, and it is often difficult to decide which one is the most important in a given material.

In the case of LiF it has been shown that the divalent impurities present in the crystal can provide a drag of the observed order of magnitude,[6,7] and that vacancy trails due to nonconservative jogs can explain the difference between the mobility of screw and edge dislocations. There is no evidence of an important Peierls energy in this case.

On the other side in the semiconductors, germanium and silicon, evidence of formation of vacancy trails has been found both for screw and 60° dislocations,[8] but a simple calculation shows that at stress levels where the dislocation velocity is only about 10^{-3} cm/sec, the work done by the applied stress is large enough to create one point defect of energy 1 eV every ten atoms along the dislocation per every Burgers vector traversed by the dislocation line.[9] Such a high defect formation rate is unreasonable without direct supporting evidence. A similar argument can be applied to the drag exerted by impurities present in the lattice.

It has been commonly supposed that the Peierls energy in the diamond lattice is high, and glide dislocations have been observed in these crystals to lie straight along crystallographic directions. This result can hardly be explained without the existence of a

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1 W. G. Johnston and J. J. Gilman, J. Appl. Phys. **30**, 129 (1959).
2 A. R. Chaudhuri, J. R. Patel, and L. G. Rubin, J. Appl. Phys. **33**, 2736 (1962).
3 M. N. Kabler, preceding paper, Phys. Rev. **130**, 54 (1963); hereafter referred to as I.
4 G. Leibfried, Z. Physik **127**, 344 (1950); see also, J. Lothe, Phys. Rev. **117**, 704 (1960).
5 A. H. Cottrell, *Dislocations and Plastic Flow in Crystals* (Clarendon Press, Oxford, 1953).
6 W. G. Johnston, J. Appl. Phys. **33**, 2050 (1962).
7 R. L. Fleischer, J. Appl. Phys. **33**, 3504 (1962).
8 W. C. Dash, J. Appl. Phys. **29**, 705 (1958).
9 If, typically, the Burgers vector is $b=4$ Å and the resolved shear stress is $\tau=25$ kg/mm², then $\tau b^3=0.1$ eV.

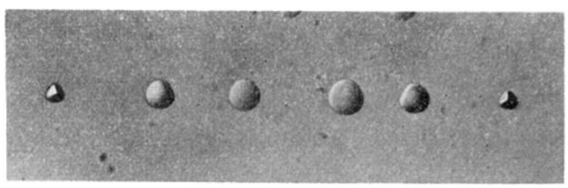

FIG. 1. A typical half-loop after two moves; $\times 400$.