

**International Symposium
on
Dislocations in Tetrahedrally Coordinated
Semiconductors**

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**International Symposium
on
Dislocations in Tetrahedrally Coordinated
Semiconductors**

Hünfeld/Fulda (FRG)

September 25-29, 1978

The Conference has been held ~~at the~~ Bonifatius-Kloster Hünfeld

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FOREWORD

The Polish Academy of Sciences has sponsored a number of summer schools on Defects in Covalent Crystals during the last decade under the leadership of J. Auleytner. In this tradition the Akademie der Wissenschaften in Göttingen invited to this symposium in the Bonifatius-Kloster in Hünfeld which turned out to be very stimulating and timely. The subject of the lively, controversial but always very interesting discussions is just about 25 years under active research since the pioneering work of W. Shockley and especially of W.T. Read jr. at Bell Labs. The participants of the Hünfeld Symposium decided to dedicate these Proceedings to Thornton Read in recognition of his lasting contribution to the field. They appreciate also the financial contributions of Deutsche Forschungsgemeinschaft, of AG Metallphysik der DPG, DGM und VdEh, and of IBM Deutschland GmbH as well as of Siemens AG. The committee enjoyed the help provided by the brothers of the monastery and of their students and wishes to thank all participants of the symposium for their cooperation and contributions.

To avoid confusion in nomenclature on dislocations in polar AB compounds the participants of this symposium recommended the use of the terms "A- or B-dislocation" for a dislocation with A or B atoms in the most distorted core positions. The older terminology " α or β -dislocation" refers to dislocations ending with rows of lower or higher valent atoms in the so-called shuffle set (s) of {111} pairs of planes. It is now evident that not these but the glide set (g) of {111} planes are sheared so that previous terminology, well related to the outer surfaces of a macroscopic specimen, has to be reinterpreted. It is hoped that future publications will state clearly whether they base their discussion on A (g) and B (g) dislocations or on A (s) and B (s) $\equiv \alpha$ and β -dislocations.

These proceedings were edited with the aid of Dr. H.G. Brion to whom we are grateful.

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MODELS OF THE DISLOCATION STRUCTURE

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Résumé.- On montre que les dislocations sont dissociées non seulement au repos mais aussi en mouvement. En utilisant les résultats obtenus par microscopie électronique, mesures électriques et spectroscopie EPR, il est montré que pendant la déformation plastique de nombreux défauts ponctuels sont relâchés par les dislocations en dessous d'une température critique. Ceci montre qu'au moins une partie des dislocations existe sous forme d'une association de dislocation dissociée avec une rangée de défauts ponctuels. Les énergies de fonte d'empilement dans le germanium (75 ± 10 mJ/m²) et le silicium (58 ± 6 mJ/m²) ont été à nouveau déterminées.

Abstract.- It is demonstrated that dislocations not merely at rest but during their motion are dissociated. Combining results of electron microscopy, electric measurements and EPR spectroscopy it is concluded that during plastic deformation below a critical temperature numerous point defects are released by the dislocations. This indicates that at least part of the dislocations exists in form of an association of a split dislocation with a linear row of point defects. The stacking fault energies of germanium (75 ± 10 mJ/m²) and silicon (58 ± 6 mJ/m²) are redetermined.

1. Introduction.- In the initial work on the dislocations in diamond like crystals Shockley /1/ and Read /2/ outlined the core structure of the dislocations in such a way that the number of severely distorted covalent (sp^3) bonds became as small as possible. From this principle followed the existence of well localized unpaired electrons, so called dangling bonds in the core of most dislocation types, only the distance between two dangling bonds depending on the dislocation character. A second assumption made in the pioneering papers concerned the choice of the glide plane: whereas there was never any doubt that the glide systems of those crystals are of the type (111) $[011]$, there remained two possibilities of shearing parallel to (111) planes. Figure 1 shows a projection of the structure on a (110) plane.

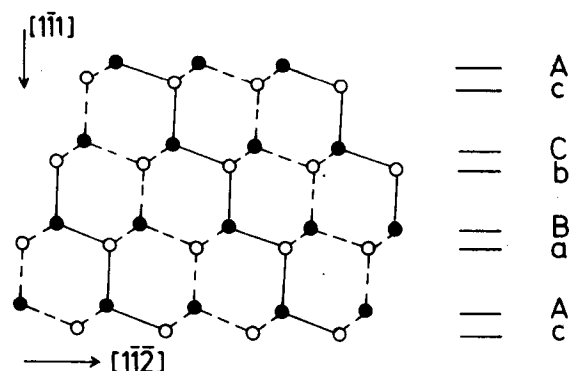


Fig. 1: Projection of the diamond structure on a (110) plane.

The stacking order parallel to $[111]$ is deduced from that of the face centred cubic (fcc) lattice A-B-C by doubling the atoms in the unit cell: A-aB-bC-cA-... We may therefore shear one part of the crystal against the other either between widely spaced planes (e.g. A and a) or between narrowly spaced ones (a and B). For a long time people followed Shockley assuming that the actual glide planes should be placed between widely spaced planes for two reasons: firstly the number of covalent bonds to be broken is three times smaller in this case and secondly the glide planes are commonly the most distant planes in a structure. These arguments lead to an idea of the dislocations in the diamond structure which was used in the following years in discussion of electrical and magnetic properties of dislocations in semiconductors. The interpretation of the results of many careful experiments in these fields turned out to be not clear-cut. It should be noted that Hornstra /3/ in 1958 in an important paper discussed a wider variety of dislocation structures which appeared to be conceivable in the diamond structure. But while an experimental decision between the various possibilities was lacking the simple dangling bond model prevailed. It was the introduction of weak beam electron microscopy /4/ which put the treatment of the single dislocation in covalent semiconductors on a more reliable base of knowledge. Since that time the number of researchers working in this field markedly increased and now the time might be appropriate to compare and to discuss the various approaches. The situa-

tion seems to be more complicated than even Hornstra's model suggested. I feel one possible reason for this could be an intimate intercorrelation between dislocations and point defects which is just coming into consideration.

2. Dislocations are dissociated into partials. - From electron microscopy using weak beam contrast since 1970 came the definite verification of former findings /5-7/ that by far most of the dislocations in silicon /8/, germanium /9/ and III-V compounds /10/ are split into two Shockley partials :

$$\frac{a}{2} [011] \rightarrow \frac{a}{6} [121] + \frac{a}{6} [\bar{1}12] \quad (1)$$

The nature of the stacking fault ribbon in most dislocations produced by glide proved to be intrinsic.⁽¹⁾ The doubling of {111} planes which converts the fcc structure into the diamond structure ($A \rightarrow A-a$) must be preserved if the tetrahedral coordination should be retained in the stacking faults. That means : a stacking fault of a reasonable energy is possible only between narrow {111} planes (aB). For that reason those dislocations which were thought to be more mobile cannot dissociate in a true sense.

Figure 2 shows a "ball and wire" model of a 60° dislocation of aB type. The 30°- and the edge-partial are to be seen at the left and at the right side of the stacking fault ribbon, respectively. Such a model presents not more than the topological situation after the shear displacement. In the cores of the partial dislocations one finds periodic rows of atoms the neighbourhood of which is no more tetrahedral. This fact results at first in a number of dangling bonds. It is one of the most important aims of theory and experiment to clarify how those topological dangling bonds minimize their energy. From the EPR spectra of deformed silicon crystals we conclude that in many cases where two atoms with dangling bonds stay opposite each other a covalent bond can be formed, the length and orientation of which differ from the tetrahedral structure. Those stretched and misoriented bonds will be called substituting bonds in the following. They are known to exist in radiation defects in silicon and were inserted by Hornstra /3/ in many of his drawings. The two electrons forming

⁽¹⁾ Some exceptions were found /11-13/. There are indications that the transition from the intrinsic dissociation to the extrinsic one is hindered by a free energy barrier which needs thermal activation to be overcome. So the predominance of intrinsic splitting is not necessarily due to a difference of the energies of intrinsic and extrinsic stacking faults, respectively. I will restrict myself here to intrinsic dissociations.

a substituting bond normally are spin paired and occupy energy levels above the valence band.

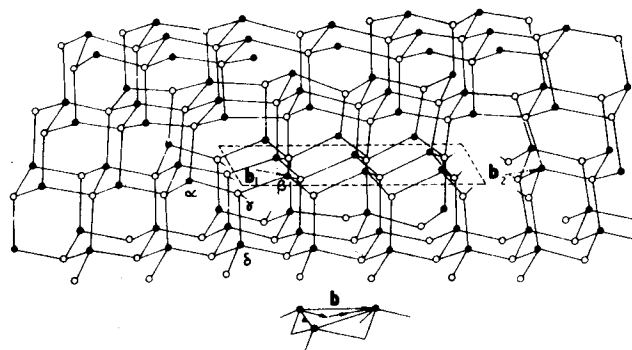


Fig. 2 : Ball and wire model of a split (aB) 60° dislocation.

In the core of the 90° partial dislocation two rows of broken bonds stand opposite to each other like two combs; they probably give rise to a linear chain of substituting bonds which are directed parallel to a $\langle 110 \rangle$ direction. In the core of the 30° partial dislocation the shear displacement of atom β produces a planar arrangement around the atom γ which has some similarity with a "molecule" bound by sp^2 hybrid bonds. As the four atoms α - δ bring in 7 valence electrons one of these should occupy the p_z orbital orthogonal to the sp^2 plane (i.e. parallel to the dislocation line). The distance between two half filled p_z orbitals being of the order of 0.4nm several possibilities of electronic rearrangement exist :

- 1) the γ atoms may be pairwise linked by a substituting covalent bond;
- 2) the unpaired electrons may be more or less delocalized, i.e. form a one dimensional half filled band of a certain width,
- 3) those electrons finally may form true localized dangling bonds the spins of which eventually may be coupled by direct or indirect exchange and dipole-dipole interaction.

The screw dislocation dissociates into two 30° partial dislocations (Fig. 3). There are strong indications that EPR spectroscopy gives information just about electrons in the core of these dislocations : Very recently E. Weber in our group succeeded by accumulation of spectra to find the hyperfine structure of the $S = \frac{1}{2}$ lines in the central part of the EPR-spectrum of dislocations in silicon /14/. It turned out, that the two lines belonging to each active glide system /15/ are due to electrons of mainly p-character with their orbitals orthogonal to the primary (P) and to the cross glide (Q) plane, respectively.

vely, of the dislocations. This symmetry shows, in our view, that the unpaired electrons causing this part of the spectrum are localized in the screw dislocations, i.e. in the core of 30° partials.

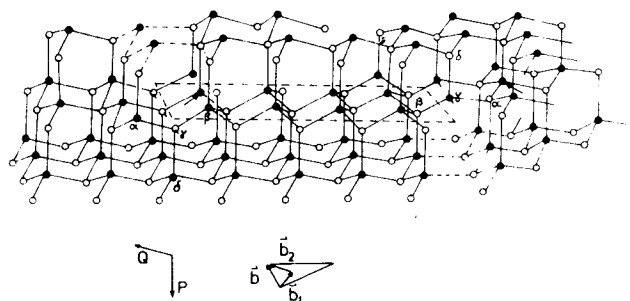


Fig. 3 : Ball and wire model of a split (aB) screw dislocation

P : normal on the primary glide plane
Q : normal on the cross glide plane

The remaining lines of the spectrum form a fine structure with its symmetry axis parallel to the Burgers vector /16,17/. This points to chains of unpaired but interacting spins parallel to \vec{b} , i.e. again the screw dislocations. Both spectra increase when the deformation is made under a high stress, enlarging the fraction of screw dislocations from a few percent to about one third. From these findings we tentatively conclude that true dangling bonds exist only in the screw dislocation which hitherto was believed to be the only dislocation type without dangling bonds /3/. The number of unpaired spins may be determined from our spectra not very exactly. It amounts to circa 10 % of the γ sites (Fig. 3) in screw dislocations. From this we conclude that pieces of 30° partials out of screw dislocations exist in at least three different states : a) those containing single spins, b) segments with two to five coupled spins, and finally c) pieces which have no dangling bonds. Transition may be induced from a) and b) to c) by annealing above 750° C (irreversibly) (R.Erdmann unpubl.) There remain several problems to be solved. Two of them I will mention : 1) Split 60° dislocations contain 30° partials too (Fig. 2). They seem to be free of dangling bonds. 2) The orientation of the p_z -orbitals in the core of the screw dislocation marks two out of three bond directions in the "sp² molecule". A future LCAO MO treatment should show if this is due to the asymmetry surrounding of the "molecule" or if half of the EPR active screw segments are split on the cross glide plane, which is difficult to believe.

Constructing ball and wire models of disloca-

tions being not parallel to a $\langle 110 \rangle$ direction generally shows the occurrence of the same topological elements as found in the 30° and 90° partials, respectively. So we may restrict our discussion to the screw and 60° dislocation.

3. Dislocations are split during their motion. - As the motion of a aB dislocation (dissociated or not) needs breaking of three times as much covalent bonds than does a A-a dislocation it was discussed for some time the carriers of plastic deformation could be A-a dislocations which produce a stacking fault ribbon on the neighbouring aB plane as soon and as long as they come to rest /18/. This ribbon should be bounded by two partial dislocations with opposite Burgers vectors. The concept of such an association of a perfect A-a dislocation D with a stacking fault was considered already by Hornstra /3/ and called by him "dissociation of the (A-a) dislocation" long before any experimental evidence of stacking faults connected with dislocations. As the driving force for the formation of the stacking fault ribbon should be the relaxation of the stress field near the core of the dislocation D the sum of the Burgers vectors of D and of the partial bounding the stacking fault on the side of D must equal the Burgers vector of the Shockley partial located at the same side in the case of splitting of a aB dislocation :

$$\frac{a}{6} [\bar{1}\bar{1}2] \xrightarrow{\text{stacking fault}} \frac{a}{6} [\bar{1}12] \quad (2)$$

$$D: \frac{a}{2} [011]$$

$$\text{sum} : \frac{a}{6} [121]$$

The resulting strain field of the two dislocations on the one side of the stacking fault is therefore always practically the same as that of the corresponding partial of a split (aB) dislocation. This is why it is impossible at present to decide between the two structures by means of the electron microscope.

During the last years several studies were made, however, which indicate that the stacking fault connected with the dislocation does not collapse when the dislocation is moving /19-21/. Meingast /19/ shot a film of partial dislocations in a thin foil of germanium moving forwards and backwards. Roehl (to be publ.) by contrast computation verified that Shockley splitting was under consideration. Similar observations are reported by Sato and Sumino /21/. The statement that dislocations move in the dissociated form does not mean, of course, that the dissociation width d should be independent of the state of motion.

Wessel and Alexander /13/ investigated dislocations in silicon which were frozen in under rather high stresses ($150-350 \frac{\text{N}}{\text{mm}^2}$), i.e. in the state of (slow) motion. They found all dislocations parallel to $\langle 110 \rangle$ directions (Peierls valleys) and without any constricted points or segments. The measured values of d were not in agreement with those calculated merely from the forces working on the individual partials. The results led to the conclusion that the lattice friction depends on the character of the partials and on its position before or behind the stacking fault. Gottschalk will report on new results later on in this conference. A special feature observed at high stress is the formation of so called "noses"; these are sites where the two partials are completely separated. In our context it seems worthwhile to note that always the trailing partial (which follows the stacking fault) is rigorously bound to $\langle 110 \rangle$ directions whereas the leading one is not /13/ (Fig. 4).

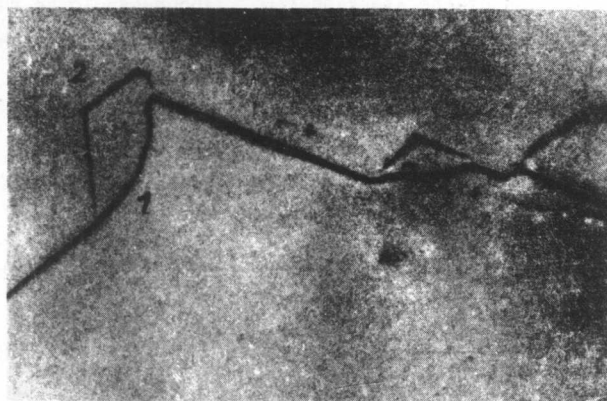


Fig. 4 : Silicon, Two noses in a dislocation frozen in under high stress

($\tau = 350 \frac{\text{N}}{\text{mm}^2}$, $T_{\text{def}} = 420^\circ \text{C}$)

1.2 : leading and trailing partial dislocation, respectively.

So there must be a principal difference between the trailing and the leading partial even in the case of two 30° partials forming a screw dislocation.

Knowing that the motion of a dislocation in Si and Ge does not need a collapse of the stacking fault, the original reason for thinking about the association ceased. But there are several other experimental results which seem to be easier to understand if one assumes that part of the dislocations might exist not simply as dissociated aB dislocation but as an association. Before discussing these observations I

should like to extend somewhat the concept of the association. Hirth and Lothe /22/ probably were the first to point out that the association of a 60° (A-a) dislocation with a stacking fault ribbon (called "shuffle set" dislocation by the authors) can be interpreted as a normal dissociated (aB) dislocation ("glide set" dislocation in /22/) which has absorbed a linear chain of point defects in the core of one of its partials. This row of vacancies (or interstitials) may formally be represented by a vertical dipole⁽²⁾ of 60° dislocations $\pm \frac{a}{2} \langle 011 \rangle$:

$$\begin{aligned} & \begin{array}{c} B \text{ --- } \frac{a}{6} [121] \text{ --- } \frac{a}{6} [\bar{1}12] \longrightarrow \\ a \text{ --- } \end{array} \\ & \left\{ \begin{array}{c} B \text{ --- } \frac{a}{2} [0\bar{1}\bar{1}] + \frac{a}{6} [121] \text{ --- } \frac{a}{6} [\bar{1}12] \\ a \text{ --- } \frac{a}{2} [011] \\ A \text{ --- } \end{array} \right. \quad (3) \\ & \equiv \left\{ \begin{array}{c} B \text{ --- } \frac{a}{6} [1\bar{1}\bar{2}] \text{ --- } \frac{a}{6} [\bar{1}12] \\ a \text{ --- } \frac{a}{2} [011] \\ A \text{ --- } \end{array} \right. \end{aligned}$$

The results is the same as in process (2) but the interpretation is different : the carriers of glide now are split (aB) dislocations which may or may not introduce into one of their partials point defects in a correlated manner⁽³⁾.

Depending on the Burgers vector of the vertical dipole the density of point defects may be varied in certain limits. Especially the split screw dislocation may absorb point defects too, which form a 60° dipole.

The transition from a split (aB) dislocation into an association is now recognized as an elementary step of climb. It may take place (weakly correlated) along limited pieces of a dislocation by the movement of a half jog along the dislocation piece.

During the last few years many results were published which indicate that a remarkable density of point defects (clusters) is produced by plastic deformation in silicon /24,25/ and germanium /26/. The concentration of EPR active point defect centres is proportional to the shear strain after deformation at 650°C and exhibits a complex dependence on

⁽²⁾ Inverting the dipole $\pm \frac{a}{2} \langle 011 \rangle$ results in a row of interstitials instead of vacancies.

⁽³⁾ The motion of such an association of a split (aB) dislocation with a row of point defects is known as shuffling /22/. Obviously this special kind of a zonal dislocation moves without difficulties from a geometrical point of view /23/, but the transport of the core region of modified density is equivalent with a component of diffusion.

the deformation temperature and shear strain rate (Erdmann unpubl.). The number of dislocation cutting processes seems to play no rôle /24/. Taking all these observations together we conclude that dislocations may loose point defects during their motion. This can be imaged more easily if one assumes that at least part of the dislocations exist in the form of an association. The process of transition of dislocation segments between the states (split aB dislocation and association, respectively) may then work as a source of point defects in the following way: possibly resting aB dislocations turn into associations producing the corresponding number of pairs of point defects by lowering their stress energy. The assumption seems reasonable that those associations have a lower mobility than pure aB dislocations. So below a critical temperature a shear stress of sufficient magnitude may separate the dislocation from its point defects, restoring its full stress field. This process may repeat when the dislocation comes to rest again. A theory of the mobility of such dislocations has to take into account the temperature and stress dependence of the binding force between point defects and dislocations. Observations concerning the dependence of the electrical /26,27/ properties of dislocations on the deformation temperature point to the fact that the mentioned critical temperature is about 400° C in Ge.

Another interesting argument for the prevailing of associations is put forward by Holt (to be publ.). He reinterprets results of Bell and coworkers /28/. These authors investigated the polarity of edge type dislocations in InSb crystals bent in opposite directions. They concluded (1965) that the polarity was in agreement with that to be expected when (A-a) dislocations produced the bending. Now, as we know that dislocations are split in InSb too /10/ this argument points to associations, because the most distorted sites in the core of split aB dislocations are occupied by In ions when the dangling bond ions in A-a dislocations are Sb and vice versa. (comp. Fig. 2).

In conclusion I want to report on a new determination of the stacking fault energies of Ge and Si which turned out to be necessary because the values accepted hitherto ($\gamma_{\text{Ge}} = 60 \pm 8 \text{ mJ/m}^2$ and $\gamma_{\text{Si}} = 51 \pm 5 \text{ mJ/m}^2$ /8/) resulted in absurd ratios α of mobilities of the partial dislocations in germanium (Eppenstein unpubl.) and doped silicon (Wendler unpubl.). Table I gives the values for γ published so far. There is a striking tendency to increasing values with time and a remarkable scatter in each determination itself. We evaluate $\gamma(\text{Ge}) = 75 \pm 10 \text{ mJ/m}^2$, $\gamma(\text{Si}) = 58 \pm 6 \text{ mJ/m}^2$ /29/. I feel there is no doubt that $\gamma(\text{Ge})$ is larger than $\gamma(\text{Si})$. The evaluation of α turned out to be a sensible test for the stacking fault energy which was used.

Table I: Measurements of the energy of intrinsic stacking faults

1) <u>Germanium</u>					
Type of dislocation	Dissociation width d(nm)	Stacking fault energy γ ($\frac{\text{mJ}}{\text{m}^2}$)	Ref.	Remarks	
Edge	5.4 \pm 1.1	(63 \pm 13)*	/9/		
Edge-60°		60 \pm 8	/30/		
Screw	2.2 \pm 0.8		/31/		
Edge	4.8 \pm 0.6	(68)*			
60°	3.0 \pm 0.3	100 \pm 10	/32/	Lattice image	
Screw		75	/33/	from cross glide experiments	
Edge-60°		75 \pm 10	this work		
2) <u>Silicon</u>					
Edge-60°		51 \pm 5	/8/		
Edge-screw		58 \pm 6	this work		

* our evaluation

Acknowledgment. The author wants to thank his coworkers, especially Dr. H. Gottschalk and Dr. E. Weber for many fruitful discussions. Dipl. Phys. R. Erdmann made unpublished results available.

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LATTICE IMAGES OF DISLOCATIONS IN GERMANIUM

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Résumé.— Des sous-joints de flexion pure dans le germanium ont été observés par microscopie électronique à haute résolution. Ces joints contiennent des dislocations coins non dissociées et des dislocations dissociées en partielles. Une énergie de faute intrinsèque relativement élevée est déduite si l'on suppose que les partielles ne tournent pas durant l'amincissement. Cependant aucune preuve expérimentale ne peut encore être fournie à l'appui de cette hypothèse.

Abstract.— Pure tilt subgrain boundaries in germanium have been studied by high resolution electron microscopy. They contain mainly undissociated edge dislocation and two types of split dislocations. A relatively high intrinsic stacking fault energy is deduced by supposing that no rotation of partials occurs during thinning. No experimental proof has yet been obtained to support this hypothesis.

1. Introduction.— Until recently it was supposed to be very difficult to interpret the lattice images of dislocations as obtained by electron microscopy. However a new development of this technique has begun due to a better understanding of imaging conditions and a good control of the defect geometry. The imaging mode (axial illumination) as applied down to a 3 \AA periodicity give an image directly related to the projected structure of the object in given conditions of thickness and of defocussing distance (this is specially true with high voltage instrument). For dislocations or any linear defect, an easy-to-read image is obtainable when the dislocation is strictly seen end-on. The small angle grain boundary produced in a bicrystal is composed of dislocations whose Burgers vector and direction are easy to control.

Germanium bicrystals with a $[011]$ common axis have been by the Czochralski method in a graphite crucible under a pure argon atmosphere. The germanium has a $40 \text{ } \Omega\text{cm}$ resistivity and is p-type. Metallic impurities as measured by activation analysis are of the order of 10^{-2} ppm. The oxygen and carbon content has not yet been measured : techniques are being developed to measure these since the carbon whose segregation is important, may influence dislocation splitting.

This paper reports the results actually obtained about the structural aspects of dislocations in subgrain boundary, and stresses the problems which remain unsolved.

2. Dislocation structure in subgrain.— The subgrain boundary structure has shown a great complexity in the dislocation arrangement /1/ (Fig. 1) possibly

due to splitting and stabilization of unusual defects by mutual interaction in the grain boundary plane. Three main types of dislocations will be discussed here.

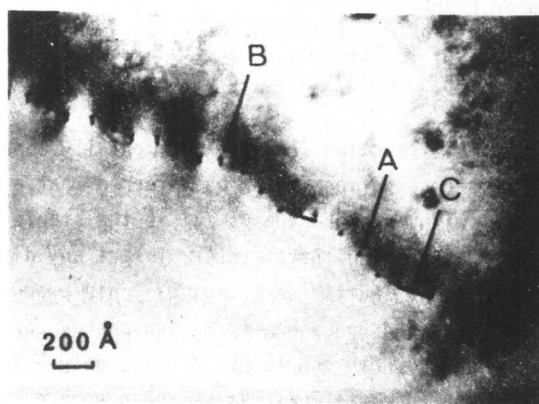


Fig. 1 : Part of a pure tilt $[011]$ grain boundary in germanium (tilt angle : $2^\circ 5'$) when viewed end-on along the $[011]$ common axis. Dislocation cores appear as black dots and stacking fault as straight segments joining cores. Three types of dislocation are recognizable : (A), non dissociated dislocation; (B), dislocation split into partials perpendicular to the grain boundary plane ; (C), three grouped dislocations separated by stacking faults. Bright field multibeam imaging mode, $E = 100 \text{ keV}$.

2.1. Pure edge dislocation (Fig. 2 A).— This dislocation has a Burgers vector $1/2 [011]$. It is Lomer dislocation as its direction contains two $\{111\}$ planes. The glide plane is (100) and therefore this type of dislocation cannot be produced in the early stages of plastic deformation.

The high resolution image taken at 500 keV shows clearly that no dissociation occurs ; furthermore a region of high contrast occurs in the dislocation core. Elasticity theory would predict a dissociated

form with a sessible configuration for this dislocation. The expected reaction scheme is :

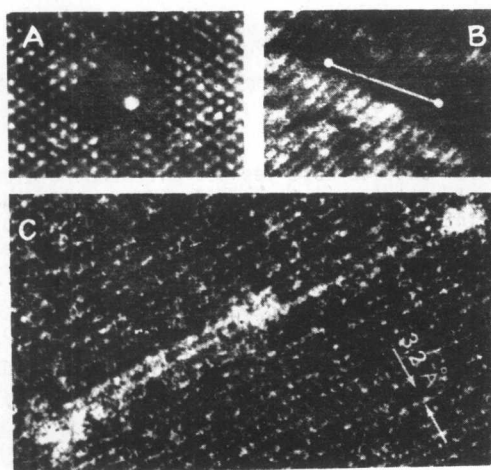
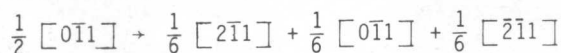


Fig. 2 : High resolution images of different type of defects in germanium : (A), edge dislocation ; (B) 60° dislocation split into two Shockley partials boarding an intrinsic fault ; (C), three identically grouped Frank dislocations separated by an intrinsic and an extrinsic stacking fault. (A) and (C) taken at 500 keV ; (B) taken at 100 keV. Axial illumination, 5 beam imaging conditions including four (111) diffraction spots.

In fact this reaction is not experimentally observed. From this observation it can be inferred that a shuffle set with no broken bonds /2/ is the stable form of this dislocation. As a matter of fact any dissociation of the shuffle set (Fig. 3) would produce broken bonds and consequently a high energy core situation which would counterbalance the decreasing elastic energy term. Furthermore the presence of a seven ring and a five ring in this type of structure could produce a bright spot in the empty tunnel and a dark area in the compact region as observed experimentally : however this direct identification although already suggested by Krivanek /3/ must be checked by image matching. It is worth noting that similar non dissociated edge dislocations have been obtained in wurtzite structure /4/.

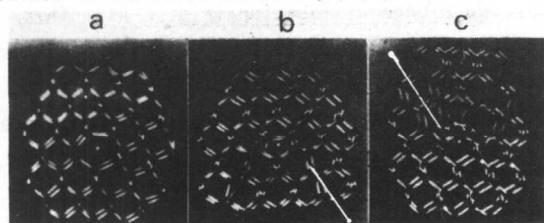


Fig. 3 : Different atomic models of the core of an edge dislocation along $[011]$. a) shuffle configuration, b) if dissociated with two intrinsic faults, the number of open bonds is 2, c) if dissociated with two extrinsic faults the number of open bonds is 6. In fact only the configuration a) is observed

2.2. 60° dislocation (Fig. 2 B).— Imaging this dislocation by two different $\{111\}$ atomic planes, one extra half plane is visible on only one set with a distinct stacking fault and no discontinuity on the other set. Thus the Burgers vector must be of the type $1/2 \langle 101 \rangle$ and these dislocations are of 60° type. They are dissociated into two partials giving an edge Shockley dislocation and a 30° Schockley dislocation. The two partials are visible even at medium range resolution when observed end-on with bright field multibeam condition (Fig. 4a). The stacking fault character is always intrinsic. This character is determined either from the high resolution image, the 30° dislocation giving rise to a smooth discontinuity in contrast to the edge partial, or from weak beam images obtained after a large specimen tilt.

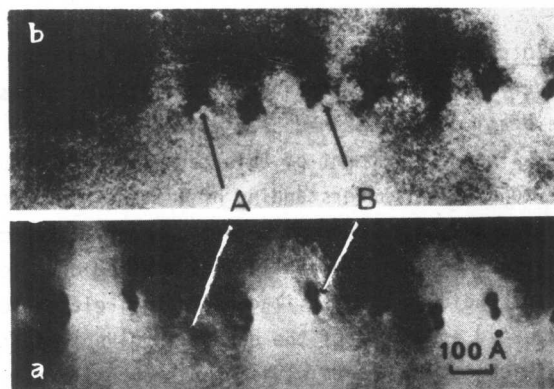


Fig. 4 : Testing the parallelism of 60° dislocation in respect to the $[011]$ direction. a) Bright field multibeam imaging in a 800 Å thick region : the two dark points indicate a very small rotation of the partials when viewed along the $[011]$ direction. b) Bright field multibeam imaging after a 3° rotation of the specimen away from the $[011]$ direction. The stacking fault is visible and is projected as a lozenge-shape (B) whose edges are slightly rotated in respect to the edge dislocation (A). The rotation angle is now measurable and is approximately $\pm 1^\circ$

The splitting distance as deduced from atomic plane imaging gives an apparent distance of 31 Å between partials. This gives an intrinsic stacking fault energy of 100 ergs/cm². This relatively higher value than that obtained by weak beam /5/ can be attributed to different impurity content or to the method of measurement itself (for discussion of the surface effect see below).

It has not yet been possible to distinguish the glide set and the shuffle set /6/ which might appear on the edge partial : however this may be possible if the partials remain strictly parallel to the $[011]$ direction even in a thin foil.

2.3. Group of Frank dislocations (Fig. 2c). - One arrangement has been isolated on figure 2c : it includes three (111) atomic planes which stop at a Frank dislocation giving successively an intrinsic and an extrinsic stacking fault. The splitting occurs in the grain boundary plane contrary to the 60° dislocation which splits perpendicular to the grain boundary plane. This dislocation arrangement is entirely edge in character and completely sessile : it accommodates misorientation between the two adjacent grains.

The splitting distance between Frank partials is not governed by the stacking fault energy angle of misorientation (1-3°), but rather by the distance between defects.

Another grouping not so frequently encountered, results from the following decomposition on two (111) planes :

$$\frac{1}{2} [\bar{2}1\bar{1}] + \frac{1}{6} [\bar{2}1\bar{1}] + \frac{1}{3} [\bar{1}1\bar{1}] + \frac{1}{3} [\bar{1}00]$$

producing always an intrinsic fault cutting an extrinsic one at an acute angle.

These peculiar dislocation groupings are probably specific to tilt subgrain boundary and have never been observed in deformed material. However they give an opportunity to produce controlled intrinsic and extrinsic stacking faults.

3. Influence of the surface of the foil. - The observation of linear defects by high resolution microscopy is easy if the lines run strictly parallel to the observation direction. In order to fulfil this requirement the $\langle 011 \rangle$ direction seems to be the most appropriate as even during thinning the dislocations would have a tendency to stay and lie along throughs of the Peierls potential. However this assumption must be checked carefully. For the defect whose character is essentially sessile it is likely that no rotation during thinning would occur : such displacement would involve climbing which is excluded at room temperature. Therefore all the defects formed with Frank partials do not move. The same argument can be applied to the edge dislocation which cannot glide on the (001) plane. The question remains open for the 60° dislocation only.

Small misalignment of the partials in respect to the $[011]$ axis is very difficult to detect in a very thin region (100 Å). Actually our check is limited to 500-1000 Å thick regions (Fig. 4) : in this range the rotation of 60° dislocation is limited to $\pm 1^\circ$ for dislocations 100 Å apart from each other. Rotations seem to be alternately in one sense

and in the other as the screw components of the 60° dislocations change sign. This misorientation would give a negligible effect in a 100 Å thick region. However this small rotation could have been limited by the presence of other segments of dislocation along the grain boundary. A crude estimate of this effect using the Hazzledine approach with no friction force /7/ shows that rotation is negligible if and only if the distance between the dislocations is smaller than half the thickness of the foil. Actually this condition is certainly not fulfilled for a very low angle grain boundary (0.5°) and small thickness where the splitting distance has been measured. Further experimental work is needed to clarify this point.

4. Conclusion. - The study of lattice defects in germanium by high resolution electron microscopy has been fruitful : Burgers vector, character of stacking fault, and analysis of complicated structures existing in small angle grain boundaries have been easily studied. However direct interpretation is actually limited to strictly end-on linear defect. This condition is verified for sessile configurations but has to be verified experimentally for glissile defects in thin foils : some efforts are necessary to find a practical way of doing this at a high resolution level.

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DISLOCATIONS IN SEMICONDUCTORS AS STUDIED BY WEAK-BEAM ELECTRON MICROSCOPY

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Résumé.— On discute la configuration des dislocations observées dans les semiconducteurs à l'aide de la technique du faisceau faible en microscopie électronique. La dissociation de ces dislocations est bien avérée. Plusieurs problèmes relatifs à la géométrie de ces dislocations sont bien résolus. Par contre les observations révèlent l'existence de nouveaux problèmes géométriques non encore résolus.

Abstract.— The configuration of dislocations in semiconductors as revealed by the weak-beam method of electron microscopy is discussed. The dissociation of these dislocations has been clearly established, but while many earlier questions concerning the dislocation geometries have been resolved, new dislocation geometries have been revealed which are as yet unexplained.

1. Introduction.— While the weak-beam method of electron microscopy /1/ has been applied to the study of many types of defect in many different materials, its application to dislocations in semiconductors has been one of the most fruitful areas. But although it has provided answers to many long-standing questions about dislocation geometries, others remain unresolved, and new questions have arisen. For example the dissociation of dislocations in silicon and germanium is no longer in dispute (which means that many theoretical analyses based upon undissociated dislocations must now be reconsidered), but whether or not these dislocations are of the glide set or the shuffle set remains unresolved. At the same time detailed weak-beam studies of these dislocations have revealed many properties which are yet to be understood.

In this paper, the properties of dislocations in semiconductors revealed by weak-beam electron microscopy are reviewed, and recent results presented. The data from these studies are compared with data from other electron microscope techniques, and in particular that available from the lattice fringe technique. Deficiencies in the present electron microscope techniques for providing information about the geometry of dislocations below 2nm are discussed, and attempts to overcome these deficiencies are outlined.

2. Principles of the weak-beam method.— The principles of the weak-beam method and its practice are now well established for defect detail greater than 2nm (for a discussion of studies of defect detail below 2nm see § 6). Detailed descriptions of the method had been given from theoretical /2,3,4/ and

experimental /5,6/ points of view. The last of these references /6/ can be considered as a companion to this paper. The influence of beam divergence upon the images has been investigated /7,8/, as has the effect of non-systematic reflections /9/. Several applications of the weak-beam method at high voltages have been reported, and some attention has been given to the theory of its use at such voltages /10/; however it would seem that further theoretical studies would be useful to establish the optimum conditions for high voltage applications.

Because of these published treatments, it will suffice to give here an outline of the technique as a basis for further discussion (for a more detailed description of the method, refer to /5/ and /6/). The principle of the method is as follows: a Bragg reflection g is chosen, and the perfect crystal is oriented to be far from satisfying the Bragg condition for this reflection. Under these conditions, a dark-field image of the perfect crystal formed with the reflection g shows only weak intensity because, in this orientation, the perfect crystal scatters only weakly into the reflection g . However if there is some small volume in the crystal where the lattice is oriented (e.g. by lattice strain) into or near to the Bragg angle for the reflection g , then this region will produce increased scattering resulting in increased intensity in the image. Confirmation of this idea is given by images calculated for a variety of defect geometries, and an example is shown in figure 1 for the case of an undissociated dislocation.

It is evident from figure 1 that the image lies to one side of the dislocation core. If the