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Plastic deformation in natural diamonds: Rose channels associated to mechanical twinning

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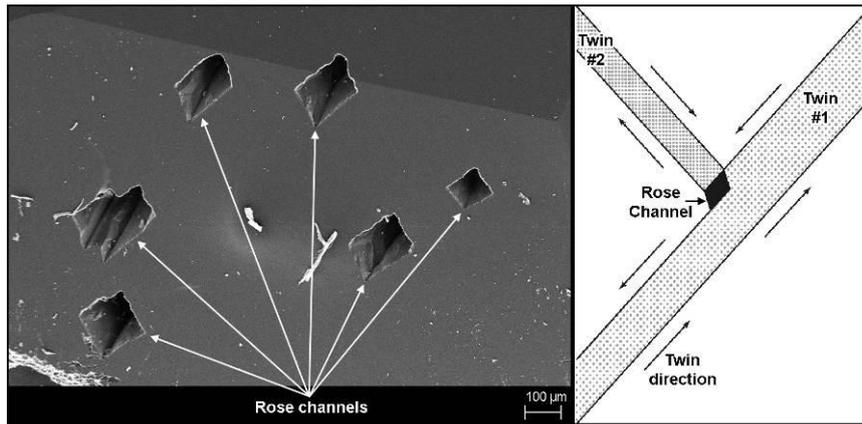
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Abstract

Hollow channels in diamond are well acknowledged to be the result of dissolution processes. In this article we demonstrate that some hollow channels in natural diamonds are the consequence of intense plastic deformation by mechanical twinning. Two mixed-habit diamonds presenting numerous geometrical hollow tubes were studied. X-ray Laue analyses showed the presence of microtwins. At the intersection of microtwins, displacements and cracks are generated, creating the hollow channels observed. The presence of the cracks seems to have released the internal stress, as there was less to no signs of deformation at and around them. Further dissolutions are sometimes but not always seen within the cavities. Mechanical twinning, so far mostly identified in pink to purple diamonds, might be more widespread than originally thought in natural diamonds.

Graphical Abstract

Macroscopic voids ($\geq 100 \mu\text{m}$ in diameter, and sometimes $\geq 1 \text{ mm}$ long) are observed in some mixed-habit growth natural diamonds. The voids are created at the intersection of two twins and are interpreted as Rose channels. This figure shows cross sections of the Rose channels by scanning electron microscope imaging and their interpretation.



Keywords

Plastic deformation, natural diamond, Rose channels, mechanical micro-twin, high pressure high temperature cracks.

Introduction

Narrow and hollow channels are uncommon defects in natural diamonds. They were first observed and sketched by Orlov [1] on Russian diamonds. Since that work, there has been little research on channels, most of which were mainly descriptive [2-8]. The main conclusions are that these open tubes occur in diamond from various geographic localities and that they are found in both types I and II diamonds.

The most complete work was reported by Lu et al. [8]. This study analyzed channels in seven natural diamonds. The channels were examined with optical microscopy, scanning electron microscopy (SEM), ultraviolet (UV) fluorescence images and UV-Visible (Vis) and Infrared (IR) (300-850 nm) spectroscopies. The authors noticed that channels were elongated parallel to the $\langle 110 \rangle$ directions. From their observations on surface appearance, elongation orientation, internal microstructures and relationships between the channels and other lattice defects they concluded that the channels were due to dissolution processes. They proposed that the formation of etch channels is most likely related to crystal defects like dislocation bundles perpendicular to $\{111\}$ planes or dislocation dipoles elongated along $\langle 110 \rangle$ directions. Let us recall that, for a long time, growth dislocations and dislocation bundles in diamond have been revealed by X-ray diffraction topography [9, 10].

Moreover it is well acknowledged that most natural diamonds have undergone one or more processes between their nucleation and their transport at the surface of the Earth. The most common are dissolution processes which leave etched trigonal depression figures on the $\{111\}$ octahedral faces and which also give rise to dissolution crystal habits with many rounded faces (like pseudo-dodecahedral or even pseudo-trioctahedral shapes) [11-13].

Later reports on hollow channels agreed with these conclusions. So far, it is well admitted that they are systematically etch channels induced by dislocations [16].

Materials and Methods

We have examined two crystals that exhibit channels visible macroscopically. Both specimens were acquired from the gem market and are reportedly from Zimbabwe. They were purchased already sliced and polished to show off a grey three-fold star.

Sample MSC19 consists in a thin slice with two large polished faces and additional smaller facets a little inclined, 10 to 16° from the upper large face (Fig. 1). It measures approximately 5 by 6 mm and 0.39 mm thick.

Sample MSC67 is cut the same way and measures 7.5 by 8.5 mm and are 0.45 mm thick. This sample was not characterized by X-ray.

The crystals were first observed with an optical microscope Leica DM 2500P, equipped with polarizers, coupled with a camera.

Transmission infrared spectra were acquired using a Bruker Vertex 70 Fourier-Transform infrared (FTIR) spectrometer, with a spectral resolution of 4 cm⁻¹, accumulating 100 scans per spectrum.

High resolution surface images were taken with a Scanning Electron Microscope (SEM) FEG Zeiss Ultra 55 of the Institut de Minéralogie, de Physique des Matériaux et de Cosmochimie, IMPMC Laboratory in Paris. Accelerating voltage varied between 5 and 15 kV and the samples were cleaned and carbon-coated before analyses.

Structure studies were conducted with an Xcalibur E (Agilent Technology) single-crystal diffractometer with Mo wavelength (0.71073 Å). The beam diameter used was 500 μm. The data were analyzed with CrysAlis Pro software.



Fig. 1: Images of sample MSC19, a mixed-habit diamond (the diameter is close to 6 mm). Notice the dark geometric channels throughout the sample. This diamond has mixed-habit growth, with grey cuboid sectors and colorless octahedral sectors.

Results

Both diamonds are type IaAB, with high concentration in nitrogen, mostly in the form of A-aggregates; they are also rich in H. Both are natural mixed-habit diamonds. They show the typical colorless sectors (octahedral growth) and brownish to grayish sectors (cuboid growth) in a three-fold symmetry (e.g. [14, 15]). The cuboid areas contain high density of **blackened graphitized** disk-crack-like defects **that appear black, which might be responsible for giving rise to** the overall gray color of these sectors. In this paper, we will focus on the large dark needle-like defects visible macroscopically in both cuboid and octahedral sectors. Under optical microscope, they look very similar to the previously described hollow channels [1-9]. Some of these defects reach the surface of the polished diamonds. The cross-sections of these

channels have the shapes of quasi-rhombs or quasi-parallelograms (Fig.2). The rhomb acute angles are close to 60° . These angles agree with channels with square (or rectangular) perpendicular section. The inner wall orientations agree with $\{100\}$ orientations. Orientations of the channels deduced from the X-ray Laue method show that they are near the $[hkk]$ directions. Additional measures with a goniometric device (Fedorov's four-axis universal stage) and geometrical measures agree with directions close to $\langle 100 \rangle$.

Examination under crossed polarizers revealed abnormal birefringence throughout the diamonds, especially around the disk-crack-like defects. Abnormal birefringence is always present in natural diamond, and represents remaining strain [e.g. 17]. Interestingly, no (to little) abnormal birefringence, therefore no remaining strain, was observed in the vicinity of the geometrical channels (Fig.3).



Fig. 2: Photomicrograph in transmitted light of the channels and their rhombic shaped openings.



Fig. 3: Photomicrograph of a channel under crossed (linear) polarizers of sample MSC67. There is less to no abnormal birefringence next to the channels, while the rest of the diamond is strained (especially around the disk-crack-like defects).

Thanks to the use of SEM, high resolution images of the channels were obtained. They revealed that the channel sections are a little concave (Fig.4). In specimen MSC1, the inner walls are smooth even under a field of view of about few dozens of microns the highest available magnification. In specimen MSC67, some dissolution figures in the inner walls exhibited a four-fold symmetry which confirmed that inner walls orientations are close to $\{100\}$ orientations (Fig.5). Moreover the channels are systematically observed at the intersection of the walls forming a 60° angle: they also reveal discontinuity at the intersection of two walls (when dissolution figures are present).

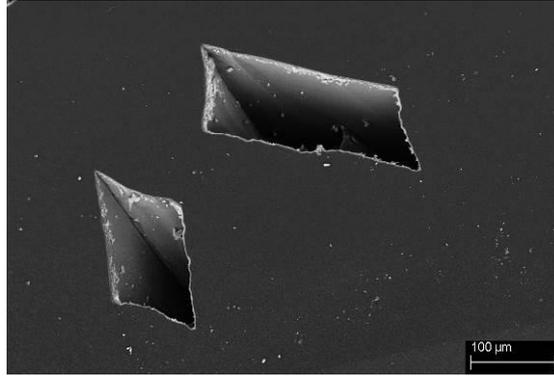


Fig. 4: Close view by SEM of the channel openings of sample MSC19. Notice the sharp concave shapes. The inner walls are smooth (even at a 10 time higher magnification). Notice the crack at the crossing of two inner walls (forming acute angle).

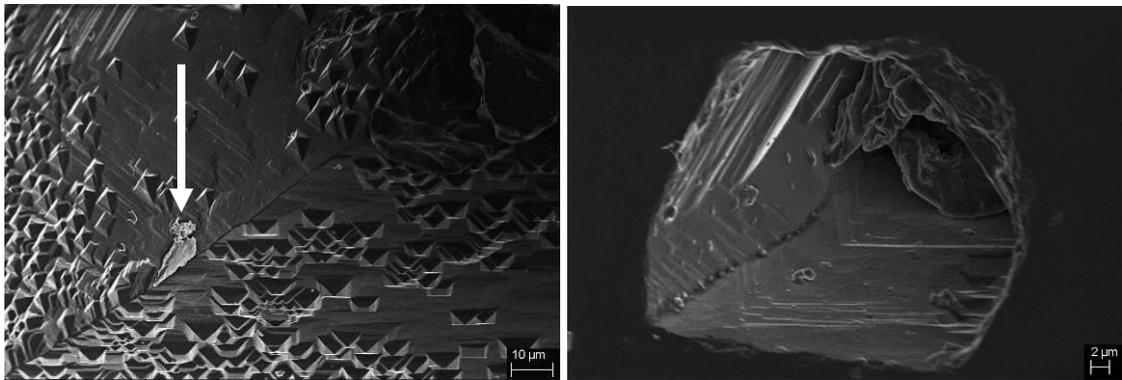


Fig. 5a and b: SEM images of two different channel interiors in sample MSC67, showing dissolution figures and a crack between the two channel walls.

A precise measure of crystallographic orientations was performed by X-ray Laue method on sample MSC1. The two large polished faces are 5° inclined from (111) face and a [110] direction is detected. Several angular scanning were performed near the center of the crystal. They did not give rise to the expected cubic cell with $a_{\text{cub}} = 3.567 \text{ \AA}$ at room conditions. The analysis of another area gave a hexagonal cell with $a_{\text{hex}} = 2.53 \text{ \AA}$ and $c_{\text{hex}} = 5.8 \text{ \AA}$. These values are close to the value $a_{\text{hex}} = 2.527 \text{ \AA}$ and $c_{\text{hex}} = 6.186 \text{ \AA}$ expected for {111} microtwins with 50%-50% percentage of both orientations [18]. The c_{hex} difference is due to difference between both orientation percentages. Similar anomalous cells found in other areas are most likely due to the presence of more complicated microtwins (there are 4 directions of diamond twins).

Discussion

Concave prismatic sections, acute angles and smooth walls observed on specimen MSC1 are in disagreement with a dissolution process proposed previously for similar features. Indeed, since the nineteenth century it was well-known that the dissolution process is more efficient on sharp parts of crystal like vertices and edges [19]. It is known to produce rounded convex shapes. Moreover dissolution figures should systematically be seen on the walls, which is not

the case for specimen MSC1. It is clear that the etch hypothesis related to dislocations or intrinsic structural channels fails to explain our observations and measurements. Another hypothesis could be that the channels come from the dissolution of preexisting foreign crystals included in the crystals. Some pyroxenes have an elongated prismatic shape with quasi-square sections. But here again the former presence of euhedral inclusions cannot explain the observed concave cross-section and the cracks.

Other explanations for hollow channels are less known. They are found for crystal deformations and more specifically deformation by mechanical twinning. Since the 19th century and the works of Mügge [20], it is known that translational glide (slip) and mechanical twinning are two main modes of plastic deformation in minerals. Ideally these deformations involve shears that preserve or reproduce the crystal structure. In the case of slip there is no change of the crystal orientation. In the case of twinning there is a change of the crystal orientation (at least a two-fold axis). The shear belong to a plane $K_{(hkl)}$ and the lattice points on one side of this plane are displaced by vector $g_{[uvw]}$ parallel to $K_{(hkl)}$. The pair $K_{(hkl)}$ and $g_{[uvw]}$ constitutes the glide system. In slip, $g_{[uvw]}$ is named the slip direction and its modulus is independent of the distance from the slip plane. In the case of twinning, the modulus of $g_{[uvw]}$ is proportional to the distance from the twin plane $K_{(hkl)}$ [21].

Twins are well known in diamond. Twinning and polysynthetic twins in natural diamonds was proposed since the early 20th century (see the study of von Fersmann and Goldschmidt [22] and references therein as well as [23] and [24]). This hypothesis was based on observations of bands on the rounded dissolution surfaces of crystal faces. The bands are always parallel to the octahedral $\{111\}$ faces. Later microtwins (lamellae) have been reported repeatedly using optical microscopy, but many X-ray diffraction studies (using Laue technique) did not reveal twins (see [25] and references therein). Microtwins have been suspected by Mineeva et al. [26] who noticed unusual electron paramagnetic resonance (EPR) spectra in pink-purple diamond from the International'naya kimberlite (Siberia, Russia). Later Titkov et al. [18], using single crystal X-ray diffraction techniques on diamond of same origin, detected a hexagonal symmetry, which is expected for (111) microtwins, with a Friedel coincidence lattice index $\Sigma=3$ [27, 28]. The authors conclude that their results suggest that natural epigenetic plastic deformation in diamond not only occurs by dislocation slipping but also by twinning. Gaillou et al. [29] reported mechanical micro-twinning in a series of pink diamonds, identified by transmission electron microscopy (TEM) coupled with X-ray diffraction. Similar lamellae and "steps" at the edge of the rough crystals can be observed in the studies of Gaillou et al. [29] on microtwinned pink diamonds and of Lu et al. [8] on diamonds with the presumed etch channels. In the work of Gaillou et al. [29], samples presented showed only one direction of twinning. No channels were observed. In a later article, Gainutdinov et al. [30] even suggested that most "ridges" seen at the surface of diamonds were microtwins, based on shape observations of such features (~~no X-ray analyses provided to prove this assumption~~). These features were described as positive forms of surface relief up to 10 μm long and $< 2 \mu\text{m}$ wide.

Let us recall that the diamond twin can be described as a 180° (hemitropy) or 60° rotation perpendicular to a (111) face (it is often referred as spinel-law twin). In term of mechanical twinning, its glide system is $K_{(111)}$ and $g_{[211]}$. The $g_{[211]}$ minimum modulus (i.e. here the slip vector from one (111) plane to the next one) is equal to $a_{\text{cub}}/\sqrt{6}$. This translation vector has the effect of a rotation (it generates a local 6-fold symmetry).

Deformation and plasticity in synthetic diamond has been a subject of substantial interest since they were made in 1950's. Several mechanisms have been proposed (see [31] and references therein). The studies were conducted on diamond powders (not single crystals). Yu

et al. [31] reported experimental results with SEM and TEM at room temperature, 1000°C and 1200°C, under 3.5 GPa. They concluded the following:

- Deformation at room temperature is essentially brittle, cataclastic with fracturing on {111} planes.

- At 1000°C the deformation mechanism changes from brittle mode to ductile mode dominated by dislocation mediated plasticity.

- At 1200°C, the plastic deformation and ductile flow is mediated by the $\langle 110 \rangle$ {111} dislocation glide and a very active {111} microtwinning. These results do not support {100} cleavages that were revealed by Humble and Hinnink by indentation experiments [32].

In another interesting paper, DeVries [33] reported the graph with P and T conditions under which microtwinning could occur. In their experiments, the authors were using both natural and synthetic diamonds put under HPHT to draw this curve of field conditions, most likely representing the best work done on that matter. If such conditions could be created in a laboratory, under conditions similar to the ones that can be reached in the Earth mantle, it is indeed not surprising that such ways to accommodate deformation could be seen in natural samples.

In some cases mechanical twinning induces hollow channels. They are known as Rose channels. Rose channels were first observed in calcite by Brewster and Stoney who described them as fibers, striae or tubes ([34] and references therein). In 1869, Gustav Rose (1798-1873) interpreted these channels as intersections of non-parallel twin lamellae and verified that compressing calcite crystal along suitable crystallographic directions can easily generate them [34]. Later they have been observed in other minerals and in various metals ([21] and references therein).

Rose gave a detailed description on the formation of the channels. The most likely situation occurs when a twin lamellae (called the “crossing twin”) encounters a preexisting twin lamella (called “crossed twin”) [35-36] (Fig.6).

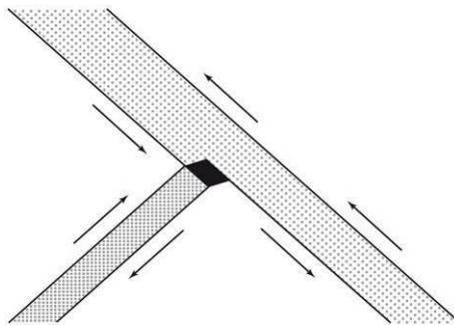


Fig. 6: When two twin lamellae meet, a hollow channel called Rose channel may appear (redrawn from [35]).

The hollow channels we have observed in the diamonds studied well agree with Rose channels. First of all, diamond MSC1 is proved to be microtwinned (hexagonal cell detected by single crystal X-ray diffraction). In diamond, there are four non-parallel {111} twin planes, so there is the potential for twin planes to intersect each other. Rose channels are compatible with the smooth inner walls. The observed cracks also agree with Rose channels [35] and also explain the absence of stress near these hollow channels.

Let us consider two {111} twinned lamellae. A priori the direction of the Rose channel is the zone axis of these lamellae. The channel inner walls are expected to be those that have the lower energy to be cleaved. The zone axis is in that case in a $\langle 110 \rangle$ direction. In diamond, the dense faces are {111}, {110} and {100}. According to first principles calculations of Telling

et al. [37], the energy ratio to cleave the {100}, {110} and {111} planes is 7 :2.8 :1. So the {111} planes are more readily cleaved.

In our case, we have $\langle 100 \rangle$ hollow channel direction and inner walls close to {100}. These facts suggest that the channels involve more than two mechanical twinings (lets us recall that in diamond, there are 4 {111} twin plane directions, 6 $\langle 110 \rangle$ and 12 $\langle 211 \rangle$ directions). The {100} inner walls agree with the Humble and Hinnink indentation experiments [32].

Nevertheless the concave walls indicate that portions of other faces may be present. It is confirmed in Fig. 7, where two neighboring channels are separated by a small {110} face portion.

Interestingly, the samples studied here not only display macroscopical but also microscopical voids. Indeed, “disk-like cracks” are observed in the cuboid sectors of the diamond.

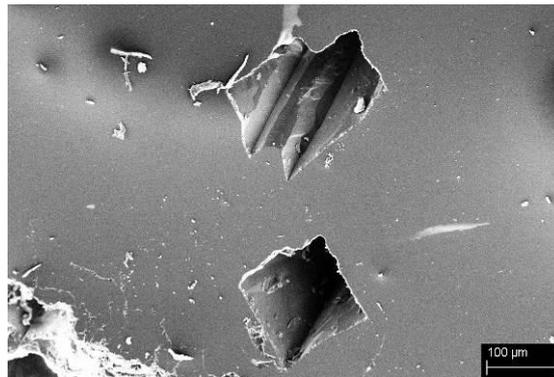


Fig. 7: A flat portion (close to (110) orientation) separates the two neighbor channels of sample MSC19.

~~Walmsley & Lang [14] showed that those cracks have a consistent diameter of about 1.2 μm, and each disk is surrounded by a stacking fault. The formation mode is not explained, but these disks contain high remaining strain, between 1.5 to 1.8 GPa. Gainutdinov et al. [30] even ascribed those disks to be microtwinned areas. This would be the subject of another study to confirm or invalidate, but the presence of those microvoids, with or without microtwinning, is coherent with the presence of the much larger Rose channels in the samples studied here.~~

Conclusion

The discovery of Rose channels in natural diamond gives rise to several conclusions:

- The etched hollow channels, mentioned in previous articles, need to be revisited. Indeed, the reported {110} directions agree with two twinned lamellae intersections, they are therefore most likely Rose channels.
- Mechanical twinning is demonstrated for the first time to be present in other diamonds than pink to purple diamonds. This phenomenon might be more widespread than originally thought. For example, we can raise the question if “graining” in the form of discrete thin deformation lamellae (that may be colored or not) could, in some cases, be related to deformation twinning.
- It is the first time that it is shown that macroscopic voids could appear in natural diamond even under HPHT (the mechanical micro-twinning is expected at HPHT).

Rose channels change the simple well-admitted scheme of diamond deformation with a low (temperature-pressure) range with brittle mode and another higher range with ductile mode. In other words, ductile mode can generate cracks in diamonds (even if room condition

experiments make it hard to believe that diamond could have deformation mechanisms similar to those of the soft calcite!). These channels open new fields of research on diamond deformations, and probably also on silicon deformation.

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We would like to dedicate this work to Professor Alfred Seeger who passed away on the 18th of October last year [38] and who was so rightly proud of the Rose scientific dynasty.

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