Effect of Dislocations on the Minority Carrier Lifetime in Semiconductors*

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The density of random dislocations in germanium and silicon crystals has been measured by means of x-ray rocking curves and by etch pit counting. Data obtained by the two methods are in good agreement, and dislocation densities in the range $10^4 - 10^7$ /cm² were found. The minority carrier lifetime was shown to vary with the dislocation density, and the results could be expressed in terms of a recombination efficiency per unit length of dislocation line, $\sigma_R = 1/N_D \tau$ (where N_D = dislocation density, $\tau =$ lifetime). σ_R was found to decrease with increasing resistivity of germanium and was higher for silicon than for germanium of comparable purity.

I. INTRODUCTION

HE present work was undertaken to investigate the relationship between the structural perfection of germanium and silicon crystals and the rate of recombination of holes and electrons.¹ Since transistor action depends on the modulating effect of a small number of holes in material where the majority of the conduction takes place by the movement of electrons, or conversely the modulating effect of a small number of electrons on the movement of positive holes, the rate of recombination of holes and electrons is an important factor in the performance of semiconductor devices. Theoretical values of the average recombination time for germanium are about one second; however, experimental values are of the order of 10^{-3} to 10^{-6} second. It was felt that the recombination process was catalyzed by the presence of imperfections such as dislocations, vacancies, low-angle boundaries, and impurity atoms, and that the minority carrier lifetime was greatly reduced by the presence of these defects.

While there was some evidence that crystal perfection influences the lifetime, previous investigations² have been qualitative in nature. In the present work, the effect of randomly dispersed dislocations on the minority carrier lifetime has been evaluated quantitatively for high- and low-resistivity germanium and for highresistivity silicon. The dislocation density was determined by etch pit counting and by means of x-ray rocking curves; the two methods gave consistent results, although the x-ray method was preferred. V correlation between the recombination time and the number of imperfections was found and some possible explanations are discussed.

II. EXPERIMENTAL METHODS

A. Metallography

Samples of single crystal germanium were prepared in order to determine the edge-type dislocation density and distribution. Specimens for this investigation were selected at random from crystals either supplied by various transistor manufacturers or grown in the laboratory and were cut to within 0.5 degree of either a (111) or (100) plane using Laue back-reflection techniques. The oriented surfaces were either mechanically polished or electropolished and then etched with CP4³ or superoxol etch.4

Under suitable etching conditions, Vogel et al.⁵ have shown that individual etch pits are nucleated by single dislocations, with the pit being formed where the dislocation singular line intersects the surface. The observed etch pits may be randomly distributed throughout the crystal or may be segregated to form low-angle boundaries. Figure 1 shows a photomicrograph of such a boundary, and Fig. 2 shows a rocking curve taken with the beam straddling the boundary. The angular mis-



FIG. 1. Low-angle boundary in germanium, electropolished and etched, dark field, $100 \times$. Area reduced approximately 66% in reproduction.

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¹W. Shockley, Electrons and Holes in Semiconductors (D. Van Nostrand Company, Inc., New York, 1950).

² Pearson, Read, and Morin, Phys. Rev. 93, 666 (1954).

³ 50 parts nitric acid, 30 parts acetic acid, 30 parts hydrofluoric acid, 1 part liquid bromine. ⁴1 part 48% hydrofluoric acid, 1 part 30% hydrogen peroxide,

⁴ parts water. ⁶ Vogel, Pfann, Corey, and Thomas, Phys. Rev. **90**, 489 (1953).



FIG. 2. Rocking curve with x-ray beam straddling boundary (111) reflection. Misorientation across boundary approximately 80 seconds.

orientation at the boundary calculated from the spacing of the etch pits along the boundary yields excellent agreement with the angular separation of the diffraction peaks, thus indicating that each pit is a single edge-type dislocation. The x-ray measurement is described in the following section. Unusual structures have been revealed in crystals where several low-angle boundaries intersect. The dislocation geometry at and near such a typical intersection is shown in Fig. 3. Here the single boundaries appear to degenerate into many subboundaries, in order to accommodate the change in crystallographic orientation. A typical crystal with randomly distributed dislocations is shown in Fig. 4.

If one assumes that the randomly-distributed etch pits are also nucleated by single dislocations, then the dislocation density may be determined by simply counting the number of pits. The values obtained ranged from about 10⁴ dislocations/cm² in the most perfect material to values as high as 10⁸ dislocations/ cm² in rather imperfect crystals. Under high magnification $(2000 \times)$ the large pits were sometimes found to encompass more than one dislocation, and this effect presumably becomes more pronounced as the dislocation density increases. Thomas⁶ has pointed out that the size of the pit may also be a function of the length of dislocation singular line just below the surface. Since an edge-type dislocation may terminate in a screw component, the length of the last segment of edge-type singular line may vary. If this length is less than the depth of a fully developed pit (several thousand interatomic spacings), the pit may appear smaller than its neighbors. It is therefore important to include all such pits when counting etch pits. It was also observed in this investigation that the shape of the pit varied as the angle between the singular line and the surface was changed. As the angle was gradually increased, the pit became shallower and the dot was displaced from the center. At an angle greater than approximately 30°, the pit essentially disappeared. All of these effects can combine to give etch pit densities lower than the true dislocation density.

B. X-Ray Measurements

Darwin has shown that the width of an x-ray diffraction line is exceedingly narrow for an ideally perfect crystal.⁷ For germanium the ideal width of a (111) reflection with Cu $K\alpha_1$ is calculated to be 15.6 seconds, and a few crystals exhibited x-ray rocking curves which approached this figure. However, the presence of random edge dislocations introduces locally disoriented regions which diffract at slightly different angles,⁸ thus broadening the rocking curve. Conversely, a measurement of the rocking curve provides some information about the imperfections in the crystal.

A double-crystal spectrometer, with the best available germanium crystal in the first position, was used to determine the x-ray rocking curves. The two crystals were in parallel positions, and Cu $K\alpha$ radiation was used to observe the (111) reflections. The angular measurement was accurate to 1 second of arc and a typical curve is shown in Fig. 5. The surfaces of each crystal were carefully polished and then etched sufficiently to remove the worked layer from the surface. A



FIG. 3. Dislocation geometry near intersection of low-angle boundaries. Electropolished and etched, $100\times$. Area reduced approximately 66% in reproduction.

⁶D. A. Thomas, Bell Telephone Laboratories Technical Memorandum, 1953 (unpublished).

⁷ R. W. James, Optical Principles of the Diffraction of X-Rays (G. Bell and Sons, London, 1950). ⁸ R. D. Heidenreich and W. Shockley, Report on the Conference

[•] R. D. Heidenreich and W. Shockley, *Report on the Conference* on Strength of Solids (Physical Society, London, 1948).

narrow x-ray beam was used and various regions of the crystal faces were analyzed.

A tilt adjustment was introduced in order to allow adjustments of the diffracting planes in the vertical direction. It was shown experimentally that additional broadening occurred unless the relative vertical tilt was less than about 5 minutes for the average germanium crystal (half-width \approx 30 sec). This adjustment was made on the spectrometer before each curve was run. The measured rocking curve must also be corrected for the broadening of the first crystal. It is easily shown⁹ that the correction takes the form

$$K = (\beta^2 - \phi^2)^{\frac{1}{2}},\tag{1}$$

where K is the true half-breadth of the second crystal, β the measured half-breadth of the second crystal, and ϕ the true half-breadth of the first crystal. This correction applies to either the half-breadth or the mean breadth, i.e., at e^{-1} of the maximum. The same type of calculation was used to correct the values of K for the natural broadening of the crystals.

With dislocations arranged randomly with an average spacing, h, the dislocation density, N_D , is given by $N_D = 1/h^2$. The angular deviation, ψ , in the region of one dislocation relative to some fixed orientation is, $\psi = b/h$, where b is the interatomic spacing in the slip plane. For the entire region about a dislocation, the average misorientation is zero, but there is an orientation distribution about this average value which is in effect measured by the rocking curve. The most probable deviation from the mean value can be determined from the corrected rocking curve, and this was used by Gay, Hirsch, and Kelly¹⁰ to calculate the dislocation density. The total dislocation density, N_D , is then given by

$$N_D = K^2 / 9b^2.$$
 (2)



FIG. 4. Random dislocations in germanium crystal. CP⁴ etch, 500×. Approximate density, 10^5 dislocations per cm². Area reduced approximately 66% in reproduction.



FIG. 5. Typical rocking curve for germanium. Cu $K\alpha$ radiation.

C. Electrical Measurements

Minority carrier lifetimes were measured by observing the rate of decay of photoconductivity in samples of known geometry. This method was developed by Haynes and Hornbeck¹¹ and refined by Stevenson and Keyes.¹² The specimens were rectangular bars with dimensions approximately 20×4×4 mm. Ohmic contacts were soldered on the two ends and the other four surfaces were either sandblasted or etched with CP4.13 A chopped light source which completely irradiated one surface was used to create excess hole-electron pairs in the sample. Since the excess conductance of the sample is proportional to the total number of hole-electron pairs in excess of the equilibrium value, an observation of the time dependence of the conductivity yielded the rate at which holes and electrons recombine. A constant current was passed through the sample, and the change in conductance was used to indicate the change in voltage across the sample. After suitable amplification, this voltage was observed on an oscilloscope, and the time required for the amplitude of the signal to decay to half of its original value was recorded. The minority carrier lifetime was then calculated.12

Electrical resistivities were determined by a voltage probe method. Ohmic contacts were soldered onto the ends of the specimen and a constant current was passed through it. The potential drop was then measured at intervals of one millimeter along the length of the sample using a microprobe.

III. EXPERIMENTAL RESULTS AND DISCUSSION

A. Comparison of Rocking Curve Measurements with Etch Pit Counting

Dislocation densities calculated from rocking curve widths (of {111} diffraction peaks) may be compared with the values determined by etch pit counting. If the two determinations were equivalent over the entire

⁹ A. Taylor, Phil. Mag. 31, 339 (1941).

¹⁰ Gay, Hirsch, Kelly, Acta Metallurgica 1, 315 (1953).

J. R. Haynes and J. A. Hornbeck, Phys. Rev. 90, 152 (1953).
 D. T. Stevenson and R. J. Keyes, Appl. Phys. 26, 190 (1955).
 Boundary conditions for the solution of the diffusion equation

for the motion of holes and electrons are determined by the state of the surface.



FIG. 6. Comparison of etch pit and x-ray dislocation density.

range, then a plot of etch pit density versus x-ray density should give a straight line with a 45° slope. Dislocation densities were determined by both methods on the same series of germanium crystals and the resulting correlation is shown in Fig. 6. For low dislocation densities the agreement with the 45° slope is quite good, but in the case of higher dislocation densities, the etch pit counting yields somewhat lower values than that obtained from x-ray broadening. This deviation may be expected since closely spaced dislocations which may not be resolved under the microscope can still contribute to the x-ray broadening. Also screw dislocations which connect straight segments of edge-type dislocations do not appear to contribute to the etch pit density but may give rise to some broadening. This would



FIG. 7. Dislocation density vs lifetime for low-resistivity germanium.

also tend to give lower etch pit values as compared with the x-ray results.

Furthermore, only those dislocation singular lines which intersect the surface at a relatively small angle (less than 30°) with respect to the surface normal will nucleate visible etch pits. One may calculate an approximate relationship between the etch pit and x-ray densities, taking this orientation effect into account. For a given (111) plane there are twelve possible orientations of the (211) singular lines with respect to the (111) pole. The singular lines (112), (211), and (121) are oriented at 90° to the (111) pole. The (211), (211), (121), (121), (112) and the (112) singular lines make angles of 62° with the (111) pole, while the (211), (112) and the (121) singular lines make an angle of about 19°. Thus for the {211} type singular lines, only three of the twelve singular lines should yield etch pits.

On the other hand, one may assume that all of the dislocations tend to broaden the rocking curve, the broadening contribution depending on the sine of the angle between the singular line and the (111) pole. Thus one must add the effective components of the variously oriented dislocations to determine the total broadening. The three singular lines at 90° to the (111) pole contribute full components, the six at 62° each contribute 0.85 while the three at 19° each contribute 0.33. Thus the ratio of etch pit density to x-ray density should be of the order of 3/[3+6(0.85)+3(0.33)] which is approximately one-third. This line is also shown in Fig. 6. From the figure it is seen that the experimentally determined correlation lies between the 45° line and this lower value. Only one set of dislocations {211} has been considered in this calculation, but the apparent correlation indicates that the contribution of other dislocation types is small.

One might conclude from this analysis that the x-ray measurements tend to give more reproducible values of dislocation densities, and this method was used generally for the quantitative determination of dislocation densities in this study. However, in order to obtain more complete information about the distribution of dislocations in the sample, both x-ray and metallographic measurements were used to supplement each other.

B. Correlation between Dislocation Density and Minority Carrier Lifetime

The experimental data indicated that edge-type dislocations markedly affect the recombination rate of holes and electrons in germanium and silicon. Dislocation densities measured by x-ray rocking curves were correlated with the minority carrier lifetime in germanium samples of two electrical resistivity ranges. Lifetime as a function of dislocation density for these two ranges is shown in Figs. 7 and 8; for the low-resistivity *n*-type germanium (3–5 ohm cm), the minority carrier lifetime varied from 700 microseconds, corresponding to about 5×10^4 dislocations per cm², to about 10 microseconds for material with 10^7 dislocations per cm². Values of the lifetime for high-resistivity germanium (30–40 ohm cm) of the same dislocation density are, of course, much higher. Similar results were obtained for silicon crystals in the 40 ohm cm resistivity range and these are shown in Fig. 9. These data indicate a hyperbolic relationship between lifetime and crystal perfection.

This functional dependence may be interpreted by assuming that each dislocation acts as a recombination center. The rate of recombination, λ , should then be proportional to the product of the excess number of minority carriers above the equilibrium value and the number of dislocations. This may be expressed as follows:

$$\lambda = \Delta P / \tau = \sigma_R N_D \Delta P, \qquad (3)$$

where ΔP is the excess number of minority carriers, N_D is the number of dislocations, τ is the mean lifetime of the minority carrier, and σ_R is a proportionality con-



FIG. 8. Dislocation density *vs* lifetime for high-lifetime germanium.

stant corresponding to the recombination efficiency per unit length of dislocation. The unit recombination efficiency, σ_R , is thus $1/\tau N_D$. A plot of $1/\tau$ versus N_D should, therefore, yield a straight line with the slope σ_R . Figure 10 is such a plot for germanium and silicon, and the resulting straight lines appear to substantiate the simple theory. All of these samples have comparable ranges of dislocation densities (10^5-10^7 dislocations per cm²) but exhibit variations in lifetime as a function of both resistivity and dislocation density. The following values of σ_R were obtained:

Material	$\sigma_R (\mathrm{cm}^{-1} \mathrm{sec}^{-1})$
high-resistivity Ge	5.5×10^{-4}
low-resistivity Ge	3.5×10 ^{−3}
high-resistivity Si	1.65×10^{-2}

It is apparent that σ_R for silicon is much larger than for germanium of comparable purity. This table also shows the large variation of σ_R with resistivity for germanium. Since σ_R is the effective recombination efficiency of a



FIG. 9. Dislocation density vs lifetime for silicon.

unit length of dislocation singular line, it would seem that an individual dislocation in high-resistivity germanium has a much smaller effect on the lifetime than in low-resistivity germanium. This is in agreement with the Shockley-Read theory of recombination¹⁴ which predicts a large variation of lifetime with resistivity. In turn, it is found that an individual dislocation in lowresistivity germanium is less harmful to the lifetime than the same dislocation in silicon. One might thus conclude that it is fundamentally more difficult to produce high-lifetime silicon as compared to germanium.

C. Recombination at Edge-Type Dislocations

In order for a hole and an electron to recombine, they must radiate energy in the form of light or thermal vibrations. The recombination time for either of these processes in a perfect crystal has been estimated to give a lifetime on the order of one second.¹ Since the measured values of the lifetime range from 10^{-5} to 10^{-3} seconds, it is apparent that some perturbation in the structure is required to catalyze the recombination process. Shifts in the energy gap, introduction of additional energy levels, or changes in the modes of vibration





¹⁴ W. Shockley and W. T. Read, Jr., Phys. Rev. 87, 835 (1952).

<i>r</i> _A Atomic Solute radius atom (A)	Germanium		Silicon		
	radius (A)	$\frac{r-r_A}{r}$	V (ev)	$\frac{r-r_A}{r}$	V (ev)
Ni Fe Cu	1.24 1.26 1.28	0.019 0.033 0.05	0.04 0.07 0.12	0.05 0.07 0.08	0.12 0.15 0.18

TABLE I. Energies of formation of Cottrell atmospheres.

of the lattice could all facilitate recombination. Several ways in which dislocations in germanium and silicon could aid recombination are: shifts in the position of the valence and conduction band edges about a dislocation, formation of impurity atmospheres near the dislocation, and introduction of unsaturated covalent bonds along the singular line.

The introduction of an edge-type dislocation in the lattice gives rise to an elastic strain that extends throughout an appreciable region in the crystal. In a material such as germanium or silicon, where the bonding is highly directional, the electronic energy is a sensitive function of the interatomic spacing. Kulin and Kurtz¹⁵ have shown that the strains resulting from the dislocation can have a large effect on the electronic energies. They show that an electron will prefer to be localized in the compression region above a slip plane, and that a hole will also tend to reside in the higherenergy valence states of the same region. Thus all along the tension side of the slip plane, holes and electrons can come together in low-lying energy states, and then recombine across a decreased gap. The effect of the dilation about the dislocation is twofold: in the region near the singular line electrons and holes can reside in close proximity, and secondly, the frequency of annihilation is greater because of the lower energy which now must be given off in the recombination process. Hence, one would expect the rate of recombination to be greater here than in the undistorted bulk of the crystal.

A second important effect associated with dislocations in germanium and silicon is the formation of an atmosphere of impurity atoms along the singular line. Because of its stress field, a dislocation will interact with other sources of internal stress in a crystal. Since the energy of interaction changes as a function of distance from the center of the dislocation stress field, it can be seen that the solute atom will experience a net force drawing it to its region where it can relieve the most stress. Cottrell¹⁶ has shown that the interaction energy for a solute atom of radius $r_A = r(1+\epsilon)$ in a solvent of atomic radius r can be expressed as

$$V = \frac{4}{3} \frac{1+\nu \sin\theta}{1-\nu R},$$
(7)

where G is the shear modulus, ν is Poisson's ratio, b is the Burgers vector, and R and θ are the polar coordi-

nates in plane of the dislocation. Approximate values of V may be determined by assuming that the impurity atom sits next to the dislocation singular line. For this case, R/b is taken as unity. These calculations have been performed for the impurities, copper, nickel, and iron, dissolved in both silicon and germanium, and the results are shown in Table I.

While these calculations indicate there is a driving force toward segregation, it should be pointed out that only the elastic interaction was considered. In a more complete treatment, one must include the effects of the changing electronic distribution and rearrangement of bonding in the neighborhood of the dislocation. However, it appears that some segregation should take place along the singular line. This should further enhance recombination by providing localized impurity trapping levels in a region where the hole-electron density is already greater than in the bulk.

Furthermore, atmosphere formation at dislocations is temperature-dependent. As the temperature increases, solute atoms will begin leaving the dislocations and will migrate through the crystal. When the thermal energy is greater than V, one can assume that the solute atoms are dispersed uniformly throughout the crystal. On fairly rapid cooling from such a high temperature, the random impurity density is frozen in because of the decreased mobility of the solute atoms at low temperatures. However, reheating to some temperature below, but in the vicinity of the escape temperature (where $T_E \equiv V/k$), should restore the atmospheres.

The third important perturbation, the introduction of acceptor levels along the dislocation singular line, has recently been treated in detail by Read.¹⁷ The lack of atomic continuity produced by the dislocation results in unsaturated bonding because of the presence of unpaired dangling electrons. These electrons may accept other electrons to form a dangling pair, thus lowering the total energy and leaving the dislocation negatively charged. Impurity atoms clustered along the singular line may also interact with the unpaired electrons. The defect structure in the nature of the region of the dislocation may affect this behavior because the tetrahedral bonding required in the bulk need not be satisfied near the dislocation. Thus atoms such as copper which ionize in the bulk (usually acting as an acceptor in *n*-type germanium) may form a Cottrell atmosphere of essentially neutral atoms. Upon heating, the copper would be dispersed throughout the volume of the crystal and would again ionize.

The effect of dislocations on the recombination process appears to be quite complex. However, all of the mechanisms discussed here appear to act in the same direction, i.e., to increase the rate of recombination of holes and electrons.

¹⁷ W. T. Read, Jr., Phil. Mag. 45, 715 (1954).

¹⁵ S. A. Kulin and A. D. Kurtz, Acta Metallurgica 2, 354 (1954). ¹⁶ A. H. Cottrell, in *Report on the Conference on Strength of Solids* (Physical Society, London, 1948).

IV. SUMMARY AND CONCLUSIONS

The results of this investigation may be summarized as follows: (1) The density of randomly distributed dislocations in germanium and silicon has been measured by x-ray rocking curves and by etch pit counting. The results obtained from the two methods were in good agreement and dislocation densities in the range $10^4 - 10^7$ /cm² were found. (2) Variations in dislocation density were shown to produce large changes in minority carrier lifetime. The lifetime, τ , was found to be related to the dislocation density, N_D , in terms of a specific recombination efficienty, $\sigma_R = 1/N_D \tau$. σ_R was found to decrease with increasing resistivity of germanium and was higher for silicon than for germanium of comparable purity.

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Simultaneous Transport of Heavy and Light Holes in Semiconductors with a Degenerate Valence Band

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A theoretical study is presented of the motion in an *n*-type semiconducting filament of an injected narrow pulse of slow and fast holes subject to drift, diffusion, recombination, and reversible interband transitions. For low injection level and for interband transition times which are small compared to the recombination lifetime and to the observation time but large compared to the time between collisions, it is shown that both sets of holes propagate and broaden as a single pulse with a group mobility and diffusivity heavily weighted by that of the slower holes. This explains why only a single pulse is observed at the collector in drift mobility experiments.

I. INTRODUCTION

HE discovery by means of cyclotron resonance experiments of two hole masses in germanium¹ has stimulated recent efforts^{2,3} to find additional evidence for the two types of holes with the use of other experimental techniques. Willardson, Harman, and Beer² have found evidence from measurements of transverse Hall effect and magnetoresistance vs temperature and magnetic field for the presence in p-type germanium of a small percentage of holes (ca 2 percent) with a mobility eight times as large as that of the majority holes. On the other hand, Harrick³ has conducted a careful search for the holes of higher mobility in *n*-type germanium, using the drift mobility technique with greatly improved sensitivity, and has been unable to observe a signal attributable to the faster set of holes. It is the purpose of the present note to suggest a possible explanation for the drift mobility observations and to provide a new interpretation of the mobility computed from such experimental data.

In semiconductors with a doubly degenerate valence band top, at temperatures high enough to permit complete occupation of acceptor levels, and in the absence of an applied electric field, it is to be expected that rapid electronic transitions will occur reversibly between the two degenerate states. This will result in the conversion of light holes into heavy holes and vice versa, the population ratio of the two states in thermal equilibrium being of the order of the effective mass ratio raised to the three-halves power for spherical energy surfaces. Now if an electric field is applied of such magnitude that the energy gained by the holes is small compared to their thermal energy, a condition which obtains in the usual drift mobility experiment, then it should be an excellent approximation to assume that reversible transitions between the levels will still occur under this nonequilibrium condition. The motion of added holes in a filament of the semiconductor will therefore be complicated by the existence of the two types of holes and their reversible conversion one into the other.

In Sec. II, we generalize the differential equations governing the transport of holes so as to include the reversible interband transitions in addition to the usually considered^{4,5} processes of drift, diffusion, and electron-hole recombination. The generalized equations. together with initial and boundary conditions appropriate to the drift mobility experiment, are then solved

 ¹ Dresselhaus, Kip, and Kittel, Phys. Rev. 92, 827 (1953).
 ² Willardson, Harman, and Beer, Phys. Rev. 96, 1512 (1954).
 ³ N. J. Harrick, Phys. Rev. 98, 1131 (1955).

⁴ W. van Roosbroeck, Bell System Tech. J. **29**, 560 (1950). ⁵ H. Brooks, Phys. Rev. **90**, 336(A) (1953).



FIG. 1. Low-angle boundary in germanium, electropolished and etched, dark field, $100\times.$ Area reduced approximately 66% in reproduction.



FIG. 3. Dislocation geometry near intersection of low-angle boundaries. Electropolished and etched, 100 \times . Area reduced approximately 66% in reproduction.



FIG. 4. Random dislocations in germanium crystal. CP⁴ etch, $500 \times$. Approximate density, 10^5 dislocations per cm². Area reduced approximately 66% in reproduction.