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A detailed fractographic analysis of cleavage steps in silicon

M. J. KAUFMAN, A. J. FORTY*
National Bureau of Standards, Gaithersburg, Maryland 20899, USA

A detailed analysis of cleavage steps present on fracture surfaces in pure silicon has been carried out using scanning electron microscopy. The results indicate that the mechanisms involved in both the formation of unfractured ligaments, produced when adjacent cleavage facets overlap, and the subsequent fracture of these ligaments to form cleavage steps, are quite complex. Specifically it is shown that, during ligament formation, the local crack fronts are deflected from their preferred (1 1 1) cleavage plane and that the fracture of these ligaments to form cleavage steps occurs in a very complex fashion producing very small microcleavage steps. It is shown that these latter steps are consistent with cleavage along both {1 1 1} and {0 1 1} planes.

1. Introduction

The propagation of cleavage cracks in a wide range of solids has been the subject of a number of previous investigations. Such behaviour occurs in very brittle materials (e.g. glass, diamond, silicon, germanium), in materials with limited room temperature ductility (e.g. NaCl, LiF, zinc), and also in some very ductile alloys under extreme conditions (e.g. [1]). Because cleavage fractures are never atomically smooth, it is important to understand their "roughness" with respect to the proposed behaviour of brittle cracking. Evident on essentially all such surfaces and responsible for this so-called roughness are cleavage steps which result when the cleavage facets, comprising the total crack and propagating on parallel, but displaced crystallographic planes (in crystalline materials), join together in a stepped fashion. These facets have been shown to overlap [2, 3] and form unfractured ligaments prior to cleavage step formation (Fig. 1). In many, if not all, materials, it is these unfractured ligaments which are believed to hinder the overall rate of crack propagation since they most likely trail behind the average crack front and become increasingly load bearing as their lag distance increases [4]. This is especially true for materials where the mechanism for fracture of the ligaments is not cleavage as is frequently the case in the more ductile alloys [4]. Thus, the energy expended by the fracture of these ligaments may, in many instances, be a large portion of the total fracture energy [5, 6].

Because of the very significant role that the unfractured ligaments are believed to play in cleavage crack propagation, it is useful to gain a better understanding of their behaviour in brittle solids. In the present paper, the nature of the cleavage steps present on the fracture surfaces of high purity silicon has been examined using scanning electron microscopy. It was felt that the information obtained from observations made on a highly brittle material such as silicon might be useful for gaining a better understanding of the mechanisms of cleavage fracture in the more ductile materials, mentioned above, where not only are the fractures more complex but where the knowledge concerning the mechanisms of cracking is presently inadequate.

1.1. Previous work on silicon-like materials

Silicon is a material of significant current interest in the semiconductor device industry; for this reason, a number of investigators have examined cleaved silicon surfaces in order to understand the precise nature of the electronic properties and the relationship of these properties to surface condition and possible subsidiary deformation. For example, Frankl [7] noted a greater density of dislocations in the immediate vicinity of cleavage steps on silicon {1 1 1} fracture surfaces and concluded that highly localized plastic deformation accompanied the cleavage step formation process. Similar conclusions were drawn by Noble and Henisch in their study of cleaved germanium crystals [8]. In both studies, the subsidiary deformation was correlated with the density of surface states. However, based on the difficulty of dislocation glide processes in silicon and germanium at room temperature, Swain *et al.* [2] proposed that the high density of dislocations, concentrated at cleavage steps in silicon-like materials, was a direct consequence of the local disregistry associated with the crack undercutting along cleavage steps and the subsequent partial healing upon release of the load rather than the local plasticity arguments proposed previously. This view is supported by some early work of Pugh and Samuels [9].

In order to gain insight into the cleavage step formation process, Swain *et al.* [2] analysed, using fracture mechanics, the local forces between two edge cracks approaching each other in a solid. These forces,

**Permanent address*: University of Warwick, Coventry, CV4 7AL, UK.

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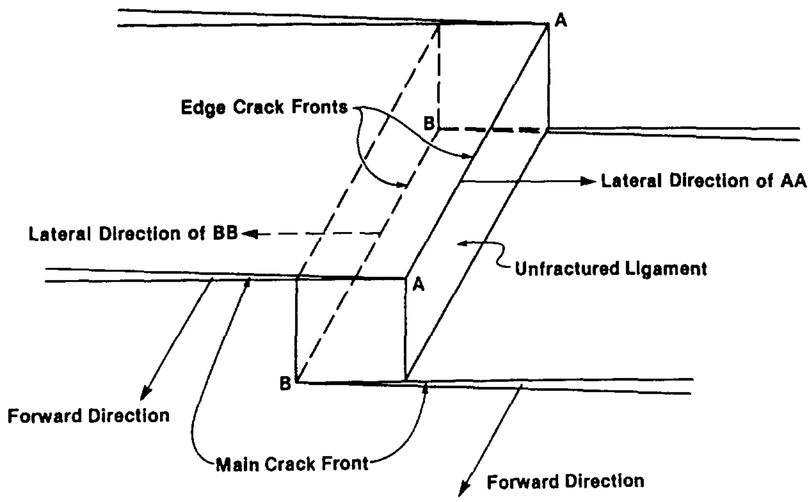

Figure 1 Schematic representation of the geometry of neighbouring cleavage cracks which overlap to form the unfractured ligaments.

they believed, could be correlated directly with the behaviour of neighbouring cleavage facets propagating on parallel planes (Fig. 1). It is the lateral movement of these facets (which Swain *et al.* considered to be edge cracks) which results in possible crack overlap (ligament formation) and subsequent cleavage step formation. In their analysis, they predicted that the forces developed between two such edge cracks upon approaching one another on parallel, slightly displaced planes would be mutually repulsive initially and then become attractive at the point of overlap (Fig. 1). From their calculations, it was determined that the magnitude of the repulsive forces in brittle materials with highly anisotropic surface energies such as silicon would be insufficient to cause the cracks to deviate from their preferred crystallographic cleavage plane. However, the mutual attractive forces were estimated to be sufficient to cause overlap prior to formation of the cleavage step. Their experimental observations of significant overlap and undercutting (schematically illustrated in Fig. 2) directly supported the cleavage crack overlap prediction indicating that the previous models [10, 11] describing step formation were probably inaccurate. Swain *et al.* also noted the presence of transverse (normal to the cleavage step) step-like markings on the cleavage steps in silicon but failed to relate them to the step formation process. The significance of these transverse markings as well as other microscopic features of the cleavage steps are discussed in the present paper.

2. Observations

Large-grained specimens of reasonably pure silicon (99.999%) were oriented randomly and tapped with a hammer and chisel to cause cleavage fracture. Various such cleaved surfaces were analysed by scanning electron microscopy (SEM). A typical cleavage fracture surface of pure silicon is shown at low magnification in Fig. 3. Apparent on this surface are cleavage steps forming a characteristic "river pattern." A large number of these steps were examined in the SEM in order to gain confidence in the prevalence of the various features described below.

Before proceeding with the present results, it is informative initially to consider an area similar to that in Fig. 5 in the paper by Swain *et al.* [2]. Fig. 4 shows one such area and it can be seen that the parallel facets do overlap and that the direction of ligament fracture switches suddenly in agreement with the observations of the previous authors (Fig. 2). Additional features evident in Fig. 4 include the transverse step-like markings mentioned above and "troughs" lying parallel to the cleavage step. These troughs were also present on the silicon fracture surfaces examined by Swain *et al.*; however, no mention was made by those authors concerning their presence or importance. Possible origins of these trough-like features will be suggested below.

A large portion of the present analysis was performed on a representative region where two relatively small cleavage steps combined to form one larger step

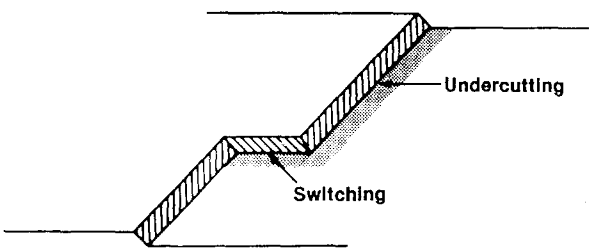

Figure 2 Schematic illustration of a typical cleavage step in pure silicon where the undercutting resulting from crack overlap is illustrated.

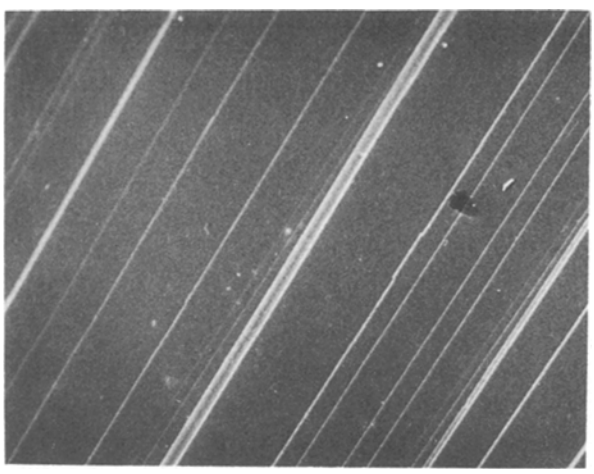

Figure 3 Low magnification scanning electron micrograph (SEM) of a typical cleavage surface in pure silicon. × 250.

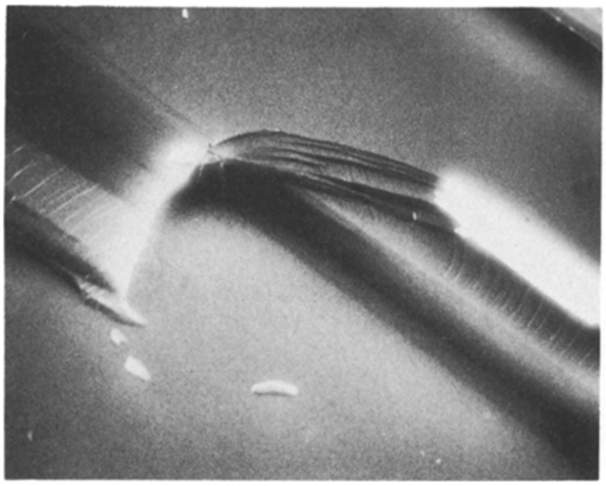

Figure 4 SEM of a cleavage step in pure silicon. Note the existence of the transverse step-like markings and the trough running parallel to the step. × 1575.

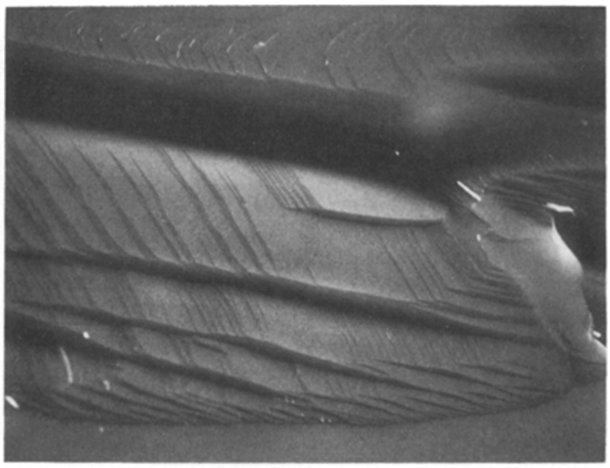

Figure 6 Higher magnification SEM of the area in Fig. 5 displaying the complex nature of the cleavage step (see text). × 2450.

(Fig. 5). At higher magnification (Fig. 6), the same features (transverse steps and troughs) noted in Fig. 4 are apparent. However, two additional features are evident in Fig. 6. Firstly, there exist small, frequently discontinuous, steps approximately parallel to the cleavage step in the vicinity of the transverse steps. Secondly, apparent on the upper cleavage surface are additional transverse step-like features. When these latter features were examined more closely using stereomicroscopy (Fig. 7), it became evident that they originated from the relatively flat cleavage facet in the vicinity of crack overlap.

In an effort to ascertain both the crystallographic nature and the possible mechanisms for production of the various steps in Fig. 6, the area was examined using selected-area channelling. By observing selected-area channelling patterns (SACPs) the fracture surface was aligned such that the macroscopic $\langle 1\,1\,1 \rangle$ cleavage plane normal was parallel to the optic axis of the SEM (Fig. 8). Subsequently, the $\langle 1\,1\,1 \rangle$ SACP was positioned carefully with respect to a corresponding image taken at the same orientation and the results are as indicated schematically in Fig. 9. From this analysis, it becomes apparent that the direction of crack propagation in this specific region is close to $[0\,1\,\bar{1}]$ while the transverse markings lie approximately along $[2\,1\,\bar{1}]$. Thus, it seems likely that the transverse steps within the cleavage step were generated by fracture on both the primary $(1\,1\,1)$ cleavage plane and a secondary plane of the $[2\,\bar{1}\,\bar{1}]$ zone (i.e. $(0\,1\,\bar{1})$, $(1\,0\,2)$, $(2\,1\,3)$, etc.). Similarly, the small, discontinuous steps most likely were generated by fracture along a plane of the $[0\,\bar{1}\,1]$ zone (i.e. $(\bar{1}\,1\,1)$, $(0\,1\,1)$, $(1\,0\,0)$, etc.). When these two types of features were examined at higher magnifications, it became apparent that the fracture of ligaments to form cleavage steps was even more complex; for example, the transverse steps frequently are comprised of three, rather than two, crystallographic planes (Fig. 10). By again utilizing stereomicroscopy it was possible to construct a three-dimensional description of the cleavage steps in this region as depicted in Fig. 11.

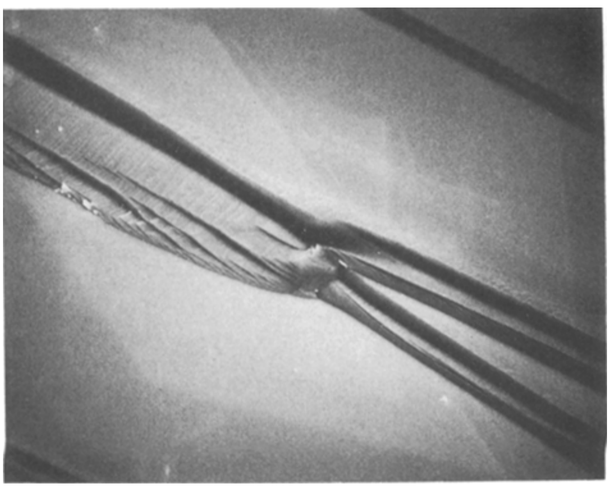

Figure 5 SEM of a region where two small cleavage steps have combined to form one larger step. × 1400.

Another feature worthy of comment in Fig. 10 is the greater density of the small transverse steps on the lower side of the discontinuous, longitudinal step. Similar regions were analysed at various orientations relative to the beam and it became apparent that the local crack front, upon propagating through the ligament in a direction nearly parallel to the small transverse steps, suffered sudden interruptions resulting in the small, discontinuous longitudinal steps. As a result of these arrest-like interruptions and accompanying displacements, the transverse steps apparently had to be reinitiated in a manner very similar in appearance to the initiation of the transverse steps on the upper surface in Fig. 7.

3. Discussion

The present observations are consistent with those of previous authors and confirm that fracture in pure silicon occurs by $\{1\,1\,1\}$ cleavage. Lawn [12] has shown that these planes have the lowest surface energy and, therefore, require the expenditure of the smallest amount of energy during fracture. However, from the detailed microscopic observations, it is apparent that the fracture of silicon to form cleavage steps is much more complex than previously suspected. The main observations from this study will be addressed separately below.

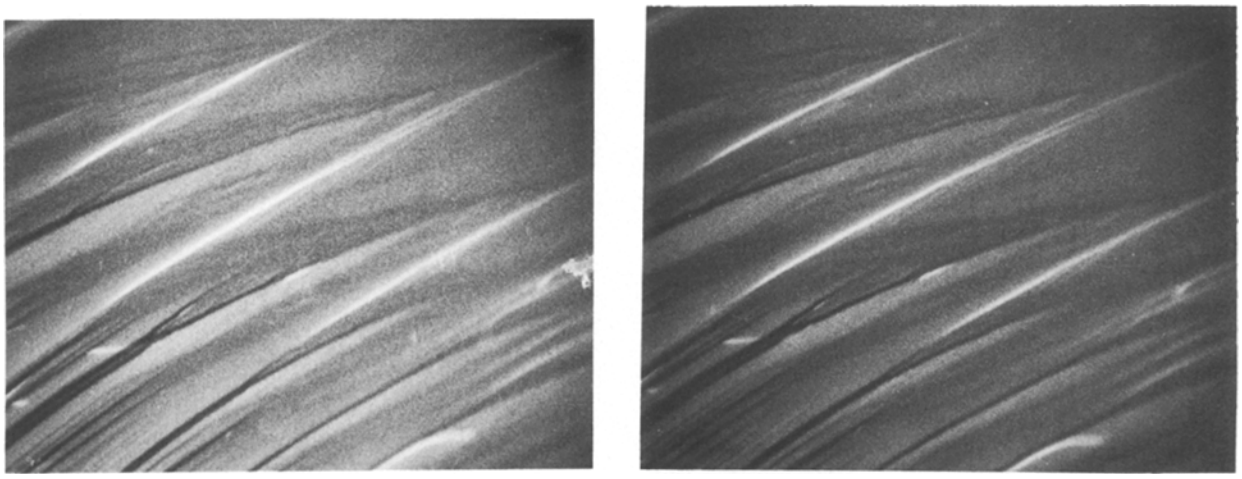

Figure 7 Stereo pairs taken of the upper surface of the large step in Figs 5 and 6. Note the transverse step-like features apparent on this surface. × 1400.

3.1. Crack deflection

As noted in Section 1.2, Swain *et al.* [2] calculated the forces present when two parallel, but displaced edge cracks approach one another as in Fig. 1. From their analysis, they proposed that the initial repulsive forces, indicated by the ratio of stress intensity factors, K_{II}/K_I, where the I and II correspond to Mode I and Mode II loading, respectively, should be of insufficient magnitude to cause any detectable deflection. However, the present observations (Figs 5 to 7) would seem to contradict their predictions, i.e. the cracks are deflected as a result of the repulsive forces which are generated.

It is of interest to discuss possible mechanisms for the deflection of the cracks from their preferred {1 1 1} orientation. As is evident in Fig. 7 the apparently flat cleavage surface becomes disrupted in the vicinity of crack overlap and transverse steps are generated on the fracture surface. The initial deviation from the planar {1 1 1} cleavage produces V-shaped markings; similar markings were produced in glass by Sommer [13] by simply altering the mode of loading from pure tension to tension plus a small amount of torsion. As noted by Sommer, the torsion load effectively altered the direction of maximum tension and the crack correspondingly changed its path in order to reorient itself normal to this new direction. As a result of this crack reorientation, the fracture surface displayed markings, termed "lances" by Sommer, which appear very similar to the V-shaped markings in Fig. 7, though on a much more macroscopic scale. Therefore, the initial deviation from {1 1 1} cleavage in the present study appears to have been caused by the change in the direction of maximum tension corresponding to the repulsive interaction between the overlapping facets. Obviously, the geometry in the present case is quite different from that produced by Sommer, but the result is similar in that the crack indeed deviates to maintain a desirable geometry relative to the direction of principal stress.

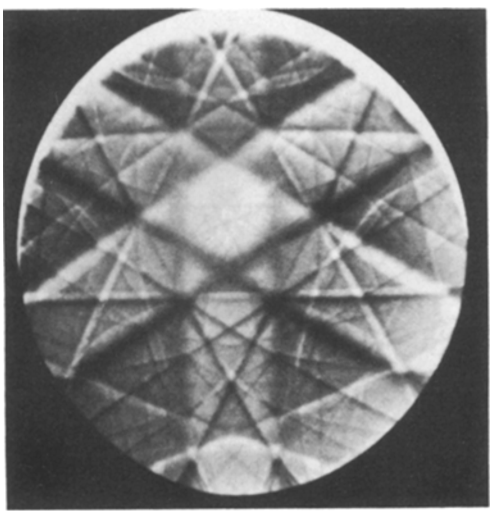

Figure 8 [1 1 1] selected-area channelling pattern obtained from the cleavage surface used to orient the various directions within the plane.

3.2. Cleavage step formation

From Fig. 7 it also may be noted that, after the initial deflection process described above, the crack bends back over, i.e. the interaction force becomes attractive in agreement with the predictions by Swain *et al.* [2]. Further support of their predictions is given in Fig. 4 (and indicated schematically in Fig. 2) where the overlapping nature and undercutting of the cleavage step is apparent. However, it is useful to consider the processes involved in the actual formation of a cleavage step which, as noted above, is much more complicated than previously suggested. An accurate description of these processes should account for the various features observed, i.e. transverse and discontinuous longitudinal steps as well as the troughs parallel to the actual cleavage steps.

Since the behaviour of the lower crack front in the vicinity of overlap should be essentially identical to that of the upper front, at least prior to the onset of ligament fracture, then the deflection arguments described above must hold for this lower surface also. Thus, in order to identify the origin of the troughs, it is useful to discuss the fracture behaviour on both sides of the step. For example, by considering the structural details of the upper side of the cleavage step in Fig. 6, it becomes evident that the fracture is comprised of an initial, relatively large, flat

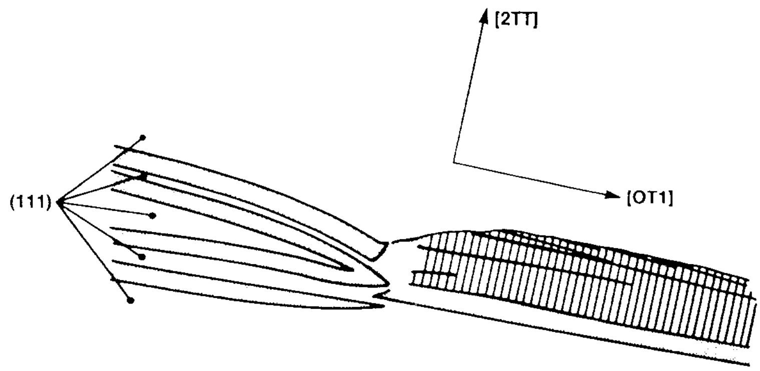

Figure 9 Schematic drawing of the geometry in Figs 5 and 6 with important crystallographic directions indicated.

step downwards followed by a series of shallower "plateaus", the surfaces of which contain the small transverse steps described above, separated by the small, sometimes discontinuous, longitudinal steps thoughout the remainder of the cleavage step. It is the initial, large step which is believed to result in the troughs on the mating surface. This is illustrated schematically in Fig. 12 where the initial step presumably occurs on another {1 1 1} cleavage plane at 70.5° to the primary {1 1 1} facet. As this step forms, the local stress apparently becomes insufficient to cause complete fracture through the ligament on this plane and, as a result, the series of plateaus and smaller steps are produced. However, from the depicted geometry (Fig. 12) it is apparent that the mating surface would appear to have a trough adjacent to the plateaus containing the small transverse steps, in accord with the observations. It should be mentioned that the above depiction is dependent on the height of the cleavage step; for smaller heights, the secondary steps should be eliminated (e.g. Fig. 13).

Next, it is interesting to analyse further the transverse steps prevalent on the majority of the cleavage step fracture surfaces. As noted in Section 2 the crystallography of these steps is consistent with fracture along the same primary (1 1 1) cleavage plane and another plane of the [$\bar{2}$ 1 1] zone. From surface energy considerations [12] the most likely secondary cleavage plane is {0 1 1} (in this case (0 1 $\bar{1}$) which is 90° from (1 1 1)), consistent with the apparent angle between these planes (Figs 6, 10). It should be noted that {0 1 1} microcleavage has been observed previously by Wolff and Broder [14] who, in addition, observed crack propagation along ⟨1 1 0⟩ consistent with the present results.

Because of the geometry in Fig. 9, it appears that the local crack front in the plateau regions propagates approximately parallel to the [$\bar{2}$ 1 1] zone until it suffers a nearly vertical displacement at the small, longitudinal steps. Subsequently, on the new, displaced plateau the transverse steps are reinitiated in a manner apparently similar to the formation of the V-shaped markings in Fig. 7. Thus, these small longitudinal steps effectively can be considered as local crack arrest markings within the ligament. Since they are frequently discontinuous, these so-called crack arrest markings demonstrate the overall complexity of the cleavage step formation process and indicate that the local crack front within the ligament propagates on parallel displaced cleavage facets in a manner which is quite similar to the propagation of the main crack front.

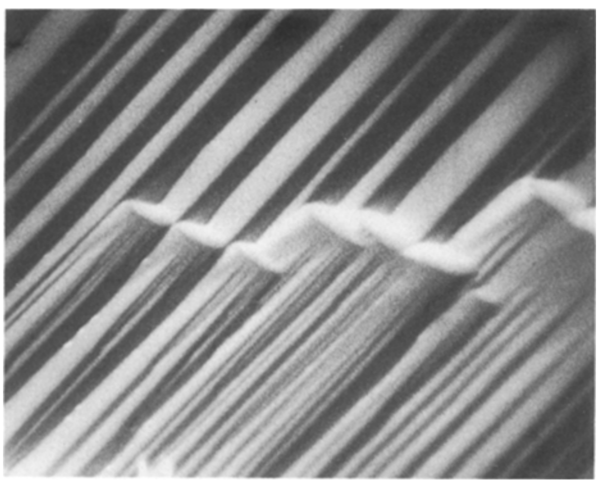

Figure 10 High magnification SEM of the transverse steps in Fig. 6 where it can be seen that some transverse steps are comprised of three, rather than two, crystallographic planes. × 14 000.

4. Conclusions

The primary results of the present study indicate that the cleavage step formation process is much more complex than previously believed. The main conclusions are:

1. When two edge cracks in silicon overlap, the interaction forces are initially repulsive becoming attractive upon overlap. This is consistent with the predictions by Swain *et al.* [2].

2. The repulsive forces can have sufficient strength to cause the cracks to deviate from their preferred {1 1 1} crystallographic cleavage plane implying that the value of K_{II}/K_I was underestimated by Swain *et al.*

3. The ligaments which are present as a result of crack overlap frequently fracture in a complex fashion on both primary and secondary {1 1 1} and secondary {0 1 1} cleavage planes. The resulting cleavage steps

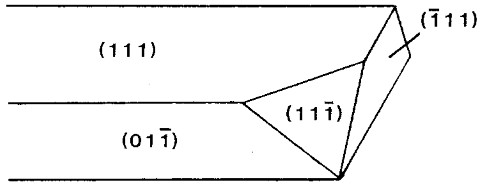

Figure 11 Schematic description of the transverse cleavage steps in Fig. 10.

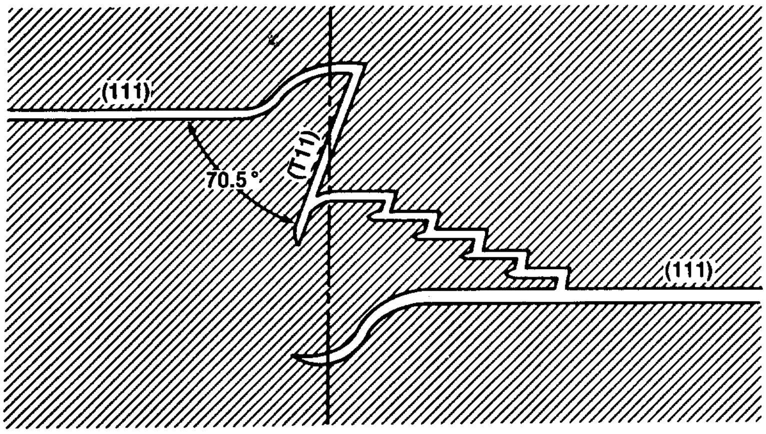

Figure 12 Schematic description of the cleavage step formation process which is believed to result in troughs in addition to the series of plateaus and smaller steps.

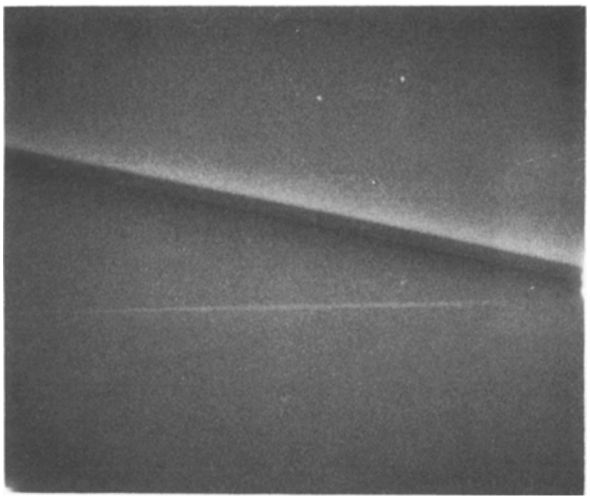

Figure 13 SEM of a smaller cleavage step where no secondary steps are evident. × 3200.

consist of complex arrays of transverse and longitudinal steps in addition to longitudinal troughs.

5. The transverse steps are composed of {1 1 1} and {0 1 1} cleavage planes except in the vicinity of longitudinal steps where a third fracture plane (probably {1 1 1}) frequently becomes evident.

6. The longitudinal steps within the overall cleavage step can be considered to be local crack arrest markings due to the fact that reinitiation of the transverse steps becomes necessary on the new, displaced plateau.

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