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AD-A243 093

RT DOCUMENTATION PAGE

1b RESTRICTIVE MARKINGS

3 DISTRIBUTION/AVAILABILITY OF REPORT
unlimited

b DECLASSIFICATION / DOWNG G SCHEDU

PERFORMING ORGANIZATION REPORT NUMBER(S)
7

5 MONITORING ORGANIZATION REPORT NUMBER(S)

a NAME OF PERFORMING ORGANIZATION
NIST

6b OFFICE SYMBOL *(If applicable)*

7a NAME OF MONITORING ORGANIZATION
ONR

c ADDRESS *(City, State, and ZIP Code)*
A329, Materials Building
Gaithersburg, Maryland 20899-0001

7b ADDRESS *(City, State, and ZIP Code)*
Code 1131
800 N. Quincy Street
Arlington, VA 22217-5000

a NAME OF FUNDING / SPONSORING ORGANIZATION
ONR

8b OFFICE SYMBOL *(If applicable)*

9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER
N00014-90-F-0011

c ADDRESS *(City, State, and ZIP Code)*

10 SOURCE OF FUNDING NUMBERS

PROGRAM ELEMENT NO	PROJECT NO	TASK NO	WORK UNIT NO

1 TITLE *(Include Security Classification)*
High Resolution Electron Microscopy of Diamond Film Growth Defects and Their Interactions

2 PERSONAL AUTHOR(S)
D. Shechtman, E. N. Farabaugh, L. H. Robins and A. Feldman

3a. TYPE OF REPORT
interim

13b. TIME COVERED
FROM ________ TO ________

14 DATE OF REPORT *(Year, Month, Day)*
91-9-27

15 PAGE COUNT
19

6 SUPPLEMENTARY NOTATION

7 COSATI CODES

FIELD	GROUP	SUB-GROUP

18 SUBJECT TERMS *(Continue on reverse if necessary and identify by block number)*
This document has been approved for public release and sale: its distribution is unlimited,

9 ABSTRACT *(Continue on reverse if necessary and identify by block number)*

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21 ABSTRACT SECURITY CLASSIFICATION
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22a NAME OF RESPONSIBLE INDIVIDUAL

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22c OFFICE SYMBOL

DD FORM 1473, 84 MAR

83 APR edition may be used until exhausted
All other editions are obsolete

SECURITY CLASSIFICATION OF THIS PAGE

Enclosure ([illegible])

OFFICE OF NAVAL RESEARCH

Contract N00014-90-F-0011

R&T Project No. IRMT 025

TECHNICAL REPORT No. 7

HIGH RESOLUTION ELECTRON MICROSCOPY OF DIAMOND FILM GROWTH DEFECTS AND THEIR INTERACTIONS

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submitted to

SPIE PROCEEDINGS 1534, DIAMOND OPTICS IV

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September 25, 1991

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ABSTRACT

High resolution electron microscopy of plasma-assisted chemical vapor deposition (CVD) diamond films was performed. The film was fine grained with a grain size of the order of 0.1 micrometer. Several features of the microstructure were studied and their importance to the understanding of the diamond film growth was evaluated. The observations include:

1. Twinning density rises as a function of the distance from the center of the crystal.
2. The twins have an important role in the rapid growth of this kind of film. The reentrant angle between intersecting twins serves as a nucleation site for the growth of new {111} planes.
3. The center point of a twin quintuplet has five reentrant angles and thus serves as a preferred nucleation site for new planes as the crystal grows.
4. Misfit boundaries, being the locus of intersection points of the growing planes on two adjacent twins can serve as an indicator for the local crystal growth direction. The central nucleation site for the growing planes can thus be traced back in many cases to a quintuplet twin point.

1. INTRODUCTION

An impressive variety of techniques have been used in recent years to deposit diamond films; however the morphologies of these films have similar features. The crystal size varies between a fraction of a micrometer to several micrometers; the crystal shapes are cubic, octahedral or icosahedral; and, in many cases twinned particles are observed. We assume therefore that the nucleation and growth of the films grown by the various techniques have much in common. Planar defects such as stacking faults and twins are readily found in CVD diamond structure, while diamonds that grow under thermodynamically stable conditions such as natural diamonds have a very low defect density. These differences are important to the understanding of the nucleation and growth of diamond films and may indicate the direction toward better controlled microstructure and properties. The fine features of these growth defects can be clearly observed and studied by high resolution electron microscopy (HREM) which provides, in addition to detailed images, crystallographic information on the defects and their boundaries. Several reported studies [1-7] used HREM and obtained a view of the nature of stacking faults, twins and twinning configurations as well as the interface between the substrate and the diamond film. It is the purpose of our study to closely investigate the crystallography of these defects, and, more importantly, to determine their role in the nucleation and growth of CVD diamond films.

2. EXPERIMENTAL

2.1 SPECIMEN PREPARATION

The deposition of free-standing CVD diamond was performed at NIST. A commercial silicon wafer 76.2 mm in diameter and 400 micrometers thick, polished on one side, was cut into square substrates 19x19 mm each. An indentation 1 mm in diameter was ground into the center of the unpolished side of each substrate. The depth of these indentations was approximately half the thickness of the wafer. The smooth side of each substrate was manually polished with 1 micrometer commercial diamond powder. The manual polish was necessary to increase the nucleation site density and thus to deposit diamond films with small grain sizes.

The procedure for the manual polishing is as follows: Diamond powder is sprinkled onto a large glass plate and then pressed into a "pancake" between two glass plates. The silicon substrate is placed, polished side down, on the diamond powder "pancake" and rubbed manually for several minutes in a circular motion. If the diamond "pancake" breaks apart it must be pressed again. Fused silica or other optical glasses can also be prepared for diamond deposition in this way. It is interesting that the use of finer diamond powder such as a 0.125 micrometer size has little effect on the diamond nucleation density. Diamond films were grown on the prepared silicon substrates in a microwave-plasma-assisted chemical vapor deposition (MPACVD) system.

The deposition conditions were as follows:

Microwave power - 1kW
Graphite susceptor temperature - 650 ^{o}C (the temperature of the growing film is known to be considerably higher as a result of the heating in the microwave plasma).
Gas pressure - 6.6x10^{3} Pa
Gas flow rate - 260 standard cm^{3}/min;
Gas composition - 99.5% H_2 , 0.5% CH_4
Deposition time - 45 min
Growth rate - 0.4 micrometers/hour
Total thickness - 0.3 micrometers

Optical reflectance measurements suggest that the root mean square surface roughness of these films is approximately 0.02 micrometers. Following deposition, the substrate was etched away chemically and squares 3 mm on the side, were cut from the free standing film. The diamond squares were then placed between electron microscope grids and thinned by ion milling.

2.1 ELECTRON MICROSCOPY

A high resolution electron microscope at Oxford university, England was used in this study. The microscope features an objective lens spherical aberration of C_S=0.9 mm and it operates at 400 kV. Its point resolution is 0.16 nm giving accurate transmission of 111 and 200 diamond reflections into the image. The information limit is 0.12 nm so that 220 reflections also contribute to the image contrast. The crystals were tilted into an exact <110> orientation for high resolution image recordings and a 500k magnification was used close to the Scherzer defocus (-48 nm).

3. RESULTS AND DISCUSSION

3.1 TWINNING ELEMENTS

Twins have been observed in natural, artificial and thin film diamond layers. These twins are of the {111} type and in all cases they form during the crystal growth (rather than deformation twins). The important twinning planes and directions in diamond are:

K1 - the twinning plane or the shear plane. It is the first undistorted plane.

η_1 - the displacement direction contained in K1. All distances and angles are left unchanged by the twinning displacement.

K2 - the second undistorted plane.

K'2- the K2 plane after twinning in the twin coordinate system.

α - the angle between K1 and K2. This angle is not changed by the twinning and is related to the displacement strain.

η_2 - a vector in the K2 plane that is at angle α from K1.

η'_2- the same direction as η_2 on K2 in the twin coordinate system. This direction is the only direction on K2 that makes the same angle before and after twining with any direction in K1.

3.2 TWIN ANALYSIS

A high resolution micrograph of a twinned region is given in figure 1. The twining plane K1 in this example is (111). The rest of the twinning elements are:

K2 - (111)
η1 - [112] } all in the matrix coordinate system.
η2 - [112]

K'2 - (111)
η'2 - [112] } in the twin coordinates.

This is illustrated in the double stereographic projection, figure 2, in which the matrix planes (and directions) are marked ● while those of the twin are marked ○ .

The twinned region in figure 1 has a high volume fraction of twins. This is not usually the case in other regions examined but it clearly indicates that the {111} twins can be readily formed and that the orientation of the crystal can switch back and forth with little energy penalty. The stacking fault energy and thus the twinning energy is relatively small in diamond.

A more complex twin structure is shown in figure 3. The zone axis of the matrix (marked M) is [110] and (111) twins are observed in the upper portion of the micrograph (the twin boundaries are marked ►. A (111) stacking fault (marked ►►) can be seen in the upper left corner. Another boundary (marked *****) cuts across the micrograph from the left center. This one progresses along a (111) plane part of the way thus forming a $\Sigma=3$ boundary and then changes direction onto a non-crystallographic surface. The fit along this boundary is not as good as along the twin boundary as manifested by the preferential etching at the edge of the foil by the ion beam during the preparation process. We will further discuss this type of boundary later in this article and refer to it as misfit boundary.

The lower part of the micrograph consists of another set of twins, one of which (marked m) is identical in orientation to the upper matrix. This part also contains a periodic set of faults running along the (111) planes of the matrix. This type of contrast has been observed in other portions of our diamond films. The faults are about 0.6 nm apart and the contrast they display may be attributed to an inclined twin boundary (overlapping {111} twins) in which every 4th (111) plane has overlapping "atom pairs".

Another example of twin formation is shown in figure 4. Four twins can be seen interacting in the lower part of the micrograph. The twins are marked T1, T'1, T2 and T3. The {111} twin boundaries are marked ► ◄ . The orientation of the T1 twin is indicated and its zone axis is [110]. As can be clearly seen, the following twin pairs match well: T1-T3, T1-T'1, and T2-T'1. All of these twin boundaries are of the $\Sigma=3$ type [8]. However, there is a mismatch at the boundary between T2-T3 (marked * *). The boundary along which T2 and T3 intersect (A-C) is thus non-crystallographic (or very close to (221) of T2, a stable surface in the diamond structure) and can run along a surface anywhere between D-C and B-C which indicate the positions of {111} planes in T2 and in T3. The mismatch angle in this case is: $\delta=38.9$ which is the angle for a $\Sigma=9$ boundary and can be measured approximately from the micrograph. The other portion of the micrograph contains a set of twins and stacking faults on the (111) plane, which is the major twinning plane (K1) for the region shown. Only T3 does not contain this plane.

3.3 DIFFRACTION FROM TWINS

A diffraction pattern from a twinned region and its analysis are shown in figure 5. A double stereographic projection which represents it is shown in figure 6. The matrix and twin diffraction spots are marked ● and ○, respectively. There are a large number of forbidden reflections that appear in the pattern (see footnote - the structure factor). These are double diffraction spots generated at the twin - matrix boundaries [9]. The streaks that appear in the pattern originate from narrow twins and indicate their twin plane normals.

3.4 MULTIPLE TWINS AND MISFIT BOUNDARIES

Grains that contain multiple twins have been observed in materials having the diamond lattice. Several examples include silicon [10-12] and diamond [13]. In many cases the diamond particles possess an icosahedral shape and numerous such examples can be observed by scanning electron microscopy. However, the formation of five twins which touch at a point in the planar arrangement and along a line in the [110] direction in space can be a local phenomenon that does not create an icosahedrally shaped crystal because other twins divert the growth direction during the continuing growth of the crystal.

One such example is shown in figure 7. In this case five twins meet at a point and the formation of the 7.5° misfit boundary is clear, as illustrated in the insert. Such a misfit boundary can stretch to the surface of the crystal or can be arrested locally at another twin boundary as shown in figure 8. In all cases the edges of the misfit boundary will end either at a twin boundary or at the surface of the crystal as in the case of other grain boundaries.

The solution of the structure factor (F) equation gives the reflection intensities of the diamond structure as a function of the atomic scattering factor f:

$|F|^2=32f^2$ when (h+k+l) is odd
$|F|^2=64f^2$ when (h+k+l) is an even multiple of 2
$|F|^2=0$ when (h+k+l) is an odd multiple of 2

Even though misfit boundaries are always formed when 5 twins meet at a point, such a misfit boundary can also result from the formation of a smaller number of twins as seen in figure 9. In this case the mismatch boundary forms between two twins and extends from one triple point to another. Misfit boundaries are usually found near the periphery of the crystal where the number of twins and their density is high (figure 10).

Misfit boundaries provide a key to understanding the growth of diamond CVD crystals. This is because misfit boundaries are the locus of the points of intersection of growing planes from two adjacent non-matching twins. The boundaries are aligned, therefore, along the local growth direction of the crystal and allow the determination of the growth direction at various points of the crystal as well as the nucleation point of the growing planes in the TEM cross-section.

An example of such a determination is shown in figure 11. A complete cross-section of a crystal is shown and the growth directions are marked by arrows along mismatch boundaries. The nucleation point of the growing planes in this cross section can be traced back to the 5-fold twin site (marked by a circle). The importance of the 5-point twin site to the nucleation and growth of CVD diamond crystals will be discussed next.

3.5 THE ROLE OF TWINNING IN THE GROWTH OF CVD DIAMOND

A large number of diamond grains have been investigated in the course of our study. In all cases the grains were twinned and the distribution of the twins was irregular. Since even small regions contain twins and multiple twinning is very common, it seems that twins play an important role in the growth of CVD diamond crystals.

The importance of twins to the growth of germanium has been studied in the past and consequently utilized for the growth of germanium dendrites from the melt [14-18]. The twin boundary reentrant angle model of Hamilton and Seidensticker [16] has shed light on the vital importance of twinning in the growth process of germanium. The model for fast growth calls for a pair of parallel twin boundaries which form a new reentrant angle as one twin boundary ceases to serve as a preferred growth site. In the case that only one twin boundary is present, a bicrystalline trigonal solid is formed provided that all three reentrant corner sites are allowed to grow.

While twins serve a crucial role in the growth of germanium crystals, this does not seem to be the case in the growth of silicon crystals. Twinned silicon crystals have been grown from the melt [19] but it is not clear that twins cause fast growth as in the case of germanium. In other metals and intermetallics, twins serve as a preferred site for the growth of dendrites. One example is the growth of cadmium crystals [20]. However, we will not discuss this case but limit ourselves to crystals with diamond cubic structure.

A key to understanding the role of twinning in the growth of diamond crystals in the CVD process is the stability of a carbon atom attached to the surface of a growing crystal at a given point. During the formation of natural diamonds (see for example [21]) or in commercial processes in which the pressure-temperature are such that the diamond phase is stable, growth does not occur in a reactive environment as in the case of CVD growth. A carbon atom attached to a free surface of the growing crystals under equilibrium growth conditions is relatively stable and can stay in position, thus forming a preferred site for the formation of a new {111} plane. In CVD diamond growth, such an atom will be likely to etch away by the atomic hydrogen. Carbon atoms which position themselves in a reentrant angle site will be more stable. When a new plane forms at the reentrant site it can grow fast since there are stable positions at the step site of the propagating plane. The reentrant site between twins can shoot out propagating planes at a fast rate which controls the local growth rate (figure 12). The local growth is on adjacent {111} planes but on a larger scale the crystal grows in the <211> directions. The best demonstration of the effect of twinning on growth in diamond cubic structure is given by Hamilton and Seidensticker [16].

A clear example of the Hamilton Seidensticker mechanism can be seen in figure 9. The part of the crystal marked T1-T2-T1 is the shape that can grow indefinitely in a lath form. To obtain fast growth in other directions more twins on other {111} planes are needed. The growth direction in this part of the crystal is marked by hollow arrows on T1 and on T2 and the intermediate position of the edge of the crystal is marked by dark arrows. The new (111) plane will start to form in point [A] and the growth continues in both directions as marked by the dark arrows along the {111} planes.

Prior to this point, a misfit boundary has formed at [B]. The formation of such a misfit boundary was discussed earlier in this report and the "v" shape with an angle of 70.5^0 between {111} planes indicates the growth direction at that point (the crystal grows from the tip of the "v" upward). The misfit boundary that starts at [B] propagates further into the periphery of the crystal along a path marked by * * * *. It is important to note that the misfit boundary propagates partly on a non-crystalline path but mainly along {111} planes. This point was also illustrated in figure 3.

The common axis of 5-fold twins is an important nucleation site for new planes along the growing crystal. This is mainly due to the five reentrant angles around the center (see figure 7). The maximum number of twins that can form around a point is twenty and an icosahedron forms as they grow. Such an arrangement of twins provides 30 reentrant angle sites and can serve therefore as a superior nucleation site for the whole crystal.

This may explain why many of the observed CVD crystals are icosahedral in shape. Following our proposed growth model (see figure 12) it is expected that the vertices (5-fold axes) and in certain cases the edges (2-fold axes) of such twinned icosahedrons will be grooved since they provide the nucleation sites for the new planes. Indeed, this is the case as illustrated in figure 13. In fact, such grooves are expected to form also in cases when only fractions of the icosahedron develop.

4. CONCLUSION

This study by high resolution electron microscopy of plasma assisted CVD diamond films has centered on the determination of twinning parameters and the role of twins in the growth of diamond crystals under the characteristic metastable conditions typical to the CVD technique. It was shown that the various configurations of twins can indicate the growth direction in certain parts of the diamond crystal, thus helping in the determination of the origin of the growing planes in various cross sections of the crystal. It has also been shown in this article that the reentrant angle between twins is a preferred nucleation site for growing planes and that a twin quintuplet is thus a superior growth center. The icosahedral shape of a large number of twinned diamond crystals and the grooves along the vertices (5-fold axes) and along the edges (2-fold axes) are explained based on the findings of this study and are consistent with an icosahedral cage compound precursor nucleation site [22].

5. ACKNOWLEDGEMENT

This work was supported in part by the Office of Naval Research.

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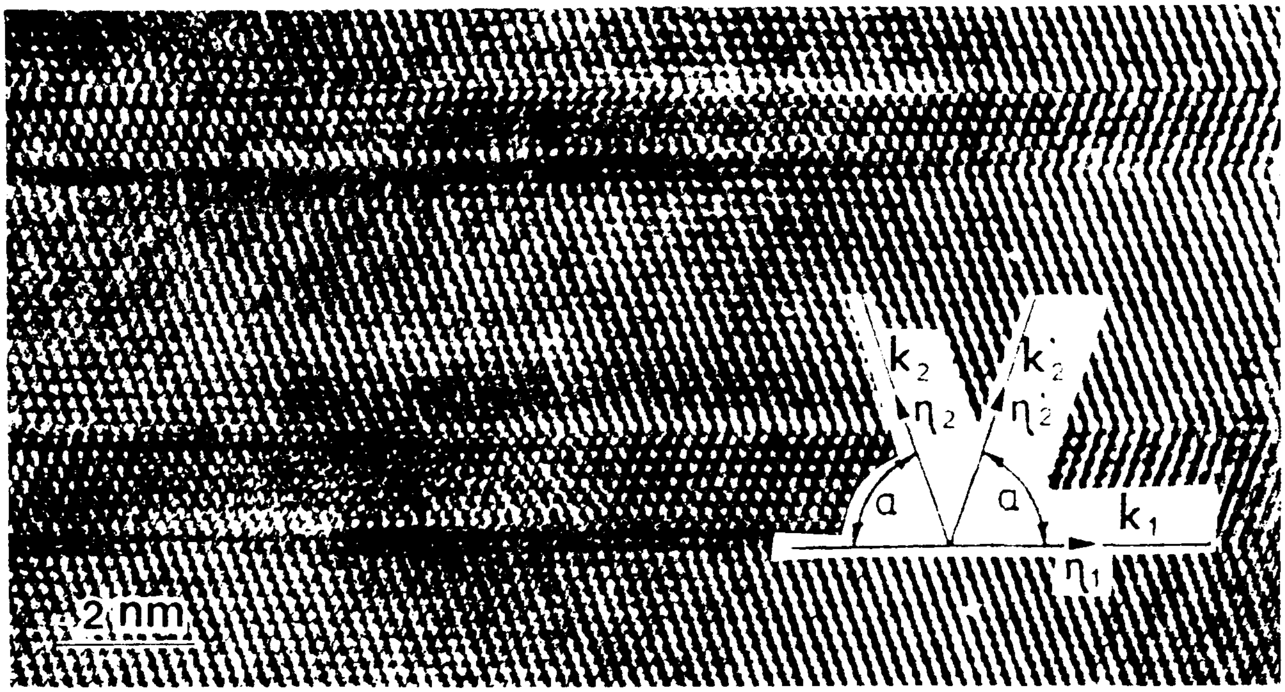

FIGURE 1 A twinned region in the diamond foil. The main twinning elements are marked on the micrograph.

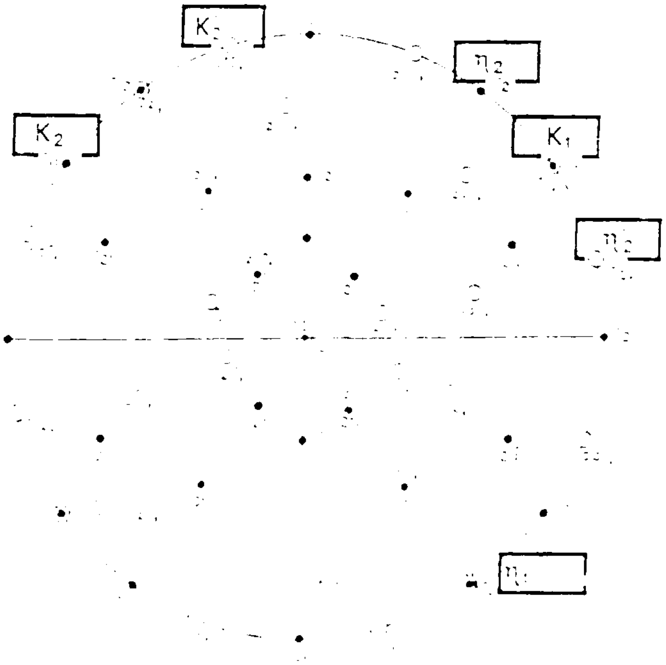

FIGURE 2 The twinning elements of the twin shown in figure 1.

FIGURE 3 Twin boundaries (case 1).

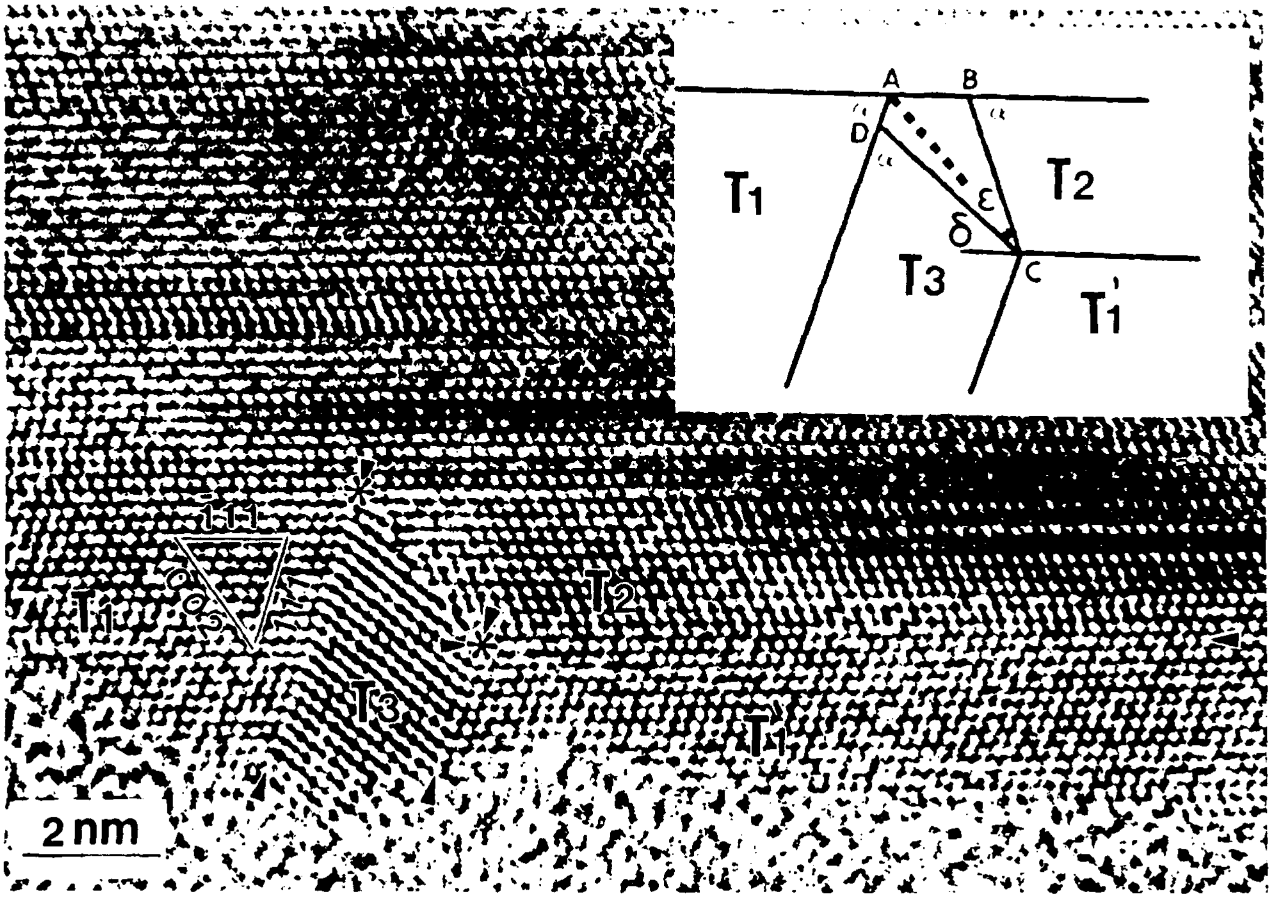

FIGURE 4 Twin boundaries (case 2).

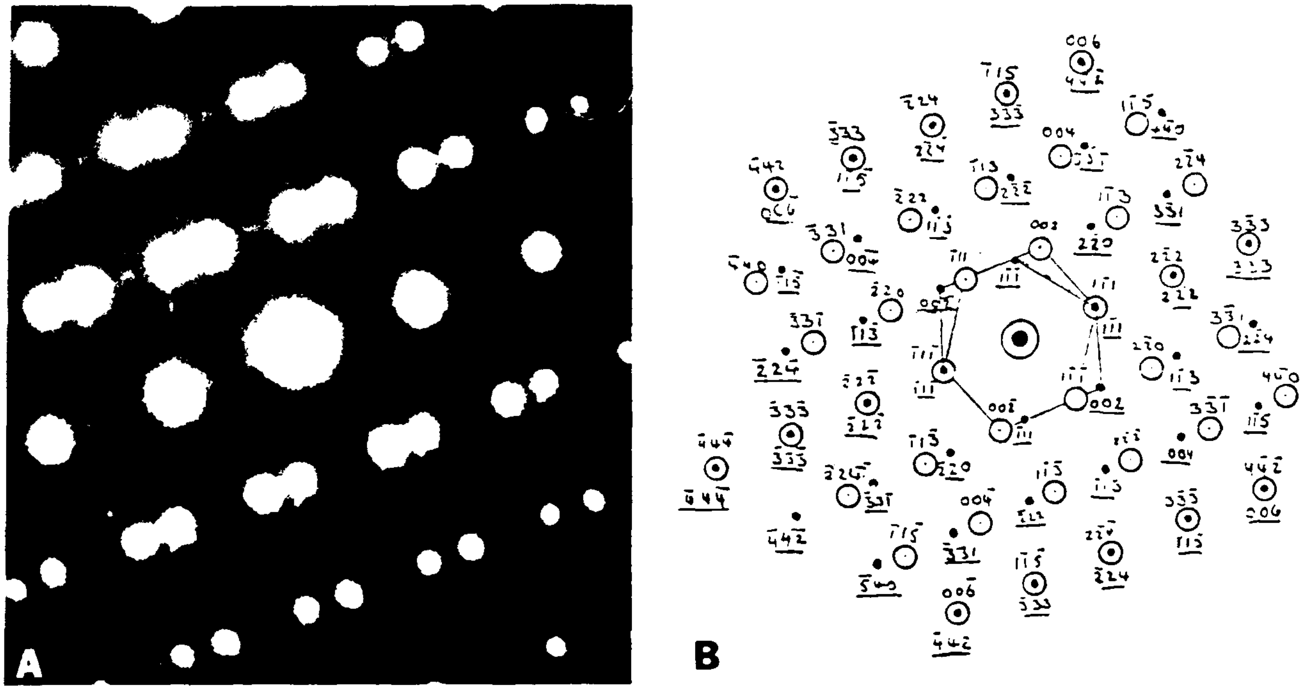

FIGURE 5 A diffraction pattern from a twinned part of the crystal [A] and its analysis [B].

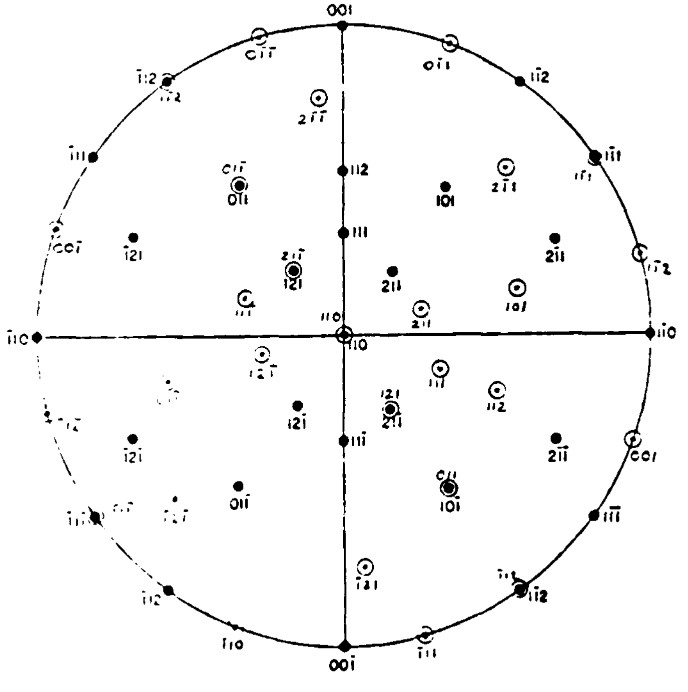

FIGURE 6 A double stereographic projection representating the diffraction pattern sown in figure 5.

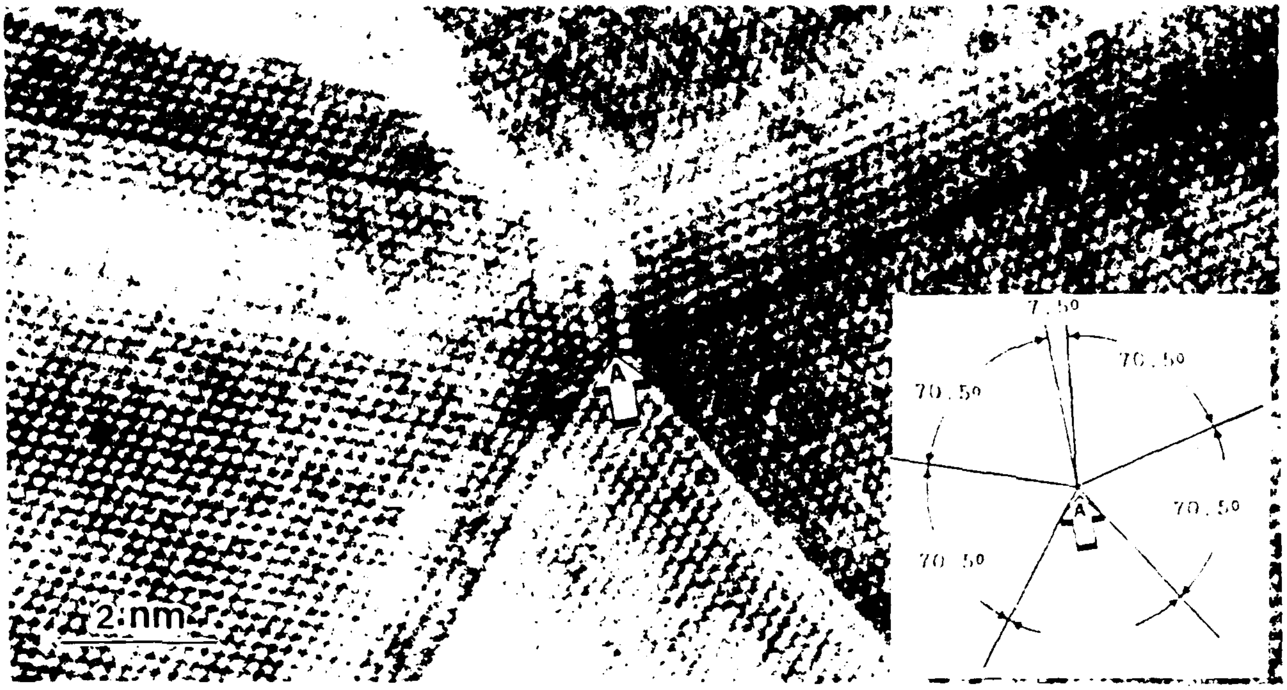

FIGURE 7 A twin quintuplet (marked A) creates a 7.5° misfit boundary along one of the twin habit planes (see insert). the misfit boundary geometry and importance are discussed in the text.

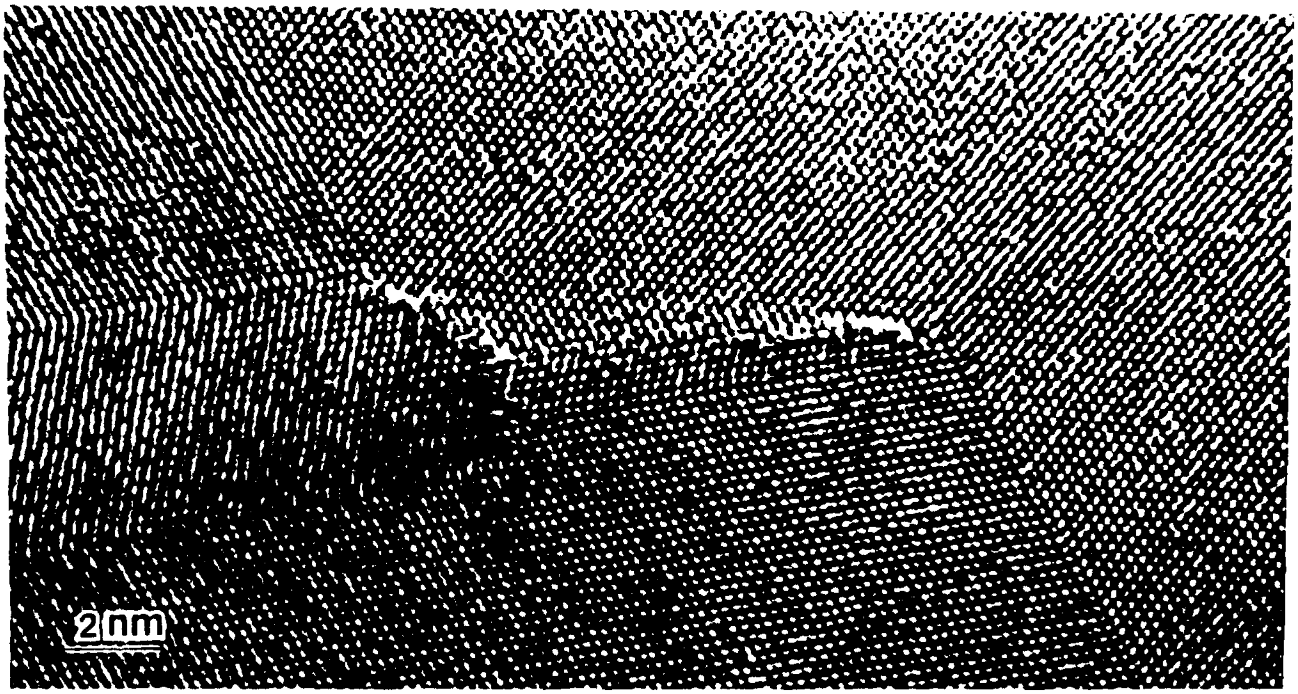

FIGURE 8 Misfit boundaries can terminate at twin habit planes (as shown in this example) or stretch to the surface of the crystal as shown in figures 9 and 11.

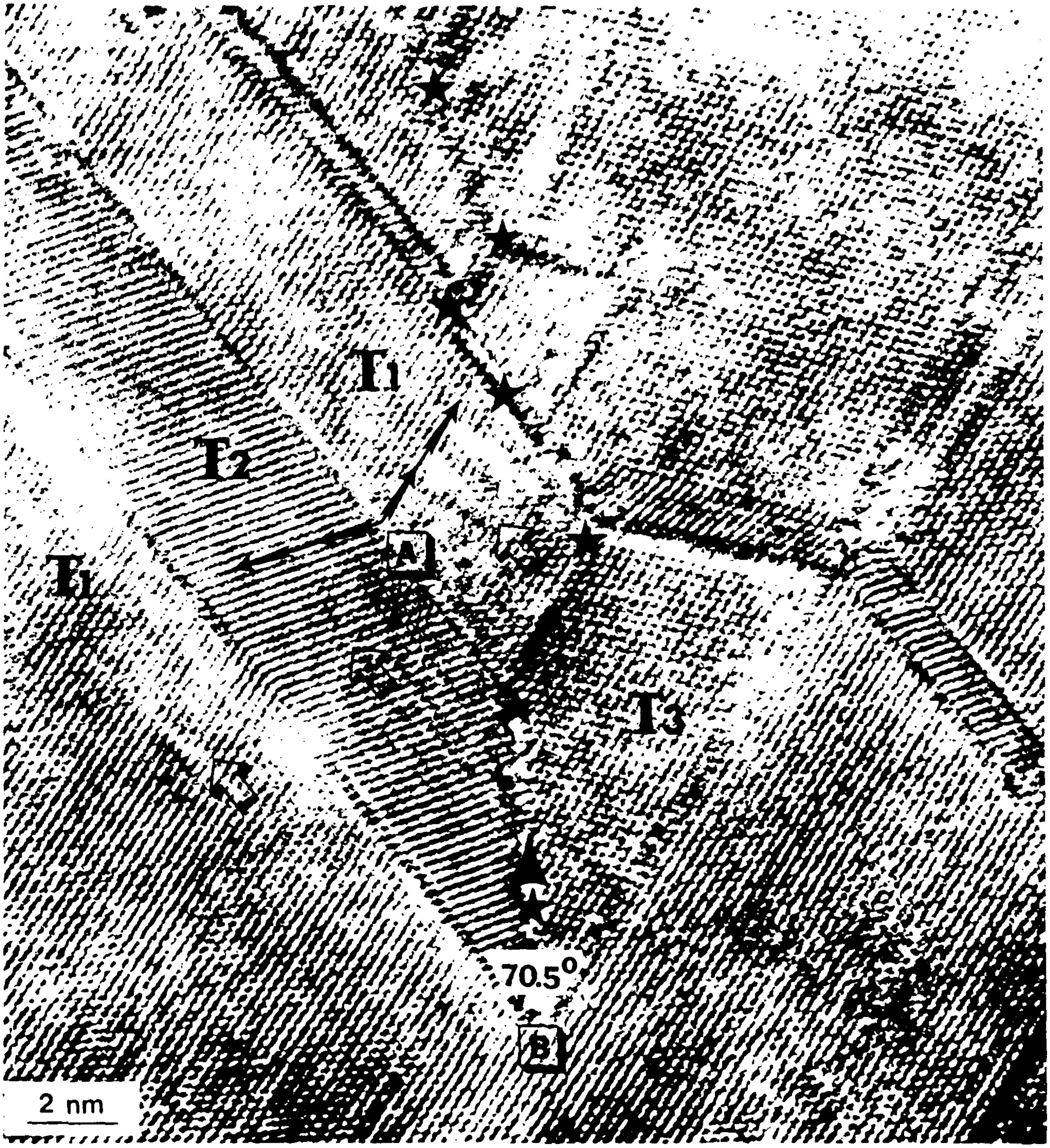

FIGURE 9 The misfit boundary at [B] between the "V" shaped T_2 and T_3 forms on a surface which is the locus of interaction points of growing planes on two adjacent non-matching twins. It thus indicates the crystal growth direction in that vicinity (marked by a dark arrow). This misfit boundary propagates in a path marked by * * * * . The reentrant point at [A] serves as a preferred nucleation site for a new plane which grows as indicated by small dark arrows along T_1 and T_2. As new planes are added the twins grow in the direction marked by hollow arrows.

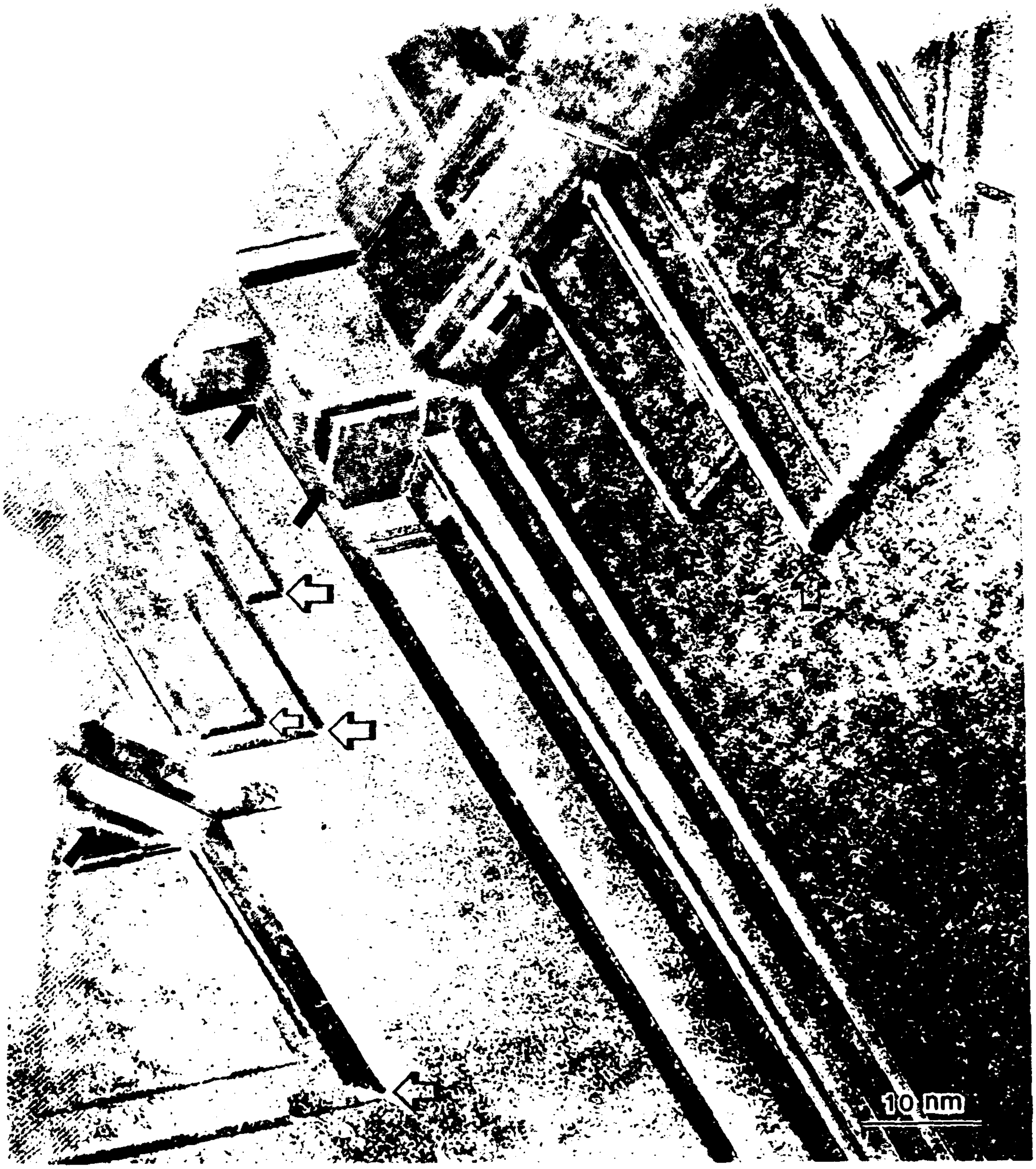

FIGURE 10 Misfit boundaries (all arrows) are more frequently found in the periphery of the crystal. Hollow arrows indicate also the local growth direction of the crystal.

FIGURE 11 Growth directions, marked by arrows in this cross section of a complete crystal can aid in tracing back the nucleation site of the growing planes to the twin quintuplet marked by a circle.

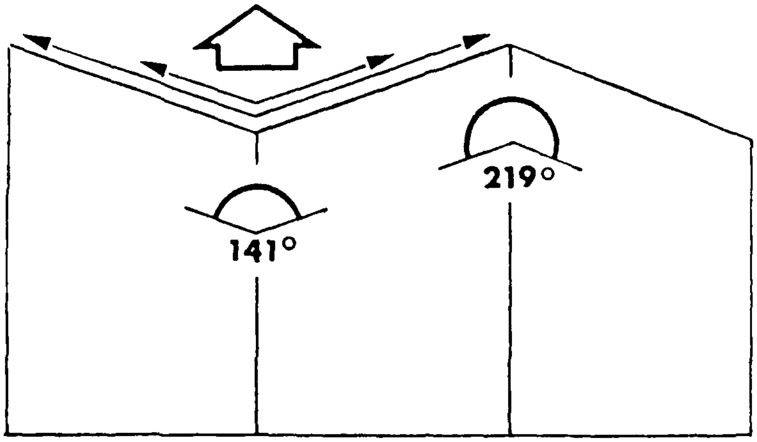

FIGURE 12 The growth of a (111) plane starts at points along the 141° reentrant line.

FIGURE 13 The grooves along the 2-fold edges and the indentation in the 5-fold vertices result from the growth mechanism described in the text. The grooves are commonly found along the intersection lines of twin habit planes and the surface of the growing diamond crystals.

6 March 1991

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