



Article Microdiamonds in Alkalic Dolerites from the North China Craton: FTIR and C Isotopic Characteristics

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Abstract: Most of the diamond deposits in China are in the North China Craton. In addition to gem diamonds in kimberlite, a large number of microdiamonds have also been discovered in alkaline dolerites. These microdiamonds show very different characteristics from those recovered in kimberlite. Here, we report the morphology, colour, nitrogen contents, and carbon isotopic compositions of the diamonds recovered from the alkalic dolerites in eastern China. The microdiamonds are mainly cube and rhombic dodecahedron with diameters of 0.2 to 0.6 mm. Infrared spectrum analysis shows that these microdiamonds are mostly type Ib with a small amount of type Ia. The Y centre is obvious in type Ib diamond. Modelling mantle residence times for the IaAB diamonds is about 550 Ma. Nitrogen contents of the diamonds varies between -18.6 and -21.1% and are similar to those of ophiolite diamond.

Keywords: microdiamond; FTIR; carbon isotopic; alkalic dolerite; North China Craton

1. Introduction

Diamonds on the Earth mainly occur in volcanic rocks such as kimberlites and lamproites [1–8], but can also be found in ultrahigh-pressure metamorphic rocks [9,10], meteorites [11] and alluvial deposits [12]. In recent years, diamonds have been recovered from ophiolites [13–16] and alkalic dolerites [17,18]. The discovery of ophiolitic diamonds and alkalic dolerites diamonds has drawn significant research interests to explore the origin of this new class of diamond source and to infer the evolution of their hosting rocks [19–21]. This new type of diamond had been initially considered as a result of contamination. However, more and more evidence either directly or indirectly demonstrates that these diamonds are of natural origin [16,18,19,22–24].

During a geological survey from 2012 to 2015, the geologist from Nanjing Centre of China Geological Survey discovered a large number of yellow microdiamonds in the Langan area in northern Anhui Province [18,25–29]. The diamond-bearing rocks of these microdiamonds mainly include dolerite and olivine basalt. From 2016 to 2018, four microdiamonds in basic rocks were recovered again in the prospecting work for primary diamond deposits in the Tashan and Zhangji areas in Xuzhou, which is geographically close to Langan [30]. All these diamonds are similar in colour and shape to ophiolite type diamonds [31] and show different characteristics of kimberlite and lamproite diamonds.

Cai et al. (2019) reported the petrological characteristics of the diamondiferous rocks [17,21,30]. In this paper, the morphology, infrared spectrum, and carbon isotope compositions of microdiamonds were analysed and discussed by Fourier infrared spectroscopy



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Copyright: © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). and carbon isotope test. The types of microdiamonds found in the North China Craton, the age of mantle occurrence, and the source of carbon isotopes were revealed.

2. Geological Background and Samples

The North China Craton (NCC) is one of the oldest cratons on Earth [32–34]. It was amalgamated after the collision of Eastern Block and Western Block at ca. 1.8 Ga, which resulted in the intervening Central Orogenic Belt [34]. The basement of the NCC mainly consists of Archean to Paleoproterozoic TTG (tonalitic–trondhjemite–granodioritic) gneisses that are covered by Mesoproterozoic to Paleozoic sediments. The basement rocks in the study area are composed of the Archaean Wuhe Group and the Paleoproterozoic Fengyang Group and are overlined by the Proterozoic and Lower-Paleozoic cover rocks, including dolomite, limestone, shale, and sandstone. Voluminous Mesozoic magmatic rocks including diabase, basalts, quartz syenite porphyries, and spessartites occur in the area [28].

Several diamond deposits have been reported in the North China Craton [33,35]. The two most significant deposits are in Wafangdian of Liaoning province and Mengyin of Shandong Province [1]. They are distributed on both sides of the Tanlu fault—the Wafangdian diamond ore area lies in the east and the Mengyin diamond ore area lies in the west. Moreover, many microdiamonds have been found in the western side of the Tanlu fault and the southern margin of the NCC, such as those in the northern Jiangsu and Anhui provinces. The area where AD microdiamonds (microdiamonds from alkalic dolerites) were found is located in the southeast margin of the NCC and west of the Tanlu fault.

Diamonds in basaltic rocks mostly coexist with high-pressure minerals such as pyrope and hessonite [25,26,28]. The AD microdiamonds obtained in this study are cube and rhombic dodecahedron with diameters of 0.2 to 0.6 mm. These microdiamonds are colourless to yellowish (Figure 1a–c) [30]. Microscopic observations revealed irregularly shaped black to light-coloured mineral inclusions. High pressure minerals were also observed in these microdiamonds. More detailed mineralogical characterization will be reported in a separate paper. The surface characteristics of diamonds, such as dissolution, can be observed on relatively large microdiamonds (Figure 1d) [30]. The characteristics of diamonds are summarized in Table 1.

Sample No.	Color	Shape	Surface Character
AD1	yellow	cubo-octahedral	Shallow depressions
AD2	Light yellow	Rounded Dodecahedra	Stacked growth layers, plastic deformation lines, terraces, elongate hillocks
AD3	colorless	fragment	Stacked growth layers,
AD4	yellow	cubo-octahedral	Stacked growth layers,
AD5	yellow	cubo-octahedral	Stacked growth layers,
AD6	yellow	cubo-octahedral	
AD7	yellow	cubo-octahedral	Challens democrations
AD8	yellow	cubo-octahedral	shanow depressions,
AD9	yellow	cubo-octahedral	abrasion, scratch mark
AD10	yellow	cubo-octahedral	

 Table 1. Characteristic of the tested Diamonds.



Figure 1. (**a**) Ib type AD microdiamond; (**b**) IaA/Ib type AD microdiamonds; (**c**) IaAB type AD microdiamond; (**d**) IaA type AD microdiamond.

3. Analytical Method

3.1. Fourier Transform Infrared Spectroscopy

Infrared absorption spectra were collected using Bruker Vertex80 (Second Institute of Oceanography, MNR) and Thermo Fisher Nicolet Nexus 470 (University of Alberta) FTIR spectrometer, equipped with a Continuum IR microscope with a motorized stage. A midinfrared light source was used to collect spectra with a spectral range of 4000–650 cm⁻¹. Diamonds were placed on a KBr plane and measured under $20 \times$ magnification infrared objective in transmission mode. Spectra were acquired by averaging 200 scans at a spectral resolution of 1 cm⁻¹ with an aperture size of $100 \times 100 \mu$ m. Baseline corrected spectra were normalized to 1 cm sample thickness employing an absorption coefficient of 11.94 cm⁻¹ for the intrinsic absorption of a diamond at 1995 cm⁻¹ [36,37].

3.2. Carbon Isotopic Composition

Carbon isotopic compositions were measured at the University of Alberta. Diamond grains weighing 129–314 ug were weighed and wrapped into a tin capsule, which was then loaded and combusted in an Elemental Analyzer (Thermal Flash 2000) at 1050 °C. The produced CO₂ was carried by a high-purity helium stream to an isotope ratio mass spectrometer (Thermo Delta V Plus) for isotopic measurement. Diamonds are resistant to combustion and complete combustion generally required multiple burning. Cumulative CO₂ yields of 100% were normally achieved after 3–4 burns. CO₂ gas from each burn was measured for δ^{13} C value unless the amount of CO₂ gas in the last burn was too low to give reliable data. The δ^{13} C reproducibility of all the burns from individual diamonds was better than 0.3‰. A weighted average δ^{13} C value of all the burns was used to represent the value of each diamond. CO₂ blanks were carefully monitored between samples to ensure no memory effect. The low-organic content soil standard (reference values: C = 1.61 wt%; δ^{13} C_{V-PDB} = -26.66‰) and high-organic content sediment standard (reference values: C = 7.45 wt%; δ^{13} C_{V-PDB} = -28.85‰) were measured in parallel to samples and used to

calibrate the carbon yield and isotopic ratios of samples. Repeated analysis of the standards gave a 2 standard deviation better than 0.2‰.

4. Analysis Result

4.1. Fourier Transform Infrared Spectroscopy

Infrared spectral data of ten samples were obtained and fitted by the OMNIC software. The DiaMap software (DiaMap_CABX 17_11_07 version) was used for spectrum analysis and N content calculation [37–40]. The concentration of C centres (in at. ppm) was calculated by multiplying the absorption coefficient at 1344 cm⁻¹ by a factor of 37 [41,42].

Microdiamonds exhibit a broad range of N concentration from 4.5–503 ppm, with a median value of 173 ppm. Eight diamonds exhibit intense N absorption, and C centres were found at 1344 cm⁻¹ in these diamonds. Three diamonds that contain N predominantly in the form of A centres also show a discernible C centre line at 1344 cm⁻¹; these diamonds are classified and referred to as Type Ia/Ib hereafter (Table 2). Bands or lines corresponding to B, B' and A centres were found co-existing in two diamonds.

Table 2. Types of tested microdiamonds and the data of the IR spectrums.

Sample No.	Major Peak (cm $^{-1}$)	Type of C-N	Туре	N Concentration (10 ⁻⁶)	N _{total} (10 ⁻⁶)
AD1	1280, 1344, 1976, 2852.2, 2925	A, C	IaA/Ib	143.0 (C centre)	143.0
AD2	1172, 1288, 1361, 1650, 1976, 1851, 2919	Α, Β΄, Β	IaAB	60.9 (A centre), 134.2 (B centre)	195.1
AD3	1650, 1976, 2850, 2919	А	Ia	4.5 (A centre)	4.5
AD4	1130, 1344, 1976	С	Ib	10.7 (C centre)	10.7
AD5	1128, 1344, 1650, 1976, 2850, 2919, 3290	С	Ib	76.9 (C centre)	76.9
AD5	1130, 1284, 1344.6, 1645, 1976.7, 2848, 2919.7, 3201, 3394	Α, C	IaA/Ib	23.6 (C centre)	23.6
AD7	1128, 1272, 1344, 1976, 2848, 2921	С	Ib	393.5 (C centre)	393.5
AD8	1128, 1282, 1344, 1596, 1976, 2854, 2919	Α, C	IaA/Ib	240.9 (C centre)	240.9
AD9	1344, 1645, 1976, 2848, 2919, 3191, 3394	С	Ib	137.0 (C centre)	137.0
AD10	1128, 1272, 1344, 1600, 1976, 2848, 2917	С	Ib	503.5 (C centre)	503.5

4.2. C Isotope

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The C isotopic results of microdiamonds are listed in Table 3. The overall range of δ^{13} C values in the microdiamonds is between -18.6 and -21.1% (Table 3).

Sample ID	δ ¹³ C (‰)		
AD5	-20.2		
AD6	-19.1		
AD7	-18.6		
AD8	-20.5		
AD9	-21.1		
AD10	-20.6		

5. Discussion

5.1. FTIR Spectra of AD Microdiamonds

5.1.1. C-C Absorption Lines

All the diamond samples showed clear absorptions between 1970 and 2300 cm⁻¹, which were the vibration absorption of C-C, mainly at 1976, 2027 and 2158, among which the absorption at 1976 was the most clear [43].

5.1.2. C-N Absorption Lines

Natural diamonds are commonly classified according to the presence or absence of nitrogen. Nitrogen is incorporated in the lattice, first as single nitrogen, and then progressively aggregated by natural annealing over time in the sequence of single nitrogen (C centre)–A-aggregate (A centre)–B aggregate (B centre) [44].

In the studied diamonds, A centre (1282 cm⁻¹) was detected in five diamonds, B centre was detected in one diamond and C-centre was detected in eight diamonds. So, these samples can be classified as type IaA (Figure 2, AD3), IaAB (Figure 2, AD2), IaA/Ib (Figure 3), and Ib (Figure 4).



Figure 2. Infrared absorption spectrum collected from diamond AD3 and AD2.



Figure 3. Infrared absorption spectrum collected from diamond AD1, AD6 and AD9 exhibiting both A and C centre.



Figure 4. Infrared absorption spectrum collected from diamonds exhibiting C centres.

The eight diamonds are distinctly of type Ib character and three of the samples contain the 1344 cm⁻¹ single nitrogen absorptions, together with the 1282 cm⁻¹ A-aggregate (Figure 3). However, the di-nitrogen (A centre) abortion was not strong, indicating that the diamonds have low nitrogen conversion rates in the mantle and still retain most of the single nitrogen. This could be attributed to 1) the mantle temperature is relatively low, and/or 2) the mantle residence time is short [45].

Bands or lines corresponding to B and B' centres were found co-existing in sample AD2, which is confirmed to be Type IaAB with a total N concentration of 195 at. ppm and a B centre proportion [$100 N_B/(N_B+N_A)$] of 68.8%.

5.1.3. H₂O and C-H Absorption Line

In these samples, the vast majority of samples showed the absorption of H_2O at about 1645 cm⁻¹, which was caused by the bending vibration of H_2O molecule [39]. Meanwhile, many samples also had the symmetric stretching vibration of H_2O at about 3200 cm⁻¹. However, the antisymmetric stretching vibration absorption of H_2O around 3630 cm⁻¹ was not detected in these samples.

C-H-related absorption was common in the spectra [39], with typical 2920 and 2850 cm⁻¹ absorption being detected in most samples. The VN₃H line at 3107 cm⁻¹ was detected in samples AD2 and AD3. The absorption near 3394 cm⁻¹ was detected in some samples, which was related to the N-H bond [39].

5.1.4. Y-Centre

In this study, Y-centres (Figure 5) were also detected in 4 microdiamonds. The centre is characterized by the dominant asymmetric absorption centred at approximately 1145 to 1150 cm^{-1} . The defect was discovered by Hainschwang (2012) through infrared spectroscopic determination and analysis of a large number of natural and synthetic type Ib yellow diamonds. The large sampling for that study shows that in natural type Ib samples from recent diamond productions the Y-centre is very common. So far, the Y-centre has neither been detected in synthetic diamonds, nor in single nitrogen free type Ia diamonds [40].



Figure 5. Y-centres in microdiamonds.

Type Ib diamonds are very rare in nature (<1% of cape series yellow diamonds in nature). So far, type Ib diamonds have only been discovered in Helam/Swartruggens in the eastern block of the Kaapvaal craton [46,47], Lac de Gras in the Slave Craton [48], Dachine in the Amazon Craton [49–51], Orapa rock tubes in the southwest of the Zimbabwe Craton [52], Qilalugaq of the Rae Craton and the Kankan region of the West African Craton [53,54], Tibet of China and Pozanti-Karsanti of Turkey [19].

5.2. Modelling of Mantle Residence Time

Modelling mantle residence times for the diamonds requires N concentration, N aggregation state and mantle residence temperature as input parameters [44,55–58]. The model is also based on the assumption that the total N concentration of diamond is reflected in the infrared absorption spectrum and that there is a smooth aggregation process from C to A to B centres [55]. The requisite nitrogen concentrations and aggregation states were determined via FTIR analyses (see above). The mantle residence temperature was assumed to be in the range of 1200–1225 °C [33].

The infrared spectra of four points from the core (location 01) to the edge (location 04) of the diamond were determined. According to the analytical results, the content and accumulation degree of N in diamonds do show spatial variations. The FTIR spectrum of sample AD3 illustrates that the absorption of diamond core was at 1370 and 1175 cm⁻¹ with strong absorption rate, which suggests a IaAB type. However, the absorption rate was weakened as the transition to the edge, where the absorption was mainly at 1282 cm⁻¹, a character of IaA type (Table 4). These characteristics may also reflect the changes of crystallization environment and conditions of diamond with complex structure in different growth stages, or the ability of diamond to capture N. The model ages calculated by N ranged from 1.29 Ga to 0.74 Ga from the core to edge, so mantle residence time modelled at 1200 °C is estimated to be 0.55 Ga (Figure 6).

Table 4. Calculation of N concentration and t_{MR} .

Location	N _A /ppm	N _B /ppm	NT	Modelling Age/Ga
	(~1282 cm ⁻¹)	$N_B(\sim 1175 \text{ cm}^{-1})$	(×10 ⁻⁶)	
01	41.590	56.615	98.205	1.29
02	58.767	71.982	130.750	0.87
03	56.229	59.316	115.545	0.85
04	74.587	109.528	184.115	0.74



Figure 6. Nitrogen content plotted against the percentage of B (100B/[A+ B]) aggregation in 550 Ma for Type IaAB diamonds [37,59].

5.3. C Isotope

Carbon isotopic compositions of diamonds from kimberlite, lamproite and metamorphic rocks have been extensively studied [23,49,60–62]. According to Cartigny (2005), eclogitic diamonds (E-type) have δ^{13} C ranging from -38.5 to +2.7% and peridotitic diamonds have δ^{13} C from -26.4 to +0.2%. Despite the different δ^{13} C ranges between eclogitic and peridotitic diamonds, both groups of diamonds are characterized by a mode at δ^{13} C $\sim -5\%$, which is consistent with the mantle range of carbon isotopic composition [62]. Ultra-high pressure (UHP) metamorphic diamonds mainly have δ^{13} C out of the normal mantle range [9,62]. Diamonds from alkalic dolerites studied here are all characterized by low δ^{13} C values with a narrow range (Figure 7). These low δ^{13} C values of dolerites-hosted diamonds overlap with the lower ends of peridotitic diamonds and metamorphic diamonds, and the upper end of the ophiolitic diamonds.



Figure 7. Comparative Column of δ^{13} C values (modified from Cartigny (2005)). **1**-Macro-diamonds from North China Craton; **2**-Macro-diamonds from Yangtze Craton; **3**-microdiamonds in Alkalic dolerites.

6. Conclusions

In the past, many deposits of macro-diamonds, mostly of type Ia or IIa, were found in the North China Craton, and they have been extensively studied. Microdiamonds which were recovered from the alkalic dolerites of the North China Craton were studied by FTIR and Carbon isotopic.

These diamonds are similar in colour and shape to ophiolite type diamonds and show different characteristics of kimberlite and lamproite diamonds. These diamonds are usually light yellow to yellow, with a few colourless, and cubic, octahedral or rhomboidal dodecahedron, and octahedron in shape. The surface characteristics of diamonds, such as dissolution, can be observed. The overall N concentration is not high, with an average of 173 ppm. The infrared spectra show that most of these diamonds were type Ib, and C centres were found at 1344 cm⁻¹ in these diamonds. Three diamonds of our samples are classified as Type Ia/Ib, because of A centres and C centres in these diamonds. Two diamonds are classified as type IaAB because B, B' and A centres were found co-existing. FTIR microscopic measurements from the core to the edge of the type IaAB diamond suggest a mantle residence time of approximately 550 Ma. The C isotopic analysis reveals that these diamonds are strongly depleted in ¹³C. These low δ^{13} C values of doleriteshosted diamonds overlap with the lower ends of peridotitic diamonds and metamorphic diamonds, and the upper end of the ophiolitic diamonds. Additionally, the reason for the strong deficit δ^{13} C shown by the carbon isotope should be studied in the future.

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