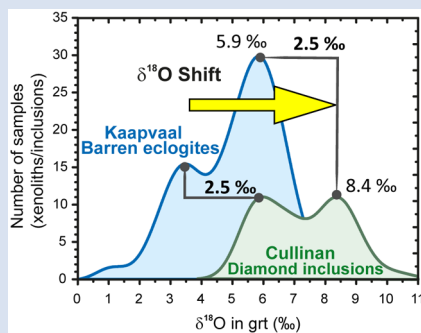# Contrasting oxygen isotopes in garnet from diamondiferous and barren eclogitic parageneses

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Abstract

Eclogite is a minor mantle lithology, present in subducted slivers in cratonic roots. Mantle eclogites carry O and C isotopic signatures from surface organic and inorganic carbon and also are modified by reaction with fluids in the lithosphere. One third of the diamonds mined worldwide are sourced from mantle eclogites, and individual eclogite xenoliths contain up to 20 vol. % diamond. It is critically important to understand where the diamond carbon comes from, and how the diamonds form, for insights on the carbon cycle, diamond exploration, and processes in the lithospheric mantle. Few samples and methods are available to constrain diamond formation in eclogites; in this work we focus on oxygen isotopes in eclogitic garnets. New analyses of garnet/majorite found as inclusions in the Cullinan diamonds reveal a statistically significant systematic difference between $\delta^{18}O$ in garnet associated, and unassociated, with diamond. This contrast persists between garnet from diamondiferous and barren eclogite xenoliths and cannot be due to shielding of diamond inclusions from equilibrating with the common mantle values of $\delta^{18}O$. We propose that diamond-forming metasomatic reactions triggered by carbonatitic fluids may contribute up to 1.5 ‰ to the shift of $\delta^{18}O$ to higher values in eclogitic diamondiferous paragenesis, but cannot fully account for the observed difference of 2.5 ‰.

Introduction

Eclogite, a high grade garnet (Grt)-clinopyroxene (Cpx) rock metamorphosed from the mafic crust, is the most diamondiferous mantle lithology. Diamond concentrations in mantle eclogite can be orders of magnitude higher than the concentration of diamonds in kimberlite – the rock from which they are mined. Over the past 40 years, the oxygen isotope composition ($\delta^{18}O$) of eclogite has become one of the most powerful indicators of its crustal origin in the cratonic mantle (Schulze *et al.*, 2003), together with stable isotopes of C, N and S, and radiogenic isotopes (Pearson *et al.*, 2003; Jacob, 2004). Diamond growth, however, is envisioned as a process overprinting the recycled shallow eclogite protolith. Crustal protoliths for the eclogite do not necessarily imply crustal sources for its diamonds, which could inherit shallow C and O, or could be introduced to the eclogite from mantle fluids. A knowledge of diamond formation in eclogites is critically important to unravel the carbon cycle and deep mantle processes. Diamond formation is considered to be partly metasomatic, as suggested by diamond distribution in eclogites (Taylor and Anand, 2004), $\delta^{13}C$ core-to-rim patterns (Smart *et al.*, 2011) and correlations of O isotopes with trace

element indicators of metasomatism (Gréau *et al.*, 2011; Huang *et al.*, 2012). Diamond precipitates from mantle C-bearing fluids percolating upward and experiencing Raleigh fractionation (Stachel and Luth, 2015; Riches *et al.*, 2016). Possible effects of metasomatic diamond formation on $\delta^{18}O$ of eclogitic minerals may be especially notable for diamondiferous parageneses. Our goal is to quantify these $\delta^{18}O$ to separate out the signatures of shallower crustal alteration from the changes introduced from deeper-seated diamondiferous fluids.

It has been noticed that garnet and clinopyroxene in diamondiferous eclogites are higher in $\delta^{18}O$ than their respective phases in barren eclogites (Pearson *et al.*, 2003). The difference was explained by the origin of garnet in diamondiferous assemblages from the shallow, more altered part of the oceanic crust where $\delta^{18}O$ is higher (McCulloch *et al.*, 1981; Alt *et al.*, 1986; Ickert *et al.*, 2013). This study aims to extend the comparison to eclogitic inclusions in diamonds and make it more statistically robust. In the last 20 years, advances in measurements of O isotopes and new kimberlite discoveries created an abundance of new data. A new summary on $\delta^{18}O$ in garnet from diamondiferous and barren parageneses is long overdue. Here we confirm the distinction between $\delta^{18}O$ of garnet equilibrated

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and unequilibrated with diamond and assess how much of this distinction can be assigned to diamond-friendly metasomatism.

Samples, Methods and Results

We studied diamond inclusions (DIs) from Cullinan Mine (Premier kimberlite) individual raw diamonds. The inclusions are associated with mafic eclogitic and majorite-bearing subli-thospheric parageneses. They are derived from a wide interval of temperatures (*T*) and pressures (*P*) of 5.5–7.5 GPa from the lithosphere and 10.5–13.5 GPa from the sublithospheric mantle (Korolev *et al.*, 2018a). Here we report major element, $\delta^{18}O$, and *P-T* data for 42 non-touching Grt-Cpx pairs and 8 majorites (Supplementary Information; Table S-5). Analysed $\delta^{18}O$ composition of garnet ranges from +5.4 to +10.2 ‰ and covers the oxygen isotope composition of majorites worldwide (+6.0 to +9.4 ‰; Burnham *et al.*, 2015; Ickert *et al.*, 2015).

This new dataset of O isotopes in eclogitic garnet DIs enables statistical comparison with global datasets. The most notable pattern is revealed by a comparison of $\delta^{18}O$ in garnet/majorite associated with diamond (DIs and diamondiferous eclogites) and garnet in barren eclogites. Statistical *t* tests determine that the average $\delta^{18}O$ and its distribution in garnet from diamondiferous eclogites are distinctly higher than the barren eclogites from the Kaapvaal craton with probability >99.99 % (Supplementary Information). The $\delta^{18}O$ compositions of the garnet/majorite inclusions from the Cullinan diamonds are higher than the $\delta^{18}O$ of garnet in Kaapvaal barren eclogites (Fig. 1a). While only 24 % of Cullinan DIs demonstrate $\delta^{18}O < +6$ ‰ and values <+5 ‰ are completely absent (Fig. 1a, Table S-5), 67.4 % of garnets from the barren eclogites have $\delta^{18}O < +6.0$ ‰. Diamondiferous eclogites globally show a narrow $\delta^{18}O$ distribution with a higher mode than the Kaapvaal barren eclogites (Fig. 1b).

Discussion

Several explanations may account for the contrasting $\delta^{18}O$ in barren and diamondiferous eclogitic parageneses. The latter may have formed deeper (>150 km), at higher pressures and temperatures. A suggested positive covariation of $\delta^{18}O_{grt}$ with equilibration temperature for Lace eclogites may hint at a wider T-$\delta^{18}O_{grt}$ correlation in the deep mantle as heavy oxygen may favour garnet with increasing *T* and *P* (Aulbach *et al.*, 2017). To test for this, we compiled data for eclogite xenoliths of the Kaapvaal craton (Fig. S-3a) and worldwide occurrences (Fig. S-4). The absence of $\delta^{18}O$ correlations of garnet DIs with the *P-T* of their formation observed in Cullinan (Fig. S-3) is repeated globally. A comparison of the $\delta^{18}O$ in Grt-Cpx pairs from eclogite xenoliths worldwide equilibrated at the widest range of temperatures (650–1500 °C) shows that there is no dependence between $\delta^{18}O_{grt}$ or $\delta^{18}O_{cpx}$ and temperature (Fig. S-4). The difference between $\delta^{18}O_{grt}$ and $\delta^{18}O_{cpx}$ is constant (±0.87 ‰, 2σ) and does not correlate with temperature (Fig. S-4a). The contrasting $\delta^{18}O$ compositions in barren and diamondiferous parageneses do not relate to pressure, which was predicted by Clayton *et al.* (1975). Only a small proportion of Cullinan Mg-rich DIs demonstrate a local $\delta^{18}O$-*T* correlation (Supplementary Information; Fig. S-2b). Thus, higher pressures and temperatures of diamondiferous eclogites and DIs cannot account for the heavier oxygen in their garnets.

An alternative explanation invokes diffusive buffering of oxygen by the surrounding mantle to explain the $\delta^{18}O$ contrast between garnet in barren (xenoliths) and diamondiferous eclogitic parageneses (DIs and xenoliths). DI garnet is shielded from
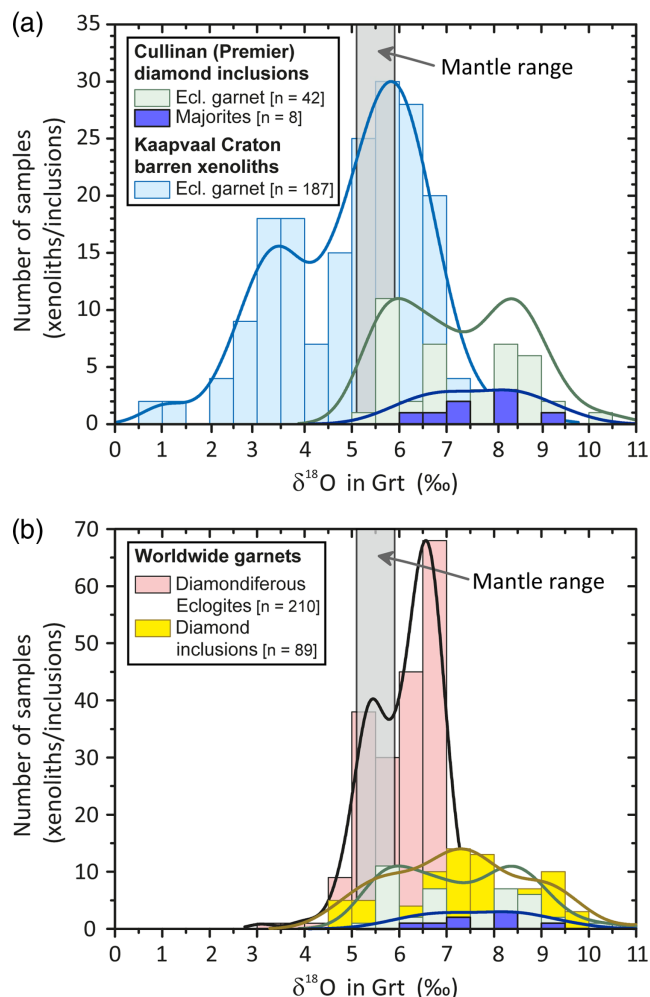

Figure 1 $\delta^{18}O$ histograms for eclogitic garnet in xenoliths and DIs. **(a)** Comparison of our data with Kaapvaal non-diamondiferous eclogites. **(b)** Comparison of global data for garnet/majorite DIs with garnets from diamondiferous eclogites worldwide (references are listed in the Supplementary Information). Lines are kernel-smoothed distribution curves.

re-equilibration with the ambient mantle oxygen ($\delta^{18}O = +5.5 \pm 0.4$ ‰; Mattey *et al.*, 1994), while the "exposed" garnet in xenoliths is not. Only silicate inclusions protected by diamonds retained the ^{18}O-enriched compositions (Schulze *et al.*, 2003; Burnham *et al.*, 2015; Ickert *et al.*, 2015) formed *via* low temperature seawater alteration of the shallowest levels of the former oceanic crust (McCulloch *et al.*, 1981; Alt *et al.*, 1986). These diamonds and their mineral inclusions originated from carbon and oxygen derived from the sedimentary organic matter or altered oceanic crust (Li *et al.*, 2019) subducted into the mantle, as evidenced by a correlation of heavy ^{18}O in silicate DIs and light, low $^{13}C/^{12}C$ carbon (Ickert *et al.*, 2015; Li *et al.*, 2019). The extent of this "diamond shielding" effect can be evaluated by comparing $\delta^{18}O$ histograms for garnet in DIs and diamondiferous eclogites. The $\delta^{18}O$ mode for the DI garnet is between +7 and +8 ‰, 1 ‰ higher than the mode for the exposed garnet in diamondiferous eclogites (Fig. 1b).

One cannot defer to the "diamond shielding" effect to explain the contrast between garnet compositions of diamondiferous and barren xenoliths. The latter show a mode at +5 to +6 ‰, at a lower $\delta^{18}O$ than diamondiferous xenoliths, and an extended "tail" of the distribution towards 0 ‰ (Fig. 1a). A clear difference in $\delta^{18}O$ was shown for both Cpx and Grt for barren and diamondiferous eclogites worldwide (Fig. 2). Traditionally,

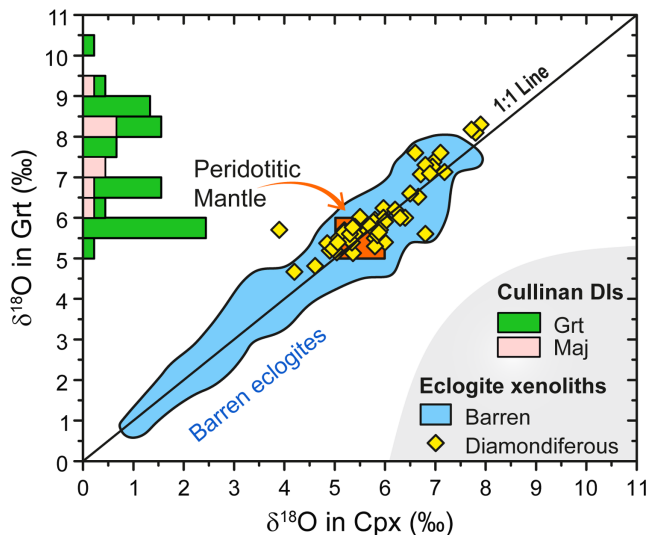**Figure 2** Comparison of $\delta^{18}O$ in eclogitic minerals for barren ($n = 183$) and diamondiferous ($n = 52$) parageneses worldwide (references are given in the Supplementary Information) with a superimposed histogram for $\delta^{18}O$ in the Cullinan DIs (this study).

Figure 3 Comparison of $\delta^{18}O$ of eclogitic garnets/majorites and $\delta^{13}C$ of the host diamond worldwide (ESM1) with $\delta^{18}O$ of Cullinan diamond inclusions. Inclusions with $\delta^{13}C$ for studied Cullinan diamonds (Korolev et al., 2018a) are plotted as symbols, $\delta^{18}O$ of eclogitic garnets with no information on the host diamond $\delta^{13}C$ are shown as the green histogram. The blue hexagon marks the initial magnesite reactant. A blue arrow connects $\delta^{18}O$ of the magnesite reactant with the Grt product for modelled combined metasomatic reactions (Reactions 1 and 8 in Table S-4); it is placed at an average mantle value of -6 ‰ for $\delta^{13}C$. The blue field corresponds to $\delta^{13}C$ in sedimentary carbonates, the yellow field represents mantle carbon, and the pink field is for organic carbon.

this difference would be explained as the contrast in $\delta^{18}O$ of the eclogite protoliths is related to their depth position within the slab and the gradual decrease of $\delta^{18}O$ with depth in the oceanic crust (McCulloch et al., 1981; Alt et al., 1986). In this model, garnet in barren eclogites might have inherited the $\delta^{18}O$ from deep gabbro layers of oceanic crust ($\delta^{18}O = 0$ to $+5$ ‰; Alt et al., 1986). Diamondiferous eclogites with higher $\delta^{18}O$, by contrast, may have recorded a higher input from altered oceanic basalts ($\delta^{18}O = +7$ to $+15$ ‰; McCulloch et al., 1981; Alt et al., 1986; Eiler, 2001; Korolev et al., 2018b).

The second model can explain light C and heavy O isotope compositions of many diamonds and their inclusions, where carbonate in altered mafic-ultramafic oceanic crust with $\delta^{18}O = +11$ to $+33$ ‰, $\delta^{13}C = -30$ to -5 ‰ (Li et al., 2019) and organic C (Fig. 3) contributed to eclogite protoliths. Yet the Cullinan diamonds with eclogitic and sublithospheric majoritic inclusions have the characteristic mantle $\delta^{13}C$ of -2.4 to -4.8 ‰ (Fig. 3) indistinguishable from Cullinan peridotitic diamonds (Korolev et al., 2018a). Thus, the model implying contribution of carbonate in altered mafic-ultramafic oceanic crust cannot be universally applied to all diamonds with inclusions enriched in heavy O, although the model adequately explains compositional patterns in many diamond occurrences.

Another factor that may contribute to contrasting $\delta^{18}O$ in barren and diamondiferous eclogites are diamond-forming metasomatic reactions. Metasomatism plays a central role in diamond formation (Stachel and Harris, 2008), and its effect on stable isotopes of diamondiferous parageneses ought to be quantitatively assessed. It was proposed that the metasomatism may have modified the eclogitic protolith by diffusional equilibration with a carbonatitic fluid (Lowry et al., 1999) or with the mantle carbonatitic fluids containing heavy oxygen ($\delta^{18}O$ of $+5$ to $+10.5$ ‰) (Gréau et al., 2011; Huang et al., 2016). However, any fluid deviating from the mantle O isotopic composition is expected to be short lived, as it would be buffered back to the mantle $\delta^{18}O$ values by re-equilibration with ambient peridotite oxygen isotope reservoirs (Riches et al., 2016).

We tested viable diamond-forming reactions that do not involve heavy oxygen-rich fluids for ^{18}O enrichment effects. Diamond can form by oxidation of methane-rich fluids, by
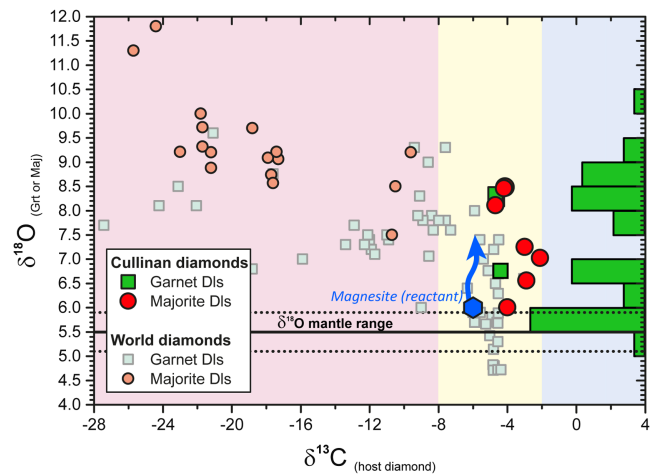
reduction of carbonatitic fluids or by isochemical precipitation from cooling or ascending C-H-O fluids (Stachel and Luth, 2015). The isochemical precipitation would not shift $\delta^{18}O$, while oxidation of methane or other reduced fluids equilibrated with H_2O would lead to metasomatic silicates with lighter oxygen compositions (Ickert et al., 2013). An origin of diamond from an oxidised medium was suggested on the basis of the core-to-rim increases in $\delta^{13}C$ composition of individual diamonds (Smart et al., 2011) and daughter minerals in fluid inclusions in diamonds (Kopylova et al., 2010). We modelled $\delta^{18}O$ effects for metasomatism by oxidising fluids in multiple feasible reactions with the realistic eclogitic mineralogy. The reactions start with the carbonatitic fluid equilibrated with the initial eclogitic garnet ($\delta^{18}O = +6.0$ ‰) and leads to a $\delta^{18}O$ value of resulting garnet elevated by as much as 1.5 ‰ (Fig. 4; Supplementary Information). Diamond-forming metasomatising reactions with the strongest $\delta^{18}O$ shift upward involve 1) production of O_2 or CO_2, 2) heavy oxygen supplied by the metasomatic fluid, 3) a sufficiently high fluid/rock ratio (1–3 moles of fluid to 1 mole of garnet), and 4) oxides (rutile or ilmenite) as products rather than reactants. In Reactions 1 and 2 (Table S-4), diamond forms by disproportionation also creating free O_2, which is immediately used up to make Fe^{3+}-bearing Grt and Cpx (Reaction 8; Table S-4). Reactions 3–7 (Table S-4) facilitate diamond production indirectly, by adding carbon dioxide to C-O-H mantle fluids that may be parental to diamonds (Stachel et al., 2022). The CO_2 concentrations in the mantle, however, are expected to be low, buffered by silicate carbonation (Kopylova et al., 2021). In CO_2 producing reactions the $\delta^{18}O$ of product garnet is elevated by 0.5 to 0.6 ‰ (Table S-4), and the strongest $\delta^{18}O$ upward shift of 1.5 ‰ is observed as a net effect of Reaction 1:

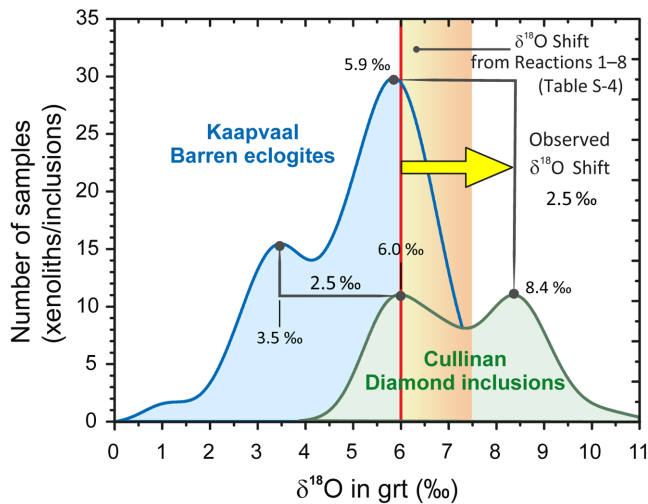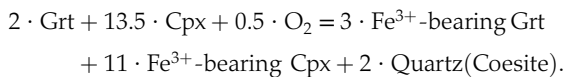$$Almandine + 3 \cdot Magnesite = Pyrope + Magnetite$$
$$+ 3 \cdot C + 2.5 \cdot O_2$$
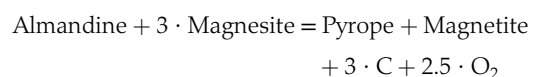
Figure 4 The observed $\delta^{18}O$ contrast between barren and diamondiferous eclogites with superimposed modelled changes of $\delta^{18}O$ composition of eclogitic garnets produced in metasomatic reactions (orange field; Table S-4). The initial $\delta^{18}O$ in eclogitic garnet (vertical line at $+6$ ‰) is chosen arbitrarily (see explanations in the text). A detailed description of the geochemical modelling is provided in the Supplementary Information.

followed by Reaction 8:

$$2 \cdot Grt + 13.5 \cdot Cpx + 0.5 \cdot O_2 = 3 \cdot Fe^{3+}\text{-bearing } Grt$$
$$+ 11 \cdot Fe^{3+}\text{-bearing } Cpx + 2 \cdot Quartz(Coesite).$$

All phases in the proposed reactions are found in cratonic eclogites (*e.g.*, Jacob, 2004), and the latter reaction is based on the observed concentrations of Fe^{3+} in eclogitic minerals (Aulbach *et al.*, 2022). A replacement of eclogitic garnet with a more magnesian garnet has been described in multiple occurrences as part of diamond-friendly metasomatism (*e.g.*, De Stefano *et al.*, 2009; Korolev *et al.*, 2021). An increase of MgO was found to be the most significant chemical change accompanying $\delta^{18}O$ enrichment in garnet from Orapa eclogite xenoliths (Deines *et al.*, 1991). It is well known that metasomatism oxidises the adjacent metasomatised mantle (Creighton *et al.*, 2009). The reactions are equally applicable to majorites in the sublithospheric mantle (Supplementary Information).

We conclude that some metasomatic reactions of diamond formation in eclogites may contribute to the observed $\delta^{18}O$ contrast between barren and diamondiferous eclogitic assemblages worldwide, yet the strongest upward $\delta^{18}O$ shift of all feasible metasomatic reactions (up to 1.5 ‰) achieved in decarbonation followed by metasomatic oxidation is not sufficient to explain the 2.5 ‰ difference in $\delta^{18}O$ (Fig. 4). Inheritance of the O isotopic composition from the crustal eclogitic protoliths is the only model that currently offers a satisfactory explanation for the contrast. This implies preferential diamond formation in eclogites with shallow basaltic protoliths with or without contribution of carbonate in altered mafic-ultramafic oceanic crust that experienced stronger low temperature alteration on the seafloor.

Acknowledgements

Additional Information

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