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Title	LA-ICP-MS U-Pb geochronology of detrital zircons from the Jining Complex, North China Craton and its tectonic significance
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2	North China	Craton a	and its	tectonic	significance

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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 Terrane of the NCC. The nearly concordant youngest age of 1842 ± 16 Ma 14 (207Pb/206Pb age) for the detrital zircons is interpreted as the maximum depositional 15 16 age of the khondalites. Overgrowth rims of detrital zircons yield an age of 1811 ± 23 Ma, which we interpret as the metamorphic age. The new age data are consistent with 17 18 the recent three-fold tectonic subdivision of the NCC and support that the Eastern and

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

Western Blocks collided at ~1.8 Ga to form the coherent NCC.

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1. Introdution

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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 reseachers in China (e.g. Lu et al., 1996). However, numerous geochronological studies on high-grade metamorphic terrains around the world have shown that igneous zircons can survive high-grade metamorphism (e.g. Vavra et al., 1996, 1999; Kröner et al., 2000; Zhao et al., 2002). Because these metasedimentary rocks were deposited on the continental margin of the Western Block (Condie et al., 1992) prior to its collision with the Eastern Block, reliable age data must be obtained in order to constrain the nature of the Western Block and the inter-block collision. For this reason, we undertook a detailed zircon geochronology to determine the depositional and metamorphic ages of the khondalites in the NCC. Whole-grain zircon analysis may result in ambiguous data since metamorphic overgrowths on zircon rims are very common in high-grade metamorphic rocks. It has been recently demonstrated that cathodoluminescence (CL) and back-scattered electron (BSE) imaging can be used to reflect internal structure of zircon (e.g. Vavra et al., 1996, 1999; Kröner et al., 2000; Zhao et al., 2002; Guan et al., 2002), which, in turn, provides important information on the origin of zircons (e.g. magmatic, metamorphic, detrital, xenocrystic, etc). Previous studies on high-grade metamorphic rocks showed that metamorphic zircons occur as either multifaceted single grains or overgrowth rims, which are structureless and highly luminescent on CL images, whereas magmatic zircons are generally euhedral with oscillatory zoning and show low to variable luminescence (e.g. Zhao et al., 2002; Corfu et al., 2003). Spot analysis of U-Pb isotope compositions on (part of) zircons can yield the protolith and

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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.

2. Geological setting

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 khondalite suites (Lu et al., 1996). The former suite consists predominantly of granulite-facies metamorphic TTG gneisses and minor enderbites and charnockites with metamorphic P-T conditions at 8.0-10.0 kbar and 750-800°C (Lu and Jin, 1993; Zhao et al., 1999a). The dominant rock types in the khondalite suite are sillimanite-garnet gneiss (SGG), quartz-garnet gneiss (QGG) and quartz-feldspar gneiss (QFG), roughly in a proportion of 5:3:2 (Liu, 1989). The SGG units are up to 100 m thick and are commonly layered on a scale of a few centimeters. The QGG units occur as massive units up to a few meters thick (most are <1 m thick) interlayered with the SGG and can often be traced along the strike for several kilometers. The QFG occurs chiefly as concordant units within the SGG and QGG 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.

3. Sampling and analytical method

Khondalite samples were collected from the Jining Complex in 2001 and four samples were selected for this study. They were processed involving crushing and initial heavy liquid and subsequent magnetic separation. Zircons were hand-picked and mounted on adhesive tape, enclosed in epoxy resin and then polished to about half their sizes and photographed in reflected and transmitted light. In order to study the structure and origin of zircons, cathodoluminescence (CL) imaging of zircon grains was taken using a scanning electron microprobe at Guangzhou Institute of Geochemistry, Chinese Academy of Sciences. The U-Pb isotope compositions of zircons were analyzed using a VG PQ Excel ICP-MS equipped with a Nd:YAG 213 laser ablation system (Microprobe2, New Wave Research, U.S.A.), installed in the Department of Earth Sciences, University of Hong Kong. The instrumental settings and detailed analytical procedures have been described by Xia et 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).

- 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
- 4 grains for each sample and ²⁰⁷Pb/²⁰⁶Pb age histograms were also plotted using Isoplot 3 to
- 5 help discuss the main age peaks of the sedimentary provenance.

4. CL imaging analysis of zircons

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

5. U-Pb zircon analytical results

2 5.1. Sample 01M020

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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 – 30µm (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 $^{207}\text{Pb}/^{206}\text{Pb}$ 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 ≤ 3%; Table 1) range between 1842 and 1960 Ma and all data are $< 1995 \pm 27$ Ma (207 Pb/ 206 Pb age, spot 12). Our results clearly indicate 20 21 that detrital zircons from this sample were exclusively derived from a 22 Paleoproterozoic provenance.

This sample is a medium-grained sillimanite-garnet-feldspar gneiss collected

5.2. Sample 01M038

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2 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 3 4 113° 57′ 46.3″, Fig. 2). Zircon grains in this sample are subhedral to euhedral, mostly 5 with clear oscillatory zoning, evidently derived from an igneous provenance. Highly 6 luminescent, structureless overgrowth rims are absent for most zircons from this 7 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 17 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"; 18 19 Fig. 2). The rock is intensively deformed. Cordierites are developed as dark band, and 20 garnet grains are elongated and rotated with pressure shadows. Zircon grains in this 21 sample are nearly equigranular, mostly rounded and coarser than those from the above

- 1 two samples. Most grains have oscillatory zoning cores and highly luminescent,
- 2 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
- 4 intercept age of 1945 ± 15 Ma (MSWD = 5.5). Of 54 analytical points, 35 points have
- 5 a discordancy degree \leq 3%, and they give 207 Pb/ 206 Pb ages ranging from 1858 Ma to
- 6 2043 Ma, with a unimodal age population at 1932 Ma on the histogram (Fig. 6). The
- 7 oldest 207 Pb/ 206 Pb age is 2196 ± 57 Ma, with a great discordancy degree of 32%.
- 8 5.4. Sample 01M053
- 9 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″;
- 11 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
- other data points cluster on or near the Concordia with two age populations. The
- bimodality of the age populations at 1894 Ma and 2086 Ma is perceptible from the
- 20 histogram (Fig. 7). The nearly concordant data-points fall into a range from 1842 Ma
- 21 to 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
- 2 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
- A key issue regarding the tectonic evolution of NCC is the depositional age of
- the khondalites. Qian et al. (1985) first proposed that these rocks possibly represent a
- 12 Neoarchean cover sequence unconformably overlying an orthogneiss basement. Shen
- 13 et al. (1992), Zhao et al. (1993), Lu et al. (1996), Qian et al. (1996), Li et al. et al.
- 14 (1996) and Wu et al. (1998) supported the hypothesis that these khondalite suites are
- of Neoarchean age based on the regional geological analysis. However, this idea has
- 16 not been supported by geochronological data. Previously published conventional
- 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
- 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
- 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 \sim 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;
- Wan et al., 2000). These metasedimentary rocks underwent high-grade metamorphism
- 8 following a clock-wise P-T path and involving a near-isothermal decompression
- 9 (Zhao et al., 1999a). There is a consensus on the high-grade metamorphism to be
- 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
- al., 2000; Kusky and Li, 2003); whereas the other suggests a later time at ~1.8 Ga (e.g.
- 213 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
- 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
- 20 protoliths of Jining khondalites could be derived from mixing of dominantly granite
- 21 sources with minor basaltic contributions. They precluded Archean TTG and Archean

1 komatiites as important contributors but a question remains concerning the source of 2 these sediments.

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

1 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

5 Block.

7. Conclusions

U-Pb detrital zircon data presented in this study show that the Jining khondalites came from a Paleoproterozoic sedimentary provenance, not an Archean source as previously considered. The Jining khondalites were initially deposited at 1.84 Ga or later and underwent granulite-facies metamorphism at ~1.81 Ga. The detrital zircon age pattern indicates that the sediments were derived from a provenance different from the Eastern Block and the Yinshan Terrane of the NCC. These new age data are consistent with the recent three-fold tectonic subdivision of the NCC and support that the Eastern and Western Blocks collided at ~1.8 Ga, but not ~2.5 Ga as previously 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 01M020. The inset is a histogram for the distribution of ²⁰⁷Pb/²⁰⁶Pb ages. Also shown 13 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 01M038. The inset is histogram for the distribution of $^{207}\text{Pb}/^{206}\text{Pb}$ ages. Also shown is 17 18 a sum curve (solid line) modeled by the Gauss distribution. 19

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Fig. 6. Concordia diagram of LA-ICPMS U-Pb zircon analytical results for sample

01M041. The inset is a histogram for the distribution of ²⁰⁷Pb/²⁰⁶Pb ages. Also shown 21

is a sum curve (solid line) modeled by the Gauss distribution.

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24 Fig. 7. Concordia diagram of LA-ICPMS U-Pb zircon analytical results for sample

01M053. The inset is a histogram for the distribution of ²⁰⁷Pb/²⁰⁶Pb ages. Also shown 25

26 is a sum curve (solid line) modeled by the Gauss distribution.

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