



## Mixed fluid sources involved in diamond growth constrained by Sr–Nd–Pb–C–N isotopes and trace elements

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### ABSTRACT

Sub-micrometer inclusions in diamonds carry high-density fluids (HDF) from which the host diamonds have precipitated. The chemistry of these fluids is our best opportunity of characterizing the diamond-forming environment. The trace element patterns of diamond fluids vary within a limited range and are similar to those of carbonatitic/kimberlitic melts that originate from beneath the lithospheric mantle. A convecting mantle origin for the fluid is also implied by C isotopic compositions and by a preliminary Sr isotopic study (Akagi, T., Masuda, A., 1988. Isotopic and elemental evidence for a relationship between kimberlite and Zaire cubic diamonds. *Nature* 336, 665–667.). Nevertheless, the major element chemistry of HDFs is very different from that of kimberlites and carbonatites, varying widely and being characterized by extreme K enrichment (up to ~39 wt.% on a water and carbonate free basis) and high volatile contents. The broad spectrum of major element compositions in diamond-forming fluids has been related to fluid–rock interaction and to immiscibility processes.

Elemental signatures can be easily modified by a variety of mantle processes whereas radiogenic isotopes give a clear fingerprint of the time-integrated evolution of the fluid source region. Here we present the results of the first multi radiogenic-isotope (Sr, Nd, Pb) and trace element study on fluid-rich diamonds, implemented using a newly developed off-line laser sampling technique. The data are combined with N and C isotope analysis of the diamond matrix to better understand the possible sources of fluid involved in the formation of these diamonds. Sr isotope ratios vary significantly within single diamonds. The highly varied but unsupported Sr isotope ratios cannot be explained by immiscibility processes or fluid–mineral elemental fractionations occurring at the time of diamond growth. Our results demonstrate the clear involvement of a mixed fluid, with one component originating from ancient incompatible element-enriched parts of the lithospheric mantle while the trigger for releasing this fluid source was probably carbonatitic/kimberlitic melts derived from greater depths. We suggest that phlogopite mica was an integral part of the enriched lithospheric fluid source and that breakdown of this mica releases K and radiogenic Sr into a fluid phase. The resulting fluids operate as a major metasomatic agent in the sub-continental lithospheric mantle as reflected by the isotopic composition and trace element patterns of G10 garnets.

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### 1. Introduction

While gem diamond is compositionally a simple mineral (pure carbon), this “purity” creates significant challenges in unveiling the story of diamond-formation. Some fibrous diamonds trap numerous minute inclusions of their parental high-density fluids (HDF) and thus carry chemical information of their origin (Navon et al., 1988). The chemistry of these fluids is our best opportunity of characterizing the fibrous diamond-forming environment.

Growth zones within diamonds that carry abundant microinclusions commonly have fibrous radiating growth habit (Kamiya and Lang, 1965), and are interpreted as reflecting rapid diamond growth (Sunagawa, 1984). Such growth zones may appear as “coats” surrounding octahedral transparent cores or may persist throughout the diamond. Internal growth zones containing abundant microinclusions, known as “clouds” (Harris and Gurney, 1979), may be also found within otherwise inclusion-poor octahedral diamonds.

Trace element studies of microinclusion-rich diamonds have found a limited range of fluid compositions that are observed in all the types of HDFs described above (e.g. Schrauder et al., 1996; Resano et al., 2003; Rege et al., 2005; Tomlinson et al., 2005, 2009; Weiss et al., 2008; Zedgenizov et al., 2007). The observed trace element patterns are similar to those of kimberlites or carbonatites and have led many

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authors to suggest a direct relationship between these mantle-derived melts and diamond-forming fluids (e.g. Schrauder et al., 1996; Tomlinson et al., 2005, 2009; Zedgenizov et al., 2007). This interpretation is reinforced by the similarity between the restricted carbon isotopic composition of the diamond matrix and convecting mantle values (Boyd and Pillinger, 1994). The only radiogenic Sr isotopic study of fibrous diamonds carried out to date revealed relatively low  $^{87}\text{Sr}/^{86}\text{Sr}$  ratios, consistent with convecting mantle Sr isotopic compositions, in five fibrous diamonds (Akagi and Masuda, 1988).

Major element studies of fluid bearing diamonds have revealed two wide compositional arrays; a) an array ranging between a silicic end-member (rich in Si, Al and water) and a low-Mg carbonatitic end-member (rich in Ca and carbonate), and b) an array extending between a saline end-member (rich in K, Cl and water) and a high-Mg carbonatitic end-member (Israeli et al., 2001; Klein-BenDavid et al., 2009; Schrauder and Navon, 1994; Weiss et al., 2009). All diamond-forming HDFs have significantly higher potassium and volatile contents than kimberlites and carbonatites erupted on the Earth's surface (up to 39 wt.% potassium on a water and carbonate free basis and up to 40 wt.% volatiles; Klein-BenDavid et al., 2007, 2009). These observations decouple the varied major element signature of the fluids from the rather conservative trace element patterns, and reveal significant differences from the kimberlitic and carbonatitic melts. The wide major element compositional spectrum of HDFs is a primary indication that several processes and sources may contribute to the composition of the final diamond growth medium. Recently it has been proposed that the observed variation is the outcome of fluid interaction with different host rocks (Weiss et al., 2009). Within such a model, fluid interaction with a peridotitic environment gives rise to fluids along the saline high-Mg carbonatitic trajectory whereas reaction with eclogite yields fluids between the silicic and low-Mg carbonatitic end-members. Such a model does not, however, further constrain the origin of the fluid.

Both major and trace element signatures can be modified by a variety of mantle processes whereas radiogenic isotopes give a clear fingerprint of the time-integrated evolution of the fluid source region. Here we present the results of the first multi-isotopic (Sr, Nd, Pb, C and N) and trace element study of fluid-rich diamonds. Trace elements and radiogenic isotope analyses were conducted using a newly developed off-line laser sampling technique that allows the determination of both elemental and radiogenic isotope data. Our results demonstrate the clear involvement of a mixed fluid with at least two distinct sources. We propose that one component originates from ancient incompatible element-enriched parts of the lithospheric mantle while the trigger for releasing this fluid source was probably carbonatitic/kimberlitic melts derived from greater depths.

### 1.1. Relationship between fibrous, fluid-rich diamonds and octahedral diamonds

Fibrous diamonds represent a small percentage of the total diamond production with different habit and growth rates (Kamiya and Lang, 1965; Sunagawa, 1984), thus, the implications of fibrous diamond-forming fluids for the general diamond growth environment have been questioned. Recent publications have shown that the trace element content of some gem diamonds share many similarities with those of fibrous diamonds (McNeill et al., 2009; Araujo et al., 2009). LREEs are generally enriched relative to HREEs, yielding steep slopes on REE diagrams. Many gem diamonds show depletion in HFSE and Sr and relative enrichment in Th and U (McNeill et al., 2009). These features are in accordance with those of fibrous diamond-forming fluids. In addition, Tomlinson et al. (2009) have found that the trace element signatures within silicate inclusions trapped within fibrous diamonds and those trapped within octahedral diamonds are very similar and may be imposed by the same fluid composition. Thus although different in habit, the composition and evolution of fibrous diamond-forming fluids may shed a light on the growth of gem diamonds.

## 2. Methods

We analyzed six diamonds from Botswana. Five of the diamonds consisted of fibrous growth pattern throughout and one diamond contained a transparent core surrounded by a fibrous coat. All diamonds were laser cut into three parallel sections, and at least one slab of each diamond was doubly polished into 0.5–1.5 mm thick slabs with two parallel faces.

### 2.1. Trace elements and radiogenic isotope chemical procedures and blanks

Sample preparation: diamond slabs were leached for two hours in 29 N ultra purity acid (UPA) HF and 16 N UPA  $\text{HNO}_3$  on a hotplate at 100 °C (acids referred to as "UPA" grade are purified by triple distillation.). The diamonds were then rinsed in MQ (milli-Q) water and dried. All samples were weighed prior to the ablation.

### 2.2. Sample ablation and collection

Diamond slabs were ablated in a custom-designed, sealed PTFE ablation cell capped with a laser window that had been previously cleaned with acid. Ablations were performed with a UP-213 New-Wave Laser ablation system, with the custom cell replacing that provided by the manufacturer. A pre-weighed diamond was brought into focus and an ablation was performed using a raster-pattern. Ablation conditions were: scan speed 50  $\mu\text{m}/\text{s}$ ; raster spacing 80  $\mu\text{m}$ ; energy output 5–6  $\text{J}/\text{cm}^2$ ; repetition rate 20 Hz; spot size 200  $\mu\text{m}$  and pass depth 2  $\mu\text{m}$ . Following ablation the laser cell window was cleaned with 6 N HCl acid which was dried and kept for analysis. One mL of 6 N HCl was added to the sample beaker and a Teflon cap replaced the laser window. The sealed cell was then placed in an ultrasonic bath for 30 min. The fluid was then collected from the ablation cell and transferred to a 3 mL Teflon beaker and dried. The diamond was rinsed in MQ water and dried. Diamonds were re-weighed and the weight loss resulting from the ablation was calculated 50 repeat weighings of a diamond yielded an uncertainty of 0.1  $\mu\text{g}$ .

#### 2.2.1. Separation chemistry

The dried samples were taken up in 200  $\mu\text{L}$  of 3 N  $\text{HNO}_3$  and placed on the hot plate for 1 h to homogenize. After cooling a ~10% aliquot by weight was transferred into a pre-leached micro-tube for trace element analysis. The remaining sample was processed for isotopic analysis. The Sr separation procedure is based on the method described by Charlier et al. (2006), using Sr-spec resin but with modifications as outlined by Harlou et al. (2009). Pb was eluted following Sr. The pre-Sr column eluant containing the REEs was collected and used for Nd isotope separation using AG50-X8 200–400# chromatographic resin.

#### 2.2.2. Trace elements – total procedural blanks and limits of quantification

To obtain a statistically valid view of the "background" corrections for any low-level chemical procedure it is necessary to have adequate knowledge of analytical blanks. Only then can confidence be placed in estimates of the limits of quantification. We use the limit of quantification (LOQ) as defined by Currie (1968) as a measure of our ability to quantitatively measure elemental abundances because this parameter is significantly more robust than defining "limits of detection" or LOD, which merely define the ability to qualitatively detect an analyte. The LOQ for a procedure with a well characterized blank is defined by Currie (1968) as:

$$\text{LOQ} = 10\sigma \quad (1)$$

where  $\sigma$  is the standard deviation of the blank for the process (here defined as the total procedural blank or TPB). This approach places

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