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Stable isotope evidence for crustal recycling as recorded by superdeep diamonds



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ABSTRACT

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Keywords: diamonds stable isotopes subduction majorite stishovite Sub-lithospheric diamonds from the Juina-5 and Collier-4 kimberlites and the Machado River alluvial deposit in Brazil have carbon isotopic compositions that co-vary with the oxygen isotopic compositions of their inclusions, which implies that they formed by a mixing process. The proposed model for this mixing process, based on interaction of slab-derived carbonate melt with reduced (carbide- or metal-bearing) ambient mantle, explains these isotopic observations. It is also consistent with the observed trace element chemistries of diamond inclusions from these localities and with the experimental phase relations of carbonated subducted crust. The ¹⁸O-enriched nature of the inclusions demonstrates that they incorporate material from crustal protoliths that previously interacted with seawater, thus confirming the subduction-related origin of superdeep diamonds. These samples also provide direct evidence of an isotopically anomalous reservoir in the deep (\geq 350 km) mantle.

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1. Introduction

Diamonds, the deepest-derived mineral grains available for scientific study, have long been used to provide insights into the nature of the mantle and chemical processes in the deep Earth. A key indicator of diamond provenance is the mineralogy and composition of trapped inclusions, the majority of which indicate diamond formation in the lithospheric mantle; a small proportion (<5%) of diamonds formed at much higher pressures, deep in the asthenospheric upper mantle or even the lower mantle (Stachel et al., 2005; Walter et al., 2011; Kaminsky, 2012).

Subduction of oceanic lithosphere returns material (sea-floor sediments, altered lavas and depleted mantle rocks) from the Earth's surface to the mantle. The fate of the various subducted lithologies remains enigmatic, with questions still unanswered regarding the extent to which these materials might be decoupled, how deep material descends into the mantle, and the extent to which subducted material is isolated from the convecting mantle. For example, subducted crust may be incorporated into the sub-cratonic lithosphere as demonstrated by mantle xenoliths and diamonds transported to the surface by kimberlite eruptions worldwide (Jacob, 2004), or it may mix back into the mantle as suggested by seismic tomography showing slabs stalled at the 660 km discontinuity and even penetrating into the lower mantle (Widiyantoro et al., 1999).

Oxygen isotopes are a powerful tracer of crustal material, only being fractionated significantly at low temperatures (Bindeman, 2008). Hence, any value outside a narrow "peridotitic mantle" range, $\delta^{18}O_{SMOW} = 5.7 \pm 0.2\%$, is diagnostic of a shallow crustal origin. For example, δ^{18} O values lie in the range 11–21‰ for mudrocks (Land and Lynch, 1996), 20-35% for marine limestones (Land and Lynch, 1996) and 3-15% for altered basaltic lavas (Gregory and Taylor, 1981). Studies of the oxygen isotopic compositions of silicates (Lowry et al., 1999; Schulze et al., 2004, 2013; Ickert et al., 2013) and the sulphur isotopic ratios of sulphides (Farquhar et al., 2002) within lithospheric diamonds testify to the subducted origin of some mineral inclusions in lithospheric diamonds. The isotopic composition of C in diamond similarly has a narrow isotopic range within typical peridotitic mantle, with $\delta^{13}C_{PDB}$ around $-5 \pm 1\%$ (Ickert et al., 2013). Lithospheric diamonds hosting eclogitic silicate inclusions often show a covariation between the δ^{13} C of the former and the δ^{18} O of the latter, as shown in Fig. 1 (Lowry et al., 1999; Ickert et al., 2013; Schulze et al., 2013). Such examples indicate that not all subducted material is recycled back into the convecting mantle, but can instead be incorporated into or accreted onto the lithospheric mantle. Those rare "superdeep" diamonds that originate in the

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Fig. 1. Previously reported co-variation of δ^{13} C and δ^{18} O for diamonds and their inclusions worldwide: majoritic (superdeep) garnets from Jagersfontein, South Africa (lckert et al., 2015), and lithospheric minerals (coesite and garnet) from Argyle, Australia (Schulze et al., 2013), Damtshaa, Botswana (lckert et al., 2013), Finsch, South Africa (Lowry et al., 1999) and Guaniamo, Venezuela (Schulze et al., 2013).

convecting mantle beneath the lithosphere provide a direct insight into the mineralogy and geochemistry of the deep mantle. Ickert et al. (2015) reported the O isotopic compositions of majorite inclusions in diamonds from South Africa (the Jagersfontein kimberlite). As with lithospheric eclogitic inclusions from other localities worldwide, these are enriched in δ^{18} O (Fig. 1), indicative of the crustal nature of the protolith.

Here we report the relationship between δ^{13} C and δ^{18} O for superdeep diamonds from Brazil (the Juina-5 and Collier-4 kimberlites; Bulanova et al., 2010; Walter et al., 2011; Thomson et al., 2014 and the Machado River alluvial deposit; Bulanova et al., 2008). We use these data to test the hypothesis that many superdeep diamonds are formed from recycled material. Additionally we assess the extent to which isotopic signatures of crustal materials are maintained throughout their passage into the deep mantle, rather than being removed and re-homogenised during the subduction process that includes dehydration and sediment melting. Although the suites of diamonds studied contain a wide variety of deep upper mantle minerals, the calibration of δ^{18} O by ion probe secondary ion mass spectroscopy (SIMS) varies considerably and unpredictably between minerals. Because of this limitation, only coesite, CaSiO₃-walstromite and garnet were analysed.

2. Methods

Inclusion-bearing diamonds were polished to expose the inclusions at the surface, mounted in indium and gold-coated. Mineral structures were verified by Raman spectroscopy using a Thermo Scientific DXRxi microscope equipped with a 455 or 532 nm excitation laser. Panchromatic cathodoluminescence (CL) images were recorded using a Hitachi S-3500N scanning electron microscope. Backscattered electron imaging and electron probe microanalysis of the major element composition were performed using a JEOL JXA8530F Hyperprobe operating at 20 kV and 3 nA (imaging) or 20 nA (compositional analysis by wavelength dispersive spectroscopy).

SIMS analysis of carbon and oxygen isotopic ratios was carried out at the Edinburgh Ion Microprobe Facility (EIMF) using a Cameca 1270 mass spectrometer operating at 10 kV and 6 nA primary Cs⁺ beam current. Electron flooding minimised charge build-up during the measurement of O isotope ratios. In-house standards were used for calibration and drift correction. The standard for diamond was synthetic diamond SYN ($\delta^{13}C = -23.9\%$), that used for coesite was Bogala quartz ($\delta^{18}O = 12.3\%$; there

is no instrumental mass fractionation (IMF) difference between guartz and coesite, Schulze et al., 2003) and for CaSiO₃ a synthetic Ca-walstromite was prepared at 3.1 GPa and 1580 °C in a piston cylinder apparatus, and characterised for δ^{18} O by laser fluorination at SUERC as $\delta^{18}O = 21.0 \pm 0.4\%$ (1 σ , n = 4). For garnet a set of 10 standards of known δ^{18} O were analysed to provide a working curve for the instrumental mass fractionation as a function of composition, with garnet 13-63-44 (Page et al., 2010) analysed throughout the session to monitor instrument drift. δ^{18} O was parameterised as a quadratic function of the Ca content of the garnet (Page et al., 2010). For the first analytical session this gave $\delta^{18}O_{\text{sample}} = \delta^{18}O_{\text{drift corrected}} + 0.652 \cdot (X_{\text{Ca}})^2 - 2.98 \cdot (X_{\text{Ca}}) + 1.34,$ which had a mean deviation from the observed IMF of 0.23‰. The larger garnet in ColN-18 was analysed the following day, on which occasion the IMF was $\sim 0.29\%$ smaller. However, the relative IMFs of the garnets studied were in excellent agreement with previous measurements made at EIMF using similar instrumental setup and differed only in the constant in the IMF equation, and hence this analysis was corrected using $\delta^{18}O_{sample} = \delta^{18}O_{drift corrected} + 0.652$. $(X_{Ca})^2 - 2.98 \cdot (X_{Ca}) + 1.05$. The standards available contained negligible Na and Ti which, respectively, are present at up to 0.19 and 0.09 atoms per formula unit (12 oxygens) in the diamond inclusions. Treating the Na component as identical to Ca for the diamond inclusions would result in $\delta^{18}O_{\text{sample}}$ values that were lower by at most 0.39% (mean = 0.19%) and therefore the neglect of this component is probably not significant. The Ti component of the garnet is lower than that of Na. and was ignored. Overall, we estimate the uncertainty of our garnet measurements as the combination of the spot-to-spot reproducibility, 0.15% (an average of 0.18% for the drift-affected morning session (n = 25) and 0.13% for the more stable afternoon session (n = 20)), and the mean deviation of the IMF-corrected measurements from the accepted values for those materials, 0.23‰. Therefore we consider the 1σ uncertainty for the garnet δ^{18} O measurements to be 0.28‰. For coesite the spot-to-spot 1σ uncertainty was 0.14%. For CaSiO₃ the uncertainty is dominated by the apparent inhomogeneity of the standard. This was tested in two sessions. During the first the spot-to spot reproducibility was 0.63% (n = 40) and during the second it was 0.57% (n = 20); excluding two markedly different values from the first session and one from the second improves the spot-to spot reproducibility to 0.44% and 0.31% respectively. These extreme outliers are probably related to the presence of decompression cracks within the synthetic material. The uncertainty on the laser fluorination value for the synthetic Ca-walstromite is \sim 0.37‰; combining this with the average reproducibility on the standard (0.38%) gives an overall uncertainty of 0.53%.

CL images were obtained after analysis to determine the internal structures of the samples and to verify the location of analytical points within the growth zones of the diamonds.

3. Results

The diamonds and inclusions studied are summarised in Table 1. EPMA data for the garnets are given in Table 2. The coesite inclusions are almost pure SiO₂ with trace amounts (<0.06 wt%) of Al₂O₃ and TiO₂; the Ca-walstromite is almost pure endmember CaSiO₃ with other components comprising <0.8% by weight (Table 3). Coesite with exsolved kyanite (Fig. 2a) probably formed as stishovite, which can accommodate much higher Al concentrations than other SiO₂ polymorphs (Liu et al., 2007), and must have formed at pressures above ~9 GPa (Zhang et al., 1993), equivalent to a minimum crystallisation depth of 270 km. The garnets have eclogitic compositions with a majoritic component and often have a rim of exsolved clinopyroxene (Fig. 2b). Several Ca-walstromite grains contain a small proportion (<10%) of exsolved CaTiO₃ grains (Fig. 2c), implying formation as Ca(Si,Ti)O₃ perovskite at pressures

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