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STRUCTURE IN DIAMONDS AS REVEALED BY ETCHING

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ABSTRACT

Structure has been revealed in diamonds by etching polished sections with fused potassium nitrate. Several very different types of structure were observed, and an account is given of the principal types, together with illustrations. Both rectilinear and curved line structures were found. The structures are discussed in relation to the kinds of crystal growth which could have caused them. Layer growth parallel to {111} was common, but was not the only kind of growth. A difference has been found in the internal structures of type 1 and type 2 diamonds (infra-red classification). Most of the observations relate to natural diamonds, but some work on synthetic diamonds is reported. No correlation was found between internal structure of natural diamonds and geographic origin.

INTRODUCTION

This paper presents results of a study of the internal structures of some diamonds as revealed by etching polished sections. Strong oxidizing agents are the most convenient etchants for diamond, and fused potassium nitrate was used as etchant in all the experiments here described. The action of fused potassium nitrate on diamond has been studied by Tolansky (1955) and his co-workers. These workers studied extensively the microstructure of etched diamond surfaces and the way etch pits develop. They showed that individual polished faces on diamond may develop regular patterns of etch pits. These patterns consist of distinct regions of comparatively heavy and of comparatively light etch, and must represent variations in the perfection of crystallization of the underlying diamond—in its impurity content perhaps, or in its defect structure. Thus they are a record of the growth history of the diamond. Tolansky also concluded that the patterns afford clear evidence that in each crystal growth has proceeded by a layer formation, the layers being parallel to {111}.

The present work has shown that the growth histories of diamonds may be more complicated than this, that there have been different types of diamond growth, that some diamonds have grown and then partially dissolved and then re-grown, that there is a relation between the kind of etch structure which is found and the type of diamond material (*i.e.* whether it is of type 1 or type 2 on the basis of its infra-red absorption), etc. Some preliminary results have been described in two previous publications (Seal 1962, 1963a) but in the present paper an attempt is made to classify the structures which have been observed and to give some interpretation of their significance.

Experimental Technique

Except for a few special cases, all the sections were approximately parallel to {100}. At first an attempt was made to use only sections which were fairly closely parallel to the true crystallographic planes (within 2° of {100}), but later work showed that somewhat greater misorientation (up to about 5°) did not add to the difficulty of interpretation. Indeed some misorientation may be helpful in that it permits the traces of different planes whose line of intersection is parallel to {100} to be distinguished. In some cases a section was prepared by polishing a flat area on the chosen diamond. Orientation was then by visual examination. In other cases the chosen diamond was sawn in two and the cut faces on the two pieces were then polished. This work was done for the author by the Diamond Research Laboratory, Johannesburg, who also oriented the diamonds by *x*-ray diffraction.

The polished diamonds were etched in fused potassium nitrate at 675° C. for 15 minutes. The etchant was contained in a platinum crucible and heated in a muffle furnace. Temperatures were measured with a platinum-platinum+10% rhodium thermocouple.

The diamonds were cleaned and then examined in a Vickers optical microscope with incident illumination as for metallography. Normally the diamonds were examined unsilvered, but in some cases where internal reflections were troublesome a thin layer of silver was deposited on the etched surface by vacuum evaporation. Interferometric techniques were used to determine surface profiles on some samples and these also were silvered by vacuum evaporation. Portions of silvered microscope cover slides were used as the reference surfaces for the interferometric work. These are smooth locally but not flat. Mercury green illumination was used. Some surfaces were examined with an electron microscope (Philips EM200) using standard replica techniques.

Description of Diamonds

In total, eighty-six diamonds were examined. The origins of these were as follows:

Birim River, Ghana,	17 diamonds;
Premier Mine, South Africa,	16 diamonds;
Kimberley area, South Africa,	10 diamonds;
Williamson Mine, Tanganyika,	10 diamonds;
Consolidated Diamond Mines, South-west Africa,	5 diamonds;
Congo,	4 diamonds;
Murfreesboro, Arkansas,	2 diamonds;
Panna, central India,	2 diamonds;
Sierra Leone,	1 diamond;
synthetic cubo-octahedra from Adamant Research Laboratory, Johannesburg,	9 diamonds;
natural diamonds of unknown origin,	10 diamonds.

There was a wide range of sizes in the samples examined, but most of the natural diamonds were in the range of 0.1 to 1.0 carat (20 to 200 mg). The synthetic diamonds were quite small—about 0.001 carat (0.2 mg) each.

The diversity of natural diamonds is such that it was impossible to examine all known kinds of diamond crystal, but representative samples of the most important crystal types and habits were examined—octahedra, dodecahedra, cubes, elongated crystals, maccles, coated crystals, etc. Nearly all the crystals were transparent and there was a range of colors including blue, white, green, yellow, and brown.

Thirty-eight of the crystals were classified on the basis of infra-red absorption and electrical conductivity as type 1, 2A or 2B. Twenty-six of these were type 1 (nine from Premier Mine, seven from Kimberley area, five from Tanganyika, and five from South-west Africa): eight were type 2A (four from Premier Mine, one each from Ghana and Arkansas, and two of unknown origin): four were type 2B (one from Premier Mine, and three of unknown origin). There was deliberate selection of type 2 diamonds and so there is no significance in the relative numbers of diamonds of each type.

Etch Patterns

The observed patterns can be classified in certain broad groupings as follows:

Uniform structure. Some diamonds showed large clear areas with virtually no gross structure. On a few crystals the entire etched cross section was apparently structureless at low magnification. Examination of such surfaces at higher magnification using electron microscopy showed that the surfaces were covered with a random distribution of small etch pits (Fig. 1). Other surfaces showed large uniform heavily etched areas.

Rectilinear patterns. Figure 2 shows a rather simple rectilinear pattern. The dark and light areas represent regions of heavy and light etch (this was shown by electron microscopy). The straight lines are parallel to the intercepts of {111} planes with the plane of section (001), and it was shown by polishing other sections through similar diamonds that they represent sections through a three-dimensional laminar structure of concentric octahedra. This is the type of pattern described by Tolansky (1955).

Figure 3 shows a somewhat more complex rectilinear pattern and Fig. 4 shows the central part of a very complex rectilinear pattern. Again the straight lines represent sections through {111} laminae but they are quite limited in lateral extent and they form a rather complex three-dimensional structure.

Rectilinear patterns were very common. Approximately 65% of the

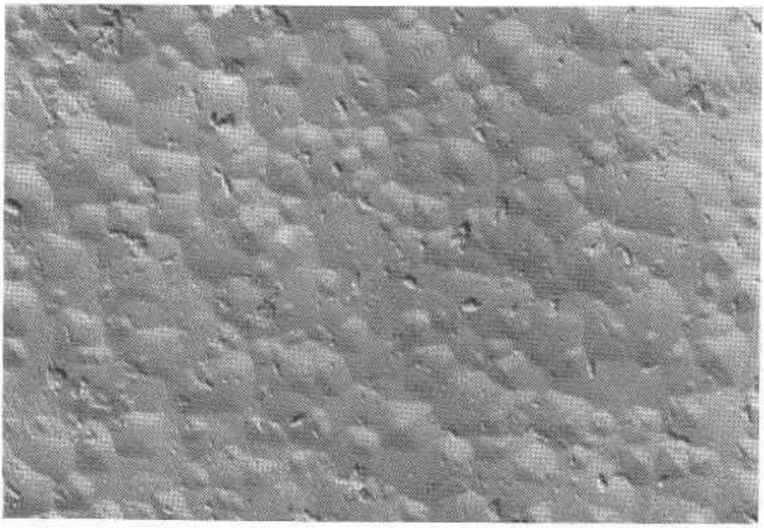


FIG. 1. Diamond no. 408 (Tanganyika, type 1)—electron micrograph showing individual etch pits on an etched polished section. Shadowed positive replica: magnification 18,000×.

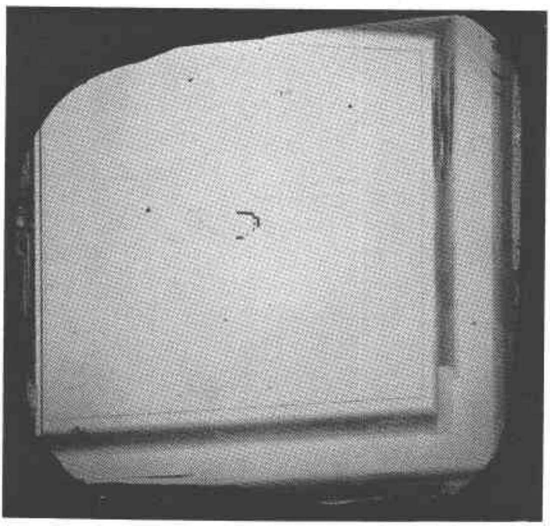
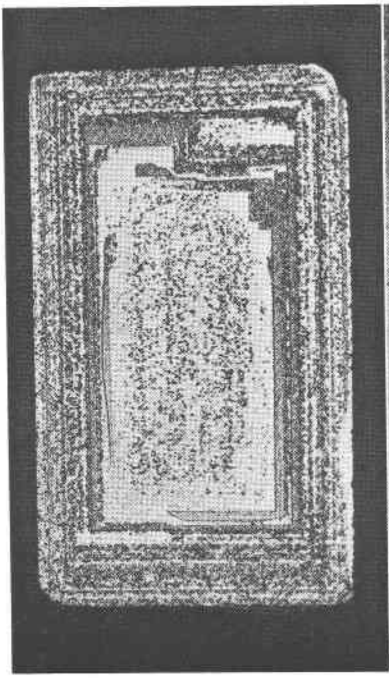


FIG. 2. Diamond no. 550A (Premier Mine, type 1). The section is approximately through the center of the diamond. At the edges it intersects curved dodecahedral faces. Magnification 19×.



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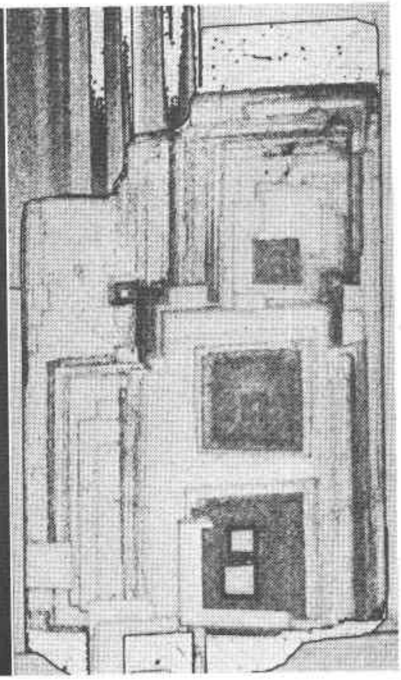

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FIG. 3. Diamond no. 559 (Ghana). This diamond was a good octahedron with sharp edges. The section passes through the diamond approximately one quarter of the way across it from corner to corner. Magnification 120×.

FIG. 4. Diamond no. 551A (Premier Mine, type 1). Region near the center of the diamond. Magnification 120×.

diamonds examined showed such patterns, though in many cases other types of patterns were also present.

Curved lines. On some diamonds, lines which were for the most part straight (forming rectilinear patterns) were in certain regions curved. Examples can be seen in Figs. 3 and 4. Sometimes it was found that a sequence of straight lines was interrupted by a curved line. This can be seen in several places in Fig. 5. Figure 6 shows the same effect in another diamond seen at higher magnification. Sometimes a curved laminar structure was observed. Examples may be seen towards the center of Fig. 5 or in Fig. 7 (another diamond). Curved line structures of these types were common and were found in one type or another on approximately three-quarters of the diamonds with rectilinear etch structures. Other types of curved structure were also seen, though less commonly, and these are described under separate headings below.

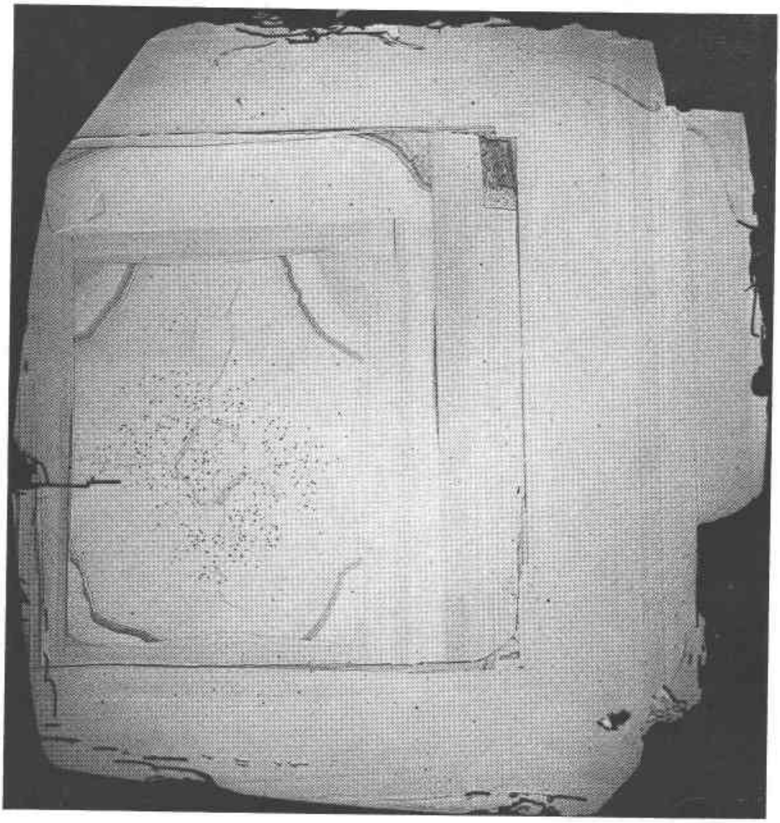

FIG. 5. Diamond no. 534A (Kimberley, type 1). The section is approximately through the center of the diamond, and at the edges it intersects curved and fractured dodecahedral faces. Magnification 28×.

Central crosses. The central part of Fig. 5 is typical of this kind of structure. For clarity this area is shown at higher magnification in Fig. 8. Such structures were seen on only four diamonds.

Other curved and cusped lines. One diamond showed some unusual features of which Fig. 9 is an illustration. Two perpendicular sections (100) and (010) were polished on this diamond. Figure 9a is of the one and Fig. 9b is of the other, in the region of their line of intersection. Thus the two views are a sectional plan and a sectional elevation of the same feature, which is seen to be a doubly curved and irregular plate of varying thickness. This same diamond in other regions showed curved and cusped lines (Fig. 10). Also sections across the extremities of this crystal (*i.e.* sections which were well off-center) had some apparently circular markings (Fig. 11). By polishing a number of sections, one of which was perpendicular to

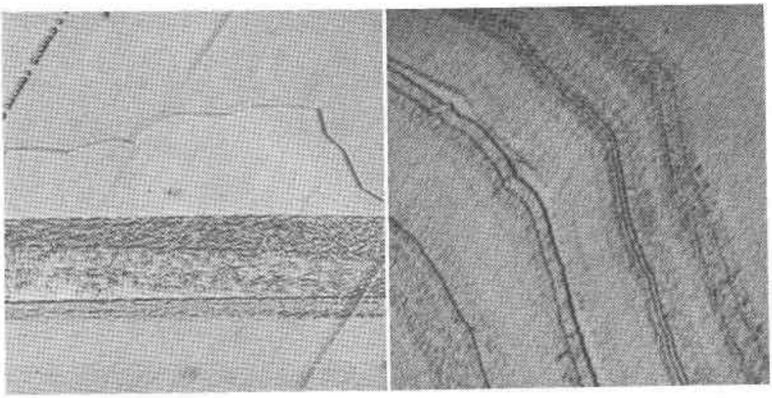

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FIG. 6. Diamond no. 552A (Premier Mine, type 1). A sequence of straight lines and a curved line on an etched section. The parallel diagonal lines represent grooves resulting from the polishing. Magnification 200×.

FIG. 7. Diamond no. 535A (Kimberley, type 1). This part of the etched section is in the outer part of the crystal near the natural surface. It shows a curved layer structure. Magnification 240×.

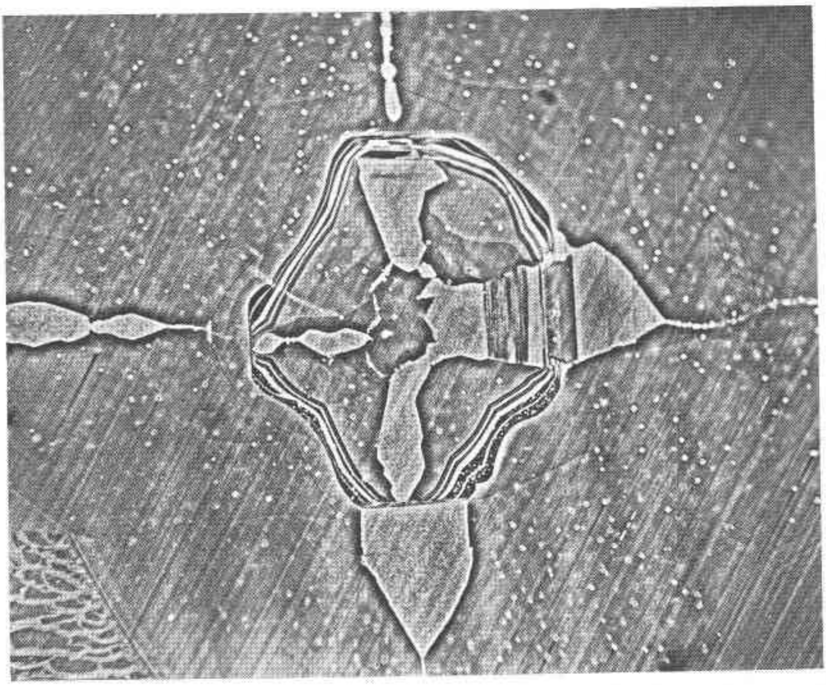


FIG. 8. The central part of Fig. 5 at higher magnification (120×)—phase contrast. The parallel diagonal lines represent grooves resulting from the polishing.

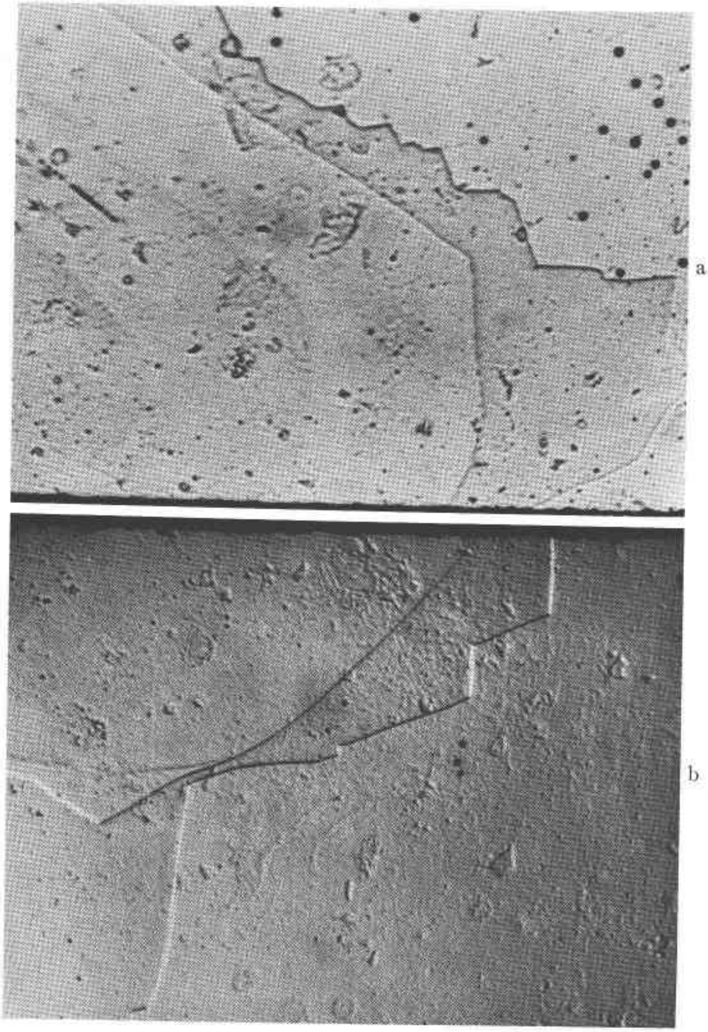


FIG. 9. Diamond no. 403 (Tanganyika, type 1). The diamond was a dodecahedron with curved faces. Two perpendicular etched sections through the same feature are shown, the one (a) being approximately (100), the other (b) being approximately (010). The common edge of the two sections which was produced by polishing appears near the lower margin of (a) and the upper margin of (b). Magnification 193×.

that of Fig. 11 and the remainder parallel to it, it was found that these apparently circular markings represent oblique sections through bent but roughly cylindrical domes sloping upwards and outwards from a central dimple (Fig. 12). It was confirmed by x-ray diffraction (done for the

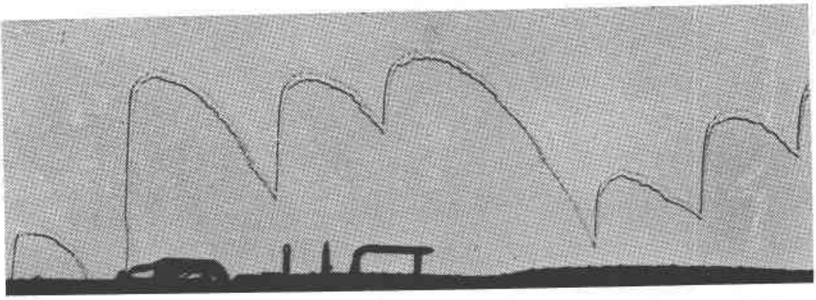


Fig. 10. Part of a different (100) etched section through the diamond shown in Fig. 9 (no. 403). This region lies close to the natural surface and the picture includes its line of intersection. Magnification 240×.

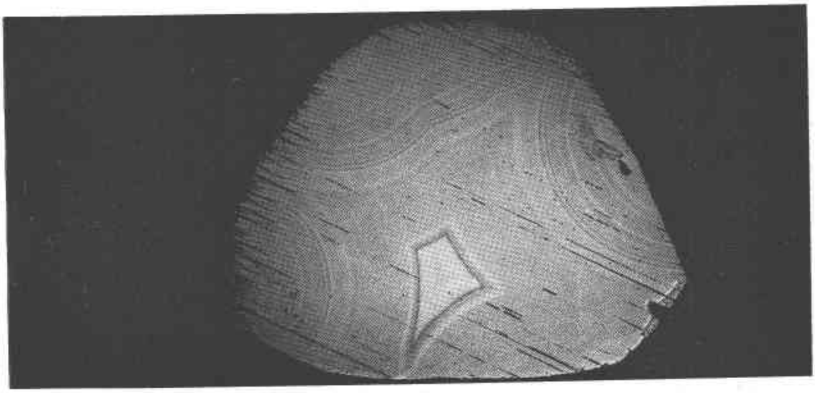


Fig. 11. An etched (100) section through one corner of the diamond shown in Figs. 9 and 10 (no. 403). The parallel lines represent grooves resulting from the polishing. Magnification 30×.

author by B. Post) that this was a single crystal, with no detected twinning or misoriented structure.

Sub-grains. Many areas which appeared not to contain much structure when examined normally were found to contain a great deal of structure when more sophisticated optical techniques were used. Figure 13 shows structure which was rendered much more clearly visible by high dispersion plural beam interferometry. Figure 14 shows another diamond where phase contrast was used. Such structures were found to be quite common.

Radial veining. A radial veining, as in Fig. 15, was observed on a total of three diamonds.

Coated diamonds. Diamonds having a greenish opaque coating around a transparent core are well known in the production of the Congo and West

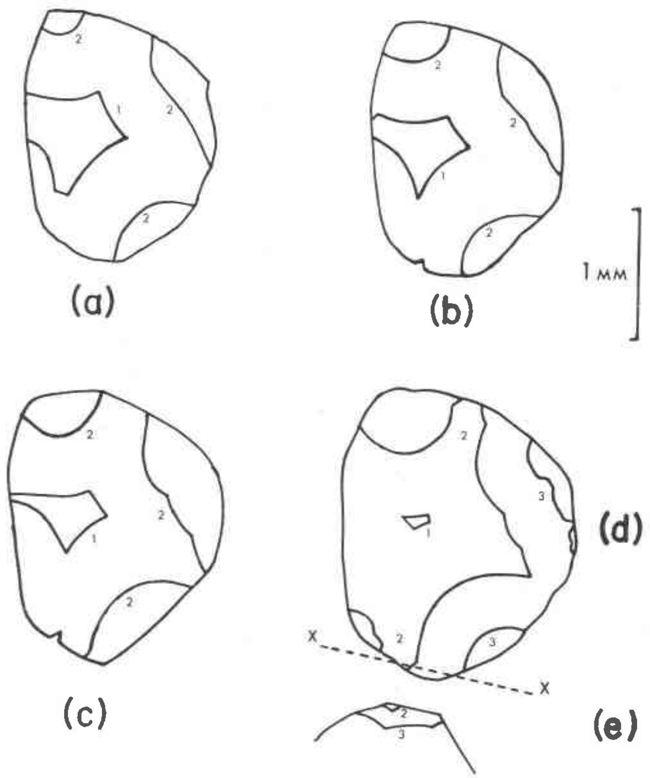


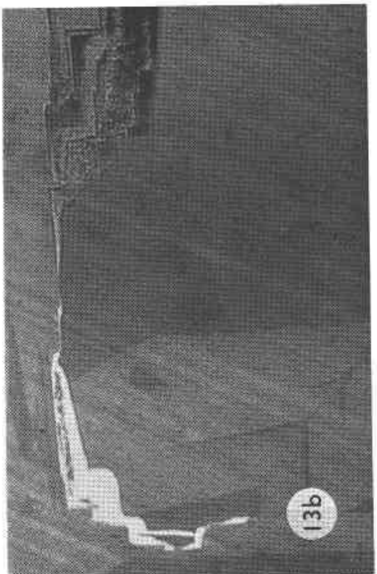
FIG. 12. Drawing showing the intersections of the same layers in diamond no. 403 by four sections all approximately parallel to (100) and by a section approximately parallel to (010). The lines marking the intersections of the same layer were identified by their position in a recognizable sequence of clearly defined lines. The four (100) sections were all polished across one corner of the diamond and (c) is a drawing of the section shown in Fig. 11. (a) was at a perpendicular distance of 0.511 mm into the diamond from the corner; (b) was 0.023 mm below (a); (c) was 0.015 mm below (b); (d) was 0.025 mm below (c). The original diamond was about 5 mm across. (e) was polished perpendicular to (d) and intersects it along the line XX.

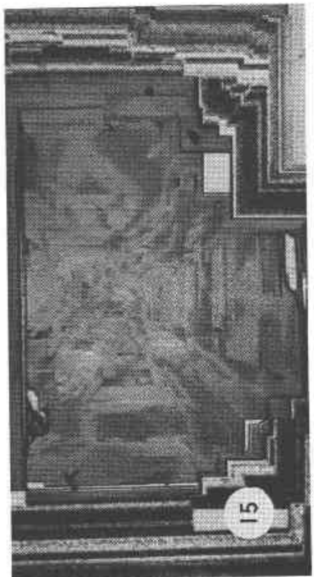
FIG. 13. Diamond no. 536A (Kimberley). Part of an etched section near the center of the diamond. (a) is a normal micrograph; (b) is a high dispersion plural beam interferogram in which small changes in surface height at the "sub-grain" boundaries are shown up by the different shades of grey. The parallel diagonal lines represent grooves resulting from the polishing. Magnification 170×.

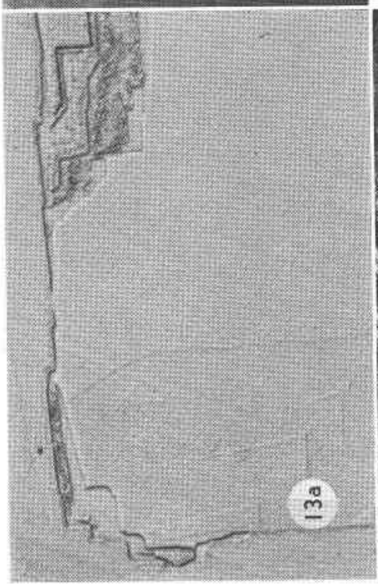
FIG. 14. "Sub-grain" structure in an etched section of diamond no. 534A (Kimberley, type 1)—phase contrast. Magnification 170×.

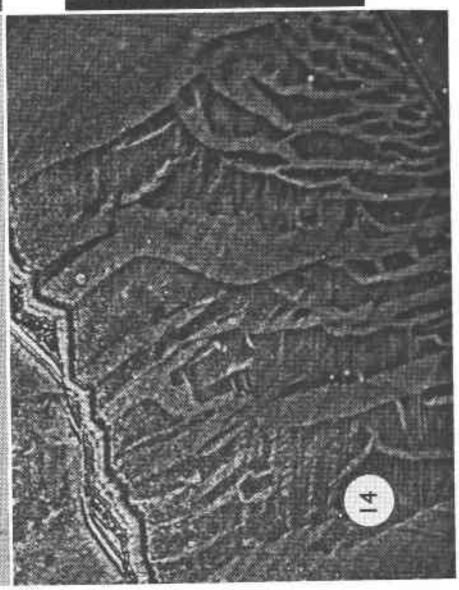
FIG. 15. Diamond no. 538A (Tanganyika). Part of an etched section near the center of the diamond. Magnification 27×.

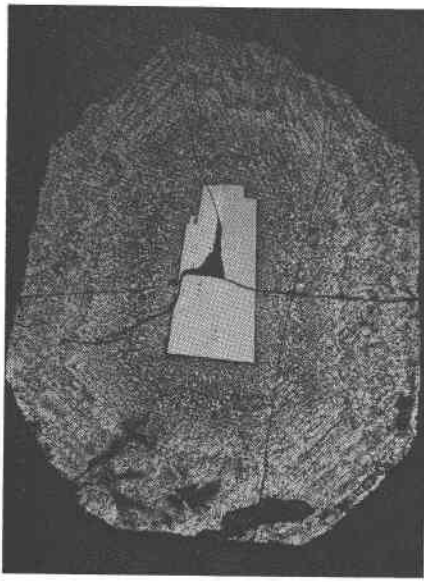
Africa. Five such diamonds were examined, and Fig. 16 shows the typical appearance. The coating has an irregular structure appearing at higher magnification as an irregular mosaic with some local orientation. The core in this case is virtually structureless. Large cracks are visible. These were evident in all five stones. It is normally fairly easy to remove the coating from such diamonds by cleavage, but there was no indication in the











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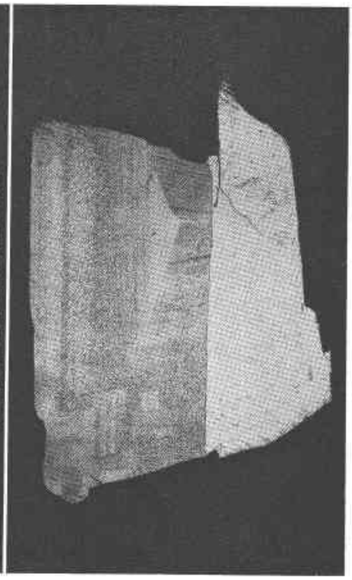

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FIG. 16. Diamond no. 487 (Congo)—a coated diamond having octahedral and stepped dodecahedral faces. This etched section was polished across one corner of the diamond and just penetrates to the central clear crystal which appears to be an octahedron with sharp edges. Magnification 31×.

FIG. 17. Diamond no. 725 (Ghana)—a maccle. This etched section intersects both portions of the twinned crystal. Magnification 17×.

samples examined of a preference for crack propagation along the interface of coating and core.

Maccles. Maccles are twinned diamonds, the twin plane being {111} (*i.e.* spinel-type twinning). A section through a maccle is shown in Fig. 17. The trace of the twin plane is clearly visible. The section is of course only parallel to {001} (approximately) for one portion of the crystal, the section through the twinned portion necessarily having a different orientation. The difference in the amount of etching of the two portions may thus be a consequence of this difference in orientation, since different crystal faces appear to etch at different rates.

Type 2 diamonds. No specific type of structure could be associated with type 1 diamonds, and all the main types of structure listed above were found in type 1 diamonds. The twelve type 2 diamonds, however, all had very similar structures. Figure 18 is an example. Etching was evidently uniform over the entire surface, except that some faint parallel lines are visible. The other eleven type 2 diamonds had substantially the same appearance. In each there was similar uniform etching. Some of the diamonds showed the faint lines, some not, but in all cases the structures were of low contrast compared with rectilinear structures such as those of figures 2, 3 and 4. No significant difference could be found between type 2A and type 2B diamonds.

Synthetic diamonds (*cubo-octahedra*). All the results described above have referred to natural diamonds of various origins. The synthetic diamonds were considerably smaller than the natural, and the only structures found on etched polished sections through them were random arrays of etch pits and rectilinear structures. Figure 19a shows a typical example with a random array of etch pits, and Fig. 19b a line a little way in from the edge of the crystal. The line runs parallel to the edge and has portions in ⟨100⟩ directions and portions in ⟨110⟩ directions.

Discussion

It is clear that natural diamonds have grown in different ways, and that some have had a rather complicated growth history. The rectilinear etch structures evidently correspond to a lamellar type of growth, and it seems that many octahedral diamonds have grown in the obvious way, by crystallization in layers. The rectilinear structures on synthetic diamonds indicate a similar type of growth, though it seems here that growth may also occur on planes other than {111}. The variations in natural diamond crystallization which are revealed by the etch patterns presumably have arisen because of changes in the environment of the diamond while it was growing, changes in temperature or pressure perhaps or in the chemical nature of the environment. There is of course no indication of the time scale of these changes except that it seems improbable that growth was slow on a geological time scale (for, if growth were slow, the significant changes in environment would have been large scale ones affecting all sides of the diamond equally, and one would then expect to observe the same sequences of layers in all directions outwards from the center of the diamond). Possibly the diamonds of Figs. 2 and 3 grew rather slowly, but the more complicated patterns such as Fig. 4 would indicate comparatively rapid growth (rapid in comparison to the time scale of diffusion processes in the environment). The more complicated rectilinear patterns must correspond to nucleation of growth at many places and the formation of octahedral projections on larger octahedral slopes, etc.

The explanation of the curved portions of otherwise straight lines also seems to be straightforward. Presumably something interfered with growth locally—a mechanical obstruction perhaps or a local deficiency of carbon in the surrounding medium.

However, some of the curved lines must be due to another cause,

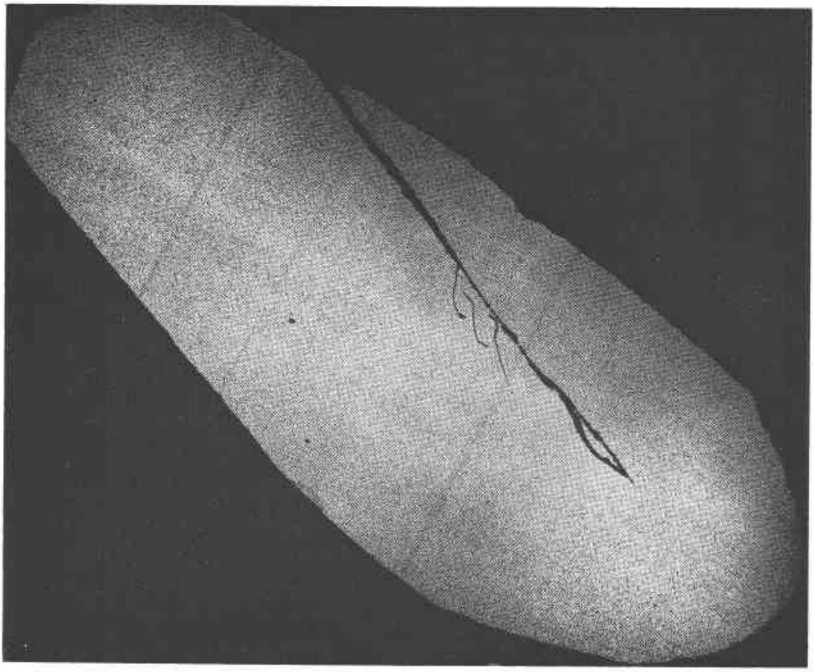

FIG. 18. Diamond no. 772A (Premier Mine, type 2A). This diamond had an irregular shape, flattened at one end and elongated to a point at the other. There were many curved and twisted faces. This etched section passes across the flattened portion. Magnification 28×.

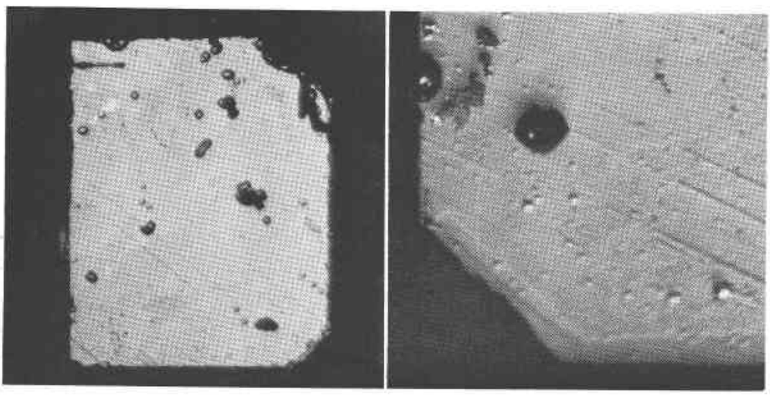

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FIG. 19. Etched polished sections through two synthetic diamond crystals. Both were cubo-octahedra. The parallel diagonal lines represent grooves resulting from the polishing. (a)—diamond no. 496; magnification 145× : (b)—diamond no. 485; magnification 370×.

namely dissolution. Consider, for example, Fig. 2. This shows a diamond which clearly grew as a series of concentric octahedra. The diamond has octahedral faces, but it also has curved dodecahedral faces, and the four edges that one sees in the figure represent the intercepts of the plane of section (001) with the four curved dodecahedral faces lying in the zone [001]. The dark heavily etched bands (which presumably represent the positions of the surface at different times during growth) intersect the present natural surface without being affected by it. Clearly then the diamond was at one time larger than it is now, for there is no other way of explaining the intersection of the growth bands and the natural surface. If there were just one growth band intersecting the natural surface, one could suppose that this represented the limiting extent of lateral growth of the last layer of diamond to be deposited, but the existence of different growth layers further out from the center indicates that growth continued

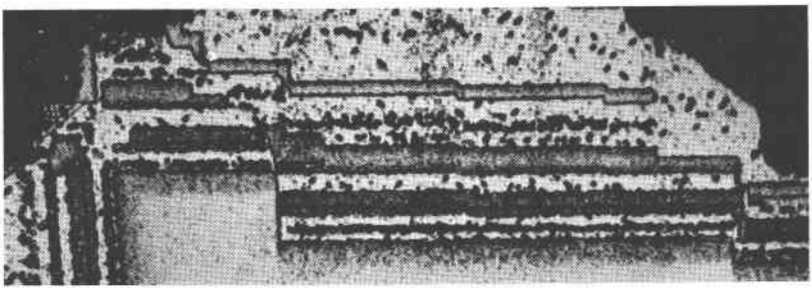


FIG. 20. Diamond no. 719 (Premier Mine). Part of an etched section near the natural surface, showing a stepped layer terminating within the crystal. Magnification 180×.

(and there is no reason to suppose that it would have continued radially, but not laterally). There is evidence that for other diamonds external conditions did change whilst particular layers were growing laterally, giving diamonds in which layers of distinctive etch character terminated within the crystal. An example is shown in Fig. 20. The effect shown in Fig. 2 is not explainable in this way, however, and it must imply that the diamond, once larger than it is now, has partially dissolved in the surrounding medium. Examination of other diamonds shows that this effect is widespread and furthermore that many diamonds have gone through cycles of growth, dissolution, and re-growth. Figure 5 is an example.

These considerations raise the question of the origin of diamond dodecahedral faces generally. Except perhaps for coated diamonds, dodecahedral faces are seldom found on natural diamonds as a well defined form in the strict crystallographic sense of plane faces having exact Miller indices {110}. Instead two types of near dodecahedral face are common. The one consists of faces which are curved and only approximately {110}. They may enclose a twelve-faced solid, each face of which is lozenge shaped but non-flat, or they may occur in association with other forms, notably {111}. Such faces are generally fairly smooth though they may be pitted to some extent. The other type of near dodecahedral face has parallel ridges and consists basically of an extended stepped edge between two {111} faces. From the present work it has been found that the first type of near dodecahedral face has invariably formed through dissolution of a larger diamond. In all cases where the etch patterns were sufficiently well developed to be informative, it was found that the patterns intersect the natural surface in a way which can only imply dissolution. Evidence that dodecahedral faces may form through partial graphitization of the diamond was presented in an earlier paper (Seal, 1963b) but this is not the only possible dissolution mechanism. It is known that dodecahedral faces are rare in synthetic diamonds and that when they occur it is under conditions where dissolution is suspected (Bovenkerk, 1961). The second, stepped type of dodecahedral face (on natural diamonds) does appear to be a growth form and in some cases to have persisted as such during the latter stages of growth. A section of such a diamond where two {111} faces meet in an approximately {110} stepped edge is shown in Fig. 21.

The central crosses as in Figs. 5 and 8 are difficult to explain and one can only conjecture as to what may have happened. Possibly something of the following kind may be responsible for the observed structures. There are four sets of curved bands in the outer central part of Fig. 5 (similar to the curved bands of Fig. 7). They presumably correspond to growth of curved faces at this stage in the history of the diamond. These bands have no exact crystallographic orientation, but the sets of bands appear to meet at crystallographically oriented planes forming the arms of the cross. At these planes of meeting, normal {111} growth was possible. Thus the planes may have thickened into bands as normal {111} growth proceeded and such bands form the arms of the cross. Impurities which were not included in the diamond crystal as the curved layers grew would then have been swept to the side (the layers growing laterally) and the concentration of such impurities would have risen along the arms of the cross. Possibly some impurity could then have been incorporated in the diamond between layers of the normal {111} growth. Some supporting evidence for this theory comes from a diamond which was found on examination of its interior through a pair of parallel polished faces to contain a faint central brown cross in an otherwise clear crystal. The arms of this cross were oriented. They radiated outwards from a central point and each arm pointed in an approximately ⟨001⟩ direction, but with a spread of angles about the true direction as shown in Fig. 22.

Some of the other curved features may have a similar origin, *i.e.*, be due to segregation of impurities during crystallization, but there are other possibilities—*e.g.* diffusion in the diamond crystal. The "sub-grains"

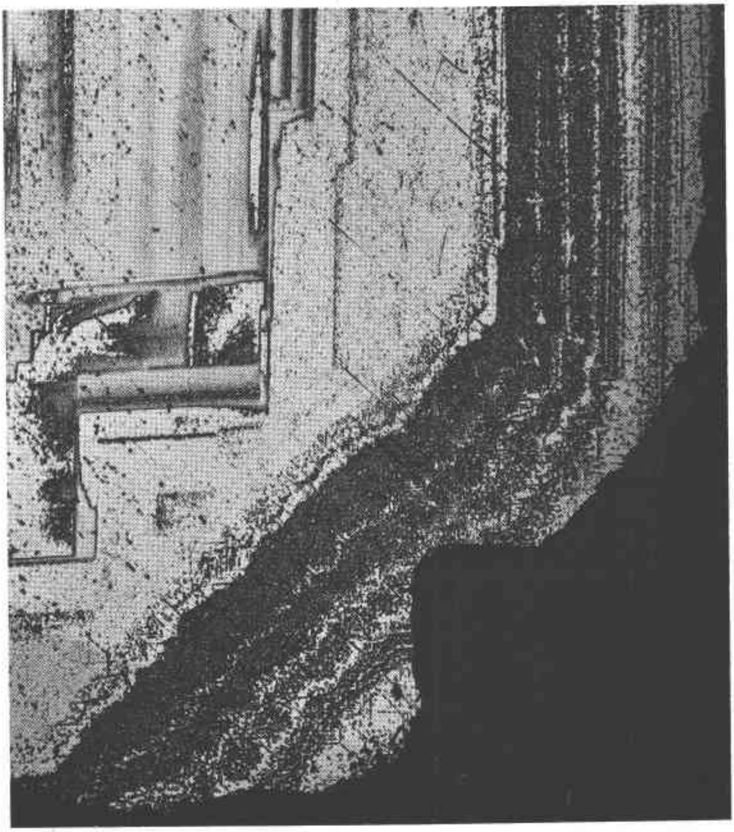


FIG. 21. Diamond no. 549A (Premier Mine, type 1). This was an octahedron having stepped dodecahedral faces at its edges. The picture shows a corner of an etched section and the intersection of one of these faces. It seems that the crystal in the earlier stages of growth had octahedral faces meeting in sharp edges, but that subsequently stepped dodecahedral faces developed and persisted through the later stages of growth. Magnification 120×.

could well have a normal structure of small angle grain boundaries, but other explanations are also possible. They could, for example, be another manifestation of a segregation of impurities during crystallization. An explanation of the patterns of radial veining could be that the diamond grew in a non-homogeneous medium, that impurities which would affect the diamond crystallization were distributed non-uniformly through this medium, and that the medium was sufficiently viscous and diffusion of impurities in it sufficiently difficult for the pattern of impurities in the medium to become imprinted in the growing diamond. Such a pattern would be irregular laterally, corresponding to the irregular distribution of impurities, but would have radial continuity due to the outward growth

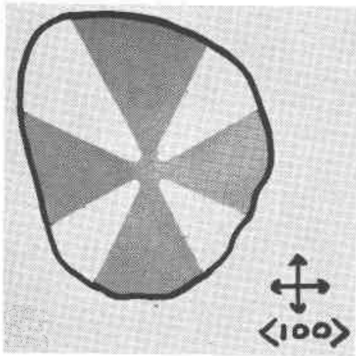
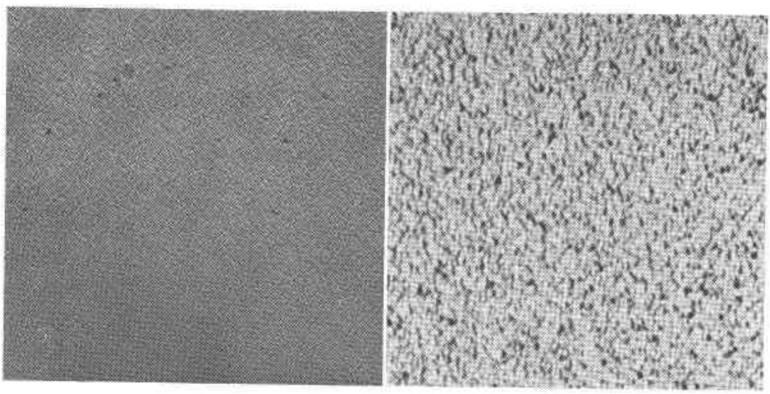


FIG. 22. Drawing showing the shape of a faint brown cross observed in the center of a diamond (no. 403, Tanganyika, type 1). The cross has six arms, two of which would be perpendicular to the paper in this representation.

of the diamond pushing back the surrounding medium. Clearly many hypotheses are possible to explain the details of the etch patterns, but there is little point in pursuing these when they become mere speculation. One conclusion is, however, inescapable—that diamonds have had varied and in many cases extremely complicated growth histories.

The differences between type 1 and type 2 diamonds are interesting and are in accord with current ideas as to the reasons for the existence of the different types of diamond. A review of these ideas has been given by Champion (1963). In the present work type 2 diamonds were found to be very uniform in their etch behavior and thus in their crystallization. Type 1 diamonds, on the other hand, are in general quite inhomogeneous in



a b

FIG. 23. Micrographs showing for comparison parts of etched polished sections of two diamonds: (a) no. 408 (Tanganyika, type 1)—part of a section showing virtually no structure when examined at low magnification. (b) no. 773A (Premier Mine, type 2A)—part of a typical section of a type 2 diamond. Magnification 250×.

their etch behavior and crystallization. A few type 1 diamonds showed virtually no structure in etched section, but these are a minority (one diamond with no detected structure, three with only a little, out of the twenty-six known type 1 diamonds). Possibly other sections through these four diamonds would have shown structure; possibly they consist of uniform type 1 material. Their appearance at high magnification is different from that of the type 2 diamonds (Fig. 23). One would expect that those physical properties of diamond which depend critically on the perfection of crystallization and the impurity content (*e.g.* infra-red absorption spectra) would show marked variation from diamond to diamond. This was confirmed by experiment, though for type 1 diamonds no correlation of etch pattern and strength of absorption bands in the infra-red could be found. Such variation in spectra from diamond to diamond is of course well known and has been reported many times in the published literature. It is worth remarking that in general an infra-red absorption spectrum of a type 1 diamond will represent a summation of the absorptions of the many different layers of the crystal through which the infra-red radiation has passed. One would expect that spectra obtained with a micro-beam spectrometer would vary from place to place on the same diamond.

No correlation could be found between the type of etch pattern observed and the origin of the diamond. If any such correlation exists, it must be on a statistical basis only and analysis of large numbers of diamonds would be necessary in order to confirm its existence.

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