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X-ray topographic study of a diamond from Udachnaya: implications for the genetic nature of inclusions

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Abstract (300 words)

One of the most debated and open question in diamond research is the distinction between syngenetic, protogenetic and epigenetic relationships between diamond and its mineral inclusions. In this light, X-ray topography (XRDT) represents a helpful non-destructive tool to verify the genetic nature of inclusions in diamond. Here we investigated by XRDT a diamond from the Udachnaya kimberlite (Russia), already characterized by single-crystal X-ray diffraction by a previous work, in order to obtain further information on its genetic nature. The diamond includes three olivine inclusions randomly oriented with the two largest ones showing a typical "diamond-imposed" shape and the smallest one apparently having its own typical morphology. The main result of our XRDT study shows that the diffraction contrast around the two larger olivines reveals that no dislocations nucleated from them. This could be explained by the following possibilities: a) the contact crystallographic planes between diamond and olivines are very well matching because they are syngeneticand show epitaxial growth relationships; b) olivines are protogenetic but, during the entrapment, at the interface with diamond a dissolution/re-crystallization process occurs producing syngenetic olivine reducing the probability to originate dislocations for the same crystallographic reason than in (a); c) the diamond-olivine interface is represented by an interposed amorphous material, as well as a fluid or a gel, that did not produce any volume distortion.

Possibility (a) could be excluded at least for olivines included in diamonds from Udachnaya as demonstrated by crystallographic relationships in a previous work; possibility (b) is in agreement with a previous work but no evidence of a secondary olivine growth has been observed; to prove possibility (c) a deep analysis of the diamond-olivine interface should be carried out.

The absence of dislocations appears to be a common feature of inclusions in diamonds regardless the composition as proven by a previous work.

Introduction

Diamonds and their mineral inclusions represent the deepest materials originating in the Earth's interior that can reach our planet surface. Their study has shed light on geochemistry (both major and trace elements), geophysics, petrology, geodynamics and mineralogy of the mantle and the growth conditions of diamond, from the lithosphere to the transition zone and lower mantle (Stachel and Harris, 2008 and references therein; Shirey et al. 2013 for a review; Pearson et al. 2014). To obtain rigorous information about the chemical and physical conditions of diamond formation it is crucial to determine if the crystallization of the inclusions occurred before (protogenetic nature), during (syngenetic nature) or after (epigenetic nature) the diamond growth. The study of inclusion-free diamonds cannot provide many geological information and thus the only way to obtain information on the diamond growth environment is the investigation of its mineral inclusions. In this light, it is evident that only in case of syngenesis between diamonds and their inclusions (they were formed under the same physical/chemical conditions) we can retrieve indications on the diamond growth formation by studying the inclusions. At present, criteria to establish the diamond-inclusions relationships were mainly based on two arguments: 1) morphology imposition by diamonds (i.e. inclusions with typical "cubo-octahedral shape"); 2) epitaxial growth relationships (Futergendler and Frank-Kamenetsky 1961; Sobolev 1977; Orlov 1977; Harris and Gurney 1979; Meyer, 1987; Bulanova 1995; Pearson and Shirey 1999).

From another side, Taylor et al. (2003) argued that rare earth elements (REE) from harzburgitic garnet inclusions (with diamond-imposed morphologies) were inconsistent with simultaneous growth with diamond and proposed a protogenetic origin for these inclusions.

Using the isotopic composition of sulfides included in diamonds, <mark>Thomassot et al. (2009)</mark> also reported arguments in favour of protogenesis. More recently, Bruno et al. (2014) and Nestola et al. (2014), studying the crystal morphology and the crystallographic orientations of olivine inclusions with diamond-imposed morphology, proposed that the morphology alone cannot be considered as unequivocal proof of syngenesis and that, at least for olivine, there is not any preferential epitaxial growth with diamonds as believed in past (see Mitchel and Giardini, 1953). Such strong different points of view demonstrate how far from the definitive answer "syngenesis/protogenesis" still we are.

In order to contribute to the study of the genetic nature of inclusions in diamond, the relationship between two inclusions and their host were investigated in a diamond from the Udachnaya kimberlite, Siberia (Russia). The diamond was the same one previously studied by Nestola et al. (2011), who was able to refine the olivine crystal structure on inclusions still trapped in this diamond in order to obtain the depth of diamond-olivine formation. Their investigation showed that the olivine crystals in that diamond had the same composition ($Fo_{92.7}$) and formed at a minimum pressure of about 5 GPa at an assumed temperature of 1300°C.

Here we have investigated the same diamond by means of X-ray Diffraction Topography (XRDT), a technique which permits the study of extended defects within a mineral and thus to reconstruct its growth history. X-ray topography is non-destructive and has a resolution limit of a few µm, being particularly suitable to investigate the structural defects in diamond because of its low attenuation coefficient of the X-ray beam. This type of study has provided minerogenetic information on tourmalines, garnets and beryls (Agrosì et al., 2006; 2011; Tempesta et al., 2011) and, recently, was used to perform a complete discrimination between growth and post-growth defects in a diamond from the Finsch mine (Agrosì et al., 2013).

Such kinds of information can provide new insight to solve to syngenesis-protogenesis debate using a completely alternative approach.

Materials and method

The diamond specimen studied in this work is colourless with longest dimension about 3 mm and has an elongate, but flattened and slightly rounded octahedral shape (Fig. 1). The sample contains three colourless olivine inclusions, (Fig. 1a); those labelled A and B were the ones

previously investigated by Nestola et al. (2011) and Nestola et al. (2014). The olivine labelled C was considered not suitable for remnant pressure investigations in Nestola et al. (2011) because it was surrounded by several cracks that could have strongly released the pressure. Our optical observations revealed the diamond to have anomalous birefringence (Fig. 1b), and under reflected light, etch pits and trigons can be observed on the flattened (111) faces (Fig. 1c).

The diamond was investigated by XRDT in transmission geometry, using MoKα_1 radiation with a conventional source. The technique, developed by Lang (1959), is a non-destructive imaging technique, sensitive to the strain associated with extended defects and yields spatial distribution and full characterization of the crystal defects in the whole sample volume. The detailed XRDT procedures used in this study are provided in Agrosì et al. (2013).

RESULTS

Optical observations

Optical observations reveal that the two largest inclusions, labelled A and C (Fig. 2a), clearly show a diamond-imposed morphology, i.e. the so-called "cubo-octahedral shape". It can be noted that several cracks surround the C inclusion while on the upper right corner of the A inclusion small healed fractures.No discontinuity was found around the B inclusion (see the image enlargement in Fig. 2c). The stereogram shown in Figure 2b represents the crystallographic orientations of these inclusions with respect to the diamond (see Nestola et al., 2014). These orientations are different from each other and appear to be randomly oriented with respect to diamond principal axes. Comparisons between the optical and crystallographic observations allowed the reconstruction, in a qualitative way, of the morphology of the inclusions. Although it was not possible to measure the true angles between the faces of olivines, it can be noted that the A and C inclusions exhibit a typical diamond-imposed morphology, whereas the B inclusion shows an elongated shape (see in Fig. 2a the green drawings). The morphology of the B inclusion was reproduced by SHAPE software, by using the equilibrium shape in the vacuum of forsterite calculated by Bruno et al. (2014) and orienting it accordingly to the orientation deduced by the stereogram of Fig. 2b. It was found that the real shape of this inclusion and its ideal reconstruction are really similar (Fig. 2c).

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Additionally, optical observations performed focusing on the interface between the A olivine inclusion and the diamond (Fig.3a) reveal unexpected piling up of laminae producing a typical stepped surface (Fig. 3b) commonly due to dissolution or growth processes (Sunagawa, 2005).

X-ray diffraction topography

Figure 4 shows a set of X-ray topographic images, which indicate that the whole diamond crystal does not diffract simultaneously. This means that the entire crystal is not in the Bragg diffraction condition and consists of different misoriented regions. As an example, in Fig. 4a) and 4b) the topographs taken under the same diffraction reflection exhibit images of different regions. In these images, there is a lack of diffraction contrast observed for both the two larger olivine inclusions, labelled A and C. Relatively to inclusion B the diffraction effects of the diamond lattice mask its small size and we cannot obtain reliable results on such small inclusion.

An analysis of the diffraction contrast reveals that the diamond exhibits deformation fields affecting the entire sample. These features are ascribable to a post growth plastic deformation (PD).The crystallographic direction of strain can be established applying the extinction criterion (Authier and Zarka, 1994) and this shows that, as expected, the deformation direction corresponds to that commonly found in *Fd*-3*m* symmetries, where the energetically most favourite slip system is <110> {111}. Because of the overlapping of the strain fields associated with the plastic deformation of the diamond, three subsequent fixed exposure topographs were taken to reduce this effect (Fig. 5). In these images, the diffraction contrast around the A and C olivines reveals, at micron-meter scale, that no dislocations nucleated from these inclusions. Only a dark halo surrounds them.

Discussion

Two extremely important results were found in this study: 1) we did not find any dislocations nucleating from the olivine inclusions into the diamond and 2) we found very characteristic "stepped surface figures" at the interface between diamond and olivine.

The absence of dislocations nucleating from the olivine inclusions toward the diamondwas also observed for garnet entrapped in diamond by Agrosì et al. (2013) and thus is independent

on the type and composition of inclusion. Before trying to explain such feature, we should remark that, normally, when a solid inclusion has been incorporated in full-grown state in another growing crystal, the associated volume distortion needs of a number of dislocations to ensure a better connection between the inclusion and the host phase. In some cases, also complex twinning can be developed (Agrosì et al., 2013). Generally, no dislocation nucleation from inclusions is rarely observed. This absence occurs only when the interface between the two crystalline phases is coherent and the better lattice matching is guaranteed, or one of the two phases is amorphous, i.e. in the case of melt and fluid inclusions. Therefore, it is evident that our observation represents a real physical anomaly. In order to explain it, here we attempt to propose different scenarios:

a) diamond and olivines are syngenetic and their contact crystallographic planes are very well matching, i.e. they growth epitaxially;

b) the bulk body of olivines are protogenetic but at the interface with diamond, during the entrapment and the imposition of the diamond morphology, a dissolution/re-crystallization process occurs producing a "new" syngenetic olivine reducing the probability to originate dislocations for the same crystallographic reason than in (a), i.e. this "new" precipitated olivine grew epitaxially with diamond;

c) at the diamond-olivine interface one of the two phases is an amorphous material, and since neither the olivine nor the diamond are amorphous, it can be hypothesized that an amorphous material, that do not produce any volume distortion, is interposed between the two crystalline phases (i.e. a fluid or a gel).

Scenario (a): the syngenetic origin could be excluded because the random orientations of these olivines are not in agreement with the better lattice matching suggested by Mithcell and Giardini (1953) that favors an epitaxial growth. On the other hand, at least for olivine included in Udachnaya diamonds, Nestola et al. (2014) reported strong evidences of protogenesis based on two crystallographic evidences: 1) olivines are randomly oriented with respect to diamonds and 2) in single diamonds they found multiple inclusions with identical orientations, which the authors interpreted as remaining not-dissolved fragments of the same single original pre-existing olivine. Scenario (a) is, instead, strongly supported by several authors, who hypothesized a process of "mutual growth" of the inclusions and diamond, during which the diamond imposes the shape to the inclusions (Harris 1968a). The "mutual growth" would presuppose a synchronous growth of inclusions and diamond and this fact has

led to consider the diamond-imposed morphology as a key proof of syngenesis (Boulanova, 1995). On the other hand, the synchronous growth of diamond and olivine was experimentally proved by tests of crystallization of melts with peridotite-carbonatite composition that produced the same mineral associations (Bobrov and Litvin, 2009; Litvin et al., 2012). But, at moment, none of these studies produced synthetic diamonds including olivines with diamond-imposed morphology, even of extremely small size. In addition, in this work we showed that one of the three olivine inclusions does not have at all a diamond-imposed morphology and therefore we are strongly confident that the inclusion morphology has no relationships with the diamond temporal growth. Indeed, it is not reasonable as possible scenario that two olivines out of three with diamond-imposed morphology are syngenetic and the third one with its own morphology is protogenetic in the same diamond with same composition and crystallized at the same pressure and temperature (Nestola et al. 2011). Resuming, in general we think that the inclusion morphology cannot be longer used as a proof of syngenesis.

Scenario (b) is in agreement with Nestola et al. (2014) but these authors did not report any observational evidences of newly grown olivine syngenetic with diamond at their interface. Indeed, the evidences in our work of "stepped surface figures" at the interface between diamond and olivine could be due to processes of dissolution and recrystallization.

Scenario (c) is extremely exciting and would be strongly in agreement with the absence of dislocations but, at present, nobody reported the presence of an interposed amorphous material (fluid or gel?) at the interface between diamond and its inclusions. This interposition would imply the presence of a void between the matrix and the inclusion filled by an amorphous material. The void formation by inclusion decohesion or cracking have been commonly found in steel and metal alloys when the stress at the interface between two materials with different lattice constants and different elastic constants exceeds some critical value (Dixit and Dixit, 2014 and references therein). However, to verify if a similar process occurs for diamond, a deep analysis of the diamond-olivine interface should be carried out.Therefore, at this moment we cannot discuss much such scenario.

Conclusions

Based on the aforementioned evidences, a synchronous growth of diamond and olivine inclusions, in our case, appears quite controversial and consequently we will try to suppose that inclusions were trapped in full-grown state. To demonstrate this hypothesis the first question to be solved regards the process under which full-grown inclusions assumed a diamond-imposed morphology. Protogenetic inclusions, having their own morphology, may have changed their shape whether by solid-state diffusion or passing through resorption-recrystallization episodes. The solid-state diffusion, comparing the physical properties of the olivine and the diamond, can be triggered by a different plastic deformation of olivine that eventually could be take the morphology imposed from the diamond. This process can be favored by dislocation creep along the slip systems of the olivine structure and "disclination" formation that can be detected only by means of investigations at nano-scale (Myazaki et al., 2013 and Cordier et al., 2014). Previously, Nestola et al. (2014) considered highly unlikely modification of the inclusion's shape after encapsulation by dislocation creep. In any case, the dislocation creep process in olivine produces crystallographic preferred orientations of grains that, in turn, involves crystallographic alignment of olivine inclusions not found in our specimen (Myazaki et al., 2013).

About the hypothesis regarding resorption-recrystallization process, it is known that the natural diamond crystals brought up from the depth of the Earth passing through the region unstable for diamond have always experienced dissolution (Sunagawa, 1984 and Sunagawa et al., 1984). On the other hand, the rounded morphology of specimen and trigons observed on the octahedral faces invariably testify dissolution. However, it is necessary to establish if the dissolution that could have determined the diamond-imposed morphology occurred during the entrapment of inclusions or after the crystallization of diamond. The rounded morphology of diamond and trigons are certainly effects of post-growth events; while the diamond-imposed morphology and the "stepped figures" observed on the surfaces of inclusion A, suggest a mechanism of selective partial dissolution occurred during the incorporation process, confirming what has been suggested by Nestola et al. (2014) and Bruno et al. (2014). In fact, a modification of the shape of inclusions after the crystallization of the diamond would have to leave any "relict" trace of the previous morphology. It is very difficult to understand that post-growth resorption-recrystallization events have been able to erase defects as dislocations nucleated during the encapsulation of olivines with early orthorhombic

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morphology. The absence of dislocations nucleated from the olivine inclusions and the "stepped figures" observed on the surface of the larger inclusions could be explained with processes of selective partial dissolution, suggesting that the diamond imposed its morphology on the olivine during their encapsulation. Therefore, as also suggested by Bruno et al. (2014), it can be concluded that the bulk of all inclusions is protogenetic, whereas more external regions of A and C olivines, closed to the diamond-inclusion interface, could be syngenetic, confirming that a diamond-imposed morphology alone cannot be considered proof of syngenetic origin of inclusions.

Acknowledgements

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Figure captions

Figure 1. Optical micrographs of sample. a) Transmitted light under parallel nicols.Note three colorless inclusions of olivine previously studied by Nestola et al. (2011), named A, B and C. b) Transmitted light under crossed nicols.Note the anomalous birefringence of diamond. c) Optical micrograph of sample under reflected light.Note the rounded cubo-octahedral morphology with two flattened parallel {111} faces showing typical growth features as well as trigons.

Figure 2. a)Optical micrograph of inclusions with the corresponding schematic drawings (in green) of the reconstructed morphology: the A and C inclusions show a very typical diamond-imposed morphology. b) Stereogram obtained by XRD data showing the relative crystallographic orientations of the olivine inclusions, labelled A and B, and their diamond host (modified from Nestola et al. 2014). c) Enlargement of the morphology reconstruction of the B inclusion taking into account its crystallographic orientation shown in Fig. 2b). The B inclusion shows an elongated morphology much closer to the typical orthorhombic morphology of olivine.

Figure 3. a) Optical micrograph taken under reflecting light, focusing the surface of inclusion A; b) enlargement of the image of a) note the "stepped" figures labelled "*o*" and "*d*".

Figure4. X-Ray Diffraction Topographs taken using MoKα_1 radiation. Arrows show the diffraction vector projection g. a) and b) g=02-2; c) g=-311; d) g= 1 -1 1.The topographic images show only the lack of contrast corresponding to inclusions A and C, whereas the inclusion B is not detectable because of its limited size. PD: plastic post growth deformation. Projection effects of the asymmetric reflections give the difference between the size of sample and the size of the different topographic images.

Figure5. Topographic sections taken to minimize the overlapping of the strain field associate with the different defects. (g=-311) A and C represent the olivine inclusions, D= dislocations. Dark contrasts surround the inclusions. No dislocations nucleated from the olivine inclusionsare observed.