



Eoarchean to Paleoproterozoic crustal evolution in the North China Craton: Evidence from U-Pb and Hf-O isotopes of zircons from deep-crustal xenoliths

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Abstract

Granulite-facies xenoliths entrained within igneous rocks shed a light on poorly understood yet critical questions about age, origin and history of the lower continental crust. Here, we present the first coupled *in-situ* U-Pb, Lu-Hf and O isotope data for the Precambrian zircons from fourteen deep-crustal xenoliths from the North China craton. The geochronological data demonstrates that the oldest lower crustal remnants formed at ~3.82 Ga and related magmatism continued until ~3.55 Ga. The Eo-Paleoarchean zircons, with exception of one analysis, have subchondritic initial Hf isotopes with negative $\epsilon_{\text{Hf}}(t)$ and are characterized by normal mantle to elevated $\delta^{18}\text{O}$ values (5.37–6.90‰), indicating a development of a low Lu/Hf reservoir and hydrosphere-crust interactions in the early Earth. Magmatic zircons from lower crustal xenoliths of North China define a strongly episodic distribution of ages at 3.82–3.55 Ga, ~2.7 Ga, ~2.5 Ga and 1.95–1.85 Ga and a non-linear Hf isotope-age array for nearly 2 Gyr, indicating episodic crustal generation and reworking through time. The lower crustal xenoliths record a change in zircon O isotope compositions from a restricted range at 3.8–2.6 Ga to more variable after ~2.5 Ga. Both heavier ($\delta^{18}\text{O}$ up to 10.25‰) and lighter ($\delta^{18}\text{O}$ low to 2.16‰) O isotope compositions than the normal mantle are observed in ~2.5 Ga magmatic zircons. The secular change in zircon O isotopes documents an increase in recycling rate of surface-derived materials (including high- $\delta^{18}\text{O}$ sediments, weathered and altered rocks and low- $\delta^{18}\text{O}$ altered oceanic crust) into magma sources at the end of Archean, which, in turn, is possibly linked to modern style subduction processes and maturation of the crust at that time.

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1. INTRODUCTION

The early evolution of continental crust, particularly its lower layer, during the first 2.0 billion years of Earth

history remains enigmatic. The ancient lower crustal rocks are very scarce because of poor preservation or difficulties in accessing. The only accessible lower crustal rocks are outcropping high-grade metamorphic terrains recording lower crustal pressures (outcropped lower crust) and granulite-facies xenoliths captured by magmas (unexposed lower crust) (Rudnick and Gao, 2014). Recent studies of deep crustal xenoliths/xenocrysts show that the

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deep crust of some cratons (e.g. North China and Yangtze cratons; Zheng et al., 2004a, 2006) are significantly older than the exposures of both lower and upper crust. These unexposed ancient materials thus are valuable archive of Earth's early history.

Phanerozoic igneous rocks that carried abundant deep crustal xenoliths with Precambrian ages, varying from >3.6 Ga to 1.8 Ga, are widespread in the North China craton (NCC; Fig. 1). These xenoliths record long and complex generation and reworking of the crust (Huang et al., 2004; Zheng et al., 2004a, 2009, 2012; Zhang et al., 2012). In particular, zircon U-Pb geochronology studies on these xenoliths have revealed the presence of Paleoarchean (as older as >3.6 Ga) lower crust beneath the younger (<2.85 Ga) exposed crust in southern part of this craton (Zheng et al., 2006; Ping et al., 2018). The Precambrian xenoliths thus not only offer an opportunity for constraining the secular evolution of lower crust beneath the NCC, but also provide a window into the nature, extent, origin and fate of the early crust, and, by implication, the early mantle differentiation.

Extracting primary isotopic information from Archean rocks is critical for understanding differentiation processes and timing of rock crystallization, but is challenged by the susceptibility of parent-daughter isotope ratios in the later geological disturbances. Owing to the resistance of zircon to later metamorphic disturbances, its U-Pb, Lu-Hf and O isotope compositions are widely used to constrain the formation and evolution of early crust and mantle differentiation (e.g., Amelin et al., 2000; Wilde et al., 2001; Zheng et al., 2004a; Harrison et al., 2005; Valley et al., 2005; Wu et al., 2008; Kemp et al., 2010; Wan et al., 2013; Hiess and Bennett, 2016; Fisher and Vervoort,

2018; Ping et al., 2018). The number of coupled U-Pb and Hf-O isotope studies of Hadean to Archean zircons has increased drastically over the past decade (e.g., Hiess et al., 2009; Wang et al., 2015; Reimink et al., 2016; Vezinet et al., 2018). In contrast, zircons from the lower crustal xenoliths of the NCC has received far less attention from the scrutiny of modern analytical methods. The available U-Pb and Lu-Hf isotope data sets have shed light on the general age framework and growth history of the lower crust beneath the NCC (Zhang et al., 2012; Zheng et al., 2012; Ma et al., 2017). However, few age data of these zircons were determined by precise and reliable SIMS technique, especially for the ancient and complexly zoned zircons, which potentially obstructs accessing the accurate knowledge of their crystallization ages, and in turn, making much of uncertainty in estimation of initial Hf isotope compositions. Moreover, despite zircon O isotope has long been taken as a powerful tool for tracing hydrosphere-crust interaction (e.g. Valley et al., 2005; Vezinet et al., 2018), so far, little is known about zircon O isotopes of lower crust beneath the NCC.

In order to develop insights into generation and evolution of ancient continental crust, we conducted the first combined *in situ* U-Pb, Lu-Hf and O isotopes of Precambrian zircons from deep-crustal xenoliths in the NCC. The results show that: (1) the oldest (3.82–3.55 Ga) known lower crustal rocks were survived in the southern part of the NCC; (2) the Eo-Paleoarchean zircons have predominant sub-chondritic Hf isotope compositions and elevated $\delta^{18}\text{O}$ values, suggesting Lu-Hf fractionation and crust-hydrosphere interactions on the Earth can be traced back to Eoarchean or even earlier; (3) the xenoliths record episodic crustal generation and reworking through time and probable recycling subducted oceanic crust in the Neoproterozoic.

2. GEOLOGICAL SETTING AND SAMPLES

The NCC is one of the few places in the world where preserves ≥ 3.8 Ga upper crustal remnants (Liu et al., 1992). The Precambrian rocks record episodic growth and/or reworking of the crust in this craton at >3.0 Ga, 2.8–2.7 Ga, 2.6–2.5 Ga and 2.0–1.8 Ga (Wu et al., 2005; Geng et al., 2012; Zheng et al., 2012; Huang et al., 2013; Zhao and Zhai, 2013). Phanerozoic magmas with abundant xenoliths are widespread (Fig. 1) and provide direct samples of the deep continent. In this study we analyzed zircons from fourteen granulite, amphibolite and metagabbro xenoliths from five localities, including the Paleozoic Fuxian kimberlites, the Mesozoic Xinyang and Ningcheng igneous rocks and the Cenozoic Nvshan and Qingyuan basalts. Petrological studies on some of the studied samples, as well as other related xenoliths, have shown that the crustal xenoliths in the NCC record high equilibrium temperatures and pressures (Table 1; Huang et al., 2004; Zheng et al., 2004a, 2004b; Ma et al., 2017), suggesting they are samples of lower part of the crust. Seven xenolith samples (Xinyang samples XY9951 and XY9928, Fuxian samples LN9821, LN9857, LN9849 and LN9841, and Ningcheng samples NC11-02) were selected for zircon U-Pb and Lu-

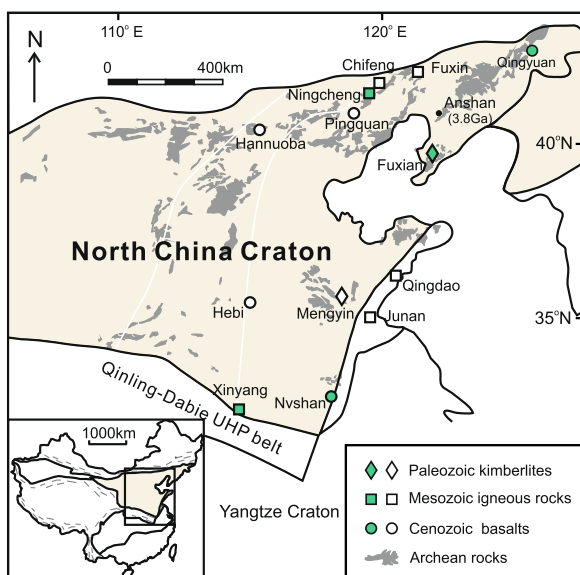


Fig. 1. Geological sketch map showing outcropping Archean rocks and localities with lower-crustal xenoliths in Phanerozoic igneous rocks in the North China craton. The sampling sites are highlighted in green. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Table 1
Summary of U-Pb ages and Hf-O isotope compositions of the studied lower crustal xenoliths in the North China Craton.

Sample	Xinyang						Qingyuan		
	XY9951	XY9928	16XY02	16XY03	16XY04	16XY01	QY1205	QY1234	
Lithology	Felsic granulite	Felsic granulite	Grt felsic granulite	Two-Py mafic granulite	Grt granulite	Felsic granulite	Felsic granulite	Two-Py felsic granulite	
Mineral assemblage	Q+Pl+Kfs+Cpx	Q+Pl+Kfs+Cpx	Q+Pl+Kfs+Grt+Cpx	Pl+Opx+Cpx+Q	Kfs+Pl+Grt+Py+Bi+Q	Q+Pl+Kfs+Cpx	Pl+Kfs+Q+Cpx	Pl+Cpx+Opx	
Pressure [#]	~30 km	~30 km							
Age (Ga)	Magmatic	~3.82/~3.78	~3.75/~3.70	~3.55	~2.48	~2.69	1.96	~2.51	~2.51
	Xenocrystic				~3.75				
E _{Hf} (t)	Magmatic	-6.6 ~ -0.8	-4.3 to -0.2	-7.6 to -4.9	-30.7 to -27.3	-2.7 to +3.2	-2.7 to -0.4	0 to +4.0	+2.1 to +7.5
	Xenocrystic				-3.7 ~ +2.3				
δ ¹⁸ O (‰)*	Magmatic	5.37–6.17	5.49–6.36	5.38–6.90	5.71–6.86	6.02–6.42	3.97–5.67	6.71–8.64	6.71–8.96
	Xenocrystic				5.94–6.73				
Sample		Fuxian			Nvshan		Ningcheng		
		LN9821	LN9857	LN9849	LN9841	11NS-71	NC11-02		
Lithology		Py amphibolite	Metagabbro	Grt granulite	Grt granulite	Two-Py felsic granulite	Two-Py mafic granulite		
Mineral assemblage		Hbl+Kfs+Pl+Py	Py+Kfs+Pl	Py+Pl+Grt	Grt+Pl+Py	Pl+Opx+Cpx	Pl+Opx+Cpx		
Pressure		7.6–8.8 kbar		>8.8 kbar	>8.8 kbar	6.2–9.5 kbar	~7.5 kbar		
Age (Ga)	Magmatic	~2.51	~2.51	~2.60	~2.50	~2.70	~1.86		
	Xenocrystic			~1.86	~1.88	1.96	~0.227		
E _{Hf} (t)	Magmatic	+2.1 to +6.6	+0.8 to +4.5	+5.1 to +6.4	+0.6 to +1.3	-4.8 to -3.0	-2.7 to +0.2		
	Xenocrystic			-11.8 to -5.1	-3.6 to -1.1	-15.4, -18.8			
δ ¹⁸ O (‰)*	Magmatic	3.46–5.86	2.72–4.61	3.34–5.44	2.25–5.47	6.68–7.28	7.93–9.63		
	Xenocrystic			8.00–10.25	7.87–9.33	7.08, 7.24			
Metamorphic									

[#] Estimated equilibration pressures are from Huang et al. (2004), Zheng et al. (2004a, 2004b) and Ma et al. (2017). It should be noted that there is no available equilibrium P-T data of the Qingyuan granulites.

* O isotopes of undisturbed grains.

Hf isotope studies by Zheng et al. (2004a, 2004b) and Ma et al. (2017). However, all but one sample (NC11-02) were previously dated using destructive LA-MC-ICP-MS technique on their zircons. So, Lu-Hf isotopes have to be analyzed in the different sites from U-Pb ages.

3. METHODS

All the analyses on zircons were conducted at Guangzhou Institute of Geochemistry, Chinese Academy of Sciences. Cathodoluminescence (CL) images of zircons were obtained prior to isotope analysis, using a Supra 55 Sapphire FE-SEM. The zircons were firstly analyzed for U-Pb isotopic compositions, and then the oxygen and Hf isotope analyses were carried out on the same sites. *In situ* U-Pb and O isotope analyses of zircon were performed on a Cameca IMS 1280-HR ion microprobe. Zircon Plesovice and Penglai were used as external standard for U-Pb dating and O isotope analyses, respectively. Detailed operating conditions, analysis procedure, and data calibration and monitoring are described by Li et al. (2009) and Yang et al. (2018). Zircon Lu-Hf isotope analyses were conducted using a Neptune Plus MC-ICP-MS in combination with a RESOLUTION M-50 laser ablation system, with a beam diameter of 45 μm . Zircon standard Penglai was used to monitor measurement procedures and data quality. All the analytical results are listed in Supplemental Table S1 and a summary is shown in Table 1.

4. RESULTS

4.1. Zircon U-Pb dating and Hf-O isotopes of Xinyang lower crustal xenoliths

4.1.1. Felsic granulite sample XY9951

The zircons from this sample are short prismatic to rounded. Most of them show core-rim structures with weak oscillatory zoned magmatic or darkgray irregular zoned recrystallized cores surrounded by unzoned metamorphic rims (Fig. 2a). The apparent $^{207}\text{Pb}/^{206}\text{Pb}$ ages of the cores and non-zoned grains range from 3.16 to 3.89 Ga and define a discordia line with upper and lower intercept ages of 3784 ± 74 Ma and 2143 ± 210 Ma, respectively (Fig. 3a), suggesting U-Pb compositions of the Eoarchean zircons were disturbed by ancient Pb loss. Two oldest concordant (degree of U-Pb discordance (disc.) < 2%; disc. = $100 \times (1 - (^{206}\text{Pb}/^{238}\text{U} \text{ age}) / (^{207}\text{Pb}/^{206}\text{Pb} \text{ age}))$) analyses yield a weighted mean $^{207}\text{Pb}/^{206}\text{Pb}$ age of 3817 ± 120 Ma, which is in agreement with the upper intercept age within the error. These zircons have subchondritic initial Hf isotope compositions (Fig. 5a) with $^{176}\text{Hf}/^{177}\text{Hf}$ (3817 Ma) of 0.280137 to 0.280300, ϵ_{Hf} (3817 Ma) of -6.6 to -0.8 and Hf depleted mantle model ages of 4.1–4.5 Ga. The $\delta^{18}\text{O}$ values are 5.4–6.8‰, 2.6–3.9‰ and 6.0–6.6‰ for the zoned magmatic, irregular zoned recrystallized and unzoned metamorphic domains, respectively (Fig. 6a).

4.1.2. Felsic granulite sample XY9928

The zircons from this sample are long prismatic to rounded. They are oscillatory or sector zoned and mostly

surrounded by narrow unzoned rims (Fig. 2b). Only one of the seventeen analyses is concordant (disc. = -1.3%) with the oldest $^{207}\text{Pb}/^{206}\text{Pb}$ age of 3748 ± 14 Ma. Other analyses are discordant and yield a discordia line with upper intercept age of 3701 ± 59 Ma (Fig. 3b). These zircons have subchondritic initial Hf isotopic compositions (Fig. 5a) with $^{176}\text{Hf}/^{177}\text{Hf}$ (3748 Ma) of 0.280248–0.280362, ϵ_{Hf} (3748 Ma) of -4.3 to -0.2 and Hf depleted mantle model ages of 4.0–4.3 Ga. All zircons yield $\delta^{18}\text{O}$ values range from 5.4‰ to 6.8‰ (Fig. 6b).

4.1.3. Grt felsic granulite sample 16XY02

The zircons from this sample are prismatic and show core-rim structures with oscillatory zoned magmatic cores surrounded by unzoned metamorphic rims (Fig. 2c). The analytical magmatic cores are discordant and yield a discordia line with upper intercept age of 3553 ± 55 Ma (Fig. 3c). Two metamorphic rims are discordant with Paleoproterozoic apparent $^{207}\text{Pb}/^{206}\text{Pb}$. The magmatic domains have subchondritic initial Hf isotopic compositions (Fig. 5a) with $^{176}\text{Hf}/^{177}\text{Hf}$ (3553 Ma) of 0.280283 to 0.280361, ϵ_{Hf} (3553 Ma) of -7.6 to -4.9 and Hf depleted mantle model ages of 4.1–4.3 Ga. All zircons yield $\delta^{18}\text{O}$ values range from 5.4‰ to 6.9‰ (Fig. 6c).

4.1.4. Two pyroxene mafic granulite sample 16XY03

The zircons from this sample are prismatic and show core-rim or core-mantle-rim structures (Fig. 2d). The cores show oscillatory or banded zoning. The mantle and rim are dark or bright and tend to homogeneous unzoned. Six oscillatory zoned cores yield a discordia line with an upper intercept age of 3734 ± 42 Ma which is within uncertainty of weighted mean $^{207}\text{Pb}/^{206}\text{Pb}$ age (3751 ± 14 Ma) of the two oldest concordant (disc. < 2%) analyses (Fig. 3d). The remaining seven zoned cores are concordant to slightly discordant (disc. $\leq 6\%$) with a weighted mean $^{207}\text{Pb}/^{206}\text{Pb}$ age of 2485 ± 60 Ma and an upper intercept age of 2481 ± 91 Ma (Fig. 3d). The unzoned rims record two episodic metamorphism at ~ 1.85 Ga and ~ 1.77 Ga.

The ~ 3.75 Ga domains have $^{176}\text{Hf}/^{177}\text{Hf}$ (3751 Ma) of 0.280262 to 0.280432 with ϵ_{Hf} (3751 Ma) of -3.7 to $+2.3$ and Hf depleted mantle model ages of 3.8–4.2 Ga. The ~ 2.48 Ga domains have $^{176}\text{Hf}/^{177}\text{Hf}$ (3751 Ma) of 0.280336 to 0.280433 with ϵ_{Hf} (3751 Ma) of -30.7 to -27.3 and meaningless Hf depleted mantle model ages of 4.7–4.9 Ga. The Paleoproterozoic metamorphic domains have initial $^{176}\text{Hf}/^{177}\text{Hf}$ of 0.280330–0.280464 with ϵ_{Hf} (t) of -47.3 to -40.5 and meaningless Hf depleted mantle model ages of 5.1–5.4 Ga. The $\delta^{18}\text{O}$ values are 5.9–6.7‰, 5.7–6.9‰ and 6.0–7.1‰ for the zircon domains of ~ 3.75 Ga, ~ 2.48 Ga and Paleoproterozoic, respectively (Fig. 6d).

4.1.5. Grt granulite sample 16XY04

The zircons from this sample are prismatic to rounded and show core-rim structures with oscillatory zoned magmatic cores surrounded by unzoned metamorphic rims (Fig. 2e). Most of the analytical magmatic cores are discordant and yield a discordia line with the upper intercept age of 2689 ± 80 Ma (Fig. 3e), which is similar to apparent $^{207}\text{Pb}/^{206}\text{Pb}$ age (2714 ± 3 Ma) of the oldest concordant (disc.

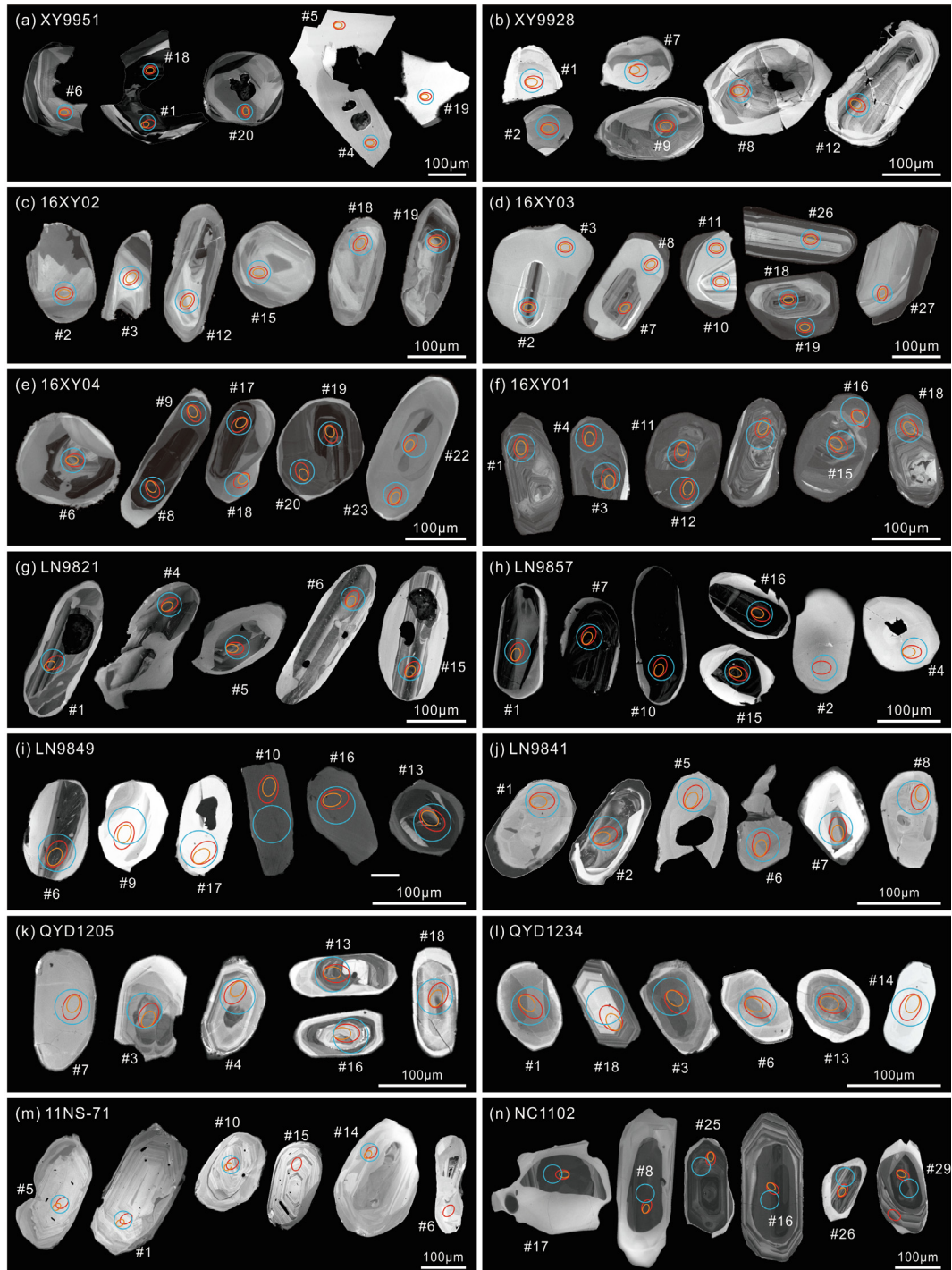


Fig. 2. Representative cathodoluminescence (CL) images of zircons from Xinyang (a–f), Fuxian (g–j), Qingyuan (k–l), Nvshan (m) and Ningcheng (n) lower crustal xenoliths in the NCC. Red ellipse, blue circle and orange ellipse denote the position of U–Pb, Lu–Hf and O isotopic analysis, respectively. The CL images of zircons from NC1102 (n) are from Ma et al. (2017). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

< 2%) analysis. The concordant to slightly concordant (disc. \leq 5%) metamorphic rims yield a weighted mean $^{207}\text{Pb}/^{206}\text{Pb}$ age of 1836 ± 64 Ma. The magmatic cores have initial $^{176}\text{Hf}/^{177}\text{Hf}$ of 0.280990–0.281154 with $\epsilon_{\text{Hf}}(t)$ of -2.7

to $+3.2$ and Hf depleted mantle model ages of 3.0–3.3 Ga. The metamorphic rims have initial $^{176}\text{Hf}/^{177}\text{Hf}$ of 0.281049–0.281278 with $\epsilon_{\text{Hf}}(t)$ of -20.1 to -12.0 and Hf depleted mantle model ages of 3.3–3.8 Ga. The $\delta^{18}\text{O}$ values

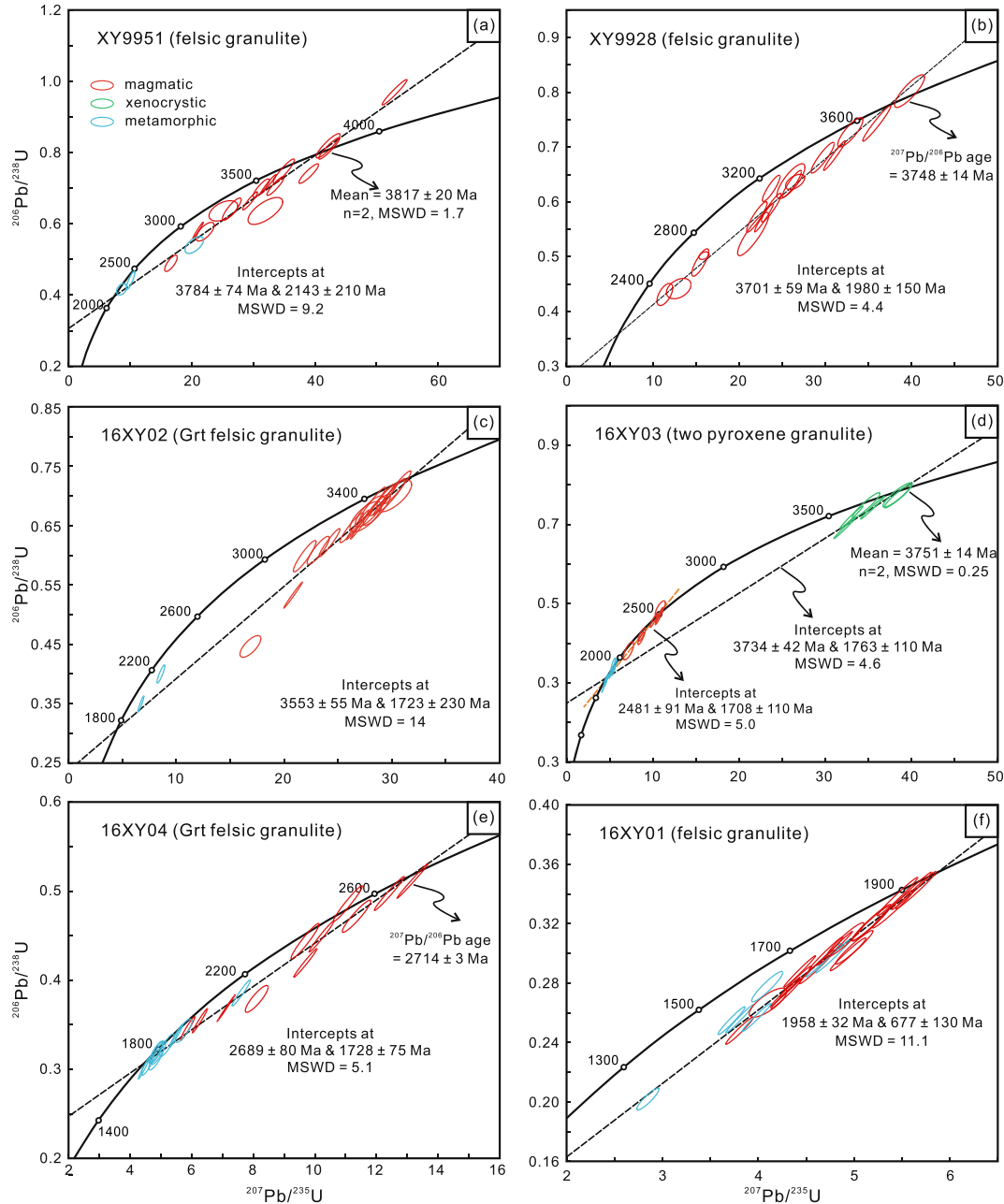


Fig. 3. U-Pb Concordia diagrams for zircons from Xinyang granulite xenoliths.

are 5.4–6.4‰ and 5.5–7.0‰ for the magmatic and metamorphic domains, respectively (Fig. 6e).

4.1.6. Felsic granulite sample 16XY01

The zircons from this sample are euhedral and oscillatory zoned with darkgray recrystallized rims (Fig. 2f). The zoned cores and recrystallized rims have identical apparent $^{207}\text{Pb}/^{206}\text{Pb}$ ages, suggesting the recrystallization occurred immediately after the magmatic zircon formed. The analytical domains yield a discordia line with upper intercept age of 1958 ± 32 Ma (Fig. 3f), which is in agreement with weighted mean $^{207}\text{Pb}/^{206}\text{Pb}$ age (1932 ± 50 Ma) of the three oldest concordant to nearly concordant (disc. < 3%) analy-

ses within errors. The magmatic and recrystallized domains, except the spot #21, have identical and restricted range of Hf isotope compositions (Fig. 5a) with $^{176}\text{Hf}/^{177}\text{Hf}$ (1958 Ma) of 0.281462–0.281526, ϵ_{Hf} (1958 Ma) of -2.7 to -0.4 and Hf depleted mantle model ages of 2.7–2.8 Ga. The $\delta^{18}\text{O}$ values range from 4.0‰ to 5.7‰ and 4.0–5.5‰ for the magmatic and recrystallized domains, respectively (Fig. 6f).

4.2. Zircon U-Pb dating and Hf-O isotopes of Fuxian lower crustal xenoliths

Zircons from the Fuxian samples are euhedral, mostly prismatic to stubby. They show core-rim structures with

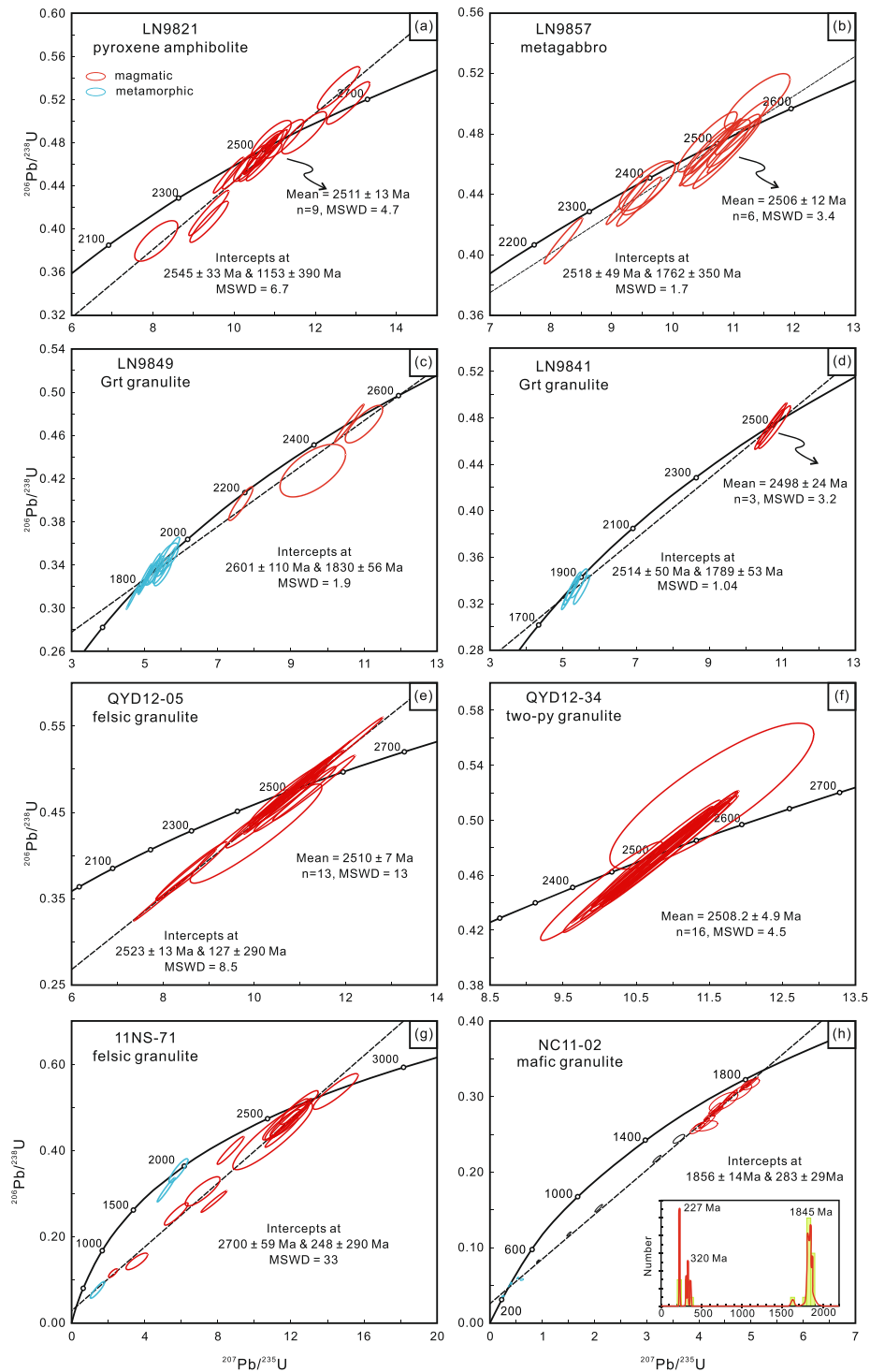


Fig. 4. U-Pb Concordia diagrams for zircons from Fuxian (a–d), Qingyuan (e–f), Nvshan (g) and Ningcheng (h) lower crustal xenoliths.

oscillatory, banded or weak zoned cores surrounded by unzoned metamorphic rims (Fig. 2g–j). Some zircons were totally recrystallized without any evidence of zoning. The zircons in the four studied Fuxian xenoliths have similar U-Pb ages and Hf-O isotopes.

4.2.1. Pyroxene amphibolite sample LN9821

Eighteen cores with typical igneous zoning were analyzed and yield an upper intercept age of 2545 ± 33 Ma, which is in agreement with weight mean $^{207}\text{Pb}/^{206}\text{Pb}$ age (2511 ± 13 Ma) of the nine concordant (disc. < 2%) analy-

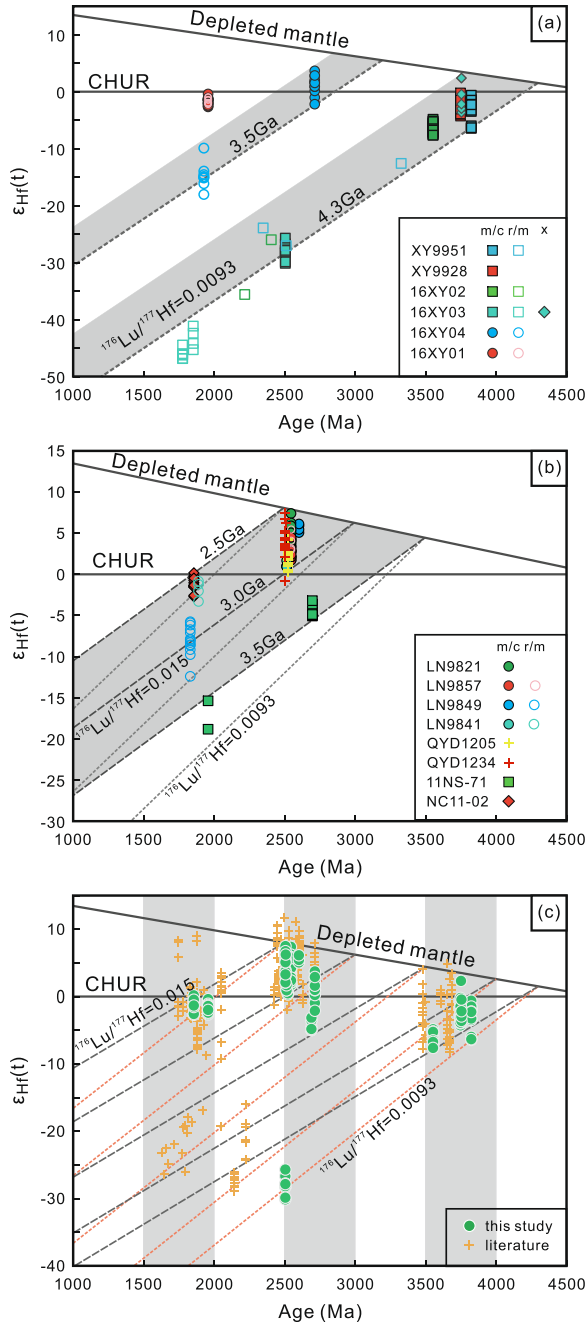


Fig. 5. Plot of zircon $\epsilon_{\text{Hf}}(t)$ versus U-Pb age for the Precambrian lower-crustal xenoliths in the NCC. (a) Xinyang xenoliths; (b) Fuxian, Qingyuan, Nvshan and Ningcheng xenoliths; (c) Precambrian zircons of igneous origin. The reworking arrays of Archean basement rocks in (a) and (b) are to adopt a source $^{176}\text{Lu}/^{177}\text{Hf}$ values of bulk continental crust (0.015; Griffin et al., 2002) or average Precambrian granitoid crust (0.0093; Vervoort and Patchett, 1996). Data on zircons from other lower-crustal xenoliths in the NCC in (c) is from a compilation by Ma et al. (2017). m/c, r/m and x represent magmatic core, metamorphic rim and xenocryst, respectively.

ses within errors (Fig. 4a). They have supra-chondritic Hf isotopes (Fig. 5b) with initial $^{176}\text{Hf}/^{177}\text{Hf}$ of 0.281239–0.281366, $\epsilon_{\text{Hf}}(t)$ of +2.1 to +6.6 and Hf depleted mantle

model ages of 2.6–2.9 Ga. The analyses yield sub-mantle $\delta^{18}\text{O}$ values range from 2.2‰ to 6.0‰ (Fig. 6g).

4.2.2. Metagabbro sample LN9857

Fifteen magmatic cores were analyzed and yield an upper intercept age of 2518 ± 49 Ma, which is in agreement with weighted mean $^{207}\text{Pb}/^{206}\text{Pb}$ age (2506 ± 12 Ma) of the six concordant (disc. < 2%) analyses (Fig. 4b). They have supra-chondritic Hf isotopes (Fig. 5b) with initial $^{176}\text{Hf}/^{177}\text{Hf}$ of 0.281207–0.281311, $\epsilon_{\text{Hf}}(t)$ of +0.8 to +4.5 and Hf depleted mantle model ages of 2.7–3.0 Ga. The analyses yield $\delta^{18}\text{O}$ values range from 2.2‰ to 4.6‰ (Fig. 6h), which are lower than both normal mantle zircon values ($5.3 \pm 0.6\text{‰}$, 2σ ; Valley et al., 2005) and most Hadean and Archean igneous zircons worldwide (5–7.5‰; Valley et al., 2005).

4.2.3. Grt granulite sample LN9849

Four analyses of magmatic cores are discordant with an upper intercept age of 2601 ± 110 Ma (Fig. 4c). They have supra-chondritic Hf isotopes (Fig. 5b) with initial $^{176}\text{Hf}/^{177}\text{Hf}$ of 0.281264–0.281294, $\epsilon_{\text{Hf}}(t)$ of +5.1 to +6.1 and Hf depleted mantle model ages of 2.71–2.78 Ga. Nine of the fourteen analyses of metamorphic domains are concordant with a weighted mean $^{207}\text{Pb}/^{206}\text{Pb}$ age of 1858 ± 42 Ma (Fig. 4c). The metamorphic domains have sub-chondritic Hf isotopes (Fig. 5b) with $^{176}\text{Hf}/^{177}\text{Hf}$ of 0.281270–0.281457, $\epsilon_{\text{Hf}}(t)$ of –11.8 to –5.1 and Hf depleted mantle model ages of 2.9–3.3 Ga. Both the magmatic and metamorphic domains yield sub-normal mantle $\delta^{18}\text{O}$ values (2.7–2.8‰ and 2.9–3.3‰, respectively; Fig. 6i).

4.2.4. Grt granulite sample LN9841

Three of the four analytical zoned cores are concordant with a weighted mean $^{207}\text{Pb}/^{206}\text{Pb}$ age of 2498 ± 24 Ma (Fig. 4d). The magmatic domains have initial $^{176}\text{Hf}/^{177}\text{Hf}$ of 0.281204–0.281225 with $\epsilon_{\text{Hf}}(t)$ of +0.6 to +1.3 and Hf depleted mantle model ages of 2.9–3.0 Ga. Three of the five analyses of metamorphic domains are concordant with a weighted mean $^{207}\text{Pb}/^{206}\text{Pb}$ age of 1877 ± 44 Ma (Fig. 4d). The metamorphic domains have sub-chondritic Hf isotopes (Fig. 5b) with initial $^{176}\text{Hf}/^{177}\text{Hf}$ of 0.281489–0.281558, $\epsilon_{\text{Hf}}(t)$ of –3.6 to –1.1 and Hf depleted mantle model ages of 2.6–2.8 Ga. Both the magmatic and metamorphic domains yield sub-normal mantle $\delta^{18}\text{O}$ values (2.9–3.0‰ and 2.6–2.8‰, respectively; Fig. 6j).

4.3. Zircon U-Pb dating and Hf-O isotopes of Qingyuan, Nvshan and Ningcheng lower crustal xenoliths

4.3.1. Qingyuan felsic granulite samples QYD1205 and QYD1234

Zircons from the Qingyuan xenoliths are euhedral and mostly show core-rim textures with oscillatory or banded zoned cores surrounded by narrow unzoned metamorphic rims (Fig. 2k–l). Recrystallized zircons without any evidence of zoning are also identified. All analyses are conducted on the cores and homogeneous grains. Both the two studied samples yield a number of concordant and near-concordant U-Pb data, with weighted mean

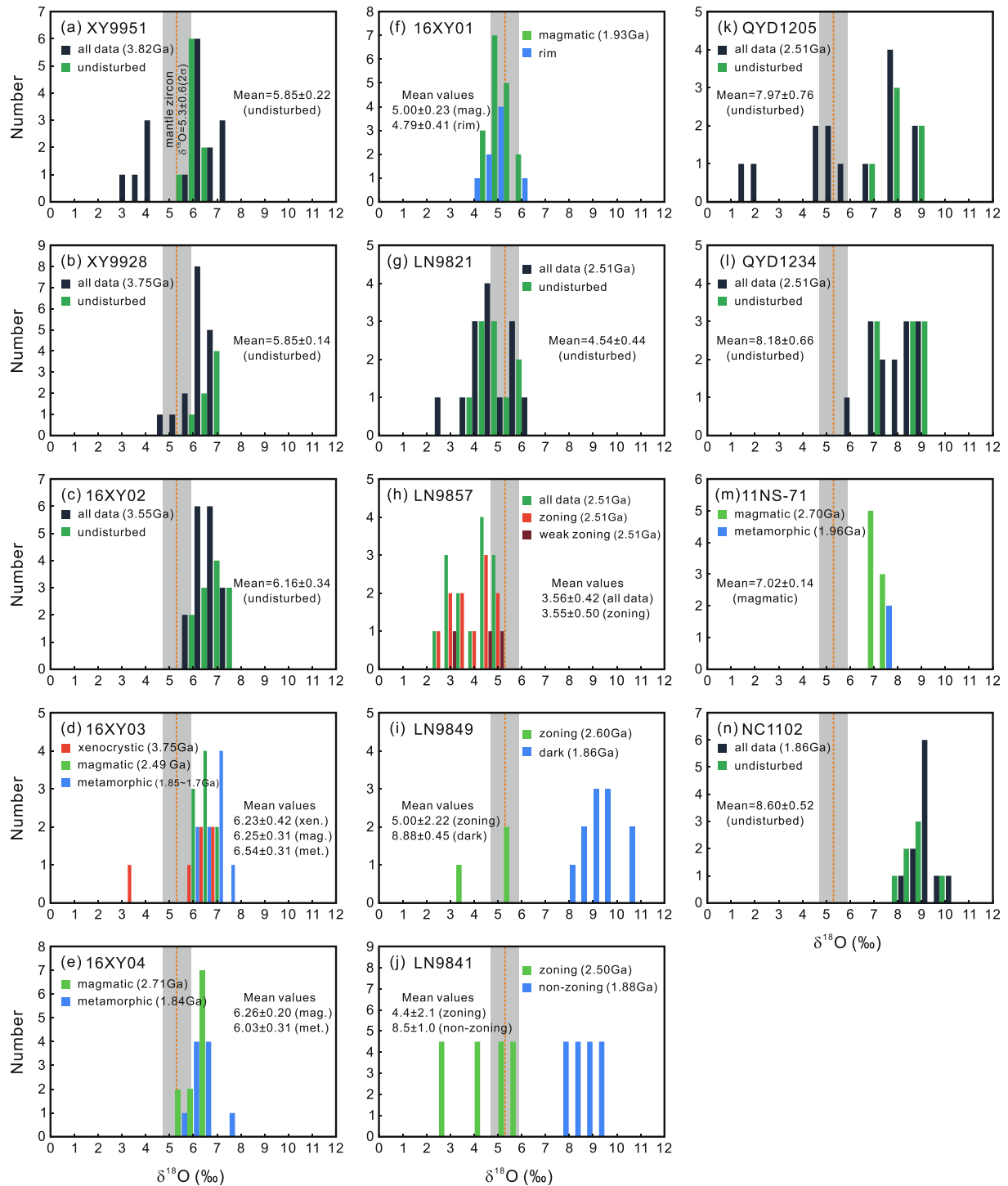


Fig. 6. Histogram of $\delta^{18}\text{O}$ values of zircon from lower crustal xenoliths in the NCC. Undisturbed data in (a, b, c, k, i and n) are from low-U, concordant to slightly discordant zircons/ domains (see text for discussion).

$^{207}\text{Pb}/^{206}\text{Pb}$ ages of 2510 ± 7 Ma ($n = 13$) and 2508 ± 5 Ma ($n = 16$) (Fig. 4e–f). The zircons have chondritic to supra-chondritic Hf isotopes (Fig. 5b) with initial $^{176}\text{Hf}/^{177}\text{Hf}$ of 0.281182–0.281394, $\varepsilon_{\text{Hf}}(t)$ of 0 to +7.5 and Hf depleted mantle model ages of 2.6–3.0 Ga. Although $\delta^{18}\text{O}$ values of the two samples are slight different (1.2–8.6‰ and 5.7–9.0‰ for QYD1205 and QYD1234, respectively), the

analyses on zircons with low U contents (≤ 500 ppm) yield identical $\delta^{18}\text{O}$ values (6.7–8.6‰ and 6.7–9.0‰ for QYD1205 and QYD1234, respectively) (Fig. 6k–l).

4.3.2. Nvshan felsic granulite sample 11NS-71

Zircons from this sample are prismatic and show core-rim structures with oscillatory zoned magmatic cores

surrounded by unzoned metamorphic rims (Fig. 2m). The analytical magmatic cores yield a discordia line with upper intercept age of 2700 ± 59 Ma (Fig. 4g). They have chondritic initial Hf isotopes (Fig. 5b) with $^{176}\text{Hf}/^{177}\text{Hf}$ (2700 Ma) of 0.280922–0.280974, $\varepsilon_{\text{Hf}}(2700 \text{ Ma})$ of -4.8 to -3.0 and Hf depleted mantle model ages of 3.4–3.5 Ga. Three metamorphic rims were analyzed and one of them is concordant with apparent $^{207}\text{Pb}/^{206}\text{Pb}$ age of 1958 ± 30 Ma. Two of them have initial $^{176}\text{Hf}/^{177}\text{Hf}$ (1958 Ma) of 0.281008 and 0.281105 with $\varepsilon_{\text{Hf}}(1958 \text{ Ma})$ of -18.8 and -15.4 and Hf depleted mantle model ages of 3.6 and 3.8 Ga. The magmatic and metamorphic domains yield identical supra-normal mantle $\delta^{18}\text{O}$ values (6.7–7.3‰ and 7.1–7.2‰, respectively; Fig. 6m).

4.3.3. Ningcheng mafic granulite sample NC11-02

Zircons from this mafic xenoliths show core-rim or core-mantle-rim structures (Fig. 2n; Ma et al., 2017). The oscillatory zoned magmatic cores yield an upper intercept U-Pb age of ~ 1856 Ma and they are mostly surrounding by metamorphic rims of late Triassic age (Fig. 4h; Ma et al., 2017). Here, we focus only on the Paleoproterozoic cores. They have chondrite-like Hf isotopic compositions with initial $^{176}\text{Hf}/^{177}\text{Hf}$ of 0.281526 to 0.281607 and $\varepsilon_{\text{Hf}}(t)$ of -2.7 to $+0.2$ (Fig. 5b; Ma et al., 2017), and yield $\delta^{18}\text{O}$ values range from 7.9‰ to 9.6‰ (Fig. 6n).

5. DISCUSSION

5.1. Initial Hf and original O isotope compositions of zircons

Extracting primary Hf-O isotopic information from zircon is a necessary requirement for constraining the formation and evolution of continental crust. Many studied xenoliths have a large apparent $^{207}\text{Pb}/^{206}\text{Pb}$ age spread for zircons from individual sample (Table S1 and Figs. 3 and 4), suggesting the U-Pb compositions of these zircons were disturbed by Pb loss. In this case, weighted mean $^{207}\text{Pb}/^{206}\text{Pb}$ age of the oldest concordant analyses and/or upper intercept value can be taken as the best estimate of the crystallization age of the zircons. It is important to further assess whether the Hf-O record is robust and meaningful. The measured $^{176}\text{Hf}/^{177}\text{Hf}$ ratios of magmatic domains from a single sample are similar (Table S1), suggesting that resetting of U-Pb age by recrystallization and Pb loss did not affect Hf isotope composition. For example, the zircons from Xinyang granulite sample XY9928 yield $^{207}\text{Pb}/^{206}\text{Pb}$ apparent ages range from 3748 Ma to 2757 Ma and define a discordia line with upper intercept age of 3701 ± 59 Ma (Fig. 3b). While these domains display ~ 1 Ga range of apparent ages, their measured $^{176}\text{Hf}/^{177}\text{Hf}$ ratio are broadly similar (0.28028–0.28039; Fig. S1). In general, the initial ε_{Hf} value for zircon is highly sensitive to the assigned age in calculation (Vervoort and Kemp, 2016). If initial Hf isotope is calculated at the measured apparent $^{207}\text{Pb}/^{206}\text{Pb}$ age, zircons from sample XY9928 yield spurious initial ε_{Hf} values, ranging over 24 ε_{Hf} units, and define a steep linear trend equivalent to derivation of a source with a $^{176}\text{Lu}/^{177}\text{Hf} = 0$ in ε_{Hf} -time space (Fig. S1). Such a trend with $^{176}\text{Lu}/^{177}\text{Hf} = 0$ is a characteristic of recrystallized zircons

with variable apparent ages and unified Hf isotope compositions, which is caused by very low Lu/Hf ratios in zircons. In contrast, a limited spread of ~ 4 ε_{Hf} units is obtained when initial Hf isotope is calculated at the crystallization age (Figs. S1 and 5a), and this result accordingly is considered as the reliable estimate of Hf isotopes first incorporated in the zircons.

Zircon has long been thought to retain its original oxygen isotopic composition even while subjected to subsequent magmatic reworking or high-grade metamorphism (Valley et al., 2005). However, the dissolution-reprecipitation, a process commonly occurring during metamictization, resets the O isotopic systems within altered zircon domains (Booth et al., 2005; Wan et al., 2013; Wang et al., 2015). Because metamictization generally results in radiogenic Pb loss and expansion of the U-rich domains, we can assess whether primary O isotope compositions have been preserved using the combination of U-Pb age, U content and O isotope (Figs. 7 and S2). Magmatic zircon domains from most studied xenoliths are characterized by homogeneous O isotopes without any correlation to discordance of age or U content (Fig. 7c and S2). The primary O isotopic signatures are thought to have been preserved in these domains. For zircons from samples XY9951, 16XY01 and QYD1205, there is a strong correlation between $\delta^{18}\text{O}$ value, degree of U-Pb discordance and U content for a single generation domains (Fig. 7a, b and S2). It appears that magmatic zircons with low U-Pb discordance ($< 10\%$) can still retain their primary O isotope compositions, while the domains with high U-Pb discordance ($> 10\%$) and high U contents have different O isotope compositions from the undisturbed domains. The deviation from the primary signature could be attributed to later alteration by hydrothermal fluids. There are some recrystallized cores with band zoning in sample XY9951. They have variable U contents and yield similar U-Pb ages within uncertainty of the crystallization age, suggesting recrystallization occurred immediately after the formation of magmatic zircon. The $\delta^{18}\text{O}$ values of the recrystallized domains are systematically lower than those of the oscillatory zoned magmatic domains from the same sample, irrespective of the discordance degree and U content. In order to avoid the potential effect by later hydrothermal alteration, we therefore used only the low-U, concordant to slightly discordant (disc. $< 5\%$; a more conservative threshold than 10%) magmatic zircons from all xenoliths to extract the primary O isotopic information at the time of the zircon crystallized from magma. It is noteworthy that the filter here just extracts the grains that are most likely to preserve their O isotope signature, but does not necessarily imply that all screened-out analyses show spurious O isotope compositions.

5.2. Eoarchean crust in southern part of the NCC

Crustal rocks older than 3.8 Ga provide direct evidence for the processes of early Earth but are limited to a few cratons (Van Kranendonk et al., 2019). Such ancient rocks are known to have survived in the northern part of the NCC (Liu et al., 1992; Fig. 1). Previous LA-ICP-MS U-Pb anal-

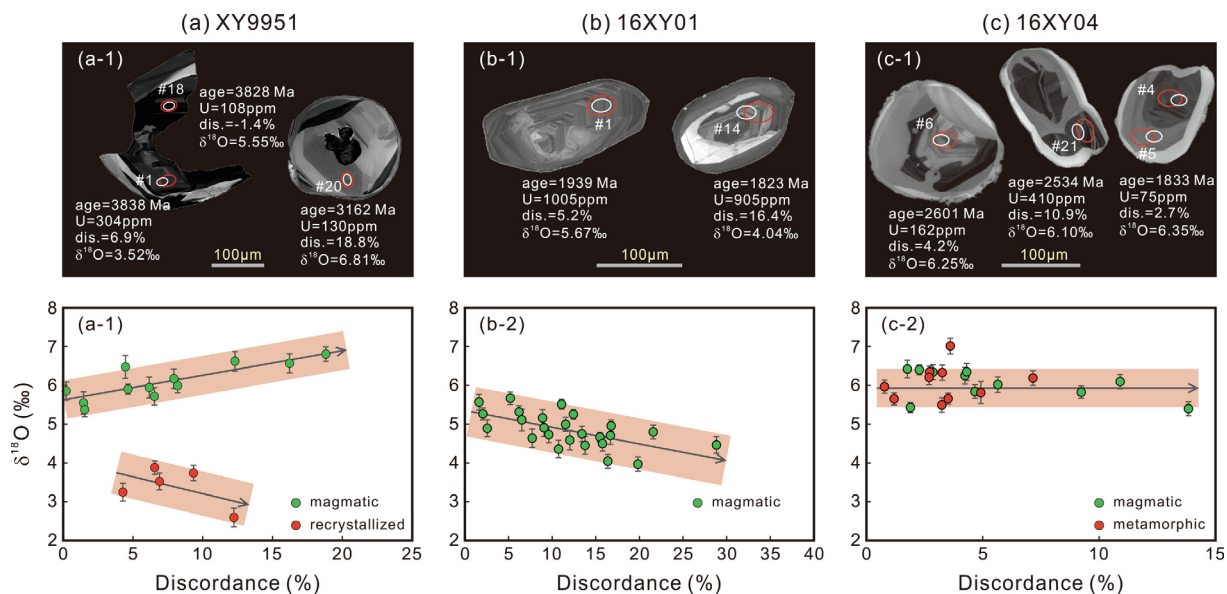


Fig. 7. Representative paired analyses and plots of $\delta^{18}\text{O}$ value versus degree of U-Pb discordance for zircons from XY9951, 16XY01 and 16XY04. Red and white ellipses in CL images denote the U-Pb and O isotopic analytical positions, respectively. The subplot a-1, b-1 and c-1 highlight the identification numbers with apparent $^{207}\text{Pb}/^{206}\text{Pb}$ age, U content, degree of U-Pb discordance (disc.) and O isotope value. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

yses of zircons from Xinyang lower crustal xenoliths have identified remnants of ≥ 3.6 Ga crust at the southern part of this craton (Zheng et al., 2004; Ping et al., 2018). However, all these data are discordant, and the extrapolated crystallized ages therefore have large errors ($\sigma > 100$ Ma, and MSWD up to 146). Zircons from samples XY9951 and XY9928, which were previously analyzed by Zheng et al. (2004) and recorded >3.6 Ga upper intercept ages, were re-analyzed by more precise and reliable SIMS technique in this study. Only a small amount of analyses are concordant (disc. $< 2\%$) and their apparent $^{207}\text{Pb}/^{206}\text{Pb}$ ages are in agreement with the upper intercept ages within the errors. Therefore, the weighted mean $^{207}\text{Pb}/^{206}\text{Pb}$ ages of concordant zircons (3.82 Ga and 3.75 Ga for XY9951 and XY9928, respectively) are interpreted to represent the emplacement time of their igneous precursors. All the analytical results on magmatic domains of 16XY02 are discordant (disc. = 2.6–28.5%) and yield a discordia line with upper intercept age of 3553 ± 55 Ma. There are two generations of magmatic cores of zircons from 16XY03, which can be explained by two plausible scenarios. One explanation is that the Paleoproterozoic magmatic zircon has been partly recrystallized at ~ 2.48 Ga. Alternatively, the protolith of this sample was emplaced at ~ 2.48 Ga, and contains inherited/xenocrystic zircons. Here, we recommend the latter option because both groups show oscillatory or banded zoning. The older population records apparent $^{207}\text{Pb}/^{206}\text{Pb}$ ages older than 3.6 Ga, and two oldest concordant (disc. $< 2\%$) analyses yield a weighted mean $^{207}\text{Pb}/^{206}\text{Pb}$ age of 3751 ± 14 Ma, which is within the uncertainty of the upper intercept age. Collectively, the U-Pb dating results of this study convincingly show the existence of Eo-Paleoproterozoic (3.82–3.55 Ga) continental crust in southern NCC.

All the Eo-Paleoproterozoic zircons, with exception of spot #2 from 16XY03, have subchondritic initial Hf isotopes with negative $\varepsilon_{\text{Hf}}(t)$ (Fig. 5a). The unradiogenic zircon Hf isotopes reflect either contamination of the original magma by older crustal materials, or the magma were derived from an evolved source with low Lu/Hf. Both two scenarios indicate that pre-existing crustal component was involved in generation of the protoliths of the Xinyang xenoliths, though no crustal component older than 3.82 Ga was survived in this area. Age of this inaccessible ancient component is roughly constrained by Hf model age of Eoarchean zircons from the oldest xenolith XY9951. The calculation of Hf model ages were based on the assumption that zircons crystallized from a crustal source with $^{176}\text{Hf}/^{177}\text{Hf}$ identical to bulk continental crust (0.015; Griffin et al., 2002), mafic crust (0.025; Payne et al., 2016) or average Precambrian granitoid crust (0.0093; Vervoort and Patchett, 1996). Chondritic model ages (expressed as $T_{\text{CHUR}}^{\text{C}}$, $T_{\text{CHUR}}^{\text{M}}$ or $T_{\text{CHUR}}^{\text{G}}$ depended on the assumed crustal source) and depleted mantle model ages (expressed as T_{DM}^{C} , T_{DM}^{M} or T_{DM}^{G}) are calculated at the extraction time of a nominal new crust from chondritic and depleted mantle compositions, respectively. It is necessary to note that model ages are not true ages in the strict geochronological sense. Moreover, the depleted mantle evolution reference line, which is projecting modern radiogenic MORB back to chondritic at 4.5 Ga, produces a bias towards artificially older depleted mantle model ages. The magmatic zircons from XY9951 have chondritic model ages range from 3.85 Ga to 4.36 Ga ($T_{\text{CHUR}}^{\text{C}} = 3.88\text{--}4.36$ Ga; $T_{\text{CHUR}}^{\text{G}} = 3.87\text{--}4.23$ Ga), which are younger than their depleted mantle model ages ($T_{\text{DM}}^{\text{C}} = 4.09\text{--}4.46$ Ga; $T_{\text{DM}}^{\text{G}} = 3.98\text{--}4.22$ Ga). These results indicate that the protoliths of XY9951, the oldest lower crustal rocks in North China,

were probably extracted from the mantle in Hadean to early Eoarchean time, perhaps as old as 3.9–4.3 Ga.

5.3. Secular evolution of crust beneath the NCC

The most salient feature of U-Pb-Hf isotopes of magmatic zircons from the studied xenoliths is that there are several magmatic episodes at >3.5 Ga, ~2.7 Ga, ~2.5 Ga and 1.95–1.85 Ga (Fig. 5c), corresponding to a secular evolution of the crust beneath the NCC, which involved episodic crustal generation and/or reworking of the continental crust.

1. *Reworking of Eoarchean crust.* The ~3.55 Ga zircons from 16XY02 and the ~2.5 Ga zircons from 16XY03 have unradiogenic Hf-isotope compositions, falling along a “reworking array” of the Eoarchean Xinyang crust (Fig. 5). This observation indicates that the earliest crust that formed before ~3.8 Ga was subsequently reworked at ~3.55 Ga and ~2.5 Ga following a low Lu/Hf crustal evolution.
2. *Generation and reworking of crust in the Neoproterozoic.* The age distribution of the analyzed magmatic zircons shows a Neoproterozoic peak, coincident with a widespread distribution of magmatic rocks in outcrop in the North China (Zhao and Zhai, 2013; Huang et al., 2013). The ~2.7 Ga magmatic zircons from two studied felsic xenoliths have subchondritic to near-chondritic Hf isotope compositions (Fig. 5) with $\epsilon_{\text{Hf}}(t)$ of -4.8 to $+3.2$ and depleted mantle Hf model ages of 2.95–3.49 Ga, indicating the ~2.7 Ga xenoliths may represent either results of reworking of pre-existing (3.5–3.0 Ga) crust or a mixing source of juvenile and ancient crust. In contrast, the ~2.5 Ga magmatic zircons from Fuxian and Qingyuan lower crustal xenoliths are characterized by chondritic to depleted Hf isotope compositions (Fig. 5). Some of them have radiogenic Hf isotopes similar to contemporary depleted mantle with depleted mantle model ages closed to the crystallization ages, indicative of juvenile crust generation. Therefore, our data provide evidence that ~2.5 Ga was a period of major crustal growth in the North China, at least in the lower crustal level.
3. *Reworking of Archean crust in the Paleoproterozoic.* Lower crustal xenoliths from the North China contain significant amounts of Paleoproterozoic zircons. Most of them occur as metamorphic rims surrounding Archean domains and have unradiogenic Hf isotopes that are consistent with those of zircons from the Archean crust. These data suggest that lower crustal rocks of the North China were subjected to Paleoproterozoic metamorphism, probably related to the assembly and breakup of the Columbia supercontinent. Magmatic zircons with Paleoproterozoic age were identified from granulite xenolith 16XY01 and NC11-02. Hf isotopes of these zircons are chondritic to slightly subchondritic ($\epsilon_{\text{Hf}} = -2.7$ to $+0.2$) with Neoproterozoic Hf model ages, suggesting reworking of the Archean crust (probably with involvement of some addition of juvenile materials).

In summary, magmatic zircons from the studied lower crustal xenoliths record broadly near-chondritic initial Hf

isotopes for nearly 2 Gyr evolution of the crust beneath the NCC. These zircons define a strongly episodic distribution of ages and a non-linear Hf isotope-age array from ~3.82 Ga to ~1.85 Ga. Combining previous studies on the exposed igneous rocks (Wu et al., 2005; Geng et al., 2012; Huang et al., 2013; Zhao and Zhai, 2013), these data reveal episodic crustal generation and reworking in the North China through time: (1) generation of new continental crust occurred as early as Eoarchean and reached its climax in the Neoproterozoic; (2) reworking of pre-existing continental crust occurred in four major episodes at >3.5 Ga, ~2.7 Ga, ~2.5 Ga and 1.95–1.85 Ga.

5.4. Implications for Eoarchean crustal evolution

U-Pb age results from this study reveal the presence of 3.82–3.55 Ga crustal fragments in southern NCC. To address the issues concerning early crustal growth and mantle differentiation, we compared Xinyang Eo-Paleoarchean magmatic zircons with other >3.55 Ga terrestrial zircon grains around the world. As shown in Fig. 8, in which only concordant to slight discordant ($\text{disc} \leq 5\%$) zircons are considered, the Eoarchean magmatic zircons are distinct from the Hadean detrital zircons in having much radiometric Hf isotopes. This temporal variation in zircon Hf isotopes probably suggests a limited involvement of evolved Hadean crustal component (or inferred early enriched reservoir) in the source of Eoarchean magmas. One possibility is that the Hadean crust, if it was voluminous, was effectively recycled back into the mantle in the Hadean to early Eoarchean and it thus was short-lived and did not survive for a long time. The Eoarchean zircons, with the exception of those from the North China, are characterized by subchondritic to slightly depleted Hf isotope compositions, suggesting the rocks were dominantly differentiated from

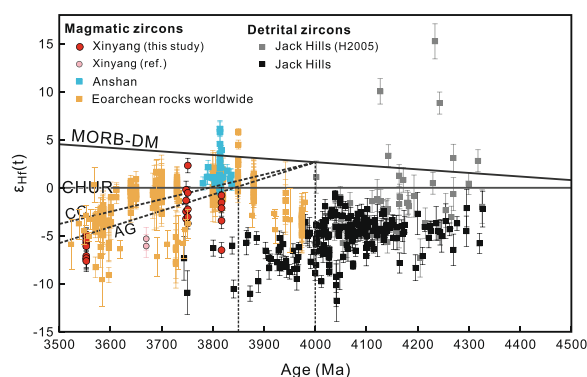


Fig. 8. $\epsilon_{\text{Hf}}(t)$ versus U-Pb age diagram of magmatic zircons from Eo-Paleoarchean rocks and detrital zircons from Jack Hills. Only concordant to slight discordant ($\text{disc} \leq 5\%$) zircons are shown. Data sources: Xinyang in southern NCC, Zheng et al. (2004), Ping et al. (2018) and this study; Anshan in northern NCC, Wu et al. (2008) and Wang et al. (2015); Eoarchean magmatic rocks worldwide, Amelin et al. (2000), Iizuka et al. (2009), Choi et al. (2006), Hiess et al. (2009), Bauer et al. (2017) and Fisher and Vervoort (2018); Jack Hills, Harrison et al. (2005) (gray square) and Kemp et al. (2010) and Harrison et al. (2008) (black square).

a chondritic-like source reservoir (Hiess and Bennett, 2016; Bauer et al., 2017; Fisher and Vervoort, 2018; Vezinet et al., 2018). Eoarchean zircons from Xinyang and Anshan in the North China show a broad range of $\epsilon_{\text{Hf}}(t)$ values varying from close to depleted mantle (Anshan) to strongly negative deviations from CHUR (Xinyang) (Fig. 8). These data indicate the development of both depleted mantle-like (high Lu/Hf) and enriched crust-like (low Lu/Hf) reservoirs before ~ 3.85 Ga.

The Eo-Paleoarchean magmatic zircons record not only the early crust-mantle differentiation, but also the early Earth hydrosphere-crust interactions. The $\delta^{18}\text{O}$ values (5.4–6.9‰) of the least disturbed and undisturbed domains within these zircons are either within or above the normal mantle range ($5.3 \pm 0.6\text{‰}$; Valley et al., 2005), similar to the Archean zircons worldwide (Figs. 6 and 9). Although $\delta^{18}\text{O}$ values of a melt can increase through fractional crystallization, zircons crystallized from such melt usually have a narrow $\delta^{18}\text{O}$ range that is insensitive to magmatic differentiation due to a concomitant increase in zircon/melt $\delta^{18}\text{O}$ fractionation (Valley, 2003; Bucholz et al., 2017). The zircons with $\delta^{18}\text{O}$ values above normal mantle range (up to $6.90 \pm 0.13\text{‰}$) reflect melting and/or contamination of near-surface rocks that experienced low temperature hydrothermal alteration (Valley et al., 2005). As we dis-

cussed previously, unradiogenic Hf isotopes of these zircons required involvement of older crustal component in generation of protoliths of the Xinyang xenoliths. The combined zircon Hf-O isotopes therefore suggest a model whereby Eo-Paleoarchean rocks were derived from a source that extracted from the mantle in Hadean to early Eoarchean and experienced interaction with low-T hydrothermal fluids, perhaps the surface water, before melting.

Elevated values of $\delta^{18}\text{O}$ have been observed in Hadean detrital zircons from Jack Hill (e.g. Wilde et al., 2001; Trail et al., 2007; Harrison et al., 2008, 2017) and southern NCC (Zhang et al., 2014, 2016). Recent studies on Acasta, Itsaq and Saglek complexes in North Atlantic craton revealed that there are many Eoarchean magmatic zircons have distinctive O isotope compositions of higher or lighter $\delta^{18}\text{O}$ values than the normal mantle range (Hiess et al., 2009, 2011; Reimink et al., 2014, 2016; Vezinet et al., 2018). The data presented here for the Eoarchean rocks in the southern NCC further demonstrated that high- $\delta^{18}\text{O}$ melts might be common in the early Earth. Altogether, the Hadean-Eoarchean zircons with $\delta^{18}\text{O}$ values greater than 5.9‰ worldwide probably indicate the presence of liquid water at or near the surface and widespread hydrosphere-crust interactions in the early Earth.

5.5. Implications for non-uniformitarian change in crustal generation and geodynamics

The changing Hf-O isotopes of magmatic zircons potentially provides a window into secular change of crustal generation/reworking and geodynamics. The primary O isotopes of 3.82–2.7 Ga zircons from the lower crustal xenoliths in the North China are similar and well within the range of $\delta^{18}\text{O}$ values displayed by global magmatic Archean zircons (Fig. 9a). There is a tendency for the younger (2.5–1.8 Ga) zircons from the xenoliths to have variable O isotopes with $\delta^{18}\text{O}$ ranging from 2.2‰ to 10.3‰ (Fig. 9a). The variability of zircon $\delta^{18}\text{O}$ is a sensitive record of recycling of supracrustal lithologies. Generally, the heavier ($>5.9\text{‰}$) and lighter ($<4.7\text{‰}$) zircon O isotope compositions are related to involvement of supracrustal materials that have undergone low-T and high-T hydrothermal alteration, respectively (Valley et al., 2005). The high $\delta^{18}\text{O}$ values (up to 10.3‰) of some ~ 2.5 Ga igneous zircons from the studied xenoliths therefore confirm previous suggestion that an increase in rate of recycling of surface-derived materials, such as high- $\delta^{18}\text{O}$ sediments, weathered rocks and rocks altered at low temperature, into crustal magmas since ~ 2.5 Ga (Valley et al., 2005). It should be noted that an enlarge of oxygen isotopes at 3.2 Ga was observed in the Pilbara Craton zircons (Van Kranendonk et al., 2015), which probably indicates locally increase of recycling rate of surface-derived materials in the Mesoarchean era.

There are many 2.6–2.5 Ga magmatic zircons, especially those form Fuxian mafic xenoliths, yield O isotopes much lighter than the values of nominal normal mantle and contemporary zircons worldwide (Figs. 6 and 9). These late Neoproterozoic zircons have supra-chondritic Hf isotopes with highest $\epsilon_{\text{Hf}}(t)$ values closed to the depleted mantle evolution

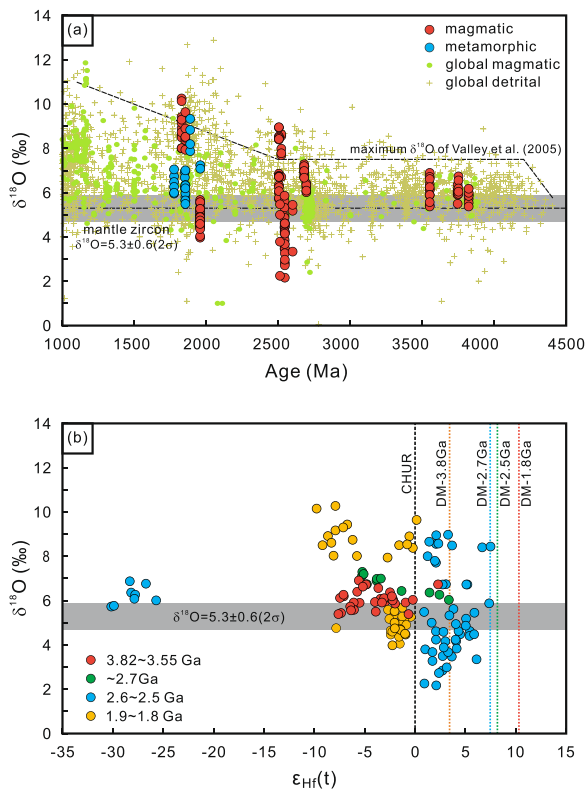


Fig. 9. Plots of zircon $\delta^{18}\text{O}$ value versus U-Pb age (a) and $\delta^{18}\text{O}$ value versus $\epsilon_{\text{Hf}}(t)$ (b) for the Precambrian lower-crustal xenoliths in the NCC. Global magmatic and detrital zircons in (a) are from the compilation of Payne et al. (2015). Only magmatic zircons and magmatic xenocrysts are shown in (b).

reference line. The lack of correlation between Hf and O isotopes of these zircons (Fig. 9b) indicates the light zircon O isotopes unlikely result from assimilation of low- $\delta^{18}\text{O}$ rocks by primary magmas. Therefore, these zircon Hf-O isotopes record a component with low $\delta^{18}\text{O}$ and high $\epsilon_{\text{Hf}}(t)$ values involved in the magma source prior to magma generation. The best candidate for this component is high-temperature altered oceanic crust, because high-temperature alteration of gabbroic lower oceanic crust often causes sub-normal mantle O isotopes but does not change primary Hf isotopes (Valley et al., 2005). This proposal has a number of important implications for growth of continental crust and geodynamics. The host rocks of Neoproterozoic low- $\delta^{18}\text{O}$ zircons are mafic and demonstrated to be derived from a mantle source (Zheng et al., 2004). Our zircon Hf-O isotope data identify altered oceanic crustal component in the mantle source of these rocks, requiring subduction processes to recycle oceanic crust back to mantle depths before magma generation at the end of Archean. Many Neoproterozoic magmatic rocks in the NCC were interpreted as products of subduction and orogeny based on petrological and geochemical observations (e.g. Huang et al., 2013; Wang et al., 2017, 2019). In the global context, modern-style plate tectonics might start at some time during the Archean eon, because many indicators for plate tectonics can be traced back at least to the late Archean (Korenaga, 2013 and references therein). The dramatic changes in composition, growth and thickness of continental crust at around 3.0 Ga is inferred to be linked to the onset of subduction-driven modern plate tectonics (e.g. Dhuime et al., 2012, 2015; Naeraa et al., 2012; Tang et al., 2016). The heavier and lighter zircon O isotopes since late Archean (Fig. 9a) reflects global increase of sedimentary and/or oceanic crustal components in the generation of magmatism, which might be associated with subduction processes. Therefore, we proposed that subduction processes may have occurred at least before the end of Archean, perhaps 3.5–3.0 Ga, and have played an important role in creating crust.

6. CONCLUSIONS

In situ U-Pb, Lu-Hf and O isotopes of zircons from lower crustal xenoliths record episodic generation and reworking in secular evolution of the crust beneath the NCC.

- (1) The Eo-Paleoproterozoic Xinyang granulite xenoliths are the oldest pieces of the crust in the southern NCC and perhaps the known oldest unexposed lower crustal rocks in the world, dating back to 3.82 ± 0.20 Ga. The Hf-O isotopes of the Xinyang 3.82–3.55 Ga magmatic zircons indicates protoliths of the granulites were derived from early formed rocks that undergone interaction with Earth's early hydrosphere at low temperature prior to partial melting. A broad range of $\epsilon_{\text{Hf}}(t)$ values varying from close to depleted mantle to strongly negative deviations from CHUR is observed in the North China Eoarchean

zircons, suggesting the development of both high Lu/Hf and complementary low Lu/Hf reservoirs before ~ 3.85 Ga.

- (2) The magmatic zircons from the studied Precambrian xenoliths define four age populations at 3.82–3.55 Ga, ~ 2.7 Ga, ~ 2.5 Ga and 1.95–1.85 Ga, and yield a non-linear Hf isotope-age array. These data reveal crustal growth in the North China occurred as early as >3.9 Ga and reached its climax in the Neoproterozoic. The pre-existing continental crust was reworked at 3.8–3.5 Ga, ~ 2.7 Ga, ~ 2.5 Ga and 1.95–1.85 Ga.
- (3) The zircons from lower crustal xenoliths record a secular change in O isotopes. An increasing range in zircon $\delta^{18}\text{O}$ values at ~ 2.5 Ga marks enhanced recycling of surface-derived materials, including high- $\delta^{18}\text{O}$ sediments, weathered and altered rocks and low- $\delta^{18}\text{O}$ altered oceanic crust. Subduction of hydrated oceanic crust back to mantle depths may have occurred at least before ~ 2.5 Ga and have played an important role in generation of continental crust.

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APPENDIX A. SUPPLEMENTARY MATERIAL

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.gca.2019.09.009>.

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