CHAPTER V

GROWTH HILLOCKS ON {0001} FACES

5.1 Introduction

Verma (1952b) reported trigonal, hexagonal, and their compound, superimposed oriented overgrowths on the basal faces of some silicon carbide crystals showing growth spirals. These oriented molecular layers, he suggested, might be silica deposits or possibly the growth of silicon carbide itself. As a possible mechanism of growth, a two dimensional nucleation around some foreign matter was proposed by him. During the present work as some observations appeared interesting, a reconsideration of this suggestion was made so as to establish any relationship of these features with the internal structure of the crystal.

Since Verma (loc. cit.), many workers have reported growth hillocks on different faces of both natural and synthetic crystals (Tolansky 1962, Sunagawa 1964, Patel and Ramanathan 1963, Patel and Ramachandran 1967, Ittyachen 1969 and Joshi and Kotru 1968), and have attributed two reasons for their formation: (i) two dimensional nucleation and (ii) growth from screw dislocations.

5.2 Correlation of Growth Hillocks with Linear Defects

Figure 23a is the (0001) face of a SiC crystal
showing a single growth hillock from which circular growth layers emanate. Figure 23b, an enlargement of the top of the hillock, reveals that the top is fairly flat with an hexagonal depression. The closed loops give the appearance of a Frank-Read source. On a close examination, a spiral from the depression and three other spirals, all of the same sense, at the centre of the flat top are very faintly visible. These four spirals, when hindered by an irregularity join together to form closed loops. Since the layers are very thick and cover the whole surface, these spirals should have helped the main growth of the crystal in the beginning. The crystal was etched in $\text{Na}_2\text{O}_2$ for 10 mts. and the region of Fig. 23b is shown in Fig. 23c after etching. All the four spirals have become more visible by etching. Besides, the hexagonal depression has grown in size and two characteristic screw dislocation etch pits (Horn 1952 and Amelinckx et al. 1960) have nucleated at the origins of the remaining three spirals. One can notice other pits due to edge dislocations also on the surface. Since no pit has been formed at the irregular feature, it is clear that the hillock is not due to a Frank-Read mechanism but due to the spirals of the same sense.

Another face shown in Fig. 24a was etched similarly. The hillock, marked A, did not show any sign of
clear spirals as in the earlier case. But in this case also a characteristic screw dislocation pit was developed on the top of the hillock along with other edge dislocation pits, as can be seen in Fig. 24b. The circular feature B might have formed due to some foreign material modifying the growth fronts from the hillock A. It cannot be due to screw dislocations as no screw dislocation pit has been developed at the centre of this feature.

The shape and symmetry of the pits formed on the tops and sides of these hillocks suggest that the material is silicon carbide.

5.3 Hillocks of Various Shapes

Figure 25 shows a cluster of triangular hillocks of different sizes. The hillocks are terraced with growth steps though the tops do not show any terracing. They overlap each other and also exist in opposite orientations. Each of these hillocks is originated from screw dislocations which are randomly distributed all over the face, and has been developed during the last stage of growth. Screw dislocations of opposite hands give rise to oppositely oriented hillocks and their corresponding size depends on the Burgers vectors of the screw dislocations from which they originate. Another factor can be the duration of the growth if they have started to grow at different times.
Figure 26a is more elucidating, where only two trigonal hillocks are present which are almost of the same size with rounded corners, but oppositely oriented. Very clear closed, large steps surrounding these hillocks over the face can be seen. The two hillocks, evidently, might have originated from two screw dislocations of equal strength but of opposite hands. At an early stage thick growth layers initiated from these screw dislocations advanced freely and interacted together to form loops of Frank-Read sources. The main part of the crystal is formed at this stage by the piling up of these layers. However, a change in the growth condition might have restricted their role in the main growth, and as Vand (1951) has suggested they will have independent growth. At this stage even, the hillocks were developed under three different conditions as can be seen in Fig. 26b.

Typical of the hexagonal hillocks observed are shown in Fig. 27a; of which one is very predominating. The kinking of the fringes in the interferogram (Fig. 27b) reveals that the steps are very closely spaced and that the predominant one is about $2\lambda$ high while the other one is about $\lambda$ high. However, the hillocks on the laboratory grown crystals were always regular in shape and hexagonal; an example is given in Fig. 28. Hillocks of circular and compound nature are shown in Figs. 23, 24 and 29. In
Fig. 29a, it can be seen that very faint growth layers emanate from the hillock. The details of the hillock are shown in Fig. 29b.

5.4 Hillocks at Twist Boundaries

The interferogram in Fig. 30a over the face in Fig. 30b suggests that the line running horizontally and then turning to the left corner of the photograph is either a tilt or a twist boundary. A tilt boundary consists of edge dislocations, whereas a twist boundary consists of screw dislocations. Actual grain boundaries have both tilt and twist components. Triangular and circular hillocks originate along this line, particularly from its end points which suggests that they are developed from screw dislocations. Similar observations are made by Sunagawa (1968) on hematite. Also visible are simple spirals on either side of the boundary and thick growth layers emanating from the boundary.

5.5 Hillocks and Spirals with 'Wrong' Sides

The polygonal spirals or features reported on SiC so far by various workers had their sides parallel to the edges of the crystal, in accordance with the symmetry of the crystal face. In the case of a crystal grown by Frank's spiral mechanism, the screw dislocation will usually emerge on the surface with its exposed ledge lying along one of the close-packed planes e.g. (101̅0)
plane; and hence the ledge winds up ultimately into spirals of the said nature. However, in the present investigations some SiC crystals have been observed to display features which do not have all their growth steps parallel to the crystal edges which are in $<11\bar{2}0>$ direction. Such two features are shown in Figs. 31 and 32.

In Fig. 31, the central dark portion forms the top of a hillock with five sides, two of which are in the $<10\bar{1}0>$ direction instead of the usual $<11\bar{2}0>$ direction. The turns of a right-handed spiral can be traversed in the inward direction from the point of the arrow. The spiral has six sides; but all of them are misoriented. The feature once again turns to five sided one with four misoriented sides. The central portion of the hillocks in Fig. 32 is irregular, though the growth fronts around it are five sided; out of which four are in the wrong orientation. The appearance of both the hillocks suggests that they may be formed at misfit boundaries, which can also provide growth centres, like in graphite (Noda et al. 1968).

Earlier, Amelinckx (1952) on gold crystals (cubic) and recently Jain and Trigunayat (1968) on CdI$_2$ (hexagonal) have reported such spirals. The 'wrong' orientation of these spirals and hence of the hillocks results from the initial ledge being in a 'wrong' orientation,
viz. a different orientation than the one along the usual close-packed plane. In SiC (1120) plane is also a close-packed plane and a misfit can occur along this plane, though the probability for this is small. The 'wrongly' oriented initial ledge is stable according to Burton et. al. (1951) and can generate a spiral in a normal way. However, the shape of a spiral is dependent on the crystallographic orientation, imparting a directional dependence of growth. If this attempt completely succeeds i.e. on all the three sides of a spiral — triangular spirals are common in SiC — each growth step will divide into two, with one branch being parallel to a crystal edge and the other retaining the original orientation of the step. The result will be a six sided spiral and similarly a five sided spiral will result if the branching occurs only on two sides. The branching takes place, depending on the extent of misorientation of growth steps of the 'wrong' spirals; the tendency to branch will be greater on the sides of greater misorientation.

5.6 Conclusions

The aforesaid observations show that these hillocks are formed by screw dislocation mechanism and that there are three types of hillocks:

1. Hexagonal and circular cones covering the whole surface (Figs. 23, 24, 29, 31 and 32).
2. Isolated hexagonal, circular and triangular hillocks (Figs. 23, 26, 28, 29 and 30).

3. Hillocks with 'wrong' orientations ; (Figs. 31 and 32).

Of these types 1 and 3 are similar to the cones of spirals observed and designated as type 3 by Sunagawa (1967). The type 2 may be growing as Vand (loc. cit.) has suggested. If this is so, these features are not a superimposed or epitaxial overgrowth of SiC or any other material as Verma (loc. cit.) suggested but are a continuation of the main growth from screw dislocations at the last stage. Hence the configuration of the surface layers of the crystal and the overgrowth cannot be different from that of an interior layer.