

Introduction

The volatile abundance, bulk composition, and nature of deep Earth are unresolved geochemical questions (e.g., Hirschmann and Dasgupta, 2009). Diamonds and their mineral inclusions are the only materials available from sublithospheric depths and provide the best geochemical information from these otherwise inaccessible regions (Stachel *et al.*, 2005). Such ultra-deep diamonds have been interpreted as having formed in subducted oceanic crust based on trace element patterns of their inclusions showing negative Eu anomalies and ^{13}C depletion of their diamond hosts (Tappert *et al.*, 2005b). However, this interpretation is non-unique and controversial (Cartigny, 2005; Corgne *et al.*, 2012). To understand Earth's deep carbon cycle it is, however, of key importance to constrain definitively or refute whether diamond acts as a carrier of crustal derived carbon in deeply subducting slabs. To resolve this question, we present the first analysis of oxygen isotope compositions of ultradeep inclusions – majoritic garnets in diamond from Jagersfontein (South Africa).

The carbon isotopic systematics of the mantle – which may contain more carbon than the crust-biosphere system (Hirschmann and Dasgupta, 2009) – are not well understood. Much of the chemical information that we have on deep carbon comes from the study of diamonds and their inclusions (Deines, 1980). From studies of the minerals included in diamonds, it is clear that the vast majority are sourced from the lithospheric mantle. However, there is a small fraction of inclusion assemblages that imply trapping in the asthenosphere, transition zone, or even the lower mantle (Stachel *et al.*, 2005). Much of the difficulty in understanding the nature of the deep carbon cycle revolves around this diamond material. The carbon isotope ratios ($\delta^{13}\text{C}$ values) in diamond have a wide range – from -41‰ to $+3\text{‰}$ (Deines, 1980; Cartigny, 2005; De Stefano *et al.*, 2009) – however, there is controversy over whether the observed variability represents sampling of distinct, unhomogenised, primitive carbon domains, fractionation at mantle conditions, or subduction of carbon that was fractionated by near-surface processes (Cartigny, 2005). Though mainly formulated in the context of cratonic diamonds, these considerations apply equally to the origin of ultradeep diamonds. $\delta^{13}\text{C}$ values for sub-lithospheric diamond have a wide range (from 0‰ to -25‰ ; Stachel *et al.*, 2002; Tappert *et al.*, 2005a; Walter *et al.*, 2011) and have often been interpreted as reflecting incorporation of subducted carbon, thereby documenting deep subduction and the subsequent return of originally crustal carbon. Nevertheless, this interpretation is not unique. For example, Mikhail *et al.* (2014) suggested that iron carbide inclusions in diamonds from Jagersfontein document isotopic fractionation ($\Delta^{13}\text{C}_{\text{carbide-diamond}} > 7\text{‰}$) sufficiently strong to cause the observed ^{13}C depleted signature. Similarly, evidence based on the rare earth element signatures (observation of negative Eu anomalies) of majoritic garnet inclusions from Jagersfontein, interpreted to reflect feldspar fractionation in crustal protoliths (Tappert *et al.*, 2005b), was disputed by Griffin and O'Reilly (2007) and Corgne *et al.* (2012) who considered Eu anomalies to reflect redox-related metasomatic signatures, or crystallisation from a melt at high pressures.

Extreme ^{18}O -enrichment in majorite constrains a crustal origin of transition zone diamonds

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Abstract

The fate of subducted oceanic lithosphere and its role in the planet-scale geochemical cycle is a key problem in solid Earth studies. Asthenospheric and transition zone minerals included in diamond have been interpreted as representing subducted oceanic crust based on inclusion REE patterns and strong ^{13}C depletion of their host diamond ($\delta^{13}\text{C}$ as low as -23‰). This view/explanation, however, has been challenged by alternative interpretations that variable carbon isotopic compositions either result from high temperature fractionation involving carbides, or reflect primordial, unhomogenised mantle reservoirs. Here, we present the first oxygen isotope analyses of inclusions in such ultradeep diamonds – majoritic garnets in diamond from Jagersfontein (South Africa). The oxygen isotope compositions provide unambiguous evidence for derivation of the inclusions from subducted crustal materials. The $\delta^{18}\text{O}_{\text{VSMOW}}$ values of the majorites range from $+8.6\text{‰}$ to $+10.0\text{‰}$, well outside that of ambient mantle ($+5.5 \pm 0.4\text{‰}$) and indicate that the protoliths were very heavily weathered at relatively low temperatures. When this information is combined with the broadly eclogitic composition of the majoritic garnets, a derivation from subducted sea-floor basalts is implied. Based on the association between the heavy oxygen and light carbon, the light carbon isotope composition cannot relate to deep mantle processes and is also ultimately derived from the crust.

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In contrast, oxygen isotope compositions ($\delta^{18}\text{O}_{\text{VSMOW}}$ values) in mantle peridotites, primitive basalts, and the moon have a narrow range, deviating only slightly from +5.5 ‰ (Eiler, 2001). This gross homogeneity is a consequence of an initially well-mixed Earth (Pahlevan and Stevenson, 2007), and small values of mass dependent stable isotope fractionation at high temperatures which prevent mantle silicates from substantially fractionating oxygen isotopes among each other (Urey, 1947; Criss, 1991). Crustal rocks typically have higher $\delta^{18}\text{O}$ values mainly within the range +6 to +12 ‰ (Simon and Lécuyer, 2005) due to low-temperature reactions involving water. In rare cases, crustal rocks can become relatively depleted in ^{18}O , with values below +5.5 ‰ (Taylor, 1968). Thus, variation makes oxygen isotope compositions a uniquely robust tracer of crustal material in the mantle (Eiler *et al.*, 2000; Schulze *et al.*, 2003). This unique feature has previously been leveraged to test the origin of carbon isotope anomalies in lithospheric diamonds. Measuring the oxygen isotope composition of coesite (Schulze *et al.*, 2003) and eclogitic garnet inclusions (Schulze *et al.*, 2013; Ickert *et al.*, 2013) in diamond, a coupled relationship of high $\delta^{18}\text{O}$ values (inclusions) and low $\delta^{13}\text{C}$ values (host diamonds) emerged, constraining a crustal origin of both the diamond carbon and the diamond substrate. For sublithospheric diamonds, similar evidence involving oxygen isotopic analyses of inclusions has so far been lacking.

Rare diamonds from Jagersfontein (South Africa) contain inclusions of majoritic garnet (Deines *et al.*, 1991; Tappert *et al.*, 2005b), an ultra-high pressure mineral reflecting the increasing solubility of pyroxene in the garnet structure at sub-lithospheric depth (Ringwood and Major, 1971). Experimental work (*e.g.*, Akaogi and Akimoto, 1977; Irifune *et al.*, 1986) demonstrated a linear relationship between the solubility of pyroxene (which can be parameterised by Si-excess over the available tetrahedral sites) and pressure, thereby providing crude minimum depth estimates. The studied inclusions from Jagersfontein have Si contents of up to 3.5 atoms per formula unit, indicating that some of the inclusions came from depths of at least 500 km (Tappert *et al.*, 2005b), well within the transition zone (Fig. 1). Based on low Cr contents (<0.4 wt. %) the majoritic garnets are classified as “eclogitic”, or based on their low Na concentrations, metapyroxenitic (Gurney, 1984; Schulze, 2003; Kiseeva *et al.*, 2012). The relatively high molar Mg/(Mg+Fe) (0.68-0.83) of these inclusions preclude derivation from typical pelitic sediments.

Methods

Mineral inclusions in diamonds have generally presented a unique analytical challenge because of their small size. Non-destructive or microbeam techniques are preferred because they consume little or no sample. Although Lowry *et al.* (1999) employed a novel, bulk laser fluorination technique to measure oxygen isotope compositions of mineral inclusions in diamond, most subsequent workers have employed secondary ion mass spectrometry (SIMS) for $\delta^{18}\text{O}$ measurements (Schulze *et al.*, 2003; Smart *et al.*, 2012; Ickert *et al.*, 2013; Schulze *et al.*, 2013).

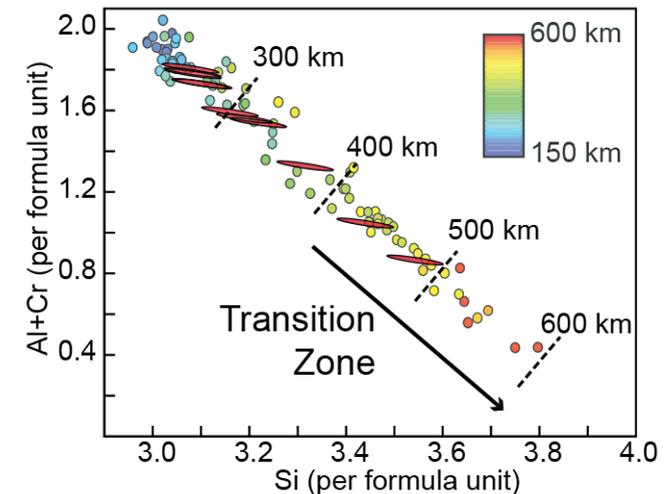


Figure 1 The chemical composition of the Jagersfontein majoritic garnet inclusions based on a formula unit containing twelve oxygens, and their relationship to minimum entrapment depth. The elongate ellipses are the chemical compositions of the garnets and the size and shape of the ellipse represents the 95 % confidence limit based on electron microprobe uncertainties. The small circles are experimental results (from the compilation of Collerson *et al.*, 2010, with the addition of data from Irifune *et al.*, 1986), with colour shading corresponding to the apparent depth that their pressure of equilibration represents. For clarity, uncertainties are not shown on the experimental results; however they are approximately the same size as the uncertainties for the natural garnets. Depth contours are derived from a linear regression of the experimental data relating Si to equilibration pressure.

We made high-precision SIMS $\delta^{18}\text{O}$ measurements on the majorite inclusions in diamond originally studied by Tappert *et al.* (2005b), who published trace element measurements of the majorities and carbon isotope compositions of their host diamonds. Analytical procedures follow Ickert and Stern (2013), with modifications described in the Supplementary Information. As in previous studies (*e.g.*, Schulze *et al.*, 2013), the inclusions, which are <200 μm in their largest dimension, were released by crushing the host diamond, and mounted in epoxy grain mounts along with reference materials. The data are summarised in Table 1, and the full dataset is available in the Supplementary Information Table S-1. Each majorite sample was analysed twice, and in each case the two spots were identical within uncertainty. A major obstacle to accurate SIMS analysis is the dependence of the instrumental mass fractionation on the chemical composition and structure of the target material, the so called “matrix effect” (Russell *et al.*, 1980; Eiler *et al.*, 1997; Vielzeuf *et al.*, 2005; Page *et al.*, 2010). We employ the technique described by Page *et al.* (2010) to correct for the bias between the primary reference material and the majorite samples based on Ca-content. However, we have no reference material for which we can correct for the majorite component. Since we observe



a uniformly heavy oxygen isotope composition at a wide range of abundances of majorite component (including nearly no majorite), we infer that the matrix effect associated with the majorite substitution is small and is not responsible for the heavy oxygen compositions. The range in majorite compositions mainly reflects the variable trapping depth, and therefore the solubility of pyroxene in the garnet. The total uncertainty for each spot analysis is approximately ± 0.4 ‰ (2σ), which is mainly due to overdispersion of the primary reference material (± 0.3 ‰) and the matrix correction (± 0.2 ‰), but includes the uncertainty in the primary reference material and the uncertainty in the session-level mass fractionation correction (see Ickert and Stern, 2013 for details).

Table 1 Oxygen and carbon isotope compositions of majoritic inclusions and their diamond hosts, respectively. The carbon isotope compositions are from Tappert *et al.* (2005b). Uncertainties on $\delta^{18}\text{O}$ values include repeatability of the reference material (RM), the calibration uncertainty, and the uncertainty in the bulk values of the RM. Note that diamonds JF-01 and JF-37 have two inclusions each.

Sample	Spot ID	$\delta^{18}\text{O}_{\text{VSMOW}}$	Mean	$\pm 95\%$ c.l.	$\delta^{13}\text{C}_{\text{VPDB}}$
JF-01A	S1318_1	8.78	8.88	0.37	-21.2
	S1318_2	8.97			
JF-01B	S1319_1	9.07	9.20	0.37	-21.2
	S1319_2	9.32			
JF-09A	S1320_1	9.94	10.00	0.37	-21.8
	S1320_2	10.06			
JF-22A	S1321_1	9.02	9.06	0.37	-17.3
	S1321_2	9.09			
JF-37A	S1322_1	9.28	9.32	0.37	-21.7
	S1322_2	9.36			
JF-37B	S1323_1	9.71	9.72	0.37	-21.7
	S1323_2	9.73			
JF-39A	S1324_1	9.19	9.09	0.37	-17.9
	S1324_2	8.98			
JF-42A	S1325_1	9.26	9.21	0.37	-17.4
	S1325_2	9.15			
JF-44B	S1326_1	8.62	8.74	0.38	-17.7
	S1326_2	8.85			
JF-55A	S1328_1	8.62	8.57	0.38	-17.6
	S1328_2	8.52			
JF-58B	S1331_1	9.14	9.21	0.38	-23.0
	S1331_2	9.28			

Discussion and Conclusions

The measured $\delta^{18}\text{O}$ values for the inclusions fall into a small range from $+8.6$ ‰ to $+10.0$ ‰ (Fig. 2). These values far exceed the range of upper mantle peridotite and derivative melts ($+5.5 \pm 0.4$ ‰; Matthey *et al.*, 1994); in the crust, values this high (in silicate rocks) are only associated with strong interactions between rocks and water at low temperature (Taylor and Sheppard, 1986). High temperature processes, such as CO_2 escape from carbon-rich melts, can fractionate both carbon and oxygen isotope ratios (Deines, 1980; Ickert *et al.*, 2013), however this will only occur under unusual circumstances, and the magnitudes of the fractionations are relatively small and cannot account for >4 ‰ shifts. Moreover, if the carbon isotope data are also considered, there are no processes that can simultaneously create depletion in ^{13}C and enrichment in ^{18}O (Ickert *et al.*, 2013). Cartigny (2010) suggested that the depleted carbon isotope signatures of the Jagersfontein transition zone-suite of diamonds were derived from a primordial mantle reservoir and not from crustal materials. The presence of a previously undetected high- $\delta^{18}\text{O}$, primordial reservoir in the deep Earth is unlikely, given the homogeneity of the mantle and the near identical oxygen isotope systematics between the Earth and Moon (Wiechert *et al.*, 2001), so we suggest that this hypothesis is now untenable.

These new data establish a link between the high $\delta^{18}\text{O}$ values of inclusions and the low ^{13}C values of the diamond hosts. This relationship is firm evidence for a subducted protolith and makes it extremely unlikely that the associated negative $\delta^{13}\text{C}$ values are related to deep mantle isotopic fractionation (Mikhail *et al.*, 2014). In a subduction scenario, the low $\delta^{13}\text{C}$ values (Fig. 2) imply that the carbon originated not as carbonate but as organic carbon – either residual from biological processes or produced abiotically by Fischer-Tropsch-type reactions. The $\delta^{18}\text{O}$ values require that the host rocks were strongly weathered, and probably underwent substantial secondary hydration on the sea floor.

This $\delta^{18}\text{O}$ - $\delta^{13}\text{C}$ relationship implies that the recovered ultradeep inclusions were derived from intensely hydrated (*e.g.*, $\delta^{18}\text{O} \gg +7$ ‰) and typically oxidised basaltic rock close to the seawater interface, and that the diamond carbon was closely associated with this protolith. Such rocks have a low relative abundance in the oceanic crust, and the coincidence of their rarity and their apparent association with diamonds most likely attests to a causal relationship. That reduced, rather than oxidised, carbon is involved suggests that after thermal cracking, the carbon is subducted as metastable graphite. Since conversion of metastable graphite to diamond is not expected to produce macrocrystals (Sung, 2000), diamond formation during a fluid/melt aided dissolution – precipitation process is likely. The volatile-rich nature of the protolith is consistent with the recent suggestion by Harte (2010) that ultradeep diamonds are associated with devolatilisation and possibly melting reactions.



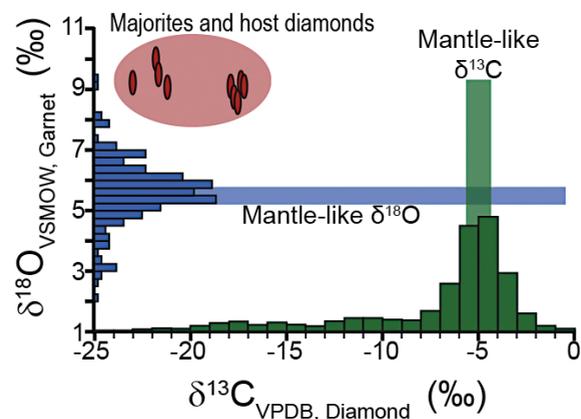


Figure 2 The $\delta^{18}\text{O}$ compositions of the majoritic garnets and the $\delta^{13}\text{C}$ values of their host diamonds. The majoritic garnets and their diamond hosts plot well away from the mantle field. The histogram on the Y-axis is of garnets from eclogite xenoliths (compilation of Ickert *et al.*, 2013), the histogram on the X-axis is of cratonic diamonds (Stachel *et al.*, 2009).

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Additional Information

Supplementary Information accompanies this letter at www.geochemicalperspectivesletters.org/article1507

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