Chapter 10
Going Inside a Diamond

Fabrizio Nestola, Martha G. Pamato, and Davide Novella

Abstract Diamonds are rare minerals but thanks to their exceptional physical properties are able to travel through time (they can be up to 3.5 Ga old) and space (from great depths within the mantle to the Earth's surface) often remaining completely uncorrupted with respect to the surrounding mantle. Time to time, diamonds can encapsulate minerals, providing a window into the deepest regions of the Earth and the processes occurring down to depths of not less than 1000 km. This chapter will outline the importance of diamonds and their mineral inclusions, covering some of the most significant aspects of diamond research.

Keywords Diamonds · Inclusions · Lithospheric mantle · Super-deep formation · Earth's deep interior

10.1 Introduction

Diamonds are not just the most valuable and sort after minerals for jewellery (Fig. 10.1) but they are among the most important geological samples in Earth Science. This is witnessed by more than 6000 published scientific articles (see www.scopus.com) focused on natural diamonds, which allow geologists to understand several deep geological processes. In addition, recently the Mineralogical Society of America (the largest mineralogy society in the world) dedicated an entire volume of 876 pages to the study of diamonds (volume 88 of the Reviews in Mineralogy and Geochemistry). Indeed, diamonds are the deepest materials reaching the surface of the Earth and exhibit an extremely wide range of formation ages, from about 3.6 to 0.5 Ga (Smit et al. 2022). From time to time, diamonds also transport mineral inclusions directly from great depths of our planet; such inclusions, which often remain pristine materials thanks to the protection of the host diamond with respect to the surrounding mantle, represent windows into the deepest regions of the Earth. These regions are completely inaccessible to any mineralogist or geologist and provide

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Fig. 10.1 A 1-carat gem quality diamond, used for jewellery (*Photo* Fabrizio Nestola)

direct insight into what happened (and when…) to the Earth over an extremely wide timespan.

Diamonds are classified in two well distinct categories: (1) lithospheric diamonds, which form between about 120 and 220 km depth, with a global mode at 175 ± 15 km depth (Nimis et al. 2020); (2) sublithospheric or super-deep diamonds, which instead originate much deeper, at depths from about 300 to up to 1000 km. These two categories of diamonds are described in detail in Sects. 10.2 and 10.3, respectively, along with their precious entrapped mineral inclusions. Sections 10.2 and 10.3 also outlines a new approach for determining the depth of formation of diamond-host systems for both lithospheric and super-deep diamonds. Known as elastic geobarometry, this is becoming a widely used technique for characterising host-inclusion geological systems (e.g., Angel et al. 2017; Alvaro et al. 2022).

Finally, Sect. 10.4 focuses on recent discoveries in one of the most debated topic in diamond research, the temporal relationships between diamond hosts and their mineral inclusions: who was born first? Are inclusions pre-existing materials which are then incorporated into younger, growing diamonds or are diamonds and their mineral inclusions formed simultaneously from the same chemical reaction? What are the geological implications of these two possibilities?

10.2 Lithospheric Diamonds

10.2.1 Morphology, Diamond Type, Age, Inclusions

Lithospheric diamonds represent about 99% of all diamonds. For a detailed introduction to lithospheric diamonds we direct readers to the recent review by Stachel et al. (2022). Lithospheric diamonds usually form between 120 and 220 km depth beneath cratonic areas (Fig. 10.2), with an average value of about 175 km (Nimis et al. 2020).

The most common temperature at which lithospheric diamonds originate within the mantle is around 1160 to 1170 (±110) °C (Pasqualetto et al. 2022); however, these precious stones can form over a wide temperature window between about 900 and 1400 °C (Stachel and Harris 2008), and at pressures between about 5 to 7 GPa.

Lithospheric diamonds are characterized generally by very regular morphologies, including the octahedron (Fig. 10.3), the most common habit for lithospheric diamonds, the cube, the dodecahedron and a series of mixed shapes or twins such as the cubo-octahedron, the macles, and others (see Harris et al. 2022 for an extensive review on the morphology of monocrystalline diamonds).

A further and very important feature of lithospheric diamonds is their nitrogen content and nitrogen aggregation state. Nitrogen is the most common impurity within the diamond crystal structure, with nitrogen atoms able to substitute carbon atoms. The nitrogen content in diamond is generally determined by secondary ion mass spectroscopy or, more commonly, by Fourier Transform infrared spectroscopy (FTIR). By using FTIR, it is also possible to determine the so-called "nitrogen aggregation state", which is crucial in classifying the diamond type. In Fig. 10.4, the classification scheme for diamonds, based on aggregation data provided by FTIR, is shown. We direct the readers to the recent and extensive review by Green et al. (2022).

Most of lithospheric diamonds belong to type I, as they have significant amounts of nitrogen impurities which can be present up to about 4000 atomic ppm (part per million), with an average value of about 90 at. ppm (Stachel and Harris 2009).

A very important piece of information about lithospheric diamonds is their age: these diamonds can date from about 3.52 Ga years ago until very recent times (some diamonds have been dated to only 0.01 Ga years ago). This means that diamonds are invaluable sources of information about the Earth over a wide geological timespan. The most recent review on the ages of diamonds is by Smit et al. (2022).

Mineral inclusions in lithospheric diamonds are distinctive of this diamond category and for an in-depth discussion we refer readers to Stachel et al. (2022). The most abundant inclusions in lithospheric diamonds can be listed as follows (mainly after

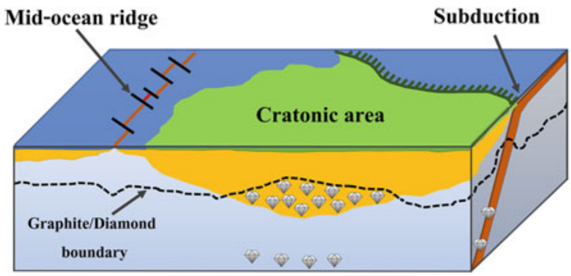

Fig. 10.2 Scheme of a cratonic area under which diamonds crystallize. These areas are far from the convergent plates boundaries. The dashed curve represents the graphite-diamond boundary, which is positioned at about 120–140 km depth; diamond symbols represent diamond formation (the figure is not in scale). All diamonds within the yellow areas are lithospheric, while all other diamonds are defined super-deep diamonds

Fig. 10.3 A lithospheric diamond showing a typical octahedral shape, with black inclusions of Cr-spinel (*Photo* Caterina Canovaro; diamond sample provided by Dr. J. W. Harris). The in this image is 2.5 mm across

Stachel and Harris 2008; the percentages reported below can vary considerably from locality to locality and must, therefore, be taken as an estimate mainly to illustrate which are the most common inclusions):

- garnet $[(Mg,Fe,Ca)_3(Al,Cr)_2Si_3O_{12}] = 32\%$
- olivine $[(Mg,Fe)_2SiO_4] = 16\%$
- Cr-spinel $[(Mg,Fe)(Cr,Fe,Al)_2O_4] = 16\%$
- iron sulphides = 15% [mainly pyrrhotite $Fe_{1\text{-}x}S$].
- clinopyroxene $[(Ca,Na,Mg,Fe,Al)_2Si_2O_6] = 13\%$
- orthopyroxene $[(Mg,Fe,Ca)_2Si_2O_6] = 7\%$
- coesite $(SiO_2) = 1\%$
- rutile $(TiO_2) = 1\%$

Many other inclusions can be found in lithospheric diamonds, but their abundancies are always below 1%.

10.2.2 Depth of Formation of Lithospheric Diamonds by Elastic Geobarometry

It is well established that lithospheric diamonds form between about 120 and 220 km depth. A recent study by Nimis et al. (2020) indicates that the global mode of depth of formation of lithospheric diamonds is at 175 ± 15 km. Most data relevant to determining depth of origin of diamonds are obtained by classical "chemical" data measured on their mineral inclusions; this approach is based on several methods,

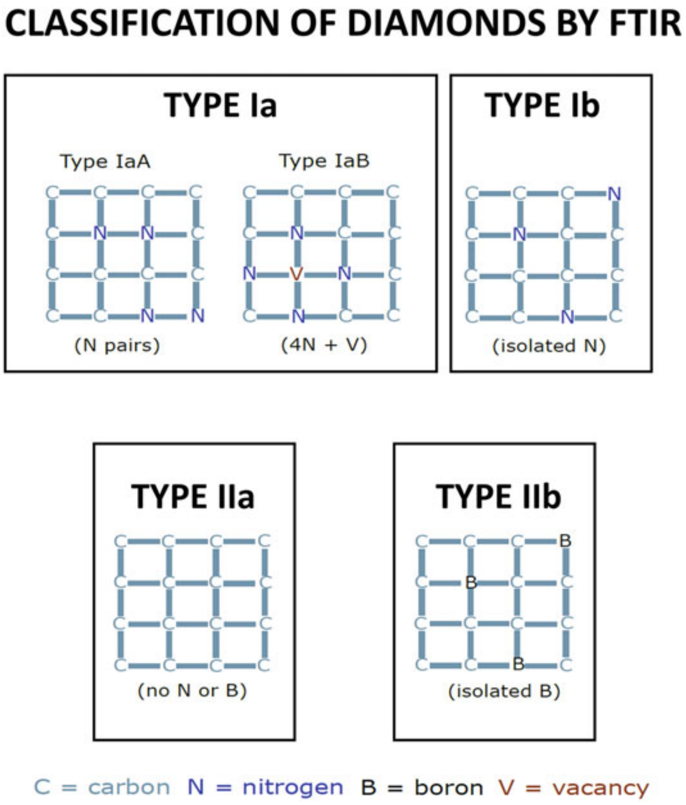

Fig. 10.4 Classification of diamonds carried out by Fourier-Transform Infrared spectroscopy (labels for each atom are reported in the figure). Redrawn after Breeding and Shigley (2009)

and we direct readers to Nimis (2022) for an extensive review. However, more recently, a new approach for determining depth of origin of lithospheric diamonds has been developed, known as "elastic geobarometry". Research by Izraeli et al. (1999), Sobolev et al. (2000), Howell and Nasdala (2008), Howell et al. (2010), Nestola et al. (2011), and Howell et al. (2012) demonstrated the considerable potential of this approach around 10–20 years ago, and the method is now being actively developed and refined, providing very reliable results. The approach is based on the thermo-elastic contrast between a diamond host and its inclusions, and importantly, can be applied to various diamond-inclusion pairs. A recent review on elastic barometry is published by Angel et al. (2022).

The elastic geobarometry method has been successfully for olivine, garnet, kyanite and Cr-spinel inclusions in diamonds. Specifically, elastic geobarometry provided pressures of formation of 4.8–6 GPa for temperatures between 930 and 1250 °C for garnet inclusions in lithospheric diamonds from Borneo (Kueter et al. 2016), a pressure of 5.2 GPa for a temperature of 1120 °C for a kyanite inclusion in diamond from Voorspoed (South Africa) (Nestola et al. 2019a), a pressure of 6.5 GPa for a temperature of 1125 °C for a Cr-spinel inclusion within a diamond from Udachnaya (Russia) and a pressure of formation of 6.2 GPa for a temperature of 1200 °C for an olivine inclusion within a Chinese diamond (Wang et al. 2023).

Summarizing, data obtained by elastic geobarometry on lithospheric diamonds have, to date, involved four types of inclusions of which at least three are among the most abundant ones (garnet, olivine, Cr-spinel). It is also evident that this approach nearly parallel the modal global depth proposed by Nimis et al. (2020), with a pressure, in average, close to 5.9 GPa, corresponding to a depth equal to about 175 km. This is very promising, and indicates that the method could be a robust approach which, in principle, can be applied to any inclusion within a diamond. As explained in Angel et al. (2022), the elastic geobarometry approach does, however, require precise and accurate constraints on the thermoelastic parameters of mineral inclusions in diamond. Unfortunately, these data are not always available, even for some common minerals.

10.3 Super-Deep Diamonds

10.3.1 Morphology, Diamond Type, Age, Inclusions

As we mentioned earlier, super-deep diamonds are those considered to have crystallized much deeper than lithospheric diamonds, between about 300 and 1000 km depth (for example, see Stachel et al. 2005; Walter et al. 2011; Pearson et al. 2014; Smith et al. 2018). Super-deep diamonds are certainly the deepest minerals reaching the Earth's surface and thus, when they entrap minerals, provide a window on the deepest regions of our planet (see Fig. 10.5 for examples of typical super-deep diamonds). For an extensive review on super-deep diamonds we direct the readers to Walter et al. (2022) and references therein. Unfortunately, for those scientists that study diamonds, super-deep diamonds only represent about 1% of all diamonds, and their extreme rarity makes their investigation very challenging.

The age of super-deep diamonds is, currently, one of the most complex, unsolved problems in diamond research. Until 2010, no age data for super-deep diamonds had been reported in the literature. Bulanova et al. (2010) published the first age for a super-deep diamond from Brazil, reporting a very young age of 0.101 ± 007 Ga, much younger than the average of lithospheric diamonds. As also reported in the extensive review by Smit et al. (2022), later, unpublished data (Smith and Shirey, unpublished data) on the same diamond studied by Bulanova et al. (2010) suggests an age of 0.650 ± 0150 Ga, which is notably older than the first age reported but still young with respect to lithospheric diamonds. Hutchison et al. (2012) reported an age of 1.27 Ga, again on a Brazilian diamond. From these studies, it is evident that, at present, any discussion about the age of super-deep diamonds is limited by a lack of data.

Fig. 10.5 Typical super-deep diamonds with irregular shapes. The diamonds are tens carats (*Photo* © GIA. Reprinted by permission)

In comparing lithospheric diamonds and super-deep diamonds it appears clear that there are many distinguishing differences, the most important of which, beyond the depth of formation, are as follows:

- super-deep diamonds often show extremely low nitrogen contents, with an average lower than 10 at. ppm and very often with values close to zero (Stachel et al. 2009); thus, super-deep diamonds are usually classified as Type II;
- super-deep diamonds lack regular morphologies, in contrast to the very regular shapes exhibited by lithospheric diamonds;
- super-deep diamonds entrapped very unusual and distinctive mineral inclusions; in detail, the most abundant mineral in super-deep diamonds is periclase (Mg,Fe)O (improperly termed 'ferropericlase' in the literature), followed by breyite $CaSiO_3$ (Joswig et al. 1999; Brenker et al. 2021) (previously referred to as $CaSiO_3$-walstromite), majoritic garnet, low-nickel enstatite (in super-deep diamond literature, enstatite associated to ferropericlase is considered a retrogressed bridgmanite when has low or no Ni), larnite Ca_2SiO_4, $CaSi_2O_5$ titanite-structure, jeffbenite (with the same stoichiometry as a garnet, Nestola et al. 2016), $CaTiO_3$ perovskite, olivine or its high-pressure form ringwoodite (see Pearson et al. 2014; Lorenzon et al. 2022 and Gu et al. 2022), in rare instances clinopyroxene, and SiO_2 (likely as a retrogressed phase from stishovite), and a series of other less abundant phases (see Kaminsky 2012 and Walter et al. 2022 for extensive reviews about inclusions in super-deep diamonds).

10.3.2 Depth of Formation of Super-Deep Diamonds by Elastic Geobarometry

The main problem in applying elastic geobarometry to super-deep diamonds is that members of this rare category of diamond show evidence for strong plastic deformation (Ragozin et al. 2020); therefore the approach, successfully developed for lithospheric diamonds, in terms of elasticity, cannot be reliably used to determine the depth of super-deep diamond formation. This means that any attempt to determine the depth of formation of super-deep diamonds using elastic geobarometry can only provide a minimum pressure determination. However, recent work on a breyite inclusion, $CaSiO_3$, (Anzolini et al. 2018) demonstrated that diamond-breyite pairs can provide meaningful results even in terms of minimum pressure determinations. Considering that breyite is the second most abundant mineral in super-deep diamonds (Brenker et al. 2021), this is certainly quite promising.

A novel approach in determining the depth of formation of super-deep origin diamond using the elastic geobarometry method was developed in 2019 by Anzolini and coauthors, who studied ferropericlase inclusions in Brazilian diamonds. Anzolini et al. (2019) attempted to develop a novel plasto-elastic geobarometry method which takes into account changes in the physical properties of a diamond host during its ascent to the Earth's surface. This model reasonably assumes that diamond behaves elastically in the upper mantle, and behaves plastically in the transition zone and lower mantle. This novel approach applied to diamond-ferropericlase from Brazil provided a depth of formation within the transition zone at about 15.7 GPa. Similar values were obtained on ferropericlase inclusions in blue diamonds (Smith et al. 2018, see Fig. 10.6). Although there is significant complexity behind this method, we consider it a good starting point in developing the most reliable methods to retrieve depth of origin for super-deep diamonds. As remarked by Anzolini et al. (2019), in order to get more reliable depth data it will be necessary to determine the strength of diamond to much higher temperatures and pressures than currently provided in the literature (e.g., 10 GPa and 1550 °C, Weidner et al. 1994).

More conventional approaches to obtaining the depth of formation or origin of super-deep diamonds are available in the literature. For example, the composition of majoritic garnets is often used for this purpose (see the recent work by Thomson et al. 2021). Pressure–temperature stability fields of specific minerals can also be used to constrain the origin of super-deep diamond. For example, the presence of ringwoodite in diamonds (see Pearson et al. 2014; Gu et al. 2022; Lorenzon et al. 2022) constrains the depth of origin to between about 525 and 660 km in the transition zone, and the association of ferropericlase and low-Ni enstatite in super-deep diamonds is often considered a typical lower mantle assemblage, i.e., a depth of origin below the 660 km depth (see Stachel et al. 2005), as enstatite is thought to be a retrograde product of bridgmanite. An inclusion of $CaSiO_3$ perovskite with a certain TiO_2 content can constrain the depth of diamond formation to more than 22 GPa (Nestola et al. 2018a, b). Furthermore, the presence of post-spinel phases constrains the formation pressure to a minimum value of 18 GPa (see Agrosì et al. 2019). Many other examples could

Fig. 10.6 Ferropericlase inclusions (50–100 μm black minerals) in blue, super-deep diamonds (Smith et al. 2018). *Photo* by Evan Smith, GIA

be cited here, however, a detailed and recent review on P–T data on diamonds can be found in Nimis et al. (2022).

10.4 Temporal Growth Relationship Between Diamond and Its Mineral Inclusions

One of the most important debates in diamond research is the temporal growth relationship between diamond and its mineral inclusions: which was born first? Were inclusions pre-existing materials before their encapsulation during later diamond growth, or were inclusions and diamonds created simultaneously as a consequence of the same or related chemical reactions? Furthermore, what are the geological implications of one or the other possibility?

Before discussing such important aspects of diamond research, it is necessary to remark that in the literature, inclusions born before their diamond hosts are called "protogenetic", while inclusions formed together with their diamond hosts are referred to as "syngenetic". Finally, those inclusions that were formed after diamond formation are termed "epigenetic" and are not usually relevant in terms of geological implication.

It is evident that if an inclusion is syngenetic, any implications regarding its petrology, geochemistry, and mineralogy can also be extended to its diamond host. In this instance, the geochemical and petrological environment under which a diamond formed within the mantle, and indeed the age of formation, are assessed by studying its mineral and/or fluid inclusions. On the other hand, the study of protogenetic inclusions cannot completely guarantee that what we get in terms of petrology, geochemistry and age can be directly applied to their diamond hosts.

How can we understand whether an inclusion is syngenetic or protogenetic? Unfortunately, this is not a trivial exercise and many authors have faced this challenging question.

In the literature, syngenesis is generally assumed based on two main arguments:

(1) Epitaxial growth between diamond and its inclusions (see Orlov 1977). Epitaxial growth, i.e., the coincidence of crystallographic planes between a host diamond and a given mineral inclusion in case of diamond-inclusion system, is not definitive evidence of syngenesis; indeed, even if we accept that a diamond and one of its inclusions have grown epitaxially, it is not clear why this should readily imply synchronicity. In fact, for epitaxial growth of a mineral on a substrate, it is necessary that the substrate is already pre-existing before mineral growth commences. For instance, it is reasonable to postulate that a diamond can grow with an epitaxial relationship to an inclusion substrate where the inclusion is 2 Ga years older than the diamond. In addition, at least for all common inclusions in diamonds (garnet, olivine, sulphides, Cr-spinel, clinopyroxenes), it has not yet been conclusively demonstrated that they show epitaxial relationships with their hosts (see Nestola et al. 2014, 2017, 2019b; Milani et al. 2016; Nimis et al. 2019; Pamato et al. 2021; Pasqualetto et al. 2022).
(2) Shape of the inclusions within their diamond hosts. Very often, inclusions in diamonds show inherited diamond shapes and not their own symmetry-allowed morphologies or habits. This is typical for almost all inclusions in diamonds from olivine to garnets, from pyroxenes to Cr-spinels, and others (see for example Fig. 10.7). This common feature has, almost always, been considered as possible evidence of syngenesis (e.g., Meyer 1985 and others). However, although the imposition of diamond shape on inclusions still needs to be addressed from a quantitative point of view, recent crystallographic research has demonstrated that even protogenetic inclusions can exhibit shapes imposed by host diamonds (see Milani et al. 2016 in which more than 60 olivine inclusions in diamonds from various provenances worldwide were investigated).

On the other hand, arguments in favour of protogenesis can be here summarized based on a series of published studies. For example, Thomassot et al. (2009) demonstrated that sulphide inclusions in Jwaneng diamonds, based on geochemical evidence, were certainly pre-existing materials, i.e., protogenetic [in accordance with the findings of Spetsius et al. (2002) and Taylor and Anand (2004)]; Bruno et al. (2016) concluded that olivine inclusions (and likely other silicates) have no chemical affinity with host diamonds; Jacob et al. (2016), based on plastic deformation features in sulphides, inferred that such minerals must have crystallized before being encapsulated by the diamond host; Milani et al. (2016), Nestola et al. (2017), Pamato et al. (2021), and Pasqualetto et al. (2022) demonstrated that olivine, clinopyroxene, Fe-sulphides, and garnet inclusions in diamonds are often clearly protogenetic, occurring as clusters of inclusions with the same crystallographic orientation, but at the same time, with a random orientation overall with respect to their diamond hosts (see Fig. 9 of Angel

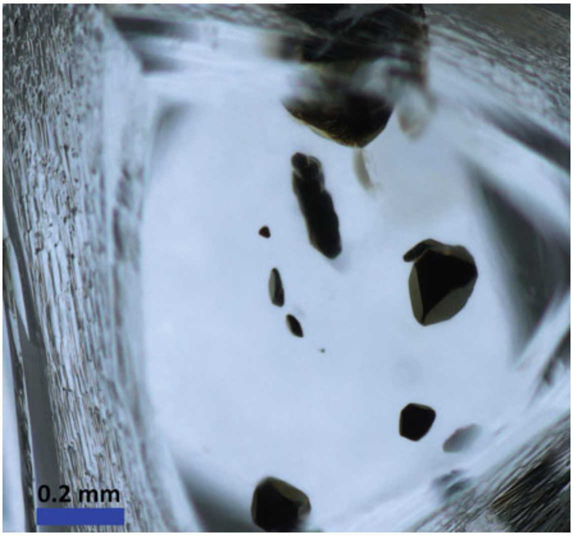

Fig. 10.7 Cr-spinel inclusions in lithospheric diamonds with characteristic inherited diamond shapes (*Photo* Caterina Canovaro; diamond sample provided by Dr. J. W. Harris)

et al. 2022 for a detailed explanation). This crystallographic evidence (also reported in some cases for Cr-spinels, Nimis et al. 2019) has been interpreted as a 'smoking gun' for pre-existing inclusions before passive entrapment by diamonds (an extensive review on this approach is given by Angel et al. 2022).

Summarizing and comparing arguments in favour of syngenesis and/or protogenesis, it is evident that arguments in favour of syngenesis are often weak and poorly supported by quantitative data, while those in favour of protogenesis are definitively more robust, at least at present. However, in general it is still reasonable to conclude that definitive evidence for whether any type of inclusion is syngenetic and/or protogenetic is lacking.

In discussing the syngenesis/protogenesis issue, it is clear that one of the most critical aspects is the age of diamond hosts. To date diamonds, it is necessary to date their mineral inclusions using the well-known Sm–Nd, Re–Os, Rb–Sr, Pb–Pb, Ar–Ar isotopic systems (see Smit et al. 2022). However, in case of protogenetic inclusions, the risk is that their age does not coincide with that of their diamond hosts. However, three recent studies demonstrated that even in case of protogenesis, model ages of inclusions and diamonds could be synchronous, e.g., encapsulation by diamond at high temperature would reset the isotopic system, and thus the model age of the inclusion would match that of diamond formation (see Nestola et al. 2019b; Pamato et al. 2021; Pasqualetto et al. 2022). This happens when cation diffusion within inclusion's crystal structure is rapid enough to allow re-equilibration and resetting of the isotopic system at the time of the diamond entrapment. Nestola et al. (2019b) demonstrated that model ages obtained from protogenetic garnets would be synchronous with formation of diamond hosts in instances where Sm–Nd systematics were applied to crystals smaller than about 0.1 mm, for temperatures of diamond crystallization exceeding 1000 °C. Similarly, Pamato et al. (2021) demonstrated that protogenetic sulphides dated using the Re-Os system are synchronous with their diamond hosts. In contrast, Pasqualetto et al. (2022) showed that dating protogenetic clinopyroxenes using Sm–Nd systematics almost never provides the age of diamond.

10.5 Conclusions and Outlook

The importance of natural diamonds in Earth Science research is evident. Diamonds and their inclusions provide key information on a wide range of geological processes, from the greatest depths to the surface and vice versa. We have learnt much from the study of natural diamonds, and diamond research has opened a new branch of science focussed on those regions of our planet that would, otherwise, remain inaccessible. Very often, investigation of diamonds and their inclusions allows us to confirm or build upon key findings and discoveries from laboratory studies (i.e., the discovery of hydrous ringwoodite in diamonds, Pearson et al. 2014, Gu et al. 2022, which had only been predicted by laboratory experiments before 2014; breyite, $CaSiO_3$, that Gasparik et al. 1994 synthesised in the laboratory before its discovery in diamonds; titanite-structured $CaSi_2O_5$, found in diamond by Brenker et al. 2005, which was first synthetized in the laboratory; and finally tetragonal ZrO_2, Lorenzon et al. 2022, which had not been found in nature before 2022). There is enormous potential for future work in this exciting research field, as we unlock new secrets of the Earth's deep interior.

In recent years, mineralogy has become a key area within the field of diamond research. Mineral inclusions allow scientists to constrain the geochemical environments and mechanisms of diamond formation, but they also allow them to understand the depths and temperatures at which they form, and not least, the age of the diamond formation. What questions remain to be answered about natural diamonds?

We have learnt much about the depths and temperatures at which lithospheric diamonds form beneath the world's cratonic areas, and about how and when lithospheric diamonds formed. However, we do not yet know why many inclusions have diamond-imposed shapes, or fully understand why abundances of mineral inclusions within lithospheric diamonds differ markedly from those we expect in diamond source regions based on laboratory experiments (e.g., experiments predict that the mineralogy of the subcratonic mantle should be approximately 60% olivine, 20% garnets, 20% pyroxene). There remain many unanswered questions regarding super-deep diamonds. Currently, we do not really know when super-deep diamonds formed, or the growth relationships between inclusions and super-deep diamonds. We do not know how deep in the lower mantle diamonds can form, and what might control the maximum depth of diamond formation. We have yet to discover demonstrable mineral inclusions of the $MgSiO_3$ bridgmanite structure within super-deep diamonds, even though this is volumetrically the most abundant mineral in the lower mantle. We do not know why this might be; super-deep diamonds instead contain inclusions of low-Ni enstatite, and perhaps we will never find bridgmanite. Although we have discovered inclusions of ringwoodite, we have yet to discover inclusions of the other major component of the mantle transition zone, wadsleyite. Why this is the case it remains a mystery. Many other unanswered questions regarding super deep diamonds could be added. Without doubt, mineralogy has much to teach us about the nature of diamond formation and of the deepest regions of the Earth.

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