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# Precisely dating Paleozoic kimberlites in the North China Craton and Hf isotopic constraints on the evolution of the subcontinental lithospheric mantle

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### ABSTRACT

Kimberlite, a deep-sourced ultramafic potassic rock, carries not only diamond, but also invaluable mantle xenoliths and/or xenocrysts, which are important for tracking the evolution of subcontinental lithospheric mantle (SCLM). However, it is challenging to accurately determine the emplacement age of kimberlite and its compositions of primary magma because of modifications by crustal and/or mantle contamination and postemplacement alteration. This paper reports emplacement ages of diamondiferous kimberlites in Mengyin and Fuxian of the North China Craton (NCC) using three different dating methods. For Mengyin kimberlite, singlegrain phlogopite Rb-Sr dating yields an isochron age of  $485 \pm 4$  Ma, U-Th-Pb analyses on perovskite give  $a^{238}U^{-206}Pb$  age of 480.6  $\pm$  2.9 Ma and  $a^{232}Th^{-208}Pb$  age of 478.9  $\pm$  3.9 Ma, and baddeleyite yields  $a^{207}Pb^{-206}Pb$ age of  $480.4 \pm 3.9$  Ma. For Fuxian kimberlite, baddelevite gives a  $^{207}$ Pb $^{-206}$ Pb age of  $479.6 \pm 3.9$  Ma, indicating that the Paleozoic kimberlites in the NCC were emplaced at ~480 Ma. Numerous lines of evidence indicate that the studied baddelevites are xenocrysts from the SCLM, and can be used to constrain Hf isotope compositions (Euf  $(t) \sim -6)$  of the SCLM when kimberlite erupted. Combined with data from Mesozoic–Cenozoic mantle-derived rocks and xenoliths, the Hf isotope evolution trend of the SCLM beneath NCC before craton destruction was tentatively constructed, which suggested that the Archean SLCM was enriched by metasomatism at ~1.3 Ga. Further Hf isotope investigations on additional SCLM-derived materials could be used to compare with the constructed Hf isotope evolution trend before craton destruction to determine when lithospheric thinning occurred.

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# 1. Introduction

The North China Craton (NCC) (Fig. 1) is one of the world's oldest Archean blocks as manifested by crustal remnants as old as 3800 Ma (Liu et al., 1992; Song et al., 1996; Wu et al., 2008a; Zheng et al., 2004a). The existence of Ordovician diamondiferous kimberlites in the NCC indicates a thick (~200 km) lithosphere in the early Paleozoic. However, at present the lithosphere is <80 km thick as revealed by seismic studies and petrologic studies of mantle xenoliths in Mesozoic-Cenozoic "intraplate" volcanism, suggesting that a significant part of the original lithospheric mantle beneath the eastern NCC was removed during the Phanerozoic (e.g. Fan and Menzies, 1992; Gao et al., 2002; Griffin et al., 1998; Menzies et al., 1993, 2007; Menzies and Xu, 1998; Xu, 2001; Zheng et al., 1998, 2007). The thick, old, cold and refractory subcontinental lithospheric mantle (SCLM) beneath the NCC was subsequently replaced by thin, young, hot and fertile mantle (e.g. Gao et al., 2002; Griffin et al., 1998; Huang et al., 2007; Menzies and Xu, 1998; Menzies et al., 2007; Wu et al., 2003, 2006a; Xu, 2001; Zhang et al., 2008; Zheng et al., 1998).

Although extensive investigations have been conducted on the lithospheric thinning process, there are still considerable debate on its mechanism, with lithospheric delamination (e.g. Deng et al., 2007; Gao et al., 2002, 2004, 2008; Wu et al., 2003, 2005a) and thermo-mechanical erosion (e.g.Griffin et al., 1998; Menzies and Xu, 1998; Xu, 2001; Zhang, 2005; Zhang et al., 2008) being commonly proposed. The delamination model, (a more rapid process), proposes that the thinning was triggered by foundering and sinking of heavy material, and predicts that the present SCLM is juvenile. In contrast, the erosion model emphasizes a slow chemical process of asthenospheric upwelling, forming a stratified SCLM with Archean relict overlying newly accreted material (Griffin et al., 1998; Menzies and Xu, 1998).

Understanding SCLM evolution is helpful in deciphering the lithospheric thinning mechanisms. For this reason, extensive studies have been conducted on mantle xenoliths and SCLM-derived maficalkaline rocks in the NCC (e.g., Chu et al., 2009; Gao et al., 2002; Wu et al., 2003, 2006a; Xu et al., 2008; Zhang et al., 2008; Zheng et al., 2009). However, our knowledge about the Paleozoic SCLM beneath the NCC is rather limited. Firstly, the age of the diamondiferous Mengyin (Shandong province) and Fuxian (Liaoning Province) kimberlites, erupted on opposite sides of the translithospheric Tanlu fault (Fig. 1), is not well determined. Available geochronological data yield a wide range from

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Fig. 1. Geological sketch map showing the Cratons in China and sample localities discussed in the text. Diamond: kimberlite. Circle: nepheline syenite.

456 Ma to 475 Ma based on phlogopite Rb–Sr and Ar–Ar, perovskite U–Pb by TIMS and LA-ICPMS methods (Dobbs et al., 1994; Li et al., 2005; Yang et al., 2009; Zhang and Yang, 2007). Secondly, although it has been proposed that the Paleozoic SCLM was Sr–Nd isotopically enriched, significantly different from that of depleted SCLM in the Cenozoic (Chi and Lu, 1996; Fan and Menzies, 1992; Griffin et al., 1998; Huang et al., 2007; Menzies and Xu, 1998; Zheng et al., 1998), this conclusion was based on the limited data from significantly altered peridotite samples. The Os isotope character of these altered samples is likely to be less disturbed and so an Archean melt-extraction age can be established (Chu et al., 2004), however other isotopic features like Sr–Nd–Hf systems of the SCLM are still in doubt or remain unknown (Wu et al., 2008b).

In this paper, we report a series of comprehensive dating results of phlogopite Rb–Sr, perovskite <sup>238</sup>U–<sup>206</sup>Pb and <sup>232</sup>Th–<sup>208</sup>Pb and baddeleyite <sup>207</sup>Pb–<sup>206</sup>Pb analyses obtained from the Mengyin and Fuxian kimberlites. The baddeleyites, considered to be mantle xenocrysts, were used to trace the Hf isotope composition of the Paleozoic SCLM.

#### 2. Geological settings and samples

The NCC is the oldest tectonic unit in China, with crustal components up to ca. 3.8 Ga exposed in the far north-east (e.g., Liu et al., 1992; Wu et al., 2008a). The Early Paleozoic Qilianshan Orogen and the Late Paleozoic Central Asian Orogenic Belt bound the craton to the west and the north, respectively, and in the south the Qinling-Dabie-Sulu ultrahigh-pressure metamorphic belt separates it from the South China Craton (Fig. 1). Based on age, lithological assemblage, tectonic evolution and P–T–t paths, the NCC has been divided into Eastern and Western blocks, which were amalgamated along the Paleoproterozoic Trans-North China Orogen (Zhao et al., 2005 and references therein). Based on today's seismology and geography, the NCC can be separated into two different tectonic domains by the N–S trending Daxinganling–Taihangshan gravity lineament (DTGL) (Ma, 1989; Menzies and Xu, 1998).

Similar to other Archean blocks around the world, the NCC contains both greenstone belts and high-grade metamorphic terrains, which were metamorphosed at 2.5 Ga and subsequently cratonized at 1.8 Ga by collision of the Eastern and Western blocks (e.g., Wu et al., 2005a; Zhao et al., 2005). After 1.8 Ga, the NCC has remained relatively stable and was covered by a thick sequence of Mesoproterozoic to Paleozoic sediments. In the Paleozoic, when the diamondiferous Mengyin and Fuxian kimberlites were emplaced in Shandong and Liaoning provinces, respectively (Zhang et al., 1989), and the NCC was characterized by thick carbonate sedimentation during the Cambrian to Early Ordovician. In the Mesozoic, extensive volcanic activity and granitoid emplacement occurred in the eastern NCC, possibly due to the interaction of the Eurasian and Pacific plates and resulting from the lithospheric thinning (Wu et al., 2003, 2005b). During the Cenozoic, numerous alkaline basalts containing mantle peridotite and minor lower-crustal granulites xenoliths were erupted throughout the central and eastern parts of the craton.

In this study, kimberlites from Mengyin and Fuxian were investigated. The Mengyin kimberlites are diamondiferous and erupted through the Archean Taishan Complex. About 100 kimberlitic dykes and pipes have been identified (Fig. 1, Chi and Lu, 1996; Wan, 1989). Among them, the pipe 1 (Shengli 1, N35°40′ and E117°47′) is the most important diamondiferous one in the area and was targeted for this study. Perovskite is relatively abundant in the Mengyin kimberlite groundmass, with concentrations up to 5% (rarely up to 20%, Yang et al., 2009). Most perovskite grains are dark orange to brown and euhedral shapes with grain sizes ranging from 30 to 200 µm. Phlogopite and perovskite were separated from sample MY12, the only one containing baddeleyite, for further investigation. The Fuxian diamondiferous kimberlites are emplaced in the Mesoproterozoic-Cambrian country rocks (Fig. 1). Samples here show more extensive alteration and weathering. Although a number of kimberlite samples were used to separate perovskite and baddeleyite, perovskite is extremely rare and baddeleyite was obtained from only one rock sample. In both localities, these kimberlites contain a variety of crustal fragments, including limestone, gneiss, amphibolite, mafic granulite xenoliths (Chi and Lu, 1996; Dong, 1994; Wan, 1989; Zheng et al., 2004a,b) and exotic zircons with ages of 2.5 Ga (Yin et al., 2005; Zheng et al., 2009).

#### 3. Analytical procedures

The kimberlite samples were crushed using a jaw crusher and bico disk mill equipped with hardened steel plates. Minerals were concentrated using a wilfley Table, heavy liquids and a Frantz Isodynamic separator. Clean and fresh mineral grains were hand-picked under a binocular microscope. Minerals fractions including phlogopite, baddeleyite and perovskite were separated from sample MY12 of the Mengyin kimberlite, and baddeleyite from sample FX-1 of the Fuxian kimberlite. Phlogopite grains were selected for single-grain Rb–Sr dating, and perovskites were analyzed for U–Pb dating, whereas baddeleyites were selected for U–Pb or Pb–Pb dating and Hf isotope analyses. All analyses were performed at the Institute of Geology and Geophysics, Chinese Academy of Sciences (IGGCAS) and are detailed in Electronic Appendix file.

## 4. Analytical results

# 4.1. Phlogopite Rb-Sr age

Single-grain phlogopite Rb–Sr isotopic data for the MY12 kimberlite sample after blank and spike corrections are shown in Fig. 2 and Appendix Table 1. Regression and age calculation of isochron were performed using the Isoplot software (Ludwig, 2003). The decay constant of <sup>87</sup>Rb is  $1.42 \times 10^{-11}$  as recommended by Steiger and Jäger (1977), giving 1.5% for errors of <sup>87</sup>Rb/<sup>86</sup>Sr ratios and 0.01% for errors of <sup>87</sup>Sr/<sup>86</sup>Sr ratios. Rb and Sr contents were calculated using estimated weight of the analyzed grains (ca. 0.1 mg per grain). Eight individual grains of phlogopite give <sup>87</sup>Rb/<sup>86</sup>Sr ranging from 18.6 to 450 and yield an isochron age of  $485 \pm 4$  Ma with an initial <sup>87</sup>Sr/<sup>86</sup>Sr of 0.7138  $\pm$  0.0029 (MSWD = 0.5, Fig. 2a).

For a comparison, Rb–Sr isochron of the Fuxian phlogopite measured in Li et al. (2005) is also shown here (Fig. 2b). Eleven analyses yielded an



Fig. 2. Single-grain phlogopite Rb-Sr isochrons of the Mengyin kimberlite (a) and Fuxian kimberlite (b).

age of  $463 \pm 7$  Ma with an initial  ${}^{87}$ Sr/ ${}^{86}$ Sr of  $0.7225 \pm 0.0057$ . This age is ~20 Ma younger than that obtained for the Mengyin phlogopite. The Fuxian phlogopites have higher initial Sr isotopic ratio than the Mengyin samples.

# 4.2. Perovskite U-Th-Pb ages

MY12 perovskites are euhedral and fresh with grain sizes ranging from 30 to 100 µm (Yang et al., 2009). Fifteen U-Pb analyses were performed in separated two sessions. The data are plotted in Fig. 3 (Appendix Table 2). All the 30 analyses show that MY12 perovskites are fairly homogeneous with a uranium content of  $72 \pm 7$  ppm (1 SD). Common lead ranges from 5 to 11 ppm, and the  $^{204}$ Pb-based  $f_{206}$  value (percentage of common lead <sup>206</sup>Pb in total <sup>206</sup>Pb) ranges from 22% to 26%. Thorium content varies significantly, with Th/U ranging from 22 to 106. Due to the tight clustering of data points, a Tera-Wasserburg plot gave an imprecise lower intercept age at 501 + 55 Ma, and an imprecise upper intercept of common  $^{207}$ Pb/ $^{206}$ Pb composition at 0.94 + 0.19 as well. However, if the terrestrial Pb model of Stacey and Kramers (1975) is applied as an estimate of common lead composition, a Concordia U–Pb age of  $480.6 \pm 2.9$  Ma can be obtained (Fig. 3). A weighted average <sup>206</sup>Pb/<sup>238</sup>U age derived by the <sup>204</sup>Pbbased common-Pb correction is  $480.9 \pm 2.8$  Ma (MSWD = 1.0), identical to the average  ${}^{206}$ Pb/ ${}^{238}$ U age of  $480.9 \pm 2.5$  Ma (MSWD = 1.1) given by the <sup>207</sup>Pb-based common-Pb correction. The rather high Th contents result in low <sup>204</sup>Pb-based  $f_{208}$  values (percentage of common lead <sup>208</sup>Pb in total <sup>208</sup>Pb) ranging from 2.3% to 6.8%. A weighted average of <sup>208</sup>Pb/<sup>232</sup>Th ages is  $478.9 \pm 3.9$  Ma (MSWD = 0.49, Fig. 3, inserted). The agreement between the <sup>238</sup>U-<sup>206</sup>Pb and <sup>232</sup>Th-<sup>208</sup>Pb ages indicates a closed U-Th-Pb system in this perovskite (Li et al., 2010b).

#### 4.3. Baddeleyite U-Pb and Pb/Pb ages

Baddeleyite grains from MY12 kimberlite are subhedral or fragmental with 10–100 µm in length (Fig. 4a). Nineteen Pb/Pb analytical data with multi-collector mode are listed in Appendix Table 3 and plotted in Fig. 4b. Uranium contents range from 84 to 467 ppm. Their measured radiogenic <sup>207</sup>Pb/<sup>206</sup>Pb are indistinguishable within analytical errors, with a weighted mean of 0.05671  $\pm$  0.00010, corresponding to a Pb/Pb age of 480.4  $\pm$  3.9 Ma (MSWD = 0.91, Fig. 4b).

Like the Mengyin kimberlite, baddelevite grains from Fuxian kimberlite (sample FX-1) are also subhedral and fragmental, and are 10-100 µm in length (Fig. 4c). Twenty five U-Pb analyses were conducted under mono-collector mode and the data are reported in Appendix Table 4, and the obtained Pb/Pb age is shown in Fig. 4d. It is noted that the FX-1 baddelevites contain high and variable U content from 628 to 2958 ppm, and many grains show extremely high Th contents (up to 1328 ppm) with Th/U up to 0.45. The calculated U-Pb ages are variable between 443 and 550 Ma and broadly positively correlate with U, Th contents and Th/U, which could be attributed to crystal orientation effect (Wingate and Compston, 2000) and/or high U effect (Li et al., 2010a; Williams and Hergt, 2000). However, radiogenic <sup>207</sup>Pb/<sup>206</sup>Pb of 25 measurements are consistent, within analytical errors, with a weighted average of  $0.05669 \pm 0.00013$ , corresponding to a Pb/Pb age of  $479.6 \pm 4.9$  Ma (MSWD = 0.71, Fig. 4d).

# 4.4. Baddeleyite Hf isotope compositions

The Hf analyses were obtained using the same mounts which were previously used for U–Pb and Pb–Pb dating. The results are shown in Fig. 5 and Appendix Table 5. All baddeleyites have very low  $^{176}Lu/^{177}$ Hf (0.000005–0.000033). However, the small grain size, mostly <30 µm in width, and needle- to wafer-shaped morphology make the laserablation measurements quite difficult. Many grains were ejected after short bombardment of laser, which resulted in big errors. Nevertheless, twenty-eight analyses on MY12 baddeleyite were obtained with

internal uncertainty ranging from 0.000031 to 0.000079 at  $2\sigma_m$  level. Though all measured <sup>176</sup>Hf/<sup>177</sup>Hf ratios show a Gaussian distribution pattern (Fig. 5a), those data with internal uncertainty larger than 0.00005 were excluded to calculate the average. A weighted average of <sup>176</sup>Hf/<sup>177</sup>Hf is 0.282286±0.000020 ( $2\sigma_m$ , n=12, MSWD=2.5), corresponding to  $\varepsilon_{Hf}$  (t=480 Ma) value of  $-6.62\pm0.64$  (Fig. 5a).

Nineteen measurements on FX-1 baddeleyite show variations of  $^{176}\text{Hf}/^{177}\text{Hf}$  ranging from 0.282283 to 0.282389 with internal uncertainties from 0.000014 to 0.000035 at  $2\sigma_m$  level. Similarly, the Hf isotopic data show a single population, and have a weighted average value of 0.282308  $\pm$  0.000010 ( $2\sigma_m$ , n = 18/19, MSWD = 1.6), corresponding to  $\epsilon_{\text{Hf}}$  (t = 480 Ma) value of  $-5.86\pm0.36$  (Fig. 5b).

# 5. Discussion

#### 5.1. Emplacement age of kimberlites

Kimberlites and related rocks are a series of volatile-rich potassic ultramafic rocks that originate from the deep lithospheric or sublithospheric mantle (Mitchell, 1986, 1995; Woolley et al., 1996). They generally contain abundant crustal and mantle xenoliths and are highly susceptible to alteration and weathering. Therefore, the combined effects of contamination and post-emplacement alteration make it difficult not only for determining the compositions of the primary kimberlite magmas, but also the age of their emplacement (Heaman, 1989; Li et al., 2010b; Mitchell, 1986).

There have been several attempts to date the emplacement age of the Mengyin kimberlite. A Rb–Sr isotopic analysis of phlogopite yielded an isochron age of  $475 \pm 3$  Ma (Dobbs et al., 1994), broadly consistent with our single-grain phlogopite Rb–Sr result of  $485 \pm 4$  Ma in this study. However, the initial <sup>87</sup>Sr/<sup>86</sup>Sr ( $I_{Sr}$ ) of the analyzed phlogopites lie between 0.704 and 0.714 (Dobbs et al., 1994; this study), variably higher than that of perovskite (0.7037 from Yang et al., 2009), suggesting a significant crustal contamination and/or later alteration. In addition to the Rb–Sr technique, Ar–Ar analyses of phlogopite were also used to date kimberlite, which yielded an age of  $465 \pm 3$  Ma for the Mengyin kimberlite (Zhang and Yang, 2007), ~15 Ma younger than the result of the Rb–Sr isochron. The Ar–Ar age spectrum, however, indicates mixture of different mineral phases and/or partial Ar loss (Zhang and Yang, 2007).

Perovskite is considered as a good mineral for dating kimberlite because it has high U content, occurs mainly in the kimberlite groundmass and is considered to have crystallized during the early stages of the magmatic history (e.g. Batumike et al., 2008; Kinny et al., 1997; Kramers and Smith, 1983; Yang et al., 2009). Unfortunately, some



**Fig. 3.** U–Th–Pb dating results for the Mengyin perovskite.

perovskites in the Mengyin kimberlite suffered different levels of alteration, and hence variable radiogenic Pb loss (Yang et al., 2009). It is also noteworthy that perovskite usually contains a high proportion of common lead, which makes it difficult to judge the concordance of U-Pb system. In this case, only an apparent  ${}^{206}Pb/{}^{238}U$  age can be obtained. Previous  $^{238}$ U $^{-206}$ Pb perovskite ages of  $456 \pm 8$  Ma obtained by TIMS (Dobbs et al., 1994) and  $470 \pm 4$  Ma by LA-ICPMS (Yang et al., 2009) can also be attributed to the alteration. In our work, perovskite was analyzed by ion probe which consumed only a small volume of sample on the least alterated part (judged by BSE images) of the mineral interior. Our consistent <sup>238</sup>U-<sup>206</sup>Pb and <sup>232</sup>Th-<sup>208</sup>Pb ages demonstrate that the dated Mengyin perovskites should be concordant in U-Th-Pb system (Li et al., 2010b). Therefore, perovskites U–Th–Pb age of  $480 \pm 4$  Ma is suggested as the best estimate of the emplacement age of the Mengyin kimberlite. Baddeleyites from the Mengyin kimberlite yielded a Pb-Pb age of  $480.4 \pm 3.9$  Ma, which is in good agreement with perovskite U–Th–Pb ages. As discussed below, the studied baddelevite may be inherited from the mantle source, but its U-Pb analyses can provide age estimation for kimberlite as some kimberlitic zircons (Belousova et al., 2001; Davis et al., 1976; Kinny et al., 1989).

As for the Fuxian kimberlite, while the phlogopite Ar–Ar plateau date of  $464 \pm 2$  Ma (Zhang and Yang, 2007) is consistent with previously reported Rb–Sr isochron dates of ~463 Ma (Dobbs et al., 1994; Li et al., 2005), mixture of different mineral phases and/or partial Ar loss was noticed (Zhang and Yang, 2007). In addition, the initial <sup>87</sup>Sr/<sup>86</sup>Sr values ( $I_{Sr}$ ) of 0.722 from phlogopite Rb–Sr isochron regression (Fig. 2b) is much higher than those of the mantle-derived magmas, indicating significant crustal contamination and/or later alteration. Therefore, both the Rb–Sr and Ar–Ar ages are likely to be questionable. Unfortunately, despite repeated attempts separation of perovskite from the Fuxian kimberlite was unsuccessful. However, baddeleyite from one sample yielded a Pb/Pb age of 479.6 ± 3.9 Ma. Based on the concordance of the perovskite U–Th–Pb age and the baddeleyite Pb/Pb age from the Mengyin kimberlite, this baddeleyite Pb/Pb age is interpreted as the age of emplacement of the Fuxian kimberlite.

Comparison with the aforementioned different isotopic systems suggests that phlogopite is not an ideal mineral for dating the kimberlite by Rb–Sr and Ar–Ar methods because of its complicated alteration and/ or contamination processes. Although dating baddeleyite can yield the precise crystallization age of a kimberlite, it is rarely found in kimberlites and could be xenocrystic in origin (see discussion below). Perovskite in the kimberlite groundmass is therefore thought to be the best candidate to date kimberlite, particularly using the ion probe technique that can effectively avoid alteration in some crystals (Yang et al., 2009). More importantly, simultaneous measurement of the perovskite U–Pb and Th–Pb age makes it possible to determine the concordance of U–Th–Pb system of the mineral, thus providing a robust constraint on the emplacement age of kimberlite (Li et al., 2010b).

#### 5.2. Xenocrystic origin of kimberlitic baddeleyite

Baddeleyite rarely occurs in kimberlite (Mitchell, 1986). Two distinct types of baddeleyite can be recognized from this kind of rocks (Heaman and LeCheminant, 1993 and reference therein). The first baddeleyite type occurs as a rim on zircon megacrysts or sometimes interfaces between zircon and rutile/ilmenite, which is interpreted as products formed by reaction between macrocrystal zircon and kimberlite melt undersaturated in SiO<sub>2</sub> and enriched in carbonate component. In fact, this type of baddeleyite forms fine idiomorphic crystals which are mostly oriented perpendicular to contours of zircon grains and they are often parallel to each other. Crystals show a zoned structure, but commonly have dark central parts and light marginal parts in backscattered electron images, indicating a higher U content in rim than core (Heaman and LeCheminant, 1993). The second type of baddeleyite occurs as xenocrysts, as documented in Mbuji–Mayi kimberlite and 'lle Bizard alnöite (Heaman and LeCheminant, 2000; Schärer et al., 1997). In this case, the baddeleyite was originally formed in the mantle and then picked up by the deep-sourced magma. No matter how these baddeleyites formed, this mineral has been proven to record the emplacement age of magmas as its U–Pb isotopic clock was triggered while being caught by the mantle-derived magmas (Heaman and LeCheminant, 1993, 2000; Schärer et al., 1997).

Several lines of evidence support the xenocrystic origin for baddeleyites from the Mengyin and Fuxian kimberlites. Firstly, baddelevite is very rare and can only be obtained occasionally. It is much easier to obtain crustal zircon xenocrysts than baddeleyites. We have not found any evidence showing reactions between xenocrystic zircon and kimberlite melt. Secondly, the CL images of the Mengyin and Fuxian baddeleyites show zoning, but commonly suggesting higher U content in core than rim, contrary to what formed by reaction between macrocrystic zircon and kimberlite melt (Heaman and LeCheminant, 1993). No zircon exists as residue in the core. Thirdly, the studied baddeleyite grains, although fragmental, have a grain size of >50 µm (Fig. 4), much bigger than the crystallized baddelevite from kimberlitic magma (Mitchell, 1986). Fourthly, the  $\varepsilon_{\rm Hf}$  (480 Ma) value of ca. –6 for baddeleyites is different from not only that of the kimberlite magma (-0.3 to -6, Zhang and Yang, 2007), but also the xenocrystic zircons (ca. -40, Zheng et al., 2009). This clearly indicates that baddelevites did not crystallize from the kimberlitic magma or were assimilated from crustal rocks.

In summary, the baddeleyites from the Mengyin and Fuxian kimberlites are most likely xenocrysts from the SCLM. These crystals formed either directly from a melt by metasomatism, or through subsolidus crystallization (Schärer et al., 1997) and stayed in the lithospheric mantle at high temperature with U–Pb isotopic system

remaining open. After eruption, its U–Pb systems were closed due to cooling and crystallization of kimberlitic magma. Therefore, these xenocrystic baddelevites record the emplacement age of kimberlite.

As for Hf isotope composition, with the exception of those measurements with large uncertainties, baddeleyites from Mengyin and Fuxian kimberlites show a narrow range of  $\pm 2 \varepsilon_{\rm Hf}$  units, which is close to the external precision of the analytical method (Wu et al., 2006b). This narrow range of  $\varepsilon_{\rm Hf}$  values might be consistent with crystallization from a single magma that was in equilibrium with the Hf composition of the SCLM (Griffin et al., 2000). Their Hf model age points to ~1.3 Ga, which may record a metasomatism event manifested by the widespread ~1.35 Ga diabase intrusion event in the NCC (Zhang et al., 2009).

# 5.3. Evolution of the SCLM of NCC

Based on Os isotope characteristics and high Fo number of olivine in peridotite xenoliths from Mengyin and Fuxian kimberlite, it is believed that the lithospheric mantle keel of NCC had an Archean melt-extraction age (e.g., Chu et al., 2009; Gao et al., 2002; Wu et al., 2003, 2006a; Zhang et al., 2008; Zheng et al., 2006). In contrast, geophysical observations and geochemical characteristics of peridotite xenoliths transported by Cenozoic basalts indicate the current SCLM beneath the eastern NCC is thin, hot and fertile (e.g. Griffin et al., 1998; Zheng et al., 2006). This contrast suggests that ~100 km of cratonic lithosphere had been removed between early Ordovician and Cenozoic, and the refractory and isotopically enriched SCLM had been changed to be fertile and isotopically depleted. However, the mechanisms and processes of this lithospheric thinning and mantle transformation have been hotly



Fig. 4. CL images and Pb/Pb ages of baddeleyite xenocrysts from kimberlites of Mengyin (a,b) and Fuxian (c,d).

debated during last decade. Obviously, to investigate how and when the craton was destroyed requires detailed physical–chemical knowledge of the original SCLM before destruction.

The deep-sourced magmas and trapped mantle xenoliths and xenocrysts provide critical information of both the chemical composition and evolution of the SCLM. Extensive studies have been conducted on mantle xenoliths and SCLM-derived mafic-alkaline rocks in the NCC (e.g., Chu et al., 2009; Gao et al., 2002; Huang et al., 2007; Qiu et al., 2005; Wu et al., 2003, 2006a; 2010; Xu et al., 2008; Ying et al., 2007; Zhang et al., 2008; Zheng et al., 2006, 2009). However, it is not known if the Paleozoic SCLM beneath the NCC was isotopically enriched or depleted since the intensive alteration makes impossible to obtain reliable isotopic compositions of the peridotite xenoliths in the Paleozoic kimberlites. It is noted that, despite many studies on the Mesozoic mantle-derived rocks, crustal contamination during crystallization similarly makes them unreliable in constraining the characteristics of the SCLM, and so only those SCLM-derived igneous rocks with little crustal contamination can provide reliable information. Fortunately, silica undersaturated nepheline syenite, although rare in the NCC, can provide useful data on the SCLM evolution in the NCC.

One important nepheline syenite is the Triassic (~225 Ma) Saima complex in the Liaodong Peninsula of northern China (Fig. 1). It is composed of an eastern syenite, central alkaline volcanic rocks (trachyte, leucite phonolite and syenitic porphyry), and western nepheline syenite (Wu et al., 2010 and references therein). It has been well documented that this nepheline syenite has fairly homogeneous Hf isotope composition of <sup>176</sup>Hf/<sup>177</sup>Hf = ~0.282330, corresponding to  $\varepsilon_{\rm Hf}$  (t) ~ - 11 (Wu et al., 2010). Another nepheline syenite is the Cretaceous (~127 Ma) Zijinshan complex, located in western NCC where a thick



**Fig. 5.** Hf isotope compositions of baddeleyite xenocrysts from kimberlites of Mengyin (a) and Fuxian (b). The grey line in (a) is from all the Hf isotope analyses from baddeleyites of Mengyin kimberlite, and dark line is from those Hf isotope data with internal uncertainty less than 0.00005 ( $2\sigma_m$ ).



**Fig. 6.** Hf isotope evolution diagram of the SCLM beneath the NCC before destruction. Data source: Baddeleyite xenocrysts in the Paleozoic kimberlites (this study); Saima nepheline syenite (Wu et al., 2010); Zijinshan monzonite (Ying et al., 2007); Cenozoic SCLM in eastern NCC (Chu et al., 2009; Qiu et al., 2005).

lithospheric keel is preserved (Fig. 1). This nepheline syenite includes monzonite in its outermost part and pseudoleucite phonolitic breccia in the center. On the basis of geochemistry the monzonite has been interpreted as mixing of lithospheric mantle-derived magma with lower-crust derived melts (Ying et al., 2007). The end member of the SCLM-derived magma has a <sup>176</sup>Hf/<sup>177</sup>Hf isotopic ratio of ~0.282335, corresponding to  $\varepsilon_{\rm Hf}(t) \sim -13$ .

When all the data discussed above are plotted together (Fig. 6), it can be seen that the Hf isotopic composition of the SCLM beneath the NCC evolved in a linear trend which intersects the depleted mantle at ~1.3 Ga. This age may record a metasomatism event and it is further supported by the widespread ~1.35 Ga diabase intrusion event in the NCC (Zhang et al., 2009). This trend can be used to determine the Hf isotopic nature of the SCLM before destruction. Interestingly, the mantle xenoliths hosted in the Cenozoic basalts in eastern NCC have much higher Hf isotopic ratios (Chu et al., 2009; Qiu et al., 2005), indicating that the SCLM in the Cenozoic is juvenile, not an ancient residue. Understanding how and when this change happened calls for further Hf isotope investigations on more SCLM-derived materials to compare with the constructed Hf isotope evolution trend of the SCLM before the craton was destroyed.

#### 6. Conclusions

We precisely determine the emplacement age of diamondiferous kimberlites from Mengyin and Fuxian in the NCC, using three different dating methods. For the Mengyin kimberlite, single-grain phlogopite Rb-Sr isochron yielded an age of  $485 \pm 4$  Ma. U-Th-Pb analyses on perovskite gave  $^{238}$ U- $^{206}$ Pb age of 480.6  $\pm$  2.9 Ma and  $^{232}$ Th- $^{208}$ Pb age of  $478.9 \pm 3.9$  Ma. The baddeleyites from the Mengyin and Fuxian kimberlites yielded almost identical Pb–Pb ages of  $480.4 \pm 3.9$  Ma and  $479.6 \pm 3.9$  Ma, respectively, which also are best explained as the emplacement age of these kimberlites. The Hf isotope compositions ( $\varepsilon_{\rm Hf}$ (480 Ma) = -6) of baddelevite xenocrysts from these diamondiferous kimberlites have been used to probe the SCLM beneath the NCC. Combined with Hf isotopic data of the Meso-Cenozoic SCLM-derived rocks and mantle xenoliths, the Hf isotope evolution trend of the SCLM beneath the NCC before destruction was constructed, which revealed a ~1.3 Ga metasomatism event. Further Hf isotope investigations on more SCLM-derived materials could be used to compare with the

constructed Hf isotope evolution trend before craton destruction to determine when the lithospheric thinning occurred.

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