1	LA-ICP-MS U-Pb geochronology of detrital zircons from the Jining Complex,
2	North China Craton and its tectonic significance
3	Xiaoping Xia, Min Sun [*] , Guochun Zhao, Yan Luo
4	Department of Earth Sciences, the University of Hong Kong, Pokfulam Road, Hong
5	Kong
6	Abstract: LA-ICP-MS U-Pb zircon dating and cathodoluminescene (CL) image
7	analysis were carried out to determine the protolith and metamorphic ages of
8	high-grade Al-rich gneisses, named as "khondalites", from the Jining Complex of the
9	North China Craton (NCC). The analytical results of more than 200 detrital zircon
10	grains from the khondalites show three main age populations: 2060 Ma, 1940 Ma and
11	1890 Ma. These data indicate that the provenance of the Jining khondalites is
12	Paleoproterozoic in age, but not Archean as previously suggested, and the sediments
13	were derived from a provenance different from the Eastern Block and the Yinshan
14	Terrane of the NCC. The nearly concordant youngest age of 1842 ± 16 Ma
15	(²⁰⁷ Pb/ ²⁰⁶ Pb age) for the detrital zircons is interpreted as the maximum depositional
16	age of the khondalites. Overgrowth rims of detrital zircons yield an age of 1811 ± 23
17	Ma, which we interpret as the metamorphic age. The new age data are consistent with
18	the recent three-fold tectonic subdivision of the NCC and support that the Eastern and
19	Western Blocks collided at \sim 1.8 Ga to form the coherent NCC.

Keywords: Detrital zircons, LA-ICP-MS, khondalites, North China Craton 21

^{*} Corresponding author: minsun@hkucc.hku.hk

2 1. Introdution

3 A major advancement in understanding the geological history of the North China Craton has been achieved in the past few years following a new tectonic subdivision 4 5 of the craton into the Eastern and Western Blocks, separated by an intervening orogenic belt, named the Trans-North China Orogen (Zhao et al., 1998, 1999a, 1999b, 6 7 2000a, 2000b, 2001). There is now a broad consensus that the assembly of the North 8 China Craton was completed by amalgamation of the Eastern and Western Blocks 9 along the Trans-North China Orogen (Zhao, 2001; Wilde et al., 2002; Li et al., 2000; 10 Kusky and Li, 2003; Kröner et al., 2005). However, controversy still remains as to 11 when the amalgamation of the two blocks occurred. One school argues that the final 12 collision took place at ~2.5 Ga (e.g. Li et al., 2000; Kusky and Li, 2003), whereas the 13 other suggests that the event occurred at ~1.8 Ga (Zhao, 2001; Wilde et al., 2002). 14 Khondalites are high-grade metamorphosed Al-rich sedimentary rocks, consisting of 15 sillimanite, garnet, K-feldspar, plagioclase, graphite and quartz (Walton et al, 1983) 16 and widely crop out in the Jining, Huai'an, and Datong areas of the North China Craton (NCC) (Fig. 1). These rocks have long been regarded as mid- or late-Archean 17 18 sequences overlying the Archean granulites and TTG gneisses (Qian et al., 1985; Lu 19 et al., 1996; Qian et al., 1999; Li et al., 1999). However, this idea is not supported by 20 the sparse U-Pb data recently obtained by the TIMS (thermal ionization mass 21 spectrometry) technique for the khondalites, which fall in the Paleoproterozoic, but 22 not Archean (Wu et al., 1997). In order to sustain the prevailing tectonic model(s),

these Paleoproterozoic ages were interpreted as "metamorphic" ages by some 1 2 reseachers in China (e.g. Lu et al., 1996). However, numerous geochronological 3 studies on high-grade metamorphic terrains around the world have shown that igneous 4 zircons can survive high-grade metamorphism (e.g. Vavra et al., 1996, 1999; Kröner 5 et al., 2000; Zhao et al., 2002). Because these metasedimentary rocks were deposited 6 on the continental margin of the Western Block (Condie et al., 1992) prior to its 7 collision with the Eastern Block, reliable age data must be obtained in order to 8 constrain the nature of the Western Block and the inter-block collision. For this 9 reason, we undertook a detailed zircon geochronology to determine the depositional 10 and metamorphic ages of the khondalites in the NCC.

11 Whole-grain zircon analysis may result in ambiguous data since metamorphic 12 overgrowths on zircon rims are very common in high-grade metamorphic rocks. It has been recently demonstrated that cathodoluminescence (CL) and back-scattered 13 14 electron (BSE) imaging can be used to reflect internal structure of zircon (e.g. Vavra 15 et al., 1996, 1999; Kröner et al., 2000; Zhao et al., 2002; Guan et al., 2002), which, in 16 turn, provides important information on the origin of zircons (e.g. magmatic, 17 metamorphic, detrital, xenocrystic, etc). Previous studies on high-grade metamorphic 18 rocks showed that metamorphic zircons occur as either multifaceted single grains or 19 overgrowth rims, which are structureless and highly luminescent on CL images, 20 whereas magmatic zircons are generally euhedral with oscillatory zoning and show 21 low to variable luminescence (e.g. Zhao et al., 2002; Corfu et al., 2003). Spot analysis 22 of U-Pb isotope compositions on (part of) zircons can yield the protolith and

metamorphic ages of zircons and further help elucidate the evolution history of high-grade metamorphic rocks. In this study, CL images were utilized to identify structure and origin of zircons from the Jining khondalites, and spot analysis of U-Pb isotope compositions was conducted using an ICP-MS (inductively coupled plasma – mass spectrometry) equipped with a UV-laser micro-probe. The new results constrain the depositional and metamorphic ages of the khondalites, as well as the time of collision between the major blocks of the North China Craton.

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9 2. Geological setting

10 The Jining Complex is located in the Zhuozi, Jining and Datong areas (Fig. 2). It is composed of two important assemblages of metamorphic rocks: the granulite and 11 12 khondalite suites (Lu et al., 1996). The former suite consists predominantly of granulite-facies metamorphic TTG gneisses and minor enderbites and charnockites 13 14 with metamorphic P-T conditions at 8.0-10.0 kbar and 750-800°C (Lu and Jin, 1993; 15 Zhao et al., 1999a). The dominant rock types in the khondalite suite are 16 sillimanite-garnet gneiss (SGG), quartz-garnet gneiss (QGG) and quartz-feldspar 17 gneiss (QFG), roughly in a proportion of 5:3:2 (Liu, 1989). The SGG units are up to 18 100 m thick and are commonly layered on a scale of a few centimeters. The QGG 19 units occur as massive units up to a few meters thick (most are <1 m thick) 20 interlayered with the SGG and can often be traced along the strike for several 21 kilometers. The QFG occurs chiefly as concordant units within the SGG and QGG 22 units and ranges from 10 cm to 3 m thick. There is an increase from east to west in the

proportion of the SGG and QFG and a corresponding decrease in the QGG. Thin lenses of marble occur in the khondalite suite and become more abundant from Datong northwest towards Zhuozi. A graphite gneiss unit up to 1 m thick in the Hunyuanyao area is persistent along strike for > 5 km, and is locally mined for graphite (Liu et al, 1989). In the region of northeastern Zhuozi, the khondalite suite is cut by the Altyn fault, a NE-SW trending shear zone. In some areas the khondalite suite is migmatitic, but we avoided sampling in these areas.

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9 3. Sampling and analytical method

10 Khondalite samples were collected from the Jining Complex in 2001 and four samples 11 were selected for this study. They were processed involving crushing and initial heavy 12 liquid and subsequent magnetic separation. Zircons were hand-picked and mounted on 13 adhesive tape, enclosed in epoxy resin and then polished to about half their sizes and 14 photographed in reflected and transmitted light. In order to study the structure and origin of 15 zircons, cathodoluminescence (CL) imaging of zircon grains was taken using a scanning 16 electron microprobe at Guangzhou Institute of Geochemistry, Chinese Academy of 17 Sciences. The U-Pb isotope compositions of zircons were analyzed using a VG PQ Excel 18 ICP-MS equipped with a Nd:YAG 213 laser ablation system (Microprobe2, New Wave 19 Research, U.S.A.), installed in the Department of Earth Sciences, University of Hong Kong. 20 The instrumental settings and detailed analytical procedures have been described by Xia et 21 al. (2004). U-Pb ages of zircons were calculated using the U decay constants of 238 U=1.55125×10⁻¹⁰ y⁻¹, 235 U=9.8454×10⁻¹⁰ y⁻¹ and the Isoplot 3 software (Ludwig, 2003). 22

1 Individual analyses are presented with 1σ error in data tables and in concordia diagrams and 2 uncertainties in age results are quoted at 95% level (2σ). Considering that zircons from the 3 Jining khondalites may have more than one age population, we analyzed at least 50 zircon grains for each sample and ²⁰⁷Pb/²⁰⁶Pb age histograms were also plotted using Isoplot 3 to 4 5 help discuss the main age peaks of the sedimentary provenance.

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4. CL imaging analysis of zircons

8 The CL images show that most zircons have a detrital core and an overgrowth 9 rim (Fig 3). The detrital cores have rounded outlines and are interpreted as a result of 10 erosion during the sedimentary transport and these cores commonly have concentric 11 oscillatory zoning. Because zircons with oscillatory zoning, low to variable luminescence are generally considered to be of magmatic origin (Hancher and Miller, 12 13 1993; Hancher and Rundnick, 1995; Kröner, et al., 2000, Corfu, et al., 2003), we 14 interpret that these zircon cores were derived from igneous rocks. The zircon rims are 15 highly luminescent and structureless. Previous studies showed that metamorphic 16 zircons often occur as multifaceted single grains or overgrowth rims surrounding 17 older zircon cores. They are structureless, highly luminescent, and have low Th/U 18 ratios (e.g. Pidgeon, et al., 2000; Zhao et al., 2002; Corfu et al., 2003). Therefore, we 19 consider that the overgrowth rims on the detrital zircon cores were formed during the 20 high-grade metamorphism.

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1 5. U-Pb zircon analytical results

2 5.1. Sample 01M020

3 This sample is a medium-grained sillimanite-garnet-feldspar gneiss collected 4 from the Manjingou area, about 30 km northeast of Huai'an city (N 40° 22' 41.4"; E 5 114° 28' 10.4", Fig. 2). It comprises of 30% garnet, 40% feldspar, 20% quartz, 5% 6 sillimanite and 5% biotite. Quartz ribbon texture is observed. The sizes of most garnet 7 grains are around 0.3 cm and up to 1 cm in some grains. Most zircons separated from the sample are nearly equigranular and highly rounded, indicating their long distance 8 9 transport. These zircons have zoned cores surrounded by structureless, highly 10 luminescent, overgrowth rims with width of $10 - 30\mu m$ (Figs. 3a, b).

11 Zircon U-Pb isotopic results of this sample are presented in Table 1 and in a concordant diagram (Fig. 4). Of 51 analyses, five data (spots 1-5, Table 1) with a 12 weighted mean 207 Pb/ 206 Pb age of 1811 ± 23 Ma (MSWD=0.03) constitute an isolated 13 14 peak on the histogram (Fig 4). This age is interpreted as the approximate time of 15 metamorphic zircon growth because the analyses were done on the highly luminescent 16 and structureless zircon rims. The rest of 46 data plot on or near the Concordia with 17 an intercept age of 1902 ± 16 Ma (MSWD=0.62) and appear as a unimodal age at about 1890 Ma on the histogram. The ²⁰⁷Pb/²⁰⁶Pb ages of 26 nearly concordant 18 19 data-points (discordance degree $\leq 3\%$; Table 1) range between 1842 and 1960 Ma and all data are $< 1995 \pm 27$ Ma (²⁰⁷Pb/²⁰⁶Pb age, spot 12). Our results clearly indicate 20 21 that detrital zircons from this sample were exclusively derived from a 22 Paleoproterozoic provenance.

1 5.2. Sample 01M038

This sample, also a sillimanite-garnet-feldspar gneiss, was collected from the north of Tian'an Village, about 20 km southwest of Xinghe city (N 40° 34' 24.9"; E 113° 57' 46.3", Fig. 2). Zircon grains in this sample are subhedral to euhedral, mostly with clear oscillatory zoning, evidently derived from an igneous provenance. Highly luminescent, structureless overgrowth rims are absent for most zircons from this sample (Figs. 3c, d).

As shown in Fig. 5, most data for this sample are discordant and scatter below 8 the concordia. Of 50 analyses, the two most concordant data give ²⁰⁷Pb/²⁰⁶Pb ages of 9 10 1857 ± 73 Ma and 2102 ± 38 Ma, with discordancy degree of 1 % and 0.7 % 11 respectively. The rest of 48 analyses show various degrees of discordancy (Fig. 5), 12 probably resulting from Pb loss. Note that most of these discordant data show ages within the range defined by the two concordant data and the oldest data-point gives a 13 207 Pb/ 206 Pb age of 2175 ± 42 Ma. Three main age peaks, 1887 Ma, 1957 Ma and 2031 14 15 Ma, can be recognized in the histogram (Fig. 5).

16 5.3. Sample 01M041

This sillimanite-garnet gneiss is collected from the Xuwujia section, located near the Dapo Village, about 12 km south of Jining city (N40° 43'36.6"; E113° 17'34.6"; Fig. 2). The rock is intensively deformed. Cordierites are developed as dark band, and garnet grains are elongated and rotated with pressure shadows. Zircon grains in this sample are nearly equigranular, mostly rounded and coarser than those from the above two samples. Most grains have oscillatory zoning cores and highly luminescent,
 structureless rims, but rims are too narrow to be analyzed (Figs. 3e, f).

Most data for this sample plot on or near the Concordia (Fig. 6) and give an intercept age of 1945 ± 15 Ma (MSWD = 5.5). Of 54 analytical points, 35 points have a discordancy degree $\leq 3\%$, and they give 207 Pb/ 206 Pb ages ranging from 1858 Ma to 2043 Ma, with a unimodal age population at 1932 Ma on the histogram (Fig. 6). The oldest 207 Pb/ 206 Pb age is 2196 \pm 57 Ma, with a great discordancy degree of 32%.

8 5.4. Sample 01M053

Sample 01M053 is a fine-grained cordierite-garnet-sillimanite gneiss collected
from the Xiaoshan quarry located at about 20 km north of Zhuozi city (N 40° 55′46.5″;
E 112° 21′55.7″, Fig. 2). Cathodoluminescence images reveal that nearly all zircon
grains from this sample are characterized by concentric, oscillatory zoning (Figs. 3g),
showing an igneous origin. Some oscillatory zoned cores are surrounded by highly
luminescent, structureless rims, which are too narrow to be analyzed (Figs. 3h).

On the concordia diagram (Fig. 7), the analytical points yield an intercept age of 16 1975 ± 71 Ma with a very large MSWD of 29. Except one strongly discordant point 17 (spot 25, with a 207 Pb/ 206 Pb age of 2384 \pm 9 Ma, the oldest age in this sample), all 18 other data points cluster on or near the Concordia with two age populations. The 19 bimodality of the age populations at 1894 Ma and 2086 Ma is perceptible from the 18 histogram (Fig. 7). The nearly concordant data-points fall into a range from 1842 Ma 2099 Ma, and the youngest concordant age of 1842 ± 20 is identical to the youngest detrital zircon age obtained from sample 01M020 and thus is referred to as the
 maximum depositional age of the khondalite suites.

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4 6. Discussion

5 U-Pb zircon geochronological data reported in this study, in conjunction with 6 some trace element data published by Condie et al. (1992) and Wan et al., (2000), 7 enable us to place constraints on a number of controversial issues regarding the 8 evolution of the North China Craton (NCC).

9 6.1. Maximum depositional age of the khondalites

10 A key issue regarding the tectonic evolution of NCC is the depositional age of 11 the khondalites. Qian et al. (1985) first proposed that these rocks possibly represent a 12 Neoarchean cover sequence unconformably overlying an orthogneiss basement. Shen et al. (1992), Zhao et al. (1993), Lu et al. (1996), Qian et al. (1996), Li et al. et al. 13 14 (1996) and Wu et al. (1998) supported the hypothesis that these khondalite suites are 15 of Neoarchean age based on the regional geological analysis. However, this idea has 16 not been supported by geochronological data. Previously published conventional 17 multigrain or single grain zircon TIMS data for the khondalites spread between 1733 18 and 2083 Ma (Shen et al., 1987; Guo et al., 1994; Wang., et al., 1995), but these 19 ages were consistently interpreted as "metamorphic" ages (e.g. Lu et al., 1996).

20 More than 200 detrital zircon grains analyzed in this study give only 21 Paleoproterozoic age, mostly < 2.1 Ga, with maximum age of 2384 ± 9 Ma. It is clear 22 that the khondalites were deposited in the Paleoproterozoic, not Archean as previously

1	considered. The minimum age of these detrital zircons is provided by two samples
2	with 1842 ± 16 Ma and 1842 ± 20 Ma. These ages can be used to constrain the
3	maximum depositional age of the khondalites at ~1.84 Ga.

4 6.2. Constraints on the time of the final assembly of the North China Craton

5 Khondalites in the North China Craton were considered to be deposited in a 6 passive margin based on major and trace element geochemistry (Condie et al., 1992; 7 Wan et al., 2000). These metasedimentary rocks underwent high-grade metamorphism following a clock-wise P-T path and involving a near-isothermal decompression 8 9 (Zhao et al., 1999a). There is a consensus on the high-grade metamorphism to be 10 related to the collision between the Western and Eastern Blocks but controversy exists on the timing of the collision. One school argues that it occurred at \sim 2.5 Ga (e.g. Li et 11 12 al., 2000; Kusky and Li, 2003); whereas the other suggests a later time at ~1.8 Ga (e.g. 13 Zhao, 2001; Wilde et al., 2002). Because our new zircon U-Pb isotopic data constrain 14 the maximum depositional age of the khondalites at about 1.84 Ga, the late Archean 15 collision is thus impossible. On the other hand, the data obtained from the zircon rims 16 clearly indicate that the granulite facies metamorphism of the khondalites and hence 17 the collision between the two blocks must have taken place at about 1.81 Ga.

18 6.3. Constraints on the sedimentary provenance of the Jining khondalites

Based on the trace element data, Condie et al. (1992) suggested that the sediment protoliths of Jining khondalites could be derived from mixing of dominantly granite sources with minor basaltic contributions. They precluded Archean TTG and Archean

komatiites as important contributors but a question remains concerning the source of
 these sediments.

3 More than 200 detrital zircon grains analyzed in this study yielded nearly 4 concordant ages ranging from 2102 ± 38 Ma to 1842 ± 16 Ma, which can be largely 5 grouped into three age populations: ~1890 Ma, ~1940 Ma and ~2060 Ma in the 6 histogram. This age pattern is in strong contrast to that of the Eastern Block, which 7 has a dominantly Archean basement. It is also different from that of the Yinshan 8 Terrane in the northern part of the Western Block. The Eastern Block is known to 9 have basements ranging from the early Archean (up to ~3.8 Ga, Liu et al., 1992; Song 10 et al., 1996), through middle Archean (3.4 - 2.9 Ga, e.g. Jahn et al., 1987; Song et al., 11 1996), to late Archean (2.9 – 2.5 Ga, e.g. Wu et al., 1991; Kröner et al., 1998) and late 12 Archean lithotectonic assemblages are exposed in the Yinshan Terrane (e.g. Wang, 1993; also see the review by Zhao et al., 2001). The high-Al characteristics of the 13 14 khondalites and rounded morphology of the detrital zircons required a long 15 transportation history and hence sampled a large area, thus, the age pattern of detrital 16 zircons should reflect, to some extent, the age frame of their provenance. At present, 17 no single Archean detrital zircon has been identified in this study, thus the Eastern 18 Block and the Yinshan Terrane are unlikely to be the provenance of these khondalites. 19 The southern part of the Western Block, namely the Ordos Terrane, is covered by the 20 thick Mesozoic to Cenozoic Ordos basin sediments. Its basement rocks are not 21 exposed and their nature remains largely unknown. Sporadic drillings (Wu et al., 1986) 22 and some aeromagnetic data (Wu et al., 1998) imply the existence of granulite-faces

basement beneath the Ordos basin. The khondalites in the NCC mainly occur
surrounding the borderland of the Ordos Terrane, and this terrane is considered to be
the provenance of the khondalites (e.g. Zhao et al., 2003; 2005). If this is the case, our
data may imply that the Ordos Terrane may have a different history from the Eastern
Block.

6

7 7. Conclusions

8 U-Pb detrital zircon data presented in this study show that the Jining khondalites 9 came from a Paleoproterozoic sedimentary provenance, not an Archean source as 10 previously considered. The Jining khondalites were initially deposited at 1.84 Ga or 11 later and underwent granulite-facies metamorphism at ~1.81 Ga. The detrital zircon 12 age pattern indicates that the sediments were derived from a provenance different 13 from the Eastern Block and the Yinshan Terrane of the NCC. These new age data are 14 consistent with the recent three-fold tectonic subdivision of the NCC and support that 15 the Eastern and Western Blocks collided at ~ 1.8 Ga, but not ~ 2.5 Ga as previously 16 considered, to form the coherent NCC.

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1	Figure Captions
2	
3	Fig 1. Spatial distribution of the khnodalites in the North China Craton (modified
4	from Zhao et al., 1999a).
5	
6	Fig 2. Generalized geological map of basement rocks in the Jining area (after Qian et
7	al., 1999).
8	
9	Fig. 3. Representative cathodoluminescence images for zircons from the khondalites.
10	Descriptions of zircons are included in the text.
11	
12	Fig. 4. Concordia diagram of LA-ICPMS U-Pb zircon analytical results for sample
13	01M020. The inset is a histogram for the distribution of 207 Pb/ 206 Pb ages. Also shown
14	is a sum curve (solid line) modeled by the Gauss distribution.
15	
16	Fig. 5. Concordia diagram of LA-ICPMS U-Pb zircon analytical results for sample
17	01M038. The inset is histogram for the distribution of 207 Pb/ 206 Pb ages. Also shown is
18	a sum curve (solid line) modeled by the Gauss distribution.
19	
20	Fig. 6. Concordia diagram of LA-ICPMS U-Pb zircon analytical results for sample
21	01M041. The inset is a histogram for the distribution of 207 Pb/ 206 Pb ages. Also shown
22	is a sum curve (solid line) modeled by the Gauss distribution.
23	
24	Fig. 7. Concordia diagram of LA-ICPMS U-Pb zircon analytical results for sample
25	01M053. The inset is a histogram for the distribution of 207 Pb/ 206 Pb ages. Also shown
26	is a sum curve (solid line) modeled by the Gauss distribution.