Timing of the granulite facies metamorphism in the Sanggan area, North China craton: zircon U-Pb geochronology

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Abstract Zircon U-Pb ages are reported for three samples of intrusive rocks in Khondalite series in the Sanggan area, North China craton. The age of meta-granite is dated as 2005 ± 9 Ma, implying that the sedimentary sequences in Khondalites series formed before 2.0Ga. The age of 1921 ± 1 Ma for the meta-diorite constrain the age of granulite facies metamorphism younger than this date. The age of 1892 ± 10 Ma for garnet granite is obtained, but the granite crystallization age seems a little younger than the date considering the morphology of zircons. On the basis of these dates and of a concise review of previous age data, it is inferred that the Khondalite series was subjected to granulite facies metamorphism at about 1.87Ga together with tonalitic granulites and HP basic granulites in the Sanggan area.

Keywords: zircon U-Pb age, granulite, metamorphism, Paleoproterozoic, North China craton.

The Sanggan area is in the north-central part of the North China craton. Here occur three types of granulite facies rock associations. They are meta-sedimentary sequences in Khondalite series, granulite of TTG compositions in the Huaian gneiss terrane, and high-pressure basic granulites distributed in the marginal area of the Huaian gneiss terrane. Metamorphic reseach revealed that all types of granulites have clockwise *P*-*T* paths with near-isothermal decompressional sections^[1-5], which are ascribed to metamorphism in collisional environment^[3, 6].

However, it has not been determined up to now that if the granulites are products of a single collisional tectonic process, and/or when the collision took place, late Archaean or Paleoproterozoic. For the granulites of TTG compositions and HP basic granulites, there are quite a lot of isotopic data to place constraints on their metamorphic ages^[7–9]. But the metamorphic age of metasedimentary rocks in Khondalite series is less constrained even though many isotopic ages existed^[10–12].

This paper aims to determine the metamorphic age of the Khondalite series. Considering the limitation of conventional multigrain chemical zircon U-Pb method, we try to avoid meta-sedimentary rocks but choose magmatic rocks, metamorphosed and unmetamorphosed, to date their formation age, and constrain the metamorphic age in return. Then, integrated with a concise review of the previous isotopic ages, the metamorphic age of the three types of granulites in the Sanggan area and the corresponding continental collision age will be discussed.

1 Geological background

The Sanggan area is in the north-central part of the North China craton. The early Precambrian basement here is made up of two lithological units: Khondalite series to the northwest and Huaian gneiss terrane to the southeast (fig. 1). The Huaian gneiss terrane is mainly composed of gneisses and granulites of TTG compositions^[13,14]. Research in structural geology suggested that the terrane is an oval gneiss dome centered near Huaian^[14–16]. Around outer part of the Huaian gneiss terrane/dome, high-pressure (HP) basic granulites are developed as small sheet like blocks or lenticular enclaves in background of tonalitic granulites^[4]. But the original geological relations between HP granulites and the country gneisses still remain to be further investigated. So, we consider HP granulite association to be an individual type in this paper.



Fig. 1. Sketch map of early Precambrian metamorphic basement in the Sanggan area, North China craton.

The Khondalite series is mainly composed of sedimentary sequences metamorphosed into low-pressure granulite facies featured by garnet + cordierite + sillimanite assemblages with graphite somewhere^[1, 17]. Large volume garnet granites occupy about 40% area of the Khondalite series. They are principally subautochthonous post tectonic S-type granites^[17–19]. Also, there are dozens of small-scale gabbroic to dioritic intrusive bodies metamorphosed to granulite facies together with sedimentary rocks. In addition, there occur some granite intrusion prior to metamorphism, and metamorphosed into orthopyroxene and perthite bearing granulite or charnokite already. But it is still recognizable from meta-sedimentary rock associations.

In this paper, representative samples of the above three types of intrusive rocks in Khondalite series are chosen to perform zircon U-Pb geochronological studies. They are meta-granite sample (NY99-11) from Niuyaogou near Zhuozi, meta-diorite and garnet granite samples (LC99-15, LC99-14) from Anzishan northwest to Liangcheng. The meta-dioritic body is 0.3 km wide and 2 km long. It intruded into the meta-sedimentary sequences, and was intruded by garnet granite.

2 Analytical methods

U-Pb analyses were performed at the isotope geochronological laboratory at the Institute of Geology and Geophysics, Chinese Academy of Sciences. Zircons were extracted from samples using standard techniques of density and magnetic separation, and grains were selected for analysis on the basis of size, clarity, color and morphology. Each analysis was performed on zircon fraction containing 4–5 grains weighing about 20 µg.

The procedures for zircon digestion, separation of U and Pb, and isotope dilution mass spectrometry using a $^{235}U^{-205}Pb$ enriched tracer-solution followed those of Krogh^[20] with modifycations detailed in Xu^[21]. During this study, total procedure blanks for Pb are less than 50 pg. Isotope ratios were measured using VG354 thermal ionization mass spectrometer. Isotopic ratios and



Fig. 2. The electronic scanning microscope photos of zircons. (a) zircons from meta-granite NY99-11; (b) zircons from meta-diorite LC99-15; (c) zircons from garnet granite LC99-14.

errors were calculated and corrected for common lead^[22], spike, fractionation and blank. U-Pb ages were calculated by a regression technique using the ISOPLOT program written by K. R. Ludwig.

3 Results

The measured and calculated zircon U-Pb compositions are listed in table 1. The electronic scanning microscope photos of zircons are shown in fig. 2. U-Pb isotopic composition and age results are shown in fig. 3. The results of each sample will be described as follow.

(1) Meta-granite NY99-11. Zircons in this sample were big, brown, clear to semitransparent, and multifaceted short to long prismatic crystals (fig. 2(a)). The length/width ratios range from 2 to 7. In general, those zircons which are long prismatic, euhedral, clear, inclusion and fracture free were selected for analysis. Four zircon analyses are plotted in concordia diagram. They confirm well to a single disconcordia with upper intercept age of 2005 ± 9 Ma. Among the four points, two are almost on the concordia, one is just below the concordia indicating slightly radiogenic Pb loss. But another point is above the concordia. This is caused by the U-lost in

		Concen	tration			Atomic ratio			Apparent age.	/Ma
$0.pmon$ $1.opmon$ $2.9 h_{\rm D}$ $2.8 \eta_{\rm U}$ $2.8 \eta_{\rm U}$ $2.9 \eta_{\rm D}$ <	Sample	[ome/11	Dh/mmol	^{206}Pb	206 Pb	207 Pb	\mathbf{P}^{207}	^{206}Pb	207 Pb	207 Pb
meta-granite. Nityaogon near ZuoziNY99-11-1852.11354.10181820.3411±0.00715.761±0.1210.12349±0.00033189219411992.8±47NY99-11-2374.51169.01952.40.3779±0.00766.402±0.1310.12338±0.00023205320132012.5±53.3NY99-11-3669.26300.48227270.3369±0.00756.307±0.1290.12361±0.00033189220132014.4±32NY99-11-4520.24268.9494340.4224±0.00875.307±0.1290.12361±0.00033202720192011.4±32NY99-11-4520.24268.9494340.4224±0.00876.307±0.1290.12361±0.00033207121362008.9±12NY99-11-4520.24268.9494340.4224±0.00865.348±0.1080.11762±0.00038183718781950.4±12LC99-15-257.7422.7733960.3392±0.00665.348±0.1080.11762±0.00038183718771950.4±12LC99-15-352.9523.050.3312±0.00675.371±0.1090.11762±0.00038183718771950.4±27LC99-15-352.9523.840000.3382±0.00655.371±0.1090.11762±0.00038183718761950.4±27LC99-15-352.9523.840000.3382±0.00655.371±0.1090.11762±0.00038187418761950.4±27LC99-14-152.9523.840000.3382±0.00655.371±0.1090.11762±0.0003818371950.4±57LC99-14-15				^{204}Pb	238 U	²³⁵ U	206 Pb	238 U	235 U	$^{206}\mathrm{Pb}$
	meta-granite, N	iuyaogou near	·Zuozi							
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	NY99-11-2	374.51	169.01	9524	0.3749 ± 0.0076	6.402 ± 0.131	0.12385 ± 0.00023	2053	2033	2012.5 ± 3.3
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meta-diorite, Anzishan near Liangcheng 5.61 679.3 0.3290±0.0068 5.354±0.114 0.11804±0.00058 1834 1878 1926.7±8.9 LC99-15-2 5.774 2.2.77 3584 0.3299±0.0066 5.348±0.108 0.11762±0.00008 1837 1877 1920.4±1.2 LC99-15-2 5.774 22.05 3906 0.3382±0.0068 5.348±0.108 0.11764±0.00035 1877 1920.7±5.4 LC99-15-3 5.2.95 22.05 3906 0.3382±0.0068 5.348±0.109 0.11762±0.00018 1877 1920.7±5.4 LC99-15-4 45.78 18.51 4386 0.3312±0.0067 5.371±0.109 0.11762±0.00018 1844 1880 1920.7±5.4 LC99-15-4 45.78 18.51 4386 0.3312±0.0067 5.371±0.109 0.11762±0.00018 1844 1880 1920.7±5.4 garnet granite, Anzishan near Liangcheng 1.8.51 4386 0.3312±0.0067 5.602±0.115 0.1164±0.00023 1932 1920.7±5.4 LC99-14-1 5.2.93 23.3 4000 0.3494±0.00058 5.401±0.110 0.11584±0.00027 1881 1897 1890.1±6.7	NY99-11-4	520.24	268.94	9434	0.4224 ± 0.0087	7.199 ± 0.158	0.12361 ± 0.00081	2271	2136	2008.9 ± 12
LC99-15-113.395.61 679.3 0.3290 ± 0.0068 5.354 ± 0.114 0.11804 ± 0.0058 1834 1878 1926.7 ± 8.9 LC99-15-2 57.74 22.77 3584 0.3298 ± 0.0068 5.348 ± 0.108 0.11762 ± 0.0008 1837 1877 1920.4 ± 1.2 LC99-15-3 52.95 22.05 3906 0.3382 ± 0.0068 5.486 ± 0.112 0.11762 ± 0.00018 18378 1898 1920.7 ± 5.4 LC99-15-4 45.78 18.51 4386 0.3312 ± 0.0067 5.371 ± 0.109 0.11762 ± 0.00018 1844 1880 1920.7 ± 5.4 LC99-15-4 45.78 18.51 4386 0.3312 ± 0.0067 5.371 ± 0.109 0.11762 ± 0.00018 1844 1880 1920.7 ± 5.4 gamet granite, Anzishan near Liangcheng 18.51 4386 0.3332 ± 0.0067 5.486 ± 0.115 0.11630 ± 0.00043 1932 1920.4 ± 2.7 LC99-14-1 52.93 23.38 4000 0.3494 ± 0.0070 5.602 ± 0.116 0.1156 ± 0.00027 1932 1976 1900.1 ± 6.7 LC99-14-2 80.76 33.76 5319 0.3387 ± 0.0068 5.410 ± 0.110 0.11516 ± 0.0022 1881 1897 1892.4 ± 3.4 LC99-14-3 119.54 51.70 868.8 0.3252 ± 0.0065 5.163 ± 0.104 0.11516 ± 0.00022 1817 1897 1892.4 ± 3.4 LC99-14-4 119.54 54.65 3401 0.3742 ± 0.0075 5.998 ± 0.121 0.11626 ± 0.0013 1976 1997.5 ± 3.0 LC99-14-4 119.54 54.05 5.998 ± 0.121 0.1161	meta-diorite, A	nzishan near L	iangcheng							
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LC99-15-3 52.95 22.05 3906 0.3332 ± 0.0068 5.486 ± 0.112 0.1176 ± 0.00035 1878 1898 1920.7 ± 5.4 LC99-15-4 45.78 18.51 4386 0.3312 ± 0.0067 5.371 ± 0.109 0.11762 ± 0.00018 1844 1880 1920.4 ± 2.7 garnet granite, Anzishan near Liangcheng 2.312 ± 0.0067 5.371 ± 0.109 0.11762 ± 0.00018 1844 1880 1920.4 ± 2.7 LC99-14-1 $5.2.93$ 23.8 4000 0.3494 ± 0.0070 5.602 ± 0.115 0.11630 ± 0.00043 1932 1916 1900.1 ± 6.7 LC99-14-2 80.76 33.76 5319 0.3387 ± 0.0068 5.410 ± 0.110 0.11584 ± 0.00027 1881 1887 1893.0 ± 4.1 LC99-14-3 127.33 51.70 8688 0.3252 ± 0.0065 5.163 ± 0.104 0.11516 ± 0.00022 1815 1847 1882.4 ± 3.4 LC99-14-4 119.54 54.65 3401 0.3742 ± 0.0075 5.998 ± 0.121 0.11626 ± 0.00013 2049 1976 1895.5 ± 2.0	LC99-15-2	57.74	22.77	3584	0.3298 ± 0.0066	5.348 ± 0.108	0.11762 ± 0.00008	1837	1877	1920.4 ± 1.2
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garnet granite, Anzishan near Liangcheng5.602±0.1150.11630±0.00043193219161900.1±6.7LC99-14-15.2.9323.840000.3494±0.00705.602±0.1150.11630±0.00043193219161900.1±6.7LC99-14-280.7633.7653190.3387±0.00685.410±0.1100.11584±0.00027188118871893.0±4.1LC99-14-3127.3351.7086.80.3252±0.00655.163±0.1040.11516±0.00022181518471882.4±3.4LC99-14-4119.5454.6534010.3742±0.00755.998±0.1210.11626±0.00013204919761899.5±20	LC99-15-4	45.78	18.51	4386	0.3312 ± 0.0067	5.371 ± 0.109	0.11762 ± 0.00018	1844	1880	1920.4 ± 2.7
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LC99-14-2 80.76 5319 0.3387±0.0068 5.410±0.110 0.11584±0.00027 1881 1887 1893.0±4.1 LC99-14-3 127.33 51.70 868.8 0.3252±0.0065 5.163±0.104 0.11516±0.00022 1815 1847 1882.4±3.4 LC99-14-4 119.54 54.65 3401 0.3742±0.0075 5.998±0.121 0.11626±0.00013 2049 1976 1899.5±2.0	LC99-14-1	52.93	23.8	4000	0.3494 ± 0.0070	5.602 ± 0.115	0.11630 ± 0.00043	1932	1916	1900.1 ± 6.7
LC99-14-3 127.33 51.70 868.8 0.3252±0.0065 5.163±0.104 0.11516±0.00022 1815 1847 1882.4±3.4 LC99-14-4 119.54 54.65 3401 0.3742±0.0075 5.998±0.121 0.11626±0.00013 2049 1976 1899.5±2.0	LC99-14-2	80.76	33.76	5319	0.3387 ± 0.0068	5.410 ± 0.110	0.11584 ± 0.00027	1881	1887	1893.0 ± 4.1
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	LC99-14-3	127.33	51.70	868.8	0.3252 ± 0.0065	5.163 ± 0.104	0.11516 ± 0.00022	1815	1847	1882.4 ± 3.4
	LC99-14-4	119.54	54.65	3401	0.3742 ± 0.0075	5.998 ± 0.121	0.11626 ± 0.00013	2049	1976	1899.5 ± 2.0

Table 1 Measured and calculated zircon II-Ph isotonic commositions^{a)}

a) Values of ""Ph/""Pb are measured in this study, other isotopic ratios are corrected for common lead (Stacey, 1975), spike, fractionation and blank; all errors are 2σ .



Fig.3. Concordia diagrams showing zircon U-Pb analyses for sample NY99-11 from meta-granite (a), sample LC99-15 from meta-diorite (b), and sample LC99-14 from garnet granite (c).

laboratory presumably, and does not affect the reliability of the date in this case. So, the U-Pb reverse discordant age of 2005 ± 9 Ma is considered to be the formation age of the granite.

(2) Meta-diorite LC99-15. Zircons in this sample were clear, yellowish, multifaceted short to long prismatic crystals (fig. 2(b)). Some grains are eroded. The length/width ratios range from 1.5 to 6. The clear, less eroded, long prismatic zircons were selected for analysis. All the four data points are plotted below but very close to the concordia (fig. 3(b)), showing that the Pb loss is very limited. The four analyses define a disconcordia line with bad probability of fit. So, we take the weighted average 207 Pb/²⁰⁶Pb apparent age of 1921±1 Ma (fig. 3(b)) as the U-Pb age of the sample. It represents the age of magma crystallization of the dioritic intrusive body.

(3) Garnet granite LC99-14. Zircons in this sample were light brown, clear to semitransparent. Most zircons (80%) are round in shape, and obviously eroded. Zircons of slightly eroded and multifaceted short to long prismatic (fig. 2(c)) also existed, and were selected for analysis. Three data points of four are plotted on or slightly below the concordia. Another point is above the concordia like the sample NY99-11, showing the U-lost in laboratory presumably. Four zircon analyses define a well fit disconcordia giving a reverse discordant age of 1892 ± 10 Ma.

4 Discussion

4.1 Significances of zircon U-Pb ages

From meta-granite and garnet granite samples (NY99-11 and LC99-14), we obtained reverse discordant dates according to analyses plotted above the concordia (fig. 3(a), (c)). In general, reverse discordance can be caused by U-lost in laboratary as well as by Pb-profit due to underesti-

mation of the common-Pb correction, if ²⁰⁴Pb was not unerringly measured^[23–25]. From the measured data, however, there is not a strong correlation between the reverse discordant data and the measured ²⁰⁶Pb/²⁰⁴Pb ratios. Possibility of the natural feature of the analysed zircons also cannot be excluded. From the previous published data, reverse discordance is more frequently seen in old zircons. Another possibility is that the zircons were not completely decomposed. No matter how the reverse discordance was caused, the disconcordant ages are still reliable in this case.

U-Pb systematics in zircon are generally considered to be the most difficult to reset completely, even by granulite facies metamorphism^[26–28]. Its closing temperature T_c is considered to be >900°C. It is likely that only very small zircons can be reset during metamorphism in continental crust^[29,30], in spite of some disturbance in their U-Pb systematics as a result of new zircon growth during later metamorphism and/or Pb-loss at low temperature.

In meta-granite sample NY99-11 and meta-diorite sample LC99-15, zircons are prismatic with varied length/width ratios (~7 for some grains) and clear facets and round edges (fig.2(a), (b)), showing features of magmatic zircons with limited erosion during metamorphism^[31,32]. Also, these zircons are quite big, >50 μ m in width and >100 μ m in length. So, we intend to consider the two U-Pb dates of 2005±9 Ma and 1921±1 Ma to be magmatic crystallization ages of granite and diorite.

Sample LC99-14 is a subautochthonous post tectonic S-type granite. Most zircons from the sample are round and short prismatic. Therefore, effect of inherited zircon cannot be excluded completely, although only multifaceted clear prismatic zircons were selected for analysis. So, the formation age of the granite should be younger than the measured age of 1892 ± 10 Ma. Combined with another zircon U-Pb concordia age of 1836 ± 18 Ma^[19], the most possible formation age of this type of granite was between the two dates presumably.

4.2 Metamorphic age of the three granulite associations in the Sanggan area

For meta-sedimentary rocks in the Khondalite series, the existing isotopic ages has already implied the late Paleoproterozoic metamorphism. Checking the published data in detail, we find some zircon U-Pb ages precise and reliable because their data points are close to the concordia. They are 1892 ± 21 Ma, 1916 ± 16 Ma and 1873 ± 32 Ma^[10-12]. All of them are for sillimanite-garnet gneisses. This type of rock is meta-pelite, and contains less inherited zircons compared with other meta-sedimentary rocks. So, the three ages, especially the youngest one, should be close to the granulite facies metamorphic age. However, they were considered as an important thermal event previously, not definitely as the age of granulite facies metamorphism. In this paper, the zircon U-Pb age of 1921 ± 1 Ma for meta-diorite strongly suggests that the granulite facies metamorphism evidenced by the Khondalite series should be later than 1.92 Ga. According to the discussion on the existing data above and the age of garnet granites, it is believed that the granulite metamorphic age was likely to be at about 1.87 Ga.

For the metamorphic age of the gneisses and granulites in Huaian gneiss terrane, the previous ideas were very different, such as end of Archaean (2.4—2.5 Ga), Paleoproterozoic (2.2—2.3 Ga), and Paleoproterozoic (1.8—1.9 Ga). Checking the existing data in detail, we find that the SHRIMP zircon U-Pb data from three samples^[7,8] provide us with reliable evidence for the metamporphic age. Their zircon U-Pb data are plotted just below the concordia between 2.7 to 1.8 Ga. There are quite a few concordia data points at 2.5 and 2.6—2.7 Ga, indicating the formation age of the TTG gneiss terrane. The other data are grouped as a straight line roughly pointing to 1.8 —2.5 Ga. And further, there are two concordia ages of 1.85 and 1.95 Ga in the lower intercept of the line with the concordia. The data pattern is typical one of gneiss terranes of the world^[30], the upper and the lower of data group exhibit formation and metamorphic ages of the gneiss terrane respectively due to the metamorphic modification to the magmatic zircons to different extent. Accordingly, the granulite facies metamorphic age of the Huaian TTG gneiss terrane has been deduced as about 1.85 Ga or a little older.

For HP basic granulites, some newly published data can place proper constraints to the metamorphic age. Two garnet + whole rock Sm-Nd isochron ages are 1842 ± 38 Ma and 1856 ± 26 Ma^[9]. Considering Sm-Nd closure temperature and the retention of Nd isotopic memory in metamorphic garnet^[33,34], these isochron ages are thought to be the beginning age of cooling after HP granulite metamorphism. The ages have been supported by a garnet ⁴⁰Ar/³⁹Ar plateau age of $1852\pm8Ma$ with 76% ³⁹Ar released and ⁴⁰Ar/³⁹Ar ratios of 4376—10666 and the corresponding ⁴⁰Ar/³⁹Ar isochron age of $1962\pm37Ma$ ^[35]. Also presented is a whole rock Sm-Nd isochron age of $1870Ma^{[9]}$, which was from HP granulite samples within meters indicating Nd resetting during granulite metamorphism^[36]. Therefore, about 1.87 Ga is closer to the HP granulite metamorphic age.

In the Khondalite series, there occur broadly pegmatites with big muscovite giving precise ⁴⁰Ar/³⁹Ar age of 1.80Ga^[37], which implies that the collisional process causing granulite facies metamorphism stopped completely at about 1.80 Ga.

4.3 Conclusion and geotectonic significance

This paper report zircon U-Pb ages of three types of intrusive rocks in Khondalite series. They are 2005 ± 9 Ma for meta-granite, 1921 ± 1 Ma for meta-diorite, and 1892 ± 10 Ma for garnet granite.

From the geological relations and metamorphic features, we can infer that the formation age of sedimentary sequences in Khondalites series was prior to 2.0Ga, the metamorphic age of Khondalite series should be later than the intrusive age (1.92 Ga) of the diorite, and earlier than the garnet granite formation age. 1.87Ga is closer to the metamorphic age of the Khondalite series. Therefore, on the basis of dates present here and a concise review of previous age data, three types of granulites of meta-sedimentary rocks in Khondalite series, tonalitic granulites in Huaian gneiss

terrane and HP basic granulites, were likely subjected to a single granulite facies metamorphism at ~1.87Ga during Paleoproterozoic in the Sanggan area.

From the metamorphic research, all these granulites have clockwise *P*-*T* paths with near isothermal decompressional sections suggesting a continental collision environment caused by the process of the assembly of the North China craton. In conclusion, the late Paleoproterozoic was the age of the assembly of the North China craton as suggested by Zhao et al.^[38,39].

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