Geochronology and Hf–Fe isotopic geochemistry of the Phanerozoic mafic–ultramafic intrusions in the Damiao area, northern North China Craton: Implications for lithospheric destruction

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Timing and source of several Fe-mineralized mafic–ultramafic intrusions in the Damiao area are investigated here by coupling new geochronological and Hf–Fe isotopic data with previous results. Although regarded as a Late Paleoproterozoic assemblage previously, two ~140 Ma intrusions are recognized by zircon U–Pb dating, indicating emplacement of these intrusions from Middle Devonian to Early Cretaceous times. Both Hf and Fe isotopic features lead to the conclusion that distinct mantle components contributed to their magma generation. As the first magmatic phase, the ~395 Ma intrusions were mainly derived from the slightly-enriched SCLM that was prevalent during the Paleozoic. However, asthenospheric material was strongly involved in the formation of the ~215 Ma Gaositai intrusion. Therefore, the initiation of lithospheric destruction in the northern NCC is inferred to have occurred in Late Triassic time, triggered by post-orogenic extension following the ~250 Ma collision between the Siberian Craton and the NCC. The ~140 Ma intrusions originated from a significantly-enriched mantle component probably resided in the predominant slightly-enriched SCLM. This mantle source would have melted in the Late Mesozoic, when the thin lithosphere enabled enhanced heat transfer from the asthenosphere. In summary, these distinct mantle sources of mafic–ultramafic magmatism provide a record of mantle heterogeneity and the gradual upward migration of the lithosphere–asthenosphere boundary during lithospheric destruction.

1. Introduction

Cratons are marked by underlying lithospheric mantle keels and thought to be the most stable tectonic units on Earth (Carlson *et al.* 2005). However, multidisciplinary investigations have unravelled that the North China Craton (NCC) experienced intense Phanerozoic reactivation and its thick subcontinental lithospheric mantle (SCLM) was significantly removed (e.g., Menzies

et al. 1993; Xu 2001, 2007; Zhang *et al.* 2002; Chen *et al.* 2008, 2009a; Tian *et al.* 2009; Yan *et al.* 2010; Dong *et al.* 2013; Li and Santosh 2013). Exposed Phanerozoic igneous rocks throughout the NCC are dominantly of felsic-intermediate compositions with only minor mafic–ultramafic products, which hinder the reconstruction of deep-level processes. Consequently, several basic issues, such as the initiation timing, duration and dynamic mechanism of the destruction of the NCC, remain open to

Keywords. Mafic–ultramafic rock; mantle source region; Late Mesozoic; lithospheric destruction; Damiao; North China Craton.

debate (e.g., Wu *et al.* 2008; Xu *et al.* 2009, 2013; Zhang 2012; Zhu *et al.* 2012a, b; Ling *et al.* 2013; Zhang *et al.* 2013).

The Damiao area is well-known in China for its unique Proterozoic massif-type anorthosite, the Damiao anorthosite complex (Xie 2005). Besides this, several mafic-ultramafic intrusions (pyroxenite-hornblendite complexes) are also distributed around or within the anorthosite complex. Owing to their imprecise K-Ar ages, location within the Trans-North China Orogen and spatial relationship with the massif-type anorthosite complex, these intrusions were previously regarded as a Late Paleoproterozoic post-orogenic assemblage (e.g., Bai *et al.* 1993; Trap *et al.* 2011). But it has been indicated recently that some of them are instead Phanerozoic in age (Zhang *et al.* 2009a; Zhou *et al.* 2012; Li *et al.* 2012a, b). The mafic-ultramafic rocks in the Damiao area, as the direct magmatic expression of deep-level processes, therefore give a chance to understand the destruction of the NCC.

In this contribution, we present new U-Pb geochronological and Hf-Fe isotopic data, and integrate the previous data to discuss the timing and source region of the mafic-ultramafic magmatism in the Damiao area. Based on this, the lithospheric destruction process beneath the northern NCC is further constrained.

2. Geological setting

The northern NCC was traditionally thought to be a passive continental margin, but many researchers argue that it was intensely modified during the tectonic evolution of the Paleasian Ocean (e.g., Zhang *et al.* 2009a, b, c, 2012; Wang *et al.* 2011; Ma *et al.* 2013). The Damiao area is situated on the north-central margin of the NCC, approximately 25 km north of Chengde, Hebei Province (figure 1a). It straddles the EW-trending Jining-Longhua fracture zone (a long-lived large-scale deep structure; HBGMR 1989), which separates the Inner Mongolia paleouplift in the north from the Yanshan fold-and-thrust belt in the south (Zhang *et al.* 2009c; Wang *et al.* 2011).

The basement rocks in the Damiao area are mainly composed of Neoproterozoic to Paleoproterozoic TTG gneisses with minor mafic granulites and amphibolites. They are unconformably overlain by Meso-Neoproterozoic, Cambrian-Ordovician, Carboniferous-Triassic, Jurassic-Cretaceous and younger unmetamorphosed strata. The Damiao anorthosite complex intruded into the Precambrian metamorphic country rocks, with an exposed area of ~80 km² (figure 1b). U-Pb isotope systematics of zircon record an emplacement age of

1739 ± 14 Ma (Zhao *et al.* 2009). Together with the Miyun rapakivi granite, it partly comprises an anorthosite-mangerite-charnockite-granite suite in the ~1.85 Ga Trans-North China Orogen (Zhang *et al.* 2007; Zhao *et al.* 2009).

There are three mafic-ultramafic intrusions exposed around the Damiao anorthosite complex: The Xiahabaqin, Gaositai, and Longwangmiao intrusions. In addition, a mafic-ultramafic intrusion beneath Dawusugou was recently discovered by drilling (named Dawusugou intrusion in this paper) and a mafic-ultramafic dyke has been newly recognized near Daheishan as a result of this work (named Heishan intrusion in this paper).

3. Occurrence, petrography and mineralization

These mafic-ultramafic intrusions occur near the branches of the EW-trending Jining-Longhua fracture zone. They are characterized by small, elliptical or rounded, and crudely concentrically zoned occurrence. Only the Gaositai intrusion displays a complete transitional sequence of zoning with dunite in the core, mantled by pyroxenite and rimmed by hornblendite; other bodies consist of pyroxenite and/or hornblendite. Mineralogically, these rock types primarily consist of olivine, pyroxene, and hornblende with minor biotite and rare plagioclase as rock-forming minerals (figure 2). In addition to chalcopyrite and titanite, zircon is also associated as accessory mineral (figure 2e), which has been reported for other mafic-ultramafic rocks (e.g., Malitch *et al.* 2010; Su *et al.* 2011; Su and Leshner 2012; Kwon *et al.* 2013). Secondary minerals mainly include calcite, epidote and serpentine. In general, these intrusions share many similarities with Alaskan/Ural-type intrusions, for which the mineralization is dominated by Fe-Ti oxides and platinum group minerals (Taylor and Noble 1969; Himmelberg and Loney 1995).

In fact, the pyroxenite and hornblendite in these intrusions are Fe-mineralized extensively with a disseminated style. As shown in figure 2, rock-forming minerals (pyroxene, hornblende and minor biotite) are subhedral to euhedral in morphology, while Fe-Ti oxides are commonly anhedral and occur as interstitial grains. However, there are also some euhedral Fe-Ti oxides enclosed by pyroxene oikocrysts, suggesting a prolonged crystallization history for Fe-Ti oxides. The textures of Fe-Ti oxides are consistent with their magmatic origin. Apatite and Fe-Ti oxides are always closely associated. The Fe₂O₃ content of mineralized samples from these mafic-ultramafic

Figure 1. (a) Tectonic sketch map of China with location of the study area (modified after Zheng *et al.* 2013) and (b) simplified geological map of the Damiao area, showing distribution of the Phanerozoic mafic-ultramafic intrusions around or within the Proterozoic anorthosite complex (modified after Zhang *et al.* 2007). In (a), WB: Western Block; EB: Eastern Block; TNCO: Trans-North China Orogen; JLFZ: Jining-Longhua fracture zone; TLFZ: Tan-Lu fracture zone; DTGL: Daxing'anling-Taihangshan gravity lineament.

intrusions varies from 13 to 35%, with an average of 16.5% (Xie *et al.* 2006). Owing to the imbalance of supply and demand of iron ores, this disseminated type of Fe mineralization is actually economical in north-central China, with a minable grade of ~15% (Li *et al.* 2009a; Xie *et al.* 2009).

4. Samples and analytical methods

To enable systematic discussion, complementary analyses have been conducted for these mafic-ultramafic intrusions. Firstly, U-Pb ages (table 1) and Hf isotopic data (table 2) are reported on zircons in sample LWM-3 (Fe-mineralized pyroxenite) from the Longwangmiao intrusion and sample HS-3 (Fe-mineralized hornblendite) from the

Heishan intrusion. Secondly, Fe isotopic data (table 3) are acquired on 17 fresh samples (Fe-mineralized pyroxenite/hornblendite) from four intrusions to better constrain the magma source. The newly-obtained ages and Hf-Fe isotopes, together with the previous results, are then summarized in table 4.

4.1 U-Pb dating

Zircons were extracted using standard crushing and separation techniques. After being hand-picked under a binocular microscope, zircon grains without cracks were then set in a resin mount and polished down to about half their size. In order to observe internal textures, cathodoluminescence (CL) imaging was conducted for the

Figure 2. Photomicrographs illustrating the petrographic and mineralization characteristics of the Phanerozoic mafic-ultramafic intrusions in the Damiao area. (a) and (b) Fe-mineralized pyroxenite and biotite-bearing pyroxenite from the Dawusugou intrusion; (c) Fe-mineralized hornblende from the Gaositai intrusion; (d) and (e) Fe-mineralized pyroxenite and hornblende from the Longwangmiao intrusion, showing zircon associated with host rock; (f) Fe-mineralized hornblende from the Heishan intrusion. Except the inset in (e) taken under cross-polarized light, the other photomicrographs were taken under plane-polarized light. Mineral abbreviations: Px: pyroxene; Hbl: hornblende; Bt: biotite; Ap: apatite; Zrn: zircon.

zircons prior to *in-situ* U–Pb dating and Hf isotope analysis.

In-situ U–Pb dating was carried out by an Agilent 7500a ICP-MS coupled with a GeoLas 200M laser-ablation system at the State Key Laboratory of Continental Dynamics, Northwest University, China. A beam diameter of 30 µm was chosen for sampling. Detailed analytical procedures are similar to those reported by Liu *et al.* (2007). Standard zircon 91500 was used for calibration, and raw data

reduction and calculation were processed with the program GLITTER4.4 (Griffin *et al.* 2008). The method designed by Andersen (2002) was applied for common Pb correction. The results were finally plotted by the program Isoplot3.0 (Ludwig 2003).

4.2 Hf isotope analysis

In-situ Hf isotope analysis was carried out by a Nu Plasma HR MC-ICP-MS equipped with a GeoLas

Table 1. Zircon U-Pb dating results of sample LWM-3 from the Longwangmiao intrusion and sample HS-3 from the Heishan intrusion.

Spot no.	Content (ppm)			Isotopic ratio				Isotopic age (Ma)			
	Th	U	Th/U	$^{207}\text{Pb}/^{206}\text{Pb}$	1 σ	$^{207}\text{Pb}/^{235}\text{U}$	1 σ	$^{206}\text{Pb}/^{238}\text{U}$	1 σ	$^{207}\text{Pb}/^{235}\text{U}$	1 σ
Sample LWM-3											
LWM-3@01	9	13	0.67	0.10181	0.00880	3.46531	0.28828	0.24687	0.00577	1519	66
LWM-3@02	384	1242	0.31	0.05108	0.00301	0.15493	0.00854	0.02200	0.00035	146	8
LWM-3@03	466	814	0.57	0.05074	0.00394	0.15195	0.01123	0.02172	0.00042	144	10
LWM-3@04	335	351	0.95	0.05192	0.00241	0.31512	0.01325	0.04401	0.00061	278	10
LWM-3@05	451	247	1.83	0.05477	0.00171	0.48079	0.01218	0.06365	0.00074	399	8
LWM-3@06	207	647	0.32	0.05247	0.00199	0.22825	0.00823	0.03155	0.00036	209	7
LWM-3@07	1	33	0.02	0.05697	0.00794	0.46656	0.06304	0.05947	0.00208	389	44
LWM-3@08	89	445	0.20	0.04957	0.00297	0.15378	0.00899	0.02250	0.00029	145	8
Sample HS-3											
HS-3@01	77	167	0.46	0.11562	0.00240	5.06262	0.08901	0.31758	0.00352	1830	15
HS-3@02	924	748	1.23	0.04952	0.00258	0.15258	0.00774	0.02235	0.00026	144	7
HS-3@03	294	241	1.22	0.07126	0.00240	0.22608	0.00635	0.02300	0.00028	207	5
HS-3@04	301	310	0.97	0.12731	0.00358	0.38843	0.00821	0.02212	0.00027	333	6
HS-3@05	3450	2120	1.63	0.04977	0.00114	0.15663	0.00231	0.02282	0.00024	148	2
HS-3@06	691	1245	0.56	0.05064	0.00248	0.17390	0.00821	0.02490	0.00032	163	7
HS-3@07	217	202	1.07	0.04948	0.00383	0.15505	0.01178	0.02273	0.00033	146	10
HS-3@08	290	223	1.30	0.04827	0.00309	0.15080	0.00945	0.02266	0.00029	143	8
HS-3@09	534	1092	0.49	0.06313	0.00290	0.18639	0.00820	0.02141	0.00029	174	7
HS-3@10	266	346	0.77	0.05608	0.00370	0.17483	0.01129	0.02261	0.00030	164	10
HS-3@11	647	384	1.68	0.05390	0.00340	0.17108	0.01056	0.02302	0.00030	160	9
HS-3@12	357	483	0.74	0.04900	0.00157	0.15393	0.00406	0.02278	0.00026	145	4
HS-3@13	298	311	0.96	0.06206	0.00360	0.20000	0.01129	0.02337	0.00031	185	10
HS-3@14	719	393	1.83	0.05053	0.00161	0.15289	0.00402	0.02194	0.00025	144	4
HS-3@15	646	794	0.81	0.05727	0.00201	0.34888	0.01164	0.04418	0.00049	304	9
HS-3@16	2954	1754	1.68	0.05765	0.00306	0.18247	0.00942	0.02296	0.00028	170	8
HS-3@17	436	728	0.60	0.05333	0.00267	0.31262	0.01519	0.04251	0.00050	276	12
HS-3@18	351	286	1.23	0.05300	0.00348	0.16377	0.01053	0.02241	0.00031	154	9
HS-3@19	800	852	0.94	0.05043	0.00261	0.15600	0.00785	0.02244	0.00027	147	7

Table 2. Zircon Hf isotope analytical results of sample LWM-3 from the Longwangmiao intrusion and sample HS-3 from the Heishan intrusion.

Spot no.	$^{176}\text{Yb}/^{177}\text{Hf}$	$^{176}\text{Lu}/^{177}\text{Hf}$	$^{176}\text{Hf}/^{177}\text{Hf}$	1σ	Hf _i	1σ	$\varepsilon_{\text{Hf}}(0)$	$\varepsilon_{\text{Hf}}(t)$	$T_{\text{DM1}}(\text{Ma})$	$T_{\text{DM2}}(\text{Ma})$	$f_{\text{Lu/Hf}}$
Sample LWM-3											
LWM-3@02	0.031168	0.001365	0.282187	0.000010	0.282183	0.000010	-20.68	-17.55	1515	2302	-0.96
LWM-3@03	0.052471	0.002000	0.282213	0.000016	0.282208	0.000016	-19.78	-16.92	1505	2255	-0.94
LWM-3@08	0.031951	0.001244	0.282162	0.000009	0.282158	0.000009	-21.58	-18.53	1546	2359	-0.96
Sample HS-3											
HS-3@02	0.048090	0.001886	0.282165	0.000011	0.282160	0.000011	-21.47	-18.54	1569	2357	-0.94
HS-3@05	0.041312	0.001678	0.282185	0.000012	0.282181	0.000012	-20.76	-17.74	1531	2310	-0.95
HS-3@07	0.050202	0.001954	0.282175	0.000011	0.282170	0.000011	-21.10	-18.09	1556	2333	-0.94
HS-3@08	0.035894	0.001329	0.282197	0.000010	0.282194	0.000010	-20.33	-17.30	1500	2282	-0.96
HS-3@10	0.030059	0.001135	0.282191	0.000012	0.282188	0.000012	-20.56	-17.51	1501	2295	-0.97
HS-3@11	0.047126	0.001823	0.282164	0.000012	0.282159	0.000012	-21.50	-18.46	1567	2357	-0.95
HS-3@12	0.046488	0.002036	0.282157	0.000009	0.282152	0.000009	-21.74	-18.92	1586	2377	-0.94
HS-3@14	0.013213	0.000673	0.282141	0.000010	0.282139	0.000010	-22.32	-19.31	1552	2404	-0.98
HS-3@18	0.041890	0.001650	0.282213	0.000011	0.282208	0.000011	-19.78	-16.72	1491	2248	-0.95
HS-3@19	0.026999	0.001070	0.282176	0.000014	0.282173	0.000014	-21.07	-18.04	1519	2327	-0.97

2005 laser-ablation system at the State Key Laboratory of Continental Dynamics, Northwest University, China. A spot size of 40 μm was selected for sampling. Detailed analytical procedures have been outlined in Yuan *et al.* (2008). Standard zircon 91500 was used for the correction of mass fractionation, time-dependent drift, and interference of ^{176}Yb and ^{176}Lu . Initial $^{176}\text{Hf}/^{177}\text{Hf}$ ratios and $\varepsilon_{\text{Hf}}(t)$ values were calculated based on the ^{176}Lu decay constant of $1.867 \times 10^{-11} \text{ yr}^{-1}$ (Söderlund *et al.* 2004) and chondritic ratios of $^{176}\text{Hf}/^{177}\text{Hf}$ ($=0.282772$) and $^{176}\text{Lu}/^{177}\text{Hf}$ ($=0.0332$) proposed by Blichert-Toft and Albarède (1997). Present-day ratios of $^{176}\text{Hf}/^{177}\text{Hf}$ ($=0.28325$) and $^{176}\text{Lu}/^{177}\text{Hf}$ ($=0.0384$) reported by Griffin *et al.* (2000) were adopted for the depleted mantle to calculate single-stage model ages (T_{DM1}). Two-stage model ages (T_{DM2}) were also calculated by assuming the average continental crust with a $^{176}\text{Lu}/^{177}\text{Hf}$ ratio of 0.015 (Griffin *et al.* 2002).

4.3 Fe isotope analysis

Bulk samples for Fe isotope analysis were crushed to less than 200 mesh, digested by concentrated HF and HNO_3 mixture, and then evaporated to dryness. In order to convert the cations to chloride-form, the residues were repeatedly treated with concentrated HCl. Subsequently anion exchange chromatography was adopted for Fe separation and purification following Tang *et al.* (2006).

Fe isotopic ratios were determined at the Laboratory of Isotopic Geology, Institute of Geology, Chinese Academy of Geological Sciences using a Nu Plasma HR MC-ICP-MS in high-resolution mode. Signal of ^{53}Cr was collected simultaneously with the Fe isotopes as an interference monitor. The standard-sample bracketing technique was used for correction of instrumental mass bias. Detailed analytical and calibration procedures have been presented by Zhu *et al.* (2008). Finally, Fe isotopic results are expressed in δ notation relative to reference material IRMM-014:

$$\delta^{56}\text{Fe}_{\text{IRMM-014}} (\text{‰}) = \left[\frac{\left(\frac{^{56}\text{Fe}}{^{54}\text{Fe}} \right)_{\text{sample}}}{\left(\frac{^{56}\text{Fe}}{^{54}\text{Fe}} \right)_{\text{IRMM-014}}} - 1 \right] \times 10^3;$$

$$\delta^{57}\text{Fe}_{\text{IRMM-014}} (\text{‰}) = \left[\frac{\left(\frac{^{57}\text{Fe}}{^{54}\text{Fe}} \right)_{\text{sample}}}{\left(\frac{^{57}\text{Fe}}{^{54}\text{Fe}} \right)_{\text{IRMM-014}}} - 1 \right] \times 10^3.$$

Long-term reproducibility in high-resolution mode is better than 0.10‰ (2σ) for $\delta^{57}\text{Fe}$, and 0.08‰ (2σ) for $\delta^{56}\text{Fe}$ based on repeated determinations of national basaltic reference material CAGSR-1.

Table 3. *Fe isotope analytical results of the Phanerozoic mafic-ultramafic intrusions in the Damiao area.*

Intrusions	Sample no.	Description	$\delta^{57}\text{Fe}$ (‰)	$\delta^{56}\text{Fe}$ (‰)
Dawusugou	WSG-2	Fe-mineralized pyroxenite	0.27	0.16
	WSG-4	Fe-mineralized pyroxenite	0.20	0.09
	WSG-5	Fe-mineralized pyroxenite	0.10	0.03
	WSG-7	Fe-mineralized pyroxenite	0.12	0.06
	WSG-8	Fe-mineralized pyroxenite	0.28	0.18
Gaositai	GST-1	Fe-mineralized pyroxenite	0.03	0.01
	GST-3	Fe-mineralized pyroxenite	0.17	0.12
	GST-4	Fe-mineralized pyroxenite	0.03	−0.01
	GST-7	Fe-mineralized hornblende	0.04	0.04
	GST-9	Fe-mineralized hornblende	0.24	0.17
	GST-12	Fe-mineralized hornblende	0.22	0.15
Longwangmiao	LWM-3	Fe-mineralized pyroxenite	0.33	0.20
	LWM-4	Fe-mineralized pyroxenite	0.26	0.15
	LWM-5	Fe-mineralized pyroxenite	0.38	0.20
	LWM-9	Fe-mineralized hornblende	0.41	0.26
Heishan	HS-3	Fe-mineralized hornblende	0.32	0.22
	HS-4	Fe-mineralized hornblende	0.37	0.24

Table 4. *Summary of ages and Hf-Fe isotopes of the Phanerozoic mafic-ultramafic intrusions in the Damiao area.*

Intrusion	Dating method	Age (Ma)	$\varepsilon_{\text{Hf}}(t)$	$\delta^{57}\text{Fe}$ (‰)	Data source
Xiahabaqin	Zircon SHRIMP U-Pb	392 ± 5	-8.01 ± 0.75	No data	Zhang <i>et al.</i> (2009a); Li <i>et al.</i> (2012b)
	Hornblende $^{40}\text{Ar}/^{39}\text{Ar}$	401 ± 8			
Dawusugou	Biotite $^{40}\text{Ar}/^{39}\text{Ar}$	396 ± 4	No data	0.19 ± 0.10	Zhou <i>et al.</i> (2012); this study
Gaositai	Zircon LA-ICP-MS U-Pb	213 ± 1	-0.23 ± 0.70	0.12 ± 0.10	Li <i>et al.</i> (2012a); this study
Longwangmiao	Zircon LA-ICP-MS U-Pb	141 ± 3	-17.90 ± 1.90	0.34 ± 0.10	This study
Heishan	Zircon LA-ICP-MS U-Pb	144 ± 1	-18.12 ± 0.59	0.35 ± 0.07	This study

5. Results

5.1 Zircon U-Pb ages

CL images of representative zircons are displayed in figure 3. The results of zircon U-Pb dating are listed in table 1 and plotted on concordia diagrams in figure 4. Only a few grains of zircon were separated from sample LWM-3, and they are mostly transparent and 100–200 μm in size. In CL images, they show oscillatory zoning with occasional homogeneous luminescence (figure 3a). Eight analyses were made on eight zircon grains, with the Th/U ratios ranging from 0.02 to 1.83 (average = 0.61). Their growth zoning and Th/U ratios are consistent with a magmatic origin. Consisting of inherited zircons, these analyses have $^{206}\text{Pb}/^{238}\text{U}$ ages varying from 139 to 1422 Ma. As the upper limit, three youngest concordant data (02, 03, 08) yielded a weighted mean $^{206}\text{Pb}/^{238}\text{U}$ age of 141.0 ± 2.5 Ma (2σ , MSWD = 0.99) (figure 4a). Since no younger mafic-ultramafic intrusions have been identified in the northern NCC, we interpret 141 ± 3 Ma as the emplacement age of the Longwangmiao intrusion.

Zircons in sample HS-3 are mostly transparent and 80–180 μm in size. Oscillatory zoning is well-developed for most zircons, suggesting a magmatic origin (figure 3b). Nineteen analyses were made on 17 zircon grains, with Th/U ratios from 0.46 to 1.83 (average = 1.07). Except for three inherited ages and six discordant ages, the remaining 10 analyses yielded a weighted mean $^{206}\text{Pb}/^{238}\text{U}$ age of 143.8 ± 1.2 Ma (2σ , MSWD = 0.93) (figure 4b). Therefore, the emplacement age of the Heishan intrusion is considered to be 144 ± 1 Ma.

5.2 Hf isotopic data

Hf isotopic analysis was made on the zircons with a concordant age of ~ 140 Ma, including three spots for sample LWM-3, and 10 spots for sample HS-3. The results are listed in table 2. Zircons in sample LWM-3 have $\varepsilon_{\text{Hf}}(t)$ values from -16.92 to -18.53 , yielding an average of -17.90 ± 1.90 (2σ). Similarly, zircons in sample HS-3 show a range of $\varepsilon_{\text{Hf}}(t)$ values from -16.72 to -19.31 , with an average of -18.12 ± 0.59 (2σ). As shown in table 4, the average $\varepsilon_{\text{Hf}}(t)$ values for the ~ 140 Ma Longwangmiao and Heishan intrusions are notably

Figure 3. CL images of representative zircons in (a) sample LWM-3 from the Longwangmiao intrusion and (b) sample HS-3 from the Heishan intrusion.

Figure 4. (a) and (b) U–Pb concordia diagrams for zircons in sample LWM-3 from the Longwangmiao intrusion and sample HS-3 from the Heishan intrusion, respectively; (c) chronogram of Phanerozoic mafic–ultramafic magmatic events in the Damiao and neighbouring areas. Data for the Damiao area are after table 4; data for the neighbouring areas are from Tian *et al.* (2007), Zhang *et al.* (2009a), Yang *et al.* (2012), and the authors unpublished data. CAOB: Central Asian orogenic belt. Error ellipses are at 2σ level.

lower than those for the ~ 215 Ma Gaositai intrusion (-0.23 ± 0.70 ; recalculated from the data of Li *et al.* 2012a) and the ~ 395 Ma Xiahabaqin intrusion (-8.01 ± 0.75 ; recalculated from the data of Zhang *et al.* 2009a). The single-stage model ages (T_{DM1}) are of 1505–1546 Ma for sample LWM-3 and of 1491–1586 Ma for sample HS-3. The two-stage model ages (T_{DM2}) were further calculated, varying from 2255 to 2359 Ma for sample LWM-3 and from 2248 to 2404 Ma for sample HS-3.

5.3 Fe isotopic data

The 17 analyzed bulk samples were collected from the Dawusugou, Gaositai, Longwangmiao and Heishan intrusions. Their $\delta^{57}\text{Fe}$ values are of 0.03–0.41‰, falling within the range of igneous basalts compiled by Zhao *et al.* (2010). The samples from the ~ 395 Ma Dawusugou intrusion have $\delta^{57}\text{Fe}$ values from 0.10 to 0.28‰, defining an average of 0.19 ± 0.10 ‰ (2σ). The samples from the ~ 215 Ma Gaositai intrusion show $\delta^{57}\text{Fe}$ values

ranging from 0.03 to 0.24‰, with an average of $0.12 \pm 0.10\text{‰}$ (2σ). However, the samples from the ~140 Ma Longwangmiao and Heishan intrusions have $\delta^{57}\text{Fe}$ values clearly heavier than the other two bodies, varying from 0.26 to 0.41‰ with an average of $0.34 \pm 0.10\text{‰}$ (2σ) and from 0.32 to 0.37‰ with an average of $0.35 \pm 0.07\text{‰}$ (2σ), respectively.

6. Discussion

6.1 Timing and mantle source of magmatism

Timing and source characteristics of mafic-ultramafic magmatism are crucially important for understanding mantle composition and processes. Based on published geochronological data and the newly-obtained zircon U–Pb ages (table 4), the mafic-ultramafic rocks in the Damiao area are known to have formed in the Middle Devonian, Late Triassic and Early Cretaceous ages.

Despite the presence of inherited zircons, the fact that all these intrusions show a limited variation of zircon $\varepsilon_{\text{Hf}}(t)$ values, tends to indicate that crustal contamination during magma ascent may be negligible (figure 5). Thus, their Hf isotopic features are primarily inherited from mantle sources. As a non-traditional stable isotope, Fe is an effective tracer for identifying the input of enriched lithospheric mantle from crustal contamination. It is because

the Fe isotopic signal of continental crust is similar to that of the magma derived from the depleted mantle source, while the Fe isotopic compositions of enriched lithospheric mantle would be modified by mantle metasomatism (figure 6; e.g., Poitrasson 2006; Weyer and Ionov 2007).

6.1.1 Middle Devonian

The emplacement age of the Dawusugou intrusion, as revealed by biotite $^{40}\text{Ar}/^{39}\text{Ar}$ dating, indicates that it is contemporaneous with the Xiahabaqin intrusion at ~395 Ma, but distinctly younger than the overlying Damiao anorthosite complex (Zhou *et al.* 2012).

The Xiahabaqin intrusion has a relatively wide range of zircon $\varepsilon_{\text{Hf}}(t)$ values from -11.59 to -4.17 (average = -8.01 ; recalculated from the data of Zhang *et al.* 2009a). Nonetheless, the zircon $\varepsilon_{\text{Hf}}(t)$ values are still plotted on the inferred evolution line of the SCLM beneath the northern NCC, implying limited contributions from ancient crustal material and its origination from the slightly-enriched SCLM that was prevalent during Paleozoic time (figure 5). Correspondingly, the contemporaneous Dawusugou intrusion displays $\delta^{57}\text{Fe}$ values that are a little higher than average for data from depleted mantle-derived magma (figure 6). This is consistent with its origination from a slightly-enriched mantle

Figure 5. Age-corrected $\varepsilon_{\text{Hf}}(t)$ vs. age diagram. Data of the mafic-ultramafic rocks in the Damiao area are from Zhang *et al.* (2009a), Li *et al.* (2012a) and this study. Data of other mafic-ultramafic rocks in the northern NCC are from Zhang and Yang (2007), Yang *et al.* (2007a, b) and Zhang *et al.* (2011). The evolution line of the SCLM beneath the northern NCC is inferred by the data of ~490 Ma mantle zircons entrained in the Xiaozhangjiakou intrusion (elliptical zone with horizontal lines; Tian *et al.* 2007). The evolution line of the average crust of the northern NCC is inferred by the data of zircons from the Archean TTGs and terrain gneisses (elliptical zone with vertical lines; Yang *et al.* 2005).

Figure 6. Correlation diagram of $\delta^{57}\text{Fe}$ of the Phanerozoic mafic-ultramafic intrusions in the Damiao area. Data of mantle xenoliths and melting products are compiled by Zhao *et al.* (2010) with additional data from Dauphas *et al.* (2009, 2010), Huang *et al.* (2011), Zhao *et al.* (2012), Poitrasson *et al.* (2013) and Teng *et al.* (2013). The dark grey zone represents the main variation range of $\delta^{57}\text{Fe}$ of komatiites, boninites and MORBs, which are formed by various degree partial melting of depleted mantle source. The vertical dashed line represents the Fe isotopic composition of continental crust ($\delta^{57}\text{Fe} = 0.10 \pm 0.03$; Poitrasson 2006).

reservoir, for which Fe isotopic compositions would be weakly elevated by mantle metasomatism. Combining the Hf and Fe isotopic features, it is concluded that the Middle Devonian mafic-ultramafic intrusions are the melting products of the prevalent SCLM beneath the northern NCC with slight crustal contamination.

6.1.2 Late Triassic

The zircon U–Pb age (280 ± 3 Ma) of the Gaositai intrusion was first reported by Chen *et al.* (2009b). But as revealed by the results of Zhang *et al.* (2009a), this weighted mean age is just a reflection of inherited zircons (Luo *et al.* 2013), which also occur in samples LWM-3 and HS-3 (table 1). Although no reliable crystallization age had been yielded, an upper limit of 265 Ma was given by Zhang *et al.* (2009a). A subsequent geochronological investigation showed a convincing emplacement age of 213 ± 1 Ma (Li *et al.* 2012a). As mentioned above, the Gaositai intrusion shares many typically geological and petrographic features with Alaskan/Ural-type intrusions, which are regarded as the hallmarks of arc magmatism (Himmelberg and Loney 1995; Pettigrew and Hattori 2006; Tseng *et al.* 2009). However, it is noteworthy that the ~ 215 Ma emplacement age postdates the final closure of the Paleasian Ocean (~ 250 Ma; Xiao

et al. 2003; Zhang *et al.* 2009c). Thus, the tectonic indication of Alaskan/Ural-type intrusions is still controversial.

A narrow range from -2.02 to 1.63 (average = -0.23) is exhibited for the zircon $\varepsilon_{\text{Hf}}(t)$ values of the Gaositai intrusion (recalculated from the data of Li *et al.* 2012a). Note that the zircon $\varepsilon_{\text{Hf}}(t)$ values are plotted above the inferred evolution line of SCLM (figure 5). In addition to an enriched mantle component, a large amount of asthenospheric material is therefore needed to involve. Indeed, this inference is further confirmed by the low $\delta^{57}\text{Fe}$ values (average = 0.12‰), which resembles the magma derived from a depleted mantle source (figure 6). Both of the Hf and Fe isotopic compositions give clear evidence for a large addition of asthenospheric material during the formation of the Gaositai (Late Triassic) intrusion.

6.1.3 Early Cretaceous

Zircon U–Pb dating carried out in this study indicates an emplacement age of ~ 140 Ma for both the Longwangmiao and Heishan intrusions. As shown in figure 4(c), Late Mesozoic mafic-ultramafic magmatism is being reported for the first time in the Damiao and neighbouring areas.

These two intrusions are characterized by notably low zircon $\varepsilon_{\text{Hf}}(t)$ values and old Hf model ages. On the $\varepsilon_{\text{Hf}}(t)$ vs. age diagram (figure 5), they are both plotted below the inferred evolution line of SCLM. Although crustal contamination likely accounts for their Hf isotopic features, the restricted range of zircon $\varepsilon_{\text{Hf}}(t)$ values implies that the parental magma should escape being intensely contaminated during ascent. Furthermore, these two intrusions clearly exhibit high $\delta^{57}\text{Fe}$ values with averages of 0.34 and 0.35‰ , respectively (figure 6). These Fe isotopic features further exclude an interpretation of the input of a continental crust component, and instead suggest that the Fe isotopic signal of the mantle source region was greatly changed by mantle metasomatism. Therefore, the parental magma of the Early Cretaceous intrusions should be produced by melting significantly-enriched mantle, which is isotopically distinct from the weakly-enriched SCLM that was predominant in the Paleozoic.

Contrasting source regions, as revealed by distinct isotopic (Hf and Fe) signals for different intrusions in the Damiao area, are consistent with their petrographic features. Only the Gaositai intrusion, the formation for which was largely involved with the asthenospheric mantle reservoir, displays the occurrence of dunite. Additionally, note that the tectonic position of Damiao during the evolution of the NCC has an important bearing on

the recognition of multistage Phanerozoic mafic–ultramafic magmatism in this area. As shown in figure 1(a), the Damiao area is located near the intersection of the northern margin of the NCC and the Daxing'anling–Taihangshan gravity lineament. Moreover, the Jining–Longhua fracture zone provides a beneficial emplacement condition for mantle-derived magma.

6.2 Implications for lithospheric destruction in the northern NCC

Lithospheric destruction of the NCC has been the subject of considerable geological interest over the last few decades. Although a broad consensus has been reached that the climax of the destruction of the NCC took place in the Late Mesozoic (e.g., Gao *et al.* 2004; Wu *et al.* 2005; Yang *et al.* 2008), there is no agreement when it comes to the initiation timing and duration (e.g., Wu *et al.* 2008; Xu *et al.* 2009; Zhu *et al.* 2012b). Much research suggests that the destruction of the NCC occurred over a long period of time, initiated in the Late Paleozoic (e.g., Xu *et al.* 2009; Li *et al.* 2009b; Zhou *et al.* 2012) or Early Mesozoic (e.g., Han *et al.* 2004; Yang *et al.* 2007a; Yang and Wu 2009; Zhang *et al.* 2012). Other research favours a short-time-scale model, beginning from the Late Jurassic (e.g., Jiang *et al.* 2005).

Magmatically, lithospheric destruction is reflected successively by asthenospheric mantle upwelling, decompression-induced melting and subsequent strong involvement of a depleted component in magma generation. As described above, the ~395 Ma intrusions in the Damiao area were derived from the prevalent slightly-enriched SCLM beneath the northern NCC. In fact, the 465 Ma Fuxian kimberlite and 280 Ma Guyang gabbro are similar cases (figure 5). However, both the Hf and Fe isotopic features lead to the conclusion that

asthenospheric material was strongly involved in the generation of the ~215 Ma Gaositai intrusion, as well as the mafic enclaves from the Xiuyan granitoid, Liaodong peninsula. It is thus implied that lithospheric destruction in the northern NCC should have been initiated in Late Triassic time, earlier than in the interior part of the NCC (Late Mesozoic; Xu *et al.* 2009). Coincidentally, asthenospheric mantle upwelling and strong involvement of depleted component in mantle-derived magma just postdate the final closure of the Paleasian Ocean (~250 Ma; Xiao *et al.* 2003; Zhang *et al.* 2009c). Accordingly, lithospheric destruction in the northern NCC was probably caused by post-orogenic extension following the collision between the Siberian Craton and the NCC. A reasonable inference is that the tectonic evolution of marginal orogenic belts around the craton may have played a key role in the widespread Late Mesozoic lithospheric destruction of the NCC (Xu 2004; Zhang *et al.* 2013).

As stated above, a significantly-enriched mantle component is identified beneath the northern NCC in addition to the prevalent SCLM. It was abruptly present as a magma source during Late Mesozoic time. Zircons from the ~140 Ma intrusions in the Damiao area record old Hf model ages of ~1.5 Ga. The lack of a coeval tectono-magmatic event in the northern NCC implies that the significantly-enriched mantle was refertilized by multistage mantle metasomatism, which may be related to the ~1.85 Ga collision between the eastern and western blocks and the continuous southward subduction of the Paleasian Ocean (Tang *et al.* 2013). We interpret this isotopically enriched mantle reservoir as 'metasomes', which are commonly resided at depths of ~100 km in the ancient cratonic lithospheric mantle (Waters and Erlank 1988; Xu 2001; Xu *et al.* 2009). They would not be melted until the lithosphere was thin enough and heat transfer was enhanced from the

Figure 7. Cartoon illustrating the lithospheric destruction process beneath the northern NCC (c.f., Xu *et al.* 2009). SCLM: subcontinental lithospheric mantle; TLFZ: Tan–Lu fracture zone; DTGL: Daxing'anling–Taihangshan gravity lineament.

asthenosphere. This explanation is further confirmed by the clearly high zircon $\varepsilon_{\text{Hf}}(t)$ values of the mafic enclaves from the Gudaoling granitoid, Liaodong peninsula (figure 5). As illustrated in figure 1(a), this area is far from the Trans-North China Orogen and thus metasomatism of the cratonic lithospheric mantle is not as intense as in the Damiao area. As a result, 'metasomes' might not be present in the Liaodong peninsula and the Hf isotopic compositions of mantle-derived magma would be as depleted as the mafic enclaves from the Xiuyan granitoid and the Gaositai intrusion owing to the large involvement of asthenospheric material. In summary, isotopically distinct mantle sources of the mafic-ultramafic magmatism in the northern NCC can be interpreted as a record of mantle heterogeneity and the gradual upward migration of the lithosphere-asthenosphere boundary during lithospheric destruction (figure 7).

7. Conclusions

- Our newly-obtained zircon U-Pb ages, coupled with previous data, reveal that the emplacement ages of the mafic-ultramafic intrusions in the Damiao area span from Middle Devonian to Early Cretaceous times. As reflected by Hf-Fe isotope systematics, distinct mantle components contributed to their magma generation.
- The ~395 Ma intrusions were derived from the slightly-enriched SCLM that was prevalent during the Paleozoic, whereas asthenospheric material was strongly involved in the formation of the ~215 Ma Gaositai intrusion. This suggests that lithospheric destruction in the northern NCC would have been initiated in Late Triassic time, probably triggered by post-orogenic extension following the ~250 Ma collision between the Siberian Craton and the NCC.
- A significantly-enriched mantle component is identified in addition to the predominant slightly-enriched SCLM by the isotopic signals of ~140 Ma intrusions. It might have resided in the ancient cratonic lithospheric mantle, and would not have melted until the Late Mesozoic when the lithosphere was thin enough and heat transfer was enhanced from the asthenosphere during lithospheric destruction.

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