

## Characterisation of sublithospheric and lithospheric diamond populations from the Candle Lake C29/30 kimberlite, Sask Craton, Canada

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### Introduction

The small Sask Craton, wedged within the Trans Hudson orogen between the much larger Superior and Hearn cratons, hosts one of the widest kimberlite fields in the world, the Fort à la Corne (FALC) kimberlite field (Harvey et al. 2001). The Candle Lake kimberlites are thought to be a satellite cluster of the major FALC field. Drill core sampling of the Candle Lake C29/30 kimberlite resulted in the recovery of ~3000 diamonds. A subsample of 674 diamonds, consisting of 280 microdiamonds (0.1-0.5 mm) and 394 macrodiamonds (0.5-6.8 mm), was classified for dimension, colour, shape, surface characteristics and presence of internal imperfections. Fourier Transform Infrared (FTIR) spectroscopy was employed to study nitrogen content and aggregation of 96 randomly selected macrodiamonds. Carbon and nitrogen isotope compositions were acquired on 35 micro- and 24 macrodiamonds through 199 Secondary Ion Mass Spectrometry (SIMS) spots. Mineral inclusions were identified and characterized *in situ* by Raman spectroscopy and Single Crystal X-ray Diffraction (SC-XRD) and a subset were extracted or exposed by polishing, for subsequent Electron Probe Microanalyzer (EPMA) analyses.

### Results

The Candle Lake diamond suite comprises both un-resorbed and highly resorbed crystals, with a high abundance of octahedral aggregates. Micro- and macro-diamonds differ in shape and degree of resorption; octahedral crystals are more frequent in the small size fraction, whereas the larger crystals are more often highly resorbed and internally pure. The macrodiamonds studied by FTIR are mostly of Type IaA to IaB and nitrogen rich (average of 805 at. ppm). Type IIa diamonds (which do not show appreciable N signal from FTIR) constitute ~14% of the sample. Absorption bands assigned to CO<sub>2</sub> were identified in the core of some diamonds. The diamond population is complex and comprises different subgroups. Assuming a Mesoproterozoic formation age (~1.2 Ga; Milne et al. this volume), the nitrogen characteristics of the diamonds yield mantle residence temperatures of 1050-1250 °C, with a main peak at ~1120 °C, consistent with a lithospheric origin.

The C and N isotope composition of the diamonds is highly variable across the suite and suggests the presence of both peridotitic and eclogitic parageneses. Carbon isotope compositions ( $\delta^{13}\text{C}$ ) vary between -25.3‰ and -0.9‰ and are not correlated with nitrogen content, whereas nitrogen isotope compositions ( $\delta^{15}\text{N}$ ) vary from -7.2‰ to +11.8‰, approaching 0‰ at high nitrogen contents.

The inclusion mineralogy of the Candle Lake diamond suite indicates carbonated peridotite and eclogite as growth substrates. Identified inclusions are olivine, garnet, pyrrhotite, enstatite, diopside, omphacite, coesite, rutile, kyanite, ferropericlaase, breyite, magnesite and a K-Ca carbonate. All the olivine inclusions occur in Type IIa diamonds. The four analyzed olivines have Mg#s of 92-93 and Cr<sub>2</sub>O<sub>3</sub> (0.05-0.08 wt.%) and CaO contents (0.05-0.07 wt.%) typical of lithospheric olivine inclusions (Stachel et al. 2022). A lithospheric origin is also indicated by relatively small residual stresses of the inclusion-diamond pairs recorded by SC-XRD analyses. Olivine coexists with ferropericlaase in one diamond.

Ferropericlaase inclusions (n=4) have Mg# 87-87.5 and NiO contents ~1.17 wt.%, consistent with ferropericlaase inclusions of lower mantle origin (Walter et al. 2022). Further evidence for a lower mantle derivation comes from coexistence of ferropericlaase with enstatite in two diamonds. The one enstatite inclusion analyzed by EPMA has Mg# 96, low Al<sub>2</sub>O<sub>3</sub> (0.28 wt.%) and low NiO (0.02 wt.%), coherent with an origin as retrogressed lower mantle bridgmanite. Multiple breyite (CaSiO<sub>3</sub>) inclusions were identified in one diamond. Breyite is often considered a retrogressed product of lower mantle Ca-perovskite (Walter, et al. 2022). One breyite was exposed and analyzed by EPMA, revealing it as a nearly pure phase with low MgO (0.38 wt.%) and TiO<sub>2</sub> (0.05 wt.%) content. Ferropericlaase, enstatite and breyite have chemical compositions consistent with a sublithospheric derivation in metaperidotite hosts. Based on the residual pressure determined at room temperature via SC-XRD analysis for one ferropericlaase inclusion, we calculated an origin from at least transition zone depth (~420 km depth for a trapping T of 1400 °C); (Angel et al. 2022).

Further barometric information on the origin of the Candle Lake sublithospheric diamonds comes from a majoritic garnet inclusion, which indicates an entrapment pressure of 9.6 ±0.4 GPa using the Thomson et al. (2021) barometer - a minimum estimate due the observed exsolution of clinopyroxene. Two other garnet inclusions analyzed by EPMA lack excess silica and are of lithospheric origin. They are compositionally classified as eclogitic, in agreement with coexisting omphacite and coesite inclusions, and the C isotope composition of the host diamond.

## Discussion

The mineralogy and chemical composition of the inclusions in Candle Lake diamonds reveals a mixture of lithospheric and sublithospheric sources. A sublithospheric origin of a subset of the diamonds is constrained by the coexistence of ferropericlaase with enstatite (retrogressed bridgmanite, two diamonds) and the identification of breyite and majoritic garnet inclusions, all occurring in Type IIa diamonds. The relative proportion of sublithospheric diamonds at Candle Lake is about 25% based on FTIR and inclusion data. This discovery adds a new sublithospheric diamond locality to the small worldwide inventory (see Shirey et al. 2024 for summary of locations).

At Candle Lake, the olivine and the sublithospheric inclusions are found in Type IIa octahedral diamonds, which often form octahedral aggregates. Ferropericlaase is associated with magnesite (in two diamonds) and is interpreted both as lithospheric (coexisting with multiple olivines, in one diamond) and sublithospheric (with retrogressed bridgmanite, in two diamonds). At this time, we have not analyzed the olivine inclusions coexisting with ferropericlaase yet, but low residual pressures of the inclusions determined via SC-XRD are not in support of a superdeep origin. We also exclude a later entrapment event because one of the olivines is in direct contact with ferropericlaase. An alternative explanation may be a common growth mechanism of diamond and ferropericlaase across different depths, for example by decarbonation of magnesite (e.g. Liu 2002). Such a decarbonation mechanism is supported by magnesite inclusions in five diamonds and CO<sub>2</sub>-related absorbance in the core of some diamonds. However, Mg-rich ferropericlaase is currently interpreted as a protogenetic inclusion in sublithospheric diamonds (Lorenzon et al. 2023). We plan to improve our characterisation of the olivine-ferropericlaase assemblage at Candle Lake to better constrain its origin.

A unique occurrence in the Candle Lake diamond inclusion suite is the mineral association of breyite and a K-Ca carbonate [ $K_2Ca(CO_3)_2$ ] interpreted as bütschliite in one Type IIa diamond. The bütschliite inclusion appears to coexist with minor amounts of other phases not yet identified. A similar inclusion was previously discovered in a nitrogen-rich diamond from Yakutia (Logvinova et al. 2019) and explained as the cooling product of a carbonatite melt that was associated with diamond growth at 1300-1400 °C. Our discovery provides further confirmation that carbonatitic melts are related to diamond formation, also at sublithospheric depths.

Finally, the primary shape of some Candle Lake diamonds that host sublithospheric inclusions is unusually well preserved, compared with the typically highly resorbed nature of sublithospheric diamonds from other locations. This observation, if it is typical of sublithospheric diamonds sampled by Sask Craton kimberlites, might assist in finding sublithospheric diamond inclusions at FALC, where their presence has been hinted at by inclusion studies (Banas et al. this volume).

## References

- Angel RJ, Alvaro M, Nestola F (2022) Crystallographic Methods for Non-destructive Characterization of Mineral Inclusions in Diamonds. *Reviews in Mineralogy and Geochemistry* 88(1):257-305 doi:10.2138/rmg.2022.88.05
- Harvey S, Kjarsgaard B, Kelley L (2001) Kimberlites of central Saskatchewan: Compilation and significance of indicator mineral geochemistry with respect to diamond potential. Summary of investigations 2:2001-2004.2002
- Liu L-g (2002) An alternative interpretation of lower mantle mineral associations in diamonds. *Contributions to Mineralogy and Petrology* 144(1):16-21 doi:10.1007/s00410-002-0389-y
- Logvinova AM, Shatskiy A, Wirth R, Tomilenko AA, Ugap'eva SS, Sobolev NV (2019) Carbonatite melt in type Ia gem diamond. *Lithos* 342-343:463-467 doi:10.1016/j.lithos.2019.06.010
- Lorenzon S, Wenz M, Nimis P, Jacobsen SD, Pasqualetto L, Pamato MG, Novella D, Zhang D, Anzolini C, Regier M, Stachel T, Pearson DG, Harris JW, Nestola F (2023) Dual origin of ferropericlasite inclusions within super-deep diamonds. *Earth and Planetary Science Letters* 608 doi:10.1016/j.epsl.2023.118081
- Shirey SB, Pearson DG, Stachel T, Walter MJ (2024) Sublithospheric Diamonds: Plate Tectonics from Earth's Deepest Mantle Samples. *Annual Review of Earth and Planetary Sciences* 52(1) doi:10.1146/annurev-earth-032320-105438
- Stachel T, Aulbach S, Harris JW (2022) Mineral Inclusions in Lithospheric Diamonds. *Reviews in Mineralogy and Geochemistry* 88(1):307-391 doi:10.2138/rmg.2022.88.06
- Thomson AR, Kohn SC, Prabhu A, Walter MJ (2021) Evaluating the Formation Pressure of Diamond-Hosted Majoritic Garnets: A Machine Learning Majorite Barometer. *Journal of Geophysical Research: Solid Earth* 126(3) doi:10.1029/2020jb020604
- Walter MJ, Thomson AR, Smith EM (2022) Geochemistry of Silicate and Oxide Inclusions in Sublithospheric Diamonds. *Reviews in Mineralogy and Geochemistry* 88(1):393-450 doi:10.2138/rmg.2022.88.07