Publisher: GSA
Journal: GEOL: Geology
DOI:10.1130/G45605.1

Depth of diamond formation obtained from single periclase inclusions

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ABSTRACT

Super deep diamonds (SDDs) are those that form between ~300 and ~1000 km in the Earth's mantle. They comprise only 1% of the entire diamond population but play a pivotal role in geology, as they represent the deepest direct samples from the interior of our planet. Ferropericlase, (Mg,Fe)O, is the most abundant mineral found in SDDs and, when associated with low-Ni enstatite, which is interpreted as retrogressed bridgmanite, is considered proof of a lower-mantle origin. As this mineral association in diamond is very rare, the depth of formation of most ferropericlase inclusions remains uncertain. Here we report geobarometric estimates based on both elasticity and elasto-plasticity theories for two ferropericlase inclusions, not associated with enstatite, from a single Brazilian diamond. We obtained a minimum depth of entrapment of 15.7(±2.5) GPa at 1830(±45) K [≈450(±70) km depth], placing the origin of the diamond-inclusion pairs at least near the upper mantle-transition zone boundary and confirming their super-deep origin. Our analytical approach can be applied to any type of mineral inclusion in diamond and will expectedly allow better insights into the depth distribution and origin of SDDs.

INTRODUCTION

Diamonds, and the mineral inclusions they trap during their growth, are pristine samples from the Earth's mantle and provide information on processes operating in inaccessible regions of our planet. This information is particularly valuable if it can be combined with depth estimates. Based on the mineral inclusions, the majority of diamonds (99%) originate within the lithosphere (Stachel and Harris, 2008). The other 1% are sub-lithospheric and formed at depths between 300 and ~1000 km, and hence are called super-deep diamonds (hereafter SDDs) (Walter et al., 2011; Pearson et al., 2014; Smith et al., 2016; Nestola et al., 2018).

Based on experimental evidence, bridgmanite and ferropericlase (fper) are the most abundant minerals in the lower mantle, comprising approximately ~75 and ~17 wt%, respectively (Stixrude and Lithgow-Bertelloni, 2012, and references therein). On decompression, bridgmanite inverts to Al-rich, low-Ni enstatite (Stachel et al., 2000), while fper can remain stable to room pressure. Early inclusion work (Harte et al., 1999; Stachel et al., 2000) concluded that fper was a lower mantle mineral, especially when found in the same diamond as low-Ni enstatite, but the findings of fper in association with olivine and jeffbenite in some SDDs (Hutchison et al., 2001) cast doubt on that conclusion. Indeed, ringwoodite is in equilibrium with fper at 24 GPa (Brey et al., 2004) and it could have later reverted to olivine, whereas jeffbenite is only stable up to 13 GPa (Armstrong and Walter, 2012), even if its origin is still controversial. In addition, the observation of droplets of Fe-Ni alloys in some Fe-enriched fpers induced Hayman et al. (2005) to outline a model which ascribes the Fe-rich character to equilibration with silicates in the deeper part of the lower mantle (1700-2900 km). On the other side, synthesis of fper and diamond by carbonate melt-peridotite reactions (Thomson et al., 2016) suggested that fpers inclusions with variable Fe contents can form at lower upper-mantle to transition-zone depths. The presence of nanometric exsolutions of magnesioferrite in some fper inclusions (Harte et al., 1999; Wirth et al., 2014; Kaminsky et al., 2015) lead Palot et al. (2016) to propose an origin in the uppermost part of the lower mantle, but Uenver-Thiele et al. (2017a,b) showed that magnesioferrite cannot exsolve directly from fper in the lower mantle.

In aiming to identify a method for determining the depth of origin of fper inclusions completely independent of mineral paragenesis, Hutchison (1997) combined sophisticated thermoelastic modelling with measurements of periclase cell parameters before and after release from diamonds from the São Luiz River, Juina, Brazil and Guinea. Hutchison and Harris (1998) reported an absolute minimum depth of formation of 320 km (equivalent to an entrapment pressure, P_{trap}, of 11 GPa) uncorrected for the brittle deformation evident in the diamond host. This study provided strong evidence for super deep origins for the samples analyzed, however, limitations were imposed by uncertainties in the Gandolfi camera measurement technique available at the time and full quantification of plastic and brittle diamond deformation. In this study, we have been able to extend the original work with improved certainty and propose an updated method for determining minimum P_{trap} applied to two fper inclusions in a further diamond from São Luiz (sample AZ1, Fig. 1). The reverse calculation of P_{trap} was performed by applying the elastic geobarometry approach (Angel et al., 2014; 2015a,b; 2017), including the full geometry of the inclusions based on a realistic 3D reconstruction (Mazzucchelli et al., 2018), coupled with a new elasto-plastic model to account for plasticity of the diamond host at high temperature.

METHODS

Sample

The diamond investigated in this study (Fig. 1) was recovered in the mid to late 1980s from alluvial deposits of the São Luiz river in the Juina area of Mato Grosso State, Brazil. The sample contains two main black tabular inclusions, identified as fper [$(Mg_{0.60}Fe_{0.40})O$; see below] by SCXRD (see Supplemental Information). The smaller one, whose longest dimension is ~160 µm, is named AZ1_1; the bigger one, whose longest dimension is ~340 µm, is named AZ1_2.

Synchrotron X-ray Tomographic Microscopy

This non-destructive, high-resolution technique creates three-dimensional maps of the variations of the X-ray attenuation coefficient within a sample. X-ray micro-tomography experiments were carried out at the Swiss Light Source (SLS) at TOMCAT, a beamline for TOmographic Microscopy and Coherent rAdiology experimenTs (Stampanoni et al., 2006). Measurements were performed at 13.5 keV in order to maximize contrast. A total of 1501 X-ray radiographs were acquired from different angular positions around a vertical rotation axis for each sample. The used imaging setup consisted of a 20 µm thick LuAG:Ce scintillator screen, a 20 × objective and a sCMOS (PCO.edge) camera. The tomographic reconstruction was performed using optimized routines based on the Fourier Transform Method (Marone and Stampanoni, 2012). The resulting volume consisted of 2160 axial slices of 2560 × 2560 pixels, with a pixel size of 0.33 µm.

Single-Crystal X-ray Diffraction (SCXRD)

SCXRD measurements were performed on the fper inclusions both before and after release from their diamond host at the Department of Geosciences (University of Padova). X-ray data were collected using a Rigaku Oxford Diffraction *SuperNova* single-crystal diffractometer, equipped with a Dectris *Pilatus* 200 K area detector and with a *Mova* X-ray microsource. A monochromatized Mo*K*a radiation ($\lambda = 0.71073$ Å), working at 50 kV and 0.8 mA, was used. The sample-to-detector distance was 68 mm. Data reduction was performed using the CrysAlisPro software (Rigaku Oxford Diffraction).

Field Emission Gun—Scanning Electron Microscopy (FEG-SEM)

The two fper inclusions were first extracted by mechanical crushing of the host, then polished in a three-step process and finally carbon coated. FEG-SEM measurements were carried out at the Department of Physics and Astronomy (University of Padova), using a *Zeiss* SIGMA HD FEG-SEM microscope operating at 20 kV, with a spot size of ~1 nm. Imaging was performed using an InLens secondary electron detector. Compositional analysis was performed using an energy dispersive X-ray spectrometer (EDX by Oxford Instruments). The spatial resolution in microanalysis was of ~1 μm.

Finite Element (FE) analysis

The FE analysis was performed on the real 3D model built from the segmentation of the X-ray microtomographic data (Fig. 2). The surface of the model was smoothed to improve the quality of the final FE mesh and the final 3D model was then assembled placing the two inclusions in the diamond host. An elastically isotropic analysis was run with Simulia Abaqus, a commercial engineering package for FE analysis (for more details see Mazzucchelli et al., 2018). For the fper inclusions we used the isothermal bulk modulus K_{0TR} = 162(14) GPa from the Equation of State (EoS) as reported in Angel et al. (2017). This EoS was obtained fitting the original P-V-T data of Mao et al. (2011) up to 2000 K and 50 GPa using a 3rd-order Birch-Murnaghan EoS combined with a Berman-type thermal expansion. The Reuss shear modulus G_{0R} = 87(2) GPa was obtained from the elastic constants reported by Jacobsen et al. (2002) for a fper with composition $(Mg_{0.63}Fe_{0.37})O$ that is close to the composition of our inclusions, $(Mg_{0.60}Fe_{0.40})O$. For diamond we used the K_{0TR} = 444(2) GPa from the P-V-T EoS reported by Angel et al. (2015a) and the G_{0TR} = 535 GPa reported by Angel et al. (2015b).

Elasto-plastic Model

The calculation is split into two steps dividing the calculation into an isothermal, quasi-static decompression from P_{trap},T_{trap} to P_{room},T_{trap}, followed by an isobaric cooling to room temperature. The model is solved by inversion. The host-inclusion system is initially at P_{room},T_{room} with the inclusion at the experimentally measured P_{inc}^{exp}. First, an entrapment temperature (T_{trap}) is chosen and the over-pressure $P_{inc}^{P_{room},T_{trap}}$ developed in the inclusion during isobaric heating to P_{room},T_{trap} is calculated adjusting the elastic properties of the host and the inclusion according to their EoS. A P_{trap} is guessed at the chosen T_{trap}, and the elasto-plastic deformation of the host and inclusion pressure are calculated during the quasi-static decompression of the host from P_{trap},T_{trap} to P_{room},T_{trap} according to Campione (2018). The guessed P_{trap} is adjusted until the pressure calculated in the inclusion at P_{room},T_{trap} matches the previously found $P_{inc}^{P_{room},T_{trap}}$. The elastic properties for diamond are from Angel et al. (2015a) and from Zouboulis et al. (1998). The variation of σ_{Y} with T (between 1273 and 1823 K) was obtained from Weidner et al. (1994). The EoS of the inclusion is from Angel et al. (2017) as discussed in the main text.

RESULTS AND DISCUSSION

Sample Analysis

The 3D reconstruction (Fig. 2) revealed the absence of significant fractures at inclusion terminations. However, graphitization in haloes around the inclusions (Fig. 1) suggests that some pressure release by brittle deformation of the host diamond may have occurred. Both inclusions after release and polishing exhibited pervasively and homogeneously distributed exsolutions of magnesioferrite of about ~200 nm size, which often coalesced into chains of 2–3 μm length and constituted ~6% of the total surface area (calculated using the ImageJ software, Abràmoff et al., 2004). EDX analyses gave a composition of $(Mg_{0.61}Fe_{0.39})O$ for AZ1_1 and $(Mg_{0.59}Fe_{0.41})O$ for AZ1_2; therefore we consider them to have a similar approximate composition of $(Mg_{0.60}Fe_{0.40})O$ (Figure DR1).

Inclusion Residual Pressures

X-ray analyses (Figure DR2) provided the lattice parameters and the relative unit-cell volumes reported in Table DR1. By comparing the unit-cell volumes before (V) and after (V_0) release from the diamond host and using the P-V-T equation of state (EoS) for fper reported in Angel et al. (2017), we obtained a residual pressure, P_{inc}, of 1.84(±0.65) GPa for inclusion AZ1_1 and of 1.48(±0.67) GPa for inclusion AZ1_2. The high uncertainties in P_{inc} are due to the high uncertainty in the bulk modulus value of fper (Mao et al., 2011). Values of P_{inc} are consistent with 1.29 (±0.38) GPa for the Guinean diamond of Hutchison and Harris (1998) where in this case the uncertainty is confined to that of measurement of cell parameters.

Depth of Formation of the Ferropericlase—Diamond Pair by Elasto-plastic Geobarometry

Given the absence of significant fracture systems around the inclusions, the calculated P_{inc} can be linked to the depth of formation by elastic geobarometry. Standard elastic methods rely on simplified models which assume the inclusion is spherical and sitting isolated in an infinitely large host (e.g., Zhang, 1998). Mazzucchelli et al. (2018) extended the model to non-spherical inclusions and showed that platy inclusions develop a lower P_{inc} compared to more rounded inclusions. This is consistent with our measurements, which show a lower P_{inc} for the platy AZ1_2 than for the more rounded

AZ1_1. The method of Mazzucchelli et al. (2018) enabled us to calculate the appropriate geometrical correction factor (Γ) for the two inclusions through an integration over their entire volumes. The Γ factors obtained in this way are –0.016(5) and –0.080(10) for inclusions AZ1_1 and AZ1_2, respectively. Applying the correction factor to our experimental determined residual pressures we obtained the corrected P_{inc} of 1.87(±0.66) GPa and 1.61(±0.73) GPa for the inclusions, respectively.

We then calculated the entrapment isomeke for the two fper–diamond pairs using the corrected values for P_{inc} and the software EosFit-Pinc (Angel et al., 2017). Since both the host and the inclusion have cubic crystallographic symmetry, the effect of anisotropic elasticity is limited (see Anzolini et al., 2018), allowing the use of current isotropic elastic geobarometry models. To maintain consistency with the calculation of the geometrical factors, we used the P-V-T EoS of fper and diamond and the shear modulus $G_{0\mathrm{TR}}$ = 535 GPa of diamond all respectively reported previously (Angel et al., 2015a,b; 2017). The intersection of the isomeke with the mantle adiabat, accounting for the isomeke and the adiabatic uncertainties, gave an entrapment pressure for AZ1_1 of P_{trap} = 13.5(±1.8) GPa at a temperature T_{trap} = 1802(60) K and for AZ1_2 of P_{trap} = 12.8(±1.8) GPa at a temperature T_{trap} = 1794(60) K (see Table DR2 and Fig. 3).

This estimate does not take into account plastic deformation in the diamond, which may accommodate part of the inclusion expansion during uplift to surface (Anzolini et al., 2016). Plastic deformation is well documented in diamond and, particularly, in SDDs (e.g., Cayzer et al., 2008), consistent with its low yield strength (σ_Y) at high temperatures (Weidner et al., 1994). Therefore, the P_{trap} calculated from a purely elastic model is likely to be underestimated. To account for plastic deformation, the elasto-plastic (EP) model for barometry proposed by Campione (2018) (see Methods) was applied to these data. The reverse calculation of $P_{\text{trap EP}}$ as a function of T was solved by adjusting the σ_Y of diamond according to the experimental measurements of Weidner et al. (1994) and the elastic parameters for diamond and fper, previously noted. Since the EP model assumes that the inclusion is spherical, we applied this method only to the most rounded of the two inclusions, i.e., AZ1_1. The best agreement between the calculated $P_{\text{trap EP}}$ (T) and the adiabat, with its uncertainty, is at 15.7(±2.5) GPa and 1830(±45) K [≈450(±70) km depth]. Considering the uncertainties, this result is compatible with an origin in the lowermost upper mantle or, more probably, in the upper transition zone (Fig. 3). Unfortunately, the depth obtained is constrained by a lack of experimental values of σ_Y when temperatures are higher than ≈1850 K (Weidner et al. (1994) and the fact that the EP model only considers the deformation caused by over-pressurization of the inclusion with respect to the external lithostatic pressure (Campione, 2018). If external tectonic stresses act on diamonds during uplift through the sub-lithospheric mantle, they may promote additional plastic deformation, which may contribute to the release of part of the P_{inc} being built on the inclusion. Therefore, the $P_{\text{trap EP}}$ value of 15.7(±2.5) GPa for AZ1_1, which corresponds to a depth of ≈450(±70) km, should be regarded as a minimum estimate.

In addition, models used in this work do not take into account the effect that the magnesioferrite exsolutions (see Supplementary Information) may have on P_{inc} and, in turn, on the calculated P_{trap}. However, given the small contrast in elastic properties between fper and magnesioferrite (Reichmann and Jacobsen, 2004) and the small volume ratio (~6%) between these two minerals, the effect is probably limited and well within the uncertainties already accounted for in the calculations.

CONCLUSIONS

Our newly devised method for inclusion barometry, which incorporates an elasto-plastic treatment of the inclusion–host system and a correction for geometrical effects based on a real 3D model, can be extended to several other types of inclusions trapped in SDDs. Our analyses demonstrate that for the fper samples studied an origin much shallower than the transition zone (Thomson et al., 2016) can certainly be excluded. Although the present results may only provide minimum pressure estimates, they yield valuable constraints independent of mineral phase relations on the depth of origin of SDDs and thereby increase our knowledge of those inaccessible regions which play a key role in the Earth’s dynamics and deep carbon cycle.

ACKNOWLEDGMENTS

This investigation was financially supported by Fondazione Cassa di Risparmio di Padova e Rovigo and by the project INDIMEDEA, funded by the ERC-StG 2012 to F.N. (grant n° 307322). M.A. has been supported by the Italian SIR-MIUR MILE DEEp (grant n° RBSI140351) and the ERC-StG 2016 TRUE DEPTHS (grant n° 714936). The synchrotron work was carried out at the TOMCAT beamline of the Swiss Light Source at the Paul Scherrer Institut, Switzerland. The authors thank M. Stampanoni, S. Milani and G. Rustioni for help with this work. L. Tauro and N. Michieli are acknowledged for their help in the SEM-EDX sample preparation and analysis, respectively. S. Castelli is thanked for providing the macrophotography of the diamond. The DeBeers Group of Companies is thanked for the donation of the diamond to J.W.H. We thank Felix V.

Kaminsky and two anonymous reviewers whose constructive suggestions helped us to improve the manuscript. We thank Chris Clark for careful editorial handling.

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FIGURE CAPTIONS

Figure 1. The inclusion-bearing diamond studied in this work.

Figure 2. 3D model of the ferropericlase inclusions built from the segmentation of the X-ray microtomographic dataset. It preserves the morphology of the two inclusions and their mutual distances and orientations and reveals the absence of significant fractures around the inclusions. The pressure calculated by FE analysis is not homogeneous within the inclusions. The final residual pressures (P_{inc}) reported in the text are obtained for each inclusion as the average of the pressure over their entire volume and include also the uncertainty in the calculation.

Figure 3. Minimum entrapment pressures of the ferropericlase inclusions determined by elastic and elasto-plastic models. The geotherm is calculated for a typical cratonic surface heat flow of 40 mW/m^2 (Hasterok and Chapman, 2011) and a mantle adiabat (Katsura et al., 2010; Trubitsyn and Trubitsyna, 2015). Entrapment pressures (P_{trap}) calculated for inclusions AZ1_1 and AZ1_2 at various T with the purely elastic model are represented by blue and green diamonds, respectively. The $P_{\mathrm{trap\ EP}}$ calculated with the elasto-plastic model for inclusion AZ1_1 at T consistent with the adiabat, and its uncertainty, is represented by the orange box.

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