



# The depth of sub-lithospheric diamond formation and the redistribution of carbon in the deep mantle



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## ABSTRACT

Most diamonds form in the Earth's lithosphere but a small proportion contain Si-rich majoritic garnet inclusions that indicate formation in the deeper mantle. The compositions of syngenetic garnet inclusions can potential yield information on both the depth and mantle lithology in which the diamonds formed. Pressure dependent changes in garnet compositions have been calibrated using the results of experiments conducted in a multi-anvil apparatus at pressures between 6 and 16 GPa and temperatures of 1000 to 1400 °C. Using the results of these experiments a barometer was formulated based on an empirical parameterisation of the two major majoritic substitutions, referred to as majorite ( $Maj$ ;  $Al^{3+} = Mg^{2+} + Si^{4+}$ ), and Na-majorite ( $Na-Maj$ ;  $Mg^{2+} + Al^{3+} = Na^{+} + Si^{4+}$ ). Moreover, previously published experimental garnet compositions from basaltic, kimberlite, komatiite and peridotite bulk compositions were included in the calibration, which consequently covers pressures from 6 to 20 GPa and temperatures from 900 to 2100 °C. Experimental pressures are reproduced over these conditions with a standard deviation of 0.86 GPa.

The barometer is used to determine equilibration pressures of approximately 500 reported garnet inclusions in diamonds from a range of localities. As the majority of these inclusions are proposed to be syngenetic this allows a detailed picture of diamond formation depths and associated source rocks to be established using inclusion chemistry. Geographic differences in diamond source rocks are mapped within the sub-lithospheric mantle to over 500 km depth. Continuous diamond formation occurs over this depth range within lithologies with eclogitic affinities but also in lithologies that appear transitional between eclogitic and peridotitic bulk compositions, with an affinity to pyroxenites. The geographic differences between eclogitic and pyroxenitic diamond source rocks are rationalised in terms of diamond formation within downwelling and upwelling regimes respectively. Macroscopic diamond formation in rocks with pyroxenite compositions are likely facilitated in the deep mantle by higher average oxidation states and low mineral H<sub>2</sub>O solubility compared to the surrounding mantle, which aid the mobility of C–O–H volatile species. The apparent lack of inclusions with a peridotite affinity may result from generally low oxygen fugacities in such lithologies, which reduces carbon mobility, and the lack of a suitable oxidising agent to allow diamonds to form from CH<sub>4</sub>. This glimpse of deep carbon cycle processes implies that heterogeneities in the carbon content, redox state and chemical composition of the mantle may be strongly coupled.

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## 1. Introduction

The existence of diamond hosted garnet inclusions that are silica-rich due to the presence of a majorite component, provides one of the only robust indicators that some diamonds have a sub-lithospheric origin (Moore and Gurney, 1985; Stachel et al., 2005).

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Although other mineral inclusions may have formed under such conditions (Harte, 2010; Stachel et al., 2005; Thomson et al., 2014), the high-pressure crystal structures that would provide unequivocal evidence for such origins are only very rarely preserved (Pearson et al., 2014). Measurable proportions of both the M<sub>3</sub>(MSi)Si<sub>3</sub>O<sub>12</sub> majorite component (*Maj*), where M is a divalent cation, and the Na<sub>2</sub>M(Si<sub>2</sub>)Si<sub>3</sub>O<sub>12</sub> sodium-majorite (*Na-Maj*) component, however, can only exist in garnets that equilibrated at depths > 150 km, as a result of the breakdown of coexisting ortho- and clinopyroxene (Akaogi and Akimoto, 1977; Irifune et al., 1986; Ringwood and Major, 1971; Sobolev and Lavrent'ev, 1971).

The dominance of different majorite components in natural samples provides evidence for the existence of different rock types in the mantle (Grütter et al., 2004; Sobolev et al., 1973). Garnets exhibiting the *Na-Maj* substitution are generally also higher in Ca and Fe and poor in Cr, consistent with formation within eclogitic rocks, while those formed within peridotite rocks, contain more Cr, less Ca, Fe and Na and exhibit the *Maj* substitution. Some majoritic garnet inclusions with low Cr, Ca and Na appear to have formed from pyroxenitic rocks (Kiseeva et al., 2013) similar to those found in Alpine peridotites (Suen and Frey, 1987) or at least from rocks that appear to be chemically intermediate between eclogitic and peridotitic rocks. Garnets inclusions can, consequently, provide by far the best window into the processes of the deep mantle because they represent a significant proportion of sub-lithospheric inclusions, formed in different rock types while recording chemical evidence of those lithologies and can allow diamond formation depths to be unambiguously determined over at least 300 km, given a sufficiently detailed calibration (Haggerty and Sautter, 1990). Furthermore, some majoritic garnet inclusions have clearly undergone retrograde reactions on decompression, causing exsolution of, for example, omphacitic clinopyroxene (Harte and Cayzer, 2007) and rutile (Zhang et al., 2003). In this case, the remaining majoritic garnet composition should characterise the depth of last equilibration, while the bulk composition of the now multiphase assemblage should characterise the original composition and formation depth of the garnet inclusion, so long as evidence exists that the inclusion was originally a single crystal. If the final and initial depth of this inclusion can be estimated, then together with mineral diffusion coefficients, this information can provide vital constraints on diamond transport rates within Earth's mantle (Harte and Cayzer, 2007; Nishi et al., 2010).

We have performed high pressure and high temperature experiments to calibrate the composition of majoritic garnet as a function of pressure and bulk rock composition, to determine minimum formation depths of garnet inclusions in sub-lithospheric diamonds. A previous similar calibration (Collerson et al., 2010) of the majoritic garnet barometer used experimental data dominated by peridotitic bulk compositions. In preliminary experiments we noticed that the formation pressures of *Na-Maj* bearing garnets were poorly predicted by this barometer, reflecting a bias in the calibrated compositional range. To address this, we performed experiments to calibrate the barometer across a range of eclogitic (basaltic) and pyroxenitic bulk compositions and employed previously published data. In order to make the calibration rigorous and applicable to the widest possible range of chemical bulk compositions, additional high pressure studies conducted in mafic and ultramafic systems were used in the refinement. The resulting barometer coupled with interpretation of the chemical variations in garnet inclusions is used to place constraints on the lithologies within which diamonds form as a function of depth beneath different cratons.

## 2. Methods

Starting compositions were chosen to cover a range of lithologies and to examine specific chemical influences on the majorite substitution mechanisms. Three of the four bulk compositions are basaltic, whereas, composition Mix-1G is pyroxenitic. OC1/2 and NMORB4 are mid-ocean ridge basalts representing an average oceanic crust with the main difference being a raised Ti content in the latter. Composition JB-1 is similar to the Japanese reference alkali basalt described by Terashima et al. (1998). Mix-1G is an average mantle pyroxenite composition based on analyses of worldwide mantle pyroxenites (Hirschmann et al., 2003), and has a higher Mg/Si ratio compared to primitive basalts. Starting ma-

terials were fabricated initially from analytical grade oxides and carbonates that were first dried to remove surface water, then weighed out and ground together under acetone in an agate mortar. Ground mixtures were first calcinated in air at 1000 °C for 3 h and then fused in a Fe saturated platinum crucible at 1600 °C, before quenching to a glass. Recovered glasses were ground and made into pellets that were then reduced in a gas-mixing furnace at 800 °C using a 50:50 CO<sub>2</sub>-H<sub>2</sub> gas mix for 15 h. This rendered an *f*O<sub>2</sub> of approximately 2 log units below the fayalite magnetite quartz (FMQ) oxygen buffer. The initial compositions were made with a deficit of Al<sub>2</sub>O<sub>3</sub> so that H<sub>2</sub>O could be added to all starting compositions as Al(OH)<sub>3</sub> in the final stage of preparation. This was in order to promote crystal growth and to aid the attainment of equilibrium. In sample OC1/2 fluorine was also added as CaF<sub>2</sub>. After additional grinding to homogenise the final mixtures they were dried at 140 °C. Bulk starting compositions are reported in Table 1.

All experiments were conducted using five hole, spark-eroded multi-chamber Au<sub>80</sub>Pd<sub>20</sub> capsules of 2 mm diameter. Sample chambers were 0.3 mm in diameter. In each experiment all four bulk compositions could be loaded into individual chambers along with a further Fe<sub>2</sub>SiO<sub>4</sub>-Mg<sub>2</sub>SiO<sub>4</sub> mixture, employed to check the pressure calibration using the coexisting compositions of olivine and ringwoodite in the products (Frost and Dolejš, 2007). The capsule was closed using two discs of Re-foil that formed a seal under pressure with the body of the capsule. An 18 mm edge length Cr<sub>2</sub>O<sub>3</sub>-doped MgO octahedral pressure media was employed, with tungsten carbide cubes with 11 mm edge length corner truncations. Capsules were placed inside a stepped graphite heater within an MgO sleeve. The temperature was monitored with a D-type thermocouple axially inserted into the octahedron inside a 4-bore Al<sub>2</sub>O<sub>3</sub> tube, with the junction in contact with the top of the multi-chamber capsule. Cavities around the thermocouple were filled with dried MgO powder. Experiments were performed in either 500 (V), 1000 (H) or 5000 t (Z) multi-anvil presses, where letters in brackets are those prefixing the corresponding run numbers in Table 2. Experiments were terminated by shutting down the electrical power to the furnace, causing the samples to quench in less than 2 s to ~100 °C. Run conditions were between 6 and 16 GPa at three different temperatures 1000, 1200, and 1400 °C. The experimental conditions are reported in Table 2.

Recovered capsules were sectioned parallel to the furnace axis using a diamond wire saw, then embedded into epoxy resin and polished for analysis. Samples were analysed using a Jeol JXA-8200 microprobe employing an acceleration voltage of 15 kV and a 15 nA beam current. Analyses were conducted using a focused beam. Counting times were 20 s on the peak and 10 s on the background. Matrix matched mineral standards have been employed for Ca (grossular), Mg (pyrope), Al (pyrope), and Si (pyrope), whereas, Fe, Mn, Ti, and Na were analysed as described in Beyer et al. (2015). A total of 37 garnet analyses (Supplementary Table S3), were collected across the different bulk compositions for use in the barometer calibration.

Several additional bulk compositions, based on analyses of natural majorite-garnet inclusions in diamonds, were fabricated in order to test the accuracy of the barometer calibration. These compositions, 105A1 (Buffalo Hills, Alberta, Canada) and KK81a (Kankan, Guinea, West Africa), were similarly synthesised from analytical grade oxides, carbonates and hydroxides that were glassed and reduced in the same way as described above. The reduced glass powders were loaded into graphite capsules that were welded into Pt capsules fabricated from tubing. These experiments were conducted in a 5000 tonne press using the same multi-anvil assembly as described above except that a stepped LaCrO<sub>3</sub> heater was employed. The inner graphite capsule completely transformed into a nano-diamond aggregate during the experiment, which made it

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