Revision 1

Positively-oriented trigons – a unique resorption feature of diamonds from Snap Lake kimberlite dyke, Canada

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Abstract

The role of fluid(s) in the formation of different lithological facies of kimberlites is still poorly understood. The ambiguity of composition of kimberlite melts hampers understanding the composition of volatiles, their depth of exsolution and the effect on magma ascent and fragmentation. Recent estimates of H₂O and CO₂ solubility in kimberlite-like magmas suggest very shallow exsolution of fluid, while many features of kimberlites indicate the presence of significant fluid fraction at depth. Deep magmatic fluid produces negative trigonal etch pits on natural diamonds, the characteristics of which depend on the temperature and composition of the fluid. Positively-oriented trigonal etch pits are very rare on natural diamonds, and are likely a feature of resorption events unique to only some kimberlite magmas. Here we present the first systematic...
study of positively oriented trigonal etch pits on natural diamonds from Snap Lake kimberlite dyke, Northwest Territories, Canada. The study used 91 micro-diamonds selected from a population of 251 diamonds representative of all six kimberlite litho-facies identified in the Snap Lake dyke. We established that unlike the majority of diamonds from kimberlite pipes in the Northwest Territories, every studied Snap Lake diamond shows positively-oriented trigons. These trigons cover the whole diamond surface starting from the \{111\} faces and continuing over the resorbed edges. They overprint negatively-oriented trigons and modify them into hexagons. Atomic force microscopy obtained detailed geometry of 154 positive trigons on fourteen diamonds. Three distinct trigon morphologies dependent on the type of the crystal lattice defect were recognized. The point-bottomed shape and positive correlation between the depth and diameter of the individual pits suggest a high CO\(_2\) content in the fluid. Comparison with the existing experimental data on positive trigons implies resorption at low-pressure conditions in the 800-1000\(^\circ\)C temperature range by trapped magmatic fluid after the dyke emplacement. The intensity of this late resorption event (and the size of the positive trigons) increases from the dyke contact with the country rock into the interior of the dyke. Such a late resorption event is absent in the majority of kimberlites, which form pipes, and might be a specific feature of hypabyssal kimberlite bodies (dykes). The absence of positive trigons on diamonds from the majority of kimberlites suggests very quick magma cooling below \(~\)800\(^\circ\)C after the pipe emplacement, precluding the development of any late resorption features. Our study shows that for kimberlitic magmas, morphological details of dissolution features on the surface of diamond and other mantle-
derived minerals can serve as a fluid proxy. Better constrains of the pressure, temperature
and oxygen fugacity of the reversal in the trigon orientation on diamond may help to
reconstruct the emplacement path of geologically diverse kimberlite bodies.

Keywords: diamond resorption, kimberlitic fluid, Snap Lake kimberlite, trigonal etch pits,
atomic force microscope

Introduction

Diamonds recovered from kimberlites and other deep-seated mantle magmas show a
combination of growth and resorption features on their surfaces. Resorption features are
products of diamond dissolution both in the mantle source and in the host magma, while
the morphology of these features depends on the conditions in both natural diamond
environments. Experiments show that kimberlitic fluid plays a major role in the
development of resorption features on diamonds (e.g. Fedortchouk et al. 2007). Thus,
better understanding the controls of diamond resorption morphology provides a tool for
examining the formation and composition of magmatic fluid during the ascent and
emplacement of kimberlite magma, independent of the uncertainty of the primary
composition of kimberlite melt (e.g. Moussallam et al. 2016).

The common resorption features found on the surfaces of natural diamonds
include: etch pits of trigonal, tetragonal, and hexagonal shape, terraces, laminae, hillocks
of rounded or sharp shape, and shallow depressions, circular pits, and corrosion
sculptures (e.g. Robinson 1979). Trigonal etch pits of negative orientation are a common
resorption features (Fig. 1a), which develop via fast propagation of step 2 (Fig. 1a)
consisting of carbon atoms doubly bonded to the crystal lattice. Positive trigons are very
rarely reported on natural diamonds (Robinson 1979) and develop via fast propagation of
step 1 consisting of carbon atoms trebly bonded to the crystal lattice (Fig. 1a) (Yamaoka
et al. 1980). Negative trigons are routinely observed in experimental studies conducted at
pressures of 1 GPa and higher (Arima and Kozai 2008; Fedortchouk 2007; Khokhryakov
and Palyanov 2010) and occur on diamonds from every kimberlite locality and any
kimberlite facies. Their maximum size depends on the temperature of the reacting fluid
(e.g. Fedortchouk 2015), while shape and abundance is very different in H₂O and CO₂
examined how the CO₂ content of the reacting fluid affects the relative lengths of the
positive (L_pos) and negative (L_neg) walls of trigonal etch pits (Fig. 1b) where C-O-H fluid
with <40 mol% CO₂ produces negative trigons with flat bottomed (f/b) and regular shape;
fluid with 40-90 mol% CO₂ produces f/b negative trigons with truncated corners; and
fluid with >90 mol% CO₂ produces pointed-bottomed (p/b) trigons and transitional
trigon-hexagon form. In addition, dissolution in CO₂-rich fluid produces negative trigons
with apparent positive correlation between the diameter and depth of the negative trigons,
while in H₂O-rich fluid this relationship is absent (Fedortchouk 2015).

Positive trigons have been very rarely reported on natural diamonds (Robinson,
1979). Typically, they occur on rare diamond crystals with extremely etched surfaces
found in parcels where other diamonds do not show positive trigons. In addition to the
positive trigons, these diamonds also show other very rare features, such as transverse
hillocks (of the opposite orientation compared to the common hillocks and imbricate
wedge markings (Fig. 1c,d). Positive trigons are the most common feature observed in
experiments conducted at 0.1 MPa but have been also produced at high pressures (high-P)
(e.g. Yamaoka et al., 1980, Harris and Vance, 1974). The causes of the reversal in the orientation of trigonal etch pits on diamonds are still not well understood. Experimental studies observed the change from negative to positive trigons at high oxygen fugacity ($fO_2$) at 0.1 MPa (Evans and Sauter 1961) and at 1.5 GPa (Yamaoka et al. 1980) and temperatures of 900°C and 1130°C respectively. These authors proposed that $T$ and $fO_2$ control the orientation of the trigons via stabilization of doubly bonded atoms of step 2 (Fig. 1a) with oxygen complexes (Evans and Sauter 1961; Yamaoka et al. 1980). Similarly, $T$ and $fO_2$ control a reversal in the orientation of tetragonal etch pits on {100} diamond faces (Fedortchouk and Canil 2009). However, experiments at 5-7 GPa and 1400 – 1750°C (Khokhryakov and Palyanov 2010) produced positive trigons in dry carbonate melt and reversed to negative trigons after addition of 8 wt% of H$_2$O at the same $fO_2$, which allowed the authors to propose that the CO$_2$:H$_2$O ratio is the main control for the trigon orientation. On the contrary, Harris and Vance (1974) recorded at 0.1 GPa and 1050°C negative trigons in carbonate-rich composition and positive in more H$_2$O rich compositions. Thus, the existing experimental data does not give an explicit answer on the causes of change in trigon orientation. A theoretical model by Angus and Dyble (1975) proposes that formation of positive trigons requires preferential removal of triply bonded carbon atoms, which requires more energy than removal of the doubly bonded atoms that accompanies negative trigon formation. Better understanding of the controls of the trigonal etch pit orientation on diamonds may help to constrain crystallization conditions of kimberlite magma.

Snap Lake kimberlite dyke (Northwest Territories, Canada) is a unique example of a kimberlite body, where all diamonds show positive trigons. What makes Snap Lake
so unique? Are the positive trigons on Snap Lake diamonds a product of deep (high-P) resorption due to the different kimberlite emplacement conditions than the majority of kimberlites, or do they represent a different resorption event happening at shallow near-surface conditions in a hypabyssal kimberlite body as opposed to the typical kimberlite that bursts through the earth’s surface to form a pipe? Can we deduce the crystallization conditions of the Snap Lake kimberlite(s) using the morphology of positive trigons? We address these questions via a detailed study of the positive trigons on diamonds from different litho-facies of the Snap Lake kimberlite dyke. Employment of an Atomic Force Microscope (AFM) for study of diamond surface features allowed us not only to detect the presence of small-sized trigons, but also to examine how the shape and dimensions of the trigons vary throughout the kimberlite body. Here we report the first systematic study of the morphology and characteristics of positive trigons on natural diamonds recovered from known sections of a single kimberlite body. The study aims to understand what factors control the presence, morphology, and dimensions of the positive trigons, and to examine their distribution within the Snap Lake dyke in order to better understand the role of volatiles in kimberlite emplacement.

**Background geology and diamond samples**

The Snap Lake Mine is located in the southeastern part of the Slave Craton about 220 km northeast of Yellowknife, Canada (Fig. 2a) (Fulop et al. 2017). The age of the kimberlite, 535-523 Ma, was determined by Rb-Sr dating of phlogopite (Agashev et al. 2001; Heaman et al. 2003, 2004). The country rocks comprise granodiorites, tonalities and granites of the Defeat Pluton Suite (2610 – 2590 Ma) and metavolcanic rocks of the Yellowknife Supergroup (Stubley 2000). There is no evidence of a thermal aureole in the
country rocks adjacent to the intrusion (Gernon et al. 2012). The orebody of the Snap Lake kimberlite is a segmented hypabyssal dyke dipping 12-15° towards the northeast flanked by a series of subparallel sheets (Gernon et al. 2012; Kopylova et al. 2010). The thickness of the dyke ranges from 0.1 to 15 m, but typically ~ 3-5 m (Gernon et al. 2012).

The Snap Lake dyke is filled with a coherent hypabyssal kimberlite HK (following the terminology of Scott Smith 2013) with minor kimberlite breccia facies dominated by granite or amphibolite xenoliths (Kopylova et al. 2010; Mogg et al. 2003).

Two models have been proposed to explain the compositional variation and the emplacement of the Snap Lake dyke. A two magma batches model suggests that the two different litho-facies (the main olivine-rich and phlogopite-poor facies, and the subordinate phlogopite-abundant facies) evolved from two magma batches which co-mingled and intruded almost simultaneously (Gernon et al. 2012; Ogilvie-Harris et al. 2008; Field et al. 2009). A single magma batch model suggests formation of one hypabyssal kimberlite with different degrees and forms of deuteric alteration and crustal contamination responsible for the generation of six sublitho-facies from HK1 to HK6, which is supported by petrographic, geochemical, and mineral chemistry data (Mogg et al. 2003, Kopylova et al. 2010, Fulop et al. 2017). The middle of the dyke is composed of the freshest phlogopite-poor HK1 and HK2 facies, with more altered HK5 surrounding granitoid xenoliths. The progressively more altered phlogopite-rich HK3, HK4 and HK6 facies developed at the contact with the country-rock granitoids (Fig. 2b).

Our study used 251 micro-diamonds provided by De Beers Canada Inc with the maximum diamond size 1.7 mm. We selected 91 diamonds that are ≥300 μm in size and can be handled under an optical microscope (diamonds <300 μm are mostly fragments...
and provide no information about resorption of crystal faces) and that are not fragments or show at least 50% of the crystal faces, so that crystal morphology can be studied. Our study focused on octahedral or tetrahedrahedral (THH) diamonds, since cubic diamonds are lacking experimental data on their resorption. The selected 91 diamonds comprise 78 octahedral and 13 THH diamonds, which represent all litho-facies at Snap Lake: HK1 (25 diamonds), HK2 (25 diamonds), HK3 (4 diamonds), HK4 (14 diamonds), and HK5-HK6 (23 diamonds). AFM was conducted on 16 diamonds representing all litho-facies and Fourier transform infrared spectroscopy (FTIR) measurements were obtained for 82 diamonds (including few fragments). The diamonds were recovered at Saskatchewan Research Council (SRC) by caustic fusion at 500°C in Na(OH)₂. Use of test synthetic diamonds with every batch confirms no etching during the recovery process.
Analytical methods

Optical and Scanning Electron Microscopy (SEM)

The diamonds were initially studied using a stereomicroscope to examine the general morphology, color, color intensity, and the presence of inclusions. Further examination of growth/resorption features on diamond surfaces and grouping into morphological types used a stereomicroscope and reflected light on a petrographic microscope with partially closed aperture stop for increased contrast. We classified diamonds with more than 10% of \{111\} faces preserved as an octahedron, and with less than 10% of \{111\} faces preserved – as a tetrahexahedron (THH). The morphologies of twinned diamonds and aggregates were classified based on the dominant shape of the constituent crystals. The octahedral diamonds were further divided into several morphological groups based on: (1) trigonal vs. ditrigonal shape of \{111\} faces, and (2) the type of resorption features on the \{111\} face including step faces, etch pits (negative and positive trigons, trigon-hexagons, hexagons), and laminae (shield-shaped, serrate- or irregular-shaped). SEM examination of 29 representative diamond crystals further refined the morphological grouping. Positive trigons larger than 1 μm were recorded with an optical microscope. The presence of positive trigonal etch pits smaller than 1 μm on the studied diamonds was confirmed with SEM. Diamonds selected for SEM were cleaned with a boiled HNO₃-H₂SO₄ mixture (3:5 volume proportion) at ~200°C for at least 30 min followed by ultrasonic cleaning in distilled water for 15 min and were then coated with gold. We used a Field Emission Scanning Electron Microscope (FE-SEM) Hitachi S-4700 FEG at the Institute of Research in Materials, Dalhousie University, with accelerating voltage of 10-15 kV and beam current of 15 μA.

Atomic Force Microscopy (AFM)
The AFM study of 16 diamonds (positive trigons were studied on 14 diamonds) was conducted using a Veeco Multimode 8 atomic force microscope at the Department of Earth Sciences, Dalhousie University. The diamonds were cleaned with aqua regia (HNO$_3$-HCl in 1:3 volume proportion) to remove the gold coating and other possible metal impurities, and then with a HNO$_3$-H$_2$SO$_4$ mixture (3:5 volume proportion) to remove the carbon from the SEM tapes and other dirt. During both treatments, the diamond crystals were boiled at ~200°C for at least 30 minutes. Finally, the crystals were cleaned in distilled water in an ultrasonic bath for 15 minutes. Prior to the AFM measurements, all selected diamonds were photographed at 100× and 200× (500× for minute features such as positive trigons) magnification using a petrographic microscope. Each diamond was mounted on a steel disk with an adhesive pad, in an attempt to orient the examined {111} face horizontally. In the case of uneven or fragmented crystals glue from the adhesive tape was placed under the crystal to minimize the tilt. All AFM images were collected using a J scanner (Bruker, maximum coverage 125 μm × 125 μm × 5 μm in x, y and z dimensions), operated in contact ScanAsyst mode using ScanAsyst Air Probes (Bruker, silicon tip on nitride lever, spring constants 0.4 to 0.8 N m$^{-1}$, resonant frequency 50-90 kHz, nominal tip radius of curvature 2 nm). The scanned areas ranged from 5 × 5 μm to 80 × 80 μm. The scanning frequency was dependent on the size of the scanned areas: we used 0.2 – 0.4 Hz for areas greater than 30 × 30 μm and 0.5 – 0.8 Hz for smaller areas to optimize the match between the trace and retrace lines. All the scans were conducted with 512 sample lines.

The images were analyzed using NanoScope software (v8.10) after a “plane fit” function to remove the tilt in both x and y directions. The parameters of individual etch pits (diameter, depth, shape, wall angles) were examined on cross-section profiles using the section analyses tool. A profile was drawn from the middle of a trigon side to the opposite vertex. For each
diamond we routinely measured 8-20 positive trigons. We measured the diameter of the pits as a
distance between the middle of one side and the opposite vertex, the depth of the pits as the
distance between the \{111\} face and the bottom of the pit, and the wall angles (\(\alpha_{\{111\}}\)) as the
angle between the plane of the wall micro-face and (111) plane. Each parameter comprises an
average of the three measurements for the profile drawn from each of the three pit vertexes. The
pits were classified based on the bottom and wall morphology (Fig. 3) (see also Zhang et al.
2015). The three sources of uncertainties in AFM measurements include: (1) the AFM
uncertainty (1.4% in x, y and z directions); (2) a possible tilt of the \{111\} diamond face; (3) the
subjective selection of the measurement (the placement of the profile line and the markers used
for the measurements). We estimate the total uncertainty <2.5% for diameter measurement, <6%
for depth measurement, and <4\(^\circ\) angle measurement (Table 1) (see also Zhang et al. 2015; Li
2017).

**Fourier transform infrared spectroscopy (FTIR)**

Nitrogen content and aggregation state were obtained with Fourier transform infrared
spectroscopy (FTIR) for the 14 diamond crystals with positive trigons studied by AFM (Table 2).
Diamond crystals were mounted on the edge of a glass slide with the (111) faces perpendicular to
the FTIR beam. We used Thermo-Nicolet Nexus 470 FTIR spectrometer fitted with a
Continuum infrared microscope equipped with a KBr beam splitter at the Department of Earth
and Atmospheric Sciences at the University of Alberta. Absorption spectra were measured in
transmission mode in the range of 4000–650 cm\(^{-1}\) with a resolution of 8 cm\(^{-1}\), and were collected
for 200 s through a 100 \(\times\) 100 \(\mu\)m\(^2\) aperture. Sample spectra were first baselined using the Basic
Macro of the OMNIC 32 software suite. A normalized Type II diamond spectrum used as a
standard for background correction was subtracted from the spectra to convert absorbance to
absorption coefficient. After the conversion, sample spectra were de-convoluted into the A, B
and D components using least square techniques (for more details see Stachel et al. 2006, Boyd
et al. 1994, 1995). The detection limit for nitrogen is about 10 ppm and the analytical precision is
10-20 % of the concentration (Stachel et al., 2006).

**Diamond etching experiments**

The temperature limit for the development of any resorption features on diamond at 0.1 MPa was
tested in experiments conducted at 700°C and 800°C at oxygen fugacity ($fO_2$) corresponding to
that of the air ($\log fO_2 = -0.68$) using a box furnace and at $fO_2$ corresponding to that of CO$_2$ gas
($\log fO_2 = -2.85$ at 700°C and -2.79 at 800°C) using a vertical-tube gas-mixing furnace in the
Department of Earth Sciences at Dalhousie University. In both cases, natural octahedral
diamonds (weight ~0.5 – 1 mg) with minimal natural resorption were imaged with AFM to
record any pre-existing features, placed inside Al$_2$O$_3$ crucible, and covered with a synthetic
mixture of 45 mol% Na$_2$CO$_3$ and 55 mol% NaCl corresponding to the eutectic composition with
melting temperature 632°C. The run duration was 2.5 hours in the air runs and 3 hours in CO$_2$
runs. After the experiments the diamonds were recovered, cleaned following the described above
procedure, and imaged with optical microscope and AFM to detect any new features on the
diamond’s surfaces.

**Results**

**General morphologies and negative trigons of Snap Lake diamonds**

We divided the octahedral and THH diamonds from Snap Lake into twelve
morphological groups based on the shape of {111} faces (trigonal vs. ditrigonal) and the type of
the surface resorption features. The details of morphological groupings and full description of the
negative trigonal etch pits will be reported elsewhere. Here we focus on the features of the
positive trigons. The 14 diamonds for which positive trigons were studied with AFM are all
colourless octahedra, which represent eight morphological groups illustrating both mantle-
derived and kimberlite-induced resorption (Fig. 4). The number of negative and positive trigons
analyzed on each diamond is reported in Table 2. AFM study of the negative trigons revealed
that they all have regular flat-bottomed (f/b) shape, diameter < 45 µm, and depth < 1 µm. The
unique feature of all negative trigons on Snap Lake diamonds is their truncated corners to a
degree that makes them transitional into hexagons (Fig. 5e,f). Many negative trigons contain
small positive trigons inside (Fig. 5b,c,f) or along their perimeter (Fig. 5a,d).

**Positive trigons on Snap Lake diamonds**

All 91 studied diamonds from Snap Lake kimberlite have positive trigons, which cover
both octahedral and THH faces. While negative trigons are limited to \{111\} faces, positive
trigons occur also on the rounded THH edges of resorbed diamonds (Fig. 6a,b). Positive trigons
are distributed uniformly (Fig. 6a) or show a patchy uneven distribution of larger and smaller
trigons in different areas of \{111\} faces (Fig. 6c). They also enhance the development of wormy
depressions (ruts) (Fig. 6h). The majority of the positive trigons have a diameter <3 µm, which
correlates with their depth (Fig. 7). Diamonds from HK1 and HK2 facies have larger trigons and
only two of the fourteen diamonds (SNP8-9 and SNP9-1) have positive trigons with diameters
larger than 4 µm (up to 13 µm). Less resorbed diamonds have positive trigons with diameter ≤
0.5 µm and depth < 20 nm, which could only be detected using SEM or AFM. The diameter of
the positive trigons roughly correlates with the kimberlite lithology and decreases from fresh
(HK1 and HK3) and slightly altered facies (HK2 and HK4) towards highly altered facies (HK5
and HK6) (Fig. 8). Diamonds from HK2 show three larger diameter trigons in Fig. 8, which
might be a result of an overprint of pre-existing pits. We found no correlation between the size of
the positive trigons and the general diamond morphology.

The detailed AFM study of 154 positive trigons on 14 Snap Lake diamonds (reported in
Supplementary Table 1) allowed us to divide them into four groups (Fig. 9):

1) point-bottomed (p/b) trigons with an irregular outline and walls consisting of multiple
micro-steps (Fig. 9b)

2) trigons similar to above but the bottoms are curved (c/b) and not pointy; the walls have
V or U shape; trigons with simple corners have more rounded bottoms (Fig. 9c), while trigons
with multiple corners have flatter bottoms (Fig. 9d)

3) asymmetric trigons with straight outlines and regular steps along one of the walls,
shallower pits are f/b (Fig. 9e) and deeper pits are p/b (Fig. 9f)

4) trigons with truncated corners and transitional trigon-hexagons with straight walls and
well-defined f/b shape (Fig. 9g)

The shape (f/b vs. p/b) of the small embryonic trigons with diameter <1 µm and depth <
100 nm (e.g. Fig. 9a) cannot be reliably distinguished with the scanner used. P/b is the most
prevalent shape of the positive trigons on Snap Lake diamonds; only transitional trigon-hexagon
pits show regular f/b shape and most likely represent an overprint of positive trigons on small
pre-existing negative trigons.

There are two distinct trends on diameter vs. depth plots (Fig. 7) where p/b trigons form a
steeper slope and c/b trigons form a shallower slope. P/b trigons are the most common and occur
on the majority of the studied diamonds. They form the only trigon type on the three diamonds
from HK1 and HK2 (SNP2-1, SNP8-9, SNP3-6). Asymmetric trigons are limited to only two
diamonds from HK1 and HK2 (SNPO13-2, SNPO9-1), that also show highly intensive etching
of \{111\} face. These trigons evolve from f/b to p/b as their depth increases. Curved c/b trigons appear alone (SNP3-8) or in combination with p/b trigons (SNP6-1, SNP9-1, SNPO12-13, SNPO12-8). Trigons with flatter bottoms mostly occur on diamonds from phlogopite-rich altered facies HK4 and HK6. Trigon-hexagon f/b pits were only observed on two diamonds from the altered facies at the contact with granitoids of HK5 (phlogopite-poor) and HK6 (phlogopite-rich) kimberlites (SNP5-3, SNPO12-2). The wall type of the studied trigons shows an evolution from U to V to Y as the depth of the trigons increases on the same diamond (Supplementary table 1).

**Experimental result**

In this study we only focused on the presence or absence of any changes to diamond surface after heating in sodium carbonate-chloride melt at 700°C and 800°C and \( \log fO_2 = -0.68 \) (air), -2.79 and -2.85 \((CO_2)\). Fig. 10 shows that etching at 800°C developed positive trigons on diamond surface easily seen on the micro-photographs taken under an optical microscope. After 700°C runs, no changes were visible under an optical microscope, but AFM showed development of positive trigons only in the run with air. AFM imaging detected no changes on diamond surface after the run at 700°C in CO₂.

**Discussion**

**Where do positive trigons form?**

In experiments positive trigons form over a wide pressure range: at 0.1 MPa (Evans and Sauter 1961), 0.1 GPa (Harris and Vance, 1974), 1.5 GPa (Yamaoka et al. 1980), 5-7 GPa (Khokhryakov and Palyanov 2010). The distribution patterns of positive trigons on natural diamonds from Snap Lake kimberlite support the conclusion of Robinson (1979) these positive trigons are a feature of the latest resorption event, which can overprint previous resorption. Indeed, positive trigons cover the whole diamond surface including the rounded resorbed faces.
(Fig. 6) and bottoms of the larger negative trigons. Furthermore, the AFM images obtained in our study demonstrate that positive trigons on Snap Lake diamonds overprint all the pre-existing negative trigons. They are present on the bottoms, along the sides or in the center of the negative trigons, and add truncated corners to all the negative trigons resulting in their transition to trigon-hexagon and hexagon shapes (Fig. 5a-f). Our results demonstrate that the presence of positive trigons on Snap Lake diamonds records conditions of a particular late resorption event.

Reversal in the trigon orientation manifested in the formation of trigon-hexagonal and hexagonal pits has been observed during diamond dissolution in CO$_2$-rich fluid (Fedortchouk et al. 2007, Khokhryakov and Palyanov, 2010). Zhang (2016) found that truncation of the corners of negative trigons starts when CO$_2$ constitutes more than 50 mol% of the fluid and hexagons form at CO$_2$ content >95 mol%, where the ratio $L_{pos}/(L_{pos}+L_{neg})$ can be used to quantify the degree of trigon to hexagon transformation (Zhang, 2016). If the development of positive walls inside the negative trigons on Snap Lake diamonds was controlled by the CO$_2$ content of the magmatic fluid, as in the Zhang (2016) experiments, both negative and positive walls of truncated trigons and trigon-hexagon pits would have developed simultaneously during a single resorption event so that the $L_{pos}/(L_{pos}+L_{neg})$ ratio would be independent of the diameter of the pit. In such a case, a correlation with the kimberlite lithology would be expected. If the formation of the positive walls overprinted the original shape of a negative trigon, then the $L_{pos}/(L_{pos}+L_{neg})$ ratio would show a negative correlation with the trigon diameter. Fig. 11a shows the correlation for the studied Snap Lake diamonds, where the smaller the diameter of the negative trigons the larger is their $L_{pos}/(L_{pos}+L_{neg})$ ratio. Fig. 11a also shows a different trend for HK1 and HK2 diamonds with a larger $L_{pos}/(L_{pos}+L_{neg})$ ratio for the same diameter compared to the diamonds from the other lithologies. Diamonds from HK1 and HK2 also have a larger absolute value of
L_{\text{pos}} (Fig. 11b) and larger diameter of the positive trigons (Fig. 8) indicating the higher intensity of the resorption event forming positive trigons in the HK1 and HK2 kimberlite facies. The micro-features on the positive walls of truncated negative trigons on Snap Lake diamonds are different from those produced in CO$_2$-rich dissolution experiments by Zhang (2016) (Fig. 5g-i).

In these experiments, the truncation by positive wall development was accompanied by a curvature of the negative trigon walls (Fig. 5g-i). All this evidence confirms that trigon-hexagonal and hexagonal pits on Snap Lake diamonds are not a product of resorption in more CO$_2$-rich fluid but a combination of two resorption events – an early (high-P) event, that produced negative trigons typical for the majority of kimberlitic diamonds, and the later (low-P) event, that produced positive trigons and truncated negative trigons, a specific feature of the Snap Lake diamonds.

**Application to Snap Lake kimberlite system**

This late resorption event in the Snap Lake kimberlite most likely occurred after dyke emplacement, while the kimberlite was still at magmatic temperature. Experimental studies of diamond etching help to constrain the temperature of this late resorption event responsible for the development of the positive trigons on Snap Lake diamonds. Yamaoka et al. (1980) estimated 1130°C as a reversal temperature from negative to positive orientation of trigonal etch pits at 1.5 GPa. Slightly lower reversal temperature between 900 and 1000°C obtained by Evans and Sauter (1961) at more oxidized conditions cannot be explained by the difference in $f_{O_2}$, which according to Yamaoka et al. (1980) should increase the reversal temperature at higher $f_{O_2}$. Most likely the reason for this discrepancy is in erroneous $f_{O_2}$ estimates in Yamaoka et al. (1980). Their study used a double capsule technique with an oxide buffer in the outer capsule and diamond crystals.
in the small capsule inside. Such an approach requires reaching equilibrium in the system, which
cannot be achieved until the diamonds are completely oxidized. As long as the diamond
oxidation reaction proceeds the \( f_{O_2} \) would be somewhere between the value imposed by the oxide
buffer and the value of the CCO buffer. Thus, we suggest 900 - 1000°C as the maximum
temperature for the development of positive trigons on Snap Lake diamonds, which is slightly
below the ~1100°C crystallization temperature of kimberlite magma (Fedortchouk and Canil
2004).

The minimum T for this resorption event is the T limit for diamond resorption. According
to the experiments of Yamaoka et al. (1980) this is 800°C. A number of studies (e.g. Evans and
Sauter 1961, Phaal 1965) observed diamond etching at temperatures as low as 650°C but only in
pure oxygen, and above 950°C in CO\(_2\) flow. Our experiments conducted at yet more oxidized
conditions than that of natural kimberlites show that at 700°C resorption occurs in the air but
does not proceed in pure CO\(_2\) gas corresponding to \( \log f_{O_2} = -2.85 \) (Fig. 10). We propose the
temperature of the late resorption event forming positive trigons on diamonds from Snap Lake
dyke between 800 – 1000°C.

Kimberlite magma typically arrives at the earth’s surface with substantial amounts of
magmatic fluid formed via CO\(_2\) degassing accompanying assimilation of silicate xenoliths by
carbonate-rich magma (e.g. Russell et al. 2012; Stone and Luth 2016) as well as from
decompressional degassing of H\(_2\)O and CO\(_2\) in the last few km of the ascent (Moussalam et al.
2016). Extensive serpentinization and other alteration reactions observed in kimberlites can be a
product of reaction with the magmatic fluid (e.g. Mitchell 2013) or can result from a substantial
contribution from meteoric fluid (e.g. Stripp et al. 2006). The high content of phlogopite in Snap
Lake kimberlite, especially closer to the contact with the country-rock granitoids (HK3,4,6 facies)
was interpreted as a product of alteration by deuteric fluid (Fulop et al. 2017). The dimensions of the positive trigons on diamonds can help to examine the nature of the fluid during the late resorption event at Snap Lake. The diameter of the trigonal etch pits on diamonds depends on T and the duration of the diamond reaction with fluids (Fedortchouk 2015). The largest diameters of the positive trigons on diamonds from our study are limited to the two freshest facies (HK1 and HK2, Fig. 8) present in the interior parts of the intrusion far from the contact with the country rock. Diamonds from the facies closer to the contact with the granitoid (HK3,4,6) or surrounding large granitoid xenoliths (HK5) (Fig. 2b) show very small sizes of the positive trigons. This implies longer interaction with fluid possibly at higher T in the interior parts of the intrusion. In combination with the estimated high T of the late resorption event, this would strongly support magmatic fluid as the resorption agent. We propose that at depth, during the ascent of the Snap Lake kimberlite magma to the surface, magmatic fluid gave rise to resorption features typical for all kimberlite-hosted diamonds (such as negative trigons). After the emplacement, the fluid was trapped in this hypabyssal intrusion close to the surface, and resulted in the later resorption event manifested in the development of the positive trigons on Snap Lake diamonds, which is not present on diamonds from both volcaniclastic and coherent lithologies in kimberlite pipes in Ekati Mine, Canada, and Orapa kimberlite cluster, Botswana (Fedortchouk et al 2010; Fedortchouk et al. 2017).

However, Snap Lake may not be that unique. Many diamonds from this study have so small trigons, which were only detected with SEM, and would have been overlooked during a routing examination under an optical microscope even with 50x objective. Indeed, AFM scanning of octahedra micro-diamonds from unknown Yakutian sources used for diamond dissolution experiments have shown presence of positive trigons on some diamonds.
(Fedortchouk, unpublished data). Furthermore, there is only very limited data on diamonds from kimberlite sheets. Some of them are not diamondiferous, others are too thin to be mined. Diamonds from kimberlite pipe are lacking such widespread development of positive trigons, and only show them on rare individual diamonds in association with other very rare features (Fig. 1c,d). It is possible that a late resorption event forming positively-oriented trigons is a feature of kimberlite sheets. The fluid responsible for the late resorption is clearly not related to serpentinization that occurs at low temperatures around 130-400°C (Afanasyev et al. 2014) and creates strongly reducing conditions (McCollom and Seewald 2013).

The shape of negative trigons on diamond can be used to deduce the H$_2$O:CO$_2$ ratio in the reacting fluid (Fedortchouk 2015). Twelve out of fourteen diamonds studied by AFM show two types of positive trigons: 1) p/b trigons with straight walls (which evolve from small flat almost f/b pits, Fig. 9a,b), and 2) curved-bottom trigons (Fig. 9c,d). Both types of trigons form distinct positive trends of diameter vs. depth (Fig. 7), the slope of which is steeper for p/b trigons and shallower for curved/bottomed trigons. Study of the negative trigons from diamond dissolution experiments (Fedortchouk 2015) suggests that such trigon morphologies and the positive correlation between the diameter and the depth are indicative of CO$_2$-rich fluid. However, applicability of this correlation to positively oriented trigons has yet to be confirmed experimentally. Negative trigons on Snap Lake diamonds show very regular f/b shape of the diamonds that suggests predominantly aqueous fluid in the earlier high-P resorption event during the magma ascent (Fedortchouk et al. 2007; Fedortchouk 2015). The shape of the positive trigons would agree with the fluid evolution towards more CO$_2$-rich composition after the emplacement during crystallization of the dyke, given that CO$_2$ effect on the shape of positive and negative trigons is similar. Assimilation reactions between the carbonate component of the melt and
silicates from the granitoids may have provided an additional CO$_2$ input. Alternatively, the
difference in the shape of negative and positive trigons can be related to the different dissolution
conditions during the earlier (high-P) and late (near-surface) resorption events. Luttge (2006)
proposed that at conditions close to equilibrium (small $\Delta G$), crystal dissolution proceeds via
formation of flat-bottomed “pancake” pits growing around kinks associated with point defects.
When the conditions are far from equilibrium (at higher $\Delta G$), dissolution opens the hollow cores
at screw dislocations forming deeper p/b etch pits. Pressure has a large control on the stability of
the diamond lattice. A decrease in pressure shifts the system very far from an equilibrium
(towards larger $\Delta G$). The presence of large diameter but shallow negative trigons and the
absence of such shallow positive trigons on Snap Lake diamonds might be due to much higher
$\Delta G$ at low P.

Development of both f/b and p/b asymmetric trigons is limited to only three diamond
crystals, of which two have no other trigon types (Fig. 7, SNPO13-2, SNPO9-1). This is likely
not due to the external conditions of the resorption but the internal properties of these diamond
crystals and their defects. Finally, truncated and trigon-hexagonal pits were only observed on
diamonds from kimberlite facies very close to the contact with large xenoliths (HK5) or country-
rock (diamond SNPO12-2 from HK6, Fig. 7). Incomplete transformation of very small (<2 $\mu$m in
diameter) negative trigons into positive trigons, suggests a very short duration of resorption and
confirms the increase in the resorption intensity towards the center of the dyke.

Mechanism of diamond resorption and development of positive trigons

Review of the existing experimental data shows uncertainty in the conditions required for
formation of negative vs. positive trigons on diamonds during dissolution. Yamaoka et al. (1980)
following Evans and Phaal (1962) and Phaal (1965) propose that T and $fO_2$ affect the activity of oxygen in stabilizing certain directions of the diamond lattice via C – O – C bridges (Fig. 1a). However, evolution from negative trigons to hexagons and positive trigons was observed in experiments at the same T, P, and $fO_2$, but conducted in H$_2$O-rich and CO$_2$-rich systems (Fedortchouk et al. 2007; Khokhryakov and Palyanov 2010; Zhang 2016) emphasizing the role of water in the orientation of etch pits on diamond. Angust and Dyble (1975) developed a theoretical model based on an ideal configuration of diamond crystal lattice (Fig. 12), which proposed that development of negatively-oriented trigons is expected for the diamond lattice because this mechanism involves preferential removal of carbon atoms bonded to fewer of the surrounding atoms. On the contrary, the development of positively-oriented trigons requires conditions with no preferential removal of triply-bonded and doubly-bonded carbon atoms. This could be achieved when the diamond crystal lattice becomes very unstable, such as at very low P or very high $fO_2$. Since diamond is a high-pressure carbon phase, emplacement of kimberlite magma at shallow levels could create conditions where the diamond lattice becomes extremely unstable, the activation energy needed to remove triply-bonded and doubly-bonded atoms become similar, and positive trigons start to form. This agrees well with the observed occurrence of positive trigons on diamonds from the shallow Snap Lake kimberlite sheet but their absence on diamonds from Ekati kimberlite pipes both located in Slave craton of Canada (e.g. Fedortchouk et al. 2010). Indeed, most of the experiments on diamond dissolution or oxidation report positive trigons at 0.1 MPa and negative trigons at elevated P. While low pressure is not the only possible way to destabilize diamond and produce positive trigons, it appears to be the most likely cause in natural kimberlitic systems.
Positive trigons developed in high-P experiments accompany features of extreme diamond etching. For example, naturally occurring rare diamonds with positive trigons shown on Fig. 1c,d best resemble figure 6 from Yamaoka et al. (1980) study, which shows experimentally oxidized diamond at 4 GPa at the extreme oxidation conditions corresponding to Mn$_2$O$_3$–MnO$_2$ oxygen fugacity buffer. We propose that there are at least two different natural mechanisms that produce positive trigons on diamonds by enhancing removal rate of triply-bonded carbon atoms (Angus and Dyble 1975). First, the extreme oxidation (and / or heating) experienced by diamonds in the mantle source, can explain presence of extremely rare individual diamond crystals found in parcels where other diamonds show no positive trigons. Second, near-surface resorption event can overprint all diamonds with tiny positive trigons similar to the ones described here on Snap Lake diamonds. As SEM imaging becomes a routine step in characterization of diamond parcels, the proposed here association of positive trigons with diamonds from kimberlite sheets can be further tested.

**Implications**

It has been shown that the common resorption features of diamonds such as negative trigons, hexagons, and circular pits show strong association with the CO$_2$/CO$_2$+H$_2$O ratio and temperature of the oxidizing fluid and hence may serve as a proxy of fluid in kimberlite systems (e.g. Fedortchouk et al. 2007; Zhang et al. 2015). By expanding this method to positive trigons in this study, we confirmed that the trigon diameter correlates with T and duration of the resorption event and may be used to examine the thermal history of fluids during crystallization of a kimberlite body. We also confirmed that the correlation of the diameter and the depth established
for negative p/b trigons exists for positive p/b trigons, and likely results from the CO₂/CO₂+H₂O ratio in the fluid.

Our findings place some constraints on the crystallization conditions of different kimberlite bodies. In the Snap Lake dyke we identified two diamond resorption events produced by the kimberlite magma. The early high-P resorption occurred in all kimberlite litho-facies during the ascent and produced negative trigons. The later low-P resorption event produced the positive trigons on all studied Snap Lake diamonds at near-surface conditions (pressures approaching 0.1 MPa) by high-temperature magmatic fluid (~ 800 – 1100°C). Since experiments at 0.1 MPa show development of positive trigons as fast as within one hour (Fedortchouk, unpublished data), absence of positive trigons on diamonds from the majority of kimberlite bodies suggests very quick magma cooling below ~800°C precluding the development of any late resorption features. The presence of positive trigons on diamonds indicates unusual emplacement conditions. Further studies are needed to clarify if such resorption events are a feature of kimberlite sheets, which do not breach the surface and better preserve magmatic volatiles.

Solubility of H₂O and CO₂ in kimberlite-like magma (Moussallam et al, 2016) suggests exsolution of both volatiles only very close to the surface within the last 2-5 km of the ascent. This could drive the explosive eruption and magma fragmentation, but would not explain the extremely fast ascent of kimberlites (e.g. Canil and Fedortchouk 1999, Kelley and Wartho 2000, Sparks et al., 2006). A reaction between silicate xenoliths and carbonate-rich kimberlite magma may exsolve CO₂ starting as deep as 70-80 km (~2.5 GPa) (Russell et al. 2012, Stone and Luth 2016) to drive the fast kimberlite ascent. However, the features of negative trigons on diamonds indicate notable variation in CO₂/CO₂+H₂O ratio of the fluid (Fedortchouk 2015), which in some kimberlites is less than 50 mol% CO₂ (Zhang, 2016). This study observed a change from the f/b
shape of the negative trigons (typical for H$_2$O-rich fluids) to p/b shape of the positive trigons
(typical for CO$_2$ –rich fluids), which may suggest CO$_2$ increase in the fluid during the ascent
through the last kilometers. Indeed, experimentally determined solubilities of H$_2$O and CO$_2$ show
an increase in CO$_2$ exsolution closer to the surface (Moussallam et al, 2016). Yet, it is not clear if
H$_2$O component in kimberlitic fluid introduced due to decompressional degassing at 2-5 km
below the surface could be responsible for all the observed resorption on kimberlitic diamonds.
Our study shows that a better understanding of P, T, and fO$_2$ of the reversal in trigon orientation
may provide a tool to establish the depth of exsolution and changes in the composition of
kimberlitic fluid. Since the development of the positive trigons is limited by P and T, this would
probably provide the most robust record of crystallization conditions and emplacement
mechanism of kimberlite magma.

Further testing of the proposed association of positive trigons on diamonds with kimberlite
sheets, can have important implications for diamond exploration. It is important to distinguish
between kimberlite sheets and hypabyssal kimberlites of the root zone of kimberlite pipes at the
early stage of a drilling program. This impact the assessment of the ore reserve calculation and
modelling of kimberlite body. Perhaps examining for the presence of positive trigons on micro-
diamonds under SEM can help better planning of drilling expenses.

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References cited


Figure Captions

Figure 1 - A) Positive and negative trigons on {111} diamond face and their formation due to relative rates of propagation of step 1 (positive trigons) and step 2 (negative trigons) (after Yamaoka et al. 1980). B) Truncated trigon on {111} diamond face with lengths of positive ($L_{pos}$) and negative ($L_{neg}$) micro-faces shown (after Zhang 2016). C) Natural diamond of unknown geographic origin with abundant positively oriented trigons and imbricate wedges. D) Natural diamond (octahedral macle twin) with positively oriented trigons, fine transverse hillocks on resorbed dodecahedral surfaces and imbricate wedge markings.

Figure 2 – Geology of the Snap Lake kimberlite dyke. A) The location and shape of the dyke. B) Distribution of the six kimberlite litho-facies within Snap Lake dyke relative to the contact with the two types of the country-rocks (after Fulop et al. 2017).
Figure 3 – Classification approach for trigonal etch pits on diamond based on the bottom and wall geometries (modified from Zhang et al. 2015). The pits are defined as having V type walls when \([\alpha_1 - \alpha_2] < 1^\circ\) and as f/b when \([\beta_1 - \beta_2] < 1^\circ\).

Figure 4 – Microphotographs of sixteen Snap Lake diamonds, on which positive and negative trigons were studied by AFM.

Figure 5 - Development of positively-oriented micro-faces on negative trigons on Snap Lake diamonds (A – F) and in experiments at 1250°C and 1 GPa with CO₂-rich fluids (modified from Zhang 2016) (G – I). A – C – development of truncated corners of trigons on Snap Lake diamonds; D – F – trigon-hexagons and hexagons on Snap Lake diamonds; G – I – negative trigons from the Zhang (2016) experiments showing curvature of trigon edges accompanying truncation of the corners with positive micro-faces. The degree of corner truncation \((L_{pos}/L_{pos}+L_{neg} \text{ ratio})\) increases from (G) to (I). All images are AFM scans.

Figure 6 – Distribution of positive trigons on Snap Lake diamonds: on resorbed THH faces (a,b), on \{111\} faces (c, d, e, f), and inside the negative trigons (g, h). The boxes on the left images show the location of the close-up area displayed on the right. a, b, c, e, g – microphotographs; d, f, h – AFM images.

Figure 7 – Diameter vs. depth of positive trigons on Snap Lake diamonds from different kimberlite litho-facies based on the AFM measurements. For the trigons types see Fig. 9.
Figure 8 – Diameter of positive trigons on diamonds from different litho-facies of Snap Lake kimberlite. A) The choice of a representative maximum trigon size of the positive trigons on individual diamonds studied with optical microscope or SEM. B) Use of the above approach to establish the proportions of diamonds with a maximum size of the positive trigons ≤ 3 μm, between 3-5 μm, and ≥ 5 μm in each kimberlite facies. C) The diameter range of the individual positive trigons measured with AFM on Snap Lake diamonds from different kimberlite facies.

Figure 9 – Morphological types of positive trigons on Snap Lake diamonds based on the AFM profiles.

Figure 10 – Etching of diamond surface in experiments with Na₂CO₃-NaCl melt conducted at 0.1 MPa, 700°C and 800°C and fO₂ corresponding to that of the air and pure CO₂ gas. AFM images of diamond surface before and after the runs at 700°C show development of positive trigons in air and but no sign of resorption in CO₂.

Figure 11 - A) The relationship between the ratio L_{pos}/L_{pos}+L_{neg} and the diameter of trigonal etch pits on diamonds from different kimberlite facies showing that the trend defined by diamonds from the fresh HK1-HK2 kimberlites is different from the trend defined by diamonds from more altered kimberlites (HK4, 5, 6). B) L_{pos} values of etch pits measured on individual diamonds from different kimberlite facies (the diamond labels are shown along the x-axes, the number of measured pits is shown in brackets).
Figure 12 - Atom removal process and development of trigonal etch pits on {111} diamond surfaces (from Angus and Dyble 1975) showing development of a negative trigon (B) from an arbitrary hole in the outer layer (A) after sequential removal of doubly-bonded atoms. X is a top layer atom bonded to the layer below. 0 is a top layer atom not bonded to the layer below.

Tables

Table 1 – Summary of the sources of uncertainty in AFM measurements.

Table 2 – AFM details and nitrogen data for the fourteen diamond from Snap Lake kimberlite studied by AFM.

Electronic supplementary materials

ES table 1 – AFM data of Snap Lake diamonds
Table 1 Summary of the sources of uncertainty in AFM measurements.

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<tr>
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<th>Source of uncertainty</th>
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Table 2 AFM details and nitrogen data (from FTIR) for the fourteen diamond from Snap Lake kimberlite studied on AFM.

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Figure 1
Figure 2
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Figure 3

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**Figure 4**

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Snap Lake diamonds (this study)

Experiments in C-O-H fluids (after Zhang, 2016)

Figure 5

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Figure 6
Figure 7
A.
The representative trigon measured to estimate the maximum size for majority of the positive trigons.

B.

Figure 8

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Figure 9
Figure 10

After 2.5 hours
700°C (air)
Initial surface
After 3 hours

700°C (CO₂)
Initial surface

800°C (air)
Initial surface
After 2.5 hours

800°C (CO₂)
Initial surface
After 3 hours

Figure 10
Figure 11
Figure 12