

# Dislocations and Plastic Flow in Germanium

**Abstract:** Single-crystal specimens of germanium have been tested in tension over a range of temperature and strain rate. Dislocation density has been determined as a function of plastic strain, and some direct measurements of dislocation velocity have been made. From these results the plastic behavior of germanium can be explained in terms of dislocation velocity and dislocation multiplication, and is analogous to the interpretation given by Johnson and Gilman for deformation of lithium fluoride. Effects of crystal purity and orientation have been examined; strain-aging effects were absent. Conjectures are made as to the causes of work hardening.

## Introduction

The study of plastic deformation and dislocation behavior in a semiconductor such as germanium is of interest for several reasons. Much of the theory of dislocations has been formulated from studies of deformation in metals but, since the crystal structure and atomic bondings of semiconductors are different from those of metals, the deformation characteristics are also different, as would be expected. An analysis of the plastic behavior of germanium will therefore permit fundamental dislocation theory to be extended.

Deformation studies may also be of value from the standpoint of solid-state device fabrication. Dislocations not only have a direct effect on the electrical properties of semiconductor devices but, by their interaction with impurities and other crystalline defects, may produce a secondary effect on the electrical properties. The limits of stress and temperature at which dislocations move and multiply may serve as indications of the limits of stress and temperature which are allowable during fabrication.

Previous work<sup>1-5</sup> has shown that germanium is plastic at temperatures above about 400°C; it exhibits during creep testing an initial region of slow plastic flow which is usually interpreted as evidence for a delay time; and it gives an exaggerated yield point in a tensile test. Attempts have been made<sup>6-8</sup> to associate both of these effects with Cottrell-type impurity locking of dislocations. However, the strong similarity between the stress-strain curves obtained during tensile tests on germanium and those for lithium fluoride suggests that the same explanation so successfully given by Johnston and Gilman<sup>9</sup> for lithium fluoride may be valid for germanium. Their theory is based on two experimental facts. First, in any given crystal, which has undergone no previous deformation, at a

constant temperature the dislocations move with a velocity which is dependent only upon the stress experienced by the dislocation. This follows if it is assumed that a moving dislocation is subjected to a frictional force which increases with increasing velocity. Secondly, it is assumed that as a dislocation moves it creates new dislocations in its wake.

## Theory

The increment in plastic strain  $d\epsilon$  contributed by a dislocation of Burgers vector  $b$  sweeping out an element of area  $da$  on the slip plane of a crystal is given by the product  $bda$  divided by the total projected area of the slip plane times the length of the crystal. For a crystal in which there are  $N$  dislocations moving, the total increment in strain

$$\Delta\epsilon = \frac{1}{V} \sum_1^N b_i da_i, \quad (1)$$

where the projected area of the slip plane times the crystal length is equal to  $V$ , the crystal volume, and  $b_i$  and  $da_i$  refer to the  $i^{\text{th}}$  dislocation. If the  $i^{\text{th}}$  dislocation has a length  $L_i$  and moves through a distance  $dx_i$  then

$$\Delta\epsilon = \frac{1}{V} \sum_1^N b_i L_i dx_i. \quad (2)$$

The strain rate  $\dot{\epsilon}$  is obtained by differentiation with respect to time

$$\dot{\epsilon} = \frac{1}{V} \sum_1^N b_i L_i v_i, \quad (3)$$

where  $v_i$  is now the instantaneous velocity of the  $i^{\text{th}}$  dislocation. If the subscript  $i$  is now transferred from the  $i^{\text{th}}$

\*Now at University of Cambridge, Cambridge, England.

dislocation to the  $i^{th}$  type of dislocation, i.e., all dislocations of Burgers vector  $b_i$ , and velocity  $v_i$ , with a length per unit volume of  $\lambda_i$  the strain rate then becomes

$$\dot{\epsilon} = \sum_1^N b_i \lambda_i v_i . \quad (4)$$

In most materials the dislocations responsible for plastic deformation can be resolved into two types, edge and screw, with identical Burgers vectors. Assuming that we can assign one velocity  $v_e$  to all edge dislocations and one velocity  $v_s$  to all screw dislocations, and that the length per unit volume of these dislocations is  $\lambda_e$  and  $\lambda_s$ , respectively, then

$$\dot{\epsilon} = b(\lambda_e v_e + \lambda_s v_s) . \quad (5)$$

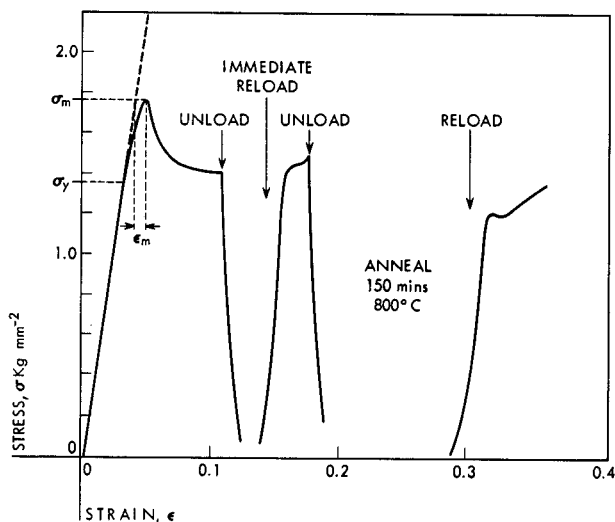
A dislocation loop lying in the slip plane will vary in character from edge to screw around its circumference. As it expands under the action of the applied stress it will be circular in shape only if the velocities of the edge and screw segments are equal. If they are not equal, the loop will assume an elliptical shape. If one type has a velocity very much higher than the other, then the segments of this type will reach the edge of the crystal before the segments of the other type have separated by very much. This leaves two straight dislocations of the slower type lying across the slip plane. The larger term thus eliminates itself from Eq. (5) and the strain rate is controlled by the smaller term and is given by:

$$\dot{\epsilon} = nbv , \quad (6)$$

where  $n$  is now defined as the *active dislocation density* in terms of the number of active dislocation lines crossing unit area, and  $v$  is the *velocity of dislocations of the slowest type*. This expression also holds if the velocities of the two types of dislocation are equal.

Figure 1a shows a typical tensile curve for germanium. Johnston and Gilman explained similar tensile curves

Figure 1a Tensile curves for Ge crystal at 650°C.



found for lithium fluoride in the following way. As the velocity of screw dislocations was found to be some 50 times slower than that of edge dislocations, they were able to describe the strain rate in terms of Eq. (6). In a tensile test using a hard machine, the crosshead is driven at a constant velocity  $S_c$ . After a time  $t$  the crosshead has moved a distance

$$S_c t = \Delta y = \Delta y_{el} + \Delta L_p , \quad (7)$$

where  $\Delta L_p$  is the plastic elongation of the specimen, and  $\Delta y_{el}$  is the elastic elongation of specimen plus machine.  $\Delta y_{el}$  is equal to  $K\sigma$ , where  $K$  is the elastic modulus of specimen plus machine, and  $\sigma$  is the stress now imposed on the specimen. The plastic strain of the specimen is

$$\epsilon = \Delta L_p / L_0 = (S_c t - K\sigma) / L_0 , \quad (8)$$

where  $L_0$  is the initial gauge length of the specimen. Differentiating with respect to time,

$$\dot{\epsilon} = (S_c - K\dot{\sigma}) / L_0 . \quad (9)$$

At the maximum in the stress-strain curve,  $\dot{\sigma} = 0$  and  $\dot{\epsilon}_m \equiv S_c / L_0$ . From Eq. (6),  $\dot{\epsilon} = nbv$  and  $v$  is some function of  $\sigma$ , say  $v = f(\sigma)$ , then:

$$nbf(\sigma) = (S_c - K\dot{\sigma}) / L_0 . \quad (10)$$

The shape of the stress-strain curve may now be explained. As the specimen is loaded, it deforms elastically until some stress  $\sigma_y$  is reached at which a significant, but initially assumed to be small, number of dislocations begin to move and to contribute to plastic strain. Continuing deformation causes both  $n$ , through dislocation multiplication, and  $\sigma$  to increase, and hence the contribution of plastic strain rate to the over-all strain rate increases. At some point,  $n$  and  $\sigma$  reach values such that the plastic strain rate is exactly equal to the rate of crosshead motion. The plastic strain rate now stays essentially constant, (ignoring the small elastic relaxation as  $\sigma$  decreases), but dislocations are still moving;  $n$  continues to increase, and the dislocation velocity, and hence  $\sigma$  decrease. This explains the yield drop; the subsequent increase in  $\sigma$  is due to strain-hardening and will not be discussed here.

The shape of the creep curves, shown in Fig. 1b, is similarly explained. This type of curve is obtained under constant stress and thus the dislocation velocity is constant. The strain rate, from Eq. (6), now depends only upon  $n$ . The initial number of active dislocations is small and increases as deformation progresses. The strain rate is therefore initially low but increases with increasing deformation until strain-hardening causes it to decrease.

A theoretical expression for the initial portion of the curve is obtained by assuming<sup>9</sup> that as a dislocation moves it leaves behind  $\delta$  new dislocations per centimeter of wake of each of the two ends of the expanding loop. At any instant the rate of production of new dislocations is:

$$dn = 2\delta n dx = 2\delta n v dt . \quad (11)$$

Integrating,

$$n = n_0 \exp(2\delta v t) , \quad (12)$$

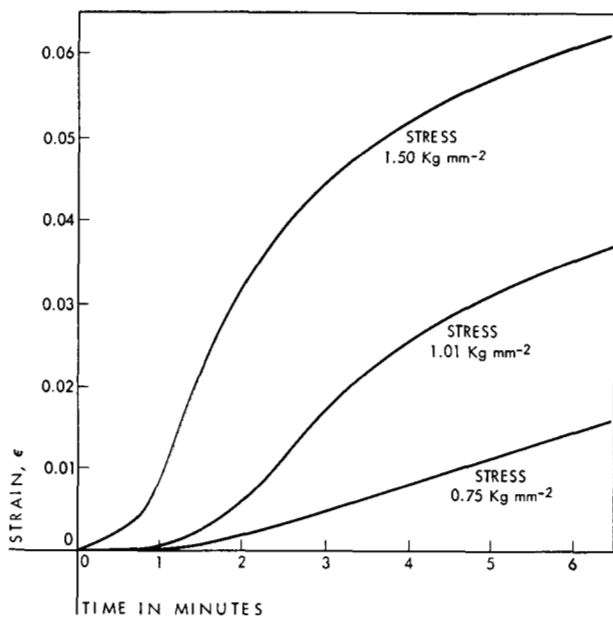


Figure 1b Creep curves for Ge crystals at 650°C.

where  $n_0$  is the initial density of active dislocations. Substituting in Eq. (6),

$$\dot{\epsilon} = bnv = bnv_0 \exp(2\delta vt). \quad (13)$$

Integrating, and applying the boundary condition that at  $t=0$ ,  $\epsilon=0$ ,

$$\epsilon = [bn_0 \exp(2\delta vt) - bn_0] / 2\delta \quad (14)$$

or

$$\epsilon = b(n - n_0) / 2\delta. \quad (15)$$

Equation (14) predicts that the initial portion of a constant-stress creep curve should show an exponential dependence of strain with respect to time. Penning and de Wind<sup>5</sup> state that their creep curves on germanium can be fitted to such an expression, but prefer to fit them to another expression based on a theory proposed by van Bueren.<sup>10</sup> However, their actual curves are not published, and therefore cannot be analyzed in terms of the above theory. Equation (15) predicts a linear dependence of dislocation density on plastic strain provided that all the dislocations stay in the material, and Johnston and Gilman<sup>9</sup> have experimentally verified this relation for lithium fluoride. The agreement between their experimental and theoretical constants is very good.

If the observed plastic behavior of germanium is to be explained by the above, three basic assumptions must be valid.

(i) As dislocations move they create new dislocations by some mechanism. The number of dislocations increases during deformation, and there is a relation between dislocation density and plastic strain.

(ii) For similar crystals, at the same temperature, dislocations move with a velocity uniquely related to the

applied stress.

(iii) The number of dislocations which are initially mobile is small. It should be noted here that the grown-in dislocations appear to play no part in the plastic deformation process in both lithium fluoride<sup>9</sup> and germanium.<sup>5</sup> Apparently they are rendered immobile by impurities.

The work described in this paper was an attempt to verify that the above assumptions are applicable to the plastic deformation of germanium.

### Experimental methods

Single crystals of germanium were grown either in a zone leveller or in a Czochralski puller from zone-refined material. In the zone leveller, the freshly grown crystal passed immediately into an annealing chamber which allowed very slow cooling of the crystal from a little below the solidification temperature. The levelled crystals contained as the major electrically active impurity approximately  $10^{13}$  atoms/cc of a deep-level acceptor, presumably copper, and a dislocation density of  $10^4$  lines/sq cc. It is believed that because of the higher dislocation density and because of the annealing treatment, the levelled crystals contain fewer vacancies than the pulled crystals. These latter had dislocation densities varying from zero to  $10^3$  lines/sq cc and contained about  $10^{13}$  atoms/cc of a donor impurity, probably arsenic. Crystals were grown with both  $\langle 321 \rangle$  and  $\langle 110 \rangle$  growth axes. One crystal containing  $5 \times 10^{19}$  atoms/cc of gallium was grown with a  $\langle 321 \rangle$  axis.

Tensile specimens with rectangular cross-section were cut from these crystals, parallel to the growth axis, with one pair of faces parallel to a  $\{111\}$  plane, by an ultrasonic impact grinding machine. The gauge length was one inch, in some cases 1.25 inches, and the cross-section  $2 \times 3$  mm. The shoulders of the specimens were rounded, and were individually lapped into the grips of the testing machine. Specimens were etched in CP4 prior to testing, and in some cases the  $\{111\}$  faces were lapped using a coarse grit after the etch. Specimens were tested over a temperature range from 575°C to 850°C in a forming gas atmosphere in a specially designed high-temperature cell mounted on an Instron tensile testing machine. A simple lever-loading creep tester was also used. On both machines the strain could be read to  $10^{-4}$ . Temperature control was  $\pm 2^\circ\text{C}$ .

Dislocation densities were determined by counting etch pits on  $\{111\}$  faces after etching in CP4 and dislocation velocities were calculated from tensile data by using formula (6). This calculation assumes that the strain rate is controlled by only one dislocation velocity. At the maximum in the stress-strain curve  $\dot{\epsilon}_m = S_c/L_0$ . The dislocation density at this point is taken to be the value corresponding to the plastic strain as given by the graph (Fig. 2) of dislocation density versus plastic strain. The value of plastic strain at the maximum is obtained directly from the stress-strain curve. The plot of  $\epsilon$  versus  $n$  was made by straining a specimen in tension by a known amount in the creep tester under a 1 kg load at 650°C

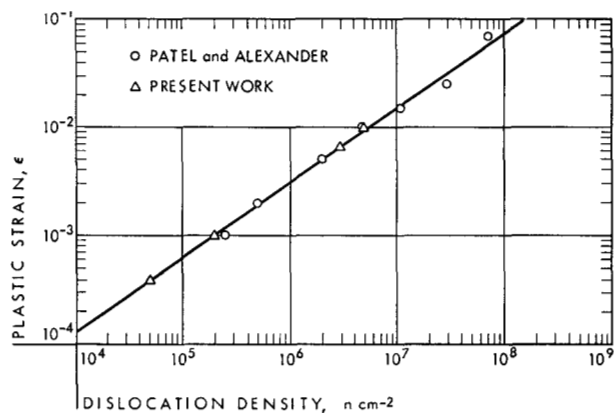


Figure 2 Dislocation density as function of plastic strain in germanium.

and then measuring the dislocation density.

Direct estimates of dislocation velocity were made in the following manner. An etched specimen was scratched with a razor blade to provide a source of dislocations. The crystal was placed under load for a known length of time, usually five or ten seconds, in the creep tester. An etch in CP4 showed that the dislocations had moved out from the scratch along slip planes. The average distance moved by the leading dislocations was measured using a microscope fitted with a micrometer stage. From this distance and the time of application of the load, the dislocation velocity was calculated. These estimates can be regarded as accurate only to within a factor of two and will tend to give values which are lower than the peak velocities attained.

Creep testing was also carried out, but the results showed such lack of reproducibility that only one or two generalizations could be drawn from them. The lack of reproducibility and the large amount of scatter in the tensile data, are both believed to result from the difficulty of exactly determining the onset of plastic deformation due to the large amount of elastic deformation in the testing machines. This problem can be overcome only by measuring strain directly from the specimen itself, a procedure which is rather difficult when the specimen must be maintained at a high temperature.

## Results and discussion

### Dislocation multiplication

Crystals with a  $\langle 110 \rangle$  tensile axis were found to deform simultaneously on two slip systems, and the majority of tests were performed on crystals with a  $\langle 321 \rangle$  orientation, in which only one slip system is operative. The first series of experiments was carried out to establish the relation between strain and dislocation density. Data were obtained on two levelled crystals and on two pulled crystals, all with a  $\langle 321 \rangle$  orientation. The results coincided with the curve given by Patel and Alexander<sup>3</sup> who measured dislocation density as a function of compressive strain at

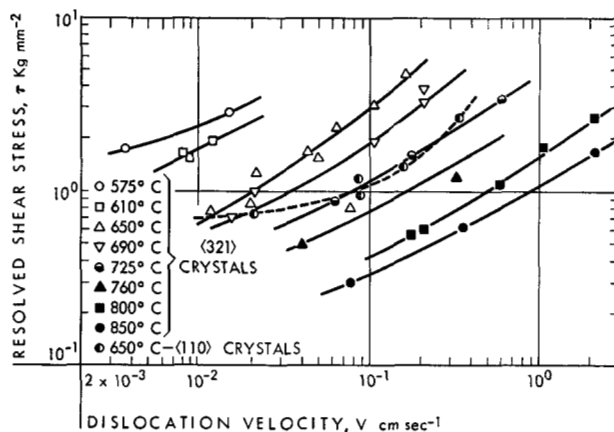


Figure 3 Dislocation velocity as function of stress for levelled germanium crystals.

600°C under an unspecified load on crystals with an orientation near to  $\langle 321 \rangle$ . The combined results are plotted in Fig. 2. The agreement between the two sets of data indicate that for a given strain, the dislocation density in similarly oriented crystals is not much affected by temperature or method of loading. Figure 2 gives an approximate relation:  $n = 5 \times 10^9 \epsilon^{3/2}$  which disagrees with the theoretical prediction of Eq. (15). Dislocation density increases more rapidly with strain in germanium than in lithium fluoride and hence the dislocation multiplication mechanism must be different in the two materials. Further evidence for this is provided by the initial portion of the creep curves, which could not be fitted to an exponential time dependence.

A model to account for dislocation multiplication in germanium must not only give the correct relation of  $n$  to  $\epsilon$ , but must also explain the observed distribution of dislocations. In deformed lithium fluoride the dislocations are congregated in slip bands, and an over-all increase in dislocation density is accomplished not only by the production of new bands, but also by the widening of bands already present. The widening of the slip bands follows directly from the model of dislocation multiplication.<sup>11</sup> Slip bands are not generally observed in germanium. The dislocations lie in individual slip lines, and though the distance between adjacent slip lines is not constant, the dislocation density over any area which includes several slip lines does tend to be quite constant.\* Thus the distribution of dislocations is much more uniform throughout a deformed germanium crystal than throughout a deformed lithium fluoride crystal. A model of dislocation multiplication for germanium which will explain the above facts has not yet been developed, but it must rely on a more complicated distribution of dislocation sources, or of source activation stresses, than envisaged by Johnston and Gilman for lithium fluoride. It is not known if cross-slip is possible in germanium but it was not observed in this investigation. This might account for the difference between germanium and lithium fluoride.

\*The tensile specimen on back cover was kindly furnished by G. E. Brock.

## Dislocation velocities

**Zone-levelled crystals.** A series of tensile tests was run on levelled crystals over a range of strain rates and temperatures. With the exception of a group of  $\langle 110 \rangle$  crystals tested at  $650^\circ\text{C}$ , all of the crystals had a  $\langle 321 \rangle$  orientation. The results are shown in terms of calculated dislocation velocity  $v$  as a function of resolved shear stress  $\tau$  in Fig. 3. Though there is some scatter, the points define fairly well a series of curves for the different temperatures, with higher velocities for the higher temperatures. Using the slowest crosshead speed available on the tensile machine, it was not found possible to test at a temperature lower than  $575^\circ\text{C}$ .

**Pulled crystals.** The above series of tests was repeated on  $\langle 321 \rangle$  pulled crystals and the results shown in Fig. 4. These crystals exhibited somewhat less plasticity than the levelled crystals and could not be tested below  $600^\circ\text{C}$ . For a given stress and temperature, dislocation velocity was generally lower in the pulled than in the levelled crystals. Direct measurements of dislocation velocities for two stresses at  $650^\circ\text{C}$  are in excellent agreement with values calculated from the stress-strain curves. This not only confirms the method of calculation but also indicates that at the maximum in the stress-strain curve the number of mobile dislocations in the crystal is equal, to within a factor of two, to the total number of dislocations present.

Figures 3 and 4 show that, within the limits of experimental accuracy, dislocation velocity (calculated from tensile data) for constant temperature, purity and orientation, is a single-valued function of the applied stress. If

the (generally small) curvature of the plots is neglected, the velocity may be represented as an exponential function of the stress, the value of the exponent lying between 1.3 and 2, with the larger values occurring at higher temperatures. This is in marked contrast to exponent values of 15 to 25 for LiF (Ref. 9) and  $\sim 40$  for silicon-iron (Ref. 20). An interesting difference appears between the levelled and the pulled crystals: the velocities are generally lower in the latter. The pulled crystals are believed to contain many more vacancies than the levelled crystals, and these extra vacancies can account for the difference in behavior. Vacancies may interact with dislocations in three ways. One is an elastic interaction as the dislocation moves through the nonuniform strain field introduced into the lattice by the presence of vacancies. Another may be called a chemical interaction and is caused by the creation of tracks of vacancies in the wake of a moving dislocation. The energy required to produce such a vacancy trail will increase as the number of vacancies originally present in the crystal increases. The third is also an elastic interaction with the strain field of collapsed sessile dislocation loops formed from the condensation of these excess vacancies. All of these interactions will cause an increase in the effective frictional force on a moving dislocation.

**Temperature dependence.** Values of velocity versus temperature were taken from the curves in Figs. 3 and 4 at three stress levels, and are plotted as in  $v$  against  $T^{-1}$  in Fig. 5. The plots for the levelled crystals (solid lines) yield reasonably straight lines which suggest that over the range of temperature and stress studied, dislocation velocity in

Figure 4 Dislocation velocity as function of stress for pulled germanium crystals.

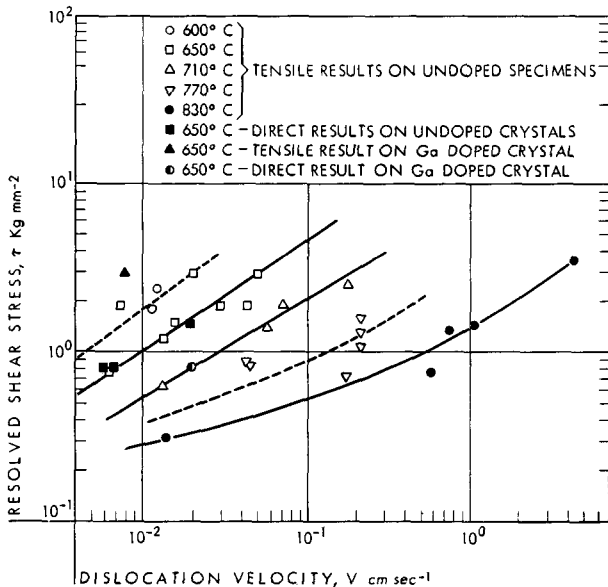
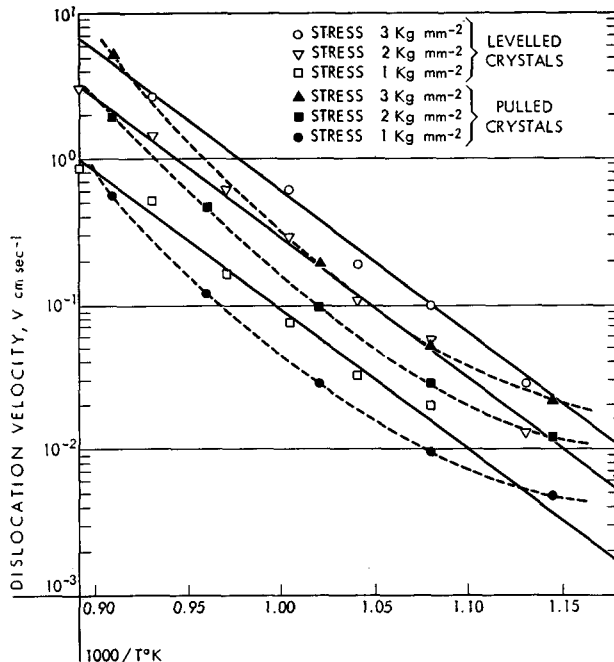


Figure 5 Temperature dependence of dislocation velocity.



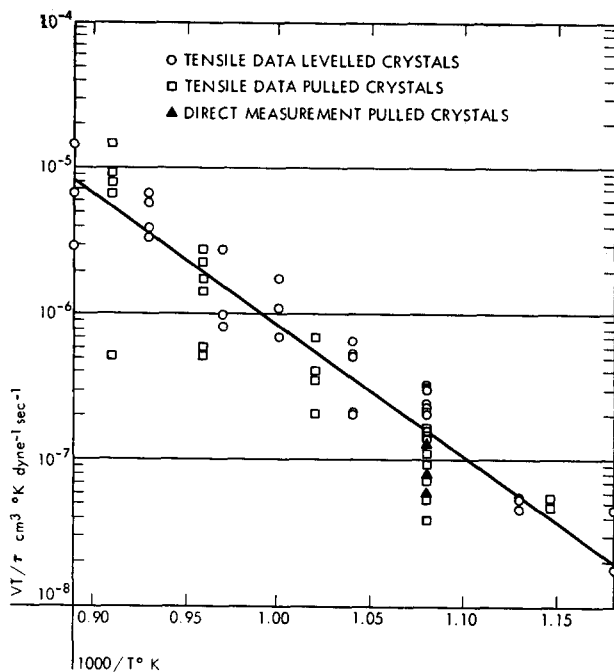


Figure 6 Diffusional motion of crack vs temperature for levelled and pulled crystals.

these crystals obeys an Arrhenius relation. Not much weight can be attached to this, as the temperature and stress range is relatively narrow, but the activation energy calculated from the plot, 1.8 ev, is very close to what is believed to be the activation energy for formation of Schottky defects in germanium.<sup>12-14</sup> This is not unexpected, as moving dislocations may leave in their wakes trails of vacancies. Plots for the pulled crystals (dashed lines) show considerable curvature, and the temperature dependence of velocity cannot be represented by an Arrhenius relation. No reasonable explanation can be offered for this at present.

An explanation for the temperature dependence of dislocation velocity in materials with the diamond cubic structure has been given by Haasen.<sup>15</sup> He suggests that there is a crack of atomic dimensions in the core of a dislocation in the diamond structure. In order to move the dislocation, the crack must diffuse with it, assisted by the applied stress. The dislocation velocity is then equal to the diffusion velocity of the crack

$$v = \frac{D}{kT} \cdot F = \frac{D_0 \tau b^2}{kT} \exp\left(-\frac{U}{kT}\right), \quad (16)$$

where  $D$  is the diffusion constant of the crack,  $F$  is the force on the dislocation, and  $U$  is the activation energy for motion of the crack. The other symbols have their usual meaning. Values of  $\ln(vT/\tau)$  versus  $T^{-1}$  are plotted for both levelled and pulled  $\langle 321 \rangle$  crystals in Fig. 6. This plot reduces the curvature in the data for the pulled crystals, and brings values for both types of crystals at different stresses closer together, though the scatter is still rather large. This indicates that a diffusion equation

of the type given by Eq. (16) more closely describes dislocation velocity than does a simple Arrhenius relation. The activation energy for the motion of the crack, calculated from Fig. 6, is approximately 1.9 ev, which is somewhat larger than that for the motion of a single vacancy in germanium (1 ev<sup>16</sup>). This is expected, as the crack will be smaller in dimensions than a single vacancy. The diffusional motion of a crack cannot completely account for the dislocation velocity, as Eq. (16) predicts a linear dependence of velocity upon stress, which is not found to be the case.

*Lapped crystals.* The experimental results on crystals with lapped surfaces lend further support to the theoretical explanation of the shape of the stress-strain rate curve. From Eq. (1), for two crystals subjected to the same strain rate, if the dislocation density in one is higher, then the dislocation velocity, and hence the stress, will be lower. Also the plastic strain at which the maximum occurs will be higher. When the crystals are lapped, the number of surface dislocation sources is increased and more dislocations are active during deformation. When compared with etched crystals tested at the same temperature and strain-rate, the lapped crystals gave tensile curves with maxima at lower stresses; the plastic strains associated with these maxima were larger, and the calculated values of dislocation velocity versus stress fell on the same curves as for the etched crystals.

*Orientation dependence.* The calculated values of dislocation velocity for the  $\langle 110 \rangle$  crystals tested at 650°C are higher than corresponding values for  $\langle 321 \rangle$  crystals. The calculations depend on a value of dislocation density which is obtained from Fig. 2. The data in Fig. 2 are plotted for  $\langle 321 \rangle$  crystals. In  $\langle 110 \rangle$  crystals deformation takes place on two intersecting slip systems simultaneously, and it may be that the rate of dislocation multiplication will be higher in these crystals. The value of  $n$  obtained from Fig. 2 will then be too low for the  $\langle 110 \rangle$  crystals, resulting in a calculated value for the velocity that is too high.

*Heavily doped crystals.* Shown also in Fig. 4 are the results of a tensile test and a direct velocity measurement at 650°C on the two specimens cut from the heavily gallium-doped crystal. The direct velocity measurement gave for the same stress a higher velocity than in the undoped crystal. In the tensile test the value of stress at the maximum was much higher than that for an undoped crystal deformed at the same strain rate, and the calculated velocity was much lower.

To explain these results it is first necessary to consider a suggestion that, since vacancies in germanium are acceptors, a heavy concentration of an acceptor impurity such as gallium will suppress the formation of vacancies.<sup>17</sup> Not only will there be few vacancies in the crystal, but the number of condensed vacancy clusters typical of all these crystals, particularly the dislocation-free crystals,\* will

\*The gallium-doped crystal was, in fact, dislocation-free.

be considerably reduced. The high measured value for dislocation velocity follows directly from the low vacancy concentration. An explanation for the very low calculated value for the dislocation velocity at a very high stress can be based on a suggestion by Wilsdorf<sup>18</sup> as to the mechanism of dislocation multiplication. Condensed vacancy clusters form prismatic loops of dislocation. Segments of these loops lie in active slip planes, and if the local stress is raised to a sufficiently high value by the passage of a nearby dislocation, then these segments can act as Frank-Read sources. With few of these loops present, the rate of dislocation multiplication will be much lower than normal, and again a value for dislocation density taken from Fig. 2 will be in error. In this instance  $n$  will be too high, and will give a calculated value of  $v$  which is too low.

### Strain aging

Some strain aging experiments were performed. A pulled crystal was tested at 650°C in the tensile machine. After the initial yield drop had occurred, the crystal was unloaded and immediately reloaded. Deformation continued at essentially the same stress at which the test had been interrupted, though a very small yield drop was found. The dislocation configuration of the crystal will, under the influence of the internal stress field, relax slightly upon unloading, and upon reloading must be "unrelaxed" before deformation can continue. This explains the small yield drop. After further plastic deformation of a few per cent, the crystal was again unloaded, annealed for 2½ hr at 800°C and then retested at 650°C. Deformation continued at a value of stress about 15 per cent lower than that at which the previous test had been stopped, and the small yield drop was more pronounced (it can clearly be seen in Fig. 1a, which shows the curves for this experiment). The annealing treatment failed to reproduce the initial tensile curve, showing that strain aging had not occurred. The lower value of the yield stress is presumably due to some dislocation rearrangement during annealing. This rearrangement has also introduced a larger relaxation which must be overcome before dislocations may start to move again, accounting for the enhancement of the small yield drop. The test was repeated on a similar specimen, and on the heavily doped gallium specimen (annealed for 20 hrs at 850°C because of the much slower diffusion of gallium in germanium) with identical results.

The absence of strain aging may be explained by considering the relative numbers of active dislocations and impurity atoms. The fact that the initial dislocation density has little effect on subsequent plastic deformation suggests that grown-in dislocations are immobile, probably due to Cottrell-type locking by impurity atoms. There are up to  $10^4$  dislocation lines/sq cc in these crystals, and the impurity content of  $10^{13}$  atoms/cc present is adequate to supply more than one atom for every atom length of dislocation line. Strain-aging effects are not observed because the dislocation density rises to between  $10^6$  and  $10^7$  lines/sq cc after yielding, and  $10^{14}$  to  $10^{15}$

impurity atoms/cc are required to provide one atom per atom length of dislocation. The crystal containing  $5 \times 10^{19}$  atoms/cc of gallium does contain enough impurity to show a strain-aging effect, provided that gallium atoms are able to lock the dislocations. This is unlikely, however, since the strain introduced into the germanium lattice by the presence of this amount of gallium is so small as to be undetected by x-ray parameter measurements,<sup>19</sup> and it is therefore not surprising that strain-aging effects are also absent in these crystals.

### Strain hardening

Although this investigation has not been concerned with strain hardening, it is perhaps worthwhile to mention some conclusions which can be drawn about this very important aspect of plastic deformation. The case of creep is the simplest to consider, and again use will be made of formula (6), and of the fact that for all creep tests made, the onset of strain hardening occurred at lower strains for lower stresses and lower temperatures. In the strain-hardening range of creep deformation, the strain-rate is decreasing. From Eq. (6) this can be due either to a decrease in the number of active dislocations or to a decrease in dislocation velocity. If, after a certain amount of plastic deformation, dislocations are blocked by obstacles of some sort, then the number of mobile dislocations is reduced. The back-stress from the blocked dislocations will also reduce the effective stress on other moving dislocations and their velocity will decrease. Blocking will occur more easily at lower stress levels and temperatures, explaining why the strain-hardening region begins after a lesser amount of total strain in crystals tested at lower stresses or at lower temperatures. The drawback to this model is that blocked dislocations have not been seen in germanium. Up to the limit at which individual dislocations can be resolved, the spacing of dislocations along slip lines is quite uniform. A technique for looking directly at dislocations which is more refined than the etch-pit method may eventually reveal blocked dislocations in germanium, but until they are seen, it is wise to consider other models for strain hardening. A dislocation will have greater difficulty in moving through a crystal as the amount of plastic deformation increases, because of trails of imperfections left by dislocations which have previously passed through the lattice. Under a constant applied stress, the dislocation velocity would decrease as the amount of deformation increases. This effect will cause some strain-hardening, but is thought to be large enough to account for only a small part of the observed hardening. Another possibility is that dislocation multiplication slows down or ceases because of exhaustion of multiplication sources. If it is assumed that these sources exist with a spectrum of operating stresses, then at lower stress levels fewer sources will be brought into operation and exhaustion will occur after lesser amounts of strain. Thermal assistance of source activation explains the temperature dependence. No proposal as to why these sources should become exhausted is made, except

that perhaps each source can operate only once. At present there is not sufficient evidence to favor any one of the proposed mechanisms for strain-hardening. The above ideas have been described in terms of creep; they apply equally well to tensile deformation, though in this case, the strain-rate is the constant in Eq. (6).

#### Effect of number of initially mobile dislocations on deformation behavior

In all materials so far examined, dislocation multiplication occurred during plastic deformation. A moving dislocation in any crystal must experience a frictional force, which is the sum of the Peierls-Nabarro force and the forces arising from the interaction of the dislocation with other imperfections, and which increases with increasing velocity of the dislocation. Thus dislocations in all materials will move with a velocity characteristic of the applied stress. This has also been demonstrated in silicon-iron.<sup>20</sup> The important criterion for the observation of a Johnston and Gilman type stress-strain curve is therefore that the number of initially mobile dislocations be small. Mathematically the condition is that  $n_0$  is small enough such that  $S_c/L_0 > n_0 b v(\sigma_y)$ , that is, the strain rate imposed on the specimen is greater than the product of

the number of mobile dislocations, their Burgers vector, and the dislocation velocity characteristic of the yield stress  $\sigma_y$ . This yield stress is the stress necessary to activate the first dislocation sources. If  $\sigma_y$  is high, or dislocation velocities are high at relatively low stresses, or if  $n_0$  is high, such that the above product is greater than the imposed strain-rate, then a stress-strain curve of this type is not obtained. The reasons that such a curve is not seen when most common metals are subjected to a tensile test are that  $n_0$  and  $v$  are both much larger than in ionic and covalent materials which do show a large yield point. Presumably this type of curve can be obtained on any material if the imposed strain-rate could be made sufficiently high.

#### Acknowledgment

The author is indebted to Dr. A. Kelly, Cambridge University, for several suggestions, and wishes also to thank colleagues at IBM, in particular G. E. Brock, W. P. Dumke and G. R. Gunther-Mohr, for many helpful discussions; B. K. Bischoff and C. J. Lent for growth and preparation of tensile specimens; and J. Grandia for help in design and for construction of experimental apparatus.

#### References

1. G. J. Gallagher, *Phys. Rev.* **88**, 721 (1952).
2. R. G. Treuting, *Trans. Am. Inst. Min. Met. Engrs.* **203**, 1027 (1955).
3. J. R. Patel and B. H. Alexander, *Acta Met.* **4**, 385 (1956).
4. H. G. van Bueren, *Physica* **24**, 831 (1958).
5. P. Penning and G. De Wind, *Physica* **25**, 765 (1959).
6. F. Seitz, *Phys. Rev.* **88**, 722 (1952).
7. J. R. Patel, *Phys. Rev.* **101**, 1436 (1956).
8. D. Dew-Hughes and G. E. Brock, *J. Appl. Phys.* **30**, 2020 (1959).
9. W. G. Johnston and J. J. Gilman, *J. Appl. Phys.* **30**, 129 (1959).
10. H. G. van Bueren, *Physica* **25**, 775 (1959).
11. W. G. Johnston and J. J. Gilman, *J. Appl. Phys.* **31**, 632 (1960).
12. W. E. Taylor, *Phys. Rev.* **86**, 642 (1952).
13. R. A. Logan, *Phys. Rev.* **101**, 1455 (1956).
14. J. N. Hobstetter, *Progress in Metal Physics*, Volume VII, p. 1, Pergamon Press, 1958.
15. P. Haasen, *Acta Met.* **5**, 598 (1957).
16. H. Letaw, Jr., *J. Phys. Chem. Solids*, **1**, 100 (1956).
17. J. R. Patel, American Physical Society Meeting, Detroit, March, 1960.
18. H. G. Wilsdorf, private communication.
19. G. E. Brock, private communication.
20. D. F. Stein and J. R. Low, Jr., *J. Appl. Phys.* **31**, 362 (1960).

Received June 9, 1961